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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 20_8260B
VOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS):
CAPILLARY COLUMN TECHNIQUE
(EPA Method 624/SW-846 METHOD 8260B)

APROVED BY:

	Date
Operations Manager	
	3/20/09
QA Manager	Date
	3/20/09
Laboratory Director	Date

MASTER

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Volatile Organic Compounds - by GC/MS Capillary Column Technique

1.0 SCOPE AND APPLICATION

1.1 This method describes the technique utilized by ESS Laboratory in the qualitative and quantitative analysis of volatile samples based on SW-846 Method 8260B. Following are compounds that may be determined by this method.

Appropriate Technique Analyte	Cas # ^b	Purge-and-Trap	Direct Injection
Acetone	67-64-1	Pp	a
Acrolein (Propenal)	107-02-8	Pp	a
Acrylonitrile	107-13-1	Pp	a
Allyl chloride	107-05-1	a	a
tert- Amyl methyl ether	994-05-8	a	a
Benzene	71-43-2	a	a
Bromobenzene	108-86-1	a	a
Bromochloromethane	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
4-Bromofluorobenzene	460-00-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
2-Butanone (MEK)	78-93-3	Pp	a
n-Butylbenzene	104-51-8	a	a
sec-Butylbenzene	135-98-8	a	a
tert-Butylbenzene	98-06-6	a	a
Carbon disulfide	75-15-0	Pp	a
Carbon tetrachloride	56-23-5	a	a
Chlorobenzene	108-90-7	a	a
1-Chlorobutane	109-69-3	a	a
Chlorodibromomethane	124-48-1	a	a
Chloroethane	75-00-3	a	a
2-Chloroethyl vinyl ether	110-75-8	a	a
Chloroform	67-66-3	a	a
Chloromethane	74-87-3	a	a

Chloroprene	126-99-8	a	a
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Appropriate Technique Analyte	Cas # ^b	Purge-and-Trap	Direct Injection
2-Chlorotoluene	95-49-8	a	a
4-Chlorotoluene	106-43-4	a	a
Cyclohexane	110-82-7	a	a
1,2-Dibromo-3-chloropropane	96-12-8	Pp	a
1,2-Dibromoethane	106-93-4	a	a
Dibromomethane	74-95-3	a	a
1,2-Dichlorobenzene	95-50-1	a	a
1,3-Dichlorobenzene	541-73-1	a	a
1,4-Dichlorobenzene	106-46-7	a	a
Cis-1,4-Dichloro-2-butene	1476-11-5	a	a
trans-1,4-Dichloro-2-butene	110-57-6	Pp	a
Dichlorodifluoromethane	75-71-8	a	a
1,1-Dichloroethane	75-34-3	a	a
1,2-Dichloroethane	107-06-2	a	a
1,1-Dichloroethene	75-35-4	a	a
cis-1,2-Dichloroethene	156-59-2	a	a
trans-1,2-Dichloroethene	156-60-5	a	a
1,2-Dichloropropane	78-87-5	a	a
1,3-Dichloropropane	142-28-9	a	a
2,2-Dichloropropane	594-20-7	a	a
1,1-Dichloropropene	563-58-6	a	a
cis-1,3-Dichloropropene	10061-01-5	a	a
trans-1,3-Dichloropropene	10061-02-6	a	a
1,4-Difluorobenzene	540-36-3	a	a
Diethyl Ether	60-29-7	a	a
1,4-Dioxane	123-91-1	Pp	a
Ethylbenzene	100-41-4	a	A
Ethyl methacrylate	97-63-2	a	a
Ethyl tertiary-butyl ether	637-92-3	a	a
Hexachlorobutadiene	87-68-3	a	a
Hexachloroethane	67-72-1	i	a
2-Hexanone	591-78-6	Pp	a

Appropriate Technique Analyte	Cas # ^b	Purge-and-Trap	Direct Injection
Iodomethane	74-88-4	a	a
Isopropylbenzene	98-82-8	a	a
Di-isopropyl ether	108-20-3	a	a
p-Isopropyltoluene	99-87-6	a	a
Methyl acetate	79-20-9	a	a
Methyl acrylate	96-33-3	a	a
Methylacrylonitrile	126-98-7	pp	a
Methyl Cyclohexane	108-87-2	a	a
Methylene chloride	75-09-2	a	a
Methyl methacrylate	80-62-6	a	a
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	a
Naphthalene	91-20-3	a	a
2-Nitropropane	79-46-9	a	a
Pentachloroethane	76-01-7	i	a
n-Propylbenzene	103-65-1	a	a
Styrene	100-42-5	a	a
1,1,1,2-Tetrachloroethane	630-20-6	a	a
1,1,2,2-Tetrachloroethane	79-34-5	a	a
Tetrachloroethene	127-18-4	a	a
Toluene	108-88-3	a	a
1,2,3-Trichlorobenzene	87-61-6	a	a
1,2,4-Trichlorobenzene	120-82-1	a	a
1,3,5-Trichlorobenzene	108-70-3	a	a
1,1,1-Trichloroethane	71-55-6	a	a
1,1,2-Trichloroethane	79-00-5	a	a
Trichloroethene	79-01-6	a	a
Trichlorofluoromethane	75-69-4	a	a
1,2,3-Trichloropropane	96-18-4	a	a
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	a	a
1,2,4-Trimethylbenzene	95-63-6	a	a
1,3,5-Trimethylbenzene	108-67-8	a	a
Vinyl acetate	108-05-4	a	a
Vinyl chloride	75-01-4	a	a
o-Xylene	95-47-6	a	a
m-Xylene	108-38-3	a	a

p-Xylene	106-42-3	a	a
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- a Adequate response by this technique.
b Chemical Abstract Services Registry Number.
i Inappropriate technique for this method
pp Poor purging efficiency results in high MDLs.
- 1.2 This method is used to determine volatile organic compounds in a variety of waste matrices. This method is applicable to nearly all types of samples, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, tars, fibrous wastes, emulsions, filter cakes, soils and sediments.
- 1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds quantitation limits are approximately ten times higher because of poor purging efficiency.
- 1.4 The quantitation limit of this method for an individual compound is approximately 1µg/L for all aqueous matrices. The Quantitation limit for soil/sediment samples is listed in SOP 20_5035.

2.0 METHOD SUMMARY

- 2.1 This SOP provides gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of volatile organic compounds.
- 2.2 This SOP describes a closed system purge and trap procedure for aqueous samples according to method 5030B.
- 2.2.1 Purged sample components are trapped in a tube containing suitable sorbent materials.
- 2.2.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly to a wide bore capillary column for analysis.
- 2.2.3 Wide-bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.
- 2.3 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS).
- 2.4 Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Absolute identifications are obtained by comparing the mass spectra of individual compounds to the reference spectra for that compound.
- 2.5 Concentrations of the identified components are measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

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 90_0001 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 MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

- 4.1 Sample preservation for soil samples is discussed in SOP 20_5035. Aqueous samples are preserved with 1:1 HCl at a ratio of 100 µL to 40 ml.
- 4.2 Samples for analysis using this SOP must be stored in tightly sealed vials with Teflon-lined silicone septum seals in which the original sample was collected. All samples are stored in a refrigerator at $4 \pm 2^{\circ}\text{C}$ within the laboratory. The soil samples are stored in a separate refrigerator from the aqueous samples in the VOC laboratory. Samples for volatile analysis must be analyzed within 14 days of sample collection
- 4.3 Samples known to contain high concentrations of VOCs are stored in a separate refrigerator located in the sample storage room. This is to prevent cross contamination.
- 4.4 Trip blanks for aqueous samples are prepared with organic free water. The water comes from a carbon filtration system located in the VOA lab and is periodically checked for low level VOCs.
- 4.5 A trip blank should accompany each batch of 20 or less VOC samples. This trip blank must accompany the associated samples at all times.
- 4.6 Refrigerator blanks are prepared like trip blanks and stored in the refrigerator with samples. These blanks are analyzed weekly to check for possible cross contamination from samples. To prevent cross contamination from sample to sample, all sample containers must be airtight. If the blank is greater than the MRL, then notify operations manager immediately.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

- 5.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealant, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.
- 5.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source. Subtracting blank values from sample results is not permitted.
- 5.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, one or more instrument blanks must be analyzed to check for cross contamination.
 - 5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.
 - 5.3.2 In extreme situations, the whole purge and trap device may require dismantling and cleaning.
- 5.4 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.
- 5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.
- 5.6 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight-halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

6.0 EQUIPMENT/APPARATUS

- 6.1 **Microsyringes:** 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL. These syringes should be equipped with a 20 gauge (0.006" ID) needle.
- 6.2 **Disposable micropipettes:** 10, 50, 100 and 200 µL volumes. Purchased from Drummond.
- 6.3 **Syringe:** 5 ml and 25 ml glass, gas-tight, with Luerlock tip.
- 6.4 **Balance:** Top-loading balance capable of weighing 0.1 g.
- 6.5 **Micro-reaction vessels:** 1.0 ml and 5.0 ml purchased from Supelco. Catalogue No. 3-3293 with Mininert Caps. Clean reaction vessels by placing in oven at 105°C overnight.
- 6.6 **Volumetric flasks:** of various sizes with ground glass stoppers –Class A.
- 6.7 **Vials:** 40 ml, with pierceable Teflon screw cap top.
- 6.8 **Spatulas:** Stainless steel.
- 6.9 **Disposable pipettes:** Pasteur.
- 6.10 **Culture tubes:** 10 x 150 mm. Kimball No. 45060-19150. Culture tubes are only used once, then discarded.
- 6.11 **Syringe Valve:** Two-way with Luerlok ends.
- 6.12 **Chamber heaters:** Tekmar sample heater, capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.
- 6.13 **Purge and trap devices:** (A) Tekmar 3100 Purge and Trap Concentrator. (B) Archon Purge and Trap Auto-Sampler System (Varian)
- 6.13.1 The Archon Purge and Trap Auto-sampler is designed to Automate the tedious sample handling procedures associated with purge and trap analyses for volatile organic compounds (VOCs) under current EPA methods. The Archon can be used for drinking water, wastewater, soil and solid analyses.

6.13.2 Function Keys:

- A) Method: Methods are entered using a keypad with LCD screen. All information can be edited using keypad. ***NOTE: Settings are variable and may change depending on analysis requirements.***

Method parameters consist of:

MS1/MS3/MS4

Sample Type (water/soil)

water

First Vial(1-51)

variable

Last Vial(1-51)	variable
Sample Volume(0-25ml)	10
Dilution Factor	variable
Rinse Volume(0-25ml)	12
# Rinses(0-20)	01
Standard 1(yes/no)	variable
Standard 2(yes/no)	variable
S. Preheat Stir(yes/no)	no
Stir(yes/no)	no
W. Stir Time(0-9.9min)	0.0
W. Settle Time(0-99sec)	0.0
Syringe Flush(0-20)	01
Preheat(yes/no)	no
Preheat Temp(amb-140C)	--
Preheat Time(0-99.9min)	--
Purge Time(0-999.9min)	--
Desorb Time(0-99.9min)	1.0
Operation Mode(Local/Remote)	Remote
Cycle Timer(0-99.9min)	0.0
Aux Timer(0-999min)	0.0
Link to Method	variable

B) **Auto:** Starts purge sequence. Initiates single blank runs. Single blank runs can be programmed after specific sample vials within a particular method. These method blanks allow the cleaning of not only the Archon, but also the purge and trap and GC. The blank water is withdrawn from the blank water reservoir, which is pressurized by helium gas at 20-25 psi. Before changing the blank reservoir, the "Blank Water Reservoir and Standard Helium" switch must turned off. This switch is located on the right rear panel inside the Archon. After refilling the reservoir, this switch must be turned on.

C) **Manual:** allows for manual injection of samples.

D) **Flush:** allows flushing of Archon components using water from Blank Water Reservoir

E) **System:** provides access to all settings, maintenance, calibration, and options of the Archon.

F) **Pause/Stop:** allows analyst to pause or abort sequence.

6.13.3 The sample is purged in a 25ml fitted purge tube. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas is introduced no more than 5 mm from the base of the water column.

6.13.4 The trap is at least 25 cm long and has an inside diameter of at least 0.105". This trap is commercially available from Supelco (Vocarb 3000, Catalogue

No. 2-4920).

- 6.13.5 The desorber is capable of rapidly heating the trap to 225°C and performing the bake out step at 260°C.
- 6.13.6 The chamber heater is capable of maintaining the purge device at 40°C ±1°C.
- 6.14 **Gas chromatograph:** HP5890 Series II or HP-6890. A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns and gases.
- 6.15 **Column:** (A). 75 M x 0.53 mm ID or (B). 30M x 0.25 mm ID, DB624 commercially available from J&W Scientific (or equivalent).
- 6.16 **Mass spectrometer:** HP5971MSD or HP5972MSD - Capable of scanning from 35 to 260 AMU every 1 second or less, utilizing 70 volts electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria listed in section 11.1 when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.
- 6.17 **Data system:**
- 6.17.1 **Computers:** The Volatiles laboratory has three GC/MS systems analyzing method 5030B/8260B. VOA MS1 has an AST computer with a Windows 95 operating system. VOA MS3 and MS4 have DELL computers with Windows NT 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.
- 6.17.2 **Software:** HP Enviroquant MSChemstation - The software is interfaced to the mass spectrometer detector and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. The most current version of the EPA/NIST Mass Spectral Library is also available. Current versions VOA MS1:G1032C version C.01.00, VOA MS3:G1701BA Version B.01.00, and VOA MS4: G1701AA version A.03.00.

7.0 REAGENTS AND STANDARDS

7.1 Reagents:

7.1.1 **Methanol:** Purge and Trap grade, purchased from Fisher, Catalog No.A453-500.

7.1.2 **Reagent Water:** Water in which interference is not observed at ½ the method reporting limit (MRL) of the parameters of interest. DI water obtained from the VOA laboratory fits the criterion.

7.2 Standards:

7.2.1 **Primary Standards:** obtained from a commercial source. They are stored in the freezer at -10°C to -20°C. After opening; they are stored in a vial with a mininert valve. All Certificates of Analysis (COAs) are marked with the appropriate Primary Standard ID and placed in a logbook. Primary standards are not to be used after the manufacturer's expiration date. Once a standard has been opened, it may not be used after 6 months. A copy of the COAs, which details the compounds found in the mixes, are in Attachment A.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Restek	Ketones	30006	5000
Restek	Acrolein	30499	10000
Restek	Oxygenates	30465	2000/10,000 (TBA)
Restek	Custom	558360	2000
Restek	Gases	30042-510	2000
Restek	Cal Mix#1	30633	2000
Restek	2-CEVE	30265	2000
Restek	Vinyl Acetate	30216	2000
Supelco	1,4-Dioxane	44-2251	10,000*
Ultra	Surrogate	STM-530-1	2500
Accustandard	1,3,5-Trichlorobenzene	As-E0176	5000

*The primary standard for 1,4-Dioxane is made up from neat by diluting 10 µL of neat 1,4-Dioxane into a 1.0 ml volumetric flask

7.2.2 **Working Calibration Standard (25 µg/ml):** Prepared standards are stored in micro reaction vessels or vials capped with mininert valve. Standards are checked frequently for signs of degradation or evaporation. Working calibration standards are used for preparing calibration curves and daily continuing calibrations.

7.2.2.1 Into 5ml volumetric flask add the following: 125 µL of Ketone std., 62.5 µL each of gas, Custom std., Cal Mix#1, Vinyl acetate, and oxygenate standards, 312.5 µL of 2-CEVE std., 12.5µL of Acrolein std, 25µL of 1,3,5-Trichlorobenzene, 237.5 µL 1,4-Dioxane and 50 µL of surrogate standard. Volumize to 5ml with MeOH. Final concentration equals 125 µg/ml for the Ketones and 2-CEVE, 500 µg/ml of 1,4-Dioxane, and 25 µg/ml for all other analytes including

surrogates.

- 7.2.3 **Secondary Source/BS/Matrix spike standards:** obtained from a commercial source. They are stored in the freezer at -10°C to -20°C. After opening; they are stored in a vial with a mininert valve. All Certificates of Analysis must be kept on file.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Accustandard	TCL Ketone Mix	CLP-022K-25x	5000
Ultra	Acrolein	RCC-150	10,000*
Accustandard	Method 502.2 VOC	M-502-10x	2000
Ultra	VOC Mix	DWM-592-1	2000
Ultra	Custom Std	CUS-10604	2000
Accustandard	2-CEVE	M-601C-10x	2000
Supelco	Vinyl acetate	4-0327	5000
Ultra	1,4-Dioxane	RCC-180	10,000*
Accustandard	tert-Butyl Alcohol	S-410	2000

*The primary standard for 1,4-Dioxane and Acrolein is made from neat product by diluting 10 µL into a 1.0 ml volumetric flask.

