USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 314

LOW LEVEL ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN AIR TO-15 SIM (SELECTIVE ION MODE)

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1 PURPOSE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the procedures used for the low level analysis of volatile organic compounds (VOCs) in air collected in specially prepared stainless steel canisters at the USEPA Region 9 Laboratory in Richmond, California. This SOP is based on EPA Method TO-15, Second Edition January 1999. Deviations from Method TO-15 are described in Appendix A. Analytes and quantitation limits (QLs) for this analysis are listed in Appendix B.

This SOP is applicable to the low level analysis of VOCs in air. The applicability of these procedures to specific data quality objectives (DQOs) must be assessed on a case-by-case basis.

2 SUMMARY

A known volume of sample is directed from the canister through a flow controller into a micro-scale purge and trap, separated in a Gas Chromatograph (GC) column, and detected by a mass spectrometer (MS). The target VOCs are identified in the sample by analyzing standards under the exact same conditions employed for samples and comparing the resulting mass spectra and GC retention times. Each target compound is quantitated using response factors from the most recent initial calibration.

3 DEFINITIONS

Absolute Pressure	Pressure measured with reference to absolute zero pressure; expressed as inches Hg, psig, or psia.
Atomic Weight	The following terms are used to describe atomic weight: Dalton - Primary unit of mass spectrometry equivalent to 1/12 mass of carbon -12 atom; amu – atomic mass unit equals one Dalton; (m/z) – mass to charge ratio equals ion mass (in Daltons) divided by number of unit charge for the ion.
BFB	Bromofluorobenzene, the compound used as the system performance check for the GC/MS
Gauge Pressure	Pressure measured with reference to the surrounding atmospheric pressure. Zero gauge pressure is equal to atmospheric pressure; expressed as inches Hg, psig, or psia.

GC/MS	Gas Chromatograph / Mass Spectrometer
LCS	Laboratory Control Sample (Blank Spike, Secondary Source Std.)
LIMS	Laboratory Information Management System
SDG	Sample Delivery Group
SIM	Selective Ion Mode

4 SAFETY & HEALTH

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN for additional information.

The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: vinyl chloride and trichloroethylene. Primary standards of these toxic compounds are prepared from commercially prepared gas reference standards that are available in various gas cylinder sizes. These standards must be prepared in a hood. If standard preparation is by dynamic dilution of the gaseous contents of a cylinder of stock gas calibration standards, then the dilution system must be vented into a hood.

The procedure described in this SOP involves the use of cryogenic liquids for cooling the Entech concentrator. Region 9 SOP 770, *Handling Cryogenic Materials*, provides guidelines for the safe handling of these materials.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or

troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

All compressed gas cylinders must be securely chained to laboratory benches or walls and placed so that the labels can be easily read.

Canisters should never be pressurized beyond 45psia, which is the maximum allowable pressure for specially prepared canisters.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Pollution Prevention Plan* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the instruments, glassware, syringes, and/or analytical balances used to prepare the solution.
Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever possible.

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure* and City of Richmond Discharge Permit. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

This procedure generates the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-regulated Waste	Not applicable
Spent stock standard cylinders (Depleted or Expired: Must be vented to ambient pressure in fume hood prior to disposal)	Recycled / Non-regulated Waste	Not applicable

Stock standard cylinders purchased from Restek Corporation (Spectra Gases, Inc.), when ready for disposal, are vented in a fume hood to < 15 psig, labeled "EMPTY", and are returned to the manufacturer: Environmental Division, Spectra Gases Inc., 80 Industrial Drive, Alpha NJ, 08865.

Stock standard cylinders purchased from Supelco (Sigma-Aldrich), when ready for disposal, are vented in a fume hood to ambient pressure, labeled empty, made unusable by drilling a hole through the cylinder, and disposed of with the non-regulated waste.

5 SAMPLE HANDLING AND PRESERVATION

5.1 Containers and Required Sample Volume

Canisters included are SilcoSteel® silica coated interior canisters, TO-Can® electro-polished interior canisters, and Silonite® coated interior canisters. Analysis of 400 ml sample load volume represents an undiluted sample (i.e. Dilution Factor = 1). Canister sizes vary from 400mL to 6 L.

5.2 Internal Chain-of-Custody

After Samples are received and logged into the Laboratory Information Management System (LIMS), they are delivered by the Sample Custodian to Room 203.

Check the following information to ensure that the information on the sample containers correspond to the information on the tracking sheets and the chain-of-custody record.

- EPA work order number

- Laboratory ID number
- Case number
- Sample delivery group (SDG) number

5.3 Preservation Verification

The analyst shall confirm that the integrity of the canister samples has not been compromised by checking the initial and final pressure of the canisters prior to and after receipt. Sample canisters upon receipt should have below ambient pressures (approximately -4 to -10 inches Hg) if passive flow controllers were used in sampling. Any problems or deviations must be noted in the LIMS work order memo ("MMO") field.

5.4 Sample Storage

Samples in canister shall be stored in the air analysis laboratory at room temperature. Upon completion of the analysis for an SDG the sample canisters for the entire SDG shall be stored, for a period of 7 days after the analysis of the entire SDG is completed. After 7 days, the canisters shall be vented in the fume hood and cleaned as specified in SOP #312, "Cleaning and Certification of Specially Prepared Canisters for Air Sampling".

5.5 Holding Time

Samples must be analyzed within 30 days from the time of collection.

6 INTERFERENCES

6.1 Contamination may occur if canisters are not properly cleaned. Before each use, canisters must be thoroughly cleaned and certified as outlined in SOP #312.

6.2 Canisters should be capped tightly during storage and shipping to prevent leakage and sample contamination.

6.3 Impurities in the calibration dilution gas and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running UHP (ultra high purity) zero grade air instrument blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with rubber components must be avoided.

