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CCP-TP-173

Revision 1

CCP
Analysis of Gas Samples for VOCs by GC/FID

EFFECTIVE DATE: 09/30/2009

Larry Porter

PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	05/03/2007	Initial Issue.
1	Xx/xx/2009	The target compound recoveries for Initial Calibration (ICAL) was set more conservative than the Permit requirement, therefore changing to match the Permit. Also updating procedure references.

1. ABSTRACT

The Environmental Chemistry Laboratory (ECL) uses this Central Characterization Project (CCP) procedure to provide instructions for the analysis of volatile organic compounds (VOCs) in headspace gas samples contained in SUMMA® canisters. This procedure is a modification of SW-846 Method 8015B, *Nonhalogenated Organics Using GC/FID.*

This method provides for the quantitative determination of the five specific volatile alcohols and ketones listed in Table 1. Normally, methanol is the only analyte determined and reported by this method; the other compounds are routinely analyzed via CCP-TP-175, *CCP Analysis of Gas Samples for VOCs by GC/MS.*

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 method is specifically designed for gas samples contained in SUMMA® canisters. Samples in other containers such as Tedlar® bags must first be transferred to SUMMA® canisters and pressurized sufficiently for the method.

2.2 This method may be used to screen gas samples for some or all of the other VOCs listed in Table 2 prior to quantitative analysis by gas chromatography/mass spectroscopy (GC/MS). A quantitative calibration is performed for 5 or 6 of the analytes. The screening data may be used to determine appropriate dilution factors for samples prior to analysis by GC/MS.

2.3 This method is appropriate for analytes ranging in concentration from approximately 2 parts per million by volume (ppmv) and up.

2.4 This method may also be applicable for chemical analysis support to other projects requiring similar determinations, provided that method performance for all analytes is successfully demonstrated prior to use.

3. DISCUSSION

SUMMA® passivated canisters containing samples are pressurized above ambient atmospheric pressure, and then attached directly to heated gas injection valves via a transfer line. The positive pressure of the sample canister fills integral fixed-volume sample loops. The sample loop aliquots are then transferred to two different wide-bore capillary columns housed in a gas chromatograph. After column separation, the analytes are detected with flame ionization detectors. Analyte identification is made by comparison of sample component peak retention times to analyte peak retention times of reference standards analyzed under identical operating conditions. Analyte identity must be confirmed on both analytical columns. Analytes are quantitated using external standardization.

Numerous VOCs may interfere by coeluting with the analytes of interest measured by this method. However, by using two different chromatographic columns for confirmation and quantitation, reporting of false positives due to interference from another VOC is greatly reduced. The chromatographic conditions employed must minimize any potential interference of Table 2 analytes with the target analytes listed in Table 1.

4. SAFETY PRECAUTIONS

4.1 Chemical Hazards

All chemicals handled in this procedure are gases. Some of the compounds that may be present in gaseous samples and standards are carcinogens, suspected carcinogens or suspected reproductive toxins. Avoid inhalation of samples and standards by ensuring that instruments and SUMMA® canisters are vented to fume hoods or drop vents. 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 Thermal Hazards

Certain portions of the analytical system (e.g., sample transfer lines and GC oven) may be maintained at elevated temperatures. Avoid direct skin contact with the high temperature components of the system. Cool heated components to room temperature or don heat shielding gloves prior to performing any maintenance procedures on the components.

4.3 Cryogenic Liquid Hazards

Liquid nitrogen may be used to provide sub-ambient cooling of the analytical system. Liquid nitrogen is a cryogenic hazard that causes severe burns upon skin contact. Follow LI-1354-07 when handling liquid nitrogen.

5. APPARATUS AND REAGENTS

5.1 Apparatus

5.1.1 Gas Chromatograph

Hewlett-Packard Model 5890 Series II Gas Chromatograph, or equivalent, equipped with dual split/splitless capillary injectors, dual fixed volume sample loops, dual flame ionization detectors, and an operating data system (computer) utilizing Hewlett Packard Chem Station or equivalent.

5.1.2 Balston 75-80 Zero Air Generator, or equivalent.

5.1.3 SUMMA® canisters, various sizes.

5.1.4 Compressed Gases

5.1.4.1 Helium, ultra high purity (UHP)

5.1.4.2 Hydrogen, UHP

5.1.4.3 Nitrogen, UHP

5.1.5 Sample Introduction System

5.1.5.1 Chromatographic columns:

Column #1: DB-1701 capillary column or equivalent, 30 m × 0.53 mm i.d., 1.0 µm film thickness.
Column #2: DB-624 capillary column or equivalent, 30 m × 0.53 mm i.d., 3.0 µm film thickness.