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- 7.2.3.1 **Initial Calibration Verification- ICV/BS/BSD/Matrix spike standard (25 µg/ml):** Used to check the primary standard. Into 5ml volumetric flask containing approximately 3 ml of MeOH add 62.5 µL each of Custom std., VOC mix , 502.2 mix, 2-CEVE std., and 25 µL of Vinyl acetate. Add 12.5 µL of Acrolein, 250 µL of 1,4-Dioxane, 100 µL TCL Ketone Mix, and 312.5 µL tert-Butyl Alcohol. Volumize to 5ml with MeOH. Individual components will have a final concentration of 25 µg/ml with the exception of 1,4-Dioxane at 500µg/ml. A copy of the COAs, which details the compounds in the mixes, is attached.

- 7.2.4 **Surrogate Standards:** The surrogate solution is made up of Dibromofluoromethane, Toluene-d₈, 4- Bromofluorobenzene, and 1,2-Dichloroethane-d₄. The mixed stock surrogate solution is purchased from Ultra at 2500 µg/ml (cat no. STM-530-1). The stock solution is logged into the primary standard logbook upon receipt. Several surrogate standard solutions are prepared from the stock at different concentrations in methanol.

- 7.2.4.1 **Surrogate mix:** For soil/sediment samples, the preparation of this solution is discussed in SOP# 20_5035.

- 7.2.4.2 **Surrogate/Internal standard QC mix MS1, MS3, and MS4:** To a 5 ml volumetric flask containing approximately 3 ml of methanol add 500 µL of stock surrogate and stock internal standard. Volumize with methanol to give a final concentration of 250 µg/ml. Transfer to reservoir #1 on the Archon. Addition of 1uL of this standard by the Archon to 10.0 ml of sample is equivalent to 25 ppb.

- 7.2.5 **Internal Standards:** The internal standard compounds are Fluorobenzene,

Chlorobenzene-d₅, and 1,4-Dichlorobenzene-d₄. The mixed internal standard solution is purchased from Ultra at 2,500 µg/ml (cat no. STM-520-1). See SOP 20_5035 for internal standard solution for soil calibration.

7.2.5.1 Internal Standard for calibration: Prepare by addition of 50 uL of Internal Standard solution into a final volume of 5 ml MeOH, final concentration is 25 ug/ml.

7.2.6 Continuing Calibration Verification Standard (CCV): MS1, MS3, and MS4: Addition of 50 uL of the working calibration standard (See 7.2.2.1) to 50.0 ml of organic free water is equivalent to 25 ppb. This solution is prepared fresh daily.

7.2.7 Blank Spike/Blank spike duplicates (BS/BSD): Prepared by adding 40 uL of Blank spike solution (7.2.3.1) to 100 ml DI water in a 100 ml volumetric flask for a final concentration of 10 ug/L. Blank spike concentrations are to be at the midpoint (median standard level) of the calibration curve.

7.2.8 4-Bromofluorobenzene (BFB) Standard: A stock solution in methanol is purchased from Supelco at a concentration of 2,000 µg/ml (cat. no. 4-8083).

7.2.8.1 Add 62.5µL of the stock solution to a 5.0-ml volumetric flask and dilute with methanol to produce a 25-µg/ml solution. A 5 ug/L standard is purged to check the instrument tune.

7.2.9 Matrix Spiking Standard: prepared as in section 7.2.3.1

7.2.9.1 Working Matrix Spike Solution: Prepare aqueous matrix spikes by adding 40 uL of matrix spike solution to 100 ml sample in a 100 ml volumetric flask for a final concentration of 10 ug/L.

8.0 PROCEDURE

8.1 Initial calibration for purge-and-trap procedures:

8.1.1 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.

8.1.2 GC/MS operating conditions: The following parameters are setup through Hewlett Packard MS EnviroQuant. A copy of an EnviroQuant method is in Attachment B.

Electron energy:	70 volts
Mass range:	35 amu - 260 amu
Scan time:	1.5 sec/scan
Initial column temp.:	40°C
Initial column hold time:	4.0 min.
Column temp. program:	Rate Final Temp Final Time

Level 1	8°C/Min	150°C	2 min
Level 2	35°C/Min	220°C	0-4.25 min
Injector temperature:	200°C		
Transfer line temperature:	280°C		
Carrier gas (Helium):	15 cm ³ /ml		

- 8.1.3 Assemble a purge-and-trap device that meets the specification in Section 6.1.3. New traps are conditioned one hour at 270°C in the bake mode. Prior to daily use bake trap for 8 minutes at 260°C
- 8.1.4 Just prior to any analysis of samples or standards, the run logbook should be completely filled out (See Attachment C). Each instrument has its own run logbook. Any unusual observations are included in the comment section of the logbook. The information that must be recorded is:
- 8.1.4.1 **Batch Date:** Date analytical sequence set-up
- 8.1.4.2 **Vial #:** Position the standard/sample vial is placed in the auto-sampler
- 8.1.4.3 **File #:** File ID assigned to standard/sample in sequence
- 8.1.4.4 **Lab ID:** The ID of the sample or standard. Sample IDs are assigned at log-in by ELEMENT (ex: 0508001) and Batch QC samples contain the batch ID (ex BG50801-blk1, BG50801-bs1, BG50801-bsd1, ...)
- 8.1.4.5 **Method:** Record the method used. The method includes instrument operating parameters and initial calibration for quantitation.
- 8.1.4.6 **Comments/Dilution/Standard ID:** Record comments and unusual observations. If a sample is run at a dilution, then record the dilution information in the comments section. (Ex. 25x dilution on aqueous sample: 0.2/5 ml) Record standard/spike ID for Tune, CCV, blank spike/blank spike duplicates, and matrix spike/matrix spike duplicates.
- 8.1.4.7 **Analyst:** Analyst must record their initials.
- 8.1.5 A tune check must be performed before the initial calibration curve. This is accomplished by purging a 5 ug/L standard of the 4-Bromofluorobenzene standard (See section 7.2.7) onto the GC/MS column. Analysis is not to begin until the 4-BFB spectra meets the criteria in Section 11.1. Evaluation of the ion abundance is performed by using one of the following scan scenarios:
- Use one scan at the peak apex,
 - Use the one scan either directly preceding or following the apex,
 - Use the mean of the apex and the scans preceding and following the scan, or
 - Use the average across the entire peak.

8.1.5.1 The analysis of all calibration standards must be accomplished within 12 hours of this tune check.

8.1.6 Prepare the standards for generating the calibration curve using the Working Standards (see section 7.2.2) and Internal Standard solution (see Section 7.2.4.1) as follows:

8.1.6.1 **Soil Method:** The calibration curve for soil/sediment samples is discussed in SOP 20_5035.

8.1.6.2 **Water Method for MS1, MS3, and MS4:** seven standards (0.4, 1.0, 5.0, 10, 25, 50, and 100 ppb) are run. The 5 ppb is prepared in a 50ml volumetric by adding 10 μ L of 25 ug/ml working calibration standard (7.2.2) to 50 ml of DI water. 50 ul of internal standard mix (7.2.5.1) is then added for a final concentration of 25 ppb. The flask is inverted three times and the solution is transferred to a 40 ml VOA vial leaving no headspace. The 0.4, 1.0, 10, 25, 50, and 100ppb standards are prepared by adding 0.8, 2.0, 20, 50, 100, and 200 μ L of working standard as described above, the amount of internal standard added remains constant for all the standards. These are then placed in the Archon for purge and trap analysis.

8.1.7 Carry out the purge-and-trap analysis procedure as described in Section 8.3.

8.1.8 EnviroQuant will tabulate the area response of the characteristic ions (see Table 2) against concentration for each compound and internal standard. It will also calculate response factors (RF) for each compound relative to its internal standards according to the calculations in Section 9.1.

NOTE: HP EnviroQuant software calculates and reports response factors for the analyst. It is each analyst's responsibility to become familiar with the software by reading the available manuals. 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 from each calibration standard's printout corresponds to the area account listed in the calibration method in Enviroquant. All of the analytes along with their retention times, retention windows, and the ions used for qualitative analysis are listed in Attachment B.

8.1.9 The average RF is calculated and recorded in the EnviroQuant Method for each compound. A system performance check is made before this calibration curve is used. Five compounds (the System Performance Check Compounds or SPCCs) are checked for a minimum average response factor. These compounds are Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane and Chlorobenzene. The minimum acceptable RFs are 0.1 for Chloromethane, 1,1-Dichloroethane, and Bromoform, and 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane. These compounds are used to check compound instability and to check for degradation of the

system, such as active sites in transfer lines or trap failure. **NOTE: The minimum response factor for all other compound should not be less than 0.01.** ESS Laboratory has found that the following poor performing compounds do not meet this criterion: Acetone, 2-Butanone (MEK), and 1,4-Dioxane.

- 8.1.10 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for all compounds using the equation in Section 9.2. The %RSD should be less than 15% for each compound, except for the following continuing calibration compounds (CCCs) 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinyl Chloride, where the %RSD must be less than 30%.
- 8.1.10.1 Linearity - If the %RSD of any compound 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.
- 8.1.10.2 When the RSD exceeds 15%, the plotting and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.
- 8.1.10.3 If a %RSD > 30 percent is measured for any method analyte (50% for poor purgers), then corrective action to eliminate a system leak and/or column reactive sites is required before re-attempting calibration. Poor purging compounds are listed in Table 2.
- 8.1.10.4 If the RSD of **any** compound exceeds 15% (never to exceed 30% except for poor purgers), then one of the following options must be applied to the GC/MS initial calibration in this situation or a new initial calibration must be performed.
- 8.1.10.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 15% QC limit. This option would apply in those instances where a linear instrument response is expected.
- 8.1.10.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points.

- 8.1.10.4.3 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards, **do not force the curve through zero**. In order to be used for quantitative purposes, the Correlation of Determination (COD/R²) must be greater than or equal to 0.99. This can easily be accomplished within the EnviroQuant software (see attachment B).
- 8.1.10.4.4 Use a non-linear calibration model. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range. (COD > 0.99) *When non-linear calibration is employed a minimum of six calibration points must be used for second order and seven calibration points for third order.*
- 8.1.11 Immediately after the initial calibration has been generated then the initial calibration has to be verified using a second source standard. The percent recovery for the independent calibration verification standard is 80-120% for all CCC compounds and 70-130% for all other compounds *When samples are to be run for the Army Corp of Engineer (Shell), all compounds must have ≤20% difference with no allowance. When samples are to be run for the Navy, DoD or AFCEE, all compounds must have ≤25% difference with no allowance.*
- 8.1.12 The RRT's are established from the 25 ppb mid-point standard in the initial calibration
- 8.2 Daily GC/MS analysis: Just prior to all samples and standard analysis, the run logs are filled out according to 8.1.4.
- 8.2.1 Prior to the analysis of samples, purge a 5 ug/L standard of 4-BFB (10 uL of standard is added into 50ml DI water, Section 7.2.8). The resultant mass spectra for BFB must meet all of the criteria in Section 11.1 before sample analysis begins. This criteria must be met each 12-hour shift.
- 8.2.2 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a calibration standard (7.2.7) at 25 µg/L (MS1, MS3, and MS4). Determine if the SPCCs meet criteria from section 8.1.9. Determine if the CCCs listed in section 8.1.10 have % Deviation less than 20% with all other analytes having a % Deviation less than 30%, poor purging compounds (Ketones, 2-CEVE) must be less than 50%. Compounds not analyzed for in the batch of samples do not need to meet the criteria. Continuing calibrations used for 8021 analysis must have <20% RSD. *When samples are to be run for the USACE/Navy/DoD/AFCEE, all compounds and surrogates should have <20% Drift/Deviation, see section 11.4 for expanded criteria.* For compounds analyzed for 624 continuing

calibration criteria are met or exceeded by 8260 criteria.

- 8.2.3 The internal standard responses and retention times in the continuing calibration standard should be evaluated immediately after data acquisition. If the area for any of the internal standards changes by a factor of two (-50% to +100%) or the retention time changes by \pm 0.5 minutes from the **mid-point standard of the initial calibration**, the mass spectrometer should be checked for defects. The relative retention time of each compound in each calibration run must agree to within 0.06 Relative Retention Time (RRT) Units. The RRT's in the continuing calibration standard is compared to the RRT's from the 25 ppb standard in the ICAL. Analysis is not to continue until problem has been resolved.
- 8.2.4 Method blank analysis should be performed at the following frequency:
- 8.2.4.1 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour shift and must be analyzed before any samples.
- 8.2.4.2 A method blank for volatile analysis should contain less than or equal to the Method Reporting limit of methylene chloride. For all other TCL compounds, the method blank should contain less than $\frac{1}{2}$ MRL of any single TCL analyte. See section 11.5 for instructions on corrective action when criteria are exceeded.
- 8.2.5 Blank Spike/Blank spike duplicate analysis must be performed once for each 12-hour shift, after the tune and CCV, and must be analyzed before any samples. BS/BSD concentrations are to be at the midpoint (median standard level) of the calibration curve. See section 10.0 for BS/BSD acceptance criteria.
- 8.2.5.1 See 7.2.7 for BS/BSD preparation.
- 8.3 GC/MS analysis: All samples and standards are analyzed under the same conditions in the associated initial calibration. Run logs are filled according to section 8.1.4.
- 8.3.1 Aqueous samples
- 8.3.1.1 All samples and working standard solutions should be allowed to warm to ambient temperature before analysis.
- 8.3.1.2 Set up the GC/MS system as outlined in Section 8.1.1.
- 8.3.1.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 11.1) before analyzing samples.
- 8.3.1.4 Sample or standard vials are placed in the Archon. The Archon will transfer 10 ml of sample/standard to a 25ml fitted purge tube (MS1, MS3, and MS4). The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future

analysis.

8.3.1.5 The Archon is programmed to add 1 μ L of Surrogate/Internal Standard (Section 7.2.4.2) to the sample. The Surrogate/Internal Standard QC Mix is to be added to each blank, sample and spiked sample.

8.3.1.6 Purge the sample for 11.0 \pm 0.1 min at ambient temperature, and then dry purge for 2 minutes.

8.3.1.7 At the conclusion of the purge time, the purge-and-trap will sound a ready signal from the GC and begin to desorb the trap while initiating the chromatographic temperature program and the CHEMSTATION data acquisition.

8.3.1.8 Before reusing any chamber, it must be rinsed with three-5 ml aliquots of reagent water.

8.3.1.9 After the sample has been desorbed for 2 minutes, the LSC 2000 will recondition the trap by heating to 260°C while back flushing with Helium. This step will take 10 minutes.

8.3.1.10 When necessary to composite samples for analysis, the following procedure is used:

8.3.1.10.1 Add 5 ml of each sample (up to 5 samples are allowed) to a 25 ml glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, if equal volumes of each sample are composited.

8.3.1.10.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

8.3.1.10.3 Mix each vial well and draw out a 5-ml aliquot with the 25-ml syringe.

8.3.1.10.4 Once all the aliquots have been combined in the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice.

8.3.1.10.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-ml sample is to be purged.

8.3.1.11 If a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. The ARCHON can be set to

dilute up to 100x. Dilutions can also be performed directly in 5-ml syringe by adding an aliquot of sample to reagent water using a micropipette. The follow procedure is used to dilute aqueous samples. All steps must be performed without delay, until the diluted sample is in a gas-tight syringe.

8.3.1.11.1 Dilutions may be made in volumetric flasks (10-100 ml). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

8.3.1.11.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

8.3.1.11.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 ml are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

8.3.1.11.4 DELETED

8.3.1.11.5 Dilutions are documented in the analytical run logbook.

8.3.1.12 When a sample is analyzed that has any target compound at a level exceeding twice the initial calibration range, this analysis should be followed by a blank reagent water analysis in the purge chamber to demonstrate that system is clean. If the blank analysis is not free of interferences, the system should be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

NOTE: Since the analytical system is automated the analyst may not be aware that a high concentration sample has been analyzed. If analysis of subsequent samples occurs, then the presence of the high concentration analyte makes re-analysis of that sample necessary to determine if carryover has occurred.

8.3.1.13 For matrix spike analysis, in a 100 ml volumetric flask add 40 μ L of the matrix spike solution (Section 7.2.3.1) to the 100 ml of sample. Invert three times and pour into 2 VOA vials.

