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CCP-TP-175

Revision 2

CCP
Analysis of Gas Samples for VOCs by GC/MS

EFFECTIVE DATE: 12/29/2010

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PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	05/02/2007	Initial Issue.
1	03/29/2010	Since sample compositing will no longer be performed, one of the criteria for determining if a non-target compound is a Tentatively Identified Compound (TIC) is being changed back to the original Permit non-composited samples requirement. The procedure is being changed back to the requirement that an unknown (non-target) compound is a TIC if the total ion area greater than 10 percent of the nearest internal standard. Deleting the use of maintenance logbook as it is not a Permit requirement and updating some procedure references.
2	12/29/2010	Revised to implement the renewal changes of the *Waste Isolation Pilot Plant Hazardous Waste Facility Permit*.

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1. ABSTRACT

The Environmental Chemistry Laboratory (ECL) uses this Central Characterization Project (CCP) procedure to provide instructions for conducting gas chromatography/mass spectrometry (GC/MS) analyses of gas samples containing high concentrations of volatile organic compounds (VOCs) collected in SUMMA® passivated canisters using a modification of USEPA Compendium Method TO-14 with mass flow controlled (MFC) sample introduction.

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. APPLICABILITY

2.1 This procedure is primarily designed to meet the requirements for analyses of headspace gas samples contained in SUMMA® canisters for CCP. This procedure may be applicable for other projects requiring similar determinations.

2.2 The VOC analytes of interest for CCP support are listed in Table 1. Method performance must be successfully demonstrated for other VOC compounds prior to use.

2.3 The calibration range of the method usually is 75 ng to 1250 ng. However, the range can vary as long as one calibration point is below the PRQL and DQOs are met.

2.4 The method is designed for sample volumes ranging from 30 to 300 mL.

3. DISCUSSION

Appropriate dilution or pressurization of the sample SUMMA®canisters is performed on a Gas Transfer Manifold per CCP-TP-179, *CCP Gas Transfer Manifold Systems and Sample Compositing*. The SUMMA® canister containing the diluted sample is attached to the analytical system and sample is introduced using a calibrated mass flow controller. The VOCs are then concentrated by collection on a solid sorbent trap. The temperature of the trap is then raised and the VOCs collected are desorbed into the gas chromatograph. The VOCs are separated on a megabore capillary column and detected using a mass spectrometer operating in the full scan mode. Internal standards and relative response factors are used for quantitation.

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4. SAFETY PRECAUTIONS

4.1 Chemical Hazards

4.1.1 Ensure that all instruments and SUMMA® canisters are vented to fume hoods or drop vents to prevent inhalation exposure to volatile organic compounds in gaseous samples and standards.

4.1.2 Wear safety glasses when injecting the liquid bromofluorobenzene (BFB) tuning standard.

4.1.3 Handle all chemicals per the requirements of LWP-14620, "Chemical Hygiene Plan." Wear appropriate personal protective equipment (PPE) per LI-1354-07-IRC/ECL, "Environmental Chemistry Laboratory (ECL) Gas Analysis Project."

4.2 Compressed Gas Hazards

4.2.1 Handle all compressed gases in accordance with LI-1354-07-IRC/ECL, "Environmental Chemistry Laboratory (ECL) Gas Analysis Project."

4.3 Physical Hazards

4.3.1 Exercise a high degree of awareness and caution when using syringes to inject the BFB tuning standard.

4.3.2 Use proper tools when connecting SUMMA® canisters to the analytical systems.

4.3.3 Exert the minimum force necessary while using hand tools to reduce the chance of tool slipping.

5. APPARATUS AND REAGENTS

5.1 Apparatus

5.1.1 Gas Chromatograph/Mass Spectrometer System

5.1.1.1 Gas Chromatograph: Hewlett Packard 5890 or equivalent.

5.1.1.2 Mass Spectrometer: Hewlett Packard 5970 or equivalent.

5.1.1.3 Purge and Trap device: Tekmar LSC-2 or equivalent.

5.1.1.4 Data System: HP Chem Station or equivalent.

5.1.1.5 J&W Scientific megabore capillary column, 0.53 mm I.D., 75 meters long with a 3.0 micrometer film thickness, or equivalent.

5.1.1.6 GC/MS Interface: jet separator.

5.1.2 10 µL blunt needle micro syringe.

5.1.3 Tekmar #8 Carbopak/Carbosieve or equivalent for trap.

5.1.4 SUMMA® passivated canisters or equivalent, Scientific Instrumentation Specialists or equivalent, 100 mL to 6000 mL capacity.

5.1.5 Mass Flow Controller, Matheson Model 8270 or equivalent.

5.1.6 Totalizer, Matheson Model SP-1793 or equivalent.

NOTE: *The mass flow controller and the totalizer are calibrated as a unit, and must have current calibration stickers from the INL Standards and Calibration Laboratory.*

5.1.7 Gas Transfer Manifold (see CCP-TP-179).

5.1.8 Wrenches, open-end, various sizes.

5.2 Reagents

5.2.1 Reagents

5.2.1.1 Methanol (CH3OH), Burdick and Jackson special purge and trap grade or equivalent.

5.2.1.2 Helium, ultra high purity (UHP).

5.2.1.3 Nitrogen, UHP.

5.2.2 Standard Gases

5.2.2.1 Calibration Stock Gas Standards

Stock gas calibration standard cylinder(s) of all analytes listed in Table 1 containing approximately 20 ng/mL of each analyte are purchased commercially (Scott Specialty Gases or equivalent). The stock gas standards must be certified by the manufacturer, and must have a known, valid relationship to a nationally-recognized standard (i.e., NIST), when available.

5.2.2.2 Internal Standards (ITSDs)

The recommended internal standards are fluorobenzene and chlorobenzene-d_5. Stock gas standards may be obtained from

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Scott Specialty Gases (or equivalent). The stock gas standard must be certified by the manufacturer, and must have a known, valid relationship to a nationally-recognized standard (i.e., NIST), when available. Prepare a secondary standard at approximately 3 ng/mL by transferring an amount (in torr) of the stock standard to a SUMMA® canister such that the ratio of the partial pressures of the ITSDs to the total pressure in the SUMMA®canister when multiplied by the initial concentration of the ITSDs gives approximately 3 ng/mL (e.g., for a 10 ng/mL stock standard, 10 ng/mL x 1100 torr/4000 torr = 2.75 ng/mL). The total pressure inside the SUMMA® canister should always be less than 3000 torr.

5.2.3 Calibration Gas Standards

Prepare a calibration gas standard containing all target analytes at approximately 5 ng/mL by dilution of the calibration stock gas standards. This standard is prepared in the same manner as the ITSDs (Section 5.2.2.2). Prepare a second standard at approximately 2.5 ng/mL by diluting the 5 ng/mL standard. These standards are prepared fresh at least every 18 months.

NOTE: *Calibration gas standards for site-specific analytes may be prepared separately.*

5.2.4 Liquid Tuning Standard

Prepare a 4-BFB standard at 25 ng/mL in methanol for injection as a liquid. This standard is remade at least every 18 months.

5.2.5 QC Standards

5.2.5.1 Continuing Calibration (CCAL) Standard

The 5 ng/mL calibration standard or equivalent (see Section 5.2.3) may be used as the CCAL standard.

5.2.5.2 Laboratory Control Sample (LCS)

Prepare the LCS from gas stock standard containing at least 6 of the target analytes in Table 1. The standard must be purchased from a different source than the calibration stock standards. This standard is remade at least once every 18 months.

5.2.5.3 Laboratory Blanks (LBs)

Laboratory blanks consist of SUMMA®canisters filled with ultra high purity nitrogen using the Gas Transfer Manifold (see CCP-TP-179).

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5.2.5.4 Method Performance Samples

Prepare standards used to determine or demonstrate method performance (e.g., MDLs, precision and accuracy) by diluting the stock gas calibration standards or the LCS gas stock standard to appropriate concentrations. Standards used for method performance demonstration must include all target analytes listed in Table 1.

NOTE: *Site-specific analytes may be contained in separate standards.*

6. SAMPLE HANDLING

6.1 IF samples were received in containers other than SUMMA® canisters, THEN transfer them to SUMMA® canisters before analysis.

6.2 Store SUMMA® canisters at room temperature (<40 °C) prior to analysis.

6.3 Use SUMMA® canisters cleaned per CCP-TP-178, *CCP SUMMA® Canister Cleaning*, for sample and standard transfers and dilutions.

6.4 Maintain the VOC analysis laboratory free of solvents in order to minimize laboratory cross-contamination.

6.5 Avoid use of non-TFE plastic coating, non-TFE thread sealants or flow controllers with rubber components in the analytical system.

6.6 Pressurize all samples to at least one atmosphere (atm) above ambient either during collection or by means of a gas transfer manifold (see CCP-TP-179). Use any resultant dilution data to calculate the final results.

6.7 Screen all samples other than blanks and those of known concentration using GC/FID (see CCP-TP-173, *CCP Analysis of Gas Samples for VOCs by GC/FID*) prior to GC/MS analysis.

6.7.1 Based on the GC/FID screen data, dilute the sample so that the compound found at the highest concentration in the screen will put approximately 850 ng (assuming an initial calibration curve of 30 ng - 1000 ng) into the GC/MS system.

Example: The GC screen gives a concentration for carbon tetrachloride (molecular weight = 153.82) of 800 ppmv in a sample diluted 3.5 times.

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1. Convert ppmv to ng/mL.

$$ng/mL = \frac{ppmv \times mw}{24.5 \times D} = \frac{800 \times 153.82}{24.5 \times 3.5} = 1435\, ng/mL$$

where: mw = molecular weight,
D = dilution factor, and
GC loop size = 1 mL.

2. Divide the sample ng/mL value by a number less than or approximately equal to 40 to yield a concentration in the approximate range of 8.5 ng/mL to 27 ng/mL, so that 30 mL to 100 mL of sample will deliver approximately 850 ng to the GC/MS system. This sample will require double dilution because:

$$\frac{1435}{40} = 35.9 \quad ng/mL$$

A possible dilution would be a 14x followed by a 12x:

$$\frac{1435}{14} = 102.5\, ng/mL \quad \text{and} \quad \frac{102.5}{12} = 8.5\, ng/mL \quad (use\, 100\, mL)$$

The total dilution factor for this sample would be:

$$3.5 \times 14 \times 12 = 588$$

7. PROCEDURES

7.1 GC/MS Operating Conditions

7.1.1 Typical GC/MS Operating Conditions

NOTE: *Operating conditions may be optimized as necessary as long as actual operating conditions used for sample analysis match those used for the associated ICAL.*

Electron Energy: 70 volts (nominal)

Mass Range: 35-260 amu, 29-260 amu (when methanol is an analyte)

Scan Time: One scan/second or enough to give 5 scan/peak

Source Temperature: According to manufacturers specifications 180-250°C

Jet Separator Heater (DetA): 220°C

Transfer Line Temperature (DetB): 225°C

Carrier Gas: UHP Helium at 30 mL/minute

BFB Column Temperature: 130 °C and hold for 8 minutes (BFB)

Sample/Standard Column Temperature: 35°C for 8 minutes
8 °C/minute to 165 °C
20 °C/minute to 220 °C.

