Waste Isolation Pilot Plant Flammable Gas Analysis

March 2007

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Flammable Gas Analysis

March 2007

Approved by: Signature on file Date: 3/19/07
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RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	11/6/06	Initial Use.
1	12/28/06	Added RRF equation and editorial changes.
1.1	1/10/07	Editorial correction to Step 4.2.4 [T].
1.2		Change to allow SRS & Hanford to perform procedure at sea level and other editorial corrections.

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1.0 PURPOSE

This document describes and implements the process to establish the concentration of flammable gas/volatile organic compounds (VOCs), hydrogen, and methane in a waste container intended for shipment in the Transuranic Package Transporter-II (TRUPACT-II) or HalfPACT packagings. An aliquot of headspace gas (HSG) is sampled from a waste container and analyzed using one analytical unit which is a gas chromatograph (GC) with two detectors, a mass spectrometer (MS) and a thermal conductivity detector (TCD). The sample introduced to the GC is split and part goes to MS and the other part goes to TCD. The MS analyzes sample for volatile organic compounds (VOC) and the TCD analyzes for hydrogen and methane. The requirements and technical bases for allowable flammable gas/VOC concentrations are described in the *Contact-Handled Transuranic Waste Authorized Methods for Payload Control (CH-TRAMPAC)*.

1.1 Scope

This document applies to system startup, tune check, calibration checks, daily checks, waste container HSG sampling and analysis, and container analysis via WIPP TRAMPAC Evaluation Software (WTES). The target analytes are listed in Table 1, Gas Target Analyte List. If necessary, a written request can be submitted (as described by the CH-TRAMPAC) by a site and must be approved by Carlsbad Field Office (CBFO) to add a compound to the Appendix B list of the CH-TRU Waste Content Codes CH-TRUCON document, then WIPP TRAMPAC Evaluation Software (WTES) will accept the new compound.

2.0 REQUIREMENTS

2.1 Training Requirements

2.1.1 Personnel performing this procedure will be trained and qualified.

2.2 Equipment List

2.2.1 Analytical System

[A] GC with TCD and MS detectors.

[B] GC/MS Interface - The GC column is coupled directly to the MS source using a heated transfer line.

[C] Data System - A data system that allows the continuous acquisition and storage of mass spectra and TCD data obtained throughout the duration of the chromatographic program is interfaced to the GC/MS-GC/TCD System. The data system has software that allows a search of a GC/MS data file for ions

of a specified mass and allows plotting of such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

[D] The data system is capable of library searching of a National Institute of Science and Technology (NIST) mass-spectral library.

2.2.2 Supplies and Equipment

[A] Gases

[A.1] Initial Calibration (ICAL) gas standards shall be procured as certified gas standards. The gas standards shall contain the target analytes listed in Table 1.

[A.2] Continuing Calibration Verification (CCV) gas standard shall be procured as certified gas standards. The gas standard shall contain the target analytes listed in Table 1.

[A.3] IS gas mixture contains the two ISs (fluorobenzene and chlorobenzene-d5) in nitrogen. The IS shall be procured as a certified gas standard.

[A.4] Tune Check: gas mixture contains BFB in nitrogen. The gas mixture will be procured at a concentration that will deliver approximately 50 ng of BFB to the mass spectrometer.

[B] Sampling Equipment

[B.1] Side port needles with Luer Lock hubs.

[B.2] 0.7 µm or less disposable syringe filter.

[B.3] 50 mL (minimum) gas tight syringes with Luer Lock hubs.

[B.4] Syringe valve with Luer Lock hubs.

[B.5] Material to cover a waste container filter.

2.3 Precautions and Limitations

2.3.1 If this procedure **CANNOT** be implemented as written, work will be STOPPED, the LO will be notified, and when this procedure is corrected, work can be resumed.

2.3.2 Radiological safety will be in accordance with the applicable radiological control plans or equivalent issued by the site.

2.3.3 This procedure may involve the use of hazardous materials, operations, and equipment. Hazards must be identified and mitigated prior to initiating sampling.

2.3.4 Consideration should be given to safety concerns regarding chemical and radiation exposure.

2.3.5 Samples and reference materials may be flammable. Flammable materials must be kept away from heat, sparks, and open flame, and adequate ventilation must be provided.

2.3.6 The certified gas standards must not be used past their expiration date.

2.4 Prerequisite Actions

2.4.1 Prerequisite activities if required will be in accordance with site procedures.

2.5 Quality Assurance Objectives (QAOs)

2.5.1 **Accuracy** - Each day of sampling operations, a CCV check will be performed. This will verify that the analytical system is capable of quantifying HSG to the accuracy percent difference (%D) requirement. The %D for the target compounds must be less than or equal to 30.

2.5.2 **Methane Precision** – Precision is a measure of the mutual agreement among multiple measurements of a single analyte, by the same method. The precision for Methane is demonstrated when meeting the coefficient of determination (r^2) requirement for an ICAL and when meeting the %D requirement for CCV.

2.5.3 **Representativeness -** Representativeness of HSG sampling and analysis is ensured with issuance of this technical standard operating procedure by CBFO.

2.5.4 **Completeness -** If the flammable gas/VOC concentration in the container headspace cannot be established, the HSG of the container shall be sampled and analyzed to determine the flammable gas/VOC concentration.

2.5.5 **Comparability -** Comparability of HSG sampling and analysis is ensured with issuance of this technical standard operating procedure by CBFO.

2.6 Quality Control (QC) Requirements

2.6.1 **BFB Tune Check** – BFB is introduced into the Gas Chromatograph/Mass Spectrometry (GC/MS) system. Upon analysis of BFB, the mass spectrum shall meet the criteria listed in Table 2, 4-Bromofluorobenzene Key Ions and Abundance Criteria. Acceptable correlation to these criteria must be demonstrated at the start of the operational shift.

2.6.2 **Initial Calibration (ICAL)** - A minimum five-point ICAL curve is generated to establish an ability to quantitate VOC compounds and serves to define the linear range of the method. The concentration of IS in these gases is constant while the target constituents vary in concentration. An ICAL is performed at startup, after major repairs, or when the quality controls can no longer be met. For the calibration curve to be valid, the relative standard deviation (RSD) of relative response factor (RRF) for each analyte must be less than 35 percent. The RRF and RSD are determined from the following equations:

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

Where:

A_x = area of the characteristic ion for the compound being measured

A_{is} = area of the characteristic ion for the specific IS

C_{is} = amount of the specific IS

C_x = concentration of VOC standard

$$\%RSD = \frac{s}{y_{mean}} * 100$$

Where:

s = standard deviation of RRF for a compound

y_{mean} = mean of initial RRFs for a compound (average RRF)

$$s = \sqrt{\frac{\sum_{i=1}^{n} (y_i - y_{mean})^2}{n-1}}$$

Where:

y_i = measured value of the i^{th} replicated value

n = number of replicate values

y_{mean} = mean value

A minimum three-point ICAL linear regression curve is generated to establish an ability to quantitate hydrogen and methane. For the calibration curve to be valid the coefficient of determination (r^2) must be greater than 0.990. The r^2 is calculated using the following equation:

$$r^2 = \left(\frac{\overline{x_i y_i} - \overline{x}_i \overline{y}_i}{\sqrt{\overline{x_i^2} - (\overline{x_i})^2}\sqrt{\overline{y_i^2} - (\overline{y_i})^2}} \right)^2$$

Where:

r^2 = the coefficient of determination
x_i = is the concentration
y_i = is the area response.

2.6.3 **<u>Continuing Calibration Verification (CCV)</u>** - CCV standard is analyzed at the start of the operational shift, but after BFB Tune Check. The CCV standard must be from a different source or lot than the standard used for the ICAL. The Internal standard responses must be within 50–200 percent of the internal standard response for the initial calibration mid-point standard. Internal standard retention time (RT) must not shift by ≥0.5 min from the RT of the mid-point standard of the current initial calibration. The Response Factor (RF) for Hydrogen and Methane is calculated using the following equation:

$$RF = \frac{A_x}{C_x}$$

Where:

A_x = area of the Hydrogen or Methane

C_x = concentration of Hydrogen or Methane standard

The %D for the target compounds must be less than or equal to 30. The %D is calculated for VOC using the following equation:

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

Where:

RRF_i = average RRF from ICAL.

RRF_c = RRF from current standard.

The %D is calculated for Hydrogen and Methane using the following equation:

$$\%D = \frac{RF_i - RF_c}{RF_i} * 100$$

Where:

RF_i = average RF from ICAL.

RF_c = RF from current standard.

2.6.4 **Method Detection Limit (MDL)** – MDL is the minimum concentration of a target analyte that can be measured and reported with 99 percent confidence. MDLs are determined at startup, after major repairs, or after column change. MDLs are determined using the following equation:

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

Where:

t (n-1,1-α=.99) = the one sided 99 percent t-statistic and n is the number of observations and s is the standard deviation of replicate measurements. Seven replicates must be analyzed. The t-statistic level is n=7 and t=3.14. The MDL for the Alcohols and Ketones must be less than or equal to 50 ppmv and all other VOCs must be less than or equal to 15 ppmv. The combined MDL for m- and p-xylene must be less than or equal to 15 ppmv. The MDL for hydrogen and methane must be less than or equal to 0.1 Volume %.