6.4 Significant contamination of the analytical system can occur whenever samples containing high VOC concentrations are analyzed. Whenever an unusually concentrated sample is

encountered (i.e. concentrations significantly greater than the calibration range of the instrument, often indicated by poor chromatographic peak shape) it should be followed by analysis of UHP zero grade air instrument blank to check for carry-over contamination.

6.5 Solvents and other compounds that are target analytes must never be introduced into the laboratory where volatiles analysis is performed. Methylene chloride, acetone, and other common laboratory chemicals that are target analytes analyzed on this system must be excluded from the room where the analysis is performed.

6.6 The analytical system is sensitive to water and the amount of humidity in a sample can affect the recovery of target analytes. Conditions of high humidity or water (i.e. rain) should be noted in the chain of custody and mentioned in the case narrative.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. Minor deviations may be made in specific apparatus and materials provided that they are documented and equivalency is maintained.

7.1 Instruments and Equipment

7.1.1 Instrument "HP5973_Air": Entech 7016CA canister auto sampler (x2), an Entech 7100 pre-concentrator, a Hewlett Packard HP6890 gas chromatograph and a Hewlett Packard HP5973 mass spectrometer. This system allows for the sequential analysis of up to 32 canisters. The gas chromatograph is equipped with a RTX-1 0.32 mm ID and 60 m length capillary column with 1.5micron film. The data system used is Hewlett Packard Chemstation operated on a 386MHz or faster computer.

7.1.2 Entech 4600 dynamic diluter: This system is used to create calibration standards by diluting stock standards. It is also used in diluting samples. The diluter uses Mass Flow Controllers to perform the dilutions.

7.2 Reagents

Enter all standards into the LIMS database. Reagents may contain impurities that may affect analytical data. Only materials that conform to the American Chemical Society (ACS) reagent grade specifications should be used. If purity of a reagent is in question, analyze fro contamination prior to use.

- Helium Gas – (Ultra High Purity)
- Liquid Nitrogen (22-50 psig)

- Nitrogen Gas
- Zero Grade Air (Ultra High Purity)

7.3 Standards

7.3.1 Calibration Gas Standard (stock) - commercially prepared certified gas standards containing nominally 100ppbv of each target compound.

7.3.2 4-Bromofluorobenzene Tuning Standard - commercially prepared certified gas standard containing nominally 100ppbv of BFB that is equivalent to 0.78ng BFB per ml of standard mix.

$$\frac{175\,g_{BFB}}{mole_{BFB}} x \frac{100*10^{-9}\,moles_{BFB}}{mole_{Nitrogen}} x \frac{1*10^{9}\,ng_{BFB}}{g_{BFB}} x \frac{mole_{nitrogen}}{22{,}400\,ml_{Nitrogen}} = .78\,ng_{BFB}/ml$$

7.3.3 Laboratory Control Sample (stock) - commercially prepared certified custom gas standard containing nominally 100ppbv of each target compound. Equivalent standard as the calibration standard derived from a secondary source.

7.3.4 All stock standards upon receipt must be recorded into the LIMS and assigned a standard ID number. Sources, expiration dates, components, and concentrations for gas mixtures must be recorded onto the LIMS. Standard ID number and expiration dates must be printed on to the standard cylinder's label.

7.3.5 A description of the preparation of all working standards is to be kept electronically in the LIMS system. Each working standard is designated a standard ID number by the LIMS.

Before use, working standards should be allowed to equilibrate for at least 2 hours after preparation.

7.3.6 Preparation of Working Calibration Standard

Calibration gas standards are prepared by diluting as necessary commercially prepared certified gas standards that typically contain 100ppbv of each target compound (stock standard). For an initial calibration, the five concentration points are typically 50pptv, 100pptv, 200pptv, 300pptv and 500pptv. A single working standard at 1000pptv is prepared from which the five concentrations

can be derived by varying the sample volumes loaded during the pre-concentration step.

Note: The concentrations of the calibration levels may be modified to achieve a desired calibration range as long as it meets QA/QC criteria as stated in section 8 of this SOP.

Calibration standard concentration calculation: $C_F = C_I (f_i / f_t)$

where: C_F is the final concentration of the calibration standard
C_I is the initial concentration of the stock standard
f_i is the flow rate from the stock standard
f_t is the total sum of all the flow rates (stock standard + dilution gas)

Replace the calibration standard every 30 days or sooner if analysis indicates that the standard has degraded.

7.3.7 Preparation of Method Blank

The method blank is prepared in a silica coated 6L canister. The canister is pressurized using the Entech 4600 dynamic diluting system. The canister is filled with humidified zero air to a final canister pressure of approximately 30 - 45psia. Replace/refill the method blank as necessary.

7.3.8 Preparation of Working LCS Standard

The working LCS standard is prepared in a silica-coated canister using the Entech 4600 dynamic dilution system to a final canister pressure of 30 - 45psia. The LCS working standard is prepared from a commercially prepared gas standard obtained from a secondary source (different form that of the calibration standard source). Replace the working LCS standard every 30 days or sooner if analysis indicates that the standard has degraded.

7.4 Supplies

- Stainless steel tubing and stainless steel fittings
- Stainless steel cylinder pressure regulator: standard two-stage cylinder regulators with pressure gauges.
- Gas-tight syringes (5-mL, 10-ml, 25-mL, 100-ml).

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Set up the GC/MS following operating instructions provided by the manufacturer. Use the operating parameters provided in Appendix D as starting point.

Ensure that appropriate waste containers are present and properly labeled.