8.3.1.14 All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

8.3.2 Dilutions for soil/sediment samples are discussed in SOP 20_5035.

8.3.2.1 The procedure for running soil/sediment samples is discussed in SOP 20_5035.

8.4 Data interpretation

8.4.1 Qualitative identification

8.4.1.1 An analyte (e.g. those listed in Table 2) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the compound obtained on the user's GC/MS. These standard reference spectra are obtained through analysis of the calibration standards. To compare the sample and reference spectra, go into the EnviroQuant software (QEDIT). The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity - any ions over 30% if less than three such ions are present. The analytes are listed in Attachment B for each analyte.

8.4.1.2 The requirements for qualitative verification by comparison of mass spectra are:

8.4.1.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine, where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time, will be accepted as meeting this criterion.

8.4.1.2.2 The RRT of the sample component is compared to the RRT from the 25 ppb standard in the ICAL and must be within ± 0.06 RRT units of the RRT of the standard component.

8.4.1.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

8.4.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. ESS Laboratory currently reports m- and p-xylanes together due to inability of resolving the isomers.

8.4.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra

containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

8.4.1.2.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are listed in section 11.9.3.

8.4.2 Quantitative Analysis

8.4.2.1 Compounds, which have been identified, are quantified by the internal standard method, utilizing the integrated abundance of the primary ion. The internal standard used shall be those listed in Section 8.1.7. The EnviroQuant method is setup to calculate the concentration of the analytes of interest in ng/ml using the initial calibration results stored in the method (Attachment B). This raw data number does not account for deviations in sample volume, sample weight, dilution factors, and % moisture. To include these other factors, see section 9.0.

9.0 CALCULATIONS

9.1 Response Factor (RF)

$$RF = (A_s \times C_s) / (A_{is} \times C_{is})$$

Where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in $\mu\text{g/L}$.

C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

9.2 Percent Relative Standard Deviation (%RSD)

$$\% RSD = (SD / RF_{Average}) \times 100\%$$

Where:

RSD = Relative standard deviation.

RF = Mean of 5 initial RFs for a compound.

SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference

$$\% \text{Difference} = ((\text{RF}_{\text{Average}} - \text{RF}_c) / \text{RF}_{\text{Average}}) \times 100\%$$

Where:

$\text{RF}_{\text{Average}}$ = Average response factor from initial calibration

RF_c = Response factor from current verification check standard.

9.4 Percent Drift

$$\% \text{Drift} = ((C_1 - C_0) / C_1) \times 100\%$$

Where:

C_1 = compound standard concentration

C_0 = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste Concentration

$$\mu\text{g/L} = (A_x)(C_s)(D) / (A_{is})(\text{RF}_{\text{Average}})(V_s)$$

Where:

A_x = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_s = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, $D = 1$. The dilution factor is always unitless.

$\text{RF}_{\text{Average}}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted or purged (ml). If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/ml, which is equivalent to $\mu\text{g/L}$.

9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste – see SOP 20_5035.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Accuracy and Precision

Laboratory personnel must demonstrate initial proficiency for each sample preparation

method/matrix that he or she performs. New employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This is accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. 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 must 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. 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:

Analysis Method:

Date Analyzed:

Accuracy:

Precision:

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

Interpretation of Results: Percent recovery is to be within 80-120% for all analytes with a RPD of $\leq 20\%$. For the full 8260B list a maximum of 5 sporadic marginal failures are allowed with the

expanded criteria of 60-140%.

- 10.1 A preparation batch is defined as a group of up to 20 field samples of similar matrix that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the samples, each prep batch will contain at a minimum, a method blank, a blank spike, blank spike duplicate, a matrix spike, and a matrix spike duplicate. An analytical/instrument batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. The analytical batch includes QC requirements as defined by the analytical (determinative) method. For example, each injection sequence would begin with a tune and CCV (or tune, calibration and ICV), followed by a method blank, BS/BSD, MS/MSD, etc. and remaining field samples. Prep batches may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP. If all field and QC samples could not be completed within the 12-hour clock, then another tune, CCV, and MB would be required. Each analytical sequence must be documented using the run log in Figure 1.
- 10.2 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 11.1. The computer software will evaluate the tune information. The analyst should be aware of the process used.
- 10.3 Run a 7-point 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 11.2.
- 10.4 Run initial calibration verification (ICV) standard using secondary standards (7.2.3) following the initial calibration curve. Acceptance criteria are listed in Section 11.3.
- 10.5 Run a mid-point Continuing Calibration Verification (CCV) at 25 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 11.4.
- 10.6 A method blank must be prepared with each batch of samples not to exceed 12 hours. The method blank should be prepared from organic-free water. The method blanks are to be related to each 12-hour sequence of samples injected. Acceptance criteria for these blanks are listed in Section 8.2.4 and corrective action is listed in Section 11.5. Instrument blanks may be injected at any time in the sequence to verify absence of contamination.
- 10.7 A Blank spike/Blank spike duplicate (BS/BSD) must be prepared and analyzed with each batch of samples. The BS/BSDs are prepared using a source different than that used to prepare the initial calibration standards and contain all method target analytes (see 7.2.3.1 and 7.2.7). Acceptance criterion is 70-130% and $\leq 25\%$ RPD.
For the Army Corp of Engineer (Shell), all compounds in the BS/BSD must be between 80-120% Rec with allowance for sporadic marginal exceedances. Sporadic marginal exceedances have the expanded criterion of 60-140%. See

Table 3 for DoD and AFCEE blank spike acceptance criteria. Sporadic Marginal Exceedance number is as follows:

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 3) or expanded criterion listed above.

NOTE: The CCV and BS are considered equivalent for aqueous samples. Only one will need to be analyzed but it must be prepared from a source different than that used to perform initial calibration. In the case of solids, the BS is extracted similar to the samples. Use CCV acceptance criteria as presented in Section 8.2.2.

- 10.7.1 Control charts will be maintained for the BS/BSD for a representative subset of target analytes and surrogate spikes. Biannually, control limits will be determined for all target analytes and surrogates for comparison to default limits.
- 10.8 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples.
 - 10.8.1 The matrix spike is prepared as described in Section 7.2.8.
 - 10.8.2 Matrix spike control limits are 70-130%. %RPD between spike and spike duplicate should be $\leq 30\%$ ($\leq 20\%$ for AFCEE). **For DoD and AFCEE matrix spike acceptance criteria use the blank spike acceptance criteria in Table 3.** For samples outside control limits, see Section 11.6 for corrective action.
- 10.9 Surrogates recoveries limits are 70-130%. **For DoD and AFCEE surrogate acceptance criteria use the blank spike acceptance criteria in Table 3.** For samples outside control limits, see Section 11.7 for corrective action.
- 10.10 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 11.8.
- 10.11 Ion abundance for target compounds and any tentatively identified compounds need to meet specific requirements. Acceptance criteria for ion abundance are presented in Section 11.9.

-
- 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 11.11.
 - 10.14 MDLs are determined in reagent water or organic-free sand/soil and verified annually. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

11.0 DATA VALIDATION

The items shall be verified and documented on the VOA Department Analysis Map (Attachment D.)

- 11.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

BFB Key Ions and Ion Abundance Criteria:

Ion	Abundance
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

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

Re-inject BFB.

Retune with PFTBA, then re-inject BFB.

Clean MS source, retune with PFTBA, and re-inject BFB.

- 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 a seven-point initial calibration curve is analyzed, ensure that the following

criteria were met.

- 11.2.1 For the RFs, the %RSD must be less than 30% for all method target analytes, except for the poor purgers where the %RSD must be less than 50%.
 - 11.2.1.1 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.
 - 11.2.1.2 If the %RSD of any method target analyte is greater than 15%, calibration curves must be constructed using first or higher order regression fits of the six calibration points. *NOTE: A minimum of 5 points are used for average and linear regression calibration, six point for second order, and seven points for third order calibrations.* The corresponding Coefficients of Determination (R^2) must be 0.99 or greater.
 - 11.2.1.3 If these 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 seven standards at different concentrations. The low end of the calibration curves must be carefully watched.
- 11.2.2 System Performance Check Compounds (SPCCs) and continuing calibration compounds (CCCs) must be evaluated and passed for initial calibration. Refer to Section 8.1.9 and 8.1.10.
- 11.3 If an initial calibration was performed, verify that initial calibration verification (ICV) was also performed. The percent difference for the CCC analytes must be within 20% (30% for all other target analytes). *When samples are to be run for the Army Corp of Engineer (Shell), all compounds must have ≤20% drift with no allowance. Samples that are to be analyzed for Navy/DoD/AFCEE must have ≤25% drift for all compounds, no allowances.* If not, reanalyze the ICV or prepare a new calibration curve as necessary.
- 11.4 Ensure the continuing calibration verification (CCV) standard is analyzed 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): A system performance check must be made during every 12-hour shift. 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. The minimum RF for volatile SPCCs is listed in Section 8.1.8.

11.4.2 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Section 8.1.9 are used to check the validity of the initial calibration with a limit of <20% drift or difference. Acceptance criteria is ≤ 30% drift for all other method target analytes (50% for the poor purgers). *When samples are to be run for the Army Corp of Engineers, all compounds must have <20% drift.*

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 six-point 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.

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 blanks. The analyst shall confirm that this blank was analyzed at the required frequency and the criteria in Section 8.3.4 are met. Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed 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 ½ the MRL 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 matrix spike/matrix spike duplicates were analyzed at required frequency. Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the control limits listed in Section 10. If results are not within criteria,

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 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, 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 of 70-130%. 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 necessary. *When samples are to be run for the Army Corp of Engineers, surrogate limits must be within 20% for method blank and BS/BSD and 30% for project samples. See Table 3 for surrogate acceptance limits for DoD/Navy/AFCEE projects.*
 - 11.7.2 If sample surrogate recoveries exceed these limits, the sample must be re-analyzed. If the results are still outside the limits, then matrix affect has been verified and results are discussed in case narrative. *NOTE: If an obvious matrix effect is seen on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT-RCP projects must have a copy of the chromatogram included with the report.*
- 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:
 - 11.8.1 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.
 - 11.8.2 Internal standard retention time:
 - 11.8.2.1 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.
 - 11.8.3 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.
 - 11.9 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.

11.9.1 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.

11.9.2 The relative intensities of ions specified in Section 11.10.1 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.)

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

11.9.2.2 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.

11.9.2.3 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.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification 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:

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

11.9.3.2 The relative intensities of the major ions should agree within $\pm 30\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).

11.9.3.3 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.

11.9.3.4 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.

11.10 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% if their associated CCV. If the internal standard area counts fail this criterion, the following corrective actions should be considered:

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

11.10.2 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.

11.10.3 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 and CT-RCP projects must have a copy of the chromatogram included with the report.*

11.11 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.

11.12 All manual integration must be printed, when made, for verification.

11.13 Besides the items listed in Sections 11.1 through 11.12, the analyst should also verify the additional items as noted in Attachment D. A second level of review must be performed by a second analyst, results are recorded on Attachment D.

12.0 REFERENCES

- 12.1 SW-846 Method 8260B, Test Methods for the Analysis of Solid Waste, Third Edition, Update III.
- 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.

13.0 ATTACHMENTS

- 13.1 Attachment A – Primary Standard Certificates of Analysis
- 13.2 Attachment B – Enviroquant method
- 13.3 Attachment C – Run logbook
- 13.4 Attachment D – Analysis Map

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 14.1 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.

15.0 METHOD PERFORMANCE

- 15.1 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 80-120% Recovery and %RSD of $\leq 20\%$. Five (5) sporadic marginal exceedances are allowed.
- 15.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

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

Analyte	Spk	Avg	%Rec	%RSD	Analyte	Spk	Avg	%Rec	%RSD
Dichlorodifluoromethane	25	24.51	98	1.3	2-Hexanone	25	118.53	95	1.7
Chloromethane	25	23.82	95	1.1	trans-1,3-Dichloropropene	25	26.19	105	1.8
Vinyl Chloride	25	23.94	96	0.7	1,1,2-Trichloroethane	25	24.73	99	1.7
Bromomethane	25	25.37	101	1.9	1,3-Dichloropropane	25	25.19	101	0.9
Chloroethane	25	25.00	100	1.6	Tetrachloroethene	25	25.15	101	1.2
Trichlorodifluoromethane	25	25.05	100	0.2	Dibromochloromethane	25	27.05	108	1.3
Diethyl ether	25	24.63	99	1.7	1,2-Dibromoethane	25	25.26	101	1.4
Acetone	25	113.63	91	1.1	1-Chlorohexane	25	24.39	98	1.0
Carbon Disulfide	25	24.35	97	1.0	Chlorobenzene	25	24.75	99	1.3
1,1-Dichloroethene	25	24.37	97	0.5	1,1,1,2-Tetrachloroethane	25	25.23	101	1.1
Methylene Chloride	25	24.55	98	1.7	Ethylbenzene	25	24.67	99	1.0
Methyl tert-Butyl Ether	25	47.42	95	1.3	Xylene P,M	25	49.08	98	1.5
trans-1,2-Dichloroethene	25	24.42	98	2.4	Xylene O	25	24.56	98	1.8
1,1-Dichloroethane	25	24.31	97	1.8	Styrene	25	25.42	102	1.2
Vinyl Acetate	25	24.78	99	1.8	Bromoform	25	26.13	105	1.1
Di-isopropyl ether	25	24.71	99	1.8	Isopropylbenzene	25	24.86	99	0.8
Ethyl tertiary-butyl ether	25	24.92	100	1.6	1,2,3-Trichloropropane	25	24.13	97	1.2
2-Butanone	25	123.99	99	2.2	Bromobenzene	25	24.95	100	1.7
cis-1,2 Dichloroethene	25	24.54	98	1.6	1,1,2,2-Tetrachloroethane	25	24.79	99	1.9
2,2-Dichloropropane	25	24.47	98	2.1	n-Propylbenzene	25	24.56	98	1.9
Bromochloromethane	25	24.14	97	1.1	2-Chlorotoluene	25	24.31	97	3.9
Tetrahydrofuran	25	23.44	94	2.1	4-Chlorotoluene	25	24.20	97	1.1
Chloroform	25	24.53	98	1.4	1,3,5-Trimethylbenzene	25	24.53	98	1.6
1,1,1-Trichloroethane	25	24.92	100	1.1	tert-Butylbenzene	25	24.52	98	1.2
1,1-Dichloropropene	25	24.41	98	0.9	1,2,4-Trimethylbenzene	25	24.55	98	0.9
Carbon Tetrachloride	25	24.99	100	1.4	sec-Butylbenzene	25	24.67	99	1.3
Benzene	25	24.23	97	1.2	1,3 Dichlorobenzene	25	24.27	97	1.4
1,2-Dichloroethane	25	25.00	100	1.6	4-Isopropyltoluene	25	24.42	98	1.4
Tertiary-amyl methyl ether	25	24.64	99	1.8	1,4 Dichlorobenzene	25	24.46	98	1.4
Trichloroethene	25	24.46	98	1.3	n-Butylbenzene	25	24.65	99	1.0
1,2-Dichloropropane	25	24.25	97	1.8	1,2 Dichlorobenzene	25	24.23	97	1.1
Dibromomethane	25	24.61	98	2.1	1,2-Dibromo-3-Chloropropane	25	25.46	102	1.8
1,4-Dioxane	25	489.19	98	11.1	1,2,4-Trichlorobenzene	25	24.36	97	1.9
Bromodichloromethane	25	25.14	101	1.9	Hexachlorobutadiene	25	26.30	105	1.9
4-Methyl-2-Pentanone	25	121.18	97	1.7	Naphthalene	25	23.59	94	1.3
cis-1,3-Dichloropropene	25	25.30	101	1.7	1,2,3-Trichlorobenzene	25	23.71	95	2.3
Toluene	25	24.70	99	1.1					

17.0 DEFINITIONS

- 17.1 **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.
- 17.2 **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.
- 17.3 **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).
- 17.4 **Control Sample:** A QC sample introduced into a process to monitor the performance of the system.
- 17.5 **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.
- 17.6 **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.
- 17.7 **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.
- 17.8 **Blank Spike (BS):** A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.
- 17.9 **Matrix:** The component or substrate (e.g., surface/water, drinking water) which contains the analyte of interest.
- 17.10 **Matrix Duplicate:** An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

- 17.11 **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.
- 17.12 **Matrix Spike Duplicates:** Intralaboratory 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.
- 17.13 **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.
- 17.14 **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.
- 17.15 **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.
- 17.16 **Records:** Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.
- 17.17 **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.
- 17.18 **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.

18.0 PERSONNEL QUALIFICATIONS

- 18.1 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.
- 18.2 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.

