Title: VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA Method 8260B by Selective Ion Monitoring (SIM)

Approvals (Signature/Date):

C. Bernaud for Robert Culver 07/25/13
Robert Culver Date
Department Manager

William Nash 07/25/2013
William Nash Date
Health & Safety Coordinator

7-25-2013
Maria Friedman Date
Quality Assurance Manager

Kirk Miltimore 07/25/13
Kirk Miltimore Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 SCOPE AND APPLICATION

The standard 8260B procedure is used to measure compounds in Selective Ion Monitoring (SIM) mode. Compounds currently quantitated by this method:

The advantage of SIM over full scan spectral acquisition is the increase in sensitivity. In a quadrupole mass spectrometer, this sensitivity enhancement is due to the increased collection time of selected ions. This increased collection time results in more ions striking the electron multiplier. In most cases a tenfold sensitivity increase is possible.

This method is used to determine volatile organic compounds in ground, surface and waste water, aqueous sludge, oily wastes, soils, and sediments. See attached Analysis Information for applicable reporting and control limits. NOTE: See LIMS system for current MDL and Control Limit values.

For work performed under State of Arizona regulations, requirements of EPA 8000C and EPA 5030C must be followed and referenced.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in “Validation of Methods” in the Quality Assurance Manual

2.0 SUMMARY OF METHOD

The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The analytes flow through a capillary column, which is temperature-programmed to optimized separation and are then detected with a mass spectrometer. Unlike standard scan mode 8260 analysis, target compounds are identified and quantitated based on retention time and a single ion mass.

3.0 DEFINITIONS

3.1 Selected Ion Monitoring (SIM) mode is the use of a single ion rather than a multi-ion mass spectrogram for the measuring of target compounds.

3.2 There are no additional specific definitions associated with this test. See the laboratory QA manual and EPA Methods 8000B, 8000C, 8260B, 5030A, 5030B, 5030C, and 5035A for general definitions.

4.0 INTERFERENCES

4.1 The major disadvantage with SIM analysis is the lack of qualitative information. A library search cannot be conducted on a single ion.

4.2 Contamination may occur when a low concentration sample is analyzed immediately after a high concentration sample.

- If the autosampler is contaminated by a sample, analyze a blank. If the target compounds are not present in the blank then analysis may continue.
- If a low concentration sample is analyzed immediately after a high concentration sample, reanalysis of the low concentration sample is necessary if the concentrations are less than 10x the reporting limit. Reanalysis is not necessary if the low concentration sample is N.D. for all target analytes at their reporting limit.

4.3 Contamination may also occur when a sample contains surfactants. Signs of surfactant are foaming and/or bubbling when the sample is purged. After a sample containing surfactants is analyzed, rinse the system carefully. Look for signs of carry over in the samples that are analyzed immediately after the surfactant sample.

4.4 The sample storage area must be free of organic solvent vapors. This is verified biweekly with the use of refrigerator blanks analyzed by GCMS.

4.5 The nano-pure water should be purged at a high flow rate for a minimum of 4-5 hours with nitrogen before use.

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Personal Protective Equipment Required: Safety glasses, labcoat, nitrile gloves.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Work under a hood when handling stock standards containing high concentrations of toxic analytes. Note that many of the compounds present in the standard mixes are classified as known or suspected carcinogens and should be treated as potential health hazards. Keep exposure to these chemicals to a minimum. Treat all compounds, not just carcinogens, as potential health hazards.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table**. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 Gas Chromatograph/Mass Spectrometer (Agilent or equivalent)
6.1.2 Purge & Trap Concentrator (O.I. Analytical or equivalent)
6.1.3 Auto Sampler (Varian Archon or equivalent)

6.2 Supplies

6.2.1 1-mL vials with polytetrafluoroethylene (PTFE)-lined screw caps
6.2.2 Teflon tape
6.2.3 Fume hood
6.2.4 5 µL,10 µL , 25 µL, 100 µL, 250 µL and 500 µL micro syringes
6.2.5 1-mL, 2.5 mL and 10-mL syringes
6.2.6 1-mL vials with mininert screw caps
6.2.7 Beakers
6.2.8 40 mL glass voa screw top vials
6.2.9 pH Test Strips
6.2.10 EncoreTM sample extrusion tool

7.0 REAGENTS AND STANDARDS

7.1 Reagents

All purchased and prepared reagents must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory.

7.1.1 Methanol (MeOH) – purge and trap grade (Burdick and Jackson or equiv.).

7.2 Standards

All purchased standards must be accompanied by a Certificate of Analysis (C of A) which is kept available at the laboratory in order to demonstrate traceability of the standard to certified (NIST-traceable, if available) source material.