8.2 Calibration and Standardization

8.2.1 GC/MS Tuning

Prior to the analysis of any calibration standards, blanks, and samples, the GC/MS system must meet the mass spectral ion abundance criteria for bromofluorobenzene (BFB). Proper tuning of the instrument is necessary to produce standardized fragmentation patterns of target and non-target compounds. Refer to Section 9.2.2 for frequency, acceptance criteria, and corrective action requirements.

8.2.2 Initial Calibration

After the instrument performance check criteria has been met but prior to analysis of samples and blanks, the GC/MS system must be calibrated at five concentrations that span the monitoring range of interest. A linear calibration curve type must be used. Refer to Section 9.3.1 for frequency, acceptance criteria, and corrective action requirements.

8.2.3 Continuing Calibration

A continuing calibration standard at a mid-level concentration (i.e. 200pptv) must be analyzed once every 24 hours. The daily calibration sequence starts with the injection of BFB and is analyzed after the BFB analysis meets the ion abundance criteria. Note that the mid level calibration standard acts as the continuing calibration verification when samples are analyzed within the same 24-hour period as the initial calibration. Refer to Section 9.4.1 for frequency, acceptance criteria, and corrective action requirements.

8.2.4 Laboratory Control Spike (LCS)

The LCS must be analyzed and reported once every 24hr analytical sequence. The LCS must be from a secondary source. (i.e. different manufacturer of the standards used for calibration). The LCS serves to confirm the concentration of the reference standard. Refer to Section 9.4.2 for frequency, acceptance criteria, and corrective

action requirements.

8.2.5 Quantitation Limit Standard

The QLS at the 50pptv level must be analyzed and reported once per 24-hour analytical sequence. If running an initial calibration, the 50pptv standard serves as the QLS. A separate QLS shall be analyzed at the end of the analytical sequence in order to "bracket" the samples to monitor the instrument's performance. Refer to Section 9.4.3 for frequency, acceptance criteria, and corrective action requirements.

8.2.6 Method Blank

A method blank must be analyzed before samples are analyzed in order to demonstrate that the instrument is free of contamination and to evaluate possible laboratory contamination. Refer to Section 9.4.4 for frequency, acceptance criteria, and corrective action requirements.

8.3 Analysis

8.3.1 Sample Preparation

Samples shall be analyzed only after the BFB tune, initial calibration or continuing calibration, quantitation limit standard, laboratory control spike, and method blank analyses meet all of the appropriate criteria specified in Section 9.4.4 of this SOP. If time still remains in the 24-hour time period after meeting the criteria, samples may be analyzed. It is not necessary to analyze a continuing calibration standard within this 24-hour time period, if the initial calibration standard that is at the same concentration as the continuing calibration standard meets the continuing calibration acceptance criteria.

Sample canister pressures are checked and recorded upon receipt. Indoorair and ambient air samples received with below ambient pressures are pressurized using the Entech 4600 dynamic diluter to a 1.5x dilution or higher if necessary to bring canister pressures above ambient (approximately 18 - 20psia). Dilution procedures and calculations are recorded in a dilution logbook. Note: Dilutions can also be performed by varying the sample load volumes through the Entech 7100 pre-concentrator. These dilutions are recorded in the daily run logbook and LIMS system.

Prior to analysis, samples should be allowed to equilibrate in the lab for at least 2 hours after collection.

The analyst shall check that the numbers on the canisters coincide with the

numbers on the routing forms to ensure that the correct sample is being analyzed.

Break the chain of custody seal (if present) on the valve of the canister with a scalpel or other appropriate implement, check the attached pressure gauge and record the canister pressure at time of analysis on the run log.

Load the closed canisters onto the auto sampler and record the canister ID number and the auto sampler position for each sample in the run log.

8.3.2 Sample Analysis and Analytical Sequence

Use the bench sheet generated from the LIMS to set up the data acquisition sequence. The sample description shall include the laboratory sample ID, client sample ID, canister ID, and the sample volume and the dilution factor (if necessary). Additional header information shall include instrument ID and the analyst's initials.

Set up the pre-concentrator system with the appropriate parameters and enter the sequence of analysis. Set up the system to take a 400 ml aliquot of the sample. If very high concentrations are suspected, screen a 40 ml aliquot of the sample first.

Setup the GC/MS system at the optimum GC and mass spectrometer conditions. See Appendix D.

8.3.3 Analyte Identification

In order for a target compound to be identified as present in a sample, both retention time and the mass spectra of the peak must match those of the standard.

SIM method looks for specific ions in the target compounds, all ions must be present for positive verification. The relative intensities of the ions must agree within 20% between the continuing calibration verification and sample spectra.
Ions present in the sample at greater than 10% abundance but not present in the standard spectra must be reviewed and accounted for by the analyst making the comparison. If a compound cannot be verified by these criteria but is present in the technical judgment of the analyst, the supporting evidence must be indicated on the raw data and the analyte reported.

8.3.4 Analyte Quantitation

Quantitate the data and print out a quantitation report and chromatogram. Use the average response factor from the initial calibration for quantitation. Review the results as discussed in Section 8.3.3 for qualitative identification of target analytes. Cross out

all reported hits that do not meet the qualitative criteria. Review all target compounds that are detected to verify they are integrated properly. Review the chromatogram for possible false negatives. Make sure that all data is transferred to the LIMS properly and that reported results are accurate.

8.3.5 Manual Integration

Where the chromatography software integrates the signal inconsistently, follow SOP 835, *Chromatographic Integration Procedures*. All manual chromatographic integration must be initialed and dated by the analyst, noted in the run log, and approved by a supervisor, Chemistry Technical Director, Quality Assurance Officer, or designees.