5.1.5.2 Sample Loops, 1.0 mL.

5.1.5.3 Mass flow controller to control flow at 20 mL/min.

5.1.5.4 Temperature monitor with Type-K thermocouple to monitor transfer line temperature.

5.1.5.5 Heated transfer line, 1/16” stainless steel.

5.1.6 Wrenches, open-end, various sizes.

5.2 Reagents

5.2.1 Gas Stock Standards

Obtain specialty gas standards containing appropriate concentrations of analytes of interest from Table 1. Also, obtain a specialty gas standard containing the GC/MS screen compounds of interest (see Table 2). Potential commercial sources for specialty standard gases are Scott Specialty Gas, Inc., Alphagaz and Matheson Gas Products, Inc. The specialty gas must have a Certificate of Analysis from the manufacturer and have a known valid relationship to a nationally recognized standard (e.g., NIST) when available. Store the specialty gas cylinder indoors at room temperature for at least 24 hours before use.

5.2.2 Gas Secondary Dilution Standards

Dilute gas stock standards on the gas transfer manifold, per CCP-TP-179, *CCP Gas Transfer Manifold Systems and Sample Compositing*.

5.2.3 Method Detection Limit (MDL) Standards

Use a gas standard prepared at approximately 25 ppmv. Analyze according to CCP-TP-176, *CCP Determination of MDLs for Gas Analysis*.

5.2.4 Laboratory Control Standard (LCS) and Initial Calibration Verification (ICV)

Prepare the LCS and ICV from a gas standard source other than that used to prepare the initial calibration. Prepare the concentration near the midpoint in the calibration range and at or above the PRQL. The LCS and ICV can be the same standard.

5.2.5 Continuing Calibration Standard

Use the gas standard that was used for the initial calibration. This standard should be diluted to approximately 150 ppmv.

6. SAMPLE HANDLING

6.1 Transfer any samples received in Tedlar® bags to SUMMA® canisters prior to analysis.

6.2 Use SUMMA® canisters cleaned per CCP-TP-178,*CCP SUMMA® Canister Cleaning*.

6.3 Store sample SUMMA® canisters at room temperature (0 to 40 º C) prior to analysis.

6.4 Pressurize all samples to above ambient pressure either during collection or by means of a Gas Transfer Manifold (see CCP-TP-179). Use the resultant dilution data to calculate the final results.

6.5 Avoid the use of non-Teflon plastic coating, non-Teflon thread sealants, or flow controllers with rubber components because they may introduce interfering VOCs into the analytical system.

7. PROCEDURES

7.1 Equipment and Instrumentation Set-up

7.1.1 Typical GC 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.*

7.1.1.1 Approximate gas flow rates at an oven temperature of –10 °C

Carrier:	Helium
Column 1:	10 mL/min
Column 2:	10 mL/min

Split Flow	
Column 1:	10 mL
Column 2:	10 mL/min

Detector Gases:	Hydrogen	Air
Column 1:	40 mL/min	400 mL/min
Column 2:	40 mL/min	400 mL/min

Purge Flow at Injector Port:	
Column 1:	5 mL/min
Column 2:	5 mL/min

7.1.1.2 Zone Temperatures

In. A	200 °C
In. B	200 °C
Det. A	250 °C
Det B	250 °C
Aux.	95 °C

7.1.1.3 GC Oven Program

Initial oven temperature: -10 ºC

Initial column hold time: 5 minutes

Column temperature program*: 1.50 ºC/min to 25 ºC
3.00 ºC/min to 50 ºC
70.0 ºC/min to 200 ºC
hold for 3.33 minutes

*Column temperature program interrupted after methanol has eluted when analyzing blanks or standards (ICAL, ICV, CCV and LCS) containing methanol only. The temperature program may be shortened after methanol elution if screening is not being performed.

7.1.2 Equipment and Instrument Checks Prior to Analyzing Samples and Standards

7.1.2.1 Check to see if proper gas flows are adequate for operation. Keep sufficient pressure available to cover weekends and holidays**.**

CAUTION

Helium flow must be continuous. Failure to maintain gas flow may result in decreased detector sensitivity or the inability to identify or confirm the analytes of interest.

7.1.2.2 Verify that the FID flames are ignited.

NOTE: *Signal light-emitting diodes (LEDs) on the GC should read approximately 2-4 picoamperes (pA)*

7.1.2.3 Verify that the mass flow controller is reading 20 mL with purge flow (nitrogen or air).

7.1.2.4 Verify that the temperature reading on the transfer line is between 75 °C and 110 °C.

CAUTION

Failure to verify the transfer line temperature may result in the introduction of an incorrect sample volume into the gas chromatographic system, poor separation or resolution of analytes, or possible condensation of sample (loss of analyte) in the system.