Trap Conditions:

Initial temperature	< 30 °C
Desorb preheat	100 ° C
Desorb	4 minutes @ 220 °C
Bake trap	260 °C (see 7.5.18)

7.2 Analytical Run Sequence for Sample Analysis

7.2.1 Initial Calibration (ICAL) Sequence

- BFB tune
- Calibration standards

7.2.2 Daily Analytical Run Sequence

- BFB Tune
- Continuing Calibration Standard*
- Laboratory Blank
- Laboratory Control Sample (one per analytical batch)
- Samples
- Sample Duplicate (one per analytical batch)

* IF samples are analyzed immediately following an ICAL, THEN use the ~500 ng ICAL standard as the daily CCAL for comparing internal standard areas and retention times.

7.3 BFB Tuning Procedures

7.3.1 At the beginning of each 12 hours of operation or prior to a calibration, inject 2 µL of the 25 ng/mL BFB Standard (50 ng on column) into the injection port of the GC using a syringe with a blunt end needle.

7.3.2 Check the key ion abundance against the criteria in Table 2 BFB Key Ion Abundance Criteria.

7.3.2.1 IF the key ion abundance criteria in Table 2 are met, THEN proceed with analyses.

7.3.2.2 IF the key ion abundance criteria in Table 2 are NOT met, THEN correct the problem and repeat the BFB tune (step 7.3) until it is acceptable.

7.4 Calibration Procedures

7.4.1 Initial Calibration (ICAL)

7.4.1.1 Meet the BFB tune criteria (see Section 7.3).

7.4.1.2 Using calibration canisters containing all analytes (approximately 2.5 ng/mL, approximately 5 ng/mL, see Section 5.2.3) and the canister containing internal standards (approximately 3 ng/mL, see Section 5.2.2.2), analyze six standards for calibration according to the procedures given in Section 7.5. See Table 3 Calibration Standards for examples.

NOTE: *Six standards are normally analyzed. Five calibration points may be used if necessary as long as the low calibration point is at or below the PRQL.*

7.4.1.3 Calculate the relative response factors (RRF) for each analyte in each of the calibration standards using equation 9.1.

7.4.1.4 Calculate the percent relative standard deviation (RSD) of the RRF over the calibration points, using equation 9.2.

7.4.1.4.1 IF the % RSD for all analytes is < 35%, THEN proceed with sample analyses..

7.4.1.4.2 IF the %RSD for any target analyte is ≥35, THEN correct the problem and repeat the ICAL (step 7.4.1.2 through 7.4.1.4).

7.4.1.5 After a valid ICAL has been achieved, analyze a LCS standard prior to analysis of any samples to provide second-source verification of the calibration.

7.4.1.5.1 Calculate the percent recovery (%R) of all target analytes in the LCS, using equation 9.6.

7.4.1.5.2 IF the %Rs for all analytes in the LCS are between 70% and 130%,
THEN proceed with continuing calibration and sample analyses.

7.4.1.5.3 IF the %R for any target analyte in the LCS does not meet the 70% to 130% criteria,
THEN perform corrective action per Section 8.1.4 before proceeding with analyses of samples.

7.4.2 Continuing Calibration (CCAL)

7.4.2.1 Meet BFB tune criteria (see Section 7.3).

7.4.2.2 Using 5 ng/mL calibration standard, deliver a midrange standard (≈ 500 ng) to the analytical system (see Section 7.5).

7.4.2.3 Using computer software, calculate the RRFs for each target analyte according to equation 9.1.

7.4.2.4 Using computer software, calculate the percent difference (%D) between the initial calibration average RRFs and the CCAL RRFs for each analyte, according to equation 9.3.

7.4.2.4.1 IF the % D ≤ 30 for each target analyte
AND the retention time (RT) of each target analyte is within 30 seconds of the average ICAL retention time,
THEN proceed with sample analyses.

7.4.2.4.2 IF the %D for any target analyte is >30,
OR the RT for any target analyte is more than 30 seconds from the average ICAL RT,
THEN perform corrective actions per Section 8.1.6 before proceeding with sample analysis.

7.5 Analysis Procedures Instrumental Operation

7.5.1 Set the mass flow on the MFC to 40 mL/min (in the Set position) with the valve in the control position.

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7.5.2 Change the position of the mass flow from Set to Read and flush the system with UHP nitrogen until a steady reading is obtained on the mass flow controller.

7.5.3 Using the current (i.e., valid calibration date) calibration correction table provided by the S&CL (see example in Exhibit 1 MFC Calibration from S&CL (Example)), find the actual flow associated with the reading obtained from the mass flow controller while it was being flushed with UHP nitrogen.

7.5.4 Use the following equations to determine the correct number of counts (displayed on the Totalizer) corresponding to the volume that is to be injected.

$$C = m \times 40\, mL / \min$$

and

$$m = \frac{V}{a}$$

where: m = minutes,
a = actual reading in mL/min (as determined by S&CL),
V = volume you want to inject (mL),
C = counts displayed on the Totalizer.

7.5.5 Calculate the desired amount of sample or standard to be introduced to the trap.

7.5.6 Attach the stainless steel transfer line from the MFC to the sample inlet port on the Liquid Sample Controller.

7.5.7 Verify that the trap temperature is less than 30 °C.

7.5.8 Attach the sample canister to the Swagelok® fitting on the Sample Introduction Manifold (SIM).

7.5.9 With the two-way valve at Vent position, open the canister valve and flip the toggle switch to allow flow through the MFC. Flush for 10 counts.

7.5.10 Turn the two-way valve to Stop position.

7.5.11 Reset the Totalizer and turn the two-way valve to Trap position.

7.5.12 When the predetermined number of counts has been reached, turn the two-way valve to Vent and close the canister valve.

7.5.13 Remove the canister from the SIM.

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7.5.14 Attach the canister containing the internal standards to the Swagelok® fitting on the SIM and introduce 250 ng of the internal standards on to the trap (Steps 7.5.9 through 7.5.13).

7.5.15 Start the acquisition form on the Chemstation so the oven will cool.

7.5.16 Complete the acquisition form.

7.5.17 Step to Desorb Ready to begin data acquisition.

7.5.18 Rinse the MFC and Tekmar lines at the completion of the run with UHP nitrogen (4-5 minutes through the vent and 13-15 minutes of the bake time through the trap).

7.5.19 IF a sample is analyzed that has saturated the detector, or shows concentrations of analytes greater than 10 times the upper limit of the initial calibration,
THEN follow that analysis by analysis of an UHP nitrogen blank.

7.5.19.1 IF the blank analysis is free of contamination (≤3 × Program-Required MDL),
THEN proceed with analysis of samples.

7.5.19.2 IF the blank analysis is not free of contamination (>3 × Program-Required MDL),
THEN decontaminate the system AND repeat the blank analysis until the ≤3 × Program-Required MDL criteria is met.

7.5.20 IF any analyte is detected during the initial analysis of a sample in an amount exceeding the initial calibration range,
THEN either dilute and reanalyze the sample or reanalyze it using a reduced sample aliquot volume until the analytes in question fall within the calibration range.

7.5.20.1 IF an analyte is not detected in the diluted or reduced volume sample but was detected in the initial analysis,
THEN report the initial analysis results AND the diluted/reduced volume results.

7.5.20.2 Report the diluted or reduced volume sample with the suffix DL appended to the sample number.

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7.6 Data Evaluation

7.6.1 Target Analyte Qualitative Analysis

7.6.1.1 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). Standard reference mass spectra are obtained through analysis of calibration standards or spectra from the NIST library. Two criteria must be satisfied to verify identification.

- The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component in the continuing calibration.
- The sample component and the standard component mass spectrum must correspond.

7.6.2 Target Analyte Quantitative Analysis

7.6.2.1 Quantitate identified target analytes based on the integrated abundance from the Extracted Ion Chromatogram Profile (EICP) of the primary characteristic ion (see Table 4 Characteristic Ions for Volatile Organic Compounds).

7.6.2.2 Use internal standard quantitation (calculations section 9.4), with the internal standard assigned in Table 5 VOC Internal Standards with Corresponding Analytes Assigned for Quantitation.

7.6.3 Tentatively Identified Compounds (TICs)

7.6.3.1 Qualitative Identification

7.6.3.1.1 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 (see Table 6).*

7.6.3.1.2 Evaluate the unknowns 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% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20 %.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting 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 coeluting peaks.

7.6.3.1.3 Report TICs which meet the above criteria and which are Appendix VIII VOC compounds.

7.6.3.2 Estimate the concentrations for TICs, using the equation in section 9.4, with the following modifications:

- the areas A_X and A_{IS} are from the total ion chromatograms,
- the RRF for the compound is assumed to be one (1).
- the nearest internal standard free of interference is used.

7.7 Data Recording and Review by Data Generator

7.7.1 Data Recording

7.7.1.1 Document analysis run sequences in the GC/MS Use Logbook. At a minimum, logbook entries must contain the analysis date, lab sample ID, dilution factor, sample volume, and data file ID.

7.7.1.2 Record preparation of BFB solutions and gas standards in GC/MS Standard Preparation Logbook.

7.7.1.3 Maintain data files on the hard drive of the computer or on diskette until the original report and raw data records are appropriately stored.

7.7.2 Data Review

7.7.2.1 Review, initial, and date all raw data generated (Exhibits 2-5).

7.7.2.1.1 Mark with an "X" on the quant report those target analytes that are not to be reported as hits.

7.7.2.1.2 On the individual target compound spectra, note whether or not the compound is to be reported as a hit (e.g., "Yes" for report, "No" for Do Not Report).

7.7.2.1.3 IF a compound is not to be reported, THEN provide justification (e.g., "Below MDL" or "Wrong RT").

7.7.2.1.4 Mark each non-target compound (TIC) as “Report” or “Do Not Report” on the raw data (see step 7.6.3).

7.7.2.1.4.1 Provide justification for any TIC marked Do Not Report (e.g., “does not meet criteria”).

7.7.2.1.4.2 Identify the compound name to be used for reportable TICs.

7.7.2.2 Complete a Supplemental Data Input Sheet (Exhibit 6) for each analytical run sequence.

7.7.2.3 Complete a Data Generator Checklist (see example in Exhibit 7) for each analytical run sequence.

7.8 Data Reporting

7.8.1 Report sample results in units of ppmv to two significant figures.

7.8.2 Use rounding rules and assign data qualifying flags per the requirements of CCP-TP-188, *CCP Analytical Data Recording, Review and Reporting*.