2.6.5 **Nonconformance** – If data are generated that DO **NOT** meet QC requirements, the issue is treated as a nonconformance, by site procedures.

2.6.6 **Batch Data Report (BDR) Number** – A batch is defined as the number of samples collected in an operational shift. The BDR is

assigned a unique identification number in the format SSYYPPNXXX where SS is the site designation, YY are the last two digits of the year, PP is the project designation, N is the instrument identifier and XXX is a sequential number starting with 001 each year. The same unique identification numbering format will be used for the ICAL and MDL report.

2.6.7 **Qualifier Flags** – Two reporting flags will be used.

- U Target analyte was not detected and concentration reported as the MDL;
- D Target analyte was quantified from a secondary dilution.

2.6.8 **Retention Time (RT) Window for H_2 and CH_4** – The RT window for each analyte is determined from the individual RTs of the ICAL standards. The RT window is calculated as the mean RT plus or minus ten percent.

3.0 RESPONSIBILITIES

3.1 Vendor/HSG Project Manager (VPM)

3.1.1 Performs the function of the Manager.

3.1.2 Oversees operations and maintenance of analytical operations and manages data generated from sampling and analysis operations.

3.2 Operator

3.2.1 Samples waste container.

3.2.2 Performs HSG analysis.

3.2.3 Prepares the BDR.

3.2.4 Maintains daily Operational Logbook per site procedures.

3.3 Lead Operator (LO)

3.3.1 Provides guidance in the event of abnormal conditions.

3.3.2 Reviews Operational Logbooks at a minimum of once per week.

3.3.3 Performs Operator duties.

3.4 Independent Technical Reviewer (ITR)

3.4.1 Performs the data generation level ITR review of the BDR. This review shall be performed by a qualified person other than the operator who is qualified to have performed the initial work.

3.5 Waste Certification Official or designee

3.5.1 Performs container evaluation using WIPP TRAMPAC Evaluation Software (WTES).

4.0 PROCEDURE

NOTE

When preparing batches that will be generated and have the ITR performed electronically, electronic signatures are utilized. The electronic signature completes the requirement for printing and signing name, and dating the batch. Additionally, printing paperwork is only required for manual batches.

NOTE

Meeting the DAC requirement is only required when sampling and analyzing for only VOCs or VOCs and hydrogen and methane together. The DAC requirement is not required for hydrogen and methane analysis alone.

4.1 Determining If Waste Container Can Be Sampled

NOTE

Reference Attachments 1 and 6 and collect necessary information as needed.

NOTE

This section may be completed simultaneously with Section 4.4, Routine Sampling and Analysis, so long as the waste container's Drum Age Criteria (DAC) is completed prior to sampling of the individual container.

4.1.1 If sampling for Hydrogen and Methane only then GO TO Section 4.1.3

4.1.2 Determine the correct Sampling Scenario to establish the DAC performing the following steps:

NOTE

DACs are defined for three unique venting and sampling scenarios. These venting and sampling scenarios are defined by the time elapsed after container closure and venting as follows:

t_1 = time (days) elapsed after container closure until venting.
t_2 = time (days) elapsed after venting.

- Scenario 1 – The drum liner headspace (under liner lid) can be sampled at the time of venting if t_1 is greater than DAC_1 (see Table 3 DAC_1 Values [in days]).

- Scenario 2 – For drums generated in an unvented condition and subsequently vented, the drum headspace can be sampled in a vented drum if t1 is greater than DAC_1, and t_2 is greater than DAC_2 (see Table 4, DAC_2 Values [in days]).

- Scenario 3 – If t_1 is less than DAC_1 when the container is vented, the container headspace can be sampled when t_2 is greater than DAC_3. Also, for newly generated containers that were vented at the time of generation, the container headspace can be sampled after DAC_3 has been exceeded (see Table 5, Packaging Configuration Groups, Table 6, Package-Specific DAC_3 Values for Solidified Waste [Waste Types I and IV], and Table 7, Package-Specific DAC_3 Values [in days] for Solid Waste [Waste Types II and III].

NOTE

Waste containers with Scenario 1, 2, and/or 3 may be combined in the same batch.

NOTE

When only analyzing for hydrogen and methane, then for sections 4.2, 4.3, 4.4, 4.5, 4.6, and 4.7 only apply requirements for Hydrogen and Methane.

4.1.3 **IF** using Scenario 1,
THEN perform, **AND** document the following on Attachment 1, Sample Container Data Form:

[A] BDR Number (enter on Page 1 and Page 2).

[B] Waste Container ID (enter on Page 1 and Page 2).

[C] Enter 1 for Sampling Scenario.

[D] Waste Type (i.e., I, II, III, IV).

[E] Enter N/A for Rigid Liner.

[F] Enter N/A for Rigid Liner Lid.

[G] Enter N/A for Rigid Liner Lid Hole Diameter.

[H] Enter N/A for Number of Inner Bags.

[I] Enter N/A for Number of Liner Bags.

[J] Enter N/A for Filter Model Number.

[K] Enter N/A for Hydrogen Diffusivity of Filter.

[L] Enter N/A for Package Configuration Group Number.

[M] Closure Date.

[N] Enter N/A for Vent Date.

[O] Required DAC Value (in days) from Table 3.

[P] Calculate and enter Drum Age (in days).

4.1.4 **IF** container can be sampled,
THEN record Y (Yes) on Attachment 1, **AND** continue.

4.1.5 **IF** container **CANNOT** be sampled,
THEN contact the LO, **AND** record N (No) on Attachment 1.

4.1.6 **IF** using Scenario 2,
THEN perform, **AND** document the following on Attachment 1:

[A] BDR Number (enter on Page 1 on Page 2).

[B] Waste Container ID (enter on Page 1 and Page 2).

[C] Enter 2 for Sampling Scenario.

[D] Waste Type (i.e., I, II, III, IV).

[E] Enter Y or N for Rigid Liner.

[F] Enter Y or N for Rigid Liner Lid.

[G] **IF** there is a Rigid Liner Lid present,
THEN enter the diameter of the hole for the Rigid Liner Lid Hole Diameter as 0.30, 0.375, 0.75, or 1.0-inch if no rigid liner is present enter 1.0 inch.

[H] Enter N/A for Number of Inner Bags.

[I] Enter N/A for Number of Liner Bags.

[J] Filter Model Number.

[K] Hydrogen Diffusivity of Filter.

[L] Enter N/A for Package Configuration Group Number.

[M] Closure Date.

[N] Vent Date.

[O] Required DAC Value (in days) from Table 4.

[P] Calculate and Enter Drum Age (in days).

4.1.7 **IF** container can be sampled,
THEN record Y on Attachment 1, **AND** continue.

4.1.8 **IF** container **CANNOT** be sampled,
THEN contact the LO, **AND** record N on Attachment 1.

4.1.9 **IF** using Scenario 3,
THEN perform, **AND** document the following on Attachment 1:

[A] BDR Number (enter on Page 1 and Page 2).

[B] Waste Container ID (enter on Page 1 and Page 2).

[C] Enter 3 for Sampling Scenario.

[D] Waste Type. (i.e., I, II, III, IV)

[E] Enter Y or N for Rigid Liner.

[F] Enter Y or N for Rigid Liner Lid.

[G] **IF** there is a Rigid Liner Lid present,
THEN enter the diameter of the hole for the Rigid Liner Lid Hole Diameter as 0.30, 0.375, 0.75, or 1.0-inch,
ELSE enter N/A

[H] Number of Inner Bags.

[I] Number of Liner Bags.

[J] Filter Model Number.

[K] Hydrogen Diffusivity of Filter.

[L] Package Configuration Group Number from Table 5.

[M] Closure Date.

[N] Vent Date.

[O] Required DAC Value (in days) from Table 6 or Table 7.

[P] Calculate and Enter Drum Age (in days).

4.1.10 **IF** the container can be sampled,
THEN record Y on Attachment 1, **AND** continue.

4.1.11 **IF** the container **CANNOT** be sampled,
THEN contact the LO, **AND** record N (NO) on Attachment 1.

4.1.12 **WHEN** Attachment 1 is complete,
THEN print name, sign, and date the Attachment 1, **AND** place in the BDR holding folder.

NOTE
When the procedure says to OPEN or CLOSE a program, it also can mean to MAXIMIZE or MINIMIZE respectively.

4.2 ICAL

NOTE
An ICAL is not a routine operation and is performed at startup, after major repairs, or when the quality controls can no longer be met.

4.2.1 BFB Tune Check

[A] On the computer, create a new folder called c:\hpchem\1\data\YYMMDDI, where YY is year, MM is month, DD is day, and I is place holder.