7.1.3 Instrument Preventive Maintenance and Troubleshooting

7.1.3.1 Refer to the instrument Reference Manuals for preventive maintenance and troubleshooting.

7.1.3.2 Record all maintenance performed on the instrument in the GC/FID Maintenance Logbook.

7.2 Instrument Calibration

7.2.1 Analysis of Calibration Standards

7.2.1.1 Prepare a minimum of five calibration standards at concentrations approximately equal to 300, 200, 150, 100, and 25 ppmv.

NOTE 1: *The 25 ppmv standard is below the PRQL.*

NOTE 2: *CCP-TP-179 provides instructions for gas standard.*

NOTE 3: *The calibration range may be increased or decreased as long as a minimum of five standards are used, the upper range is within the linear range of the instrument, and at least one standard has a concentration less than the PRQL.*

7.2.1.2 Analyze each standard three times using instruction in Section 7.3.

7.2.1.3 Construct a calibration curve using the equation in Section 9.1.

7.2.1.4 Evaluate the calibration curve per the regression coefficient acceptance criteria defined in Section 8.1.

7.2.1.5 IF the regression coefficient is acceptable,
THEN update the concentrations in the calibration table to reflect the true values of the calibration standards.

7.2.1.6 Evaluate the calibration curve per the standard recovery acceptance criteria defined in Section 8.1.

7.2.1.7 IF the standard recoveries are acceptable,
THEN analyze an ICV standard.

7.2.1.8 IF the ICV standard analysis meets Section 8.2 acceptance criteria,
THEN GO TO Step 7.2.2

7.2.1.9 IF the calibration or the ICV do not meet acceptance criteria,
THEN correct the problem and repeat the calibration.

7.2.2 Retention Time (RT) Window Determination

7.2.2.1 Establish retention time (RT) windows for each analyte on each column from the ICAL data, using the calculation in Section 9.2.

7.2.2.2 Redetermine RT windows whenever a new ICAL is performed.

7.2.2.3 IF RTs show a major change (i.e., analyte peaks outside the determined window),
THEN recalibrate and redetermine the RT windows.

7.2.3 GC/MS Screen Calibration

7.2.3.1 Calibrate for the GC/MS analytes with a minimum of three standards using purchased gas standards.

7.2.3.2 Analyze each standard, using the procedure in Section 7.3.

7.2.3.3 Update the concentrations in the calibration table to reflect the true values of the standards used.

7.2.3.4 Evaluate the areas of the calibration standards against the new calibration curve (i.e., plug the areas into the regression and calculate percent recovery) using equations in sections 9.1 and 9.3.

7.2.3.5 IF the percent recoveries for each standard are within 30% of true value,
THEN use the calibration for screening.

7.2.3.6 IF the percent recoveries for each standard are not within 30% of true value,
THEN correct the problem and repeat the screening calibration.

7.3 Analysis Procedures for Samples and Standards

7.3.1 Record all pertinent information in the GC/FID Use Logbook (i.e. date, Sample ID, instrument operator, analytical batch, data file name, dilution factor).

7.3.2 Start the HP ChemStation and enter the correct laboratory sample ID and dilution factor, if appropriate.

7.3.3 Connect the sample SUMMA® canister to the transfer line.

7.3.4 Open the valve on the SUMMA® canister.

7.3.5 Verify that the mass flow controller reads 20 mL/min.

7.3.6 After approximately 30 seconds have lapsed, close the SUMMA® canister valve and allow the flow registered on the mass flow controller to reach zero flow.

7.3.7 Push the START button on the data station when the flow controller shows zero flow.

7.3.8 Analyze samples in the following order during a day's run.

- CCV (each 12 hours of continuous instrument operation)
- Laboratory Blank
- LCS (if starting new analytical batch, see NOTE)
- Samples (including sample duplicate and LCS duplicate, see Sections 8.6 and 8.7 for frequency).

NOTE: *The LCS may be analyzed later in the analytical batch. However, analysis at the beginning is strongly recommended to avoid potential sample reanalysis if the LCS fails.*

7.3.9 Remove the sample canister after sample injection is complete.

7.3.10 Attach the nitrogen purge line to the sample transfer line and purge for five minutes.

7.3.11 Dilute and reanalyze any sample having target analyte concentrations that exceed the highest calibration standard concentration.

7.3.12 IF a sample having concentrations exceeding the calibration range by an order of magnitude is analyzed,
THEN analyze a blank (ultra-high purity nitrogen or air) immediately after the sample.

7.3.12.1 IF the results of the blank are less than or equal to three times the program-required MDL in Table 1,
THEN proceed with sample analysis.

7.3.12.2 IF the results of the blank are greater than 3× program-required MDL,
THEN continue analyzing blanks until results ≤3× program-required MDL are obtained.

7.3.13 After completing a sample run, integrate and print the data.

7.3.13.1 Print the external standard report for each sample and quality control (QC) sample, and print the area percent report for each sample.