7.8.3 Generate reporting forms for sample and QC results (see examples in Exhibits 8-17).

7.8.4 Assemble a batch data report in the following format:

- Data Report Cover page and Table of Contents
- Sample Identification Table/Analysis Request Form

- Sample Custody Documents and Sample Tag
- Analysis Results
  - Sample Results Forms
- Quality Control Measurements Results
  - Laboratory Duplicate Comparison Form
  - Laboratory Blank Summary Form
  - Laboratory Blank Results Form
  - Internal Standard Area Form
  - Laboratory Control Sample Results Form
- Calibration Results
  - BFB Tune Form
  - Initial Calibration Form
  - Continuing Calibration Forms
  - MDL Reporting Form
- Data Review Checklists

8. QUALITY CONTROL REQUIREMENTS

8.1 Calibration

8.1.1 An ICAL relating instrument response to nanograms 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.

8.1.2 An ICAL is valid if the %RSD of the RRFs for each target analyte is less than 35% (see Section 7.4.1). If this specification is not met, corrective action must be taken (i.e., identify and correct problem, and recalibrate).

8.1.3 After each ICAL, an LCS is analyzed to provide second-source verification of the ICAL.

8.1.4 Recovery of all analytes in the LCS must be between 70% and 130%. If this specification is not met, the LCS standard may be remade and reanalyzed to eliminate LCS standard preparation as the source of error. If the reanalysis of the LCS still does not meet specification, further corrective actions must be implemented.

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

8.1.6 A CCAL is valid if the %D between ICAL and CCAL RRFs is ≤ 30% for all target analytes and the RT of each analyte is within 30 seconds of the average ICAL retention time (see Section 7.4.2). If this specification is

not met, corrective action must be taken (e.g., identify and correct problem, recalibrate) before sample analysis can proceed.

8.2 Internal Standard Areas (ISAs)

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

8.2.2 The ISAs for each sample must be within 50 to 200% of the associated CCAL ISAs. If this specification is not met, the problem must be identified and corrected before sample analysis can proceed.

8.3 Laboratory Blank (LB)

8.3.1 One LB (usually 100 mL UHP nitrogen) must be analyzed daily after the CCAL but before sample analysis.

8.3.2 LB results are acceptable if the amount of any individual analyte in the blank is less than or equal to three times the Program-Required MDL (see Table 1). If this specification is not met, the source of the contamination must be identified and eliminated before sample analysis can proceed.

8.4 Laboratory Control Sample (LCS)

8.4.1 One LCS must be analyzed per analytical batch. The LCS standard must be second-source to the calibration standards.

8.4.2 LCS results are acceptable if the percent recovery of all analytes is 70%-130%. If the LCS results do not meet specification, the LCS may be remade and reanalyzed to eliminate LCS preparation as the source of error. If the reanalysis is still noncompliant, sample analyses must be stopped until the problem is identified and corrected. All associated samples in the analysis batch must then be reanalyzed.

8.5 Laboratory Duplicates

8.5.1 A field canister must be analyzed in duplicate at a frequency of one per analytical batch.

8.5.2 For samples having analyte concentrations greater than or equal to the respective PRQLs, laboratory duplicate RPD must be ≤ 25. If this specification is not met, a nonconformance report (NCR) must be initiated per CCP-QP-005, *CCP TRU Nonconforming item Reporting and Control.*

8.5.3 If none of the field samples in an analytical batch contain any of the target analytes, the LCS must be analyzed in duplicate for that batch. Specification limits for duplicate LCSs are the same as those for field sample laboratory duplicates.

8.6 Method Detection Limits (MDLs)

8.6.1 Method detection limits (MDLs) are expressed in nanograms for VOCs, and must be demonstrated to be less than or equal to the Program-Required MDLs (see Table 1).

8.6.2 MDLs are determined as described in CCP-TP-176, *CCP Determination of Method Detection Limits for Gas Analysis*. MDLs must be determined semiannually.

8.7 Precision and Accuracy (P&A) Demonstrations

8.7.1 P&A must be acceptably demonstrated by initially analyzing seven replicate method performance samples (see Section 5.2.5.4) and achieving precision of ≤ 25 % relative standard deviation (%RSD) and mean accuracy of 70% to 130%.

8.7.2 After the initial P&A performance is acceptable, acceptable performance must be demonstrated semiannually by analyzing at least four replicate method performance standards and evaluating those results against the initial P&A specifications.

9. CALCULATIONS

9.1 Relative Response Factors (RRFs)

$$RRF = \frac{A_X C_{IS}}{A_{IS} C_X}$$

where: A_x = area of characteristic ion for compound being measured,
A_{IS} = area of characteristic ion for the specific internal standard,
C_{IS} = concentration of the specific internal standard,
C_x = concentration of the compound being measured.

9.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{s}{\bar{x}} \times 100$$

where: %RSD = percent relative standard deviation,
$\bar{x}$ = mean of initial calibration RRFs for a compound,
s = standard deviation of initial calibration RRFs for a compound.

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

where: x_i = measured value, i^{th} replicate sample analysis,
n = number of replicate analyses.

9.3 Percent Difference (%D)

$$\%D = \frac{\left|RRF_1 - RRF_C\right|}{RRF_1} \times 100$$

where: RRF_I = average relative response factor from ICAL,
RRF_C = relative response factor from CCAL.

9.4 Internal Standard Quantitation

$$Concentration\,(ppmv) = \frac{A_x \times C_{IS} \times R \times T \times D}{A_{IS} \times RRF \times V_0 \times C \times MW}$$

where: A_x = area of the characteristic ion for compound of interest,

$$Nanograms\ of\ analyte\ (value\ on\ quant\ report) = \frac{A_x \times C_{IS}}{A_{IS} \times RRF}$$

C_{IS} = amount (nanograms) of the internal standard,
A_{IS} = area of the characteristic ion of the internal standard,
D = dilution factor (listed on bench sheet),
C = 1 (correction factor if needed),
MW= molecular weight (g/mol) of compound being measured,
RRF= average ICAL RRF for compound being measured,
RT = Universal Gas Constant times temperature,
V_O = volume (mL) of sample delivered to the analytical system.

9.5 Relative Percent Difference (RPD)

$$RPD = \frac{\left|C_S - C_D\right|}{\left(\frac{C_S + C_D}{2}\right)} \times 100$$

where: C_S = sample concentration (ppmv),
C_D = duplicate concentration (ppmv).

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9.6 LCS Percent Recovery (%R)

$$\%R = \frac{C_m}{C_t} \times 100$$

where: C_m = measured value (ng) of LCS,
C_t = true (known) value of LCS standard (ng).

10. RECORDS

Records generated during the performance of this procedure are maintained as QA records in accordance with CCP-QP-008, *CCP Records Management*. The records are the following:

QA/Nonpermanent

- ICAL
- MDL
- Precision and Accuracy (P&A) Documentation

The following records generated during the performance of this procedure will be compiled into the Data Report in accordance with CCP-TP-188, *CCP Analytical Data Recording, Review and Reporting.*

QA/Nonpermanent

- Copy of applicable pages of GC/MS Use Logbook
- Copy of applicable pages of GC/MS Standard Preparation Logbook
- Raw Data/Instrument Printouts

11. REFERENCES

11.1 U.S. Environmental Protection Agency (U.S. EPA), 1988a, Compendium Method TO-14, The Determination of Volatile Organic Compounds (VOC) in Ambient Air Using SUMMA Passivated Canister Sampling and Gas Chromatographic Analyses, May 1988, Quality Assurance Division, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina.

11.2 U.S. EPA, 1988b, Compendium Method TO-2, The Determination of Volatile Organic Compounds in Ambient Air by Carbon Molecular Sieve Absorption and Gas Chromatography/Mass Spectrometry (GC/MS), Quality Assurance Division, Environmental Monitoring Systems Laboratory, Research Park, North Carolina.

11.3 J. H. Myron Stephenson, F. Allen, and T. Slagle, 1990, EPA/Air and Waste Management Association-International Symposium on Measurement of Toxic and Related Air Pollutants.

11.4 LI-1354-07-IRC/ECL, “Environmental Chemistry Laboratory (ECL) Gas Analysis Project.”

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11.5 LWP-14620, "Chemical Hygiene Plan".

11.6 CCP-PO-001, *CCP Transuranic Waste Characterization Quality Assurance Project Plan.*

11.7 CCP-QP-002, *CCP Training and Qualification Plan.*

11.8 CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control.*

11.9 CCP-QP-008, *CCP Records Management*

11.10 CCP-TP-173, *CCP Analysis of Gas Samples for VOCs by GC/FID.*

11.11 CCP-TP-176, *CCP Determination of Method Detection Limits for Gas Analysis*

11.12 CCP-TP-178, *CCP SUMMA® Canister Cleaning.*

11.13 CCP-TP-179, *CCP Gas Transfer Manifold Systems and Sample Compositing.*

11.14 CCP-TP-188, *CCP Analytical Data Recording, Review, and Reporting*.

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Exhibit 1. MFC Calibration from S&CL (Example)