NOTE
The naming convention for icons (e.g. HGAS 05), methods (e.g. VOCGAS.m), data files, and folders that are used in this procedure are included as examples. Site specific procedure may define these items differently.

[B] OPEN HGAS 05 icon.

[C] From the pull down menu METHOD, select LOAD.

[D] From the directory c:\HPCHEM\1\Methods, select Tune.m.

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR field.

[H] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDI\BIYYMMDD.d, where YY is year, MM is month, and DD is day.

[I] Click START RUN Button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** push START on GC/MS.

[L] CLOSE BFB valve.

[M] Enter the following on Attachment 2, Flammable Gas Analysis Daily Run Sheet:

[M.1] BDR Number.

[M.2] Date.

[M.3] BFB for Analysis Type.

[M.4] Laboratory File Name.

[M.5] Not applicable (N/A) for Waste Container ID (Identification).

[M.6] N/A for Dilution Factor (DF).

[M.7] N/A for Sample/Standard Pressure.

[M.8] Operator Initials.

[N] **WHEN** analysis is complete,
THEN OPEN ANALYSIS.

[O] From pull down menu METHOD, select LOAD METHOD.

[P] Click YES to question popup dialog box “Be sure changes are saved. Load Now?”.

[Q] From the directory c:\HPCHEM\1\Methods, select Tune.m, **AND** click OK.

[R] From pull down menu FILE, select LOAD DATA FILE.

[S] From the directory c:\HPCHEM\1\Data\YYMMDDI, select BIYYMMDD.d file, **AND** click OK.

[T] Double Right click on the BFB spectra.

[U] From pull down menu TUNER, select EVALUATE TO SCREEN.

[V] **IF** the key ion abundance criteria in Table 2 are met, **THEN** proceed.

[W] **IF** the key ion abundance criteria in Table 2 are **NOT** met, **THEN** return to step 4.2.1 [T], **AND** select an alternate scan(s) to evaluate.

[X] **IF** an acceptable BFB scan(s) cannot be found, **THEN** contact the LO to correct the problem, **AND** rerun BFB Tune Check.

[Y] Print BFB Tune Evaluation Report (see Attachment 9, BFB Tune Evaluation Report for an example).

[Z] CLOSE MULTIVU, leaving ANALYSIS open.

4.2.2 VOCGAS.m

[A] From pull down menu METHOD, select LOAD METHOD.

[B] Click YES to question popup dialog box “Be sure changes are saved. Load Now?.

[C] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m, **AND** click OK.

[D] From pull down menu CALIBRATE, select EDIT COMPOUNDS.

[E] Select FLUOROBENZENE, **AND** click VIEW.

[F] In the box labeled SAMPLE ISTD CONCENTRATION, ensure the IS concentration is X times the concentration of IS cylinder

to be used, where X is the ratio between IS and sample loop volume.

[G] **IF** new concentration is to be entered,
THEN enter the IS concentration in the box labeled SAMPLE ISTD CONCENTRATION.

[H] Click Page 3

[I] In the Conc column check to see if the IS concentrations in Levels 1 through 5 is the same as Page 1.

[J] **IF** the Concentration is not the same as Page 1
THEN make the correction.

[K] In the Conc column and response column for the CC check to see if there are no entries.

[L] **IF** there are entries
THEN delete them.

[M] Click OK.

[N] **IF** a change has been made,
THEN click YES in box labeled SAVE CHANGES TO COMPOUND,
ELSE, Click NO.

[O] Repeat steps 4.2.2[E] through 4.2.2[N] substituting Chlorobenzene-d5 for Fluorobenzene.

[P] Starting with Target Compound 1, repeat steps 4.2.2[P.1] through 4.2.2[P.7] for each target compound:

[P.1] Select COMPOUND.

[P.2] Click VIEW.

[P.3] Click PAGE 3.

[P.4] In column CONC for each LVL ID column used, compare the different entered levels of ICAL and CC concentrations to the ICAL and CC concentration to be used.

[P.5] **IF** a new concentration is to be used,
THEN enter the concentration in the appropriate box.

[P.6] Click OK.

[P.7] **IF** a change has been made,
THEN click YES in box labeled SAVE CHANGES TO COMPOUND?,
ELSE click NO.

[Q] Click EXIT.

[R] From the pull down menu METHOD, select SAVE METHOD.

[S] Save method as VOCGAS.m.

[T] Click OK.

[U] CLOSE ANALYSIS.

4.2.3 Analyze ICAL

NOTE
The ICAL3 concentration must be approximately equal to the CCV standard concentration. The CCV concentration is compared to the ICAL3 concentration for the %D.

[A] **IF** it is necessary to prepare a calibration standard less than the stock calibration standard,
THEN calculate the DF using the following equation:

[A.1] DF = initial volume / final volume

[B] Use the appropriate syringe(s) to take an aliquot of the stock calibration standard.

[C] Perform serial dilutions, if necessary.

[D] Record the following information on Attachment 6 for each standard prepared:

[D.1] Date.

[D.2] Standard Cylinder Number.

[D.3] Stock Standard Expiration Date.

[D.4] Initial Standard Concentration.

[D.5] Initial Volume.

[D.6] Final Volume.

[D.7] Dilution Factor.

[D.8] Final Standard Concentration.

[D.9] Operator Initials.

[E] OPEN HGAS 05.

[F] From pull down menu METHOD, select LOAD.

[G] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m.

[H] Click OK.

[I] Click on GREEN ARROW.

[J] Enter initials in OPERATOR field.

[K] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDI\ICALX.d, where X is a standard level, **AND** in the miscellaneous box enter the calibration cylinder number and expiration date.

[L] Click START RUN Button.

[M] OPEN vent valve, **AND** using a suitable syringe, flush sample loop with approximately 30 to 50 milliliter (ml) of nitrogen or air.

[N] Attach standard syringe to inlet.

[O] Flush sample loop with approximately 10 ml of standard.

[P] OPEN IS valve.

[Q] Fill sample loop to 775 plus or minus 10 torr or mmHg, with standard by opening and closing vent valve.

[R] START GC/MS.

[S] CLOSE IS valve.

[T] Remove standard syringe.

[U] Enter the following on Attachment 2:

[U.1] ICALX for Analysis Type, where X is the ICAL level standard number.

[U.2] Laboratory File Name.

[U.3] N/A for Waste Container ID.

[U.4] DF (if applicable) or N/A

[U.5] Sample/Standard Pressure (Torr).

[U.6] Operator Initials.

4.2.4 Evaluate ICAL

[A] **AFTER** analysis is complete,
THEN OPEN ANALYSIS.

[B] From pull down menu FILE, select LOAD DATA FILE.

[C] From the directory c:\HPCHEM\1\Data\YYMMDDI\, select ICAL1.d, **AND** click OK.

[D] From the pull down menu QUANTITATE, select CALCULATE.

[E] Exit MULTIVU.

[F] OPEN ANALYSIS.

[G] From pull down menu VIEW, select QEdit QUANT Results.

[H] Verify and correct if necessary the integration for each compound.

[I] Select EXIT.

[J] Select YES or NO in the SAVE CHANGES TO QUANITATION RESULTS? dialog box depending if changes were made.

[K] From pull down menu QUANTITATE, select GENERATE REPORT.

[L] In the QUANTITATE REPORT OPTIONS dialog box select SUMMARY REPORT TO PRINTER.

[M] From pull down menu CALIBRATE, select UPDATE.

[N] Select UPDATE ONE LEVEL in SELECT UPDATE OPTION dialog box.

[O] Click OK.

[P] Select NO to REQUANTITATE.

[Q] Select to RE-CALIBRATE LEVEL 1, **AND** REPLACE RESPONSES.

[R] Click DO UPDATE.

[S] Click EXIT.

[T] Repeat steps 4.2.3[A] through 4.2.4[S] for ICAL2, ICAL3, ICAL4, and ICAL5.

[U] From the directory c:/HPCHEM/1/Data/YYMMDDI/, select ICAL3.d, **AND** click OK.

[V] From pull down menu CALIBRATE, select UPDATE.

[W] Select UPDATE ONE LEVEL in SELECT UPDATE OPTION dialog box.

[X] Click OK.

[Y] Select NO to REQUANTITATE.

[Z] Select to RE-CALIBRATE LEVEL CC and REPLACE RESPONSES, REPLACE RETENTION TIME, **AND** check box to REPLACE QUALIFIER ION RELATIVE RESPONSES.

[AA] Click DO UPDATE.

[BB] Click EXIT.

[CC] Select YES in UPDATE REFERENCE SPECTRA? dialog box.

[DD] From the pull down menu HGas, select RESPONSE FACTORS TO SCREEN.

[EE] **IF** the %RSD for any VOC is greater than or equal to 35 percent,
THEN contact the LO to correct the problem,
AND repeat the individual standard level or entire ICAL as appropriate.

[FF] **IF** the %RSD for all VOCs is less than 35 percent,
THEN proceed.