8.3.6 Calculations

Results for target analytes are calculated using the following equation:

(Cx) = (Ax)(DF)/(RFx)

Where:

Cx = Concentration of compound X
Ax = Area of quantitation ion of compound x
DF = Dilution factor
RFx = Response factor from the initial calibration for compound x

8.3.7 QC Review

Initial Calibration, continuing calibration, quantitation limit standard, laboratory control spike, and method blank must pass all QC criteria as described in Section 9 prior to any analyses of samples.

Check that target analyte results are within range of the initial calibration. In addition to the undiluted analysis, the sample must be diluted and re-analyzed if any of the target analytes exceed the calibration range of the instrument. The sample should be diluted so that the target compound that was originally outside of the calibration range will fall within the middle range of the initial calibration.

Sample results are reported to no less than ½ the quantitation limit. Sample results less than the quantitation limit are reported as “estimated” and flagged appropriately in the report.

Check laboratory sample duplicate results and make sure that QC limits were not

exceeded as described in Section 9.4.5.

Make sure that the end-of-sequence QLS passes all QC limits as described in Section 9.4.3.

8.3.8 Data Export and LIMS entry

Generate epatemp.txt files for field and QC samples by printing the report to the screen; these files are used by the LIMS Data Tool module to import the instrument results into the Data Entry/Review table.

Copy sample data files from the local drive to the appropriate instrument subdirectory on the Region 9 LAN to make them available to LIMS and archive them.

Create an empty upload file containing the samples analyzed in the LIMS batch or sequence. Import and merge data files using the LIMS Data Tool module. Load the resulting merged data file into the LIMS Data Entry/Review table. See LIMS manual for detailed procedure.

Edit dilutions in Data Tool or LIMS entry table as needed.

Check data entered into LIMS to make sure that proper transcription has occurred, all dilutions are correct, and that all outliers are properly flagged.

8.4 Maintenance

Preventive maintenance of GC/MS system is performed by Full Spectrum Analytical Services on a regularly scheduled basis.

The analyst may need to clean the source or perform leak checks if the need arises. Source cleaning may be necessary when a significant drop in response is observed or difficulty in passing QC limits for initial and continuing calibrations are encountered.

9 QUALITY CONTROL

9.1 Demonstration of Capability

The EPA Region 9 Laboratory operates a formal quality control program. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that

are generated. A summary of QC criteria is provided in Appendix C.

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in EPA Region 9 Laboratory SOP 880 for more details.

9.2 Instrument QC

9.2.1 Mass calibration.

Mass calibration of the analytical system shall be performed whenever the mass spectrometer is shut down for maintenance, repair, or whenever BFB tuning criteria are not met. Mass calibration is performed to ensure the accurate assignment of masses to ions generated in the ion volume of the mass spectrometer. Perfluorotributylamine (PFTBA) is the compound that shall be used to perform the mass calibration of the instrument. The PFTBA spectrum must meet the following criteria:

Mass	Target % of Mass 69
69	100
219	>30
502	>1

Isotope Ratio

Mass Ratio	Target %
70/69	0.54-1.6
220/219	3.2-5.4
503/502	7.9-12.3

If the PFTBA spectrum does not meet the criteria listed above, corrective action must be taken. The corrective action may be as simple as adjusting the voltages/retuning the MS. If retuning the MS does not produce adequate PFTBA spectra, further maintenance such as cleaning the ion source may be required.

9.2.2 GC/MS System Performance Check (BFB Tune Check)

BFB must be injected once at the beginning of each 24-hour period during which standards, blanks and samples are analyzed. The 24-hour time period begins at the moment of injection of the BFB. The time period ends after twenty-four hours have elapsed. If a sample is analyzed after the 24-hour time period has elapsed it must be re-analyzed.The analysis of the GC/MS performance check standard is performed by introducing approximately 30ng of BFB into the analytical system.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.

The ion abundance ratios must meet the following criteria:

Mass (m/z)	**Relative Ion Abundance Criteria***
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

*All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

If the BFB acceptance criteria are not met, the MS must be re-tuned. It may be necessary to clean the ion source or take other necessary actions to achieve the acceptance criteria.

9.3 Instrument QC

9.3.1 Initial Calibration

The GC/MS system must be re-calibrated whenever a corrective action that may change instrument response (e.g., ion source cleaning, column replacement, etc.) is performed. Re-calibration is also required if the continuing calibration acceptance criteria cannot be met

Calculate the response factor (RF) for each target compound for all five calibration standards using the following equation. The quantitation ions are listed in Appendix B.

$$RF = A_x / C_x$$

where: Ax = Area of quantitation ion of compound x
Cx = Concentration of compound x in ppbv

Calculate the mean RF for each compound using the equation:

$$RF_{avg} = \sum_{i=1}^{n} (RF_i / n)$$

where: RF_{avg} = Mean response factor
RF_i = RF of the compound at concentration i
n = Number of concentration values (i.e. 5)

Calculate the percent relative standard deviation (%RSD) of the RF values for each compound using the following equations:

$$\%RSD = (SD/RF_{avg}) x 100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - RF_{avg})}{n-1}}$$

Immediately after the initial calibration is performed, the data files shall be processed and checked to ensure that the following technical acceptance criteria have been satisfied.

The calculated %RSD for the RF for each compound must be less than 30%.

The retention time (RT) for each target compound at each concentration level must be within 0.06min of the mean RT for the compound.

If any of the criteria above have not been met because of a single concentration point in the curve, then that single point can be re-analyzed, and the data checked against the criteria. The system must be repaired so that the criteria are satisfied before any samples are analyzed. If repairs are made to the system, then a new initial calibration must be performed. The initial calibration should be checked for misidentified peaks due to retention time shifts.

If the initial calibration meets all the specified criteria, the remainder of the analytical period may be used for the analysis of blanks and samples. The initial calibration's average response factors are used to quantitate results.

