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CCP-TP-143

Revision 0

CCP
Carlsbad Environmental Monitoring and Research Center Headspace Gas Analysis

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RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	08/18/2006	Initial issue.

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1.0 PURPOSE

This procedure describes and implements the process for analyzing headspace gas (HSG) in transuranic (TRU) waste using the gas chromatograph/mass spectrometer (GC/MS) and gas chromatograph/thermal conductivity detector (GC/TCD) methods. The requirements for waste characterization are addressed in CCP-PO-001, *CCP Transuranic Waste Characterization Quality Assurance Project Plan*.

1.1 Scope

This procedure applies to sample receiving, sample handling, system startup, calibration checks, daily checks, and sample and standard analysis. The analytical process is based on the introduction of samples via an autosampler onto a capillary column for Volatile Organic Compounds (VOC) analysis and onto a Porous Layer Open Tubular (PLOT) column for hydrogen and methane (H_2 and CH_4) using fixed-volume injection loops. Internal standards are introduced with each standard and sample. A Mass Spectrometer (MS) is used for detection of the VOCs and Thermal Conductivity Detector (TCD) is used for the detection of H_2 and CH_4. The target compounds are listed in Table 1, Gas Target Analyte List and Quality Assurance Objectives.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- Compendium Method TO-14A, *Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. Second Edition. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, January 1999

- DOE/CAO-95-1076, *Performance Demonstration Program Plan for Analysis of Simulated Headspace Gas*

Referenced Documents

- DOE/WIPP 01-3194, *CH TRU Waste Content Codes,* Appendix B, List of Additional Flammable Volatile Organic Compounds Evaluated by the CH-TRAMPAC Methodology
- *40 Code of Federal Regulations* (CFR), Part 261, Appendix VIII, *Hazardous Constituents*
- National Institute of Science and Technology Mass Spectral Library
- CCP-PO-001, *CCP Transuranic Waste Characterization Quality Assurance Project Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*
- CCP-QP-008, *CCP Records Management*
- CCP-QP-011, *CCP Notebooks and Logbooks*
- CCP-QP-022, *CCP Software Quality Assurance Plan*
- CCP-TP-056, *CCP HSG Performance Demonstration Plan*
- CCP-TP-093, *CCP Sampling of TRU Waste Containers*
- CCP-TP-142, *CCP Preparation of Canisters for Headspace Gas Sampling for Carlsbad Environmental Monitoring and Research Center*

2.2 Training Requirements

2.2.1 Personnel performing this procedure will be trained and qualified in accordance with CCP-QP-002, *CCP Training and Qualification Plan* prior to performing this procedure.

2.3 Equipment List

2.3.1 GC/MS-GC/TCD Analytical System

[A] GC with TCD.

NOTE
The length and diameter of the column used must be documented in the GC/MS Operator's Logbook. The length and diameter must be consistent with that used to analyze the most recent HSG Performance Demonstration Program (PDP) blind sample analysis cycle.

[B] Column A (for VOC analysis) - 60-meter (m) X 0.250-millimeter (mm) ID 1.4-micrometer (µm) stationary film thickness; Model RTX-624 or equivalent.

[C] Column B (for H_2 and CH_4 analysis) - HP Mole SIV, 30m X 0.32mm X 25 micrometer, or equivalent..

[D] MS (for VOC analysis) - Capable of scanning from 30 - 300 atomic mass unit (amu) every three seconds or less, using 70 volts (nominal) electron energy in the electron impact mode.

[E] Entech 7032A-L MiniCan Autosampler or equivalent.

[F] Entech 4600A Dynamic Diluter or equivalent.

[G] Data System - Allows the continuous acquisition and storage of mass spectra and TCD data obtained throughout the duration of the chromatographic program is interfaced to the GC/MS-GC/TCD Analytical System. The data system has software that allows a search of a GC/MS data file for ions of a specified mass and allows plotting of such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The data system is capable of forward library searching of the National Institute of Science and Technology (NIST) Mass Spectral Library, which include Title 40 *Code of Regulations* (CFR), Part 261, Appendix VIII, *Hazardous Constituents* compounds.

[H] Liquid, fixed needle syringes, ranging in volume from 1 microliter (µL) to 500 µL.

NOTE
Laboratory canisters DO **NOT** required pressure gauges.

[I] SUMMA® canisters or equivalent with pressure gauge. The pressure gauge must be helium leak tested to 1.5 X 10E-7 standard cubic centimeters per second (cc/s). The canister size can have 100 mL to 6000 mL capacity.

[J] Calibrated Thermometer(s). The ambient air temperature sensor shall be calibrated or replaced annually.

[K] Calibrated Barometer.

[L] Calibrated pressure gauge.

[M] Nano Pure Water System, capable of producing American Society of Testing and Materials (ASTM) Type 1 water.

2.3.2 Reagents

[A] Ultra High Purity (UHP) helium.

[B] UHP nitrogen.

[C] 4-Bromofluorobenzene (BFB).

2.3.3 Standard Gases

[A] The H_2 and CH_4 standards shall be procured as compressed gas standards and certified by the manufacturer. H_2 and CH_4 standards in nitrogen are at the following approximate concentrations:

Level 1 0.075 volume percent (vol%)
Level 2 1.0 vol%
Level 3 10 vol%
Level 4 20 vol%

[B] Internal standard gas mixture of the two internal standards (fluorobenzene and chlorobenzene-d5) in nitrogen. The internal standard shall be procured as a compressed gas standard and certified by the manufacturer and have a known valid relationship to a nationally recognized standard (e.g., NIST), when available.

2.3.4 Liquid Standards

[A] The VOC calibration standards shall be procured certified by the manufacturers and have a known valid relationship to a nationally recognized standard (e.g., NIST), when available.

[B] The Laboratory Control Standard (LCS) shall be procured certified by the manufacturers or have a known valid relationship to a nationally recognized standard (e.g., NIST), when available. The LCS must be from a second supplier or lot number, different from the initial calibration (ICAL) standard.

2.4 Software

2.4.1 The software listed below complies with the requirements of CCP-QP-022, *CCP Software Quality Assurance Plan.*

[A] Agilent MSD Chemstation®.

[B] Entech SmartLab 7032 Autosampler Software.

[C] HSG_StdCalc.xls spreadsheet.

[D] HSGDP.xls spreadsheet.

[E] HSG_MDL_Calc.xls spreadsheet.

[F] Entech SmartLab 4600A Dynamic Diluter Software.

2.5 Precautions and Limitations

2.5.1 If this procedure CAN **NOT** be implemented as written, work will be STOPPED, and the HSG Technical Supervisor (TS) will be notified. Work will **NOT** be resumed until the procedure has been corrected.

2.6 Prerequisite Actions

2.6.1 None.

2.7 Definitions

2.7.1 None.

2.8 Quality Control Requirements

NOTE

Calculations are performed with software for quality control (QC) samples and samples. The equations are shown in Attachment 1, Calibration and Quality Control (QC) Equations.

NOTE

All standards used for calibrations and/or QC checks shall be used or taken out of service prior to the expiration date of the standard itself or the primary standard from which it was made.

2.8.1 BFB Tune Check

[A] BFB is introduced into the GC/MS system. Upon analysis of BFB, the mass spectrum shall meet the criteria listed in Table 1, 4-Bromoflourobenzene (BFB) Key Ions and Abundance Criteria. Acceptable correlation to these criteria must be demonstrated for every 12-hour period the system is used for analysis.

2.8.2 Initial Calibration (ICAL)

[A] An ICAL relating instrument response to parts per million volume (ppmv) of analyte is required to implement the analysis method. A new ICAL is required whenever indicated as corrective action for noncompliant QC results or whenever the instrument operating conditions or chromatographic column are changed.

[B] An ICAL for VOCs is valid if the percent relative standard deviation (%RSD) of the Relative Response Factors (RRFs) for each analyte is less than 35 percent. If this specification is not met, corrective action must be taken (i.e., identify and correct problem, and recalibrate).

[C] An ICAL for H_2 and CH_4 is valid if the correlation coefficient (r^2) is greater than 0.990. If this specification is not met, corrective action must be taken (i.e., identify and correct problems and recalibrate).

2.8.3 Continuing Calibration (CCAL)

[A] A CCAL is required at the beginning of every 12 hours of instrument operation.

[B] A CCAL is valid if the percent difference (%D) between average RRF of ICAL and the RRF of CCAL is less than or equal to 30 percent for all target analytes and the retention time (RT) of each analyte is within 30 seconds of the average ICAL RT. If this specification is not met, corrective action must be taken (e.g., identify and correct problem, recalibrate) before sample analysis can proceed.

2.8.4 Internal Standard Areas (ISAs)

[A] The ISAs of the CCAL must be within 50 percent to 200 percent of the average ISAs from the current valid ICAL. If this specification is not met, corrective action must be taken (e.g., identify and correct problem, recalibrate) before analysis can proceed.

[B] The ISAs of each sample must be within 50 percent to 200 percent of the associated CCAL ISAs. If this specification is not met, corrective action must be taken (e.g., identify and correct problem, recalibrate) before analysis can proceed. Any sample not meeting this requirement must be reanalyzed.

2.8.5 Laboratory Blank (LB)

[A] One LB must be analyzed daily after the CCAL but before sample analysis.

[B] LB results are acceptable if the amount of any individual analyte in the blank is less than or equal to 3 times the Method Detection Limit (MDL).

[C] If an analyte is detected above the MDL but less than or equal to 3 times MDL in the LB, the analytical results must be B flagged.

[D] If an analyte is greater than 3 times the MDL, then initiate a Nonconformance Report (NCR) in accordance with CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*, and the appropriate qualifier flag in Table 4, Data Flags, assigned to data.

2.8.6 LCS

[A] An LCS contains at least six of the target compounds listed in Table 1.

[B] One LCS must be analyzed per analytical batch

[C] LCS results are acceptable if the %R of all analytes is 70 percent to 130 percent. If the LCS results do not meet specifications, the LCS may be remade or reanalyzed to eliminate LCS preparation as the source of error.

[D] If the criteria in step 2.8.6[C] cannot be met, then initiate an NCR in accordance with CCP-QP-005, and the data will be assigned a qualifier flag in Table 4.

2.8.7 Laboratory Duplicates (LDs)

[A] A field canister must be analyzed in duplicate at a frequency of once per analytical batch.

[B] For samples having analyte concentrations greater than or equal to the respective Program Required Quantitation Limits (PRQL), LD RPD must be less than or equal to 25 percent. If there are no targets above the PRQL detected in the field canister, then a second LCS will be used as the LD. The two LCSs must meet the same duplicate sample criteria. If this specification is not met, an NCR must be initiated per CCP-QP-005.

2.8.8 MDLs and Method Performance Samples

[A] Demonstration of the MDL shall be achieved by analyzing Method Performance Samples. The analysis of seven replicates shall meet the criteria specified for precision, accuracy, and MDL in Table 1.

[B] MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero.

[C] MDLs are expressed in ppmv for VOCs and vol% for H_2 and CH_4, and must be demonstrated to be less than or equal to the program-required MDLs (see Table 1).

[D] MDLs must be determined semiannually, at a minimum.

[E] After the initial MDL demonstration is acceptable, acceptable performance must be demonstrated semiannually by analyzing at least four replicate method performance standards.

2.8.9 RT Windows

[A] Identify the target analytes, H_2 and CH_4, by RT. The RT for each must be within the RT window determined from the initial calibration analyses. The RT windows are calculated as the mean RT plus or minus five percent of the mean RT.

2.8.10 Representativeness

[A] Representativeness is the degree to which sample data represent the population being studied.

[A.1] Sampling Representativeness - Steps to ensure samples are representative include:

(a) Selection of the correct Drum Age Criteria (DAC) Scenario and waste packaging configuration and meeting DAC equilibrium times.

(b) Sample canister cleaning and leak check after assembly.

(c) Sampling equipment leak check after sample collection.

(d) Sampling equipment cleaning or disposal after use.

(e) Use of sample canisters with passivated internal surfaces.

(f) Use of low-internal-volume sampling equipment.

(g) Collection of samples with low-sample volume to available headspace volume ratio, less than 10 percent.

(h) Performance audits.

(i) Documented pressure regulation of all activities associated with sampling.

(j) Collection of Equipment Blanks (EBs), Field Blanks (FBs), and Field Duplicates (FDs) at the specified frequencies.

(k) Pressure sensors and temperature sensors calibrated before initial use and annually using NIST or equivalent standards.

[B] Analytical Representativeness is achieved by collecting sufficient number of samples using clean sampling equipment that does not introduce sample bias.

2.8.11 Comparability

[A] The degree to which one data set can be compared to another. For HSG analysis, data generated through analysis of samples from different sites shall be comparable. Each site shall achieve comparability by using standardized methods and traceable standards and by successfully participating in the PDP.

2.8.12 Completeness

[A] Analytical completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Valid results are defined as results that meet the data usability criteria based on application of the QC Criteria specified in Table 1, and meet the detection limit, calibration representativeness, and comparability criteria. Completeness, expressed as the percent complete (%C), is calculated as follows:

$$\%C = \frac{V}{n} * 100$$

Where:

V = the number of valid analytical results obtained.

n = the number of samples submitted for analysis.

%C must be greater than or equal to 90 percent. The completeness is calculated at the project level.

2.8.13 Analytical Batch

[A] An analytical batch may consist of up to 20 samples excluding laboratory QC samples. Prior to sample analysis the BFB Tune Check, CCAL, and LB must be analyzed.

3.0 RESPONSIBILITIES

3.1 HSG Operator

3.1.1 Operates the HSG instruments and equipment.

3.1.2 Starts documentation for the Batch Data Report (BDR).

3.2 Lead Operator (LO)

3.2.1 Oversees all operations for HSG analysis.

3.3 Independent Technical Reviewer (ITR)

3.3.1 Responsible for reviewing 100 percent of the BDRs and completes Attachment 3, Independent Technical Reviewer Checklist to document the review.