[GG] Print Response Factor Report (see Attachment 12, ICAL Response Factor Report for an example).

[HH] EXIT the MULTIVU screen.

[II] From the pull down menu CALIBRATE, select EDIT COMPOUNDS.

[JJ] Select HYDROGEN.

[KK] Select VIEW.

[LL] Select Page 3.

[MM] For each LVL ID not used for the hydrogen curve delete the CONC and RESPONSE.

[NN] Select PLOT.

[OO] **IF** r2 is less than or equal to 0.990 for hydrogen
THEN contact LO to correct the problem,
AND repeat the individual standard level or entire ICAL as appropriate.

[PP] If the r^2 is greater than 0.990 for hydrogen, then proceed

[QQ] Select PRINT.

[RR] Select OK.

[SS] Select OK.

[TT] **IF** values were changed then click YES to SAVE CHANGES TO COMPOUND?, ELSE click NO. ,

[UU] Repeat steps 4.2.4[JJ] through 4.2.4[TT] substituting methane for hydrogen.

[VV] Click EXIT.

[WW] **AFTER** all ICAL analyses have been performed,
THEN from the pull down menu METHOD, select SAVE METHOD.

[XX] Click OK.

[YY] Save method as VOCGAS.m.

[ZZ] Select EXIT.

[AAA] Select YES from BE SURE CHANGES ARE SAVED EXIT NOW? Dialog box.

4.2.5 ICAL Report

[A] Assemble ICAL Report with the following information:

[A.1] Attachment 7, Data Report Cover Page.

[A.2] ICAL Response Factor Report.

[A.3] BFB Evaluation Report.

[A.4] Quantitation Reports (see Attachment 13, Quantitation Report for an example) for ICAL standards.

[A.5] Hydrogen r^2 Plot Sheet (See Attachment 5, Hydrogen and Methane r^2 Plot Sheet for an example) from VOCGAS.m.

[A.6] Methane r^2 Plot sheets (See Attachment 5, Hydrogen and Methane r^2 Plot Sheet for an example) from VOCGAS.m.

[A.7] Attachment 6, Standard Preparation Worksheet.

[A.8] Attachment 2.

[B] Paginate ICAL Report.

[C] Complete Data Report Cover Page.

[D] Print name, sign, and date Data Report Cover Page.

[E] Submit the ICAL Report to the ITR.

ITR

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

[F.1] BFB Tune Check met acceptance criteria of Table 2.

[F.2] ICAL standards were updated correctly into VOCGAS.m.

[F.3] Concentrations of ISs used for ICAL standard analysis are correct.

[F.4] That the %RSD for all VOCs is less that 35 percent.

[F.5] That the r^2 is greater than 0.990 for hydrogen and methane.

[F.6] Notify the Operator of all errors or omissions found during the review, if applicable.

[F.7] Recheck the data after the errors or omissions have been rectified, if applicable.

[G] Print name, sign, and date Data Report Cover Page.

[H] Forward the ICAL Report to Records according to site procedures.

NOTE

The MDL is performed at startup, after major repairs, or after column change.

Operator

4.3 MDL

4.3.1 Tune Check

[A] On the computer, create new folder called c:\hpchem\1\data\YYMMDDM, where YY is year, MM is month, DD is day, and M is place holder.

[B] OPEN HGAS 05 icon.

[C] From the pull down menu METHOD, select LOAD.

[D] From the directory c:\HPCHEM\1\Methods, select Tune.m.

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR field.

[H] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDM\BMYYMMDD.d, where YY is year, MM is month, and DD is day.

[I] Click START RUN Button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** push START on GC/MS.

[L] CLOSE BFB valve once the sample has been injected.

[M] Enter the following on Attachment 2, Flammable Gas Analysis Daily Run Sheet:

[M.1] BDR Number.

[M.2] Date.

[M.3] BFB for Analysis Type.

[M.4] Laboratory File Name.

[M.5] Not applicable (N/A) for Waste Container ID (Identification).

[M.6] N/A for Dilution Factor (DF).

[M.7] N/A for Sample/Standard Pressure.

[M.8] Operator Initials.

[N] **WHEN** analysis is complete,
THEN OPEN ANALYSIS.

[O] From pull down menu METHOD, select LOAD METHOD.

[P] Click YES to question popup dialog box “Be sure changes are saved. Load Now?”.

[Q] From the directory c:\HPCHEM\1\Methods, select Tune.m, **AND** click OK.

[R] From pull down menu FILE, select LOAD DATA FILE.

[S] From the directory c:\HPCHEM\1\Data\YYMMDDM, select BMYYMMDD.d file, **AND** click OK.

[T] Double Right click on the BFB spectra.

[U] From pull down menu TUNER, select EVALUATE TO SCREEN.

[V] **IF** the key ion abundance criteria in Table 2 are met, **THEN** proceed.

[W] **IF** the key ion abundance criteria in Table 2 are **NOT** met, **THEN** return to step 4.3.1 [T], **AND** select an alternate scan(s) to evaluate.

[X] **IF** acceptable BFB scan(s) cannot be found, **THEN** contact the LO to correct the problem, **AND** rerun BFB Tune Check.

[Y] Print BFB Tune Evaluation Report.

[Z] CLOSE MULTIVU.

4.3.2 Analyze MDL

[A] From pull down menu METHOD, select LOAD METHOD.

[B] Click YES to question popup dialog box “Be sure changes are saved. Load Now?”.

[C] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m, **AND** click YES.

[D] **IF** it is necessary to prepare a MDL standard less than the stock calibration standard, **THEN** calculate the DF using the following equation:

[D.1] DF = initial volume / final volume

[E] Use the appropriate syringe(s) to take an aliquot of the stock calibration standard.

[F] Perform serial dilutions, if necessary.

[G] Record the following information on Attachment 6 for each standard prepared:

[G.1] Date.

[G.2] Standard Cylinder Number.

[G.3] Stock Standard Expiration Date.

[G.4] Initial Standard Concentration.

[G.5] Initial Volume.

[G.6] Final Volume.

[G.7] Dilution Factor.

[G.8] Final Standard Concentration.

[G.9] Operator Initials.

[H] OPEN HGAS 05.

[I] From pull down menu METHOD, select LOAD.

[J] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m.

[K] Click OK.

[L] Click on GREEN ARROW.

[M] Enter initials in OPERATOR field.

[N] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDM\MDLX.d, where X is the number MDL standard and in the miscellaneous box enter the calibration cylinder number and expiration date.

[O] Click START RUN Button.

[P] OPEN vent valve, **AND** using a suitable syringe, flush sample loop with approximately 30 to 50 ml of nitrogen or air.

[Q] Attach standard syringe to inlet.

[R] Flush sample loop with approximately 10 ml of standard.

[S] OPEN IS valve.

[T] Fill sample loop to 775 plus or minus 10 torr or mmHg, with standard by opening and closing vent valve.

[U] START GC/MS.

[V] CLOSE IS valve.

[W] Remove standard syringe.

[X] Enter the following on Attachment 2:

[X.1] MDL for Analysis Type.

[X.2] Laboratory File Name.

[X.3] N/A for Waste Container ID.

[X.4] DF (if applicable) or N/A.

[X.5] Sample/Standard Pressure (Torr).

[X.6] Operator Initials.

[Y] After analysis is finished, OPEN ANALYSIS.

[Z] From pull down menu FILE, select LOAD DATA FILE.

[AA] From the directory c:\HPCHEM\1\Data\YYMMDDM\, select MDL1.d, **AND** click YES.

[BB] From the pull down menu QUANTITATE, select CALCULATE.

[CC] Exit MULTIVU.

[DD] OPEN ANALYSIS.

[EE] From pull down menu VIEW, select QEdit QUANT Results.

[FF] Verify and correct if necessary the integration for each compound.

[GG] Select EXIT.

[HH] Select YES or NO in the SAVE CHANGES TO QUANITATION RESULTS? dialog box depending if changes were made.

[II] From pull down menu QUANTITATE, select GENERATE REPORT.

[JJ] In the QUANTITATE REPORT OPTIONS dialog box select SUMMARY REPORT to PRINTER.

[KK] Repeat steps 4.3.2[D] through 4.3.2[JJ] for all MDLs.

4.3.3 Determine MDLs

[A] Determine MDLs by entering the concentration for all of the target compounds in the Flammable Gas Analysis MDL Spreadsheet (see Attachment 11, Flammable Gas Analysis MDL Spreadsheet for an example).

[B] **IF** the MDLs meet the requirement of step 2.6.4, **THEN** proceed.

[C] **IF** the MDLs DO **NOT** meet the requirements, **THEN** contact the LO to correct.

[D] Print Flammable Gas Analysis MDL Spreadsheet.

4.3.4 MDL Report

[A] Assemble MDL Report with the following information:

[A.1] Attachment 7, Data Report Cover Page.

[A.2] Flammable Gas Analysis MDL Spreadsheet.