9.4 Batch QC

9.4.1 Continuing Calibration Verification

A continuing calibration standard at a mid-level concentration (i.e. 200pptv) must be analyzed once every 24 hours. The daily calibration sequence starts with the injection of BFB and is analyzed after the BFB analysis meets the ion abundance criteria. Note that the mid level calibration standard acts as the continuing calibration verification when samples are analyzed within the same 24-hour period as the initial calibration.

Calculate the response factor (RF) for each target compound using the equation in Section 9.3.1.

For each target compound, calculate the percent difference (%D) between the continuing calibration RF and the mean RF in the most recent initial calibration using the following equation:

$$\%D = \frac{RF_c - RF_{avg}}{RF_{avg}} \, x\,100$$

where: RF_c = RF of the compound in the continuing calibration standard.
RF_{avg} = Mean RF of the compound in the most recent initial

calibration.

The %D for each target compound in the daily calibration must be within ± 30%.

If these criteria are not satisfied, rerun the continuing calibration standard, or run a new initial calibration. If the system undergoes corrective action (i.e. cleaning the ion source, changing the GC column) then a new initial calibration must be performed. Check the continuing calibration for mis-identified peaks due to retention time shifts.

If the continuing calibration meets all the specified criteria, the remainder of the analytical period may be used for the analysis of blanks and samples, using the initial calibration's average response factors to quantitate results.

9.4.2 Laboratory Control Spike - Secondary Source Standard analysis

The LCS must be analyzed and reported once every 24hr analytical sequence. The LCS must be from a secondary source. (i.e. different manufacturer of the standards used for calibration). If no secondary source standard is available, then a standard with a different lot number from the same manufacturer can be used, but it must be noted in the case narrative.

Calculate the percent recovery for each target compound in the LCS using the following equation:

$$\% \text{ Recovery} = \frac{\text{Concentration Reported}}{\text{Concentration Spiked}} \times 100$$

The percent recovery for each of the compounds in the LCS must be within the recovery limits of 70 to 130 percent. In addition, the retention time shift between the LCS and the most recent valid calibration for each of the target compounds must be within " 0.33 minutes.

If the technical acceptance criteria for the LCS are not met, this must be documented in the case narrative and associated results must be qualified.

9.4.3 Quantitation Limit Standard

The QLS at the 50pptv level must be analyzed and reported once per 24-hour analytical sequence. If running an initial calibration, the 50pptv standard serves as the QLS.

The QLS quantitation report must be checked immediately after it is analyzed, or as soon as possible, to determine that the following criteria have been met:

The concentration of each target compound must be within ± 40% of the expected concentration of QLS (i.e. percent recovery of 60-140%).

Calculate the percent recovery for each target compound in the QLS using the equation in Section 9.3.2.

If the technical acceptance criteria for the QLS are not met, this must be documented in the case narrative and associated results must be qualified.

A separate QLS shall be analyzed at the end of the analytical sequence in order to "bracket" the samples to monitor the instrument's performance.

9.4.4 Method Blank

A method blank must be analyzed before samples are analyzed (at least one (1) per batch of twenty (20) environmental samples), in order to demonstrate that the instrument is free of contamination and evaluate possible laboratory contamination.

The method blank reconstructed ion chromatogram (RIC) and quantitation report must be checked immediately after it is analyzed to determine that the following criteria have been met:

The method blank should not contain any target analytes at a concentration greater than the reporting limits and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

If the method blank result is greater than the reporting limit and contributes greater than 10% of the total amount of analyte found in the sample, the source of contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report. (Reference Appendix M of R9 Lab QA Plan)

If the method blank does not meet the technical acceptance criteria, a new blank shall be analyzed before any samples are analyzed. If the technical acceptance criteria are still not met, the Laboratory must ensure that method interferences caused by contaminants in solvents, reagents, canisters, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the

contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds.

If an analyte in the blank is found to be outside of control limits (i.e., contaminated) and the analyte is also found in associated samples, those sample results must be qualified in the report.

9.4.5 Laboratory Duplicate

Laboratory duplicate (sample duplicate) shall be analyzed after a sample is analyzed to evaluate analytical variability and reproducibility. Samples that are designated as trip blanks must not be used as a laboratory duplicate.

Laboratory duplicates shall be analyzed at a minimum of one (1) per batch of twenty (20) samples or SDG.

Calculate the relative percent difference (RPD) between the results of the sample and duplicate sample using the following equation:

$$RPD = \frac{/\ MS\ recovery - MSD\ recovery\ /}{(MS\ recovery + MSD\ recovery)/2} \times 100$$

The %D between the results of the sample and duplicate sample cannot exceed $\pm$ 20%. Poor performance in the duplicate may indicate a problem with the sample composition/matrix and associated results must be documented in the case narrative and qualified in the report.

9.4.6 Matrix Spikes and Matrix Spike Duplicate

The use of matrix spikes and matrix spike duplicates are not required by EPA Method TO-15 however shall be analyzed if requested by the client.

Upon request of the client, a matrix spike and matrix spike duplicate shall be analyzed concurrently at a minimum of once per batch of twenty (20) samples or SDG.

Calculate the percent recovery for each target compound in the matrix spike and matrix spike duplicate using the equation in Section 9.3.2.

The percent recovery for each of the compounds in the matrix spike and matrix spike duplicate must be within the recovery limits of 70 to 130 percent.

Calculate the relative percent difference (RPD) for each target compound in the

matrix spike and matrix spike duplicate using the equation in Section 9.4.5.

The relative percent difference (RPD) between the matrix spike and matrix spike duplicate must be #20%. If the technical acceptance criteria for the matrix spike and matrix spike duplicate are not met, this must be documented in the case narrative and associated results must be qualified.

9.5 Sample QC

No internal standard is used in this analysis. Target compounds are quantitated using an external calibration.