3.4 Facility Records Custodian

3.4.1 Receives, processes, and transmits records generated by this procedure in accordance with CCP-QP-008, *CCP Records Management*.

4.0 PROCEDURE

NOTE
Steps 4.1 through 4.5 are for preparation of standards and are performed as needed.

HSG Operator

4.1 Preparation of BFB Tune Check Standard

4.1.1 Remove the liquid BFB (neat) from the freezer, **AND** gently shake.

4.1.2 **IF** using a new ampule,
THEN transfer the contents to a crimp-top glass vial, **AND** seal.

4.1.3 Transfer the vendor label to the glass vial.

4.1.4 Attach the liquid injection fitting to the Autosampler 2 pressurization port.

NOTE
The regulator pressure should be set to approximately 40 pounds per square inch (psi). The 0.075 percent H_2 and CH_4 standard is a recommendation only. Any of the H_2 and CH_4 standards can be used in preparing the tuning standard.

4.1.5 OPEN the 0.075 percent H_2 and CH_4 standard cylinder valve.

4.1.6 OPEN the Autosampler software for Autosampler 2.

4.1.7 Press the FILL Button to enter the canister pressurization controls screen.

4.1.8 Set the flush time to one minute, **AND** press the GO Button.

4.1.9 OPEN the fitting valve by attaching a quick-connect fitting to the liquid injection fitting, **AND** press the FLUSH Button on the Autosampler 2 to purge with gas.

4.1.10 Take a cleaned and evacuated one-liter canister, **AND** confirm its evacuation with a pressure gauge.

[A] **IF** evacuated,
THEN attach the canister to the liquid injection fitting.

[B] **IF NOT** evacuated,
THEN select another cleaned and evacuated canister. **AND** attach to the liquid injection fitting.

4.1.11 Set the Autosampler software to dilute to a target pressure of 25 pounds per square in absolute (psia), **AND** press the GO Button.

4.1.12 OPEN the canister, **AND** inject approximately 4.6 µL of BFB into the injection fitting.

4.1.13 Press the PRESSURIZE Button on Autosampler 2.

4.1.14 **AFTER** the canister is pressurized,
THEN CLOSE the canister valve, **AND** remove the canister from the liquid injection fitting.

4.1.15 CLOSE the 0.075 percent H_2 and CH_4 standard cylinder valve, **AND** return the BFB liquid standard to the freezer.

4.2 Preparation of Initial Calibration Standards

NOTE

The required volume injected for each standard is calculated using the Excel workbook template, HSG_StdCalc.xls spreadsheet (see Attachment 13, Standard Preparation VOC and H_2 and CH_4 for an example), for the particular primary VOC standard.

For example, standards prepared on 5/1/04 using primary standard 001-026-002 would use the template 001-026-002_Template and have a workbook saved as 001-026-002_050104.xls spreadsheet.

4.2.1 OPEN the appropriate template in C:\headspace gas\standard preparation, **AND** save the file as the organic compound (OC) primary standard ID with a date extension.

NOTE

The worksheet will calculate the approximate injection volume for each standard.

4.2.2 Record the current barometric pressure, room temperature, canister volume, and date/time into the appropriate worksheet cells.

4.2.3 Remove the multi-compound liquid VOC calibration standard from the freezer, **AND** gently shake.

[A] **IF** using a new ampule,
THEN transfer the contents to a crimp-top glass vial and seal, **AND** transfer the vendor label to the glass vial.

4.2.4 Attach the liquid injection fitting to the Autosampler 2 pressurization port**.**

4.2.5 Attach the gas transfer line from Autosampler 2 to the regulator of the 0.075 percent H_2 and CH_4 standard cylinder.

NOTE

The regulator pressure should be set to approximately 40 psi.

4.2.6 OPEN the 0.075 percent H_2 and CH_4 standard cylinder valve.

4.2.7 OPEN the Autosampler software for Autosampler 2.

4.2.8 Press the FILL Button to enter the canister pressurization controls screen.

4.2.9 Set the flush time to one minute, **AND** press the GO Button.

4.2.10 Attach a vented quick-connect fitting to the liquid injection fitting to open the fitting valve, **AND** press the FLUSH Button on the Autosampler 2 to purge with H_2 and CH_4 standard.

4.2.11 Note the current psia reading in the appropriate software box (local absolute pressure reading) when complete.

[A] Type this same value into the software TARGET PRESSURE field.

4.2.12 Take a cleaned and evacuated canister, **AND** confirm its pressure is below zero psig with a pressure gauge.

[A] **IF** evacuated,
THEN attach the canister to the injection fitting.

[B] **IF NOT** evacuated,
THEN select another cleaned and evacuated canister, **AND** attach to the liquid injection fitting.

[C] Using a clean syringe, aliquot the appropriate amount of VOC calibration standard for the first calibration standard using the HSG_StdCalc.xls spreadsheet as a guideline.

[D] Enter the actual amount of standard drawn into the syringe into the ACTUAL INJECTION field in HSG_StdCalc.xls spreadsheet for that standard level.

[E] Set the Autosampler software to dilute to target pressure, **AND** press the GO Button.

[F] OPEN the canister, **AND** inject the VOC calibration standard into the injection fitting.

[G] Press the PRESSURIZE Button on Autosampler 2.

[H] **AFTER** the software shows the canister is pressurized, **THEN** CLOSE the canister valve, **AND** remove it from the liquid injection fitting.

[I] Prepare ICAL5 on Autosampler 2 by repeating steps 4.2.12[A] through 4.2.12[H].

[J] CLOSE the 0.075 percent H_2 and CH_4 cylinder valve.

[K] Prepare ICAL2 on Autosampler 2 using the 1 percent H_2 and CH_4 standard in accordance with steps 4.2.6 through 4.2.12[H].

[L] CLOSE the 1 percent H_2 and CH_4 cylinder valve after preparation of the ICAL2.

[M] Prepare ICAL3 on Autosampler 1 using the 10 percent H_2 and CH_4 standard in accordance with steps 4.2.6 through 4.2.12[H].

[N] CLOSE the 10 percent H_2 and CH_4 cylinder valve after preparation of the ICAL3.

[O] Prepare ICAL4 on Autosampler 1 using the 20 percent H_2 and CH_4 standard in accordance with steps 4.2.6 through 4.2.12[H].

[P] CLOSE the 20 percent H_2 and CH_4 cylinder valve after preparation of the ICAL4.

[Q] Return the VOC calibration standard to the freezer.

[R] Attach a vented quick-connect fitting to the Autosampler 3 pressurization port to allow for port flushing.

NOTE

The regulator pressure should be set to approximately 40 psi.

[S] OPEN the internal standard cylinder valve.

[T] OPEN the Autosampler software for Autosampler 3.

[U] Press the FILL Button to enter the canister pressurization control screen, set the flush time to one minute, **AND** press the GO Button.

[V] Attach a vented quick-connect fitting to the FILL port on Autosampler 3.

[W] Press the FLUSH Button on Autosampler 3 to purge with internal standard.

[W.1] Remove the vented quick-connect fitting when purging is completed.

[X] Attach a calibration standard canister to the pressurization port, **AND** OPEN valve.

[Y] Set the Autosampler software to dilute to a relative dilution factor of 2.0 times, **AND** press the GO Button.

[Z] Press the PRESSURIZE Button on Autosampler 3.

[AA] **AFTER** the Autosampler software indicates the pressurization is done,
THEN CLOSE the canister valve, **AND** remove it from the pressurization port.

[BB] Repeat steps 4.2.12[X] through 4.2.12[AA] for the remaining four standards.

[CC] CLOSE the internal standard cylinder valve.

[DD] **AFTER** the HSG_StdCalc.xls spreadsheet is completely filled out,
THEN save it, print a hardcopy, **AND** print name, sign, and date.

[EE] Place the hard copy in the BDR holding folder.

[FF] Place a tag to the canister to label the canisters with the following information:

[FF.1] Calibration Standard.

[FF.2] Intermediate ID.

[FF.3] Date Prepared.

[FF.4] Preparer's Initials.

[FF.5] Level of Concentration.

4.3 Preparation of LCS

NOTE

A second source H_2 and CH_4 standard is **NOT** required. The 10 percent H_2 and CH_4 calibration standard is used, but a different concentration could be used if needed. The required volume injected for the LCS is calculated using the HSG_StdCalc.xls spreadsheet for the particular primary VOC standard.

For example, an LCS prepared on 5/1/04 using primary standard 001-026-008 would use the template 001-026-008_Template and have a workbook saved as 001-026-008_050104LCS.xls spreadsheet. The master workbook used to make the templates for the primary VOC standards is called HSG_StdCalc.xls spreadsheet.

4.3.1 OPEN the template for the primary standard that will be used to prepare the LCS, **AND** save the file as the primary ID with a date extension and LCS.

NOTE

The worksheet will calculate the approximate injection volume for the LCS.

[A] Record the current barometric pressure, room temperature, canister volume and date/time into the appropriate worksheet cells.

[B] Remove the multi-compound liquid VOC standard from the freezer, **AND** gently shake.

[B.1] **IF** using a new ampule,
THEN transfer the contents to a crimp-top glass vial and seal, **AND** transfer the vendor label to the glass vial.

[C] Attach the liquid injection fitting to the Autosampler 1 pressurization port.

[D] Attach the Autosampler 1 gas transfer line to the 10 percent H_2 and CH_4 standard gas regulator.

[E] OPEN the 10 percent H_2 and CH_4 standard cylinder valve.

[F] OPEN the Autosampler 1 software, **AND** press the FILL Button to enter the canister pressurization controls screen.

[G] Set the flush time to one minute, **AND** press the GO Button.

[H] Attach a vented quick-connect fitting to the liquid injection fitting to open the fitting valve, **AND** press the FLUSH Button on the Autosampler 1 to purge with standard.

[H.1] Note the current psia reading in the Autosampler software, **AND** type this same value into the TARGET PRESSURE field when the purge is finished.

[I] Remove the vented quick-connect fitting from Autosampler 1.

[J] Take a cleaned and evacuated canister, **AND** confirm its evacuation with a pressure gauge.

[J.1] **IF** evacuated,
THEN attach the canister to the injection fitting.

[J.2] **IF NOT** evacuated,
THEN select another cleaned and evacuated canister,
AND attach to the liquid injection fitting.

[K] Using a clean syringe, aliquot the appropriate amount of VOC standard for the LCS using the HSG_StdCalc.xls spreadsheet approximate injection value as a guideline.

[L] Enter the actual amount of standard drawn into the syringe into the ACTUAL INJECTION field in the HSG_StdCalc.xls spreadsheet for that standard.

[M] Set the Autosampler software to dilute to target pressure, **AND** press the GO Button.

[N] OPEN the canister, **AND** inject the LCS standard into the injection fitting.

[O] Press the PRESSURIZE Button on Autosampler 1.

[P] **AFTER** the software shows the canister is pressurized, **THEN** CLOSE the canister valve, **AND** remove it from the liquid injection fitting.

[Q] CLOSE the 10 percent H_2 and CH_4 cylinder valve.

[R] Return the LCS standard to the freezer.

[S] Attach a vented quick-connect fitting to the Autosampler 3 pressurization port to allow for port flushing.

NOTE
The regulator pressure should be set to approximately 40 psi.

[T] OPEN the internal standard cylinder valve.

[U] OPEN the Autosampler 3 software.

[V] Press the FILL Button to enter the canister pressurization control screen, set the flush time to one minute, **AND** press the GO Button.

[W] Attach a vented quick-connect fitting to the FILL port on Autosampler 3.

[X] Press the FLUSH Button on Autosampler 3.

[X.1] Remove the vented quick-connect fitting when purging is completed.

[Y] Attach the LCS standard canister to the pressurization port, **AND** OPEN valve.

[Z] Set Autosampler software to dilute to a relative dilution factor of 2.0 times, **AND** press the GO Button.

[AA] Press the PRESSURIZE Button on Autosampler 3.

[BB] **AFTER** the Autosampler software indicates pressurization is done,
THEN CLOSE the canister valve, **AND** remove from the pressurization port.

[CC] CLOSE the internal standard cylinder valve.

[DD] Save the template using the following format:

[DD.1] PRIMARYID_DATE_LCS.xls spreadsheet

[EE] Print a hardcopy, **AND** print name, sign, and date.

[FF] Place the hard copy in the BDR holding folder.

[GG] Place a tag to label the canister with the following information:

[GG.1]LCS.

[GG.2]Intermediate Standard ID.

[GG.3]Date Prepared.

[GG.4]Preparer's Initials.

[GG.5]Level of Concentration.

4.4 Preparation of the LB

4.4.1 OPEN the UHP nitrogen cylinder valve.

4.4.2 OPEN the Diluter software, **AND** press the FLUSH Button to enter the canister pressurization controls screen.

4.4.3 Set the flush time to one minute, **AND** press the GO Button.

4.4.4 Press the FLUSH Button on the diluter to purge it with UHP nitrogen.

4.4.5 Note the current psia reading in the Diluter software, **AND** type this same value into the TARGET PRESSURE field allowing the Diluter software to dilute to zero psig when purging is complete.

[A] Take a cleaned and evacuated canister, **AND** confirm its evacuation with a pressure gauge.

[B] **IF** evacuated,
THEN attach the canister to the pressurization port.

[C] **IF NOT** evacuated,
THEN select another cleaned and evacuated canister, **AND** attach to the pressurization port.

[D] Set the Autosampler software to dilute to target pressure, **AND** press the GO Button.

[E] OPEN the canister, **AND** press the PRESSURIZE Button on the diluter.

[F] **AFTER** the software shows the canister is pressurized, **THEN** CLOSE the canister valve, **AND** remove it from the liquid injection fitting.

[G] CLOSE the UHP nitrogen cylinder valve.

[H] Attach a vented quick-connect fitting to the Autosampler 3 pressurization port to allow for port flushing.

NOTE

The regulator pressure should be set to approximately 40 psi.

[I] OPEN the internal standard cylinder valve.