All prepared standards must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory

7.2.1 1000 µg/mL stock 1,2,3-Trichloropropane stock solution, primary source (O2Si or equivalent)
7.2.2 2000 µg/mL stock 1,2,3-Trichloropropane stock solution, second source (Restek or equivalent)
7.2.3 2000 µg/mL 1,2,3-Trichloropropane-d5 stock solution (O2Si or equivalent)
7.2.4 2000 µg/mL stock 1,4-Dioxane stock solution, primary source (Absolute or equivalent)
7.2.5 2000 µg/mL stock 1,4-Dioxane stock solution, second source (Restek or equivalent)
7.2.6 10,000 ug/mL 1,4-Dioxane-d8 stock solution (Absolute or equivalent) .
7.2.7 2000 µg/ml Pentafluorobenzene Internal standard stock solution (Ultra Scientific or equivalent)
7.2.8 2000 µg/mL Dibromofluoromethane Surrogate standard solution, primary source (Ultra Scientific or equivalent)
7.2.9 2000 µg/mL Dibromofluoromethane Surrogate standard solution, second source (Supelco or equivalent)
7.2.10 1000 µg/ml Vinyl Chloride stock solution, primary source (O2Si or equivalent)
7.2.11 1000 µg/ml Vinyl Chloride stock solution, second source (O2Si or equivalent)
7.2.12 20000 µg/mL t-Butyl Alcohol stock solution, primary source (Absolute or equivalent)
7.2.13 2000 µg/mL t-Butyl Alcohol stock solution, second source (O2Si or equivalent)
7.2.14 2000 µg/mL MTBE stock solution, primary source (Restek or equivalent)
7.2.15 2000 µg/mL MTBE stock solution, second source (Ultra Scientific or equivalent)

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters - Preserved	40-mL VOA vials	3 vials	HCl, Cool >0 to 6°C No headspace	14 Days	EPA 624 & EPA 5030B, C
Waters – Unpreserved	40-mL VOA vials	3 vials	Cool >0 to 6°C No headspace	7 Days (see Note 1)	EPA 624
Soils	Jar, Brass Sleeve	100 grams	Cool >0 to 6°C	14 Days	EPA 5030B, C
Soils	VOA vial	3 vials of 5g each	Freeze -10 to -20°C	7 Days	CA DTSC Guidance Document
Soils	VOA vial	3 vials of 5g/with 10 mL H2O each	Freeze -10 to -20°C	7 Days	EPA 5035A

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	VOA vial	1 vial of 5g/with 5mL MeOH or 10g/with 10mL MeOH	Methanol	14 Days	EPA 5035A
Soils	VOA vial	3 vials of 5g/with 5mlLNAHSO 4	Sodium Bisulfate	14 Days	EPA 5035A
Encore or TerraCore	Encore or TerraCore	3 Encores of 5g each	Cool >0 to 6°C Or freeze -10 to -20°C	48 hours (see Note 2)	EPA 5035A
Air	Tedlar Bag	1 bag	N/A	72 Hours	N/A

Note 1: 7 day holding time for unacidified water samples.
Note 2: Encores used as a transfer device only; samples must be extruded into a preserved or frozen sealed VOA vial within 48 hours from collection; holding time for VOA vial is then 14 or 7 days from collection.

9.0 QUALITY CONTROL

9.1 Sample QC

The following quality control samples are prepared with each batch of samples. Each of these QC samples may be re-analyzed once if it doesn't pass, prior to sample analysis, in order to verify the failure wasn't due to a physical or mechanical problem.

9.1.1 Method Blank (MB)

Prepare and analyze a method blank (MB) after each CCV check standard every 12 hours, for each matrix and with every batch of 20 samples, or less. Check that there are no analytes detected at or above the reporting limit. If the method blank shows contamination, re-prepare and re-analyze all samples in the batch unless:

- The samples are ND (qualify the result accordingly).
- The sample result is > 10x the blank level (qualify the result accordingly).

9.1.2 Laboratory Control Sample (LCS)

Prepare and analyze a second source laboratory control sample (LCS) for every batch of 20 samples or less. The recovery and relative percent difference must be within laboratory acceptance limits which are evaluated semi-annually by in-house statistical analysis (see Attachment 1). If the LCS is outside of these limits, re-prepare the whole batch and/or re-calibrate the system unless:

- The LCS recovery is above the upper limit and samples are ND. Report the LCS with an NCM.
- If the LCS is out below the acceptance limits and the sample results are ND, reanalyze once. If it is still out, re-prepare a fresh LCS solution and re-analyze. If the LCS fails twice, two consecutive LCS standards must pass.