PROPERTY # 711205 DATE:13 May 1996

UUT IN SCCM	0.000	.100	.200	.300	.400	.500	.600	.700	.800	.900
20.00:	20.4669	20.5772	20.6875	20.7979	20.9082	21.0185	21.1288	21.2391	21.3494	21.4597
21.00:	21.5699	21.6802	21.7905	21.9008	22.0110	22.1213	22.2316	22.3418	22.4521	22.5623
22.00:	22.6725	22.7828	22.8930	23.0032	23.1135	23.2237	23.3339	23.4441	23.5543	23.5645
23.00:	23.7747	23.8849	23.9951	24.1053	24.2154	24.3256	24.4358	24.5459	24.6561	24.7663
24.00:	24.8764	24.9866	25.0967	25.2068	25.3170	25.4271	25.5372	25.6473	25.7574	25.8676
25.00:	25.9777	26.0878	26.1979	26.3079	26.4180	26.5281	26.6382	26.7483	26.8583	26.9684
26.00:	27.0785	27.1885	27.2986	27.4086	27.5187	27.6287	27.7387	27.8488	27.9588	28.0688
27.00:	28.1788	28.2888	28.3988	28.5088	28.6188	28.7288	28.8388	28.9488	29.0588	29.1688
28.00:	29.2787	29.3887	29.4987	29.6086	29.7186	29.8285	29.9385	30.0484	30.1583	30.2683
29.00:	30.3782	30.4881	30.5980	30.7079	30.8178	30.9278	31.0377	31.1475	31.2574	31.3673
30.00:	31.4772	31.5871	31.6969	31.8068	31.9167	32.0265	32.1364	32.2462	32.3561	32.4659
31.00:	32.5758	32.6856	32.7954	32.9052	33.0151	33.1249	33.2347	33.3445	33.4543	33.5641
32.00:	33.6739	33.7837	33.8934	34.0032	34.1130	34.2228	34.3325	34.4423	34.5520	34.6618
33.00:	34.7715	34.8813	34.9910	35.1007	35.2105	35.3202	35.4299	35.5396	35.6493	35.7591
34.00:	35.8688	35.9784	36.0881	36.1978	36.3075	36.4172	36.5269	36.6365	36.7462	36.8559
35.00:	36.9655	37.0752	37.1848	37.2945	37.4041	37.5137	37.6234	37.7330	37.8426	37.9522
36.00:	38.0618	38.1714	38.2810	38.3906	38.5002	38.6098	38.7194	38.8290	38.9386	39.0481
37.00:	39.1577	39.2673	39.3768	39.4864	39.5959	39.7055	39.8150	39.9245	40.0341	40.1436
38.00:	40.2531	40.3626	40.4721	40.5817	40.6912	40.8007	40.9102	41.0196	41.1291	41.2386
39.00:	41.3481	41.4576	41.5670	41.6765	41.7859	41.8954	42.0048	42.1143	42.2237	42.3332
40.00:	42.4426	42.5520	42.6615	42.7709	42.8803	42.9897	43.0991	43.2085	43.3179	43.4273
41.00:	43.5367	43.6461	43.7554	43.8648	43.9742	44.0835	44.1929	44.3023	44.4116	44.5210
42.00:	44.6303	44.7396	44.8490	44.9583	45.0676	45.1769	45.2863	45.3956	45.5049	45.6142
43.00:	45.7235	45.8328	45.9420	46.0513	46.1606	46.2699	46.3792	46.4884	46.5977	46.7069
44.00:	46.8162	46.9254	47.0347	47.1439	47.2532	47.3624	47.4716	47.5808	47.6900	47.7993
45.00:	47.9085	48.0177	48.1269	48.2361	48.3452	48.4544	48.5636	48.6728	48.7820	48.8911
46.00:	49.0003	49.1094	49.2186	49.3277	49.4369	49.5460	49.6552	49.7643	49.8734	49.9825
47.00:	50.0917	50.2008	50.3099	50.4190	50.5281	50.6372	50.7463	50.8554	50.9644	51.0735
48.00:	51.1826	51.2917	51.4007	51.5098	51.6188	51.7279	51.8369	51.9460	52.0550	52.1640
49.00:	52.2731	52.3821	52.4911	52.6001	52.7091	52.8181	52.9271	53.0361	53.1451	53.2541
50.00:	53.3631	53.4721	53.5810	53.6900	53.7990	53.9079	54.0169	54.1258	54.2348	54.3437
51.00:	54.4527	54.5616	54.6705	54.7795	54.8884	54.9973	55.1062	55.2151	55.3240	55.4329
52.00:	55.5418	55.6507	55.7596	55.8685	55.9773	56.0862	56.1951	56.3039	56.4128	56.5216
53.00:	56.6305	56.7393	56.8482	56.9570	57.0658	57.1747	57.2835	57.3923	57.5011	57.6099
54.00:	57.7187	57.8275	57.9363	58.0451	58.1539	58.2627	58.3714	58.4802	58.5890	58.6977
55.00:	58.8065	58.9153	59.0240	59.1327	59.2415	59.3502	59.4590	59.5677	59.6764	59.7851
56.00:	59.8938	60.0025	60.1112	60.2199	60.3286	60.4373	60.5460	60.6547	60.7634	60.8720
57.00:	60.9807	61.0894	61.1980	61.3067	61.4153	61.5240	61.6326	61.7413	61.8499	61.9585
58.00:	62.0672	62.1758	62.2844	62.3930	62.5016	62.6102	62.7188	62.8274	62.9360	63.0446
59.00:	63.1531	63.2617	63.3703	63.4788	63.5874	63.6960	63.8045	63.9131	64.0216	64.1301
60.00:	64.2387	64.3472	64.4557	64.5642	64.6728	64.7813	64.8898	64.9983	65.1068	65.2153
61.00:	65.3238	65.4322	65.5407	65.6492	65.7577	65.8661	65.9746	66.0831	66.1915	66.3000
62.00:	66.4084	66.5168	66.6253	66.7337	66.8421	66.9506	67.0590	67.1674	67.2758	67.3842
63.00:	67.4926	67.6010	67.7094	67.8178	67.9261	68.0345	68.1429	68.2513	68.3596	68.4680
64.00:	68.5763	68.6847	68.7930	68.9014	69.0097	69.1180	69.2264	69.3347	69.4430	69.5513
65.00:	69.6596	69.7679	69.8762	69.9845	70.0929	70.2011	70.3094	70.4177	70.5259	70.6342
66.00:	70.7425	70.8507	70.9590	71.0672	71.1755	71.2837	71.3920	71.5002	71.6084	71.7166
67.00:	71.8249	71.9331	72.0413	72.1495	72.2577	72.3659	72.4741	72.5823	72.6905	72.7986
68.00:	72.9068	73.0150	73.1231	73.2313	73.3395	73.4476	73.5558	73.6639	73.7720	73.8802
69.00:	73.9883	74.0964	74.2045	74.3127	74.4208	74.5289	74.6370	74.7451	74.8532	74.9613

STD FLOW SCCM

PROPERTY # 711205 DATE:13 May 1996

Exhibit 2. Quant Report (Example)

Quantitation Report

Data File : C:\INSTR_E\ECL02175\A02EG.D Vial: 1
Acq On : 2 Apr 2002 1:41 pm Operator:
Sample : 02087017C 3.0933X 100ML Inst : E
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Apr 2 14:12 2002 Quant Results File: WPED047.RES

Quant Method : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
Title : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
Last Update : Wed Jan 09 10:17:40 2002
Response via : Initial Calibration
DataAcq Meth : WIPP75

Internal Standards	R.T.	QIon	Response	Conc Units		Dev(Min)
1) Fluorobenzene	12.62	96	1992231	270.00 ng		0.00
19) Chlorobenzene-d5	19.31	117	1156096	260.00 ng		0.00

System Monitoring Compounds

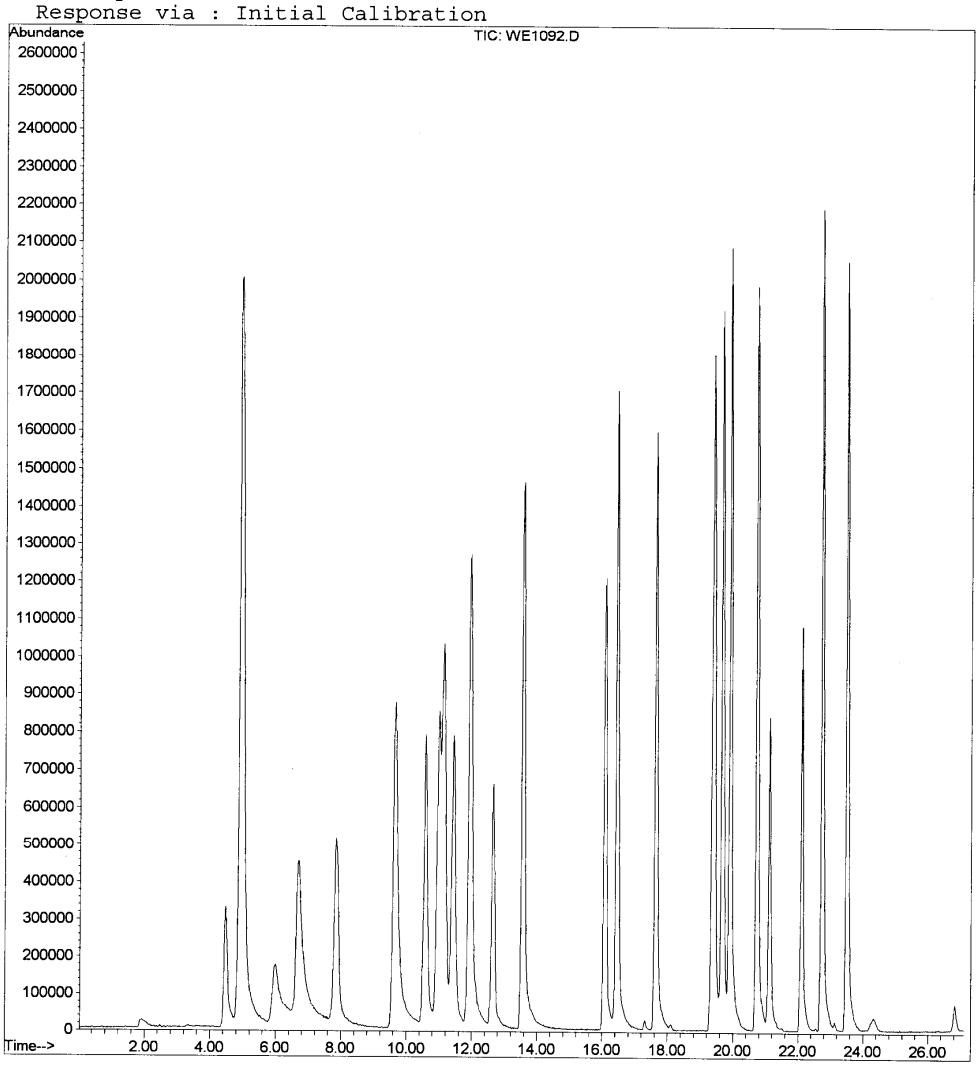
Target Compounds						Qvalue
3) Methylene Chloride	5.97	84	136041	68.67 ng		96
4) 1,1,2-Trichlorotrifluoroet	4.91	101	13747	~~X~~3.11 ng	#	62
5) 1,1 Dichloroethene	4.86	96	6176	~~X~~3.11 ng	#	80
6) 1,1-Dichloroethane	7.80	63	315175	72.02 ng		97
8) Chloroform	10.53	83	52234	11.45 ng		99
9) 1,2-Dichloroethane	11.93	62	16191	5.21 ng		92
10) 1,1,1-Trichloroethane	10.93	97	2557896	595.11 ng		100
11) Carbon Tetrachloride	11.37	117	16498	4.55 ng		89
12) Trichloroethene	13.52	130	489882	221.35 ng		99
13) Cyclohexane	11.29	56	113383	~~X~~30.24 ng	#	8
14) Benzene	11.87	78	91788	15.48 ng		98
15) Acetone	4.94	43	1796087m	635.61 ng		99
16) 2-Butanone	9.63	72	143958	122.12 ng		93
17) 1-Butanol	13.55	56	50983	42.02 ng		94
23) Toluene	16.41	92	1751854	451.69 ng		93
24) Chlorobenzene	19.37	112	10510	~~X~~2.54 ng		95
25) Ethylbenzene	19.64	106	5471	~~X~~2.70 ng	#	82
26) Xylene (meta & para)	19.88	106	20234	7.67 ng		95
27) Xylene (ortho)	20.70	106	7371	~~X~~3.09 ng		80
28) 1,3,5-Trimethylbenzene	22.70	105	11764	~~X~~2.03 ng		75
29) 1,2,4-Trimethylbenzene	23.45	105	15745	~~X~~2.83 ng		96
30) 4-Methyl-2-Pentanone	16.04	43	25570m	5.33 ng		77

(#) = qualifier out of range (m) = manual integration JJH 4-2-02
A02EG.D WPED047.M Tue Apr 02 14:12:17 2002 R 4-2-02 Page 1

Exhibit 3. Reconstructed Total Ion Chromatogram (Example)