The use of surrogates is not required by the TO-15 method however shall be used if requested by the client.

9.6 Method Performance

Analysis of Volatile Organic Compounds in Soil Vapor 10/01/05 – 9/30/06

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2 s)
Vinyl chloride	Air	LCS	21	101	87.3-114
1,3-Butadiene	Air	LCS	21	94.0	78.6-109
1,1-Dichloroethene	Air	LCS	21	98.1	86.4-110
1,1,2-Trichloro-1,2,2-trifluoroethane(Freon 113)	Air	LCS	21	98.3	84.3-112
Dichloromethane	Air	LCS	21	96.1	84.1-108
1,1-Dichloroethane	Air	LCS	21	97.0	82.3-112
cis-1,2-Dichloroethene	Air	LCS	21	95.2	78.7-112
Chloroform	Air	LCS	21	98.4	84.4-112
1,1,1-Trichloroethane	Air	LCS	21	97.4	83.9-111
Carbon tetrachloride	Air	LCS	21	98.7	84.2-113
1,2-Dichloroethane	Air	LCS	21	94.9	79.2-111
Benzene	Air	LCS	21	94.9	75.4-114
Trichloroethene	Air	LCS	21	92.4	74.7-110

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2 s)
1,2-Dichloropropane	Air	LCS	21	97.0	78.3-116
Tetrachloroethene	Air	LCS	21	93.6	74.4-113
1,2-Dichlorobenzene	Air	LCS	21	88.3	74.4-102

10 DOCUMENTATION

Refer to SOP #845 and #846 for data package elements, requirements, and review procedures.

10.4 Standards

All standards (ICAL, ICV/CCV, QL, MS/MSD, and LCS) are recorded in the LIMS. A copy of each Analytical Standard Record associated with sample analysis must be included in the data package.

10.5 Reagents

Record all reagents used in this SOP in the LIMS.

10.6 Analytical sequence

The analytical sequence is documented in the Element database or in the instrument Run Log. Case Number, SDG number, date of analysis, QC solution IDs, analyst initials, lab sample IDs, client sample IDs, dilution factors and comments, if any, are recorded.

10.7 Analytical Report and Data Package

Analytical reports are produced using the Element database. The data package is produced from Element database and manual log records. Appendix E provides the typical format for data package deliverables.

10.8 Maintenance Logbook

Maintain a maintenance logbook for each instrument covered in this SOP. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control. All preventive or routine maintenance performed are to be documented, as well as repairs or corrective or remedial actions in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*. Major changes or upgrades to instrument hardware and software must be documented in the maintenance logbook.

10.9 SOP Distribution and Acknowledgement

Distribute the approved SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP; should be documented using the SOP Distribution and Acknowledgement List as shown in Appendix H.

11 REFERENCES

Entech Instruments, Inc.; 7000 Operators Manual; Version 1.1.

Entech Instruments Air Academy Course Manual.

Hewlett-Packard *MS ChemStation User=s Guide.*

Hewlett-Packard HP5890 Operating and Reference Manuals.

Hewlett-Packard *HP5972 Hardware Manual.*

NELAC Quality Systems, *2002 Standard*

U.S. Environmental Protection Agency, 1999. *Compendium Method TO-15; Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS).*

U.S. Environmental Protection Agency, 1990. *Statement of Work (SOW) for the Analysis of Air Toxics from Superfund Sites.*

U.S. Environmental Protection Agency Region 9 Laboratory, 2005. *Laboratory Quality Assurance Plan, Revision 8, January 31, 2005.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 110; Sample Receiving and Login.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 312; Cleaning and Certification of Specially Prepared Canisters for Air Sampling.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 820; Laboratory Discrepancy and corrective Action Reporting Practices.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 835; Chromatographic Integration Procedures.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 845; Internal Data Review*

and Report Preparation.

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 846; Internal Laboratory EPA Review of ESAT and EPA Generated Data.*

U.S. Environmental Protection Agency Region 9 Laboratory. *SOP 880; Demonstration of Capability*

Colorado Department of Public Health and Environment; Hazardous Materials and Waste Management Division, 2000. *Guidance for Analysis of Indoor Air Samples.*

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD

1. No internal standard is used. Target analyte concentrations are calculated using an external standard.

2. Method TO-15 does not require routine analysis and control of a Laboratory Control Standard, Quantitation Limit Standard or Matrix Spikes.

3. Method TO-15 does not implicitly include holding times for samples in canisters. A holding time of 30 days is included in this SOP based on studies referenced in Method TO-15 that indicate stable storage of up to 30 days for most VOCs.

4. This SOP includes a requirement that freshly prepared working standards and field samples be allowed to equilibrate for at least 2 hours in the Air Lab prior to analysis. This is not a requirement of Method TO-15.

5. Method TO-15 addresses sampling procedures. This SOP does not.

6. Method TO-15 addresses canister cleaning and certification. Canister cleaning and certification are not addressed in this SOP but are addressed in Region 9 Lab SOP #312.

7. Method TO-15 outlines typical Pre-concentrator and GC operating conditions. This SOP included Pre-concentrator and GC operating conditions that have been found to provide optimum results.

8. Method TO-15 requires 50ng BFB to be used for checking the instrument tune. This SOP requires a more stringent amount of approximately 30ng of BFB.