9.1.3 Laboratory Control Sample Duplicate (LCSD)

LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract. LCSD recovery criteria is same as that for the LCS. The RPD between the LCS and LCSD must be within in-house/method-specified limits. If the RPD is outside of limits but the individual LCS/LCSD recoveries pass, the data may be reported with an NCM.

9.1.4 Matrix Spike and Matrix Spike Duplicate

The sample for MS/MSD is randomly selected, unless specifically requested by a client. Prepare and analyze a matrix spike (MS) and a matrix spike (MSD) duplicate for each matrix and with every batch of 20 samples, or less. The recovery and relative percent difference must be within laboratory acceptance limits which are evaluated semi-annually by in-house statistical analysis (see Attachment 1).

- If the MS/MSD are outside of the acceptance limits due to matrix interference, flag accordingly.
- If the MS/MSD are outside of the acceptance limits due to instrument problems or due to analyst error, re-analyze the MS/MSD if possible. If re-analyzing the MS/MSD is not possible, fill out an NCM with detailed explanation.

9.1.5 Surrogates

Surrogates are added to each sample, calibration checks, method blank, LCS, MS and MSD. The surrogate recoveries must fall within the acceptance limits evaluated semi-annually by in-house statistical analysis. If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective action.

- If the surrogate is out due to sample matrix, note this information on the results when reporting by flagging the results using the LIMS flagging suite followed with a non-conformance memo.
- If the cause is not due to obvious chromatographic matrix interference, the sample must be reanalyzed to confirm matrix effects.

9.1.6 Internal Standards

For every sample, determine that the retention times for any internal standard have not changed by more than 30 seconds from the midpoint level standard of the most recent initial calibration.

Determine that the absolute areas of the quantitation ions of the internal standards in each sample have not changed by a factor of two (-50% to +100%) from the IS area measured from the current daily midpoint check. The continuing calibration verification retention times and areas must be compared to the mid-point standard level of the most recent initial calibration

- If the retention times or areas have changed by more than these amounts, reanalyze the sample once.
- If internal standard criteria are still not met (due to matrix effects), flag accordingly.

9.2 Instrument QC

The following instrument QC samples are run with each analytical sequence. Each of these QC samples may be re-analyzed once if it does not pass, in order to verify the failure wasn't due to a

physical or mechanical problem. Re-analysis must be performed before any batch QC or client samples are analyzed.

9.2.1 Initial Calibration Verification (ICV)

Calibrations are initially verified immediately following the calibration. The ICV is prepared from a second source. All compounds in the ICAL must be within LCS water control limits.

- If any compounds do not meet LCS limits, re-prepare and re-analyze the ICV.
- If the ICV is still out of control, re-calibrate the system.

9.2.2 Continuing Calibration Verification

Continuing Calibration consists of two steps that are performed at the beginning of each 12-hour analytical shift before any samples are analyzed:

1) Analyze a midpoint calibration standard (CCV). If the response varies from the predicted response by more than ±20%, take any necessary corrective action, prepare a new CCV standard, and reanalyze.

2) Analyze a method blank to ensure that the total system is free of contaminants.

9.2.3 Calibration Acceptance Summary

Refer to the "Calibration Curves" SOP and the "Selection of Calibration Points" SOP for more information on calibrating the GCMS.

Perform initial calibrations on an as needed basis and after major instrument maintenance. The surrogates are calibrated in conjunction with the calibration standards. Major maintenance may include:

- Cleaning the MS source
- Replacing analytical column
- Replacing the detector
- Prepare the ICAL by plotting the response (peak area) against the concentration of at least 5 standards (6 for quadratic).

Prepare the ICAL by plotting the response (peak area) against the concentration of at least 5 standards (6 for quadratic). The following criteria must be met:

- The lowest standard must support the reported detection limit.
- Use average response factor (RF) to determine acceptability. The RF from the initial calibration curve must have an RSD ≤ 15% for each analyte.

Since the curve is not forced through the origin, inaccuracies may be present near the low end of the curve or negative values may be obtained at the reporting limit. In such cases, implement the following corrective actions

- Re-evaluate the curve without the highest calibration standard
- Re-prepare the calibration standards and repeat the calibration

- The cause is sometimes related to the slight bending in the curve at higher analyte concentrations and a 1st order curve is used. If this is the case, consider using a weighted 1st order curve, or using a 2nd order fit.
- Note **that a quadratic regression should be used only as a last resort**, as it can mask instrument response problems and, if used, the plot **must be visually inspected** to insure the absence of an inflection point.