Quantitation Report

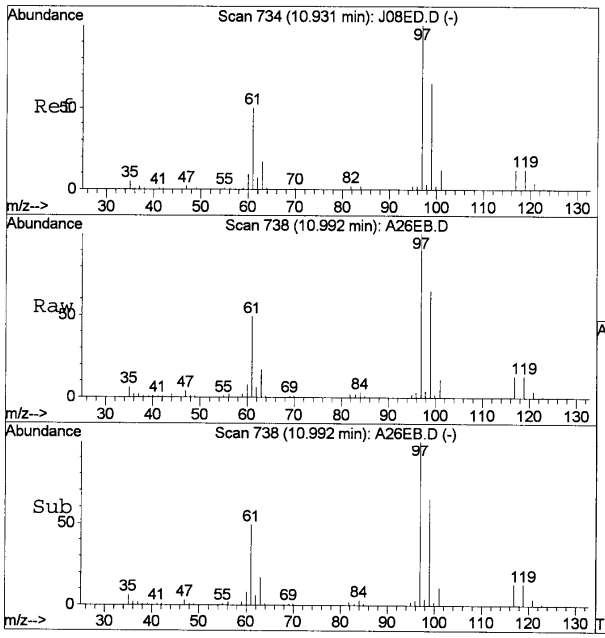
```
Data File : C:\INSTR_E\ECL02228\WE1092.D                Vial: 1
Acq On    : 26 Apr 2002   7:49 am                   Operator:
Sample    : ACMM-9930-CCAL   1X  100ML              Inst    : E
Misc      :                                         Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Apr 26  8:20 2002              Quant Results File: WPED047.RES

Method       : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
Title        : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
Last Update  : Wed Jan 09 10:17:40 2002
Response via : Initial Calibration
```



WE1092.D WPED047.M Fri Apr 26 08:20:36 2002 Page 2

4/28/02

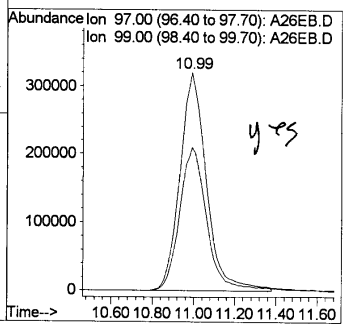
Exhibit 4. Target Analyte Mass Spectra (Example)

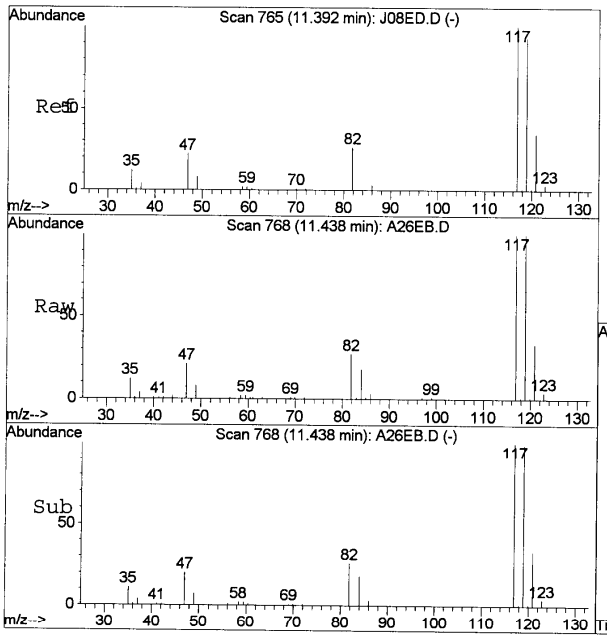


#10
1,1,1-Trichloroethane
Concen: 511.07 ng
RT: 10.99 min Scan# 738
Delta R.T. 0.06 min
Lab File: A26EB.D
Acq: 26 Apr 2002 1:22 pm

Tgt Ion: 97 Resp: 2865842

Ion	Ratio	Lower	Upper
97	100		
99	65.3	45.4	85.4

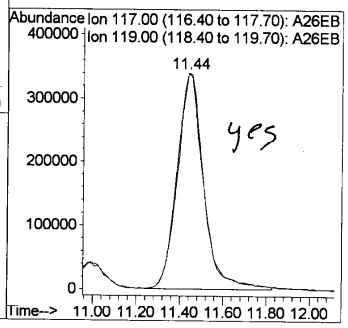




#11
Carbon Tetrachloride
Concen: 615.54 ng
RT: 11.44 min Scan# 768
Delta R.T. 0.05 min
Lab File: A26EB.D
Acq: 26 Apr 2002 1:22 pm

Tgt Ion:117 Resp: 2909078

Ion	Ratio	Lower	Upper
117	100		
119	99.7	73.1	113.1



A26EB.D WPED047.M Acq :26 Apr 2002 1:22 pm
Sample = LCSMEA26 35.0000X Misc = LCS30399SF21B

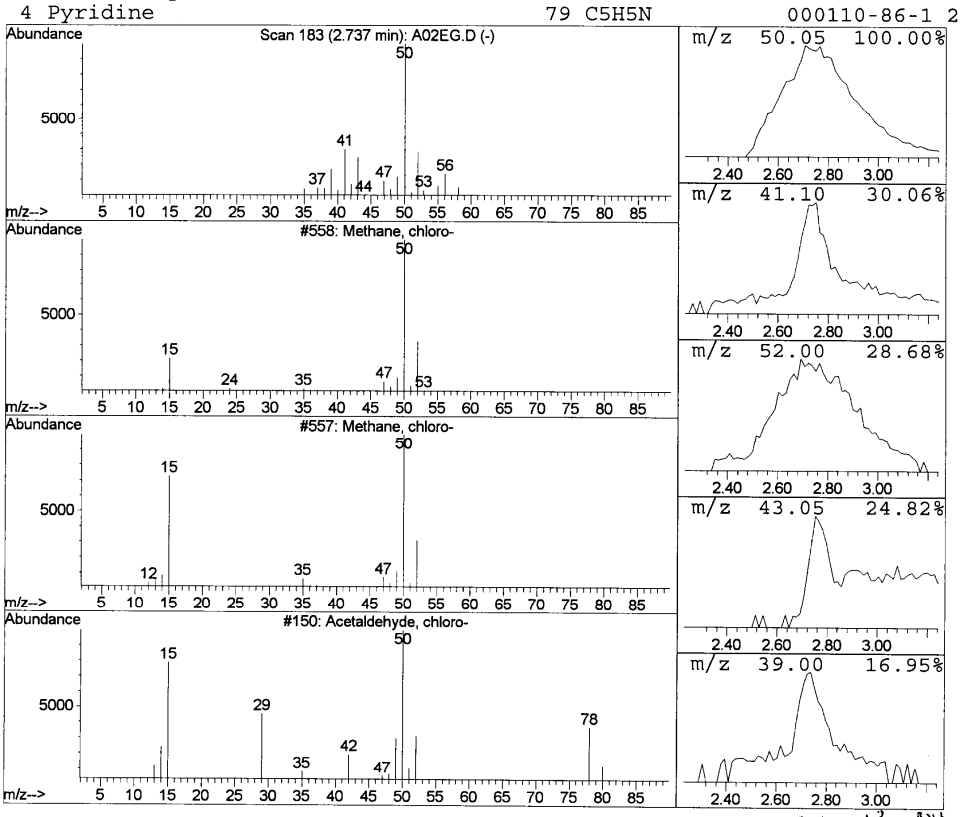
Exhibit 5. TIC Mass Spectra (Example)

Library Search Compound Report

```
  Data File : C:\INSTR_E\ECL02175\A02EG.D                    Vial: 1
  Acq On    :  2 Apr 2002   1:41 pm                     Operator:
  Sample    : 02087017C    3.0933X  100ML               Inst    : E
  Misc      :                                           Multiplr: 1.00
  MS Integration Params: rteint.p
```

Report

```
  Quant Method : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
  Title        : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
  Library      : C:\DATABASE\NISTAPX8.L
*********************************************************************
Peak Number  1  Methane, chloro-                Concentration Rank  1

  R.T.   EstConc          Area    Relative to ISTD   IS Area   R.T.
---------------------------------------------------------------------
  2.74   97.61 ng       1885550    Fluorobenzene     5215810  12.62

 Hit# of  5    Tentative ID                MW  MolForm       CAS#        Qual
---------------------------------------------------------------------------
 1 Methane, chloro-                        50  CH3Cl         000074-87-3 83
 2 Methane, chloro-                        50  CH3Cl         000074-87-3 64
 3 Acetaldehyde, chloro-                   78  C2H3ClO       000107-20-0 9
 4 Pyridine                                79  C5H5N         000110-86-1 2
```



DJH 4-2-02 BJH 4-2-02

4-2-02

A02EG.D WPED047.M Tue Apr 02 16:49:09 2002 Page 1

Exhibit 6. Supplemental Data Input Sheet (Example)

TWCP GC GAS ANALYSIS SUPPLEMENTAL DATA INPUT SHEET

DATA REPORT #:	**METHOD:**	**ANALYST:** **DATE:**
ANALYSIS BATCH #:	**INSTRUMENT:**	
CCAL #:	**REPORT TICs:**	
REPORT GROUP(s):		

FILE #	LABORATORY SAMPLE ID	DILUTION #1	DILUTION #2	DILUTION #3	DILUTION #4	FINAL DILUTION	VOLUME INJECTED mLs	TIME ANALYZED	DATE ANALYZED

FORM DATE 04/02/02

Exhibit 7. Data Generator Checklist (Example)

Environmental Chemistry Laboratory
Data Generator Checklist

ACMM-9930 (GC/MS VOCs)

Analytical Batch#:	Procedure Revision:
Analyst Signature:	Analysis Date:

Criteria	Yes	No	N/A	Comments
1. Samples analyzed in accordance with ACMM-9930 requirements.				
2. BFB tune analyzed at the beginning of the run (once every 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 meet 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 >0.5% 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 ACMM-9930.				
9. All logbook entries completed in accordance with MCP-2001.				
10. All raw data corrections made per MCP-2008 requirements [lined out, initialed, dated, and justified (as necessary)].				
11. All raw data evaluated, signed/initialed and dated in indelible black ink.				
12. Raw data reviewed for completeness and accuracy.				