[J] OPEN the Autosampler 3 software, **AND** press the FILL Button to enter the canister pressurization controls screen.

[K] Set the flush time to one minute, **AND** press the GO Button.

[L] Press the FLUSH Button on the Autosampler 3 to purge with internal standard.

[M] Remove the vented quick-connect fitting when purging is complete.

[N] Attach the LB canister to the pressurization port, **AND** OPEN valve.

[O] Set the Autosampler software to dilute to a relative dilution factor of 2.0 times, **AND** press the GO Button.

[P] Press the PRESSURIZE Button on Autosampler 3.

[Q] **AFTER** the Autosampler software shows the canister pressurization is done,
THEN CLOSE the canister valve, **AND** remove from the pressurization port.

[R] CLOSE the internal standard cylinder valve.

[S] Place a tag to label the canister with the following information:

[S.1] LB.

[S.2] Date Prepared.

[S.3] Preparer's Initials.

NOTE

Standards used for method performance demonstration must include all target analytes listed in Table 1.

4.5 Preparation of MDLs

4.5.1 Prepare standards used to determine or demonstrate method performance (e.g., MDLs, precision and accuracy) in the same manner as the ICAL1 calibration standard.

4.6 Sample Receiving

NOTE

Step 4.6.1 may be performed at any time during the sample receiving and log-in process, but must be completed prior to performing Section 4.8.

4.6.1 Inspect the custody seal(s) placed over the shipping container closure before opening the shipping container.

[A] Verify that the custody seal(s) meets the following conditions:

[A.1] The seal is intact.

[A.2] The seal is dated and signed/initialed **OR** otherwise traceable to the person applying the seal.

[A.3] The seal is placed such that the container CAN **NOT** be opened without damage to the seal.

[A.4] The seal CAN **NOT** be removed without damage to the seal.

[B] **IF** the seal(s) is missing, **OR** DOES **NOT** meet the conditions above (e.g., damaged, not completed [not signed/initialed and dated or traceable], placed such that the container could be opened without damage to the seal, or can be removed without damage to the seal),
THEN initiate an NCR in accordance with CCP-QP-005.

4.6.2 Break the custody seal, **AND** verify the sampling Chain of Custody (COC) generated from CCP-TP-093, *CCP Sampling of TRU Waste Containers*, is present in the shipping container.

[A] **IF** the samples are **NOT** accompanied by a COC,
THEN contact LO for instructions.

[B] **IF** the samples are accompanied by COC,
THEN remove COC form from the shipping container, sign the Received By section, **AND** record the Date/Time of the transfer (date and time that container custody seal was broken).

[C] Document any problems identified with the container custody seal (step 4.6.1) on COC.

[D] Inspect the custody transfers documented on the COC for completeness, consistency and continuity.

[D.1] Verify that a final relinquishing signature is present on the COC.

[D.2] Verify that transfers of custody are documented in chronological order.

(a) **IF** discrepancies in the document custody transfers are identified,
THEN initiate an NCR in accordance with CCP-QP-005.

[D.3] Remove the canisters from the shipping container.

4.6.3 Inspect the COC and the canisters.

[A] Verify that all canisters present in the shipping container are identified on the COC.

[B] Verify that all canisters identified on the COC are present in the shipping container.

[C] Verify that each canister has an accompanying gas sample tag.

[D] Verify that each canister has a custody seal on the valve cap meeting the following criteria:

[D.1] The seal is intact.

[D.2] The seal is dated and signed/initialed **OR** otherwise traceable to the person applying the seal.

[D.3] The seal is placed such that the valve cap CAN **NOT** be opened without damage to the seal.

[D.4] The seal CAN **NOT** be removed without damage to the seal.

[E] Inspect each canister for physical signs of damage (i.e., dents, broken valves).

[F] Verify that all canister valves are in the CLOSED position.

[G] Verify that the following minimum information is recorded on the COC:

[G.1] Signature of individual initiating custody along with date and time.

[G.2] Field sample Identification(s).

[G.3] Signature of persons relinquishing and accepting custody with date and time of transfer.

[H] Document any problems found with the canisters in the preceding steps on the COC.

[I] Inspect the Attachment 3, Gas Sample Canister Tag, from CCP-TP-142, *Preparation of Canisters for Headspace Gas Sampling* accompanying the canisters.

[J] Verify that all field sample numbers on the SUMMA® gas sample canister tags correspond to those listed on The COC.

[K] Verify that the following minimum information is recorded on the gas sample canister tags.

[K.1] Correct analytical requests.

[K.2] Sample description.

[K.3] Field Sample ID.

[K.4] Sampler initials and organization.

[K.5] Ambient pressure and temperature.

[K.6] Date and time of sample collection.

[K.7] QC designation.

[L] Verify that information on the gas sample canister tags matches that on The COC.

[M] Verify that the canister ID (XXXXX) on the gas sample canister tags **OR** the COC matches the canister ID found on the metallic silver label on the bar code attached to the canister.

[N] Document any problems found in the proceeding steps on the COC.

NOTE
Gas sample canister tags remain with the canisters until sample analysis is complete.

4.6.4 Complete the following fields in the Analytical Laboratory portion of the gas sample canister tags:

[A] Canister Gauge Pressure.

[B] Units (psi if pressurized, inches Hg if below ambient, either units acceptable if a gauge zero).

[C] Ambient Temperature.

[D] Ambient Pressure.

[E] Date.

[F] Time.

4.6.5 Notify the LO of any discrepancies found in the documentation **OR** any issues with sample integrity (e.g., broken custody seals) identified during the receiving process.

NOTE
The Sample Receiving Logbook SHALL be a bound, paginated book. At a minimum, fields to be recorded SHALL include Receipt Date, HSG Operator's Signature, Receiving Group Number, Field Batch Number, and Analytical Batch Number in accordance with CCP-QP-011, *CCP Notebooks and Logbooks*.

[A] Resolve the discrepancies with sampling organization, **AND** document the resolution on the Sample Receiving Logbook.

[B] Initiate an NCR in accordance with CCP-QP-005, if necessary.

[C] **IF** problems identified during receipt necessitate the return of the sample(s) to the sampling organization,
THEN perform the following:

[C.1] Replace the sample(s) in the shipping container.

[C.2] Sign the COC as the relinquisher.

[C.3] Place the COC and gas sample canister tag(s) inside the shipping container.

[C.4] CLOSE the container, **AND** apply custody seals across the container closure such that they meet the criteria for step 4.6.1[A].

[C.5] Contact the sampling organization to arrange transport of the sample(s) back to the sampling location.

[C.6] Record the return of the samples in the Sample Receiving Logbook.

4.7 Sample Log-In

NOTE
All samples received at the lab are logged in by a HSG Operator prior to analysis.

During the log-in process, laboratory sample numbers are assigned and generated to track the samples through the laboratory.

NOTE
The Receiving Number is an 8-charcter alphanumeric code in the format CEYYXXX where YY is the last 2 digits of the calendar year and XXX is a sequential number that starts with 001 at the beginning of each calendar year.

4.7.1 Assign a Receiving Group Number to the samples.

[A] Determine the next available Receiving Number from the last entry in the Sample Receiving Logbook.

[B] Record the Receiving Group Number of the samples in the Sample Receiving Logbook.

NOTE
Lab Sample ID is the same as the Drum Number found on the Gas Sample Canister Tag.

4.7.2 Assign lab sample ID to each sample.

4.7.3 Place each lab sample ID label on a paper tag (approximately 31/4" X 11/2"), **AND** attach it to the fastener on the appropriate sample canister.

[A] Verify that the label is being placed on the appropriate canister by comparing the canister ID (sliver metallic tape label or bar code) to the canister ID portion of the field sample ID shown on the label.

4.7.4 Sign and date the Sample Receiving Logbook after verifying that all fields are complete.

4.7.5 Place all sample canisters in secured storage area.

NOTE
The temperature device must have a current calibration sticker.

[A] **IF** the storage temperature exceeds 0 to 40 °C, **THEN** contact the LO for instructions.

4.8 Analytical Run Sequence for Sample Analysis

4.8.1 ICAL or MDL Sequence (to be run as needed)

[A] BFB Tune Check.

[B] Calibration Standards.

4.8.2 Daily Analytical Run Sequence

NOTE
The LCS and Sample Duplicate can be analyzed anytime after the LB.

NOTE
If samples are analyzed immediately following an ICAL, the level 3 ICAL standard SHALL be used as the daily CCAL for comparing ISAs and RTs.

[A] BFB Tune Check.

[B] CCAL.

[C] LB.

[D] LCS.

[E] Samples.

[F] Laboratory Duplicate.

4.9 BFB Tune Check

4.9.1 Analyze the BFB standard at the beginning of each 12 hours of operations **OR** prior to a calibration.

4.9.2 Check the key ion abundance against the criteria in Table 2.

[A] **IF** the key ion abundance criteria in Table 2 are met, **THEN** print out the BFB Evaluation Report (see Attachment 14, BFB Tune Form for an example).

[B] Print name, sign, and date the BFB Tune Form.

[C] Place BFB Tune Form in the BDR holding folder.

[D] **IF** the key ion abundance criteria in Table 2 are **NOT** met, **THEN** correct the problem, **AND** repeat the BFB Tune Check until it is acceptable.

4.10 ICAL

4.10.1 Verify that the BFB Tune Check meets the criteria in Table 3, Independent Technical Reviewer Checklist before calibrating.

4.10.2 Ensure that the pressure in each ICAL standard canister is above 4 psig, **AND** load the 5 ICAL canisters onto Autosampler(s).

4.10.3 Enter the calibration standards into the GC/MS, **AND** Autosampler sequence tables.

4.10.4 Verify that the Autosampler positions specified in the sequence tables are the ones being used.

4.10.5 Choose the first calibration standard, **AND** begin the sequence in both the GCMS and Autosampler software.

4.10.6 After the analysis of all initial calibration standards is complete, OPEN the ENVIRONMENTAL DATA ANALYSIS window.

4.10.7 Load the data analysis method which includes the most recent calibration responses (filename “mmddyy”).

4.10.8 Clear all the calibration responses.

4.10.9 Type ENHANCED 2 in the EXECUTE line, **AND** hit EXECUTE.

4.10.10 GO TO CALIBRATION on the toolbar of the enhanced data analysis screen, **AND** select EDIT COMPOUNDS.

NOTE

The following will bring up the page 1 of the chosen compound. For H_2 and CH_4 only, make sure that the Quant Signal in the box SIGNALS TO BE USED FOR QUANTITATION is BC SIGNAL 1. For the VOC compounds, the Quant Signal must be TARGET ION.

4.10.11 **WHEN** the EDIT COMPOUNDS screen comes up, **THEN** select the first compound, **AND** click on VIEW.

NOTE

The following will bring up the Calibration Level ID and Concentration screen.

4.10.12 Select PAGE 3.

4.10.13 Fill out 0.075%, 1%, 10% and 20% for H_2 and CH_4, in the CONC. column, **AND** for CALIBRATION LEVEL ID 1, 2, 3, 4, and 5, respectively.

4.10.14 Fill out the concentrations for the VOC compounds according to the actual concentrations calculated during the preparation of the standard.

4.10.15 Enter the certificate concentration for the internal standards in the appropriate fields on page 1 and page 3 of the quantitation database.

4.10.16 Save the changes to each compound.

4.10.17 Save the method as MMDDYY.m (e.g., “031804.m” is the method saved on March 18th 2004).

4.10.18 Exit the ENHANCED DATA ANALYSIS screen by typing ENHANCED in the Execute line and selecting EXECUTE.

4.10.19 Load the method just created.

4.10.20 OPEN the first calibration data file (ICAL 1).

4.10.21 Calculate and generate the Quantitation Report (see Attachment 16, Quantitation Report for an example).

4.10.22 Enter Qedit mode, **AND** check to be certain that each peak and its qualifier ions were correctly integrated.

4.10.23 Exit Qedit, saving changes to the quantitation.

4.10.24 Update the initial calibration for the first level.

4.10.25 Repeat steps 4.10.20 through 4.10.24 for each calibration level.

4.10.26 **AFTER** calibration update has been performed for all calibration levels,
THEN display the calculated RRFs for the calibration.

4.10.27 **IF** the %RSD for all the VOC target analytes listed is less than 35 percent,
THEN proceed with analysis.

4.10.28 **IF** the %RSD is greater than or equal to 35 percent for any target analytes,
THEN correct the problem, **AND** repeat the ICAL.

4.10.29 **IF** the r^2 value for H_2 and CH_4 is greater than or equal to 0.990,
THEN save the method, **AND** proceed to sample analyses.

4.10.30 **IF** the r^2 value for H_2 and CH_4 is less than 0.990,
THEN correct the problem, **AND** repeat ICAL.

4.10.31 Calculate the mean RT of both H_2 and CH_4, including the RTs from ICAL1 through ICAL4, if necessary.

4.10.32 Calculate the acceptable H_2 and CH_4 RT windows by using the mean RT from the ICAL plus or minus five percent of the mean RT, if necessary.

[A] Assemble ICAL Report with the following information:

[A.1] ICAL Response Factor Report

[A.2] BFB Evaluation Report

[A.3] H_2 and CH_4 r^2 Calibration Curve.

[A.4] Standard Preparation Worksheet

[A.5] Raw Data

[B] Submit the ICAL Report to the ITR.

ITR

[C] Review ICAL Report, **AND** ensure the following:

[C.1] ICAL standards were updated correctly into VOCGAS Method.

[C.2] All of target analytes meet the less than 35 percent RSD.

[C.3] The r^2 for H_2 and CH_4 is greater than 0.990.

[D] Notify the HSG Operator of all errors or omissions found during the review.

[E] Recheck the data after the errors or omissions have been rectified.

[F] Print name, sign, and date top sheet of the ICAL Response Factor Report to document the ITR review.

[G] Paginate the ICAL Report.

[H] Make a copy of the ICAL Response Factor Report for the BDR, **AND** place in the BDR holding file.

[I] Forward the ICAL Report to Facility Records Custodian.