If the %RSD >15%, generation of a first order (linear) or second order (quadratic) regression may be used for quantitation (not forced through the origin). The Coefficient of Determination (r2) must be > 0.99 (Correlation Coefficient (r) > 0.995) for the curve to be acceptable. If r2 is < 0.99, then the instrument must be recalibrated.

10.0 PROCEDURE

10.1 Standard Preparation

Prepare standards monthly or sooner as needed.

Transfer the vendor-supplied stock standards from their 1 mL ampules to 2 mL vials with PTFE-lined screw caps. Label the vials and enter the information into LIMS and ensure each is peer-reviewed. Store the stock standards in the freezer (-10 to -20°C) for no longer than six months or manufacturers' expiration date whichever is sooner. Replace the standards when a change in response is observed.

10.1.1 Sim High Level Calibration Standard Mix

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
Dibromofluoromethane (Surr)	10 µL	2000 µg/mL	4 µg/mL	5mL
Vinyl chloride	50 µL	1000 µg/mL	20 µg/mL	5mL
TBA	25 µL	20,000 µg/mL	100 µg/mL	5mL
1,4-Dioxane	500 µL	2000 µg/mL	200 µg/mL	5mL
MTBE	50 µL	2000 µg/mL	20 µg/mL	5mL
1,2,3-Trichloropropane	5 µL	1000 µg/mL	1 µg/mL	5mL

10.1.2 Sim Low Level Calibration Standard Mix.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
Sim High Level Standard Mix	50 µL	4/20/100/200/1 µg/mL	0.2/1/5/10/0.5 µg/mL	1mL

10.1.3 Prepare the LCS standard

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
1,4-dioxane (2nd source)	100 µL	2000 µg/mL	20 µg/mL	10mL
1,2,3-Trichloropropane (2nd source)	0.5 µL	2000 µg/mL	0.1 µg/mL	10mL
Vinyl Chloride (2nd source)	20 µL	1000 µg/mL	2 µg/mL	10mL
TBA (2nd source)	50 µL	2000 µg/ml	10 µg/mL	10mL
MTBE (2nd source)	10 µL	2000 µg/ml	2 µg/mL	10ml

10.1.4 Prepare the CCV Standard

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
1,4-dioxane (1st source)	100 µL	2000 µg/mL	20 µg/mL	10mL
1,2,3-Trichloropropane (1st source)	1 µL	1000 µg/mL	0.1 µg/mL	10mL
Vinyl Chloride (1st source)	20 µL	1000 µg/mL	2u µg/mL	10mL
TBA (1st source)	5 µL	20000 µg/mL	10 µg/mL	10mL
MTBE (1st source)	10ul	2000 µg/L	2 µg/mL	10mL

10.1.5 Prepare IS/Surrogate (IS/SURR) .

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
1,4-Dioxane-d8 stock	20 µL	10,000 µg/mL	20 µg/mL	10mL
Dibromofluoromethane (Surr) Stock	2ul	2000 µg/mL	0.4 µg/mL	10mL
1,2,3-Trichloroproane-d5 stock	1 µL	2000 µg/mL	0.2 µg/mL	10mL
Pentafluorobenzene Internal standard stock	2 µL	2000 µg/mL	0.4 µg/mL	10mL

10.1.6 Prepare Internal Standard (IS) only

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MEOH
1,4-Dioxane-d8 stock	20 µL	10,000 ug/mL	20 µg/mL	10mL
1,2,3-Trichloroproane-d5 stock	1 µL	2000 µg/mL	0.2 µg/mL	10mL
Pentafluorobenzene Internal standard stock	2 µL	2000 µg/mL	0.4 µg/mL	10mL

10.1.7 Calibration curve standards are prepared as follows in a 10 mL purge volume of DI water:

STD Level	Volume (IS) Only (µL)	Volume for Low Level STD. Mix (µL)	Volume for High Level std mix (µL)	TBA ppb	MTBE ppb	1,4-Dioxane ppb	1,2,3-TCP ppb	Vinyl Chloride ppb	Dibromofluoromethane (Surr) ppb
1	5	1	----	0.5	0.1	1	.005	0.1	0.02
2	5	2	----	1	0.2	2	.01	0.2	0.04
3	5	5	----	2.5	0.5	5	.025	0.5	0.1
4	5	10	----	5	1	10	.05	1	0.2
5	5	----	1	10	2	20	0.1	2	0.4
6	5	----	2.5	25	5	50	0.25	5	1
7	5	----	5	50	10	100	0.5	10	2
8	5	----	10	100	20	200	1	20	4

10.1.8 CCV/Midpoint. Prepare the daily CCV/midpoint with 10-mL reagent grade water and add 5 uL of the CCV standard solution and 5 uL of the internal standard/surrogate solution.