19.0 TROUBLESHOOTING

- 19.1 If BFB criteria are not met, the analysis must be repeated. Analysis can not begin until BFB 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.
- 19.2 If manual or auto-tune does not produce BFB spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.
- 19.3 Method blanks must not contain any target compound greater than $\frac{1}{2}$ the MRL with the exception of methylene chloride (see section 11.5 for expanded criteria). If the method blank does not meet criteria, then system must be cleaned before processing samples. This includes washing the purge vessel and baking the trap.
- 19.4 See laboratory supervisor or operations manager for all other maintenance problems.
- 19.5 Record all maintenance in the instrument's maintenance logbook.

20.0 Data Management And Records

- 20.1 **Data Management** - ESS Laboratory's utilizes the Premium 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.
- 20.2 **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.

TABLE 2 -Internal Standards with their associated compounds

FLUOROBENZENE (IS)	CHLOROBENZENE-d5 (IS)	1,4-DICHLOROBENZENE-d4 (IS)
Dichlorodifluoromethane	Toluene - d ₈	Isopropylbenzene
Chloromethane	Toluene	trans-1,4-Dichloro-2-Butene
Vinyl Chloride	2-Hexanone (P)	1,2,3-Trichloropropane
Bromomethane	Ethyl methacrylate	Bromobenzene
Chloroethane (P)	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Trichlorofluoromethane	Tetrachloroethene	n-Propylbenzene
Diethyl Ether	Dibromochloromethane	2-Chlorotoluene
Acrolein (P)	1,2-Dibromoethane	4-Chlorotoluene
1,1,2-Trichloro-1,2,2-trifluoroethane	1-Chlorohexane	1,3,5-Trimethylbenzene
Acetone (P)	Chlorobenzene	tert-Butylbenzene
Iodomethane (P)	1,1,1,2-Tetrachloroethane	Pentachloroethane
Carbon Disulfide (P)	Ethylbenzene	1,2,4-Trimethylbenzene
1,1-Dichloroethene	Xylene, M,O	sec-Butylbenzene
Allyl Chloride	Xylene P	1,3-Dichlorobenzene
Methyl acetate	Styrene	4-Isopropyltoluene
Methylene Chloride	Bromoform	1,4-Dichlorobenzene
Methyl tert-butyl ether	cis 1,4-Dicchboro-2-butene	n-Butylbenzene
Acrylonitrile (P)	4-Bromofluorobenzene*	1,2-Dichlorobenzene
trans-1,2-Dichloroethene		Hexachloroethane
1,1-Dichloroethane		1,2-Dibromo-3-chloropropane (P)
Vinyl Acetate		1,2,4-Trichlorobenzene
Chloroprene		Hexachlorobutadiene
Di-isopropyl ether		Naphthalene
Ethyl tertiary butyl ether		1,2,3-Trichlorobenzene
2-Butanone (P)		
cis-1,2-Dichloroethene		
2,2-Dichloropropane		
Methyl acrylate		
Bromochloromethane		
Methacrylonitrile		
Tetrahydrofuran		
Chloroform		
Dibromofluoromethane*		
1,1,1-Trichloroethane		
Cyclohexane		
1-Chlorobutane		
1,1-Dichloropropene		
Carbon Tetrachloride		
1,2-Dichloroethane-D4*		
Benzene		
1,2-Dichloroethane		
Tertiary-amyl methyl ether		
Trichlorethene		
Methylcyclohexane		
1,2-Dichloropropane		
Dibromomethane		
1,4-Dioxane (P)		
Bromodichloromethane		
2-Nitropropane		
2-Chloroethyl vinyl ether (P)		
4-Methyl-2-pentanone (P)		
cis-1,3-Dichloropropene		
Toluene		
trans-1,3-Dichloropropene		
1,1,2-Trichloroethane		

IS = Internal Standard; * = Surrogate; (P) = Poor purger

Table 3 DoD Quality Systems Manual and AFCEE QAAP Blank Spike QC Limits

Analyte	DoD				AFCEE			
	LCL	UCL	LMEL	UMEL	LCL	UCL	LMEL	UMEL
1,1,1,2-Tetrachloroethane	80	130	75	135	81	129	75	135
1,1,1-Trichloroethane	65	130	55	145	67	132	55	145
1,1,2,2-Tetrachloroethane	65	130	55	140	63	128	55	140
1,1,2-Trichloroethane	75	125	65	135	75	125	65	135
1,1-Dichloroethane	70	135	60	145	69	133	60	145
1,1-Dichloroethene	70	130	55	140	68	130	55	140
1,1-Dichloropropene	75	130	65	140	73	132	65	140
1,2,3-Trichlorobenzene	55	140	45	155	67	137	45	155
1,2,3-Trichloropropane	75	125	65	130	73	124	65	130
1,2,4-Trichlorobenzene	65	135	55	145	66	134	55	145
1,2,4-Trimethylbenzene	75	130	65	140	74	132	65	140
1,2-Dibromo-3-Chloropropane	50	130	35	145	50	132	35	145
1,2-Dibromoethane	80	120	75	125	80	121	75	125
1,2-Dichlorobenzene	70	120	60	130	71	122	60	130
1,2-Dichloroethane	70	130	60	140	69	132	60	140
1,2-Dichloropropane	75	125	65	135	75	125	65	135
1,3,5-Trimethylbenzene	75	130	65	140	74	131	65	140
1,3-Dichlorobenzene	75	125	65	130	75	124	65	130
1,3-Dichloropropane	75	125	65	135	73	126	65	135
1,4 Dichlorobenzene	75	125	65	130	74	123	65	130
2,2-Dichloropropane	75	135	60	150	69	137	60	150
2-Butanone	30	150	10	170	49	136	49	136
2-Chlorotoluene	75	125	65	135	73	126	65	135
2-Hexanone	55	130	45	140	-	-	-	-
4-Chlorotoluene	75	130	65	135	74	128	65	135
4-Methyl-2-Pentanone	60	135	45	145	58	134	45	145
Acetone	40	140	20	160	40	135	20	160
Benzene	80	120	75	130	81	122	75	130
Bromobenzene	75	125	70	130	76	124	70	130
Bromochloromethane	65	130	55	140	65	129	55	140
Bromodichloromethane	75	120	70	130	76	121	70	130
Bromoform	70	130	60	140	69	128	60	140
Bromomethane	30	145	10	165	30	141	10	165
Carbon Disulfide	35	160	15	185	-	-	-	-
Carbon Tetrachloride	65	140	55	150	66	138	55	150
Chlorobenzene	80	120	75	130	81	122	75	130
Dibromochloromethane	60	135	45	145	66	133	45	145
Chloroethane	60	135	50	145	58	133	50	145
Chloroform	65	135	50	150	69	128	50	150

Chloromethane	40	125	25	140	56	131	25	140
cis-1,2 Dichloroethene	70	125	60	135	72	126	60	135
cis-1,3-Dichloropropene	70	130	60	140	69	131	60	140
Dibromomethane	75	125	65	135	76	125	65	135
Dichlorodifluoromethane	30	155	10	175	30	153	10	175
Ethylbenzene	75	125	65	135	73	127	65	135
Hexachlorobutadiene	50	140	35	160	67	131	35	160
Isopropylbenzene	75	125	65	135	75	127	65	135
m,p-Xylene	75	130	65	135	76	128	65	135
Methylene Chloride	55	140	40	155	63	137	40	155
Methyl tert-Butyl ether	65	125	55	135	65	123	55	135
Naphthalene	55	140	40	150	54	138	40	150
n-Butylbenzene	70	135	55	150	69	137	55	150
n-Propylbenzene	70	130	65	140	72	129	65	140
o-Xylene	80	120	75	130	80	121	75	130
p-Isopropyltoluene	75	130	65	140	73	130	65	140
sec-Butylbenzene	70	125	65	135	72	127	65	135
Styrene	65	135	55	145	65	134	55	145
tert-Butylbenzene	70	130	60	140	70	129	60	140
Tetrachloroethene	45	150	25	165	66	128	60	135
Toluene	75	120	70	130	77	122	70	130
trans-1,2-Dichloroethene	60	140	45	150	63	137	45	150
trans-1,3-Dichloropropene	55	140	40	155	59	135	40	155
Trichloroethene	70	125	60	135	70	127	60	135
Trichlorofluoromethane	60	145	45	160	57	129	45	160
Vinyl Chloride	50	145	35	165	50	134	35	165
<i>Surrogates:</i>								
Dibromofluoromethane	85	115	-	-	85	115	-	-
Toluene-d8	85	120	-	-	81	120	-	-
4-Bromofluorobenzene	75	120	-	-	76	119	-	-
1,2-Dichloroethane-d4	70	120	-	-	72	119	-	-

LCL = Lower Control Limit

UCL = Upper Control Limit

LMEL = Lower Marginal Exceedance Limit

UMEL = Upper Marginal Exceedance Limit

Table 4

Summary of Method Quality Objectives for Method 8260B
Volatile Organic Compounds

OC Element	Frequency	Target Analyte/Surrogate	Corrective action
Tune	Every twelve hours	Criteria in Table 1	Corrective action
Initial Calibration	Instrument set up. Each time the ICV or CCV can not meet criteria.	RSD \leq 15%, R \geq 0.995, R ² \geq 0.99 Do not force through origin	Suspend analysis until criteria met.
ICV	Immediately following initial calibration.	%Rec = 70-130% USACE: 80-120% DoD/AFCEE: 75/125%	Recalibrate as required.
CCV	At the beginning of each analytical sequence. No more than 20 samples or 12 hours between CCVs.	CCCs must be < 20%, all other analytes < 30% drift or difference. USACE/DoD/AFCEE: 80-120% all compounds.	Re-analyze once, if still fails criteria recalibrate instrument.
Blank spike/ Blank spike duplicate	At the beginning of each analytical sequence. Prepared using source different than that used to prepare initial calibration standards and contain all target analytes.	All target analytes < 30% drift or difference and \leq 25%RPD, except for 1,4-dioxane (screen), where lab CLs are used. USACE: 80-120% for all compounds, allows for 5 sporadic marginal failures. <i>See Table 3 for DoD/AFCEE criteria</i>	Re-analyze once with associated samples. If still outside criteria, report exceedance in project narrative. The lab is to identify those difficult analyte that routinely are outside the 70-130% criterion, disclose in P/N.
Method Blank	One per analytical batch of 20 or fewer samples. Use second source standard MS/MSD	Analytes < $\frac{1}{2}$ MRL 1 per 20 samples.	See Section 11.0. Report nonconformance in case narrative.
Surrogates	Added to all samples and standards.	% Rec = 70-130%, except for 1,4-dioxane (screen), where lab CLs are used; \leq 30% RPD. <i>See Table 3 for DoD/AFCEE criteria</i>	Check BS, if BS is within criteria then report non-conformance in project narrative. If BS fails, then re-analyze.
Internal Standards	Added to all samples and standards.	$\frac{1}{2}$ MRL 50-200% of area counts in the associated continuing calibration standard.	If one or more is outside criteria, then re-analyze sample unless an obvious interference is present (UCM).



CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

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

Catalog No.: 30006 Lot No.: A034309
Description: VOA Calibration Mix #1
Expiration Date¹: February 2008 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Acetone	67-64-1	99%	5000 ug/mL	+/- 0.1
2	2-Butanone (MEK)	78-93-3	99%	5000 ug/mL	+/- 0.1
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5000 ug/mL	+/- 0.1
4	2-Hexanone	591-78-6	99%	5000 ug/mL	+/- 0.1

Solvent: P&T Methanol/Water (90:10) 67-56-1/7732-18-5 99%

Column:
105m x .32mm x 3.0um
Rtx-502.2 (cat.#10910)

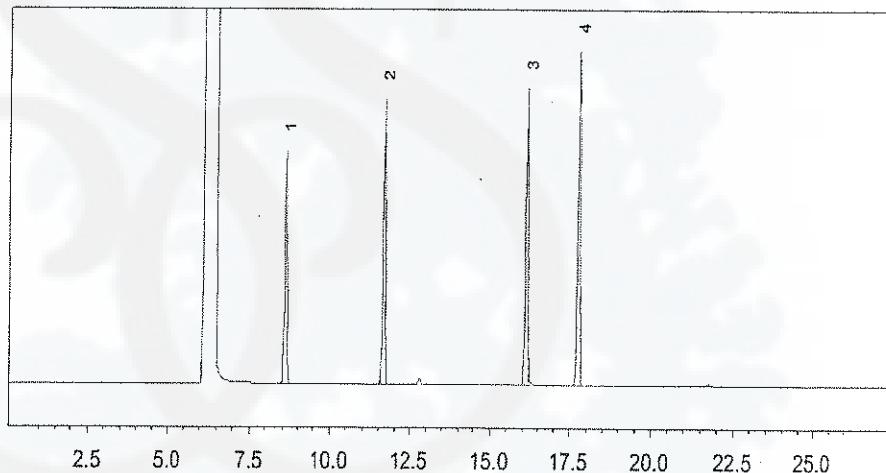
Carrier Gas:
hydrogen @ 40 cc/min.

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: MEW

John Lidgett

John Lidgett - Q.A. 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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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

Catalog No.: 30042 _____ Lot No.: A034239 _____

Description: 502.2 Calibration Mix #1 _____

Expiration Date¹: July 2011 _____ Storage: Freezer _____

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Dichlorodifluoromethane (CFC-12)	75-71-8	99%	2003 ug/mL	+/- 1.3
2	Chloromethane (methyl chloride)	74-87-3	99%	1997 ug/mL	+/- 1.3
3	Vinyl chloride	75-01-4	99%	2002 ug/mL	+/- 2.1
4	Bromomethane (methyl bromide)	74-83-9	99%	1995 ug/mL	+/- 1.3
5	Chloroethane (ethyl chloride)	75-00-3	99%	1996 ug/mL	+/- 1.3
6	Trichlorofluoromethane (CFC-11)	75-69-4	99%	2000 ug/mL	+/- 0.3

Solvent: P&T Methanol 67-56-1 99%

Column:

105m x .32mm x 1.8um
Rtx-502.2 (cat.#10921)

Carrier Gas:

helium @ 2.2 ml/min.

Temp. Program:

50°C (hold 9.5 min.) to 50°C
@ 8°C/min.

Inj. Temp:

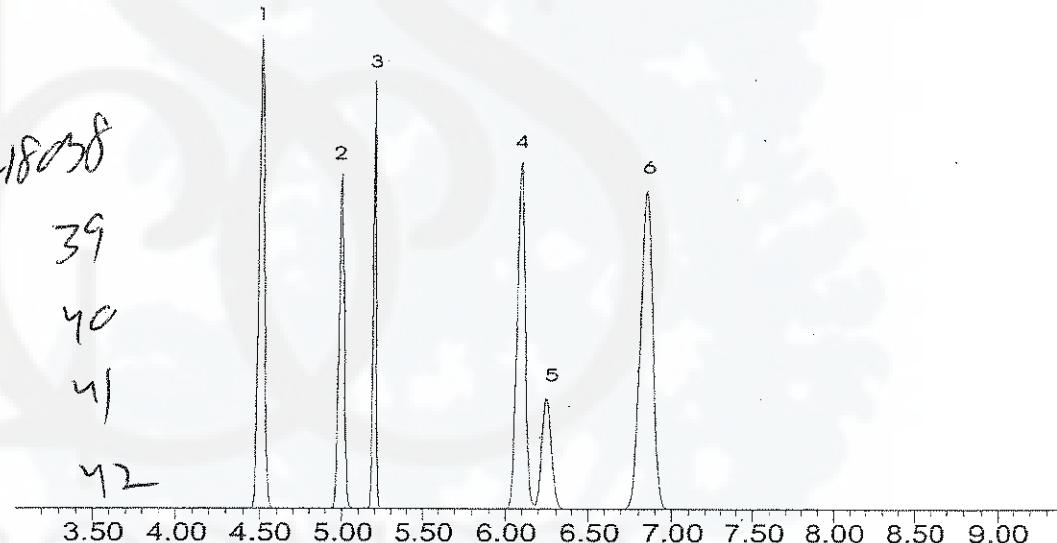
200 °C

Det. Temp:

250 °C

Det. Type:

MSD



Manufactured By: MEW

John Lidge

John Lidge - 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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

⁴ Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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CERTIFICATE OF COMPOSITION

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.: 558360 Lot No.: A036277
Description: Custom VOA Additions Standard
Expiration Date¹: October 2006 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Methyl acetate	79-20-9	99%	2000 ug/mL	+/- 0.1
2	1-Chlorobutane (Butyl chloride)	109-69-3	99%	2000 ug/mL	+/- 0.1
3	Cyclohexane	110-82-7	99%	2000 ug/mL	+/- 0.1
4	Methylcyclohexane	108-87-2	99%	2000 ug/mL	+/- 0.1
5	1-Chlorohexane	544-10-5	99%	2000 ug/mL	+/- 0.1
6	Hexachloroethane	67-72-1	99%	2000 ug/mL	+/- 0.1
Solvent: P&T Methanol		67-56-1	99%		

Column:

105m x .32mm x 1.8um
Rtx-502.2 (cat.#10921)

Carrier Gas:
helium @ 2.2 ml/min.