[A.3] BFB Evaluation Report.

[A.4] Quantitation Report for each MDL.

[A.5] Attachment 2.

[A.6] Attachment 6 (if applicable).

[B] Paginate MDL Report.

[C] Complete Data Report Cover Page, except for signature.

ITR

[D] Review MDL Report.

[D.1] Ensure the BFB Tune Check met criteria in Table 2.

[D.2] Ensure the concentrations from the MDL analyses have been correctly entered into the Flammable Gas Analysis MDL Spreadsheet.

[D.3] Ensure MDLs meet requirements of step 2.6.4.

[D.4] Notify the Operator of all errors or omissions found during the review, if applicable.

[D.5] Recheck the data after the errors or omissions have been rectified, if applicable.

[E] Open Analysis.

[F] From pull down menu METHOD, select LOAD METHOD.

[G] Click YES to question popup dialog box “Be sure changes are saved. Load Now?”

[H] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m and click OK.

[I] From pull down menu CALIBRATE, select EDIT COMPOUNDS.

[J] Starting with Target Compound 1, repeat steps 4.3.4 [J.1] through 4.3.4 [J.5] for each target compound:

[J.1] Select COMPOUND.

[J.2] Click VIEW.

[J.3] Click Page 2. In column MDL and row MATRIX enter MDL value or verify value is correct.

[J.4] Click OK.

[J.5] **IF** a change has been made,
THEN click YES in box labeled SAVE CHANGES TO COMPOUND?,

[K] Click EXIT.

[L] From the pull down menu METHOD, select SAVE METHOD.

[M] Save method as VOCGAS.m

[N] Click OK.

[O] Print name, sign, and date the Data Report Cover Page.

[P] Submit to Operator.

[Q] Repeat steps 4.3.4 [E] through 4.3.4 [O] THEN return to step 4.3.4[R].

[R] Submit the MDL Report to Records according to site procedures.

Operator

4.4 Routine Sampling and Analysis

4.4.1 Tune Check

[A] On the computer, create new folder called c:\hpchem\1\data\YYMMDDC, where YY is year, MM is month, DD is day, and C is place holder.

[B] OPEN HGAS 05 icon.

[C] From the pull down menu METHOD, select LOAD.

[D] From the directory c:\HPCHEM\1\Methods, select Tune.m.

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR field.

[H] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDC\BCYYMMDD.d, where YY is year, MM is month, and DD is day.

[I] Click START RUN Button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** push START on GC/MS.

[L] Once the sample has been injected, CLOSE BFB valve.

[M] Enter the following on Attachment 2, Flammable Gas Analysis Daily Run Sheet:

[M.1] BDR Number.

[M.2] Date.

[M.3] BFB for Analysis Type.

[M.4] Laboratory File Name.

[M.5] Not applicable (N/A) for Waste Container ID (Identification).

[M.6] N/A for Dilution Factor (DF).

[M.7] N/A for Sample/Standard Pressure.

[M.8] Operator Initials.

[N] **WHEN** analysis is complete,
THEN OPEN ANALYSIS.

[O] From pull down menu METHOD, select LOAD METHOD.

[P] Click YES to question popup dialog box “Be sure changes are saved. Load Now?”.

[Q] From the directory c:\HPCHEM\1\Methods, select Tune.m, **AND** click OK.

[R] From pull down menu FILE, select LOAD DATA FILE.

[S] From the directory c:\HPCHEM\1\Data\YYMMDDC, select BCYYMMDD.d file, **AND** click OK.

[T] Double right click on the BFB spectra.

[U] From pull down menu TUNER, select EVALUATE TO SCREEN.

[V] **IF** the key ion abundance criteria in Table 2 are met,
THEN proceed.

[W] **IF** the key ion abundance criteria in Table 2 are **NOT** met,
THEN return to step 4.4.1 [T], **AND** select an alternate scan(s) to evaluate.

[X] **IF** an acceptable BFB scan(s) cannot be found,
THEN contact the LO to correct the problem, **AND** rerun BFB Tune Check.

[Y] Print BFB Tune Evaluation Report.

[Z] CLOSE MULTIVU.

4.4.2 CCV

[A] OPEN HGAS 05.

[B] From pull down menu METHOD, select LOAD.

[C] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m.

[D] Click OK.

[E] Click on GREEN ARROW.

[F] Enter initials in OPERATOR field.

[G] In the DATA FILE NAME field enter c:\hpchem\1\data\YYMMDDC\CCYYMMDD.d in the DATA FILE NAME field, where YY is year, MM is month, and DD is day and in the miscellaneous box enter the calibration cylinder number and expiration date.

[H] Click START RUN Button.

[I] OPEN vent valve, **AND** using a suitable syringe, flush sample loop with approximately 30 to 50 milliliter (ml) of nitrogen or air.

[J] Attach standard syringe to inlet.

[K] Flush sample loop with approximately 10 ml of standard.

[L] OPEN IS valve.

[M] Fill sample loop to 775 plus or minus 10 torr or mmHg, with standard by opening and closing vent valve.

[N] START GC/MS.

[O] CLOSE IS valve.

[P] Remove standard syringe.

[Q] Enter the following on Attachment 2:

[Q.1] CCV for Analysis Type.

.

[Q.2] Laboratory File Name.

[Q.3] N/A for Waste Container ID.

[Q.4] N/A for DF.

[Q.5] Sample/Standard Pressure (Torr).

[Q.6] Operator Initials.

[R] After analysis is finished, OPEN ANALYSIS.

[S] From the pull down menu METHOD, select LOAD.

[T] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m.

[U] Click OK.

[V] From pull down menu FILE, select LOAD DATA FILE.

[W] From the directory c:\HPCHEM\1\Data\YYMMDDC\, select CCYYMMDD.d **AND** click YES.

[X] From the pull down menu QUANTITATE, select CALCULATE.

[Y] Print Quantitation Report

[Z] Exit MULTIVU.

[AA] OPEN ANALYSIS.

[BB] From pull down menu VIEW, select QEdit QUANT Results.

[CC] Verify and correct the integration for each compound, if necessary.

[DD] Select EXIT.

[EE] Select YES or NO in the SAVE CHANGES TO QUANITATION RESULTS? dialog box depending if changes were made.

[FF] From pull down menu HGas, select EVALUATE DATA FILE AS CONTINUING CAL TO SCREEN.

[GG] **IF** the %D for each of the target compounds in the CCV is less than or equal to 30 percent,
THEN proceed.

[HH] **IF** the %D is greater than 30 percent,
THEN contact the LO to correct problem, **AND** repeat CCV.

[II] Print the CCV Evaluation Report (see Attachment 10, CCV Evaluation Report for an example).

[JJ] EXIT MultiVu screen.

[KK] CLOSE ANALYSIS.

4.4.3 Sampling

[A] Flush sample syringe that is in good working order five times with nitrogen or air.

WARNING

The disposable filter must be added to control potential radiological contamination when obtaining gas samples.

[B] Use a syringe, a syringe valve, a disposable filter and a needle for sampling a waste container.

WARNING

Applicable personal protective equipment (PPE) must be worn when performing HSG waste container sampling activities.

CAUTION

Any material determined by radiation contamination survey to have measurable contamination will be handled in accordance with site procedures.

[C] Review the Sample Container Data Form to make sure the waste container is on the form and ready to be sampled.

NOTE

Steps 4.4.3 [D] through 4.4.3 [F] may be performed any time before a sample is taken.

[D] Remove the sample port seal screw, if necessary, from the filter on the drum to be sampled.

[E] Cover or block the filter with a material before sampling to prevent intrusion of air.

[F] Ensure all filters not used for sampling are covered or blocked as applicable.

[G] Insert syringe with a syringe valve open **AND** withdraw a minimum of 50 mL of sample, then close syringe valve.

[H] Remove the needle/filter subassembly from the sampling syringe, **AND** discard the disposable filter.

NOTE

Steps 4.4.3 [I] and 4.4.3 [J] can be performed any time after sampling is complete or at the end of the operational shift.

[I] Remove all covers and/or blocks from all of the filters, unless present for contamination control.

[J] Reinstall sample port seal screw, if necessary.

[K] Take sample to analytical instrument.

[L] Open HGAS 05.

[M] Click on GREEN ARROW.

[M.1] Enter initials in OPERATOR field.

[M.2] **IF** this is the first analysis of this waste container, **THEN** perform the following:

(a) Enter XXXXXXXX.d, where XXXXXXXX is the Waste Container ID. Enter alpha site container characters prior to the numerical portion of the container as applicable.

(b) Enter the type of container (e.g., 55-gallon drum) and where sample was taken (e.g., under the container lid) in the miscellaneous box.

NOTE

The analysis date is the same as the sampling date because the analysis is always performed the same day as sampling.

[M.3] **IF** this is a sample of a waste container which has been diluted,

(a) **THEN** enter diluteN.d where N is dilution number and in the miscellaneous box enter waste container ID, XXXXXXXX (enter alpha site

container characters prior to the numerical portion of the container as applicable) and DF.