10.1.9 Laboratory Control Spike (LCS). Prepare an LCS by adding 5 uL of the LCS standard and 5 uL of the internal standard/surrogate solution into 10ml ultrapure water.

10.1.10 Method Blank. Prepare a method blank with 10-mL of ultrapure water. Add 5 µL of internal standard/surrogate solution to the ultrapure water.

10.1.11 Internal Standard/Surrogate

- Use 1,4-Dioxane-d8 as the internal standard for 1,4-Dioxane at a concentration of 10 µg/L.
- Use 1,2,3-Trichloropropane-d5 as the internal standard for 1,2,3-Trichloropropane at a concentration of 0.1 µg/L.
- Use dibromofluromethane as the surrogate at a concentration of 0.2 µg/L.

- Use pentaflurobenzene as the internal standard for the surrogate at a concentration of 0.2 µg/L.

10.2 Waters Sample Preparation

10.2.1 Use a 10 mL sample purge volume.

10.2.2 Add 5 µL of IS/SURR solution to each sample to be purged

10.3 Soil Sample Preparation

10.3.1 Weigh out the sample near the required weight quickly into a tared VOA with stir bar or purge tube. The weight will be electronically transferred directly to the worklist in the system.

10.3.2 Immediately after weighing the sample and while still at the fume hood, add water and add the required standards. The purging vessel needs to be sealed quickly to keep the compounds from releasing into the air.

10.3.3 Refer to the current Volatiles Sample Preparation SOP for details on the amount of sample, spikes/surrogates, and amount of solvent to be used.

10.4 Instrument Initialization

The following are general instrument conditions. These conditions may vary between instruments because of necessary instrument maintenance (e.g. column trimming), type of column or column age.

- Column: DBVRX 20m x 0.180mm (1 micron)
- Carrier gas (He) flow rate: 1.2ml/min
- Purge gas (He) flow rare: 40ml/min

10.4.1 Purge-and-Trap Conditions

- Pre-Heat sample to 55° C for approximately 1.5 minutes
- Purge for 11 minutes.
- Dry purge for 2 minutes
- Pre-heat desorb at 135° C.
- Desorb at 190° C for 1 minute.
- Bake out at 210° C for 10 minutes.
- Transfer line (set temperature at 120°C), and valve (set temperature at 150° C).

10.4.2 Detection/Integration Parameters

- Set to SIM mode
- Characteristic Ions monitored

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
TBA	59	57
Vinyl Chloride	62	64
MTBE	73	57
Pentafluorobenzene (IS)	99	137
Dibromofluoromethane (SU1)	113	111,192
1,4-Dioxane-d8 (IS)	64	96
1,4-Dioxane	88	58, 87
1,2,3-Trichloropropane-d5 (IS)	79	114, 63
1,2,3-Trichloropropane	75	110,61

10.5 Calibration

For a new calibration, load the calibration standards in ascending concentration order followed by the ICV. Verify all calibration criteria are met before proceeding.

To continue using a previous calibration, recall the calibration file, analyze a calibration blank and midpoint standard. Verify acceptance criteria are met before proceeding.

10.6 Sample Analysis

A typical daily run sequence is listed below:

1 Daily midpoint (CCV)
2 LCS
3 Method blank
4 Samples and MS/MSD
5 [end of run after 12 hours from daily midpoint]

10.7 Preventative Maintenance

10.7.1 Replace OI #10 trap when necessary.
10.7.2 Replace injector septa and deactivate liners when necessary.
10.7.3 Clean the ion source and replace filaments when necessary.
10.7.4 Change the oil in the foreline pump as needed and record in the maintenance logbook.
10.7.5 Check the diffusion pump oil and replace as necessary and record in the maintenance logbook.
10.7.6 Replace the carrier gas as needed and record in the maintenance logbook.
10.7.7 Record all performed maintenance in the instrument maintenance logbook.
10.7.8 If an instrument is unusable or has limitation to its use (bad port, not for low level samples, etc), it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Percent Difference (%D) for CCV

$$\text{\% Difference} = \frac{[\text{Apparent conc. (µg/l) - True conc. (µg/L)}] \times 100}{\text{True conc. (µg/l)}}$$