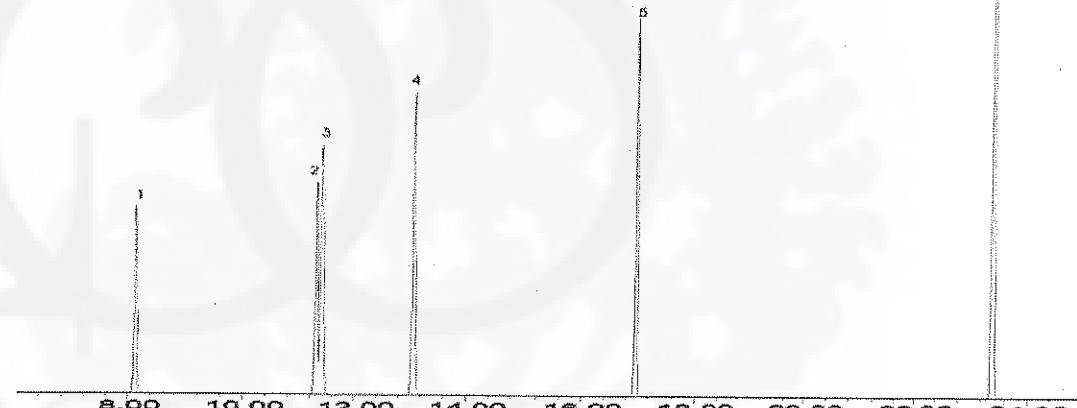
Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
250°C

Det. Temp:
250°C

Det. Type:
MSD

5/12/04 7 thru 5/12/058



Manufactured By: JP

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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

⁴ Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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CERTIFICATE OF ANALYSIS

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

Catalog No.: 30475A Lot No.: A036598
Description: 8260B Calibration Mix #1
Expiration Date: April 2008 Storage: Freezer

SE DC68
b9
7075

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Diethyl ether (ethyl ether)	60-29-7	99%	2000 ug/mL	+/- 0.3
2	1,1,2-Trichlorotrifluoroethane (CFC-113)	76-13-1	99%	2000 ug/mL	+/- 0.3
3	1,1-Dichloroethylene	75-35-4	99%	2000 ug/mL	+/- 0.3
4	Acetonitrile	75-05-8	99%	2000 ug/mL	+/- 0.3
5	Iodomethane (methyl iodide)	74-88-4	99%	2000 ug/mL	+/- 0.3
6	Allyl chloride (3-chloropropene)	107-05-1	99%	2000 ug/mL	+/- 0.3
7	Methylene chloride (dichloromethane)	75-09-2	99%	2000 ug/mL	+/- 0.3
8	Carbon disulfide	75-15-0	99%	2000 ug/mL	+/- 0.3
9	Acrylonitrile	107-13-1	99%	2000 ug/mL	+/- 0.3
10	trans-1,2-Dichloroethylene	156-60-5	99%	2000 ug/mL	+/- 0.3
11	1,1-Dichloroethane	75-34-3	98%	2000 ug/mL	+/- 0.3
12	Chloroprene (2-chloro-1,3-butadiene)	126-99-8	99%	1961 ug/mL	+/- 1.6
13	Propionitrile	107-12-0	99%	2000 ug/mL	+/- 0.3
14	2,2-Dichloropropane	594-20-7	99%	2000 ug/mL	+/- 0.3
15	cis-1,2-Dichloroethylene	156-59-2	98%	2000 ug/mL	+/- 0.3
16	Methacrylonitrile	126-98-7	99%	2000 ug/mL	+/- 0.3
17	Methyl acrylate	96-33-3	99%	2000 ug/mL	+/- 0.3
18	Isobutanol (2-Methyl-1-propanol)	78-83-1	99%	2000 ug/mL	+/- 0.3
19	Chloroform	67-66-3	99%	2000 ug/mL	+/- 0.3
20	Bromoform	74-97-5	99%	2000 ug/mL	+/- 0.3
21	Tetrahydrofuran	109-99-9	99%	2000 ug/mL	+/- 0.3
22	1,1,1-Trichloroethane	71-55-6	99%	2000 ug/mL	+/- 0.3
23	1,1-Dichloropropene	563-58-6	99%	2000 ug/mL	+/- 0.3
24	Carbon tetrachloride	56-23-5	99%	2000 ug/mL	+/- 0.3
25	1,2-Dichloroethane	107-06-2	99%	2000 ug/mL	+/- 0.3
26	Benzene	71-43-2	99%	2000 ug/mL	+/- 0.3
27	2-Chloroethanol (ethylene chlorhydrin)	107-07-3	99%	2000 ug/mL	+/- 0.3
28	Trichloroethylene	79-01-6	99%	2000 ug/mL	+/- 0.3
29	1,2-Dichloropropane	78-87-5	98%	2000 ug/mL	+/- 0.3
30	Methyl methacrylate	80-62-6	99%	2000 ug/mL	+/- 0.3
31	Bromodichloromethane	75-27-4	99%	2000 ug/mL	+/- 0.3
32	1,4-Dioxane	123-91-1	99%	2000 ug/mL	+/- 0.3
33	2-Nitropropane	79-46-9	98%	2000 ug/mL	+/- 0.3
34	Dibromomethane	74-95-3	99%	2000 ug/mL	+/- 0.3
35	cis-1,3-Dichloropropylene	10061-01-5	99%	2000 ug/mL	+/- 0.3
36	Toluene	108-88-3	99%	2000 ug/mL	+/- 0.3
37	Ethyl methacrylate	97-63-2	99%	2000 ug/mL	+/- 0.3
38	trans-1,3-Dichloropropylene	10061-02-6	99%	2000 ug/mL	+/- 0.3
39	1,1,2-Trichloroethane	79-00-5	99%	2000 ug/mL	+/- 0.3
40	1,3-Dichloropropane	142-28-9	99%	2000 ug/mL	+/- 0.3
41	Tetrachloroethylene	127-18-4	99%	2000 ug/mL	+/- 0.3
42	Dibromochloromethane	124-48-1	99%	2000 ug/mL	+/- 0.3
43	1,2-Dibromoethane (EDB)	106-93-4	99%	2000 ug/mL	+/- 0.3
44	Chlorobenzene	108-90-7	99%	2000 ug/mL	+/- 0.3
45	1,1,1,2-Tetrachloroethane	630-20-6	99%	2000 ug/mL	+/- 0.3
46	Ethylbenzene	100-41-4	99%	2000 ug/mL	+/- 0.3
47	m-Xylene	108-38-3	99%	2000 ug/mL	+/- 0.3
48	p-Xylene	106-42-3	99%	2000 ug/mL	+/- 0.3
49	o-Xylene	95-47-6	99%	2000 ug/mL	+/- 0.3
50	Styrene	100-42-5	99%	2000 ug/mL	+/- 0.3
51	Isopropylbenzene (cumene)	98-82-8	99%	2000 ug/mL	+/- 0.3
52	Bromoform	75-25-2	99%	2000 ug/mL	+/- 0.3
53	cis-1,4-Dichloro-2-butene	1476-11-5	97%	2000 ug/mL	+/- 0.3
54	1,1,2,2-Tetrachloroethane	79-34-5	99%	2000 ug/mL	+/- 0.3
55	1,2,3-Trichloropropene	96-18-4	99%	2000 ug/mL	+/- 0.3
56	trans-1,4-dichloro-2-butene	110-57-6	98%	2000 ug/mL	+/- 0.3
57	n-Propylbenzene	103-65-1	99%	2000 ug/mL	+/- 0.3
58	Bromobenzene	108-86-1	99%	2000 ug/mL	+/- 0.3



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

Catalog No.: 30216 Lot No.: A035108
Description: Vinyl Acetate Standard
Expiration Date¹: December 2009 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Vinyl acetate	108-05-4	99%	2000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

Carrier Gas:
hydrogen @ 40 cm/sec.

Temp. Program:
40°C (hold 2 min.) to 130°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID

Manufactured By: MEW

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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

⁴ Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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Tech Tip:

Vinyl acetate is a volatile organic ester included in the target lists of several US EPA and other methods. Under acidic conditions, esters react with alcohols to form new esters (transesterification). Methanol-based mixes containing halogenated compounds are slightly acidic, so it is important to minimize exposure of vinyl acetate to mixes of halogenated compounds in methanol. For this reason, we offer vinyl acetate in individual solution, and suggest that it be introduced into the working level calibration solution immediately before use. This will minimize problems and ensure more consistent results.



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

Catalog No.: 30475B Lot No.: A031415
 Description: 8260B Calibration Mix #2
 Expiration Date¹: January 2008 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³ (weight/volume)	Percent Uncertainty ⁴
1	2-Chloroethyl vinyl ether	110-75-8	99%	2000 ug/mL	+/- 0.04
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

det. Temp:
250°C

Det. Type:
FID

Manufactured By: JAM

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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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

Catalog No.: 30465 Lot No.: A031618

Description: California Oxygenates Mixture #1

Expiration Date¹: April 2007 Storage: Freezer

Julien Order	Compound	CAS#	Percent Purity ²	Concentration (weight/volume) ³	Percent Uncertainty ⁴
1	tert-Butanol (TBA)	75-65-0	99%	10000 ug/mL	+/- 0.1
2	Methyl-tert-butyl ether (MTBE)	1634-04-4	99%	2000 ug/mL	+/- 0.1
3	Diisopropyl ether (DIPE)	108-20-3	99%	2000 ug/mL	+/- 0.1
4	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	2000 ug/mL	+/- 0.1
5	tert-Amyl methyl ether (TAME)	994-05-8	99%	2000 ug/mL	+/- 0.1
Solvent: P&T Methanol		67-56-1	99%		

Column:
 105m x .32mm x 1.8um
 Rtx-502.2 (cat.#10921)

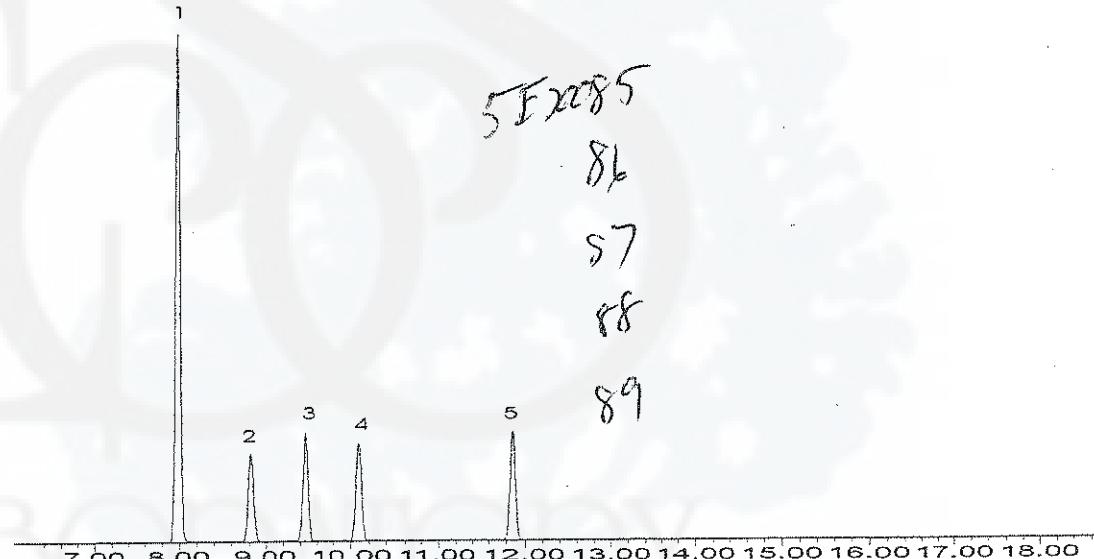
Carrier Gas:
 helium @ 2.2 ml/min.

Temp. Program:
 40°C (hold 2 min.) to 175°C
 @ 8°C/min.

J. Temp:
 200°C

Det. Temp:
 250°C

Det. Type:
 MSD



Manufactured By: MEW

John Lidgett
 John Lidgett - Q.A. 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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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

Catalog No.: 30499 Lot No.: A037729
Description: Acrolein Standard
Expiration Date¹: October 2005 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity ²	Concentration ³	Percent Uncertainty ⁴
1	Acrolein	107-02-8	99%	10000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um Rtx-502.2 (cat.#10910)

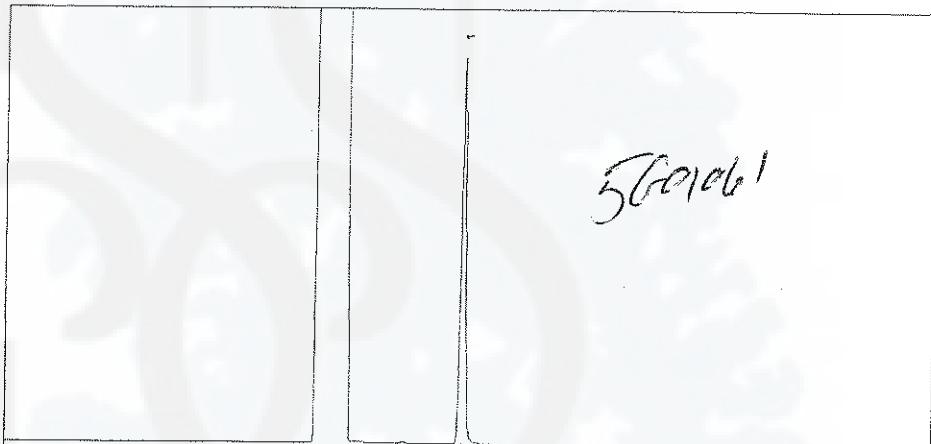
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min.) to 200°C @ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



1 2 3 4 5 6 7 8 9 10 11 12

Manufactured By: n/a

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 addition 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 preparation with balance calibration verified using NISTtraceable weights (7 mass levels).

⁴ Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



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Certificate #FM80397

Certificate of Analysis

Internal Standard Mixture

Product STM-520
Lot Number: CA-2291

Expiration Date: Jan-2008
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
chlorobenzene-d5	003114-55-4	PR-12098	2509 ± 13 µg/mL
1,4-dichlorobenzene-d4	003855-82-1	16304CA	2503 ± 13 µg/mL
fluorobenzene	000462-06-6	CS10731MG	2507 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

5H01052
53
54
55
56
57
58
59

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.



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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

Method 8260 Surrogate Standard Mixture

Product STM-530
Lot Number: CB-1395

Expiration Date: Jul-2008
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
4-bromofluorobenzene	000460-00-4	11203KS	2503 ± 13 µg/mL
dibromofluoromethane	001868-53-7	90004843	2506 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO2F-623	2508 ± 13 µg/mL
toluene-d8	002037-26-5	PSOAG-433	2506 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

5 Holo 1
62
63
64
65
66
67
68

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.



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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

Custom Standard

Product Number: CUS-6492

Expiration Date: Apr-2007

Lot Number: CB-0748

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
diisopropyl ether	000108-20-3	02151KQ	2005 ± 10 µg/mL
ethyl tert-butyl ether	000637-92-3	MN-17917	2006 ± 10 µg/mL
TAME	000994-05-8	JN-12512EN	2005 ± 10 µg/mL
1-chlorohexane	000544-10-5	232-8B	2008 ± 10 µg/mL
cyclohexane	000110-82-7	380707	2005 ± 10 µg/mL
methyl acetate	000079-20-9	I-136A	2004 ± 10 µg/mL
methylcyclohexane	000108-87-2	DR-00844CR	2005 ± 10 µg/mL
1,1,2-trichlorotrifluoroethane	000076-13-1	KN-09246KN	2005 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

5005d0
61
62
63
64

65
66 67

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.