Facility Records Custodian

[J] Receive, process, and transmit the ICAL Report in accordance with CCP-QP-008.

4.11 CCAL

4.11.1 Ensure that the BFB Tune Check meets the criteria in Table 2 before analysis of the CCAL.

4.11.2 Load the CCAL canister onto an Autosampler.

4.11.3 Enter the sample information and data file information into the sequence log of Chemstation® software.

4.11.4 Enter the sample information into the Autosampler sequence log.

4.11.5 Select the CCAL in both sequence tables, **AND** start the sequence in both the Chemstation® and Autosampler software.

4.11.6 **AFTER** the completion of the run,
THEN double click on the ENVIRONMENTAL DATA ANALYSIS icon on the Desktop to OPEN the ENVIRONMENT DATA ANALYSIS software.

4.11.7 Select FILE on the toolbar and click LOAD METHOD.

4.11.8 Select mmddyy.m (the latest calibration date) when the LOAD METHOD screen appears to open the current method, **AND** click OK.

4.11.9 Select CALCULATE AND GENERATE REPORT under the QUANTITATE pulldown menu.

4.11.10 Enter Qedit mode, **AND** check to be certain that each peak and its qualifier ions were correctly integrated.

4.11.11 EXIT Qedit saving changes to the quantitation.

4.11.12 Select EVALUATE FILE AS CONTINUING CALIBRATION TO PRINTER from the ConCal pulldown menu in the ChemStation® software.

[A] Review the report produced by this action.

4.11.13 Proceed with sample analysis if the following criteria are met:

[A] Percent D less than or equal to 30 for each target analyte

[B] RTs are within plus or minus 0.5 minutes from the average ICAL RT

[C] The internal standard areas are within 50 percent to 200 percent of ICAL3 internal standard responses.

4.11.14 **IF** the CCAL DOES **NOT** meet the criteria, **THEN** reanalyze.

4.12 LB

4.12.1 Add the LB to both the Chemstation® and Autosampler software sequence tables.

4.12.2 Attach the LB canister to the appropriate Autosampler position, **AND** OPEN its valve.

4.12.3 Select the LB in both sequence tables, **AND** begin the run.

4.12.4 Load the LB data file in the ENVIRONMENTAL DATA ANALYSIS screen following the run.

4.12.5 CALCULATE AND GENERATE the Quantitation Report for the LB data file.

4.12.6 Enter the Qedit mode, **AND** ensure that each peak and its qualifier ions were correctly integrated.

4.12.7 EXIT Qedit, saving changes to the quantitation.

4.12.8 Review the Quantitation Report for the LB.

4.12.9 Proceed if the following criteria are met:

[A] Internal standard responses are within 50 percent to 200 percent of the CCAL internal standard responses.

[B] RTs of all VOC target compounds are within plus or minus 0.5 minutes from the CCAL RTs.

[C] RTs of H_2 and CH_4 are within the CCAL RT window.

[D] Concentrations of all target analytes are less than or equal to 3 times the currently established MDLs.

4.12.10 **IF** the criteria are **NOT** met,
THEN initiate an NCR in accordance with CCP-QP-005, **AND** assign the appropriate qualifier flag to the data.

4.13 LCS

4.13.1 Add the LCS to both the Chemstation® and Autosampler software sequence tables.

4.13.2 Attach the LCS canister to the appropriate Autosampler position, **AND** OPEN its valve.

4.13.3 Select the LCS in both sequence tables, **AND** begin the run.

4.13.4 Load the LCS data file in the ENVIRONMENTAL DATA ANALYSIS screen following the run.

4.13.5 CALCULATE AND GENERATE the Quantitation Report for the LCS data file.

4.13.6 Enter the Qedit mode, **AND** ensure that each peak and its qualifier ions were correctly integrated.

4.13.7 Save changes to the quantitation, **AND** EXIT Qedit.

4.13.8 Review the Quantitation Report for the LCS.

4.13.9 Calculate the %R of all target analytes in the LCS using Equation 6 from Attachment 1.

[A] **IF** the %R for all analytes in the LCS are between 70 percent and 130 percent,
THEN proceed with sample analysis.

[B] **IF** the %R for all analytes in the LCS are **NOT** between 70 percent and 130 percent,
THEN reanalyze, **OR** prepare a new LCS and reanalyze, **OR** recalibrate as needed.

[B.1] **IF** reanalysis DOES **NOT** meet the criteria,
THEN notify the LO.

4.14 MDL Determination

4.14.1 Analyze replicates of the MDL samples in accordance with step 4.16.3.

4.14.2 Complete the spreadsheet template HSG_MDL.xls spreadsheet, **AND** SAVE the completed template as MDL-MMDDYY.xls spreadsheet.

[A] Print a hardcopy, print name, sign, and date.

[B] Assemble MDL Report with the following information:

[B.1] MDL –mmddyy (see Attachment 12, MDL Reporting Form for an example).

[B.2] BFB Tune Form

[B.3] Raw Data

[C] Submit the MDL Report to the ITR.

ITR

[D] Review MDL Report.

[D.1] Ensure the concentrations from the MDL analyses have been correctly entered into Flammable Gas Analysis MDL Excel Spreadsheet.

[E] Notify the HSG Operator of all errors or omissions found during the review.

[F] Recheck the data after the errors or omissions have been rectified.

[G] Print name, sign, and date the MDL-mmddyy Spreadsheet to document the review.

[H] Paginate the MDL Report.

[I] Submit the MDL Report to the Facility Records Custodian.

Facility Records Custodian

[J] Receive, process, and transmit the MDL Report in accordance with CCP-QP-008.

4.15 PDP Samples

4.15.1 Prepare PDP Samples prior to analysis by transferring an aliquot of the sample from the 6L SUMMA® canister supplied to a cleaned and evacuated 380 to 1000mL canister.

4.15.2 Ensure that the pressure in the 380 to 1000mL canister is approximately 0 psig, **AND** proceed with addition of the internal standard and analysis according to Section 4.16.

4.15.3 Submit the PDP results in accordance with CCP-TP-056, *CCP HSG Performance Demonstration Plan*.

4.16 Analyzing Samples

4.16.1 Sample Preparation

NOTE

Prior to analysis, all samples will be pressurized to twice their original pressure with internal standard gas mixture. Samples with analytes whose concentrations exceed the calibration curve will be diluted and reanalyzed. Sample dilutions will be recorded in the Attachment 11, Headspace Gas Sample Preparation Form. Samples may be diluted with internal standards or diluted with H_2 and CH_4 standards (only if H_2 and CH_4 analysis was completed from the undiluted sample), or ultra high purity nitrogen. Care must be taken during sample dilutions to ensure that changes in the proportional quantity of internal standards to sample are considered in determining the effective dilution factor.

[A] Add the internal standards to sample canisters by attaching the canister to the pressurization port on Autosampler 3.

[B] Set the Autosampler 3 software to 2 times the pressure reading on individual canisters, **AND** click GO on the Autosampler 3 software.

[C] OPEN the canister, **AND** press the PRESSURIZATION Button on Autosampler 3.

[D] **WHEN** the pressurization with internal standard is completed,
THEN remove it from the pressurization port.

[E] Record following information on Attachment 11:

[E.1] Client Sample ID (Drum Number)

[E.2] Canister ID

[E.3] Original Pressure (before addition of internal standard)

[E.4] Final Pressure (after addition of internal standard)

[E.5] Dilution Factor (Current Dilution)

[E.6] Dilution Number (“0”, if addition of internal standard only.

[E.7] Pressurization Gas

[E.8] Internal Standard Mix ID

[E.9] Current Total Dilution Factor (“1”, if addition of internal standard only)

[F] Repeat this for all the samples from the batch.

4.16.2 Sample Dilution

[A] Sample dilutions will be recorded in the Attachment 11 using one complete line for each step of dilution. There are three dilution methods that may be used:

[A.1] Dilution with 2X Diluted Internal Standard:

A large (6 or 15 L) clean, evacuated canister is pressurized to 20 ± 5 psia with Internal Standard Gas, then diluted 2X using UHP nitrogen. This canister is then attached to the gas supply line on Autosampler 3.

After analysis, the pressurized sample is vented to atmospheric pressure. Next, the sample is pressurized to twice atmospheric pressure with the 2X diluted Internal Standard Gas, using the pressurization port on Autosampler 3. This step will result in a 2X dilution of the sample because the ratio of sample to internal standard mix is maintained.

A series of dilutions may be conducted if larger dilution factors are required, so long as the 2X dilution of Internal Standard Gas is used for all dilutions on a given sample. The final dilution factor is simply the product of all dilutions performed. (e.g., if three 2X serial dilutions are performed, the final dilution factor would be 2*2*2=8X.)”

[A.2] Dilution with Internal Standard Gas:

After analysis, the pressurized sample is vented to the atmosphere, so that part of the sample and a proportional amount of internal standard remain in the canister.

Next, the canister is pressurized with nitrogen containing internal standards, using the pressurization port on the Autosampler. This is set up for 2 times pressurization (pressure in the canister will be increased two times in absolute pressure readings). After the pressurization the canister will contain a fraction of the original sample and an increased amount of IS. Because 2 times pressurization was used for the original pressurization and for the second pressurization, the dilution factor will be 3. See the NOTE below for a detailed explanation.

NOTE

Dilution factor calculations (all pressures in discussion below) are in absolute units.

Assume a sample is collected to the pressure "x" in absolute pressure units.

Next the sample is pressurized to 2 times the initial pressure (with N_2 + IS mixture), so the final pressure will be "2x".

After analysis, the still-pressurized sample is vented to the atmosphere, so that the final pressure in the canister will be "y"

And again, the remaining gas (containing part sample and part ISs) in the canister is pressurized with the same N_2 + IS mixture to 2 times the initial pressure (y). Final pressure will be "2y".

Based on the pressures, after venting the canister will contain:

"y/2x" fraction of the sample, and

"y/2x" fraction of the IS

The second pressurization adds more IS to the canister. The amount of IS added to the canister is proportional to the pressure increase (e.g., "y"). Relatively (to the initial amount of sample – "x"), the amount of IS increased by "y/x".

After venting and the second pressurization, the canister will contain:

"y/2x" part of the initial sample, and

"y/2x+y/x=3/2*y/x" parts (relative to initial sample amount) of IS.

Because we are changing the ratio of IS to the sample, the dilution factor will be:

$$\frac{1}{\text{fraction of the sample left in diluted sample}} \qquad (10)$$

Using our pressure symbols:

$$dilution\ \ factor = \frac{1}{y/2x} * \frac{3y}{2x} = 3$$

This dilution factor is for volatile compounds only. Because the internal standard is not used to quantitate H_2 and CH_4 concentrations, the resulting dilution factor for H_2 and CH_4 will be two.

[A.3] Dilution with UHP Nitrogen:

The original sample is vented to atmospheric pressure (0 psig) so that half of the sample and half of ISs remain in the canister.

Next, the canister is pressurized with UHP Nitrogen using the pressurization port on Diluter set up for 2x pressurization. After pressurization, the canister will contain 50 percent of the original sample and 50 percent of the ISs (if compared to the undiluted sample). The dilution factor will be 1 (in terms of response factor there is no dilution). A decreased amount of internal standards in the diluted sample should be taken into account when internal standard area counts are compared. See NOTE under Section 4.16.2[A][A.1] for a detailed explanation.

4.16.3 Sample Analysis

[A] Load sample canisters onto the Autosampler(s).

[B] Enter the sample information and data file information into the sequence log of Chemstation® software, **AND** include a sample duplicate in the sequence (one for each batch of 20 or less).

[C] Enter the sample information and data file information for all samples in the batch into the sequence log of Autosampler software, **AND** save both sequence logs.

[D] Select the first sample in both sequence logs, **AND** START the sequences.

[E] **IF** any analyte is detected during the initial analysis of a sample in an amount exceeding the initial calibration range, **THEN** dilute, **AND** reanalyze until the analytes in question fall within the calibration range.

[E.1] **IF** an analyte is **NOT** detected in the diluted sample but was detected in the initial analysis, **THEN** report the initial analysis results and the diluted results.

[E.2] Report the diluted analyte with a D flag.

4.16.4 Duplicates

[A] The duplicate sample will be run according to steps 4.16.3[A] through 4.16.3[E.2].

[B] Determine the RPD for all analytes present in the sample/sample duplicate at concentrations greater than or equal to the respective PRQLs using HSGDP.xls spreadsheet.

[C] Verify the RPD is less than or equal to 25 percent for all target analytes present in the samples at concentrations greater than or equal to PRQL.

[D] **IF** there are **NO** VOC target analytes above the PRQL detected in the field canisters,
THEN analyze a second LCS to be used as the duplicate.

4.17 Data Evaluation

4.17.1 Target Analyte Analysis

NOTE

For VOCs, standard reference mass spectra are obtained through analysis of calibration standards or spectra from the NIST Mass Spectral Library. Two criteria must be satisfied to verify identification.

- The sample component RRT must compare within plus or minus 0.06 RRT units of the RRT of the standard component in the CCAL.
- The sample component and the standard component mass spectrum must correspond.

[A] Identify target analytes by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference mass spectrum).

[B] Identify H_2 and CH_4 by comparison of the sample RTs to the established RT windows (see steps 4.10.31 through 4.10.32).

4.18 TICs

4.18.1 Qualitative Identification

[A] Perform a search against a NIST Mass Spectral Library for all unknown (non-target) compounds having a total ion area greater than 10% of the nearest internal standard.

NOTE

At a minimum the NIST library must contain all available spectra for VOC compounds that appear in the 20.4.1.200 NMAC (incorporating 40 CFR 261) Appendix VIII list.

[B] Evaluate the unknown against the results of the library search for tentative identification according to the following guidelines:

- Relative intensities of major ions in the reference spectrum (ions greater than 10 percent or most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within plus or minus 20 percent.
- Molecular ion that is present in the reference spectrum should be present in the sample spectrum.
- Ions that are present in the sample spectrum but **NOT** present in the reference spectrum should be reviewed for possible background contamination **OR** presence of co-eluting compounds.
- Ions present in the reference spectrum but **NOT** in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination **OR** co-eluting peaks.