[M.4] Click START RUN Button.

[N] OPEN vent valve, **AND** using a suitable syringe, flush sample loop with approximately 100 to 200 ml of nitrogen or air.

[O] Attach sample syringe to inlet and open syringe valve.

[P] Flush sample loop with approximately 10 ml of sample.

[Q] OPEN IS valve.

[R] Fill sample loop to 775 plus or minus 10 torr or mmHg, with standard or sample, by opening and closing vent valve.

[S] START GC/MS.

[T] CLOSE IS valve.

[U] Close syringe valve and remove sample syringe once the sample has been injected.

[V] Enter the following on Attachment 2:

[V.1] Sample for Analysis Type.

[V.2] Laboratory File Name.

[V.3] Waste Container ID.

[V.4] DF (if applicable) **OR** N/A.

[V.5] Sample introduction pressure for Sample/Standard Pressure (Torr).

[V.6] Operator Initials.

[W] After analysis is finished, OPEN ANALYSIS.

[X] From the pull down menu METHOD, select LOAD.

[Y] From the directory c:\HPCHEM\1\Methods, select VOCGAS.m.

[Z] Click OK.

[AA] From pull down menu FILE, select LOAD DATA FILE.

[BB] From the directory c:\HPCHEM\1\Data\YYMMDDC\, select XXXXXXXX.d or diluteN.d, **AND** click OK.

[CC] From the pull down menu QUANTITATE, select CALCULATE.

[DD] Exit MULTIVU.

[EE] OPEN ANALYSIS.

[FF] From pull down menu VIEW, select QEdit QUANT Results.

[GG] Verify and correct, if necessary, the integration for each compound.

[HH] Select EXIT.

[II] Select YES or NO in the SAVE CHANGES TO QUANITATION RESULTS? dialog box depending if changes were made.

[JJ] From pull down menu QUANTITATE, select GENERATE REPORT.

[KK] In the GENERATE REPORT dialog box select DETAILED REPORT to PRINTER.

[LL] **IF** the Internal standard responses are not within 50–200 percent of the internal standard response for current CCV or Internal standard RT has shifted by >0.5 min from the RT of the current CCV and are due to Matrix affects
THEN document on the quantitation report of waste container that the internal standard and/or RT does not meet the requirement due to Matrix affects **ELSE** contact LO.

[MM] **IF** any analyte is detected during the analysis of a sample in the amount exceeding the ICAL range,
THEN calculate the DF using the following equation so the analyte on reanalysis will be approximately in the mid range of the calibration curve:

[MM.1] DF = initial volume / final volume

[NN] Perform serial dilutions, if necessary.

[OO] For each sample diluted, record the following information on Attachment 3, Sample Dilution Worksheet:

[PP] BDR Number (only during first sample).

[QQ] Date (only during first sample).

[RR] Waste Container ID.

[SS] Initial Volume.

[TT] Final Volume.

[UU] Dilution Factor.

[VV] Operator Initials.

[WW] **IF** the waste container sample was diluted repeat steps 4.4.3[L] through 4.4.3[VV],
ELSE repeat steps 4.4.3[A] through 4.4.3[VV] until samples are complete, including dilutions.

4.5 Data Evaluation

NOTE

The software automatically identifies the target analytes by comparing the sample mass spectrum with the mass spectrum of a standard of the suspected compound. Standard reference mass spectra are obtained through analysis of calibration standards.

The software automatically quantitates target analytes based on the integrated abundance from the EICP of the primary characteristic ion listed in Table 8.

NOTE

Tentatively Identified Compound (TIC) is equivalent to saying Non-listed Flammable VOC.

4.5.1 Tentatively Identified Compounds (TICs)

[A] Qualitative Identification

NOTE

The software automatically performs a search against a NIST Mass Spectral Library for all unknown (non-target) compounds having a total ion area greater than 10 percent of the nearest IS.

NOTE

IF the unknown has a total ion area greater than 10 percent of the nearest IS, **THEN** the unknown meets the first criteria for being reportable.

NOTE

IF the unknown meets all of the following guidelines, **THEN** the unknown meets the second criteria for being reportable in accordance with the following guidelines:

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

NOTE

The concentration of the reportable TIC compound shall be estimated using the internal standard nearest the retention time of the TIC. Quantitation shall use a response factor of one. Quantitation is performed by first multiplying the area count of the TIC by the concentration of internal standard and then dividing by the area count of the internal standard.

[B] **IF** an unknown has been determined to be a reportable TIC and meets the following guideline of flammability **THEN** Report the identified TIC in the Identified TIC Compound portion of the Flammable Gas Analysis Form (see Attachment 4, Flammable Gas Analysis Form for an example).

- **IF** a TIC VOC has an NFPA 704 Hazard Rating Index for flammability of 4 (Danger: Flammable gas or extremely flammable liquid), 3 (Warning: Flammable liquid flash point below 100F), or 2 (Caution: Combustible liquid flash point of 100F to 200F), **THEN** it is considered flammable.

4.6 Data Reporting

4.6.1 Report the following information on the Flammable Gas Analysis Form (see Attachment 4, Flammable Gas Analysis Form for an example) for each target compound:

[A] BDR Number.

[B] Waste Container ID.

[C] For hydrogen and methane report Measured Volume % or MDL which ever is appropriate, **AND** Reporting Flag.

[D] For VOCs report Measured ppmv or MDL which ever is appropriate, **AND** Reporting Flag.

4.6.2 **IF** TICs are reportable,
THEN report on Flammable Gas Analysis Form for each TIC the following information:

[A] CAS Number.

[B] Identified TIC Compound.

[C] Estimated Amount (ppmv) as calculated by the instrument software.

4.6.3 Assemble the BDR as follows:

[A] Attachment 7, Data Report Cover Page.

[B] Attachment 8.

[C] BFB Tune Evaluation Report.

[D] CCV Evaluation Report.

[E] Flammable Gas Analysis Form for each waste container.

[F] Attachment 1.

[G] Attachment 2.

[H] Attachment 3, if necessary.

[I] Quantitation Report for each sample and sample dilution analysis.

[J] Copy of NCR(s), if applicable.

4.6.4 Record the following information on Attachment 7:

[A] BDR Number.

[B] NCR Number, if applicable.

[C] ICAL BDR Number.

[D] MDL BDR Number.

[E] Add information to Content Column in Table of Content portion of Data Report Cover Page (Attachment 7).

4.6.5 Print name, sign, and date Attachment 7, **AND** forward to the ITR.

4.7 Data Review

ITR

4.7.1 Review the BDR in accordance with Attachment 8, **AND** perform data generation level review.

4.7.2 Notify the HSG Operator of all errors or omissions found during the review.

4.7.3 Recheck the data after the errors or omissions have been rectified.

4.7.4 Print name, sign, and date Attachment 8.

4.7.5 Paginate the BDR, **AND** add page numbers to Attachment 7.

4.7.6 Submit the BDR to Records according to site procedures.

4.8 Container Analysis via WIPP TRAMPAC Evaluation Software (WTES)

Waste Certification Official or Designee

NOTE

Upon completion of the Waste Certification process for the waste containers sampled in this procedure, the following data must be submitted to the WIPP Waste Information System (WWIS) for each container in order for the WIPP TRAMPAC Evaluation Software (WTES) portion of the WWIS to evaluate compliance with transportation requirements. If using electronic file transfer methods, refer to site's CBFO-approved program procedures for WWIS data submittal, otherwise refer to the sections below.

NOTE

WWIS User's Manual referenced herein is defined as the latest version of DOE/CBFO 97-2273.

4.8.1 Refer to Figure 9-2 and appendices F.1.7 and F.1.8 of WWIS User's Manual; collect the applicable information needed for each container.

4.8.2 Log onto the WWIS as a shipper/generator user with privileges for the site for which data are being entered.

4.8.3 From the main screen, draw down the "cErtification" menu and select "Certification data entry".

4.8.4 Enter data using main screen and all subscreens (main, radioisotopes, material parameters, filters, assay methods, characterization methods, hazardous codes, methods, comments).

4.8.5 After all data have been entered, select the Save button (disc icon) and then press the (e) button to run a Preliminary Evaluation for each container.

4.8.6 If the container fails to pass any of the WIPP TRAMPAC Evaluation Software (WTES) checks, the database will return a list of error messages. Contact the LO or the VPM/HSG Project Manager, otherwise select lightning bolt to formally process the container data. When the preliminary WIPP TRAMPAC Evaluation Software (WTES)container evaluation is run successfully, the user will receive a summary of the results of the evaluation as shown in Figure 9-3, WIPP TRAMPAC Evaluation Software (WTES) *Container Evaluation Results*.