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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

VOC Mixture

Product Number: DWM-592

Expiration Date: May-2007

Lot Number: W-0239

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
acetone	000067-64-1	43034	2008 ± 10 µg/mL
acrylonitrile	000107-13-1	HI11505EI	2009 ± 10 µg/mL
allyl chloride	000107-05-1	MC070777	2009 ± 10 µg/mL
2-butanone	000078-93-3	CA-02161PO	2010 ± 10 µg/mL
carbon disulfide	000075-15-0	AI12076LU	2008 ± 10 µg/mL
chloroacetonitrile	000107-14-2	CF01026AF	2005 ± 10 µg/mL
1-chlorobutane	000109-69-3	07247HF	2007 ± 10 µg/mL
trans-1,4-dichloro-2-butene	000110-57-6	JN01422DG	2008 ± 10 µg/mL
1,1-dichloro-2-propanone	000513-88-2	LY02111DL	2007 ± 10 µg/mL
diethyl ether	000060-29-7	MA01256EA	2008 ± 10 µg/mL
ethyl methacrylate	000097-63-2	AO09819AS	2008 ± 10 µg/mL
hexachloroethane	000067-72-1	DN06203HF	2005 ± 10 µg/mL
2-hexanone	000591-78-6	AI11228KS	2009 ± 10 µg/mL
methacrylonitrile	000126-98-7	LT00427ET	2009 ± 10 µg/mL
methyl acrylate	000096-33-3	HF01125BF	2008 ± 10 µg/mL
methyl iodide	000074-88-4	KA20329DA	2008 ± 10 µg/mL
methyl methacrylate	000080-62-6	HN03906DN	2007 ± 10 µg/mL
4-methyl-2-pentanone	000108-10-1	05756LQ	2007 ± 10 µg/mL
tert-butylmethyl ether	001634-04-4	36032	2009 ± 10 µg/mL
nitrobenzene	000098-95-3	HK2925LK	2008 ± 10 µg/mL
2-nitropropane	000079-46-9	BZ01727PY	2004 ± 10 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.



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Senior Scientist

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Certificate of Analysis**VOC Mixture****Product Number:** DWM-592**Expiration Date:** May-2007**Lot Number:** W-0239**Page:** 2 of 2

Analyte	CAS#	Analyte Lot	True Value
pentachloroethane	000076-01-7	232-36B	2007 ± 10 µg/mL
propionitrile	000107-12-0	BI18125KS	2008 ± 10 µg/mL
tetrahydrofuran	000109-99-9	EN02837AN	2008 ± 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.



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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

VOC Mixture

Product Number: DWM-588

Expiration Date: Apr-2008

Lot Number: CB-0526

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
bromochloromethane	000074-97-5	JS-16015HS	2006 ± 10 µg/mL
bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 µg/mL
bromoform	000075-25-2	DU-06126KS	2004 ± 10 µg/mL
carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
chloroform	000067-66-3	BS-03041BS	2005 ± 10 µg/mL
dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
dibromomethane	000074-95-3	EM-01514TJ	2005 ± 10 µg/mL
methylene chloride	000075-09-2	43041	2006 ± 10 µg/mL
trichlorofluoromethane	000075-69-4	DR-16417BR	2007 ± 10 µg/mL
1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethane	000075-34-3	64552/1	2005 ± 10 µg/mL
1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
1,1-dichloroethene	000075-35-4	CA-02207CA	2005 ± 10 µg/mL
cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
1,1,1,2-tetrachloroethane	000630-20-6	CO-12312LI	2005 ± 10 µg/mL
1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
1,1,1-trichloroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL
1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene	000079-01-6	KN-08846KN	2005 ± 10 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.



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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

VOC Mixture

Product Number: DWM-588

Expiration Date: Apr-2008

Lot Number: CB-0526

Page: 2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2006 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2006 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2007 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34257-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	PB-082017	2006 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	EY-01328EY	2006 ± 10 µg/mL
naphthalene	000091-20-3	N960012	2006 ± 10 µg/mL
benzene	000071-43-2	31072	2005 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2006 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2005 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2006 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2005 ± 10 µg/mL
styrene	000100-42-5	MQ-11228MQ	2005 ± 10 µg/mL
toluene	000108-88-3	34117	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2005 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CI	2005 ± 10 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.



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Certificate of Analysis

VOC Mixture

Product Number: DWM-588

Expiration Date: Apr-2008

Lot Number: CB-0526

Page: 3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2004 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2006 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2005 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2008 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2008 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 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.



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AccuStandard Inc.

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www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO. M-601C-10X

DESCRIPTION: 2-Chloroethylvinyl ether
LOT: B4100153
SOLVENT: MeOH

EXPIRATION: Oct 20, 2007

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200 μ g/mL. For concentrations greater than 200 μ g/mL the uncertainty is <2%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration ¹ (μ g/mL)	Analyte Concentration ² (μ g/mL)
2-Chloroethylvinyl ether	110-75-8	99.7	2000	1994

5E70098

99

100

101

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480.
2. Analyte Concentration = Purity x Gravimetric Concentration
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/NO-001
Rev. 11/02



AccuStandard Inc.

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CATALOG NO. APP-9-007-10X

CERTIFICATE OF ANALYSIS

DESCRIPTION: Acrolein

LOT: B5060041

SOLVENT: MeOH

EXPIRATION: Oct 5, 2005

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200 μ g/mL. For concentrations greater than 200 μ g/mL, the uncertainty is <2%.

Component	CAS #	Purity %	Gravimetric Concentration ¹ (μ g/mL)	Analyte Concentration ² (μ g/mL)
Acrolein	107-02-8	90.0	1112*	1001

560638

39

* Weight compensated to 100% purity

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
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

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CERTIFICATE OF ANALYSIS

CATALOG NO. APP-9-211-20X

DESCRIPTION: Vinyl acetate

LOT: B5020138-1A

SOLVENT: MeOH

EXPIRATION: Nov 20, 2005

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200 μ g/mL. For concentrations greater than 200 μ g/mL the uncertainty is <2%.

Component	CAS #	Purity % (GC/FID)	Gravimetric Concentration ¹ (μ g/mL)	Analyte Concentration ² (μ g/mL)
Vinyl acetate	108-05-4	99.9	2001	1999

5606040

41

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
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

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Certificate of Analysis

1,4-Dioxane

Product Number: RCC-180

Expiration Date: Jan-2008

Lot Number: NT02237

Page: 1 of 1

This reference material has been analyzed by high resolution gas chromatography or high performance liquid chromatography, and found to meet the specifications stated below. The uncertainty of the purity measurement is $\pm 0.5\%$.

Compound	CAS #	Purity
1,4-dioxane	000123-91-1	99%

Storage: May be stored at room temperature

5G11049

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Dr. Edward Fitzgerald,
Senior Scientist

Certificate of Analysis

DESCRIPTION: 1,4-Dioxane

MFG. DATE: Nov 2004

CATALOG NO.: 442251 (1)

LOT NO.: LB25729

EXP. DATE: Nov 2007

CAS NUMBER: 123-91-1

MOLECULAR FORMULA: C₄H₈O₂

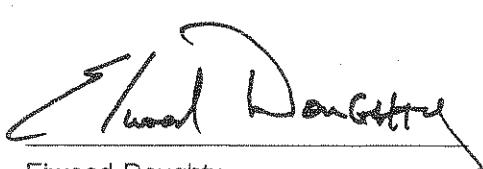
MOLECULAR WEIGHT: 88.1

PHYSICAL PROPERTIES ASSAY

FTIR	Matches: SEA 207	Lib. No.: 1563
GC - Mass Spec	Matches: NIST	Lib. No.: 62899
Purity (2)	99.9%	
Refractive index	1.4213 @ 20.0 deg C	

5C04061

- (1) This product is packaged from R472225 Lot number LB11219.
(2) Determined by GC-FID unless otherwise noted.



Eiwood Doughty
Quality Control Supervisor

Supeco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

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TOLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\AQ080505.M

Method Sections To Run:

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

Method Comments:

Method 8260

MS-3

END OF TOLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Rear
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	200 C
Detector A:	Off	50 C
Detector B:	On	280 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.20 minutes
Oven Max:	260 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	40 C
Initial Time:	4.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	8.0	150	2.00
2 (A)	35.0	220	4.25
3 (B)	0.0	0	0.00

Next Run Time: 26.00 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: On
Constant Flow Pressure: 19.4 psi
Constant Flow Temperature: 40 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: 60.00 m
Column Diameter: 0.250 mm
Gas: He
Vacuum Compensation: Off

HP5890 Inlet Split Flow Control

Channel C:

Carrier gas supply for channel C is not linked to a split vent.

Channel D:

Carrier gas supply for channel D is not linked to a split vent.

Channel E:

Carrier gas supply for channel E is not linked to a split vent.

Channel F:

Carrier gas supply for channel F is not linked to a split vent.

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Auxiliary Pressure Programs

Channel C:

Comment:

Pressure Program:

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	

Make-up Gas Compensation: None

Channel D:

Comment:

Pressure Program:

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	

Make-up Gas Compensation: None

Channel E:

Comment:

Pressure Program:

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	

Make-up Gas Compensation: None

Channel F:

Comment:

Pressure Program:

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	

Make-up Gas Compensation: None

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.00	0.00	No
B	On	0.00	0.00	No

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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	Testplot	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 : BFB.U
Acquisition Mode : Scan

MS Information

Solvent Delay : 3.70 min

EM Absolute : True
Resulting EM Voltage : 1941.2

[Scan Parameters]

Low Mass : 35
High Mass : 260
Threshold : 800
Sample # : 4 A/D Samples 16
Plot 2 low mass : 50
Plot 2 high mass : 550

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

Uncontrolled Document

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\AQ080505.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
c:\database\nbs75k.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: Summary

Output Destination

Screen: No
Printer: No
File: No

Generate Report During Run Method: Yes

ELEMENT ID: 0508010

Calibration Last Updated: Tue Aug 30 15:15:34 2005

Reference Window: 10.00 Percent

Non-Reference Window: 5.00 Percent

Method: AQ080505.M

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Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

1) Fluorobenzene (ISTD)

Ret. Time 12.61 min., Extract & Integrate from 12.11 to 13.11 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	96.00		*** METH DEFAULT ***
Q1	70.00	16.80	30.0 *** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	1793239
10	25.000	1801571
5	25.000	1838338
1	25.000	1704091
50	25.000	1818145
100	25.000	1899842
0.5	25.000	1716216

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l
Curve Fit: Avg. RF

2) Dichlorodifluoromethane ()

Ret. Time 4.15 min., Extract & Integrate from 3.65 to 4.65 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	85.00		*** METH DEFAULT ***
Q1	87.00	33.20	30.0 *** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	416355
10	10.000	172274
5	5.000	90302
1	1.000	18792
50	50.000	836422
100	100.000	1823963
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

3) Chloromethane ()

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	50.00		*** METH DEFAULT ***
Q1	52.00	33.10	30.0 *** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	597482
10	10.000	266628
5	5.000	137597
1	1.000	30884
50	50.000	1203850
100	100.000	2480172
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

4) Vinyl Chloride ()

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	62.00		*** METH DEFAULT ***
Q1	64.00	38.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	437518
10		10.000	192044
5		5.000	101731
1		1.000	19224
50		50.000	882458
100		100.000	1837800
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

5) Bromomethane ()

Ret. Time 5.47 min., Extract & Integrate from 4.97 to 5.97 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	94.00		*** METH DEFAULT ***
Q1	96.00	95.80	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	301032
10		10.000	135055
5		5.000	77283
1		1.000	18213
50		50.000	649844
100		100.000	1436601
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

6) Chloroethane ()

Ret. Time 5.72 min., Extract & Integrate from 5.22 to 6.22 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	64.00		*** METH DEFAULT ***
Q1	66.00	32.00	*** METH DEFAULT ***
Q2	49.00	32.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	256692
10		10.000	112808
5		5.000	57979
1		1.000	16574
50		50.000	495501
100		100.000	1066476
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

7) Trichlorofluoromethane ()

Ret. Time 6.68 min., Extract & Integrate from 6.18 to 7.18 min.

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Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 101.00 67.50 30.0 *** METH DEFAULT ***
Q1 103.00

Lvl ID Conc (ug/l) Response
25 25.000 399557
10 10.000 165001
5 5.000 81556
1 1.000 19818
50 50.000 799662
100 100.000 1678945
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

8) Diethyl ether ()

Ret. Time 7.09 min., Extract & Integrate from 6.59 to 7.59 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 59.00 *** METH DEFAULT ***
Q1 45.00 102.20 30.0 *** METH DEFAULT ***
Q2 74.00 63.70 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 220431
10 10.000 102527
5 5.000 52949
1 1.000 18591
50 50.000 468962
100 100.000 1051883
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Linear

9) Acrolein ()

Ret. Time 6.68 min., Extract & Integrate from 6.18 to 7.18 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 56.00 *** METH DEFAULT ***
Q1 55.00 71.60 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 42726
10 10.000 18902
5 5.000 -1
1 1.000 -1
50 50.000 72056
100 100.000 165907
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

10) Acetone ()

Ret. Time 6.88 min., Extract & Integrate from 6.38 to 7.38 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 58.00 *** METH DEFAULT ***
Q1 43.00 388.40 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 125.000 99057
10 50.000 47222
5 25.000 30787
1 5.000 -1
50 250.000 176366
100 500.000 382429
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Linear

11) Iodomethane ()

Ret. Time 7.60 min., Extract & Integrate from 7.10 to 8.10 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	142.00		*** METH DEFAULT ***
Q1	126.90	54.90	*** METH DEFAULT ***
Q2	141.00	15.60	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 500804
10 10.000 206208
5 5.000 106626
1 1.000 31182
50 50.000 1120745
100 100.000 2489139
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Linear

12) 1,1,2-Trichloro-1,2,2-trifluoroethane ()

Ret. Time 7.88 min., Extract & Integrate from 7.38 to 8.38 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	101.00		*** METH DEFAULT ***
Q1	151.00	59.40	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 331875
10 10.000 145444
5 5.000 87959
1 1.000 16874
50 50.000 695665
100 100.000 1381718
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

13) Methyl Acetate ()

Ret. Time 7.90 min., Extract & Integrate from 7.40 to 8.40 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	43.00		*** METH DEFAULT ***
Q1	74.00	14.90	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 226125
10 10.000 108222
5 5.000 60912
1 1.000 -1

50 50.000 467109
100 100.000 960526
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg., RF

14) Allyl Chloride ()

Ret. Time 7.94 min., Extract & Integrate from 7.44 to 8.44 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	41.00		*** METH DEFAULT ***
Q1	76.00	25.20	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	671399
10		10.000	288531
5		5.000	162257
1		1.000	36376
50		50.000	1561946
100		100.000	3343008
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg., RF

15) Carbon Disulfide ()

Ret. Time 8.12 min., Extract & Integrate from 7.62 to 8.62 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	76.00		*** METH DEFAULT ***
Q1	78.00	9.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1537785
10		10.000	650059
5		5.000	331521
1		1.000	70014
50		50.000	3411160
100		100.000	7456603
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg., RF

16) 1,1-Dichloroethene ()

Ret. Time 7.55 min., Extract & Integrate from 7.05 to 8.05 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	96.00		*** METH DEFAULT ***
Q1	61.00	114.60	*** METH DEFAULT ***
Q2	63.00	37.30	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	300278
10		10.000	128316
5		5.000	62540
1		1.000	20157
50		50.000	621349
100		100.000	1302793
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

17) Methylene Chloride ()

Ret. Time : 7.79 min., Extract & Integrate from 7.29 to 8.29 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	84.00		*** METH DEFAULT ***
Q1	86.00	64.60	*** METH DEFAULT ***
Q2	49.00	147.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	359532
10		10.000	154815
5		5.000	83431
1		1.000	23476
50		50.000	726794
100		100.000	1554380
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

18) Methyl tert-Butyl Ether ()

Ret. Time 9.06 min., Extract & Integrate from 8.56 to 9.56 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	73.00		*** METH DEFAULT ***
Q1	57.00	25.90	*** METH DEFAULT ***
Q2	41.00	29.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	800869
10		10.000	335324
5		5.000	149753
1		1.000	31545
50		50.000	1817014
100		100.000	4152248
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

19) Acrylonitrile ()

Ret. Time 7.66 min., Extract & Integrate from 7.16 to 8.16 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	53.00		*** METH DEFAULT ***
Q1	52.00	82.60	*** METH DEFAULT ***
Q2	51.00	38.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	92809
10		10.000	44811
5		5.000	23381
1		1.000	-1
50		50.000	190253
100		100.000	405352
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

20) trans-1,2-Dichloroethene ()

Ret. Time 8.86 min., Extract & Integrate from 8.36 to 9.36 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	96.00		*** METH DEFAULT ***
Q1	61.00	113.30	*** METH DEFAULT ***
Q2	98.00	66.50	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	473996
10		10.000	200838
5		5.000	97241
1		1.000	18775
50		50.000	1052229
100		100.000	2340829
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