[C] Report all flammable TICs (excluding the VOCs that have been evaluated for shipment in the TRUPACT-II and HalfPACT shipping packages as listed in the current revision of DOE/WIPP 01-3194, *CH TRU Waste Content Codes*, Appendix B) that exceed a concentration of 500 ppmv as an NCR in accordance with CCP-QP-005.

[D] Report TICs which meet the above criteria per step 4.19.5[A.1].

4.19 Data Generation

4.19.1 From the Chemstation® software, generate the following:

[A] BFB Tune Form

[B] ICAL Response Factor Summary

[C] H_2 and CH_4 Plot Curve

[D] Continuing Calibration Evaluation Report (see Attachment 15, Continuing Calibration Evaluation Report for an example)

[E] Quantitation Reports (for QC samples and samples)

4.19.2 From the HSGDP.xls spreadsheet, generate the following:

[A] Laboratory Control Sample Data Sheet

[B] Sample Duplicate Precision Data Sheet

[C] Blank Summary

[D] Headspace Gas Canister Internal Standard Area and RT Summary

[E] Headspace Gas Sample Analysis Results Summary (see Attachment 7, Headspace Gas Sample Analysis Results Summary for an example)

4.19.3 From the HSG_MDL_Calc.xls spreadsheet, generate the following:

[A] MDL Report

4.19.4 Fill out the Attachment 17, Headspace Gas Batch Data Report Cover Page, to include the following information:

[A] Analytical Batch Number.

[B] Batch Date.

[C] Procedure Number/Revision Number.

[D] Sample/Container Numbers.

[E] Print name, sign, and date.

4.19.5 Data Review

[A] Review, initial, and date all raw data generated.

[A.1] **IF** a non-target compound (TIC) is to be reported **THEN** record YES on the TIC Raw Data.

[A.2] **IF** a TIC is **NOT** to be reported **THEN** record NO on the TIC Raw Data.

[B] Print name, sign, and date all forms generated.

[C] Complete Attachment 10, HSG Operator Checklist, for each analytical run sequence.

4.20 Data Reporting

4.20.1 Report sample results in units of ppmv and vol% to minimum of two significant figures.

4.20.2 Generate a Case Narrative (see Attachment 19, Case Narrative for an example) which includes the following:

[A] Laboratory Name.

[B] BDR Number.

[C] Procedure Number and Revision Number.

[D] Analysis Date.

[E] Discussion of QC Samples outside the control limits and the associated NCRs.

[F] Print name, sign, and date the Case Narrative.

4.20.3 Use rounding rules and assign data qualifying flags per the requirements of Attachment 2, Data Reporting Guidance.

NOTE
When assembling the Batch Raw Date Report Cover Page and Table Contents, any items that are not applicable can be recorded with N/A.

4.20.4 Assemble the Batch Raw Data Report as follows:

[A] Headspace Gas Batch Data Report Cover Page (see Attachment 17, Headspace Gas Batch Data Report Cover Page for an example).

[B] Headspace Gas Batch Data Report Table of Contents (see Attachment 18, Headspace Gas Batch Data Report Table of Contents for an example).

[C] Raw Data

4.20.5 Submit the Batch Raw Data Report to the Facility Records Custodian.

Facility Records Custodian

4.20.6 Receive, process, and transmit the MDL Report in accordance with CCP-QP-008.

4.20.7 Assemble a Batch Data Report (BDR) as follows:

[A] Attachment 17

[B] Attachment 18

[C] Analysis Request Form

[D] Case Narrative

[E] Sample Custody Documents and Sample Tag

[E.1] Copy of the COC generated in CCP-TP-093

[E.2] Gas Sample Canister Tag

[F] Analysis Results

[F.1] Headspace Gas Sample Analysis Results Summary

[F.2] Attachment 11

[G] Quality Control Measurements Results

[G.1] BFB Tune Form (see Attachment 14, BFB Tune Form for an example)

[G.2] LCS Duplicate Precision Data Sheet (see Attachment 5, LCS Duplicate Precision Data Sheet for an example)

[G.3] Sample Duplicate Precision Data Sheet (see Attachment 6, Sample Duplicate Precision Data Sheet for an example)

[G.4] Blank Summary (see Attachment 8, Blank Summary for an example)

[G.5] Laboratory Blank Results Form

[G.6] Headspace Gas Canister Internal Standard Area and RT Summary (see Attachment 9, Headspace Gas Canister Internal Standard Area and RT Summary for an example)

[G.7] Laboratory Control Sample Data Sheet (see Attachment 4, Laboratory Control Sample Data Sheet for an example)

[H] Calibration Results

[H.1] BFB Tune Form

[H.2] Initial Calibration Form

[H.3] Continuing Calibration Verification Form

[H.4] MDL Reporting Form (Attachment 12, MDL Reporting Form for an example)

[I] Data Review Checklists

[I.1] Attachment 3

[I.2] Attachment 10

[J] Copy of Temperature Log

[K] Copy of NCRs, if applicable

4.20.8 Submit the BDR to the ITR.

ITR

4.20.9 Review BDR according to Attachment 3.

4.20.10 Notify the HSG Operator of all errors or omissions found during the review, if applicable.

4.20.11 Recheck the data after the errors or omissions have been rectified, if applicable.

4.20.12 Document the independent technical review by printing name, signing, and dating Attachment 3.

4.20.13 Submit the BDR to the Facility Record Custodian.

Facility Records Custodian

4.20.14 Paginate the BDR, **AND** fill out the Attachment 18.

4.20.15 Paginate the Batch Raw Data Report, **AND** fill out Attachment 18.

4.20.16 Receives, processes, and transmits all records generated by this procedure in accordance with CCP-QP-008.

5.0 RECORDS

5.1 The following documents are maintained in accordance with CCP-QP-008.

5.1.1 QA/Lifetime

[A] Batch Data Report

5.1.2 QA/Non Permanent

[A] Certification of Standard

[B] MDL Report

[B.1] MDL-mmddyy Spreadsheet

[B.2] BFB Tune Report

[B.3] MDL Raw Data

[C] ICAL

[C.1] ICAL Response Factor Report

[C.2] BFB Evaluation Report

[C.3] H_2 and CH_4 Calibration Curve

[C.4] Standard Preparation Worksheet

[C.5] ICAL Raw Data

[D] Attachment 11, Headspace Gas Sample Preparation Form

Table 1. Gas Target Analyte List and Quality Assurance Objectives

Compound	CAS No.	Precision[a] (%RSD or RPD) Less than or equal to value below	Accuracy[a] (%R)	MDL[b] (ng)	PRQL (ppmv)	Completeness
Benzene	71-43-2	25	70-130	10	10	90%
Bromoform	75-25-2	25	70-130	10	10	90%
Carbon Disulfide	75-15-0	25	70-130	10	10	90%
Carbon tetrachloride	56-23-5	25	70-130	10	10	90%
Chlorobenzene	108-90-7	25	70-130	10	10	90%
Chloroform	67-66-3	25	70-130	10	10	90%
Cyclohexane[c]	110-82-7	25	70-130	10	10	90%
Chloromethane	74-87-3	25	70-130	10	10	90%
1,1-Dichloroethane	75-34-3	25	70-130	10	10	90%
1,2-Dichloroethane	107-06-2	25	70-130	10	10	90%
1,1-Dichloroethylene	75-35-4	25	70-130	10	10	90%
cis-1,2-Dichloroethyene	156-59-2	25	70-130	10	10	90%
trans-1,2-Dichloroethyene	156-60-5	25	70-130	10	10	90%
1,2 Dichloropropane	78-87-5	25	70-130	10	10	90%
Ethyl benzene	100-41-4	25	70-130	10	10	90%
Ethyl ether	60-29-7	25	70-130	10	10	90%
Methylene chloride	75-09-2	25	70-130	10	10	90%
1,1,2,2-Tetrachloroethane	79-34-5	25	70-130	10	10	90%
Tetrachloroethene	127-18-4	25	70-130	10	10	90%
Toluene	108-88-3	25	70-130	10	10	90%
1,1,1-Trichloroethane	71-55-6	25	70-130	10	10	90%
Trichloroethene	79-01-6	25	70-130	10	10	90%
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	25	70-130	10	10	90%
1,2,4-Trimethylbenzene[c]	95-63-6	25	70-130	10	10	90%
1,3,5-Trimethylbenzene[c]	108-67-8	25	70-130	10	10	90%
m-Xylene[c]	108-38-3	25	70-130	10	10	90%
o-Xylene	95-47-6	25	70-130	10	10	90%
p-Xylene[c]	106-42-3	25	70-130	10	10	90%
Acetone	67-64-1	25	70-130	150	100	90%
1-Butanol	71-36-3	25	70-130	150	100	90%
Methanol	67-56-1	25	70-130	150	100	90%
2-Butanone	78-93-3	25	70-130	150	100	90%
4-Methyl-2-pentanone	108-10-1	25	70-130	150	100	90%
Hydrogen	1333-74-0	25	70-130	0.05vol%	0.1 vol%	90%
Methane	74-82-8	25	70-130	0.05vol%	0.1 vol%	90%

[a]Criteria apply to PRQL
[b]Values based on delivering10 mL to the analytical system
[c]These isomers cannot be resolved by this analytical method

NOTES:
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MDL = Method detection limit
PRQL = Program required quantitation limit

Table 2. 4-Bromofluorobenzene (BFB) Key Ions and Abundance Criteria

Mass (amu)	**Ion Abundance Criteria** (as determined by peak areas)
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	Base peak, 100% relative abundance
96	5% to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5% to 9% of mass 174
176	Greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 3. Characteristic Ions for VOCs

Volatile Organic Compound	**Primary Ion** (amu)	**Secondary Ion(s)** (amu)
Acetone	58	43
Benzene	78	52,71
Bromoform	171	173,175,252
Butanol	56	31,55
Carbon Disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114,77
Chloroform	83	85,47
Cyclohexane	56	84,41
Chloromethane	50	52
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	64,98
1,1-Dichloroethylene	96	61,98
cis-1,2-Dichloroethylene	96	61,98
trans-1,2-Dichloroethylene	96	61,98
1,2 Dichloropropane	63	62
Ethyl benzene	106	91
Ethyl ether	31	59
Methanol	31	-
Methyl ethyl ketone	72	43,57
Methyl isobutyl ketone	100	57,58,43,85
Methylene chloride	84	49,51,86
1,1,2,2-Tetrachlorothane	85	85,131,133,83
Tetrachloroethylene	166	129,131
Toluene	92	91,65
1,1,1-Trichloroethane	97	99, 61
Trichloroethylene	130	95,97,132
1,1,2-Trichloro-1,2,2-trifluoroethane	101	155,85,151
1,2,4-Trimethylbenzene	120	105
1,3,5-Trimethylbenzene	120	105
m-Xylene	106	91
o-Xylene	106	91
p-Xylene	106	91
Chlorobenzene-d5 (IS)	117	82,119
Fluorobenzene (IS)	96	77,70,50

(IS) = Internal Standard

Table 4. Data Flags

Data Flag	Indicator
B	Analyte detected in blank
D	Analyte was quantitated from a secondary dilution, or reduced sample aliquot
E	Analyte exceeds calibration curve
J	Analyte less than PRQL but greater than or equal to the MDL
U	Analyte was not detected and value is reported as the MDL
Z	One or more QC samples do not meet acceptance criteria

Attachment 1 – Calibration and Quality Control (QC) Equations

NOTE

The minimum number of significant figures required is 2.

1. Determining Relative Response Factor (RRF)

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

Where:

A_x = area of the characteristic ion for the compound being measured.
A_{is} = area of the characteristic ion for the specific internal standard.
C_{is} = amount of the specific internal standard (ng).
C_x = amount of the compound being measured (ng).

2. Determining Percent Relative Standard Deviation (%RSD)

$$\%\,RSD = \frac{s}{x} * 100$$

Where:

s = standard deviation of RRF for a compound.
x = mean of five initial RRFs for a compound (average RRF).

3. Determining Standard Deviation

$$s = \sqrt{\sum_{i=1}^{n} \frac{\left(y_i - \bar{y}\right)^2}{n-1}}$$

Where:

y_i = measured value of the i^{th} replicated sample analysis measurement.
n = number of replicate analyses.

$\bar{y}$ = average measured value.

4. Determining percent difference (%D)

$$\%\,D = \frac{RRF_i - RRF_c}{RRF_i} * 100$$

Where:

RRF_i =average RRF or average RRF from initial calibration (area/ng).
RRF_c =RRF or RRF from current midpoint standard (area/ng).

Attachment 1 – Calibration and Quality Control (QC) Equations (Continued)

5. Determining Method Detection Level (MDL)

$$MDL = t_{(n-1,1-\alpha=.99)} * s$$

Where:

t = t-distribution value appropriate to a 99 percent confidence level.
n -1 = degrees of freedom.
n = number of observations.
s = standard deviation of replicate measurements.

6. Determining Percent Recovery (%R)

$$\%R = \frac{C_m}{C_{srm}} * 100$$

Where:

C_m = measured concentration value obtained by analyzing the sample.
C_{srm} = "true" or certified concentration of the analyte in the sample.

7. Determining Relative Percent Difference (RPD)

$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} * 100$$

Where:

C_1, C_2 = Two values obtained by analyzing the duplicate samples, C_1 is the larger of the two observed values.

Attachment 1 – Calibration and Quality Control (QC) Equations (Continued)

8. Determining Concentration (C_{VOC})

$$C\,(VOC) = \frac{As\ x\ Cis\ x\ V\ x\ D}{Ais\ x\ RRF}$$

Where:

A_s = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
C_{is} = Concentration of the internal standard in the sample loop in ppmv.
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made D=1. The dilution factor is always dimensionless.
V = Volume of the extract injected (μL). The normal injection volume for samples and calibration standards must be the same. For headspace gas analysis, V is not applicable and set at 1.
RRF = Relative response factor from the initial calibration.