5.0 RECORDS

5.1 Records generated during the performance of this procedure are maintained as QA records. The records are as follows:

5.1.1 QA/Nonpermanent Records

[A] ICAL Report

[B] MDL Report

5.1.2 QA/Lifetime Records

[A] BDR

6.0 REFERENCES

6.1 U.S. Department of Energy (DOE). Contact-Handled Transuranic Waste Authorized Methods for Payload Control (CH-TRAMPAC), NRC Docket No. 71-9218, current revision, U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico.

6.2 U.S. Department of Energy (DOE). Quality Assurance Program Plan for TRUPACT-II Gas Generation Test Program, DOE/WIPP 01-3187, current revision, U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico.

6.3 U.S. Department of Energy (DOE). Quality Assurance Program Document, DOE/CBFO 94-1012, current revision, U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico.

Table 1. Gas Target Analyte List

COMPOUND	CAS No.
Benzene	71-43-2
Chlorobenzene	108-90-7
Cyclohexane	110-82-7
1,1-Dichloroethane	75-34-3
1, 2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
cis-1,2-Dichloroethylene	156-59-2
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Toluene	108-88-3
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
m-Xylene[a]	108-38-3
o-Xylene	95-47-6
p-Xylene[a]	106-42-3
Acetone	67-64-1
1-Butanol	71-36-3
Methanol	67-56-1
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Hydrogen	1333-74-0
Methane	74-82-8

[a] These xylene isomers **CANNOT** be resolved by the analytical methods employed in this procedure.

Table 2. 4-Bromofluorobenzene Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8% to 40% of m/z 95
75	30% to 66% of m/z 95
95	Base peak, 100% relative abundance
96	5% to 9% of m/z 95
173	Less than 2% of m/z 174
174	50% to 120% of m/z 95
175	5% to 9% of m/z 174
176	93% to 101% of m/z 174
177	5% to 9% of m/z 176

Table 3. DAC_1 Values (in days)

Waste Type	DAC_1 (days)
Waste Type I and IV	127
Waste Type II and III	53

Note: Containers that are sampled using the Scenario 1 DAC DO **NOT** require information on the packaging configuration because the Scenario 1 DAC are based on a bounding packaging configuration. In addition, information on the rigid liner vent hole presence and diameter DO **NOT** apply to containers that are sampled using the Scenario 1 DAC because they are unvented prior to sampling and not dependent upon a package configuration.

Table 4. DAC_2 Values (in days)

	Waste Type I and IV				Waste Type II and III			
Drum Filter Minimum Hydrogen Diffusivity	Rigid Liner Vent Hole Diameter (inch)				Rigid Liner Vent Hole Diameter (inch)			
(mol/s/mf)	0.30	0.375	0.75	1.0	0.30	0.375	0.75	1.0
1.9×10^{-6}	36	30	23	22	29	22	13	12
3.7×10^{-6}	30	25	19	18	25	20	12	11
3.7×10^{-5}	13	11	11	11	7	6	6	4

Table 5. Packaging Configuration Groups

Packaging Configuration	Waste Types II and III	Waste Types I and IV
Packaging Configuration 1 (55-gallon drums)	No inner bags, no liner bags	
Packaging Configuration 2 (55-gallon drums)	Up to 4 bag layers, up to 1 of which is a liner bag	Any configuration with 1 liner bag
Packaging Configuration 3 (55-gallon drums)	Up to 6 bag layers, up to 2 of which are liner bags	Any configuration with 2 liner bags
Packaging Configuration 4 (pipe components)	Up to 2 inner bags and 1 filtered metal can inside a pipe component (headspace sample taken inside the pipe component)	
Packaging Configuration 5 (SWBs and TDOPs)	SWB or TDOP with up to 1 bag layer (inner or liner)	
Packaging Configuration 6 (SWBs and TDOPs)	SWB or TDOP with up to 6 bag layers, up to 1 of which is a liner bag	
Packaging Configuration 7 (85- and 100-gallon drums)	85-gallon or 100-gallon drum with filtered inner lid (no inner or liner bags and no rigid liners)	
Packaging Configuration 8 (85- and 100-gallon drums)	Up to 4 inner bags and 2 liner bags, no rigid liner, and filtered inner lid.	

SWB – Standard Waste Box
TDOP – Ten Drum Overpack

Table 6. Package-Specific DAC_3 Values for Solidified Waste (Waste Types I and IV)

Packaging Configuration Group 1						
	Rigid Liner Vent Hole Diameter					
Filter Minimum Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	131	95	37	24	4	4
3.7×10^{-6}	111	85	36	24	4	4
3.7×10^{-5}	28	28	23	19	4	4

Packaging Configuration Group 2						
	Rigid Liner Vent Hole Diameter					
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	213	175	108	92	56	18
3.7×10^{-6}	188	161	105	90	56	17
3.7×10^{-5}	80	80	75	71	49	10

Packaging Configuration Group 3						
	Rigid Liner Vent Hole Diameter					
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	283	243	171	154	107	34
3.7×10^{-6}	253	225	166	151	106	31
3.7×10^{-5}	121	121	115	110	84	13

mol/s/mf = moles/second/moles factor
SWB = Standard Waste Box
TDOP = Ten Drum Overpack

Table 6. Package-Specific DAC_3 Values for Solidified Waste (Waste Types I and IV) (Continued)

Packaging Configuration Group 4	
Pipe Component Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Pipe Component**
1.9×10^{-6}	152

Packaging Configuration Group 5	
Minimum Total Filter Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Direct Load SWB/TDOP**
7.4×10^{-6} (SWB)	15
3.33×10^{-5} (TDOP)	15

Packaging Configuration Group 6	
Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Direct Load SWB/TDOP**
7.4×10^{-6} (SWB)	56
3.33×10^{-5} (TDOP)	56

Packaging Configuration Group 7 [a]			
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Inner Lid Filter Vent Minimum Hydrogen Diffusivity (mol/s/mol fraction)**		
	7.4×10^{-6}	**1.85×10^{-5}**	**9.25×10^{-5}**
3.7×10^{-6}	13	7	2
7.4×10^{-6}	10	6	2
1.85×10^{-5}	6	4	2

mol/s/mf = moles/second/moles factor
SWB = Standard Waste Box
TDOP = Ten Drum Overpack

Table 6. Package-Specific DAC_3 Values for Solidified Waste (Waste Types I and IV) (Continued)

Packaging Configuration Group 8	
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Inner Lid Filter Vent Minimum Hydrogen Diffusivity (mol/s/mol fraction)**
	7.4×10^{-6}
3.7×10^{-6}	21

mol/s/mf = mole/second/moles fraction
SWB = Standard Waste Box
TDOP = Ten Drum Overpack
[a] Headspace sample taken between inner and outer drum lids. If headspace sample is taken inside the filtered inner drum lid prior to placement of the outer drum lid, then a DAC value of 2 days may be used.

Table 7. Package-Specific DAC_3 Values (in days) for Solid Waste (Waste Type II and III)

Packaging Configuration Group 1						
	Liner Lid Opening					
Drum Filter Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	131	95	37	24	4	4
3.7×10^{-6}	111	85	36	24	4	4
3.7×10^{-5}	28	28	23	19	4	4

Packaging Configuration Group 2						
	Liner Lid Opening					
Drum Filter Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	175	138	75	60	30	11
3.7×10^{-6}	152	126	73	59	30	11
3.7×10^{-5}	58	57	52	47	28	8

Table 7. Package-Specific DAC_3 Values (in days) for Solid Waste (Waste Type II and III) (Continued)

Packaging Configuration Group 3						
Liner Lid Opening						
Drum Filter Hydrogen Diffusivity (mol/s/mf)	0.3-inch Diameter Hole	0.375-inch Diameter Hole	0.75-inch Diameter Hole	1-inch Diameter Hole	No Liner Lid	No Liner
1.9×10^{-6}	199	161	96	80	46	16
3.7×10^{-6}	175	148 [a]	93	79	46	16
3.7×10^{-5}	72	72	67	62	42	10

Packaging Configuration Group 4	
Pipe Component Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Pipe Component**
1.9×10^{-6}	152

Packaging Configuration Group 5	
Minimum Total Filter Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Direct Load SWB/TDOP**
7.4×10^{-6} (SWB)	15
3.33×10^{-5} (TDOP)	15

Packaging Configuration Group 6	
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Headspace Sample Taken Inside Direct Load SWB/TDOP**
7.4×10^{-6} (SWB)	56
3.33×10^{-5} (TDOP)	56

mol/s/mf =
SWB = Standard Waste Box
TDOP = Ten Drum Overpack

Table 7. Package-Specific DAC_3 Values (in days) for Solid Waste (Waste Type II and III) (Continued)

Packaging Configuration Group 7 [b]			
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Inner Lid Filter Vent Minimum Hydrogen Diffusivity (mf)**		
	7.4×10^{-6}	**1.85×10^{-5}**	**9.25×10^{-5}**
3.7×10^{-6}	13	7	2
7.4×10^{-6}	10	6	2
1.85×10^{-5}	6	4	2

Packaging Configuration Group 8	
Drum Filter Minimum Hydrogen Diffusivity (mol/s/mf)	**Inner Lid Filter Vent Minimum Hydrogen Diffusivity (mol/s/mol fraction)**
	7.4×10^{-6}
3.7×10^{-6}	21

mol/s/mf =
SWB = Standard Waste Box
TDOP = Ten Drum Overpack
[a] DAC of 142 days is applicable provided that the packaging configuration does not exceed 3 inner bags and 2 liner bags.
[b] Headspace sample taken between inner and outer drum lids. If headspace sample is taken inside the filtered inner drum lid prior to placement of the outer drum lid, then a DAC value of 2 days may be used.