Form Revision: 0
E:\ECL\checklists\ACMM-9930 Data Generator Rev 0.doc
Created on 04/03/02 12:10 PM

Exhibit 8. Analysis Data Sheet (Example)

GAS VOC ANALYSIS DATA SHEET

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Lab Sample ID:	02087017C34		Field Sample ID:	ID032702EI352
Analytical Batch:	ECL02175M		Sampling Batch:	PRF020145
Data Report:	ECL02175M		Method:	ACMM-9930
Date/Time Analyzed:	4/2/02	1341	Date Sampled:	3/27/02
Lab File ID:	A02EG		Date Received:	03/28/02
Instrument ID:	GCMS-E		Composite:	Yes 50 mL aliquot

Compound Name	Concentration (ppmv)	Qualifier
Acetone	21	J
Benzene	0.38	J
Bromoform	0.027	U
Butanol	1.1	J
Carbon tetrachloride	0.057	J
Chlorobenzene	0.091	U
Chloroform	0.18	J
Cyclohexane	0.11	U
1,1-Dichloroethane	1.4	J
1,2-Dichloroethane	0.10	J
1,1-Dichloroethylene	0.13	U
cis-1,2-Dichloroethylene	0.091	U
trans-1,2-Dichloroethylene	0.14	U
Ethyl benzene	0.12	U
Ethyl ether	0.11	U
Methyl ethyl ketone	3.3	J
Methyl isobutyl ketone	0.10	J
Methylene chloride	1.6	J
1,1,2,2-Tetrachloroethane	0.069	U
Tetrachloroethylene	0.048	U
Toluene	9.5	J
1,1,1-Trichloroethane	8.6	J
Trichloroethylene	3.2	J
1,1,2-Trichloro-1,2,2-trifluoroethane *	0.049	U
1,3,5-Trimethylbenzene	0.066	U
1,2,4-Trimethylbenzene	0.074	U
p/m-Xylene	0.14	J
o-Xylene	0.091	U

Form Rev 04-02

Exhibit 9. TIC Analysis Data Sheet (Example)

GAS VOC ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Lab Sample ID:	02087017C34		Field Sample ID:	ID032702EI352
Analytical Batch:	ECL02175M		Sampling Batch:	PRF020145
Data Report:	ECL02175M		Method:	ACMM-9930
Date/Time Analyzed:	4/2/02	1341	Date Sampled:	3/27/02
Lab File ID:	A02EG		Date Received:	03/28/02
Instrument ID:	GCMS-E		Composite:	Yes 50 mL aliquot

CAS Number	Compound Name	Concentration (ppmv)	Qualifier		Retention Time (Minutes)
000074-87-3	Chloromethane	3.8	J	N	2.74

Number of TICs Found: 1

Form Rev 04-02

Exhibit 10. Laboratory Duplicate Form (Example)

LABORATORY DUPLICATE FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Method:	ACMM-9930	Data Report:	ECL02175M
Instrument ID:	GCMS-E	Analytical Batch:	ECL02175M
Lab Sample ID:	02087009C15	Dup Lab Sample ID:	02087009C15D
Field Sample ID:	ID032602EI672	Dup Field Sample ID:	ID032602EI672D
Date/Time Analyzed:	4/1/02 1356	Date/Time Dup Analyzed:	4/1/02 1539
Lab File ID:	A01EC	Dup Lab File ID:	A01EE

COMPOUND	SAMPLE CONCENTRATION (PPMV)	FLAG(S)	DUPLICATE CONCENTRATION (PPMV)	FLAG(S)	RPD* (%)
Acetone	1.34341	J	1.15575	J	15.02
Benzene	0.26644	J	0.25561	J	4.15
Bromoform	0.028	U	0.028	U	NC
Butanol	0.70790	J	0.68932	J	2.66
Carbon tetrachloride	0.12777	J	0.11306	J	12.21
Chlorobenzene	0.093	U	0.093	U	NC
Chloroform	0.064	U	0.064	U	NC
Cyclohexane	0.49511	J	0.38571	J	24.84
1,1-Dichloroethane	7.60096	J	7.66360	J	0.82
1,2-Dichloroethane	0.068	U	0.068	U	NC
1,1-Dichloroethylene	0.21918	J	0.22222	J	1.38
cis-1,2-Dichloroethylene	0.093	U	0.093	U	NC
trans-1,2-Dichloroethylene	0.15	U	0.15	U	NC
Ethyl benzene	0.12	U	0.12	U	NC
Ethyl ether	0.12	U	0.12	U	NC
Methyl ethyl ketone	0.93361	J	0.86759	J	7.33
Methyl isobutyl ketone	0.092	U	0.09369	J	200.00
Methylene chloride	0.15	U	0.15	U	NC
1,1,2,2-Tetrachloroethane	0.070	BU	0.070	BU	NC
Tetrachloroethylene	0.049	U	0.049	U	NC
Toluene	15.86156		15.27253		3.78
1,1,1-Trichloroethane	16.16940		15.78673		2.40
Trichloroethylene	9.71008	J	9.14798	J	5.98
1,1,2-Trichloro-1,2,2-trifluoroethane	0.050	U	0.050	U	NC
1,3,5-Trimethylbenzene	0.067	U	0.067	U	NC
1,2,4-Trimethylbenzene	0.075	U	0.075	U	NC
p/m-Xylene	0.26546	J	0.25294	J	5.21
o-Xylene	0.09487	J	0.09358	J	1.38

NC = NOT CALCULABLE
* Specification limit <= 25% for analytes present in the sample and duplicate at concentrations greater than or equal to the PRQL

Form Rev 04-02

Exhibit 11. Laboratory Blank Summary Form (Example)

LABORATORY BLANK SUMMARY FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD:	ACMM-9930	ANALYTICAL BATCH:	ECL02175M
INSTRUMENT:	GCMS-E	DATA REPORT:	ECL02175M
LAB SAMPLE ID:	LBMEM31.	FIELD SAMPLE ID:	LB04098M27
LAB FILE ID:	M31EA		
DATE/TIME ANALYZED:	3/31/02 0827		

THIS LABORATORY BLANK APPLIES TO THE FOLLOWING SAMPLES:

FIELD SAMPLE ID	LAB SAMPLE ID	DATE/TIME ANALYZED
LCS30399SF21A	LCSMEM31	3/31/02 1456
ID032602EI721	02087005	3/31/02 1538
ID032602EI370	02087006	3/31/02 1651
ID032602EIC02	02087007	3/31/02 1730
ID032602EI601	02087008C15	3/31/02 1813
ID032602EI278	02087008C25	3/31/02 1813
ID032602EIC76	02087008C35	3/31/02 1813
ID032602EI012	02087008C45	3/31/02 1813
ID032602EI565	02087008C55	3/31/02 1813

Form Rev 04-02

Exhibit 12. Internal Standard Area Summary Form (Example)

8A
VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name : Environmental Chemistry Contract : INEEL
Project : WIPP Site : Location : Group :
Lab File ID (Standard): WE1065.D Date Analyzed : 31 Mar 2002
Instrument ID : E Time Analyzed : 7:47 am
GC Column : DB-624 ID : 0.53 Heated Purge (Y:N) : N

	IS1 Area	#	RT	#	IS2 Area	#	RT	#	IS3 Area	#	RT	#
12 HOUR STD	1995633		12.62		1218662		19.31		0		0.00	
UPPER LIMIT	3991266		13.12		2437324		19.81		0		0.00	
LOWER LIMIT	997817		12.12		609331		18.81		0		0.00	
File ID Sample												
M31EA LBMEM31	2276155		12.61		1347068		19.32		0		0.00	
M31EG LCSMEM31	2517371		12.63		1585850		19.32		0		0.00	
M31EH 02087005	2558520		12.63		1550905		19.32		0		0.00	
M31EI 02087006	2323184		12.63		1434494		19.33		0		0.00	
M31EJ 02087007	2357625		12.63		1411849		19.34		0		0.00	
M31EK 02087008C	2586806		12.63		1548254		19.33		0		0.00	

IS1 = Fluorobenzene
IS2 = Chlorobenzene-d5
IS3 =
AREA UPPER LIMIT = 200% of internal standard area
AREA LOWER LIMIT = 50% of internal standard area
RT UPPER LIMIT = 0.5 minutes of internal standard RT
RT LOWER LIMIT = -0.5 minutes of internal standard RT

Column to be used to flag values outside QC limit with an asterisk
* Values outside of contract required QC limits

Page 1 FORM VIII VOA 3-90

Exhibit 13. Laboratory Control Sample Results Form (Example)

LABORATORY CONTROL SAMPLE FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Method: ACMM-9930
Instrument ID: GCMS-E

Data Report: ECL02175M
Analytical Batch: ECL02175M

Lab Sample ID: LCSMEM31
Field Sample ID: LCS30399SF21A
Date/Time Analyzed: 3/31/02 1456
Lab File ID: M31EG

COMPOUND	FOUND CONCENTRATION (PPMV)	KNOWN CONCENTRATION (PPMV)	Recovery* (%)
Acetone	118.0613	99.90	118.18
Carbon tetrachloride	86.1988	99.70	86.46
Chloroform	87.2297	100.20	87.06
Cyclohexane	94.8334	100.00	94.83
Methylene chloride	94.5517	99.70	94.84
Toluene	94.8112	99.70	95.10
1,1,1-Trichloroethane	84.5689	99.80	84.74
Trichloroethylene	94.9277	99.60	95.31
1,1,2-Trichloro-1,2,2-trifluoroethane	85.1940	99.80	85.36

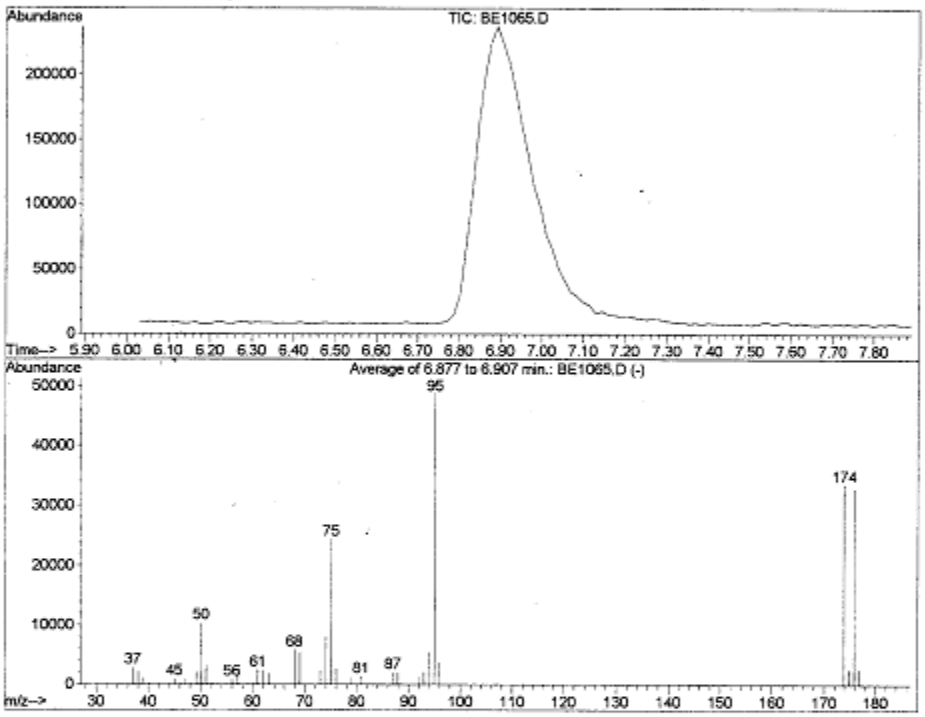
* Recovery specification limits 70 - 130%

Form Rev 04-02

Exhibit 14. BFB Tune Form (Example)