21) 1,1-Dichloroethane ()

Ret. Time 9.25 min., Extract & Integrate from 8.75 to 9.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	63.00		*** METH DEFAULT ***
Q1	65.00	32.60	*** METH DEFAULT ***
Q2	83.00	14.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1084415
10		10.000	469088
5		5.000	228337
1		1.000	50777
50		50.000	2235019
100		100.000	4726697
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

22) Vinyl Acetate ()

Ret. Time 9.49 min., Extract & Integrate from 8.99 to 9.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00		*** METH DEFAULT ***
Q1	86.00	7.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	969560
10		10.000	398756
5		5.000	192408
1		1.000	39563
50		50.000	2294813
100		100.000	5426019
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

23) Chloroprene ()

Ret. Time 9.83 min., Extract & Integrate from 9.33 to 10.33 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	53.00		*** METH DEFAULT ***
Q1	88.00	66.80	*** METH DEFAULT ***
Lvl ID	Conc (ug/l)	Response	
25	25.000	730206	
10	10.000	278040	
5	5.000	133241	
1	1.000	27007	
50	50.000	1598316	
100	100.000	3503002	
0.5	not used for this compound		

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

24) 2-Butanone ()

Ret. Time 9.95 min., Extract & Integrate from 9.45 to 10.45 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	72.00		*** METH DEFAULT ***
Q1	43.00	695.40	*** METH DEFAULT ***
Lvl ID	Conc (ug/l)	Response	
25	125.000	143841	
10	50.000	62943	
5	25.000	33898	
1	5.000	-1	
50	250.000	307714	
100	500.000	668126	
0.5	not used for this compound		

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

25) Di-isopropyl ether ()

Ret. Time 9.98 min., Extract & Integrate from 9.48 to 10.48 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	45.00		*** METH DEFAULT ***
Q1	43.00	48.10	*** METH DEFAULT ***
Q2	87.00	16.50	*** METH DEFAULT ***
Lvl ID	Conc (ug/l)	Response	
25	25.000	2337668	
10	10.000	942710	
5	5.000	426010	
1	1.000	76440	
50	50.000	4992657	
100	100.000	10505860	
0.5	not used for this compound		

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

26) Methacrylonitrile ()

Ret. Time 10.10 min., Extract & Integrate from 9.60 to 10.60 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	41.00		*** METH DEFAULT ***
Q1	67.00	42.30	*** METH DEFAULT ***
Lvl ID	Conc (ug/l)	Response	

25 25.000 281255
10 10.000 123283
5 5.000 56666
1 1.000 8378
50 50.000 615653
100 100.000 1310989
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

27) cis-1,2 Dichloroethene ()

Ret. Time 10.16 min., Extract & Integrate from 9.66 to 10.66 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	96.00		*** METH DEFAULT ***
Q1	61.00	95.50	*** METH DEFAULT ***
Q2	98.00	66.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	558271
10		10.000	219798
5		5.000	109332
1		1.000	21754
50		50.000	1147128
100		100.000	2455295
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

28) Methyl Acrylate ()

Ret. Time 10.59 min., Extract & Integrate from 10.09 to 11.09 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	55.00		*** METH DEFAULT ***
Q1	85.00	18.50	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	374149
10		10.000	160341
5		5.000	81772
1		1.000	17877
50		50.000	777455
100		100.000	1646033
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

29) Ethyl tertiary-butyl ether ()

Ret. Time 10.60 min., Extract & Integrate from 10.10 to 11.10 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	59.00		*** METH DEFAULT ***
Q1	87.00	41.30	*** METH DEFAULT ***
Q2	57.00	32.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1452475
10		10.000	584977
5		5.000	267663
1		1.000	46902

50 50.000 3036504
100 100.000 6314530
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

30) 2,2-Dichloropropane ()

Ret. Time 10.60 min., Extract & Integrate from 10.10 to 11.10 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	77.00		*** METH DEFAULT ***
Q1	97.00	28.60	*** METH DEFAULT ***
Q2	41.00	112.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	710782
10		10.000	293585
5		5.000	146387
1		1.000	30152
50		50.000	1349364
100		100.000	2709502
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

31) Bromochloromethane ()

Ret. Time 10.39 min., Extract & Integrate from 9.89 to 10.89 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	128.00		*** METH DEFAULT ***
Q1	49.00	140.90	*** METH DEFAULT ***
Q2	130.00	130.70	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	249703
10		10.000	106943
5		5.000	49929
1		1.000	11192
50		50.000	535335
100		100.000	1137973
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

32) Tetrahydrofuran ()

Ret. Time 11.00 min., Extract & Integrate from 10.50 to 11.50 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	42.00		*** METH DEFAULT ***
Q1	72.00	28.80	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	88497
10		10.000	39414
5		5.000	22651
1		1.000	-1
50		50.000	174067
100		100.000	441670
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

33) Chloroform ()

Ret. Time 10.48 min., Extract & Integrate from 9.98 to 10.98 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	83.00		*** METH DEFAULT ***
Q1	85.00	67.60	*** METH DEFAULT ***
Q2	47.00	26.00	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	995164
10		10.000	420641
5		5.000	198177
1		1.000	46237
50		50.000	1982965
100		100.000	4108998
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

34) Dibromofluoromethane(SURR) ()

Ret. Time 10.66 min., Extract & Integrate from 10.16 to 11.16 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration	
Tgt	111.00		*** METH DEFAULT ***	
Q1	113.00	98.00	30.0	*** METH DEFAULT ***
Q2	192.00	9.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	561295
10		10.000	234152
5		5.000	115870
1		1.000	25783
50		50.000	1136923
100		100.000	2389781
0.5		0.500	13072

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

35) 1-Chlorobutane ()

Ret. Time 11.63 min., Extract & Integrate from 11.13 to 12.13 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration	
Tgt	56.00		*** METH DEFAULT ***	
Q1	41.00	71.30	30.0	*** METH DEFAULT ***
Q2	43.00	34.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1098968
10		10.000	440748
5		5.000	202368
1		1.000	39169
50		50.000	2282885
100		100.000	4790439
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

36) 1,1,1-Trichloroethane ()

Ret. Time 11.64 min., Extract & Integrate from 11.14 to 12.14 min.

Signal Rel Resp. Pct. Unc. (abs) Integration

Tgt	97.00		*** METH DEFAULT ***	
Q1	99.00	66.20	30.0	*** METH DEFAULT ***
Q2	61.00	34.90	30.0	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 721155
10 10.000 293809
5 5.000 147853
1 1.000 30957
50 50.000 1440140
100 100.000 2962268
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

37) 1,1-Dichloropropene ()

Ret. Time 11.94 min., Extract & Integrate from 11.44 to 12.44 min.

Signal Rel Resp. Pct. Unc. (abs) Integration

Tgt	75.00		*** METH DEFAULT ***	
Q1	110.00	50.40	30.0	*** METH DEFAULT ***
Q2	77.00	31.70	30.0	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 632648
10 10.000 249432
5 5.000 115808
1 1.000 23846
50 50.000 1371122
100 100.000 2910338
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

38) Cyclohexane ()

Ret. Time 12.07 min., Extract & Integrate from 11.57 to 12.57 min.

Signal Rel Resp. Pct. Unc. (abs) Integration

Tgt	56.00		*** METH DEFAULT ***	
Q1	84.00	79.60	30.0	*** METH DEFAULT ***
Q2	41.00	70.50	30.0	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 907077
10 10.000 355664
5 5.000 169423
1 1.000 35456
50 50.000 1863476
100 100.000 3891957
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

39) Carbon Tetrachloride ()

Ret. Time 12.21 min., Extract & Integrate from 12.01 to 12.41 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	117.00		*** METH DEFAULT ***
Q1	119.00	95.40	*** METH DEFAULT ***
Q2	121.00	32.30	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	535324
10	10.000	216339
5	5.000	101142
1	1.000	21638
50	50.000	1109861
100	100.000	2361524
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

40) Benzene ()

Ret. Time 12.28 min., Extract & Integrate from 11.78 to 12.78 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	78.00		*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	1904939
10	10.000	784884
5	5.000	378428
1	1.000	70104
50	50.000	3854560
100	100.000	8180736
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

41) 1,2-Dichloroethane-d4 (SURR) ()

Ret. Time 11.38 min., Extract & Integrate from 10.88 to 11.88 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	65.00		*** METH DEFAULT ***
Q1	102.00	38.00	30.0
			*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	589788
10	10.000	252323
5	5.000	131353
1	1.000	31041
50	50.000	1195026
100	100.000	2489303
0.5	0.500	14936

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

42) 1,2-Dichloroethane ()

Ret. Time 11.49 min., Extract & Integrate from 11.29 to 11.69 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	62.00		*** METH DEFAULT ***
Q1	98.00	15.80	30.0
Q2	49.00	46.90	30.0
			*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 728730
10 10.000 312418
5 5.000 158587
1 1.000 36484
50 50.000 1471334
100 100.000 3064482
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

43) Tertiary-amyl methyl ether ()

Ret. Time 12.55 min., Extract & Integrate from 12.05 to 13.05 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	73.00		*** METH DEFAULT ***
Q1	43.00	42.10	*** METH DEFAULT ***
Q2	55.00	23.60	*** METH DEFAULT ***
Q3	87.00	26.10	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 909250
10 10.000 381598
5 5.000 178280
1 1.000 28156
50 50.000 1984661
100 100.000 4429119
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

44) Trichloroethene ()

Ret. Time 13.29 min., Extract & Integrate from 12.79 to 13.79 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	95.00		*** METH DEFAULT ***
Q1	97.00	66.10	*** METH DEFAULT ***
Q2	130.00	130.10	*** METH DEFAULT ***
Q3	132.00	124.60	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 525298
10 10.000 209767
5 5.000 101671
1 1.000 20367
50 50.000 1098688
100 100.000 2293127
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

45) 1,2-Dichloropropane ()

Ret. Time 13.22 min., Extract & Integrate from 12.72 to 13.72 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	63.00		*** METH DEFAULT ***
Q1	112.00	6.00	*** METH DEFAULT ***
Q2	61.00	14.20	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 612692
10 10.000 260437
5 5.000 122156
1 1.000 28518
.50 50.000 1253960
100 100.000 2644077
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

46) Dibromomethane ()

Ret. Time 13.16 min., Extract & Integrate from 12.66 to 13.66 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	93.00		*** METH DEFAULT ***
Q1	95.00	85.70	*** METH DEFAULT ***
Q2	174.00	84.70	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 299812
10 10.000 121141
5 5.000 62564
1 1.000 14370
50 50.000 632274
100 100.000 1349994
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

47) 2-Nitropropane ()

Ret. Time 13.31 min., Extract & Integrate from 12.81 to 13.81 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	43.00		*** METH DEFAULT ***
Q1	41.00	89.50	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 115216
10 10.000 46205
5 5.000 26023
1 1.000 4671
50 50.000 238414
100 100.000 420606
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

48) Bromodichloromethane ()

Ret. Time 13.35 min., Extract & Integrate from 12.85 to 13.85 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	83.00		*** METH DEFAULT ***
Q1	85.00	62.60	*** METH DEFAULT ***
Q2	127.00	12.70	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 699569
10 10.000 281122
5 5.000 129756

1 1.000 30183
50 50.000 1384589
100 100.000 2844463
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

49) 1,4-Dioxane ()

Ret. Time 13.56 min., Extract & Integrate from 13.06 to 14.06 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	88.00		*** METH DEFAULT ***
Q1	58.00	56.10	*** METH DEFAULT ***
Q2	43.00	70.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		500.000	33325
10		200.000	22779
5		100.000	11540
1		20.000	6822
50		1000.000	74642
100		1500.000	187314
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

50) Methyl Methacrylate ()

Ret. Time 13.62 min., Extract & Integrate from 13.12 to 14.12 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	41.00		*** METH DEFAULT ***
Q1	69.00	57.10	*** METH DEFAULT ***
Q2	100.00	31.30	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	426428
10		10.000	161949
5		5.000	74582
1		1.000	-1
50		50.000	976712
100		100.000	2135220
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

51) 2-Chloroethyl vinyl ether ()

Ret. Time 14.04 min., Extract & Integrate from 13.54 to 14.54 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	63.00		*** METH DEFAULT ***
Q1	65.00	30.70	*** METH DEFAULT ***
Q2	106.00	34.60	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	1201319
10		50.000	472654
5		25.000	194776
1		5.000	-1
50		250.000	2677002
100		500.000	6006774

0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

52) Methyl Cyclohexane ()

Ret. Time 14.10 min., Extract & Integrate from 13.60 to 14.60 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	83.00		*** METH DEFAULT ***
Q1	55.00	85.60	*** METH DEFAULT ***
Q2	98.00	52.50	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	573799
10		10.000	226672
5		5.000	119751
1		1.000	19461
50		50.000	1188169
100		100.000	2513523
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

53) 4-Methyl-2-Pentanone ()

Ret. Time 14.59 min., Extract & Integrate from 14.39 to 14.79 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	58.00		*** METH DEFAULT ***
Q1	43.00	312.30	30.0 *** METH DEFAULT ***
Q2	85.00	38.90	30.0 *** METH DEFAULT ***
Q3	100.00	43.10	30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	628870
10		50.000	261111
5		25.000	114609
1		5.000	29151
50		250.000	1393379
100		500.000	3030687
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

54) cis-1,3-Dichloropropene ()

Ret. Time 14.39 min., Extract & Integrate from 13.89 to 14.89 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	75.00		*** METH DEFAULT ***
Q1	77.00	31.40	30.0 *** METH DEFAULT ***
Q2	39.00	53.80	30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	677818
10		10.000	257094
5		5.000	120916
1		1.000	23281
50		50.000	1483726
100		100.000	3268559
0.5		0.500	11596

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

55) trans-1,3-Dichloropropene ()

Ret. Time 15.08 min., Extract & Integrate from 14.58 to 15.58 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	75.00		*** METH DEFAULT ***
Q1	77.00	31.90	*** METH DEFAULT ***
Q2	39.00	61.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	553563
10		10.000	216459
5		5.000	91643
1		1.000	22578
50		50.000	1252755
100		100.000	2813004
0.5		0.500	7469

Qualifier Peak Analysis ON
Curve Fit: Linear

56) 1,1,2-Trichloroethane ()

Ret. Time 15.33 min., Extract & Integrate from 14.83 to 15.83 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration	
Tgt	83.00		*** METH DEFAULT ***	
Q1	97.00	126.30	30.0	*** METH DEFAULT ***
Q2	85.00	62.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	325064
10		10.000	139694
5		5.000	69384
1		1.000	13659
50		50.000	682634
100		100.000	1473009
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

57) Toluene ()

Ret. Time 15.66 min., Extract & Integrate from 15.16 to 16.16 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration	
Tgt	92.00		*** METH DEFAULT ***	
Q1	91.00	160.60	30.0	*** METH DEFAULT ***
Q2	65.00	14.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1086020
10		10.000	439079
5		5.000	197248
1		1.000	35619
50		50.000	2260646
100		100.000	4739947
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

58) Chlorobenzene-d5

(ISTD)

Ret. Time 17.92 min., Extract & Integrate from 17.42 to 18.42 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 117.00 *** METH DEFAULT ***
Q1 119.00 31.70 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1149114
10 25.000 1152088
5 25.000 1198151
1 25.000 1051571
50 25.000 1180885
100 25.000 1210800
0.5 25.000 1056414

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l

Curve Fit: Avg. RF

59) Toluene-d8 (SURR)

()

Ret. Time 15.54 min., Extract & Integrate from 15.04 to 16.04 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 98.00 *** METH DEFAULT ***
Q1 100.00 72.40 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1457463
10 10.000 566890
5 5.000 248627
1 1.000 44328
50 50.000 3183076
100 100.000 6956506
0.5 0.500 20355

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

60) Ethyl Methacrylate

()

Ret. Time 15.79 min., Extract & Integrate from 15.29 to 16.29 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 69.00 *** METH DEFAULT ***
Q1 41.00 91.80 30.0 *** METH DEFAULT ***
Q2 99.00 27.60 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 317915
10 10.000 120387
5 5.000 54326
1 1.000 -1
50 50.000 729766
100 100.000 1581816
0.5 not used for this compound

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

61) 2-Hexanone

()

Ret. Time 15.99 min., Extract & Integrate from 15.49 to 16.49 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	43.00		*** METH DEFAULT ***
Q1	58.00	44.90	*** METH DEFAULT ***
Q2	57.00	12.90	*** METH DEFAULT ***
Q3	100.00	9.80	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	1400232
10		50.000	572507
5		25.000	264854
1		5.000	52715
50		250.000	2993528
100		500.000	6284389
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