7.3.13.2 Initial/sign and date the printouts.

7.4 Corrective Action for Failed Analyte Recovery in the CCV

7.4.1 Remake and re-analyze the CCV.

7.4.2 IF the reanalysis of the CCV meets acceptance criteria,
THEN proceed with sample analysis.

7.4.3 IF the reanalyzed CCV does not meet acceptance criteria,
THEN GO TO Step 7.2 and repeat the initial calibration for target analytes.

7.5 Analyst Review of Data

7.5.1 Check that the retention times and peak sizes are consistent with the chromatogram and the integrated values.

7.5.2 Qualitatively identify analytes as present in the sample if the analyte is detected above the MDL on both columns and if the analyte RT for each column is within the window determined by the current ICAL.

7.5.3 Check for consistent chromatography and quantitation on the primary and confirmation columns.

NOTE: *Coeluting compounds should be suspected if there are major differences between column concentrations.*

7.5.4 Verify that the peaks identified are within the retention time window.

7.5.5 Verify correct baseline and peak integration.

7.5.5.1 Manually integrate if necessary to correct erroneous integration.

7.5.5.2 Document the justification for manual integration on the chromatogram.

7.5.6 Use column 1 (front column) as the quantitation column (reported concentration) and column 2 (rear column) as the confirmation column, unless one of the following conditions occurs:

7.5.6.1 IF more than one peak is within the retention time window on both columns,
THEN determine the most likely peak and column to use for reporting. Report the column with the least amount of interference for each analyte when possible.

7.5.6.2 When concentrations between columns are widely divergent, report the lower concentration as required by a qualified independent technical reviewer.

7.5.7 Complete a Data Generator checklist (see example in Exhibit 10) for each day of sample analysis within the analytical batch.

7.6 Data Reporting

Perform generation level review and report data in the following format, per CCP-TP-188, *CCP Analytical Data Recording, Review and Reporting*. See Exhibits 1 – 9 for examples of reporting forms.

- Data Report Cover page and Table of Contents
- Sample Identification Table/Analysis Request Form
- Sample Custody Documents and Sample Tags
- Sample Results
- Quality Control Measurement Results
  - Laboratory Duplicate Comparison Form
  - Laboratory Blank Summary/Results Form
  - Laboratory Control Sample Results Form
  - Analyte ID Forms
- Calibration Results
  - Initial Calibration Forms
  - Continuing Calibration Forms
  - MDL Reporting Form
- Data Review Checklists

8. QUALITY CONTROL REQUIREMENTS

8.1 Initial Calibration (ICAL)

Use a linear least squares regression to determine the external calibration. The regression calculation will generate a correlation coefficient (r) that is a measure of the fit. The value for "r" must be greater than or equal to 0.99. In order to verify the linearity of the calibration curve, plug the areas of each standard back into the calibration regression. The recovery for each target analyte in each standard must be 70% to 130% of true values for both columns. If this criterion is not met, corrective action must be taken (reanalyze, remake, recalibrate, etc.).

8.2 Initial Calibration Verification (ICV)

After the initial instrument calibration has been completed, analyze the second-source initial calibration verification standard (LCS may be used). The recovery for each target analyte must be 85%-115% of true value for both columns. Additionally, the ICV RTs must fall within the RT windows determined from the current ICAL for each target analyte on both columns. If these criteria are not met, repeat the initial calibration.

8.3 Continuing Calibration Verification (CCV)

Analyze a CCV standard at the beginning of each day or every 12 hours of continuous instrument operation, whichever is more frequent. Recoveries of each target analyte must be 85%-115% on both columns. The target analyte RTs for the CCV must fall within the RT windows determined from the current ICAL for both columns. If these criteria are not met, corrective action must be taken. If no source of the problem can be determined after corrective action has been taken, a

new initial calibration curve must be generated. CCV acceptance criteria must be satisfied before sample analysis begins.

8.4 Laboratory Blank (LB)

Analyze a Laboratory Blank daily or after every 12 hours of continuous instrument operation, whichever is more frequent. Analyze the LB after the CCV but before analysis of any field sample. Laboratory Blank (LB) results are acceptable if the concentrations of all target analytes are less than or equal to three times the program-required MDL in Table 1. Sample analysis cannot proceed until the blank criteria have been met. If LB results are not with the specification limits, the source of contamination must be identified and eliminated. If any data associated with a noncompliant LB are reported, data must be flagged with B and Z qualifiers.