APPENDIX B.
ANALYTES AND QUANTITATION LIMITS

The following table provides the target analytes list for this SOP with the Chemical Abstracts Registry Number (CASRN) and quantitation limits (QL). m^3

Analyte	CASRN	Air QL pptv	Air QL µg/m^3
Vinyl chloride	75-01-4	50	0.13
1,3-Butadiene	106-99-0	50	0.11
1,1-Dichloroethene	75-35-4	50	0.20
1,1,2-Trichloro-1,2,2-trifluoroethane(Freon 113)	76-13-1	50	0.38
Dichloromethane	75-09-2	50	0.17
1,1-Dichloroethane	75-34-3	50	0.20
cis-1,2-Dichloroethene	156-59-2	50	0.20
Chloroform	67-66-3	50	0.24
1,1,1-Trichloroethane	71-55-6	50	0.27
Carbon tetrachloride	56-23-5	50	0.31
1,2-Dichloroethane	107-06-2	50	0.20
Benzene	71-43-2	50	0.16
Trichloroethene	79-01-6	50	0.27
1,2-Dichloropropane	78-87-5	50	0.23
Tetrachloroethene	127-18-4	50	0.34
1,2-Dichlorobenzene	95-50-1	50	0.30

APPENDIX C.
QUALITY CONTROL MEASURES AND CRITERIA

QC Measure	Criteria	Frequency
BFB	See 9.2.1	Once every 24-hours
IC RSD	≤ 30%	
CCV %D	≤ 30%	Once every 24-hours
LCS %Recovery	70-130%	Once every 24-hours
MB	<½ QL	Once every 24-hours
QLS %Recovery(	60-140%	Two every 24-hours, must bracket sample runs
Matrix Duplicate Precision, RPD	≤ 20%	If requested. one per SDG or 20 field samples
Sample Retention Time Drift	±0.33 minutes (20 sec.) from CCV	
Demonstration of Capability	≤ 30%	Annually
Precision and Accuracy Study	70-130%	Annually

QUANTITATION IONS

Analyte	Primary Ion, m/z	Secondary Ion(s), m/z
Vinyl chloride	62	64
1,3-Butadiene	54	39, 53
1,1-Dichloroethene	61	96, 63
1,1,2-Trichloro-1,2,2-trifluoroethane(Freon 113)	151	101, 103
Dichloromethane	49	84, 86
1,1-Dichloroethane	63	27, 65
cis-1,2-Dichloroethene	96	61, 98
Chloroform	83	85, 47
1,1,1-Trichloroethane	97	99, 61
Carbon tetrachloride	117	119, 121
1,2-Dichloroethane	62	27, 64
Benzene	78	77
Trichloroethene	130	132, 95
1,2-Dichloropropane	63	41, 62
Tetrachloroethene	166	164, 131
1,2-Dichlorobenzene	146	148, 111

APPENDIX D.
INSTRUMENT PARAMETERS

ENTECH 7000/7100 PRE-CONCENTRATOR PARAMETERS

TO15 CONCENTRATION EVENTS

1. Wait for temperature to reach setpoints
2. Wait for GC Ready
3. Cool Module 1 to trapping temperature
4. Preflush with internal standard
5. Trap internal standard
6. Preflush with analytical standard (BFB)
7. Trap analytical standard (BFB)
8. Preflush with sample
9. Trap sample
10. Preflush with Helium sweep/purge Gas
11. Trap Helium sweep/purge gas
12. Preheat Module 1
13. Transfer VOCs from M1 to M2
14. Wait for GC Ready
15. Cool focusing trap
16. M2 preheat
17. Transfer M2 to M3
18. Heat M3, Inject, Start GC
19. Preflush with next sample
20. System Bakeout
21. Wait time after injection

BFB ANALYSIS

PARAMETER	SETTING
Sample:	
Preflush (sec)	10
Trap (cc/min)	100
Volume (cc)	160 (Zero Air Blank)
Internal Standard:	(BFB tuning standard is ran without internal standard)
Preflush (sec)	5
Trap (cc/min)	100
Volume (cc)	0
Analytical Standard (BFB):	
Preflush (sec)	5
Trap (cc/min)	100
Volume (cc)	40
Sweep/Purge:	
Preflush (sec)	10
Trap (cc/min)	100
Volume (cc)	75

SAMPLE ANALYSIS **Note: These parameters may be adjusted in order to achieve optimum results.**

PARAMETER	SETTING
Sample:	
Preflush (sec)	10
Trap (cc/min)	100
Volume (cc)	400
Internal Standard:	(SIM analysis does not use an internal standard)
Preflush (sec)	5
Trap (cc/min)	100
Volume (cc)	0
Analytical Standard (BFB):	
Preflush (sec)	5
Trap (cc/min)	100
Volume (cc)	0
Sweep/Purge:	
Preflush (sec)	10
Trap (cc/min)	100
Volume (cc)	75
M1? M2:	
Preflush (sec)	N/A
Trap (cc/min)	10
Volume (cc)	40
Module 1:	
Trap temp(^{o}C)	-150
Preheat?	Yes
Preheat temp (^{o}C)	10
Desorb temp (^{o}C)	12
Bake temp (^{o}C)	150
Bake time (min)	7
Bulkhead 1:	
Trap temp (^{o}C)	10
Desorb temp (^{o}C)	12
Bake temp (^{o}C)	150
Module 2:	
Trap temp(^{o}C)	-30

Preheat?		No
Preheat temp (°C)		N/A
Desorb temp (°C)		180
Bake temp (°C)		190
Desorb (min)		2.5
Bulkhead 2:		
Trap temp (°C)	30	
Desorb temp (°C)		60
Bake temp (°C)		150
Module 3:		
Trap temp(°C)	-160	
Focus?	Yes	
Inject time (min)		3
Bake temp (°C)		100
Bake time (min)		4
Bake on event #		3
Wait (min)		0
Sample Transfer temp (°C)		80
GC Transfer temp (°C)	100	
MPOS Valve temp (°C)		100

OPTIONS

Wait for GC before final focusing

Use Pressure Compensation

- Pressure Compensation Factor: 14

Additional Cryogenic Temperature Control

-Raise M2 temperature before Helium sweep
M2 Helium sweep temperature (CTD): -10 deg C

-Max temperature below set point before adding heat
Control heaters during trapping
Cryo Module 1: 10 deg C
Cryo Module 2: 10 deg C

Extra M2 ! M3 transfer after inject: 2 min

GC PARAMETERS

HP5890/HP6890

Note: These parameters may be adjusted in order to achieve optimum results.