9. ICAL Calibration Equation for Hydrogen or Methane

An initial calibration curve is constructed by using a least squares linear regression to plot area responses versus concentration of analyte for all calibration standards analyzed. The calibration equation is of the following general form.

$A_x = mC_x + b$

Where:

A_x =Total area response for analyte.
C_x =Amount (Vol%) of analyte.
m =Slope of line (proportionality constant).
b =Intercept (regression constant).

10. Analyte Concentration for Hydrogen or Methane

$C_x = ((A_x - b)/m)\ X\ DF$

Where: A_x, C_x, m, and b are defined above and

DF = dilution factor

Attachment 2 – Data Reporting Guidance

A. Document information clearly and legibly to allow reconstruction and verification of the analysis process by another party at a later date.

B. Record entries in logbooks, computer-generated worksheets, or other documentation in chronological order, with the run sequence evident for instrumental analyses. Include time of analysis as necessary to verify the run sequence.

C. Record data clearly and legibly with indelible ink.

NOTE
Medium point pens are recommended because they produce records that are more legibly copied than do fine point pens.

a. Do not use pencil or erasable ink for analytical record keeping.

b. Check felt-tip and roller-ball pen ink for permanency before using them for analytical record keeping. If the ink smears after rubbing an entry with a damp fingertip, do not use the pen.

D. For computer-controlled instruments and equipment, always check the computer clock time for accuracy before starting any analyses that use the computer clock for a date/time stamp.

E. Clearly identify reportable results on the analytical records.

a. Explain (i.e. justify) any renalyses, and indicate if the original analysis, the reanalysis, or both are to be reported.

b. If multiple dilutions of the same sample are analyzed clearly indicate which dilution is to be reported.

c. In the case of multi-analyte methods, indicate which analyte is to be reported from which dilution or reanalysis.

F. Use the following steps to correct any error or omissions on hard copy (i.e. paper) data records:

a. Draw a single line through the error and enter the correct information near the line-out.

b. Initial and date all changes, including additions.

Attachment 2 – Data Reporting Guidance (Continued)

c. If necessary, add a brief explanation to clarify the reason for the change.

d. Do not overwrite or obliterate erroneous entries and never use correction fluid.

e. Ensure that any corrections made to paper records are also made to any associated computer-resident data whenever possible.

f. If it is not possible to correct computer-resident data, note this on the associated paper-copy record.

g. If the error or omission is identified on a photocopy of the original record (e.g., during review or reporting), correct the error on the original record and recopy it.

G. Use the following rounding rules when recording or reporting data:

a. If the figure following the one to be retained is less than 5, drop it (round down).

b. If the figure following the one to be retained is greater than 5, drop it and increase the last digit to be retained by 1 (round up).

c. If the figure following the last digit to be retained is equals 5 and there are no digits to the right of the 5 equal zero then round up. If the digit to be retained is odd, or round down if the digit is even. If there are digits other than zero to the right of the 5, round up.

d. If a series of multiple operations is to be performed (add, subtract, divide, multiply), carry all figures through the calculations, then round the final answer to the proper number of significant figures.

Attachment 3 – Independent Technical Reviewer Checklist

BDR Number _____

REQUIREMENT	Yes	No	COMMENT
1. The data have been reviewed for transcription errors.			
2. Data generation and reduction were conducted in a technically correct manner in accordance with the methods used (procedure with revision). Data was reported in the proper units (ppmv and volume %) and correct number of significant figures? (Minimum of 2)			
3. Were all calculations verified by a valid calculation program, a spot check of verified calculation programs, and/or 100% percent check of all hand calculations. Values that are not verifiable to within rounding or signicant differences must be rectified prior to completion of ITR.			
4. The sampling and analytical data QA documentation for Batch Data Report is complete and includes raw data, chain-of-custody (COC), ICAL reponse factor report and Hydrogen and Methane calibration curve, QC sample results, copies or originals of gas canister sample tags.			
5. QC sample results are within established control limits, and if not, the data have been appropriately qualified in accordance with data useablility criteria. Data outside of established control limits will be qualified as appropriate, assigned an appropriate qualifier flag, discussed in the case narrative.			
6 Did BFB tunes meet acceptance criteria of Table 2?			
7. Were all samples run within 12 hours of the injection of BFB?			
8. Did initial calibration meet acceptance criteria for each compound of < 35 %RSD?			
9. Did continuing calibration meet acceptance criteria for each compound of less or equal to 30 %D.?			
10. Did the internal standard responses meet acceptance criteria of ≥ 50% to ≤ 200%?			
11. Did the internal standard retention times (RTs) meet acceptance criteria of ± 30 seconds?			
12. Did the laboratory blank meet acceptance criteria of ≤ 3 X MDL?			

Attachment 3 – Independent Technical Reviewer Checklist (Continued)

BDR Number _____

REQUIREMENT	Yes	No	COMMENT
13. If the LCS/LCS duplicate was used did the analysis meet the acceptance criteria of ≤ 25 %R?			
14. Did the LCS recovery meet the acceptance criteria of 70 to 130 %R?			
15. If used, did the Drum duplicate meet the acceptance criteria of ≤ 25 %R?			
16. Was the MDL study performed semiannually?			
17. Did the H_2 and CH_4 RTs meet acceptance criteria of established window?			
18. Reporting flags were assigned correctly as specified in the Table 4.			
19. Sampling preservation requirements were met. (0 to 40 °C).			
20. Were the TICs correctly identified and reported?			
21. Were there any NCR and are they included in the batch data report?			
22. QAO described in Section 2.8 of this procedure were met.			
23. Field sampling records are complete.			

Print Name _____ Signature _____ Date _____

Attachment 4 – Laboratory Control Sample Data Sheet (Example)

LABORATORY CONTROL SAMPLE DATA SHEET

Sample ID:_LCS ___ Date Analyzed:_____ Instrument ID:_____
Lab File ID: _____ Time Analyzed:_____ Column ID:_____
Injection Volume (cm^3): _____

	Concentration (ppbv, except hydrogen and methane)			%	
CAS RN	COMPOUND NAME	Spiked	Reported	Recovery	Q
1333-74-0	hydrogen(in V/V%)				
74-82-8	methane(in V/V%)				
67-56-1	Methanol				
60-29-7	Ethyl Ether				
75-35-4	1,1-Dichloroethene				
76-13-1	1,1,2-Trichloro-1,2,2-Trifluoroethane				
67-64-1	Acetone				
75-15-0	Carbon Disulfide				
75-09-2	Methylene Chloride				
156-60-5	trans-1,2-Dichloroethene				
75-34-3	1,1-Dichloroethane				
156-59-2	cis-1,2-Dichloroethene				
78-93-3	Methyl ethyl ketone				
67-66-3	Chloroform				
71-55-6	1,1,1-Trichloroethane				
110-82-7	Cyclohexane				
56-23-5	Carbon Tetrachloride				
71-43-2	Benzene				
107-06-2	1,2-Dichloroethane				
79-01-6	Trichloroethene				
71-36-3	Butanol				
108-10-1	Methyl isobutyl ketone				
108-88-3	Toluene				
127-18-4	Tetrachloroethene				
108-90-7	Chlorobenzene				
100-41-4	Ethylbenzene				
108-38-3	m and p-xylene				
95-47-6	o-xylene				
75-25-2	Bromoform				
79-34-5	1,1,2,2-Tetrachloroethane				
108-67-8	1,3,5-Trimethylbenzene				
95-63-6	1,2,4-Trimethylbenzene				

LCS Recovery:_____outside limits out of _____total.
Comments:

Attachment 5 – LCS Duplicate Precision Data Sheet (Example)

LCS DUPLICATE PRECISION DATA SHEET

Client:_____ Contract:
Lab File ID: _____ Canister No.:_____
Lab File ID:_____
Client Sample No.: _____ Lab Dup File ID:_____
Instrument ID:_____ _____ GC Column ID:_____

COMPOUND NAME	Sample Conc.	Duplicate Conc.	RPD
hydrogen(in V/V%)			
methane(in V/V%)			
Methanol			
Ethyl Ether			
1,1-Dichloroethene			
1,1,2-Trichloro-1,2,2-Trifluoroethane			
Acetone			
Carbon Disulfide			
Methylene Chloride			
trans-1,2-Dichloroethene			
1,1-Dichloroethane			
cis-1,2-Dichloroethene			
Methyl ethyl ketone			
Chloroform			
1,1,1-Trichloroethane			
Cyclohexane			
Carbon Tetrachloride			
Benzene			
1,2-Dichloroethane			
Trichloroethene			
Butanol			
Methyl isobutyl ketone			
Toluene			
Tetrachloroethene			
Chlorobenzene			
Ethylbenzene			
m and p-xylene			
o-xylene			
Bromoform			
1,1,2,2-Tetrachloroethane			
1,3,5-Trimethylbenzene			
1,2,4-Trimethylbenzene			

U = Concentration Result Below Method Detection Limit
NA = Not Applicable

Attachment 6 – Sample Duplicate Precision Data Sheet (Example)

SAMPLE DUPLICATE PRECISION DATA SHEET

Client:_____ Contract:
Lab File ID: _____ Canister No.:_____
Lab File ID:_____
Client Sample No.: _____ Lab Dup File ID:_____
Instrument ID:_____ _____ GC Column ID:_____

COMPOUND NAME	Sample Conc.	Duplicate Conc.	RPD
hydrogen(in V/V%)			
methane(in V/V%)			
Methanol			
Ethyl Ether			
1,1-Dichloroethene			
1,1,2-Trichloro-1,2,2-Trifluoroethane			
Acetone			
Carbon Disulfide			
Methylene Chloride			
trans-1,2-Dichloroethene			
1,1-Dichloroethane			
cis-1,2-Dichloroethene			
Methyl ethyl ketone			
Chloroform			
1,1,1-Trichloroethane			
Cyclohexane			
Carbon Tetrachloride			
Benzene			
1,2-Dichloroethane			
Trichloroethene			
Butanol			
Methyl isobutyl ketone			
Toluene			
Tetrachloroethene			
Chlorobenzene			
Ethylbenzene			
m and p-xylene			
o-xylene			
Bromoform			
1,1,2,2-Tetrachloroethane			
1,3,5-Trimethylbenzene			
1,2,4-Trimethylbenzene			

U = Concentration Result Below Method Detection Limit
NA = Not Applicable

Attachment 7 – Headspace Gas Sample Analysis Results Summary (Example)

GAS ANALYSIS DATA SHEET

CARLSBAD ENVIRONMENTAL MONITORING AND RESEARCH CENTER

TRU WASTE CHARACTERIZATION PROGRAM

Lab Sample ID:		**Field Sample ID:**	
Analytical Batch:		**Sampling Batch:**	
Data Report:		**Method:**	GC/MS - GC/TCD
Procedure:	CCP-TP-143	**Revision:**	0
Date/Time Analyzed:		**Date Sampled:**	
Lab File ID:		**Date Received:**	
Instrument ID:		**Composite:**	

GC/MS - Organic Compounds	Concentration (ppmV)	Qualifiers
Acetone		
Benzene		
Bromoform		
Butanol		
Carbon disulfide		
Carbon tetrachloride		
Chlorobenzene		
Chloroform		
Chloromethane		
Cyclohexane		
1,1-Dichloroethane		
1,2-Dichloroethane		
1,1-Dichloroethylene		
cis- 1,2-Dichloroethylene		
trans- 1,2-Dichloroethylene		
1,2-Dichloropropane		
Ethyl benzene		
Ethyl ether		
Methyl ethyl ketone		
Methyl isobutyl ketone		
Methylene chloride		
1,1,2,2-Tetrachloroethane		
Toluene		
1,1,1-Trichloroethane		
Trichloroethyelene		
1,1,2-Trichloro-1,2,2-trifluoroethane		
1,3,5-Trimethylbenzene		
1,2,4-Trimethylbenzene		
p/m-Xylene		
o-Xylene		

TCD	Concentration (VOL%)	Qualifiers
Hydrogen		
Methane		

Print Name: _____

Signature: _____ Date: _____

Attachment 7 – Headspace Gas Sample Analysis Results Summary (Example) (continued)

GAS ANALYSIS DATA SHEET

CARLSBAD ENVIRONMENTAL MONITORING AND RESEARCH CENTER

TENTATIVELY IDENTIFIED COMPOUNDS

TRU WASTE CHARACTERIZATION PROGRAM

Lab Sample ID:		**Field Sample ID:**	
Analytical Batch:		**Sampling Batch:**	
Data Report:		**Method:**	GC/MS - GC/TCD
Procedure:	CCP-TP-143	**Revision:**	0
Date/Time Analyzed:		**Date Sampled:**	
Lab File ID:		**Date Received:**	
Instrument ID:		**Composite:**	

Compound Name	Concentration (ppmV)	Qualifiers	Retention Time (Minutes)

Attachment 8 – Blank Summary (Example)

BLANK SUMMARY

Client:_____ Contract:

Blank ID:_Blank_____ Date Analyzed:_____ Instrument ID:_____
Blank File ID:_____ Time Analyzed:_____ Column ID: _____

THIS BLANK APPLIES TO THE FOLLOWING SAMPLES:

	Sample No.	Laboratory ID			Analysis	
		Sample	File	Canister	Date	Time
1						
2						
3						
4						
4						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						

Comments:

Attachment 9 – Headspace Gas Canister Internal Standard Area and RT Summary (Example)

HEADSPACE GAS- CANISTER

INTERNAL STANDARD AREA AND RT SUMMARY

Client: _____ Contract: _____
Lab File: _____ Date Analyzed: _____

		Fluorobenzene				Chlorobenzene-d_5			
		Area	#	RT	#	Area	#	RT	#
	ICAL3								
	Upper Limit	0		0.50		0		0.50	
	Lower Limit	0		-0.50		0		-0.50	
	Sample No.								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

AREA: Upper Limit: 200% of internal standard area. RT: Upper Limit: +0.5 minutes of internal standard RT.
Lower Limit: 50% of internal standard area. Lower Limit: -0.5 minutes of internal standard RT.