8.5 Laboratory Control Sample (LCS)

Analyze an LCS at a minimum frequency of one per analytical batch. The LCS results are acceptable if the recovery of each analyte is within the range of 70% to 130% on both columns. If the LCS fails, the LCS may be reanalyzed once. If it still fails, remake the LCS and repeat the analysis. If the re-prepared LCS still fails, then recalibrate before continuing with the batch analysis. Samples associated with a failed LCS should be reanalyzed whenever possible. If any data associated with a noncompliance LCS are reported, an NCR must be initiated per CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control.*

8.6 Laboratory Duplicates

Analyze a field sample canister in duplicate at a frequency of one per analytical batch. Laboratory duplicate results are acceptable if the Relative Percent Difference (RPD) is ≤ 25% for analyte concentrations ≥ PRQL in both the original analysis and the duplicate. If laboratory duplicate results do not meet the acceptance criteria, initiate a NCR per CCP-QP-005.

8.7 Laboratory Control Sample Duplicate (LCSD)

If none of the field samples in the analytical batch contain all quantitated compounds from Table 1 in concentrations greater than the PRQL, analyze a Laboratory Control Sample Duplicate (LCSD). LCSD results are acceptable if the % RPD is ≤ 25%. If LCSD results do not meet the acceptance criteria, initiate a NCR per CCP-QP-005.

8.8 Precision and Accuracy (P&A) Demonstrations

Demonstrate method precision and accuracy by initially analyzing seven replicate method performance samples (see Section 5.2.4) and achieving precision of ≤ 25% relative standard deviation and mean accuracy of 70% to 130%. After the initial Precision and Accuracy (P&A) performance is acceptable, demonstrate continuing acceptable performance semiannually by analyzing at least four

replicate method performance standards and evaluating those results against the initial P&A specifications.

9. CALCULATIONS

9.1 Initial Calibration (ICAL) Calculation

Plot of area versus concentration (least squares linear regression).

$$ppmv = DF[Am + b]$$

where: ppmv = concentration of analyte in parts per million by volume

A = area of analyte from External Standard Report

m = slope of linear regression

b = y-intercept of linear regression

DF = Dilution Factor

9.2 Retention Time (RT) Window Calculations

RT windows are calculated using all the retention times from all concentration levels of the ICAL for each target analyte on each column. The RT window is determined as the mean RT of the ICAL plus or minus three times the standard deviation of the determination. If the standard deviation is zero, use 0.01 minutes as a default standard deviation.

9.3 Percent Recovery (%R)

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

where: C_m = Measured concentration (ppmv), and

C_t = True concentration (ppmv).

9.4 Relative Percent Difference (RPD)

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

where: C_s = Sample concentration (ppmv), and

C_d = Duplicate concentration (ppmv).

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

- MDL Documentation
- ICAL Documentation
- P&A Documentation

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

QA/Nonpermanent

- Copy of applicable pages of GC/FID Use Logbook
- Copy of applicable pages of GC/FID Maintenance Logbook
- Raw Data/Instrument Printouts

11. REFERENCES

11.1 EPA SW-846, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, 3rd Edition, Method 8015B, *Nonhalogenated Organics Using GC/FID*, 1996.

11.2 CCP-QP-002, *CCP Training and Qualification Plan*

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

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

11.5 CCP-TP-175, *CCP Analysis of Gas Samples for VOCs by GC/MS*

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

11.7 CCP-TP-178, *CCP SUMMA Canister Cleaning*

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

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

11.10 LI-1354-07-IRC/ECL, "Environmental Chemistry Laboratory (ECL) Gas Analysis Project."

11.11 LWP-14620, "Chemical Hygiene Plan"

Exhibit 1. Analysis Data Sheet (Example)

GAS VOC ANALYSIS DATA SHEET

FIELD SAMPLE ID
ID040202EI722

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD:	ACMM-9910	SAMPLING BATCH:	PRF020158
INSTRUMENT:	GC-2	DATA REPORT:	ECL02193G
LAB SAMPLE ID:	02095018	ANALYTICAL BATCH:	ECL02193G
DATE RECEIVED:	04/05/02	DATE SAMPLED:	4/2/02
		COMPOSITE:	No

COLUMN 1 (FRONT):		COLUMN 2 (REAR):	
LAB FILE ID:	2F003168.D	LAB FILE ID:	2R003168.D
DATE ANALYZED:	4/9/02	DATE ANALYZED:	4/9/02
TIME ANALYZED:	0739	TIME ANALYZED:	0739

CAS #	ANALYTE	CONCENTRATION	
		PPMV	FLAG(S)
67-56-1	Methanol	22	J

COMMENTS

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Exhibit 2. Laboratory Duplicate Form (Example)

LABORATORY DUPLICATE FORM
GAS VOC ANALYSIS

FIELD SAMPLE ID
ID040202EI125D

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD: ACMM-9910
INSTRUMENT: GC-2