BFB Tune Evaluation

```
Data File : C:\INSTR_E\ECL02163\BE1065.D                    Vial: 1
Acq On    : 31 Mar 2002   6:33 am                       Operator: J. Wilson
Sample    : 50ng BFB                                    Inst    : E
Misc      : N/A                                         Multiplr: 1.00
MS Integration Params: RTEINT.P
Method    : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
Title     : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
```



Peak Apex is scan: 59 (6.89 min)
Average of 3 scans: 58,59,60 minus background scan 39 (6.59 min)

Target Mass	Rel. to Mass	Lower Limit,%	Upper Limit,%	Rel. Abn,%	Raw Abn	Result Pass/Fail
50	95	15	40	21.0	10302	PASS
75	95	30	60	49.6	24312	PASS
95	95	100	100	100.0	48992	PASS
96	95	5	9	7.6	3710	PASS
173	174	0	2	0.0	0	PASS
174	95	50	100	68.4	33488	PASS
175	174	5	9	7.5	2498	PASS
176	174	95	101	97.7	32731	PASS
177	176	5	9	7.9	2587	PASS

BE1065.D WPED047.M Sun Mar 31 07:34:13 2002

JW 3/31/02

Exhibit 15. ICAL Form (Example)

Response Factor Report E

Method : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
Title : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
Last Update : Wed Jan 09 10:17:40 2002
Response via : Initial Calibration

Calibration Files
75 =J08EF.D 250 =J08EE.D 500 =J08ED.D
750 =J08EC.D 1000 =J08EB.D 1250 =J08EA.D

		Compound	75	250	500	750	1000	1250	Avg	%RSD
1)	I	Fluorobenzene	----------ISTD----------							
2)	T	Ethyl Ether	0.202	0.211	0.220	0.225	0.223	0.223	0.217	4.14
3)	T	Methylene Chloride	0.264	0.284	0.273	0.272	0.262	0.255	0.268	3.87
4)	T	1,1,2-Trichlorotrif	0.597	0.603	0.601	0.601	0.597	0.591	0.598	0.69
5)	T	1,1 Dichloroethene	0.287	0.307	0.305	0.264	0.225	0.228	0.269	13.59
6)	T	1,1-Dichloroethane	0.583	0.627	0.632	0.641	0.543	0.533	0.593	7.99
7)	T	cis-1,2-Dichloroeth	0.309	0.326	0.325	0.337	0.330	0.317	0.324	3.08
8)	T	Chloroform	0.578	0.621	0.632	0.640	0.626	0.612	0.618	3.52
9)	T	1,2-Dichloroethane	0.407	0.421	0.423	0.432	0.426	0.418	0.421	2.01
10)	T	1,1,1-Trichloroetha	0.541	0.580	0.580	0.596	0.604	0.594	0.583	3.85
11)	T	Carbon Tetrachlorid	0.460	0.489	0.487	0.507	0.507	0.496	0.491	3.53
12)	T	Trichloroethene	0.282	0.301	0.297	0.302	0.311	0.306	0.300	3.29
13)	T	Cyclohexane	0.505	0.517	0.514	0.513	0.512	0.489	0.508	2.04
14)	T	Benzene	0.823	0.819	0.792	0.801	0.803	0.785	0.804	1.84
15)	T	Acetone	0.421	0.398	0.392	0.380	0.354	0.352	0.383	6.94
16)	T	2-Butanone	0.140	0.159	0.163	0.168	0.168	0.160	0.160	6.46
17)	T	1-Butanol	0.127	0.153	0.168	0.177	0.184	0.178	0.164	12.93
18)		trans-1,2-Dichloroe	0.299	0.335	0.337	0.329	0.326	0.314	0.323	4.44
19)	I	Chlorobenzene-d5	----------ISTD----------							
20)	T	Bromoform	0.340	0.390	0.416	0.457	0.462	0.481	0.424	12.51
21)	T	Tetrachloroethene	0.415	0.451	0.436	0.440	0.429	0.431	0.434	2.73
22)	T	1,1,2,2-Tetrachloro	0.602	0.586	0.585	0.615	0.591	0.587	0.594	1.98
23)	T	Toluene	0.856	0.919	0.875	0.865	0.858	0.861	0.872	2.72
24)	T	Chlorobenzene	0.908	0.919	0.937	0.964	0.933	0.924	0.931	2.08
25)	T	Ethylbenzene	0.418	0.458	0.457	0.484	0.468	0.453	0.456	4.79
26)	T	Xylene (meta & para	0.567	0.571	0.605	0.629	0.592	0.595	0.593	3.84
27)	T	Xylene (ortho)	0.480	0.522	0.537	0.586	0.549	0.548	0.537	6.53
28)	T	1,3,5-Trimethylbenz	1.191	1.236	1.294	1.393	1.367	1.355	1.306	6.10
29)	T	1,2,4-Trimethylbenz	1.142	1.191	1.210	1.325	1.315	1.314	1.250	6.29
30)	T	4-Methyl-2-Pentanon	1.045	1.121	1.101	1.077	1.069	1.055	1.078	2.62

L = Linear LO = Linear+Origin Q = Quad QO = Quad+Origin R = Corr. Coef
(#) = Out of Range
WPED047.M Wed Jan 09 10:33:40 2002 SJM 1-9-02 Page 1

Exhibit 16. CCAL Form (Example)

Evaluate Continuing Calibration Report

Data File : C:\INSTR_E\ECL02175\WE1066.D Vial: 1
Acq On : 1 Apr 2002 8:59 am Operator: J. Wilson
Sample : ACMM-9930-CCAL 100ml Inst : E
Misc : N/A Multiplr: 1.00
MS Integration Params: RTEINT.P

Method : C:\HPCHEM\1\METHODS\WPED047.M (RTE Integrator)
Title : GC/MS-E Wipp Initial Calibration #047 ACMM-9930
Last Update : Wed Jan 09 10:17:40 2002
Response via : Multiple Level Calibration

Min. RRF : 0.000 Min. Rel. Area : 50% Max. R.T. Dev 0.50min
Max. RRF Dev : 30% Max. Rel. Area : 200%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	Fluorobenzene	1.000	1.000	0.0	101	0.03
2	T	Ethyl Ether	0.217	0.204	6.0	94	0.02
3	T	Methylene Chloride	0.268	0.274	-2.2	102	0.03
4	T	1,1,2-Trichlorotrifluoroeth	0.598	0.590	1.3	99	0.02
5	T	1,1 Dichloroethene	0.269	0.305	-13.4	101	0.02
6	T	1,1-Dichloroethane	0.593	0.600	-1.2	96	0.03
7	T	cis-1,2-Dichloroethene	0.324	0.313	3.4	98	0.03
8	T	Chloroform	0.618	0.605	2.1	97	0.02
9	T	1,2-Dichloroethane	0.421	0.387	8.1	93	0.03
10	T	1,1,1-Trichloroethane	0.583	0.541	7.2	95	0.06
11	T	Carbon Tetrachloride	0.491	0.447	9.0	93	0.03
12	T	Trichloroethene	0.300	0.294	2.0	100	0.02
13	T	Cyclohexane	0.508	0.502	1.2	99	0.03
14	T	Benzene	0.804	0.804	0.0	103	0.02
15	T	Acetone	0.383	0.390	-1.8	101	0.02
16	T	2-Butanone	0.160	0.151	5.6	94	0.03
17	T	1-Butanol	0.164	0.161	1.8	97	0.03
18		trans-1,2-Dichloroethene	0.323	0.335	-3.7	101	0.03
19	I	Chlorobenzene-d5	1.000	1.000	0.0	96	0.03
20	T	Bromoform	0.424	0.408	3.8	94	0.03
21	T	Tetrachloroethene	0.434	0.443	-2.1	97	0.03
22	T	1,1,2,2-Tetrachloroethane	0.594	0.637	-7.2	104	0.03
23	T	Toluene	0.872	0.936	-7.3	102	0.03
24	T	Chlorobenzene	0.931	0.977	-4.9	100	0.03
25	T	Ethylbenzene	0.456	0.473	-3.7	99	0.03
26	T	Xylene (meta & para)	0.593	0.632	-6.6	100	0.03
27	T	Xylene (ortho)	0.537	0.569	-6.0	101	0.02
28	T	1,3,5-Trimethylbenzene	1.306	1.359	-4.1	100	0.02
29	T	1,2,4-Trimethylbenzene	1.250	1.293	-3.4	102	0.03
30	T	4-Methyl-2-Pentanone	1.078	1.123	-4.2	97	0.03

4/1/02

(#) = Out of Range SPCC's out = 0 CCC's out = 0
WE1066.D WPED047.M Mon Apr 01 11:26:53 2002 Page 1

Exhibit 17. MDL Reporting Form (Example)

MDL REPORTING FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD: ACMM-9930
Instrument ID: GCMS-E MDL Determination Date: 1/9/02

ANALYTE	MOLECULAR WEIGHT	MDL (ng)	PRQL (ppmv)
Acetone	58.08	6.3	100
Benzene	78.12	3.4	10
Bromoform	252.75	3.6	10
Butanol	74.12	5.1	100
Carbon tetrachloride	153.82	2.9	10
Chlorobenzene	112.56	5.3	10
Chloroform	119.38	3.9	10
Cyclohexane	84.16	4.9	10
1,1-Dichloroethane	98.94	5.1	10
1,2-Dichloroethane	98.94	3.4	10
1,1-Dichloroethylene	96.94	6.3	10
cis-1,2-Dichloroethylene	96.94	4.6	10
trans-1,2-Dichloroethylene	96.94	7.2	10
Ethyl benzene	106.17	6.5	10
Ethyl ether	74.12	4.4	10
Methyl ethyl ketone	72.11	6.6	100
Methyl isobutyl ketone	100.16	4.7	100
Methylene chloride	84.93	6.3	10
1,1,2,2-Tetrachloroethane	167.85	6.0	10
Tetrachloroethylene	165.83	4.1	10
Toluene	92.15	4.6	10
1,1,1-Trichloroethane	133.41	3.3	10
Trichloroethylene	131.39	4.3	10
1,1,2-Trichloro-1,2,2-trifluoroethane	187.38	4.8	10
1,3,5-Trimethylbenzene	120.2	4.1	10
1,2,4-Trimethylbenzene	120.2	4.6	10
p/m-Xylene	106.17	4.3	10
o-Xylene	106.17	5.0	10

Form Rev 01-02

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Table 1. Target Analyte list for CCP-TP-175

ANALYTE	MOLECULAR WEIGHT	Required MDL[a] (ng)	PRQL (ppmv)
Acetone[c]	58.08	150	100
Benzene	78.12	10	10
Bromoform	252.75	10	10
Butanol[c]	74.12	150	100
Carbon disulfide[d]	76.14	10	10
Carbon tetrachloride	153.82	10	10
Chlorobenzene	112.56	10	10
Chloroform	119.38	10	10
Chloromethane[d]	50.49	10	10
Cyclohexane[b]	84.16	10	10
1,1-Dichloroethane	98.94	10	10
1,2-Dichloroethane	98.94	10	10
1,1-Dichloroethylene	96.94	10	10
cis-1,2-Dichloroethylene[b]	96.94	10	10
trans-1,2-Dichloroethylene	96.94	10	10
1,2-Dichloropropane[d]	112.99	10	10
Ethyl benzene	106.17	10	10
Ethyl ether	74.12	10	10
Methanol[c]	32.04	150	100
Methyl ethyl ketone[c]	72.11	150	100
Methyl isobutyl ketone[c]	100.16	150	100
Methylene chloride	84.93	10	10
1,1,2,2-Tetrachloroethane	167.85	10	10
Tetrachloroethylene	165.83	10	10
Toluene	92.15	10	10
1,1,1-Trichloroethane	133.41	10	10
Trichloroethylene	131.39	10	10
Trichlorofluoromethane[d]	137.37	10	10
1,1,2-Trichloro-1,2,2-trifluoroethane	187.38	10	10
1,3,5-Trimethylbenzene[b]	120.20	10	10
1,2,4-Trimethylbenzene[b]	120.20	10	10
p/m-Xylene	106.17	10	10
o-Xylene	106.17	10	10

a Values based on delivery of 10 mL to the analytical system
b Analysis of these analytes not required for WIPP WAP compliance.
c These analytes may be analyzed via GC/FID (CCP-TP-173).
d Site-specific analyte; analysis may not be required for samples from all TRU Waste Generator/Storage Sites.