All values outside of the QC limits must be followed by an "*" under the "#" column.

Attachment 10 – HSG Operator Checklist

Analytical Batch Number:	Procedure Revision:
HSG Operator Signature:	Analysis Date:

Criteria	Yes	No	Comments
1. Samples analyzed in accordance with CCP-TP-143.			
2. BFB tune analyzed art the beginning of the run (once very 12 hours of instrument operation), and meets acceptance criteria.			
3. CCAL analyzed at the beginning of the run (once every 12 hours of instrument operation), and meets acceptance criteria.			
4. LB analyzed after the CCAL, and meets acceptance criteria.			
5. Analytical batch QC (LCS, laboratory duplicate or LCS duplicate) analyzed if appropriate, and meets acceptance criteria. (N/A if these analyses are performed on another day within the batch)			
6. All samples quantitated within the calibration range for all target analytes, diluted and reanalyzed, or scheduled for reanalysis within the batch.			
7. All non-target analyte peaks having total ion areas > 10% of the nearest internal standard evaluated against a NIST Appendix VIII VOC mass spectral library for tentative identification.			
8. All TICs identified using criteria defined in CCP-TP-143.			
10. LB analyzed after the CCAL, and meets acceptance criteria.			
11. All raw data evaluated, signed/initialed and dated in indelible ink.			
12. Raw data reviewed for completeness and accuracy.			

Attachment 11 – Headspace Gas Sample Preparation Form

RECEIVING NUMBER:									
Client Sample ID	**Canister ID**	**Original Pressure (psia or psig)**	**Final Pressure (psia or psig)**	**Dilution Factor (Current Dilution)**	**Dilution Number**	**Pressurization Gas**	**Internal Standard Mix ID**	**Current Total Dilution Factor**	**Comments**

Attachment 12 – MDL Reporting Form (Example)

	A	B	C	D	E	F	G	H	I	J	K
1	Name	Bulb Vol(mls)	Gas Bulb Molar Volume (ambient)	Target Conc. ppmv	Target moles analyte	Bulb Inj Vol(uls)	Density (g/ml)	MW g/mol	mls/Moles	[Expected] PPMV	mgs compound
2	***HGAS Calcs Rev. 2***										
3	*Benzene*	500.00	1.625E-02	**10.00**	1.625E-07	0.014	0.8765	78.00	88.99	9.0429	0.0127
4	*Bromoform*	500.00	1.625E-02	**10.00**	1.625E-07	0.014	2.8899	253.00	87.55	9.0429	0.0411
5	*Chlorobenzene*	500.00	1.625E-02	**10.00**	1.625E-07	0.017	1.1058	113.00	102.19	9.0429	0.0184
6	*Chloroform*	500.00	1.625E-02	**10.00**	1.625E-07	0.013	1.484	119.50	80.53	9.0429	0.0194
7	*Cyclohexane*	500.00	1.625E-02	**10.00**	1.625E-07	0.018	0.7785	84.00	107.90	9.0429	0.0136
8	*1,1-Dichloroethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.014	1.1757	99.00	84.21	9.0429	0.0161
9	*1,2-Dichloroethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.013	1.2351	99.00	80.16	9.0429	0.0161
10	*1,1-Dichloroethylene*	500.00	1.625E-02	**10.00**	1.625E-07	0.013	1.218	97.00	79.64	9.0429	0.0158
11	*cis-1,2-Dichloroethene*	500.00	1.625E-02	**10.00**	1.625E-07	0.012	1.2837	97.00	75.56	9.0429	0.0158
12	*Ethylbenzene*	500.00	1.625E-02	**10.00**	1.625E-07	0.020	0.867	106.00	122.26	9.0429	0.0172
13	*Ethyl Ether*	500.00	1.625E-02	**10.00**	1.625E-07	0.017	0.7138	74.00	103.67	9.0429	0.0120
14	*Methylene chloride*	500.00	1.625E-02	**10.00**	1.625E-07	0.010	1.3348	85.00	63.68	9.0429	0.0138
15	*1,1,2,2-Tetrachloroethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.017	1.5953	168.00	105.31	9.0429	0.0273
16	*Tetrachloroethene*	500.00	1.625E-02	**10.00**	1.625E-07	0.017	1.5866	166.00	104.63	9.0429	0.0270
17	*Toluene*	500.00	1.625E-02	**10.00**	1.625E-07	0.017	0.8669	92.00	106.13	9.0429	0.0149
18	*1,1,1-Trichloroethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.016	1.339	133.00	99.33	9.0429	0.0216
19	*Trichloroethene*	500.00	1.625E-02	**10.00**	1.625E-07	0.015	1.4642	131.00	89.47	9.0429	0.0213
20	*1,1,2-Trichloro-1,2,2-Trifluoroethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.019	1.5635	187.00	119.60	9.0429	0.0304
21	*1,2,4-Trimethylbenzene*	500.00	1.625E-02	**10.00**	1.625E-07	0.022	0.8758	120.00	137.02	9.0429	0.0195
22	*1,3,5-Trimethylbenzene*	500.00	1.625E-02	**10.00**	1.625E-07	0.023	0.8652	120.00	138.70	9.0429	0.0195
23	*m-Xylene*	500.00	1.625E-02	**5.00**	8.123E-08	0.010	0.8642	106.00	122.66	4.5215	0.0086
24	*o-Xylene*	500.00	1.625E-02	**10.00**	1.625E-07	0.020	0.8802	106.00	120.43	9.0429	0.0172
25	*p-Xylene*	500.00	1.625E-02	**5.00**	8.123E-08	0.010	0.8611	106.00	123.10	4.5215	0.0086
26	*Carbon Tetrachloride*	500.00	1.625E-02	**10.00**	1.625E-07	0.016	1.589	154.00	96.92	9.0429	0.0250
27	*Acetone*	500.00	1.625E-02	**100.00**	1.625E-06	0.119	0.7899	58.00	73.43	90.4293	0.0942
28	*Butanol*	500.00	1.625E-02	**100.00**	1.625E-06	0.148	0.8098	74.00	91.38	90.4293	0.1202
29	*Methanol*	500.00	1.625E-02	**100.00**	1.625E-06	0.066	0.7914	32.00	40.43	90.4293	0.0520
30	*Methyl ethyl Ketone*	500.00	1.625E-02	**100.00**	1.625E-06	0.145	0.8054	72.00	89.40	90.4293	0.1170
31	*Methyl isobutyl ketone*	500.00	1.625E-02	**100.00**	1.625E-06	0.203	0.801	100.00	124.84	90.4293	0.1625
32	*trans-1,2-Dichloroethene*	500.00	1.625E-02	**10.00**	1.625E-07	0.013	1.2565	96.94	77.15	9.0429	0.0157
33	*Carbon Disulfide*	500.00	1.625E-02	**10.00**	1.625E-07	0.010	1.2632	76.13	60.27	9.0429	0.0124
34	*1,2-Dichloropropane*	500.00	1.625E-02	**10.00**	1.625E-07	0.016	1.156	112.99	97.74	9.0429	0.0184
35	*Chloromethane*	500.00	1.625E-02	**10.00**	1.625E-07	0.009	0.9159	50.49	55.13	9.0429	0.0082
36	**Column Heading Totals**										1.0534
37											
38	Ambient Pressure (Torr)	**626.50**		**Actual Injection Volume (uls) =**			**1.00**				
39	Correction Factor/Pressure	1.21									
40	Bottle Temperature (Celcius)	**36.00**									
41	Bottle Temperature (Kelvin)	309.00									
42	Correction Factor/Temp	1.13									
43	Molar Gas Volume (mls)	30775.68									
44											

Attachment 12 – MDL Reporting Form (Example) (continued)

	L	M	N	O	P	Q	R	S	T	U	V
1	Ordered wt/wt%	A2	Actual wt/wt%	Actual PPMV	Mole Ratio	total mls	volume fraction	# of moles	GC INJ VOL(mls)	Moles On Col	ng on column
2											
3	1.20295	0.9975	1.20	9.02074	1	88.99	0.01308	1.470E-07	0.1	3.249E-11	2.534
4	3.90188	0.9995	3.90	9.03857	1	87.55	0.01287	1.470E-07	0.1	3.249E-11	8.221
5	1.74274	0.9755	1.70	8.82117	1	102.19	0.01502	1.470E-07	0.1	3.249E-11	3.672
6	1.84298	0.9767	1.80	8.83202	1	80.53	0.01184	1.470E-07	0.1	3.249E-11	3.883
7	1.29549	0.9695	1.26	8.76730	1	107.90	0.01586	1.470E-07	0.1	3.249E-11	2.729
8	1.52682	0.9824	1.50	8.88406	1	84.21	0.01238	1.470E-07	0.1	3.249E-11	3.217
9	1.52682	0.9824	1.50	8.88406	1	80.16	0.01178	1.470E-07	0.1	3.249E-11	3.217
10	1.49598	1.0027	1.50	9.06724	1	79.64	0.01171	1.470E-07	0.1	3.249E-11	3.152
11	1.49598	1.0027	1.50	9.06724	1	75.56	0.01111	1.470E-07	0.1	3.249E-11	3.152
12	1.63478	0.9787	1.60	8.85054	1	122.26	0.01797	1.470E-07	0.1	3.249E-11	3.444
13	1.14126	0.9638	1.10	8.71599	1	103.67	0.01524	1.470E-07	0.1	3.249E-11	2.404
14	1.31091	0.9917	1.30	8.96768	1	63.68	0.00936	1.470E-07	0.1	3.249E-11	2.762
15	2.59097	1.0035	2.60	9.07443	1	105.31	0.01548	1.470E-07	0.1	3.249E-11	5.459
16	2.56013	1.0156	2.60	9.18377	1	104.63	0.01538	1.470E-07	0.1	3.249E-11	5.394
17	1.41887	0.9867	1.40	8.92269	1	106.13	0.01560	1.470E-07	0.1	3.249E-11	2.989
18	2.05119	1.0238	2.10	9.25813	1	99.33	0.01460	1.470E-07	0.1	3.249E-11	4.322
19	2.02034	0.9899	2.00	8.95188	1	89.47	0.01315	1.470E-07	0.1	3.249E-11	4.257
20	2.88400	1.0055	2.90	9.09310	1	119.60	0.01758	1.470E-07	0.1	3.249E-11	6.076
21	1.85070	1.0266	1.90	9.28384	1	137.02	0.02014	1.470E-07	0.1	3.249E-11	3.899
22	1.85070	1.0266	1.90	9.28384	1	138.70	0.02039	1.470E-07	0.1	3.249E-11	3.899
23	0.81739	0.9787	0.80	4.42527	0.5	61.33	0.00902	7.351E-08	0.1	1.625E-11	1.722
24	1.63478	0.9787	1.60	8.85054	1	120.43	0.01771	1.470E-07	0.1	3.249E-11	3.444
25	0.81739	0.9787	0.80	4.42527	0.5	61.55	0.00905	7.351E-08	0.1	1.625E-11	1.722
26	2.37506	1.0105	2.40	9.13789	1	96.92	0.01425	1.470E-07	0.1	3.249E-11	5.004
27	8.94503	10.0615	9.00	909.85050	10	734.27	0.10795	1.470E-06	0.1	3.249E-10	18.846
28	11.41262	9.9889	11.40	903.29302	10	913.81	0.13435	1.470E-06	0.1	3.249E-10	24.045
29	4.93519	9.9287	4.90	897.84553	10	404.35	0.05945	1.470E-06	0.1	3.249E-10	10.398
30	11.10417	9.9962	11.10	903.95332	10	893.97	0.13143	1.470E-06	0.1	3.249E-10	23.395
31	15.42246	9.9854	15.40	902.97608	10	1248.44	0.18354	1.470E-06	0.1	3.249E-10	32.493
32	1.49505	1.0033	1.50	9.07285	1	77.15	0.01134	1.470E-07	0.1	3.249E-11	3.150
33	1.17411	1.0220	1.20	9.24232	1	60.27	0.00886	1.470E-07	0.1	3.249E-11	2.474
34	1.74258	0.9756	1.70	8.82195	1	97.74	0.01437	1.470E-07	0.1	3.249E-11	3.671
35	0.77868	1.0274	0.80	9.29052	1	55.13	0.00810	1.470E-07	0.1	3.249E-11	1.641
36			99.86			6801.83		1.13E-05			
37											
38											
39											
40											
41											
42											
43											
44											

Attachment 13 – Standard Preparation VOC and H_2 and CH_4 (Example)