SAMPLING BATCH: PRF020157
ANALYTICAL BATCH: ECL02187G
DATA REPORT: ECL02187G

LAB SAMPLE ID: 02093022

LAB DUPLICATE ID: 02093022D

	COLUMN 1	COLUMN 2		COLUMN 1	COLUMN 2
LAB FILE ID:	2F003124.D	2R003124.D	LAB FILE ID:	2F003132.D	2R003132.D
DATE ANALYZED:	4/5/02	4/5/02	DATE ANALYZED:	4/6/02	4/6/02
TIME ANALYZED:	1518	1518	TIME ANALYZED:	0849	0849

COLUMN 1

ANALYTE	CONCENTRATION (PPMV)				RPD * (%)
	SAMPLE	FLAG(S)	DUPLICATE	FLAG(S)	
Methanol	14.35700	U	14.35700	U	NC

COLUMN 2

ANALYTE	CONCENTRATION (PPMV)				RPD * (%)
	SAMPLE	FLAG(S)	DUPLICATE	FLAG(S)	
Methanol	11.20600	U	11.20600	U	NC

* CONTROL LIMITS: RPD <= 25% FOR ALL ANALYTES PRESENT AT CONCENTRATIONS >= PRQL
NC = NOT CALCULABLE

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Exhibit 3. 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-9910	ANALYTICAL BATCH:	ECL02187G
INSTRUMENT:	GC-2	DATA REPORT:	ECL02187G
LAB SAMPLE ID:	LBG2A05	FIELD SAMPLE ID:	LB04103A04

COLUMN 1 (FRONT)		COLUMN 2 (REAR)	
LAB FILE ID:	2F003116.D	LAB FILE ID:	2R003116.D
DATE ANALYZED:	4/5/02	DATE ANALYZED:	4/5/02
TIME ANALYZED:	0721	TIME ANALYZED:	0721

CAS #	ANALYTE	CONCENTRATION	
		PPMV	FLAG(S)
67-56-1	Methanol	4.1	U

THIS LABORATORY BLANK APPLIES TO THE FOLLOWING SAMPLES:

FIELD SAMPLE ID	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED
ID040202EI392	02093015	4/5/02	0758
ID040202EIB22	02093016C15	4/5/02	0913
ID040202EIB35	02093016C25	4/5/02	0913
ID040202EIE06	02093016C35	4/5/02	0913
ID040202EI169	02093016C45	4/5/02	0913
ID040202EI300	02093016C55	4/5/02	0913
ID040202EIC73	02093017C15	4/5/02	1003
ID040202EI121	02093017C25	4/5/02	1003
ID040202EIB50	02093017C35	4/5/02	1003
ID040202EIB40	02093017C45	4/5/02	1003
ID040202EI633	02093017C55	4/5/02	1003
ID040202EI004	02093018C15	4/5/02	1105
ID040202EI281	02093018C25	4/5/02	1105
ID040202EI670	02093018C35	4/5/02	1105
ID040202EI502	02093018C45	4/5/02	1105
ID040202EI460	02093018C55	4/5/02	1105

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Exhibit 4. Laboratory Control Sample Form (Example)

LABORATORY CONTROL SAMPLE FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD:	ACMM-9910	ANALYTICAL BATCH:	ECL02187G
INSTRUMENT:	GC-2	DATA REPORT:	ECL02187G
LAB SAMPLE ID:	LCSG2A04	FIELD SAMPLE ID:	LCS30385SM09
		LCS STD SOURCE:	MATHESON CYL #30385

COLUMN 1 (FRONT)		COLUMN 2 (REAR)	
LAB FILE ID:	2F003111.D	LAB FILE ID:	2R003111.D
DATE ANALYZED:	4/4/02	DATE ANALYZED:	4/4/02
TIME ANALYZED:	1628	TIME ANALYZED:	1628

COLUMN 1

ANALYTE	CONCENTRATION (PPMV)		RECOVERY (%)
	FOUND	TRUE	
Methanol	150.450	150.9	99.7

COLUMN 2

ANALYTE	CONCENTRATION (PPMV)		RECOVERY (%)
	FOUND	TRUE	
Methanol	167.854	150.9	111.2

CONTROL LIMITS: 70% <= %R <= 130%

Form Rev 10-01

Exhibit 5. Analyte Identification Form (Example)

ANALYTE IDENTIFICATION FORM
GAS VOC ANALYSIS

FIELD SAMPLE ID
ID040202EI722

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD:	ACMM-9910	SAMPLING BATCH:	PRF020158
INSTRUMENT:	GC-2	DATA REPORT:	ECL02193G
LAB SAMPLE ID:	02095018	ANALYTICAL BATCH:	ECL02193G

COLUMN 1 (FRONT)		COLUMN 2 (REAR)	
LAB FILE ID:	2F003168.D	LAB FILE ID:	2R003168.D
DATE ANALYZED:	4/9/02	DATE ANALYZED:	4/9/02
TIME ANALYZED:	0739	TIME ANALYZED:	0739