11.4 Response Factors (RFs)

$$RF = \frac{(R_A)(C_{IS})}{(C_A)(R_{IS})}$$

$$RF_{avg} = \sum RF_{std}$$

$$C_A = \frac{R_S \times C_{IS}}{R_{IS} \times RF_{avg}}$$

R_A = analyte response
C_A = analyte concentration
R_{IS} = internal standard response
C_{IS} = internal standard concentration
RF = response factor
RF_{avg} = mean response factor for an analyte from the ICAL

11.5 Concentrations

11.5.1 Water Samples

$C_f = C_i \times PF \times DF$

C_f = Final concentration in μg/L or μg/Kg
C_i = Concentration in μg/L from instrument
PF = Preparation Factor
DF = Any additional Dilution Factor

11.5.2 Low Level Soils

$C_f = C_i \times DF \times CF$

C_f = Final concentration in μg/L or μg/Kg
C_i = Concentration in μg/L from instrument
DF = Dilution Factor (5g / wt of soil purged, in g)
CF = Calibration Factor (2x soils)

11.5.3 High Level Soils (MeOH extracts)

$C_f = C_i \times PF \times DF$

C_f = Final concentration in μg/L or μg/Kg
C_i = Concentration in μg/L from instrument
PF = Prep Factor (Vol of MeOH in ml / Weight of soil, in g)
DF = Dilution Factor (10 ml / actual volume injected, in ml)

11.5.4 Surrogate Spike Results in High Level Soils (MeOH extracts)

$C_f = C_i \times SF \times DF$

C_f = Final concentration in μg/L or μg/Kg
C_i = Concentration in μg/L from instrument
SF = Surrogate Factor (Final Prep Vol of MeOH in ml / 10 ml)
DF = Dilution Factor (100 μl / actual volume injected, in μl)

12.0 METHOD PERFORMANCE

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure as described in laboratory's SOP, IR-QA-MDL. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples. An IDOC consists of 4 consecutive LCS samples spiked at 10 - 50 times the MDL for each analyte in that matrix with an average recovery and RSD within laboratory acceptance limits. An on-going DOC must be performed annually. An ODOC can be 4 consecutive LCSs at mid-level.

12.3 Training Requirements

The analyst must have documented training, including reading of the SOP and source methods, conducted by the department manager, senior chemist, or other analyst with training documentation and a passing DOC.

13.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in the "Waste Management and Pollution Prevention" section of the Corporate Environmental Health and Safety Manual (CW-E-M-001).

14.0 WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the laboratory's Waste Disposal SOP (IR-EHS-WASTE). The following waste streams are produced when this method is carried out:

- **Methanol waste** is generated when preparing samples and standards. This waste is collected into 4 L bottles located in the Volatiles fume hoods. Sample archive technicians remove the bottles from the lab to the main waste storage area twice a week. This waste is bulked as Mixed flammable solvents
- **Solid lab trash waste** (includes plastic pipets and gloves used in handling hazardous samples). This waste is stored in a pink plastic bag. Sample archive technicians remove this waste from the lab to the main waste storage area twice a week. This hazardous waste is lab packed.
- **EnCores waste**. This waste is generated when analyzing soil that came in encores. Usually a set of three encores per sample is received in a plastic bag. The analyst opens and transfers the soil sample from the encore to the appropriate container according to the procedures in their SOPs. The empty encore(s) is returned to the original bag. After at least 21 days from sample received sample archive technicians remove the encores to the main waste storage area. This waste is disposed of in the regular trash.

Vacuum oil waste. Collected in a 5-gallon container located in the main waste storage area and taken for recycling when full.

- **Tedlar bags**. Once the analysts are done analyzing the samples from the tedlars bags, they are stored in a cardboard box for about one week in case they are needed for re-testing. After a week the analyst place them in a black plastic bag, where they are kept for about 1 month. The Analyst empties the contents of the bag in the VOA fume hood. After this time the tedlar bags are disposed into the regular trash.
- **Foreign soil** - Generated by analyst/technicians when preparing samples and from samples after being analyzed (samples from Mexico, Canada and Hawaii, etc). This waste is collected in a glass jar, which is placed in the volatiles fume hood. Volatiles analyst/technicians remove the waste from the lab into the main waste storage area once a month or more often if necessary. This waste is bulked as RCRA foreign soils.
  The water waste generated after the samples have been analyzed is collected in a 4 L bottle container. This waste is removed from the lab to the main waste storage area once

a month or more often if necessary. This waste is bulked in the same drum of RCRA foreign soil.