H_2/CH_4 Stock Ids :	001-028-002	001-026-014	001-026-015	001-026-016	001-028-002	
% vol/vol :	0.075%	1.00%	10.00%	20.00%	0.075%	
Calibration Standard Injection Volume & Canister Standard Concentration						Date & Time of Preparation - 8/10/06 1100
Intermediate Ids :	I-005-006-001	I-005-006-002	I-005-006-003	I-005-006-004	I-005-006-005	PSIA (Meter ID 0223) = 29.00
desired ppmv :	9	20	60	100	200	Temp (K, Therm. Serial# 16953) = 298
approx inject (uL) :	4.95	11.00	32.99	54.98	109.96	Canister Volume (mL) = 1000
actual inject (uL) :	4.96	11.03	33.00	55.01	110.98	
Canister Standards Concentrations (ppmv)						Compound
	0.00	0.00	0.00	0.00	0.00	formaldehyde
	9.28	20.63	61.72	102.88	207.56	chloromethane
	89.54	199.11	595.71	993.03	2003.38	methanol
	8.69	19.32	57.81	96.36	194.41	ethyl ether
	9.06	20.15	60.28	100.49	202.74	freon-113
	90.72	201.75	603.59	1006.17	2029.91	acetone
	9.06	20.15	60.27	100.47	202.70	1,1-dichloroethylene
	8.96	19.93	59.62	99.39	200.51	methylene chloride
	9.23	20.52	61.39	102.34	206.46	carbon disulfide
	9.06	20.15	60.27	100.47	202.70	trans-1,2-dichloroethylene
	8.87	19.73	59.04	98.42	198.56	1,1-dichloroethane
	90.12	200.41	599.59	999.50	2016.45	2-butanone
	9.06	20.15	60.27	100.47	202.70	cis-1,2-dichloroethylene
	8.83	19.63	58.73	97.90	197.52	chloroform
	9.22	20.49	61.32	102.21	206.21	1,1,1-trichloroethane
	9.04	20.11	60.17	100.30	202.35	cyclohexane
	9.13	20.31	60.78	101.31	204.39	carbon tetrachloride
	8.87	19.73	59.04	98.42	198.56	1,2-dichloroethane
	8.99	20.00	59.84	99.75	201.25	benzene
	90.05	200.24	599.10	998.68	2014.79	1-butanol
	8.91	19.82	59.29	98.84	199.40	trichloroethene
	8.81	19.59	58.61	97.69	197.09	1,2-dichloropropane
	90.02	200.18	598.90	998.35	2014.12	4-methyl-2-pentanone
	8.89	19.78	59.18	98.65	199.02	toluene
	9.18	20.41	61.07	101.80	205.39	tetrachloroethene
	8.84	19.66	58.83	98.07	197.84	chlorobenzene
	8.82	19.62	58.70	97.85	197.41	ethylbenzene
	8.82	19.62	58.70	97.85	197.41	m & p-xylenes
	8.82	19.62	58.70	97.85	197.41	o-xylene
	9.03	20.09	60.11	100.20	202.15	bromoform
	9.07	20.17	60.34	100.58	202.91	1,1,2,2-tetrachloroethane
	9.26	20.58	61.58	102.65	207.08	1,3,5-trimethylbenzene
	9.26	20.58	61.58	102.65	207.08	1,2,4-trimethylbenzene

VOC Stock ID - 001-026-010
Internal Standard Stock ID - 001-026-024

VOC Stock Opened Date - 8/10/06
VOC Stock Exp. Date - 12/11/06
VOC Intermediate Exp. Date - 10/11/06
Internal Standard Stock Exp. Date - 4/27/07
H_2CH_4 Stock Exp. Date - 2/17/07

Prepared By: KP

Printed Name: Karl Pennock

Signature: Karl Pennock

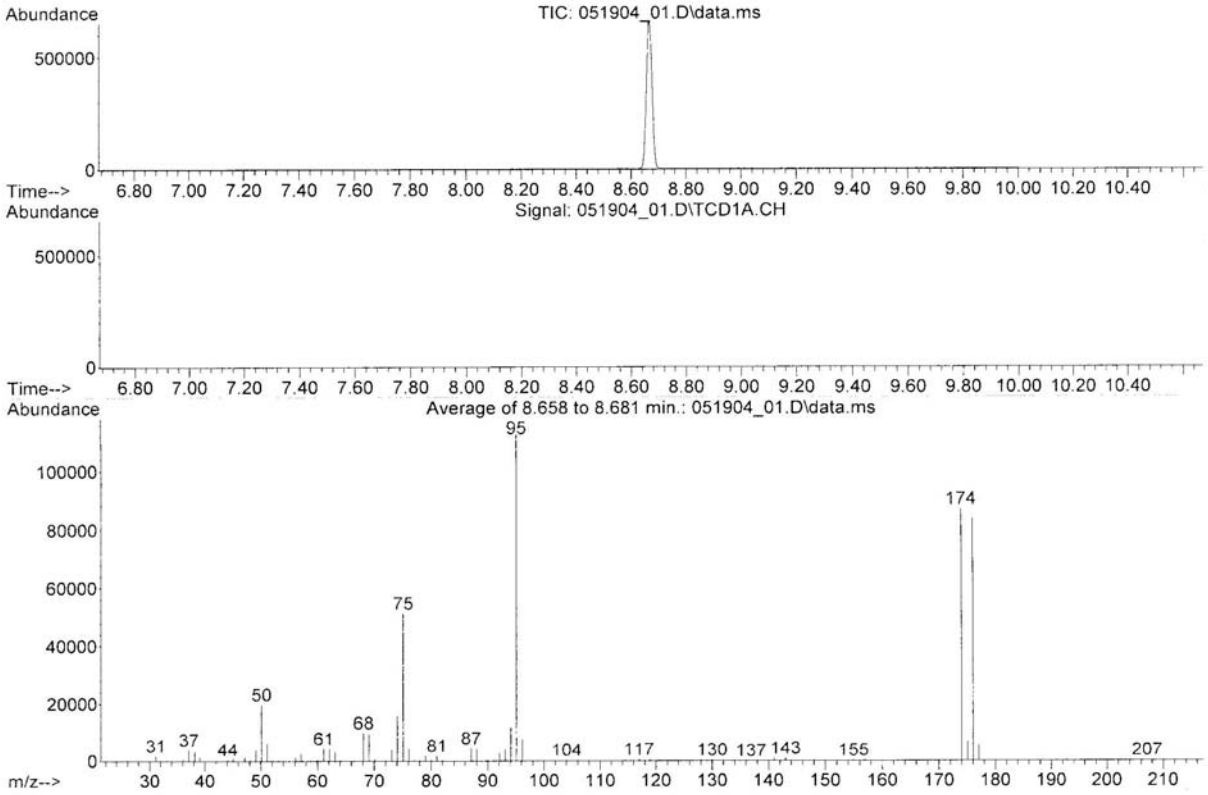
Comments: N/A=Not applicable

Attachment 14 – BFB Tune Form (Example)

```
Data Path : C:\Archives\MSDChem\1\DATA\051904\
Data File : 051904_01.D
Acq On    : 19 May 2004    8:58 am
Operator  : CS
Sample    : 0.075% H2/CH4 + BFB
Misc      : Tune_051904 (Sig #1);  (Sig #2)
ALS Vial  : 13   Sample Multiplier: 1

Integration File signal 1: RTEINT.P
Integration File signal 2: RTEINT2.P

Method    : C:\Archives\MSDChem\1\METHODS\050604.M
Title     : CEMRC Headspace Gas VOC/H2/C2H4 Calibration
Last Update  : Fri Jul 16 10:33:56 2004
```



Spectrum Information: Average of 8.658 to 8.681 min.

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	17.5	19705	PASS
75	95	30	60	45.5	51282	PASS
95	95	100	100	100.0	112669	PASS
96	95	5	9	6.8	7648	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	77.3	87058	PASS
175	174	5	9	7.7	6661	PASS
176	174	95	101	96.3	83870	PASS
177	176	5	9	6.6	5537	PASS

Attachment 15 – Continuing Calibration Evaluation Report (Example)

```
Data Path : C:\Archives\MSDChem\1\DATA\051904\
Data File : 051904_02.D
Acq On    : 19 May 2004    9:47 am
Operator  : CS
Sample    : CCAL
Misc      : CCAL_051904 (Sig #1);  (Sig #2)
ALS Vial  : 14   Sample Multiplier: 1

Quant Time: May 19 12:14:28 2004
Quant Method : D:\MSDCHEM\1\METHODS\050604.M
Quant Title  : CEMRC Headspace Gas VOC/H2/C2H4 Calibration
QLast Update : Fri May 07 10:19:59 2004
Response via : Initial Calibration


Min. RRF     :   0.000  Min. Rel. Area :  50%  Max. R.T. Dev  0.50min
Max. RRF Dev :  25%     Max. Rel. Area : 150%
```

		Compound	AvgRF	CCRF		%Dev	Area%	Dev(min)
1		Hydrogen	168.888	171.404	E3	-1.5	101	0.00
2		Methane	76.117	77.668	E3	-2.0	102	0.02
3	I	Fluorobenzene (IS1)	1.000	1.000		0.0	99	0.00
4		Formaldehyde	0.041	0.040		2.4	98	0.00
5		Chloromethane	0.151	0.148		2.0	100	0.00
6		Methanol	0.064	0.064		0.0	102	0.00
7		Ethyl Ether	0.180	0.174		3.3	98	0.00
8		Freon 113	0.460	0.455		1.1	100	0.00
9		Acetone	0.120	0.119		0.8	100	0.00
10		1,1-Dichloroethene	0.379	0.372		1.8	99	0.00
11		Methylene Chloride	0.280	0.281		-0.4	101	0.00
12		trans-1,2-Dichloroethene	0.372	0.360		3.2	100	0.00
13		1,1-Dichloroethane	0.457	0.455		0.4	101	0.00
14		2-Butanone	0.130	0.127		2.3	99	0.00
15		cis-1,2-Dichloroethene	0.358	0.353		1.4	100	0.00
16		Chloroform	0.494	0.493		0.2	102	0.00
17		1,1,1-Trichloroethane	0.504	0.505		-0.2	101	0.00
18		Carbon Tetrachloride	0.507	0.505		0.4	100	0.00
19		1,2-Dichloroethane	0.349	0.346		0.9	100	0.00
20		Benzene	0.817	0.791		3.2	99	0.00
21		1-Butanol	0.156	0.152		2.6	100	0.00
22		Trichloroethene	0.356	0.351		1.4	101	0.00
23	I	Chlorobenzene-d5 (IS2)	1.000	1.000		0.0	96	0.00
24		4-Methyl-2-Pentanone	0.137	0.136		0.7	97	0.00
25		Toluene	1.145	1.136		0.8	98	0.00
26		Tetrachloroethene	0.547	0.547		0.0	97	0.00
27		Chlorobenzene	0.938	0.893		4.8	94	0.00
28		Ethylbenzene	1.527	1.464		4.1	95	0.00
29		m & p - Xylenes	1.193	1.125		5.7	93	0.00
30		o - Xylene	1.236	1.124		9.1	91	0.00
31		Bromoform	0.545	0.537		1.5	96	0.00
32		1,1,2,2-Tetrachloroethane	0.785	0.707		9.9	90	0.00

```
   (#) = Out of Range                SPCC's out = 0  CCC's out = 0
```

Attachment 16 – Quantitation Report (Example)

```
Data Path : C:\Archives\MSDChem\1\DATA\051904\
Data File : 051904_02.D
Acq On    : 19 May 2004    9:47 am
Operator  : CS
Sample    : CCAL
Misc      : CCAL_051904 (Sig #1);  (Sig #2)
ALS Vial  : 14   Sample Multiplier: 1

Quant Time: May 19 12:14:28 2004
Quant Method : D:\MSDCHEM\1\METHODS\050604.M
Quant Title  : CEMRC Headspace Gas VOC/H2/C2H4 Calibration
QLast Update : Fri May 07 10:19:59 2004
Response via : Initial Calibration
```

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
3) Fluorobenzene (IS1)	6.184	96	53806	83.60	ppm	0.00
23) Chlorobenzene-d5 (IS2)	7.923	117	39842	72.30	ppm	0.00
Target Compounds						Qvalue
1) Hydrogen	1.729	GC1	1714035	10.35	pct	100
2) Methane	3.206	GC1	776677	10.55	pct	100
4) Formaldehyde	3.638	30	5954	225.68	ppm	100
5) Chloromethane	3.813	50	9218	94.82	ppm	97
6) Methanol	3.980	31	23625	570.08	ppm	98
7) Ethyl Ether	4.494	59	6454	55.66	ppm	97
8) Freon 113	4.642	101	16925	57.18	ppm	99
9) Acetone	4.654	58	43905	567.81	ppm	97
10) 1,1-Dichloroethene	4.692	61	13889	57.01	ppm	98
11) Methylene Chloride	4.920	49	10408	57.66	ppm	99
12) trans-1,2-Dichloroethene	5.072	61	13528	56.49	ppm	100
13) 1,1-Dichloroethane	5.289	63	17067	58.06	ppm	98
14) 2-Butanone	5.522	72	47105	564.35	ppm	99
15) cis-1,2-Dichloroethene	5.579	61	13255	57.45	ppm	99
16) Chloroform	5.712	83	18261	57.49	ppm	99
17) 1,1,1-Trichloroethane	5.872	97	18792	57.96	ppm	99
18) Carbon Tetrachloride	5.975	117	18684	57.29	ppm	100
19) 1,2-Dichloroethane	6.047	62	12893	57.45	ppm	99
20) Benzene	6.074	78	29442	55.98	ppm	99
21) 1-Butanol	6.150	41	56699	566.24	ppm	100
22) Trichloroethene	6.382	130	12888	56.29	ppm	99
24) 4-Methyl-2-Pentanone	6.884	85	48441	641.92	ppm	98
25) Toluene	7.086	91	35936	56.93	ppm	100
26) Tetrachloroethene	7.425	166	17499	58.03	ppm	99
27) Chlorobenzene	7.946	112	28582	55.28	ppm	98
28) Ethylbenzene	7.961	91	46600	55.39	ppm	99
29) m & p - Xylenes	8.022	91	71825	109.28	ppm	99
30) o - Xylene	8.300	91	35337	51.87	ppm	100
31) Bromoform	8.494	173	17192	57.24	ppm	99
32) 1,1,2,2-Tetrachloroethane	8.696	83	22597	52.23	ppm	99

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Attachment 17 – Headspace Gas Batch Data Report Cover Page

Lab Name: CEMRC

Analytical Batch Number: _____

Batch Date: _____

Procedure/Rev. Number: _____

Procedure/Rev. Number: _____

Sample Matrix/Type: Headspace Gas

Sample/Container Numbers: _____

Print Name: _____

Signature: _____

Date: _____

Attachment 18 – Headspace Gas Batch Data Report Table of Contents

Lab Name: CEMRC

Analytical Batch Number:

Instrument ID:

Batch Date:

Attachment 19 – Case Narrative (Example)

Laboratory Name: _____

Batch Data Report Number: _____

Analysis Date: _____

Procedure and Version Number: _____

QA/QC _____

NCRs: N/A _____

Print Name _____ Signature _____ Date _____