ANALYTE	COL	QUANT	RETENTION TIME	RT WINDOW		CONCENTRATION	
				FROM	TO	PPMV	FLAG(S)
Methanol	1	*	2.815	2.535	3.198	22.369	J
	2		4.196	4.101	4.393	38.873	J

COLUMN USED FOR QUANTITATION MARKED WITH *

COMMENTS

Form Rev 10-99

Exhibit 6. Initial Calibration Form (Example)

IDAHO NATIONAL ENGINEERING AND EVIRONMENTAL LABORATORY

BECHTEL BWXT IDAHO, LLC

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD	INSTRUMENT	ICAL ID	ICAL STD. SOURCE
ACMM-9910	GC-2	ICAL2-06	AERIFORM ECL# 30395

	COLUMN 1	COLUMN 2
ANALYSIS METHOD	17012-06	6242-06
CALIBRATION DATE	27-Mar-01	27-Mar-01
CALIBRATION TIME	1322	1333
INSTRUMENT METHOD	WIPPT2.MTH	WIPPT2.MTH

COLUMN 1

STD#	STD LOG#	TRUE VALUE (PPMv)	AVERAGE VALUE (PPMv)	%R
1	WC-03-07-09	24.848	23.373	94.1%
2	WC-03-06-09	99.248	97.392	98.1%
3	WC-03-09-09	150.901	151.403	100.3%
4	WC-03-08-09	199.488	197.759	99.1%
5	WC-03-01-10	299.880	301.518	100.5%
REGRESSION COEFFICIENT: r =	1.000		MEAN RT: 2.867	minutes

COLUMN 2

STD#	STD LOG#	TRUE VALUE (PPMv)	AVERAGE VALUE (PPMv)	%R
1	WC-03-07-09	24.848	23.985	96.5%
2	WC-03-06-09	99.248	97.511	98.2%
3	WC-03-09-09	150.901	150.878	100.0%
4	WC-03-08-09	199.488	198.452	99.5%
5	WC-03-01-10	299.880	301.226	100.4%
REGRESSION COEFFICIENT: r =	1.000		MEAN RT: 4.247	minutes

COMMENTS: (1) FOR A VALID ICAL, 85% ≤ %R ≤ 115%

Exhibit 7. Initial Calibration Verification Form (Example)

INITIAL CALIBRATION VERIFICATION FORM

GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD: ACMM-9910
INSTRUMENT: GC-2
LAB SAMPLE ID: ICV2-07
ICV STD SOURCE: MATHESON ECL# 30385

ANALYSIS BATCH NO: CALIBRATION
DATA REPORT NO: CALIBRATION
ICAL DATE: 20-Apr-02
ICAL ID: ICAL2-07
ICAL STD SOURCE: AERIFORM ECL# 30395

LAB FILE ID: 2F003276.D
DATE ANALYZED: 20-Apr-02
TIME ANALYZED: 1312

LAB FILE ID: 2R003276.D
DATE ANALYZED: 20-Apr-02
TIME ANALYZED: 1312

COLUMN 1

ANALYTE	CONCENTRATION (PPMV)		RECOVERY	RETENTION
	TRUE	FOUND	(%)	TIME
Methanol	150.900	143.469	95.08	2.901

COLUMN 2

ANALYTE	CONCENTRATION (PPMV)		RECOVERY	RETENTION
	TRUE	FOUND	(%)	TIME
Methanol	150.900	148.766	98.59	4.854

CONTROL LIMITS: 70% ≤ %R ≤ 130%

4/20/02

FORM ICV (10/17/01)

Exhibit 8. Continuing Calibration Form (Example)

CONTINUING CALIBRATION FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

METHOD:	ACMM-9910	ANALYTICAL BATCH:	ECL02187G
INSTRUMENT:	GC-2	DATA REPORT:	ECL02187G
LAB SAMPLE ID:	CCV20357	ICAL DATE:	3/27/01
		ICAL ID:	ICAL2-06

COLUMN 1 (FRONT)		COLUMN 2 (REAR)	
LAB FILE ID:	2F003109.D	LAB FILE ID:	2R003109.D
DATE ANALYZED:	4/4/02	DATE ANALYZED:	4/4/02
TIME ANALYZED:	1557	TIME ANALYZED:	1557

COLUMN 1

ANALYTE	CONCENTRATIONS (PPMV) TRUE	FOUND	RECOVERY (%)	RETENTION TIME (MIN)
Methanol	150.000	140.272	93.5	2.824

COLUMN 2

ANALYTE	CONCENTRATIONS (PPMV) TRUE	FOUND	RECOVERY (%)	RETENTION TIME (MIN)
Methanol	150.000	154.358	102.9	4.196

CONTROL LIMITS: 85% <= %R <= 115%

Form Rev 10-99

Exhibit 9. Method Detection Limit Form (Example)