The operating methods for the Gas Chromatograph that is interfaced with the HP5972MS/HP5973MS are as follows:

BFB / Air Sample Analysis

Parameter	Setting
Solvent Delay	5.6 minutes
Injector Temperature	100EC
Oven Equib. Time	0.5 minutes
Detector B	280EC
Initial Oven Temp	35EC
Initial Oven Time	6.0 minutes
Temperature Ramp - level 1	8EC/minute
Final Temp - level 1	100°C
Final Hold Time - level 1	0.0 minutes
Temperature Ramp - level 2	10EC/minute
Final Temp - level 2	160°C
Final Hold Time - level 2	0.0 minutes
Temperature Ramp - level 3	12EC/minute
Final Temp - level 3	220°C

Parameter	Setting
Final Hold Time - level 3	4.88 minutes
Column Flow rate	1.2mL/min
EM Voltage	ABS (varies with tune)
Inlet B Pressure	19.1psi

APPENDIX E.
CHEMSTATION FILE NAMING CONVENTIONS

File data, methods, and sequences on ChemStation computers and the LAN using the following naming conventions:

Directories

On the Workstation:
Data: C:\MSDCHEM\1\Data\MDDY or D:\MSDCHEM\1\Data\MDDYS
Methods: C:\MSDCHEM\1\Methods or D:\MSDCHEM\1\Methods
Sequences: C:\MSDCHEM\1\Sequence or D:\MSDCHEM\1\Sequence
For system controlling multiple instruments, 1 may be changed to reflect the instrument number

System running ChemStation versions C & D HPCHEM is named as MSDCHEM

On the LAN:
Data: I:\Room Number\Instrument\Year\MDDYS
Methods: I:\Room Number\Instrument\Methods
Sequences: I:\ Room Number\Instrument\Sequence

Methods
MDDYITA

Sequence
MDDYS

Data Files
For GC:
MDDYICSS

For GC/MS
MDDYIQSS

Variables

A: Enter analysis, as follow:

1,4-Dioxane	X
504	E
TO15	A
BNA	B
BNA-L (SIM)	L
Congeners	C
P/P	P

PCB	P
RSK175	R
Soil Gas/Air	A
TPH-G	G
TPH-D	D
VOA	V

C: Channel: A = front
B = back (if applicable)

DD: Day

I: Instrument
6890 series GCs by last number in name: e.g. 6890-1 = 1 except 5890-2 = A
All GC/MSs by last letter in name: e.g. 5973L = L

M: Month 1-9, A: October, B: November, C: December

Q: QC type

BFB	F
Blank	B
CV	C
Degradation	P
DFTPP	D
IB	Z
IC	I
LCS	L
LCV	Q
Second Source	S
MS/MSD	M

S: Sequential number 1,2 3, ….

T: Matrix Type (if applicable)

Water	W
Solid	S
Air	A
Oil	O
Other	X

Y: Year i.e. 5 for 2005

APPENDIX F.
PREVENTATIVE MAINTENANCE REQUIREMENTS

GC Maintenance

Item	Frequency	Actions/Comments
Gas purifiers (carrier gas & detector gas)	Annually	Replacement schedule is based on capacity and grade of gases. In general, replace non-indicating traps every 6-12 months or when indicating traps start to change color. Replace indicating traps when indicating material is spent.
Flow meter calibration	2 years	Manual flow meters only.
Inlet Hardware	Annually	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Inlet Septum	As needed	Replace ferrules when changing columns and or inlet parts.
Column Maintenance	As needed	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules/Column Union	As needed	Replace ferrules when changing columns and or inlet parts.
Purge/Sample Lines	As needed	Bake out and purge. Clean with organic free water if necessary.
Trap	As needed	Replace when loss of performance.

MS Maintenance

Task	Every Week	Every 6 Months	Every Year	As Needed
Tune the MSD				✓
Check the foreline pump oil level	✓			
Check the calibration vials		✓		
Replace the foreline pump oil		✓		
Clean the ion source				✓
Check the carrier gas traps on the GC				✓
Replace worn out parts				✓
Lubricate side-plate or vent valve O-rings				✓

APPENDIX G.
REGION 9 LABORATORY TO-15 RUN LOG

Analyst: _____ **Internal Std. ID:** _____ **Calibration Std. ID:** _____ **Method:** TO-15 SIM

Analysis Date: _____ **Internal Std. Volume:** _____ **LCS ID:** _____ **Instrument:** _____

BFB Std. ID: _____ **EM Volts** _____

	Filename	Can #	Time	Case#	SDG	Sample ID	Lab ID	Sample Vol (mL)	Dilution	Dilution Calc.	M*	Comments
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

M* = Manual Integration, enter the compound number(s) of manually integrated peak(s)

Dilution Calc. = Dilution logbook # and page # where dilution calculations are present.

Reviewed By:_____ Date:_____

APPENDIX H.
SOP DISTRIBUTION AND ACKNOWLEDGEMENT LIST

STANDARD OPERATING PROCEDURE: 314
Revision: 1, Effective: 11/17/06

LOW LEVEL ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN AIR
TO-15 SIM (SELECTIVE ION MODE)

I have received, read, and understood the above referenced SOP and agree to comply with the SOP when performing the subject procedure until the effective date of future revisions.

NAME	DATE DISTRIBUTED	SIGNATURE	DATE