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Table 2. BFB Key Ion Abundance Criteria

BFB KEY ION ABUNDANCE CRITERIA	
MASS	Ion Abundance Criteria
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. Calibration Standards

Calibration* Point (ng)	Concentration of Standard (ng/mL)	Calibration Standard Aliquot (mL)	Internal Standard Aliquot (mL)
75	2.5	30	100
250	5	50	100
400	5	80	100
500	5	100	100
600	5	120	100
750	5	150	100
1000	5	200	100
1250	5	250	100

* If calibrating for methanol the six calibration points may differ from those listed in this table.

Table 4. Characteristic Ions for Volatile Organic Compounds

VOC Analytes	Primary Ion	Secondary Ion(s)
Acetone	43	58
Benzene	78	52, 77
Bromoform	173	171, 175, 252
Butanol	56	41
Carbon disulfide	76	44
Carbon tetrachloride	117	119, 121
Chlorobenzene	112	114, 77
Chloroform	83	85
Chloromethane	50	52
Cyclohexane	56	84
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	63	65
1,1-Dichloroethylene	96	98, 61
cis-1,2-Dichloroethylene	96	61, 98
trans-1,2-Dichloroethylene	96	61, 98
1,2-Dichloropropane	63	62, 76
Ethyl benzene	106	91
Ethyl ether	59	74, 45
Methanol	31	-
Methyl ethyl ketone	72	57, 43
Methyl isobutyl ketone	43	58, 100
Methylene chloride	84	49, 51, 86
1,1,2,2-Tetrachloroethane	83	85, 131, 166
Tetrachloroethylene	164	129, 131, 166
Toluene	92	91, 65
1,1,1-Trichloroethane	97	99, 117
Trichloroethylene	130	95, 97, 132
Trichlorofluoromethane	101	103
1,1,2-Trichloro-1,2,2-trifluoroethane	101	85
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
m-Xylene	106	91
o-Xylene	106	91
p-Xylene	106	91
Chlorobenzene-d_5 (IS)	117	82, 119
Fluorobenzene (IS)	96	77

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Table 5. VOC Internal Standards with Corresponding Analytes Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d_5
Acetone	Bromoform
Benzene	Chlorobenzene
Butanol	Ethyl benzene
Carbon disulfide	Methyl isobutyl ketone
Carbon tetrachloride	1,1,2,2-Tetrachloroethane
Chloroform	Tetrachloroethylene
Chloromethane	Toluene
Cyclohexane	1,3,5-Trimethylbenzene
1,1-Dichloroethane	1,2,4-Trimethylbenzene
1,2-Dichloroethane	m-Xylene
1,1-Dichloroethylene	o-Xylene
cis-1,2-Dichloroethylene	p-Xylene
trans-1,2-Dichloroethylene	
1,2-Dichloropropane	
Ethyl ether	
Methanol	
Methyl ethyl ketone	
Methylene chloride	
1,1,1-Trichloroethane	
Trichloroethylene	
Trichlorofluoromethane	
1,1,2-Trichloro-1,2,2-trifluoroethane	

Table 6. Appendix VIII VOC Compounds

Compound Name	CAS #	Compound Name	CAS #	Compound Name	CAS #
Acetonitrile	75-05-8	1,3-Dichloropropene	542-75-6	Nickel carbonyl	13463-39-3
Acetophenone	98-86-2	1,2,3,4-Diepoxybutane	1464-53-5	Nitrobenzene	98-95-3
Acetyl chloride	75-36-5	Diethylarsine	692-42-2	Nitrogen dioxide	10102-44-0
Acrolein	107-02-8	1,4-Diethyleneoxide	123-91-1	Nitrogen mustard	51-75-2
Acrylonitrile	107-13-1	N, N'-Diethylhydrazine	1615-80-1	2-Nitropropane	79-46-9
Allyl alcohol	107-18-6	Diisopropylfluorophosphate (DFP)	55-91-4	N-Nitrosodiethylamine	55-18-5
Allyl chloride	107-05-1	Dimethylcarbamoyl chloride	79-44-7	N-Nitrosodimethylamine	62-75-9
Aniline	62-53-3	1,1-Dimethylhydrazine	57-14-7	N-Nitrosomethylethylamine	10595-95-6
Benzal chloride	98-87-3	1,2-Dimethylhydrazine	540-73-8	N-Nitroso-N-methylurethane	615-53-2
Benzene*	71-43-2	2,4-Dimethylphenol	106-67-9	N-Nitrosomethylvinylamine	4549-40-0
Benzotrichloride	98-07-7	Dimethyl sulfate	77-78-1	N-Nitrosopiperidine	100-75-4
Benzyl chloride	100-44-7	Di-n-propylnitrosamine	621-64-7	Osmium tetroxide	20816-12-0
Bromoacetone	598-31-2	Epichlorohydrin	106-89-8	Paraldehyde	123-63-7
Bromoform*	75-25-2	Ethyl carbamate (urethane)	51-79-6	Pentachloroethane	76-01-7
Carbon disulfide*	75-15-0	Ethyl cyanide	107-12-0	Phenol	108-95-2
Carbon oxyfluoride	353-50-4	Ethylene dibromide	106-93-4	Phosgene	75-44-5
Carbon tetrachloride*	56-23-5	Ethylene dichloride	107-06-2	Phosphine	7803-51-2
Chloral	76-87-6	Ethylene glycol monoethyl ether	110-80-5	2-Picoline	109-06-8
Chloroacetaldehyde	107-20-0	Ethyleneimine	151-56-74	Potassium dimethyldithiocarbamate	128-03-0
Chlorobenzene*	108-90-7	Ethylene oxide	75-21-8	Potassium n-methyldithiocarbamate	137-41-7
p-Chloro-m-cresol	59-50-7	Ethylidene dichloride	75-34-3	1,3-Propane sultone	1120-71-4
2-Chloroethyl vinyl ether	110-75-8	Ethyl methacrylate	97-63-2	n-Propylamine	107-10-8
Chloroform*	67-66-3	Ethyl methanesulfonate	62-50-0	Propargyl alcohol	107-19-7
Chloromethyl methyl ether	107-30-2	Fluorine	7782-41-4	Propylene dichloride	78-87-5
o-Chlorophenol	95-57-8	Fluoroacetamide	640-19-7	1,2-Propylenimine	75-55-8
Chloroprene	126-99-8	Formaldehyde	50-00-0	Pryidine	110-86-1
3-Chloropropionitrile	542-76-7	Formic acid	64-18-6	Selenourea	630-10-4
Coal tar creosote	8007-45-2	Glycidylaldehyde	765-34-4	1,2,4,5-Tetrachlorobenzene	95-94-3
Cresol (Cresylic acid)	1319-77-3	Hexachlorobutadiene	87-68-3	Tetrachloroethane, NOS	25322-20-7
Crotonaldehyde	1470-30-6	Hexachloroethane	67-72-1	1,1,1,2-Tetrachloroethane	630-20-6
Cyanogen	460-19-5	Hexachloropropene	1888-71-7	1,1,2,2-Tetrachloroethane*	79-34-5
Cyanogen bromide	506-68-3	Hydrazine	302-01-2	Tetrachloroethylene*	127-18-4
Cyanogen chloride	506-77-4	Hyrogen cyanide	74-90-8	Tetraethyl lead	78-00-2
1,2-Dibromo-3-chloropropane	96-12-8	Hydrogen fluoride	7664-39-3	Tetranitromethane	509-14-8
o-Dichlorobenzene	95-50-1	Hydrogen sulfide	7783-06-4	Thallium(I) acetate	563-68-8
m-Dichlorobenzene	541-73-1	Isobutyl alchohol	78-83-1	Thallium(I) carbonate	6533-73-9
p-Dichlorobenzene	106-46-7	Maleic anhydride	108-31-6	Thioacetamide	62-55-5
Dichlorobenzene, NOS 1	25321-22-6	Malononitrile	109-77-3	Thiomethanol	74-93-1
1-4-Dichloro-2-butene	764-41-0	Methacrylonitrile	126-97-7	Thiophenol	108-95-8
Dichlorodifluoromethane	75-71-8	Methyl bromide	74-83-9	Thiosemicarbazide	79-19-6
Dichloroethylene, NOS 1	25323-30-2	Methyl chloride*	74-87-3	Toluene*	108-88-3
1,1-Dichloroethylene*	75-35-4	Methyl chlorocarbonate	79-22-1	o-Toluidine	95-53-4
1,2-Dichloroethylene*	156-60-5	Methyl chloroform	71-55-6	o-Toluidine hydrochloride	636-21-5
Dichloroethyl ether	111-44-4	Methylene bromide	74-95-3	p-Toluidine	106-49-0
Dichloroisopropyl ether	108-60-1	Methylene chloride*	75-09-2	1,2,4-Trichlorobenzene	120-82-1
Dichloromethoxy ethane	111-91-1	Methyl ethyl ketone (MIBK)*	78-93-3	1,1,2-Trichloroethane	79-00-5
Dichloromethyl ether	542-88-1	Methyl hydrazine	60-34-4	Trichloroethylene*	79-01-6
2,4-Dichlorophenol	120-83-2	Methyl iodide	74-88-4	Trichloromethanethiol	75-70-7
Dichlorophenylarsine	696-28-6	Methyl isocyanate	624-83-9	Trichloromonofluoromethane*	75-69-4
Dichloropropane, NOS 1	26638-19-7	2-Methylactonitrile	75-86-5	Trichloropropane, NOS 1	25735-29-9
Dichloropropanol, NOS 1	26545-73-3	Methyl methacrylate	80-62-6	1,2,3-Trichloropropane	96-18-4
Dichloropropene, NOS 1	26952-23-8	Methyl methanesulfonate	66-27-3	Triethylamine	121-44-8
				Vinyl chloride	75-01-4

* analyzed as target analytes