MDL REPORTING FORM
GAS VOC ANALYSIS

IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

ENVIRONMENTAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Method: ACMM-9910
Instrument ID: GC-2 MDL Determination Date: 2/27/02

Column 1

ANALYTE	MDL (ppmv)	MDL (ng)*	PRQL (ppmv)
Methanol	4.1	5.4	100

Column 2

ANALYTE	MDL (ppmv)	MDL (ng)*	PRQL (ppmv)
Methanol	3.2	4.2	100

* Assumes a 1 mL sample.

The MDLs are presented in units of ppmv and ng. The listed MDLs are approximately 15 times lower than the required values 150 ng. The listed MDLs are applicable to undiluted samples, standards, and lab blanks only; for diluted samples and standards, the MDL value is dependent on the multiplier/dilution factor (found on the external standard report).

Form Rev 10-99

Exhibit 10. Data Generator Checklist (Example)

Environmental Chemistry Laboratory
Data Generator Checklist

ACMM-9910 (GC/FID VOCs)

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

Criteria	Yes	No	N/A	Comments
1. Samples analyzed in accordance with ACMM-9910 requirements.				
2. CCV analyzed at the beginning of the run (once every 12 hours of instrument operation), and meets acceptance criteria.				
3. LB analyzed after the CCV, and meets acceptance criteria.				
4. 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 this batch.]				
5. All samples quantitated within the calibration range, diluted and reanalyzed, or scheduled for reanalysis within the batch.				
6. All user modifications to chromatogram integration documented and justified. [N/A if no user modifications were made to any chromatogram.]				
7. All logbook entries completed in accordance with MCP-2011.				
8. All raw data corrections made per MCP-2008 requirements [lined out, initialed, dated, and justified (as necessary)].				
9. All raw data evaluated, signed/initialed and dated in indelible black ink.				
10. Raw data reviewed for completeness and accuracy.				

Form Revision: 0
E:\ECL\checklists\ACMM-9910 Data Generator Rev 0.doc
Created on 04/03/02 12:01 PM

Table 1. VOCs by GC/FID Target Analytes

Target Analyte (Alternate names)	CAS Number	TWCP-Required Method Detection Limit (MDL)* (ppmv)	TWCP Program-Required Quantitation Limit (PRQL) (ppmv)
Acetone (2-propanone) (dimethyl ketone)	67-64-1	6.3	100
1-Butanol (n-Butyl alcohol)	71-36-3	5.0	100
Methyl ethyl ketone (2-Butanone)	78-93-3	5.1	100
Methanol (Methyl alcohol)	67-56-1	11	100
Methyl isobutyl ketone **(4-Methyl-2-pentanone)** (Hexone)	108-10-1	3.7	100

95. Assume 10 mL sample, 150 ng MDL

Table 2. Analytes of Interest for Screening

Target Analyte	CAS Number
1. Acetone	67-64-1
2. Benzene	71-43-2
3. Bromoform	75-25-2
4. Butanol	71-36-3
5. Carbon disulfide**	75-15-0
6. Carbon tetrachloride*	56-23-5
7 Chlorobenzene	108-90-7
8. Chloroform	67-66-3
9. Chloromethane**	74-87-3
10. Cyclohexane	110-82-7
11. 1,1-Dichloroethane	75-35-3
12. 1,2-Dichloroethane	107-06-2
13. 1,1-Dichloroethylene	75-35-4
14. 1,2-Dichloroethylene, cis	156-59-2
15. 1,2-Dichloroethylene, trans	156-60-5
16. 1,2-Dichloropropane**	78-87-5
17. Ethyl benzene	100-41-4
18. Ethyl ether	60-29-7
19. Methyl ethyl ketone	78-93-3
20. Methyl isobutyl ketone	108-10-1
21. Methylene chloride*	75-09-2
22. 1,1,2,2,-Tetrachloroethane	79-34-5
23. Tetrachloroethylene*	127-18-4
24. Toluene*	108-88-3
25. 1,1,1-Trichloroethane*	71-55-3
26. Trichloroethylene**	79-01-6
27. 1,1,2-Trichloro-1,2,2-trifluoroethane*	76-13-1
28. Trichlorofluoromethane	75-69-4
29. 1,2,4-Trimethylbenzene	95-63-6
30. 1,3,5-Trimethylbenzene	108-67-8
31. m-Xylene	108-38-3
32. o-Xylene	95-47-6
33. p-Xylene	106-42-3

* Calibrated Analytes for Screening

** Site-specific analyte; analysis may not be required for samples from all TRU Waste Generator Storage Sites.