- **VOA vials with water**. – (Purge VOA vials) VOA vial waste contains water and small amounts of Hydrochloric acid. Collected in a 2-1/2 gallon container marked as "Hazardous Waste". The pH of the collected waste is measured, neutralized using soda ash or lime, and drained into the sewer system. The empty VOA vials are placed in the regular trash.
- **VOA vials with soil and water**. – (Purge VOA vials) VOA vial contains soil and water. The water waste is collected in non-hazardous waste containers (beakers or jars), and then draining the contents into the sewer system, the VOA vials with soil are placed in the regular trash
- **VOA vials with soil and Methanol**. – (Purge VOA vials) VOA vial contains soil and Methanol. The VOA vials are collected and then taken to the main waste storage area. The VOA vials are bulked in a drum and shipped as vials with flammable solvents.
- **Unused standards** are placed on the shelves labeled "hazardous waste" in the main waste storage area by analyst/technicians. The containers must be labeled as hazardous waste with a date they were taken to the main waste storage area. This waste is lab packed.

15.0 REFERENCES / CROSS-REFERENCES

15.1 EPA Method 8260B, EPA SW-846 Update III, December 1996

15.2 EPA Method 8000B, EPA SW-846 Update III, December 1996

15.3 EPA Method 8000C, SW-846, March 2003

15.4 EPA Method 5035A, SW-846, July 2002

15.5 EPA Method 5030A, SW-846, Update I, July 1992

15.6 EPA Method 5030B, SW-846, Update III, December 1996

15.7 EPA Method 5030C, SW-846, May 2003

15.8 CA-Q-S-005, Calibration Curves (General)

15.9 CA-T-P-002, Selection Of Calibration Points

16.0 METHOD MODIFICATIONS

Item	Method Ref	Modification
1	5030A, B, C	The purge is performed at 55 ± 1°C to increase the purge efficiency.
2	SW846, Chapter 4	Holding time for samples that are not acidified to pH<2 is set at 7 days.

17.0 ATTACHMENTS

17.1 **Attachment 1**: Analysis Information
17.2 **Attachment 2**: Data Review Checklist
17.3 **Attachment 3**: ICAL Review Checklist
17.4 **Attachment 4**: Track sheets

18.0 REVISION HISTORY

18.1 **Revision 0, dated 02 September 2008**

- Integration for TestAmerica and STL operations
- Supersedes 14DIOX.SOP (revision 0, 04/02/03) and 123TCP_R1.SOP (revision 0, 09/02/04

18.2 **Revision 1, dated 24 November 2010**

- Supersedes IR-MSV-SIM, revision 0, 09/02/08
- Prepared by VS

18.3 **Revision 2, dated 26 July 2013**

- Supersedes IR-MSV-SIM, revision 1 (11/24/10) and IR-MSV-SIM_r1-CF1 (03/21/12)
- Added references to Calibration Curves and Selection Of Calibration Points corporate SOPs.
- Updated SOP title. SOP title references EPA 8260B
- Updated IDOC requirements, IDOCs must be spiked @ 10 to 50 times the MDL per EPA 8000 methods
- Remove the requirement to run an BFB tune
- Updated standard preparation procedure (tables)
- Removed references to 1,1,2,2-Tetrachloroethane and Chloroform from the SOP. The laboratory no longer analyze for these two compounds.
- Updated Response Factor Calculation Section to include formulas for the calculation of RF_{ave} and analyte concentration
- Updated References section
- Added track sheet as attachment 4
- Added soil preparation section
- Updated Waste Management Section
- Prepared by GK

Attachment 1
Analysis Information

TestAmerica Irvine

4/13/2013

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8260B-SIM in Water (EPA 8260B-SIM)								
Preservation: 4 C, HCL								
Container: 40 mL Amber VOA Vial		**Amount Required:** 3 VOA				**Hold Time:** 14 days		
1,4-Dioxane	1.0	2.0 ug/l			70 - 130	30	70 - 125	30
1,2,3-Trichloropropane	0.0035	0.0050 ug/l			55 - 135	30	60 - 130	20
Chloroform	0.010	0.020 ug/l			65 - 135	20	70 - 130	20
1,1,2,2-Tetrachloroethane	0.010	0.020 ug/l			55 - 135	30	55 - 130	25
Methyl-tert-butyl Ether (MTBE)	0.020	0.10 ug/l			55 - 145	25	60 - 135	25
tert-Butanol (TBA)	0.40	0.50 ug/l			65 - 140	25	70 - 135	20
surr: Dibromofluoromethane			80 - 120					