62) 1,3-Dichloropropane ()

Ret. Time 15.73 min., Extract & Integrate from 15.53 to 15.93 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	76.00		*** METH DEFAULT ***
Q1	78.00	33.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	636645
10		10.000	276904
5		5.000	139114
1		1.000	25367
50		50.000	1328764
100		100.000	2671607
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

63) Tetrachloroethene ()

Ret. Time 16.85 min., Extract & Integrate from 16.35 to 17.35 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration	
Tgt	164.00		*** METH DEFAULT ***	
Q1	129.00	119.50	30.0	*** METH DEFAULT ***
Q2	131.00	111.80	30.0	*** METH DEFAULT ***
Q3	166.00	126.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	377976
10		10.000	148543
5		5.000	73358
1		1.000	16445
50		50.000	804360
100		100.000	1781312
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

64) Dibromochloromethane ()

Ret. Time 16.16 min., Extract & Integrate from 15.66 to 16.66 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	129.00		*** METH DEFAULT ***

Q1 127.00 75.20 30.0
Q2 131.00 24.50 30.0

Lvl ID Conc (ug/l) Response
25 25.000 392387
10 10.000 153553
5 5.000 74019
1 1.000 13781
50 50.000 863711
100 100.000 1921566
0.5 0.500 6279

*** METH DEFAULT ***
*** METH DEFAULT ***

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

65) 1,2-Dibromoethane ()

Ret. Time 16.57 min., Extract & Integrate from 16.07 to 17.07 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	107.00		*** METH DEFAULT ***
Q1	109.00	95.30	*** METH DEFAULT ***
Q2	188.00	1.90	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 353284
10 10.000 146149
5 5.000 70101
1 1.000 12382
50 50.000 796363
100 100.000 1791032
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

66) 1-Chlorohexane ()

Ret. Time 17.86 min., Extract & Integrate from 17.36 to 18.36 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	91.00		*** METH DEFAULT ***
Q1	93.00	33.30	*** METH DEFAULT ***
Q2	55.00	55.90	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 495953
10 10.000 171862
5 5.000 76808
1 1.000 15541
50 50.000 1082560
100 100.000 -1
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

67) Chlorobenzene ()

Ret. Time 17.98 min., Extract & Integrate from 17.48 to 18.48 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	112.00		*** METH DEFAULT ***
Q1	77.00	37.00	*** METH DEFAULT ***
Q2	114.00	32.80	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1174907
10 10.000 465819
5 5.000 228308
1 1.000 50309
50 50.000 2445956
100 100.000 5301175
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

68) 1,1,1,2-Tetrachloroethane ()

Ret. Time 17.85 min., Extract & Integrate from 17.35 to 18.35 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	131.00		*** METH DEFAULT ***
Q1	133.00	93.40	*** METH DEFAULT ***
Q2	119.00	63.20	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 391021
10 10.000 158174
5 5.000 79069
1 1.000 16868
50 50.000 846551
100 100.000 1888814
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

69) Ethylbenzene ()

Ret. Time 18.32 min., Extract & Integrate from 17.82 to 18.82 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00		*** METH DEFAULT ***
Q1	106.00	35.90	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1893065
10 10.000 726723
5 5.000 311946
1 1.000 52033
50 50.000 4103410
100 100.000 8979616
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Linear

70) Xylene P,M ()

Ret. Time 18.66 min., Extract & Integrate from 18.16 to 19.16 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	106.00		*** METH DEFAULT ***
Q1	91.00	170.30	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 50.000 1422204
10 20.000 550441
5 10.000 240023
1 2.000 35277

50 100.000 3044004
100 200.000 6571882
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Linear

71) Xylene O ()

Ret. Time 19.42 min., Extract & Integrate from 18.92 to 19.92 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	106.00		*** METH DEFAULT ***
Q1	91.00	180.90	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	693942
10	10.000	278413
5	5.000	122731
1	1.000	17454
50	50.000	1469054
100	100.000	3137818
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

72) Styrene ()

Ret. Time 19.29 min., Extract & Integrate from 18.79 to 19.79 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	104.00		*** METH DEFAULT ***
Q1	78.00	30.20	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	1099006
10	10.000	397107
5	5.000	160783
1	1.000	24153
50	50.000	2427566
100	100.000	5388566
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

73) Bromoform ()

Ret. Time 18.86 min., Extract & Integrate from 18.36 to 19.36 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	173.00		*** METH DEFAULT ***
Q1	175.00	46.90	*** METH DEFAULT ***
Q2	254.00	4.00	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	247606
10	10.000	94230
5	5.000	44415
1	1.000	9375
50	50.000	557107
100	100.000	1276653
0.5	not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

74) cis-1,4-Dichloro-2-butene ()

Ret. Time 19.11 min., Extract & Integrate from 18.61 to 19.61 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	75.00		*** METH DEFAULT ***
Q1	53.00	79.90	30.0 *** METH DEFAULT ***
Q2	88.00	85.10	30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	70035
10		10.000	31849
5		5.000	14487
1		1.000	3206
50		50.000	177757
100		100.000	-1
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

75) Bromofluorobenzene (SURR) ()

Ret. Time 20.20 min., Extract & Integrate from 19.70 to 20.70 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	95.00		*** METH DEFAULT ***
Q1	174.00	68.90	30.0 *** METH DEFAULT ***
Q2	176.00	63.40	30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	618813
10		10.000	249771
5		5.000	113774
1		1.000	20232
50		50.000	1303299
100		100.000	2673692
0.5		0.500	8751

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

76) 1,4 Dichlorobenzene-D4 (ISTD)

Ret. Time 22.11 min., Extract & Integrate from 21.61 to 22.61 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	152.00		*** METH DEFAULT ***
Q1	115.00	76.60	30.0 *** METH DEFAULT ***
Q2	150.00	156.60	30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	588311
10		25.000	569637
5		25.000	572894
1		25.000	527165
50		25.000	605755
100		25.000	644006
0.5		25.000	505141

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l

Curve Fit: Avg. RF

77) Trans-1,4-Dichloro-2-Butene ()

Ret. Time 19.75 min., Extract & Integrate from 19.25 to 20.25 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	53.00		*** METH DEFAULT ***
Q1	88.00	52.00	*** METH DEFAULT ***
Q2	75.00	147.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	121372
10		10.000	51739
5		5.000	25474
1		1.000	3142
50		50.000	227308
100		100.000	503763
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

78) 1,2,3-Trichloropropane ()

Ret. Time 19.69 min., Extract & Integrate from 19.19 to 20.19 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	75.00		*** METH DEFAULT ***
Q1	77.00	32.10	*** METH DEFAULT ***
Q2	110.00	46.70	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	322230
10		10.000	130377
5		5.000	61639
1		1.000	16957
50		50.000	636122
100		100.000	1399251
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

79) Isopropylbenzene ()

Ret. Time 20.14 min., Extract & Integrate from 19.64 to 20.64 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	105.00		*** METH DEFAULT ***
Q1	120.00	27.20	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1594156
10		10.000	590306
5		5.000	243620
1		1.000	33892
50		50.000	3528560
100		100.000	7744816
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Linear

80) Bromobenzene ()

Ret. Time 20.55 min., Extract & Integrate from 20.05 to 21.05 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 156.00 *** METH DEFAULT ***
Q1 77.00 104.60 30.0 *** METH DEFAULT ***
Q2 158.00 98.30 30.0 *** METH DEFAULT ***

Lvl ID Conc.(ug/l) Response
25 25.000 487021
10 10.000 186519
5 5.000 81412
1 1.000 15101
50 50.000 1085416
100 100.000 2405392
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

81) 1,1,2,2-Tetrachloroethane ()

Ret. Time 19.41 min., Extract & Integrate from 18.91 to 19.91 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 83.00 *** METH DEFAULT ***
Q1 131.00 12.30 30.0 *** METH DEFAULT ***
Q2 85.00 63.10 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 417593
10 10.000 181461
5 5.000 88743
1 1.000 21859
50 50.000 895802
100 100.000 1844826
0.5 0.500 11426

Qualifier Peak Analysis ON
Curve Fit: Linear

82) n-Propylbenzene ()

Ret. Time 20.87 min., Extract & Integrate from 20.37 to 21.37 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 91.00 *** METH DEFAULT ***
Q1 120.00 25.90 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 2113209
10 10.000 787297
5 5.000 337422
1 1.000 49059
50 50.000 4566162
100 100.000 9961710
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Linear

83) 2-Chlorotoluene ()

Ret. Time 21.00 min., Extract & Integrate from 20.50 to 21.50 min.

Signal Rel Resp. Pct. Unc. (abs) Integration
Tgt 91.00 *** METH DEFAULT ***
Q1 126.00 41.80 30.0 *** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1353830
10 10.000 539378
5 5.000 239869
1 1.000 40555
50 50.000 2800815
100 100.000 5954898
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

84) 4-Chlorotoluene ()

Ret. Time 21.12 min., Extract & Integrate from 20.62 to 21.62 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	91.00		*** METH DEFAULT ***
Q1	126.00	42.70	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1420409
10 10.000 558637
5 5.000 249781
1 1.000 39577
50 50.000 2921318
100 100.000 6131601
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

85) 1,3,5-Trimethylbenzene ()

Ret. Time 21.31 min., Extract & Integrate from 20.81 to 21.81 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	105.00		*** METH DEFAULT ***
Q1	120.00	58.70	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 1362287
10 10.000 526400
5 5.000 222983
1 1.000 28916
50 50.000 2882721
100 100.000 6254356
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Linear

86) Pentachloroethane ()

Ret. Time 21.39 min., Extract & Integrate from 20.89 to 21.89 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	119.00		*** METH DEFAULT ***
Q1	117.00	105.30	*** METH DEFAULT ***
Q2	167.00	84.50	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 288430
10 10.000 119191
5 5.000 56051
1 1.000 11799

50 50.000 616309
100 100.000 1338507
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg., RF

87) tert-Butylbenzene ()

Ret. Time 21.68 min., Extract & Integrate from 21.18 to 22.18 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	119.00		*** METH DEFAULT ***
Q1	91.00	52.00	*** METH DEFAULT ***
Q2	134.00	23.50	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1093230
10		10.000	422799
5		5.000	175015
1		1.000	23912
50		50.000	2385444
100		100.000	5211855
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

88) 1,2,4-Trimethylbenzene ()

Ret. Time 21.83 min., Extract & Integrate from 21.33 to 22.33 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	105.00		*** METH DEFAULT ***
Q1	120.00	50.30	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1427065
10		10.000	544152
5		5.000	238792
1		1.000	32487
50		50.000	3002805
100		100.000	6409908
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

89) sec-Butylbenzene ()

Ret. Time 21.98 min., Extract & Integrate from 21.48 to 22.48 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	105.00		*** METH DEFAULT ***
Q1	134.00	20.40	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1552406
10		10.000	623645
5		5.000	266416
1		1.000	39345
50		50.000	3273552
100		100.000	7095447
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

90) 1,3 Dichlorobenzene ()

Ret. Time 22.07 min., Extract & Integrate from 21.57 to 22.57 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	146.00		*** METH DEFAULT ***
Q1	111.00	42.50	*** METH DEFAULT ***
Q2	148.00	64.50	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	868503
10		10.000	352801
5		5.000	158563
1		1.000	34723
50		50.000	1824661
100		100.000	3967013
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

91) 4-Isopropyltoluene ()

Ret. Time 22.22 min., Extract & Integrate from 21.72 to 22.72 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	119.00		*** METH DEFAULT ***
Q1	134.00	22.60	*** METH DEFAULT ***
Q2	91.00	18.70	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1378866
10		10.000	529417
5		5.000	238557
1		1.000	38649
50		50.000	2886552
100		100.000	6026376
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

92) 1,4 Dichlorobenzene ()

Ret. Time 22.16 min., Extract & Integrate from 21.66 to 22.66 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	146.00		*** METH DEFAULT ***
Q1	111.00	41.20	*** METH DEFAULT ***
Q2	148.00	63.70	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	899117
10		10.000	367083
5		5.000	178481
1		1.000	39315
50		50.000	1815588
100		100.000	4043026
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

93) n-Butylbenzene ()

Ret. Time 22.77 min., Extract & Integrate from 22.27 to 23.27 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	91.00		*** METH DEFAULT ***
Q1	92.00	59.50	*** METH DEFAULT ***
Q2	134.00	27.60	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1220286
10		10.000	481734
5		5.000	216979
1		1.000	36480
50		50.000	2534892
100		100.000	5411888
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

94) 1,2 Dichlorobenzene ()

Ret. Time 22.64 min., Extract & Integrate from 22.14 to 23.14 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	146.00		*** METH DEFAULT ***
Q1	111.00	42.90	*** METH DEFAULT ***
Q2	148.00	63.90	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	764113
10		10.000	305758
5		5.000	139625
1		1.000	28586
50		50.000	1623859
100		100.000	3622555
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

95) 1,2-Dibromo-3-Chloropropane ()

Ret. Time 23.26 min., Extract & Integrate from 22.76 to 23.76 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	75.00		*** METH DEFAULT ***
Q1	155.00	129.90	*** METH DEFAULT ***
Q2	157.00	166.10	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	59974
10		10.000	26954
5		5.000	14735
1		1.000	-1
50		50.000	122449
100		100.000	268077
0.5		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

96) Hexachloroethane ()

Ret. Time 23.35 min., Extract & Integrate from 22.85 to 23.85 min.

Method: AQ080505.M

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Signal	Rel Resp.	Pct.	Unc. (abs)	Integration
Tgt	117.00			*** METH DEFAULT ***
Q1	119.00	98.30	30.0	*** METH DEFAULT ***
Q2	201.00	35.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	241403
10		10.000	98031
5		5.000	44965
1		1.000	8703
50		50.000	541780
100		100.000	1239041
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

97) 1,2,4-Trichlorobenzene ()

Ret. Time 25.18 min., Extract & Integrate from 24.68 to 25.68 min.

Signal	Rel Resp.	Pct.	Unc. (abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	88.60	30.0	*** METH DEFAULT ***
Q2	145.00	39.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	450909
10		10.000	185804
5		5.000	83302
1		1.000	17048
50		50.000	995540
100		100.000	2279041
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

98) Hexachlorobutadiene ()

Ret. Time 25.65 min., Extract & Integrate from 25.15 to 26.15 min.

Signal	Rel Resp.	Pct.	Unc. (abs)	Integration
Tgt	225.00			*** METH DEFAULT ***
Q1	223.00	59.00	30.0	*** METH DEFAULT ***
Q2	227.00	59.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	174024
10		10.000	78851
5		5.000	39992
1		1.000	10151
50		50.000	366529
100		100.000	831959
0.5		0.500	3888

Qualifier Peak Analysis ON
Curve Fit: Linear

99) Naphthalene ()

Ret. Time 25.57 min., Extract & Integrate from 25.07 to 26.07 min.

Signal	Rel Resp.	Pct.	Unc. (abs)	Integration
Tgt	128.00			*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 706440
10 10.000 293145
5 5.000 128247
1 1.000 27241
50 50.000 1634760
100 100.000 3893262
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

100) 1,2,3-Trichlorobenzene ()

Ret. Time 25.88 min., Extract & Integrate from 25.38 to 26.38 min.

Signal	Rel Resp.	Pct. Unc. (abs)	Integration
Tgt	180.00		*** METH DEFAULT ***
Q1	182.00	90.30	*** METH DEFAULT ***
Q2	145.00	41.40	*** METH DEFAULT ***

Lvl ID Conc (ug/l) Response
25 25.000 369683
10 10.000 161568
5 5.000 77129
1 1.000 16679
50 50.000 828650
100 100.000 1900077
0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

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ESS LABORATORY MS-3 RUN LOG

Attachment C

Run Sequence Confirmation

Control Number 20.0020-0604A

Control Number 20-0020-0604A
All Standards must be noted with a primary or secondary ID

Surrogate:

On-column |S: _____ Page _____

ESS Laboratory
Cranston, RI

Procedure: 20_8260B R. 10
VOC by GC/MS

ATTACHMENT D

Page _____ of _____

VOA Analysis Map

SYNTHESIS

ESS Work Order/Samples

Comments 2.

VOC Spika II™
Spika Anti-
Final Volumer

1 Q/NQA™ Instr? /
2 All QC SOP compliant unless otherwise noted.

Date: _____

All QC SOP compliant unless otherwise noted.
Control # 20.0027-0901a

Reviewed by: _____ (second review includes a check and approval of manual integrations)

Q:\VOA\VOCAWork_Map.xls