TestAmerica Irvine

4/13/2013

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8260B-SIM in Soil (EPA 8260B-SIM)								
Preservation: 4 C, Cool								
Container: 4 oz Jar/BrassSleeve		**Amount Required:** 100 grams				**Hold Time:** 14 days		
1,4-Dioxane	1.1	2.5 ug/kg			70 - 130	30	70 - 130	30
1,2,3-Trichloropropane	0.0044	0.010 ug/kg			50 - 150	30	60 - 135	25
Chloroform	0.020	0.040 ug/kg			65 - 135	20	70 - 130	20
1,1,2,2-Tetrachloroethane	0.020	0.040 ug/kg			40 - 160	30	55 - 140	30
surr: Dibromofluoromethane			80 - 125					

Attachment 2
Data Review Checklist

GCMS DAILY DATA CHECK LIST
EPA 8260B-SIM

2nd Level Review:	_____	Analyst:	_____
Date:	_____	Analysis Date:	_____
QC Batches:	_____	GCMS #:	_____
Data Upload by:	_____		
Date:	_____		

2nd Level Rev	Analyst Rev	
		Mid-point check (ICV/CCV) :
_____	_____	• Every 12-hr shift
_____	_____	• % recovery of all target analytes met (refer to in-house limits)
_____	_____	**Method blank :** after each Mid-point check (< RL or accordingly flagged)
_____	_____	**2nd source LCS :** every batch of 20 samples or less (refer to in-house limits)
_____	_____	**MS/MSD :** every batch of 20 samples or less (refer to in-house limits)
		All samples check for:
_____	_____	• Unit, Dilution Factor
_____	_____	• Manual Integration, Spectra Match
_____	_____	• Area of internal standards met (-50% to + 100% from the daily CCV)
_____	_____	• Surrogates within limits (refer to in-house limits)
		• All samples analyzed within 12-hour from the CCV
_____	_____	Update Analyst initials in TALS
_____	_____	Non-conformance memo (if applicable)

Comments:

_____
_____
_____
_____

G:\Depts\Quality\Data_rev\chklist\GCMS\8260_SIM_chk_v062113.doc
Rev 06/21/2013

Attachment 3
ICAL Review Checklist

GCMS INITIAL CALIBRATION CHECK LIST
EPA 8260B -SIM

2nd Level Review:		Analyst:	
Date:		Initial Calibration Date:	
Initial Cal updated by:		Initial Calibration File:	
Date		GCMS #:	

2nd Level Rev	Analyst Rev	
_____	_____	Minimum 5-point calibration – lowest standard at Reporting Limit (6-point for quadratic regression)
		RSD of RF:
_____	_____	• **All compounds:** ≤15 %
_____	_____	• If RSD >15% and r ≥ 0.99: generate a linear or quadratic curve.
		ICV
_____	_____	• Meets all LCS-water %recovery limits
_____	_____	• If any compound out high, ONLY ND results may be reported with an NCM
		Review calibration curve (include software or calculator-generated r coefficient)
_____	_____	• Review calibration history report
_____	_____	• Review the initial calibration %Drift report (all compounds within limits)
_____	_____	• Review calibration history summary
_____	_____	• Ensure that all co-eluted compounds have correctly associated spectral masses
_____	_____	• Ensure that all isomers (same spectral masses) have correctly identified retention times
_____	_____	Initial Calibration date and analyst initial in LIMS system
		Check all levels for:
_____	_____	• Manual integration
_____	_____	Calibration performed within 12-hour period
		Review Internal Standard Report

Comments: _____

G:\Depts\Quality\Data_rev\chklist\GCMS\8260_SIM_CAL_v06213.doc
Rev 6/21/2013

Attachment 4
Track Sheet

EPA METHOD 8260 SIM GC/MS DAILY LOG SUMMARY

DATE: *AM / PM* Worklist:

Prep Batch: 1st Level date_____ initial._____

INSTRUMENT: GC/MS # Batch: 2nd Level date_____ initial._____

**Check box if sample pH <2, if not, record actual pH in the box.* *Final volume for all matices = 10 ml*

#ALS	SAMPLE NAME / LIMS ID#	PH <2*	INSTRUMENT FILE NAME	MTX	LOADER	CHROM	Comments
			V				

GCMS_TRACK_#44_R.0
Rev. 10/05/12