Table 8. Characteristic Ions

Volatile Organic Compound	Primary Ion	Secondary Ion(s)
Acetone	58	43
Benzene	78	52,71
Butanol	56	31,55
Chlorobenzene	112	114,77
Cyclohexane	56	84,41
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	64,98
1,1-Dichloroethylene	96	61,98
cis-1,2-Dichloroethylene	96	61,98
Ethyl benzene	106	91
Ethyl ether	31	59
Methanol	31	-
Methyl ethyl ketone	72	43,57
Methyl isobutyl ketone	100	57,58,43
Toluene	92	91,65
1,2,4-Trimethylbenzene	120	105
1,3,5-Trimethylbenzene	120	105
m-Xylene	106	91
o-Xylene	106	91
p-Xylene	106	91
Chlorobenzene-d5 (IS)	117	82,119
Fluorobenzene (IS)	96	77,70,50

(IS) = Internal Standard

Attachment 1 –Sample Container Data Form (Example)

BDR Number: _____

Waste Container ID		1 Sampling Scenario	2 Waste Type	3 Rigid Liner	4 Rigid Liner Lid	5 Rigid Liner Lid Hole Diameter	6 Number of Inner Bags	7 Number of Liner Bags
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								

Attachment 1 –Sample Container Data Form (Example) (Continued)

BDR Number:_____ Page 2 of 2

Waste Container ID		8	9	10	11	12	13	14	15
		Filter Model Number	Hydrogen Diffusivity of Filter (mol/s/mf)	Package Configuration Group Number	Closure Date	Vent Date	Required DAC Value (in days)	Drum Age (in days)	Can this container be sampled?
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									

Operator: _____ _____ _____

Printed Name Signature Date

Attachment 2 – Flammable Gas Analysis Daily Run Sheet (Example)

BDR Number: _____
Date: _____

Analysis Type	Laboratory File Name	Waste Container ID	DF	Sample/ Standard Pressure (Torr)	Operator Initials

Attachment 3 –Sample Dilution Worksheet (Example)

BDR Number: ________________________
Date:___________________________

Waste Container ID	Initial Volume	Final Volume	Dilution Factor	Operator Initials

Attachment 4 – Flammable Gas Analysis Form (Example)

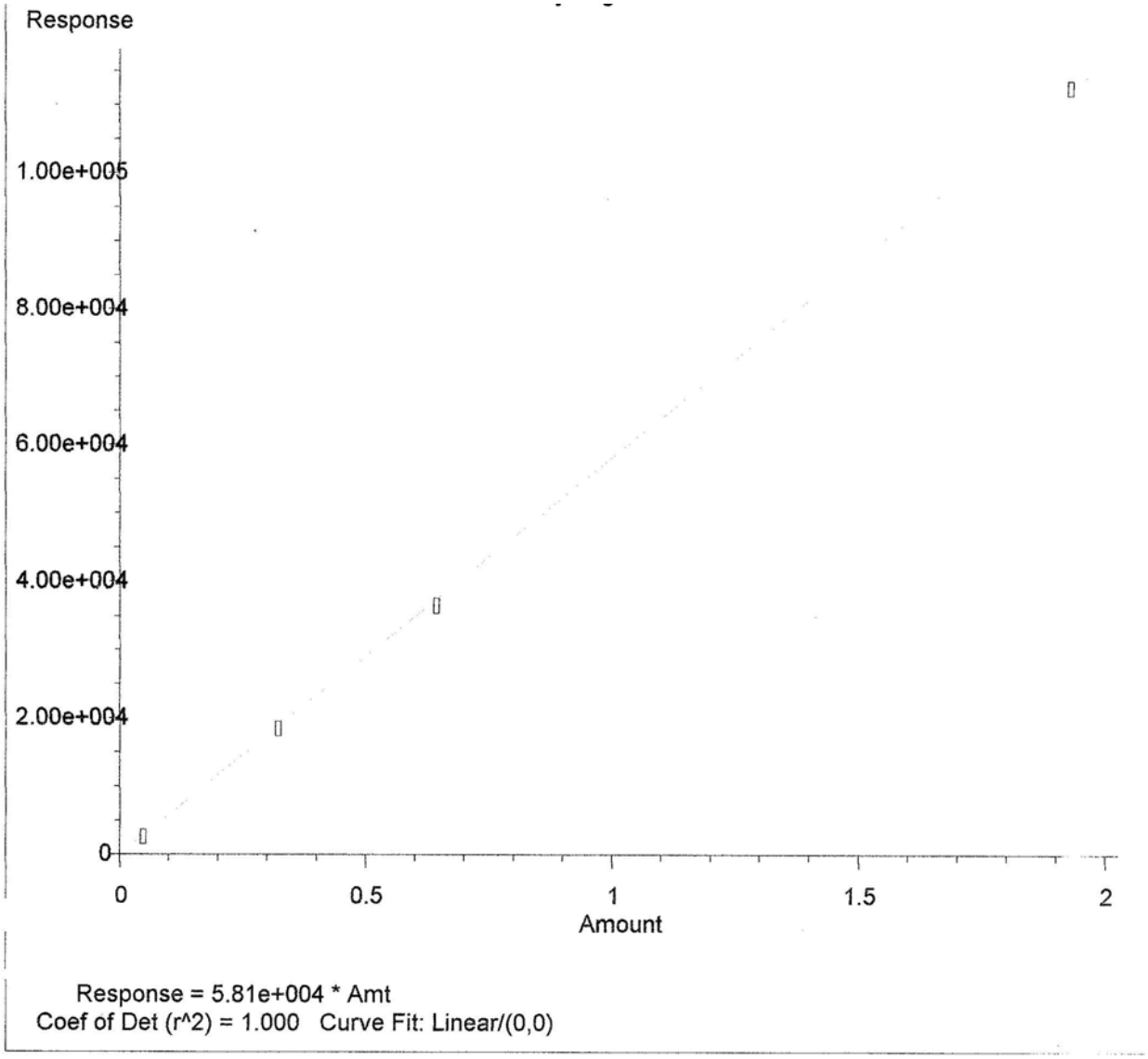
Batch Data Report No.: _____

Waste Container ID: _____

CAS NUMBER	COMPOUND	Measured Volume %	Reporting Flag
1333-74-0	Hydrogen		
74-82-8	Methane		
CAS NUMBER	COMPOUND	Measured ppmv	Reporting Flag
67-56-1	Methanol		
60-29-7	Ethyl Ether		
75-35-4	1,1-Dichloroethylene		
67-64-1	Acetone		
75-34-3	1,1-Dichloroethane		
156-59-2	cis-1,2-Dichloroethene		
78-93-3	Methyl ethyl ketone		
110-82-7	Cyclohexane		
71-43-2	Benzene		
107-06-2	1,2-Dichloroethane		
71-36-3	1-Butanol		
108-10-1	Methyl isobutyl ketone		
108-88-3	Toluene		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
108-38-3/106-42-3	m- and p-Xylene		
95-47-6	o-Xylene		
108-67-8	1,3,5-Trimethylbenzene		
95-63-6	1,2,4-Trimethylbenzene		

CAS NUMBER	IDENTIFIED TIC COMPOUND	Est. Amount ppmv

Attachment 5 – Hydrogen and Methane r^2 Plot Sheet (Example)



Method Name: C:\HPCHEM\1\METHODS\VOCGAS.M
Calibration Table Last Updated: Thu Mar 24 10:25:42 2005

Attachment 6 – Standard Preparation Worksheet (Example)

Standard Cylinder No: _____ Standard Expiration Date:_____

Initial Volume: _____ Final Volume: _____ Dilution Factor: _____

Operators Initial: _____ Date: _____

Target Compound	Initial Standard Concentration	Final Standard Concentration
Benzene		
Chlorobenzene		
Cyclohexane		
1,1-Dichloroethane		
1, 2-Dichloroethane		
1,1-Dichloroethylene		
cis-1,2-Dichloroethylene		
Ethyl benzene		
Ethyl ether		
Toluene		
1,2,4-Trimethylbenzene		
1,3,5-Trimethylbenzene		
m- Xylene		
o- Xylene		
p- Xylene		
Acetone		
1-Butanol		
Methanol		
Methyl ethyl ketone		
Methyl isobutyl ketone		
Hydrogen		
Methane		

Attachment 7 – Data Report Cover Page (Example)

COVER PAGE

BDR Number: _____

NCR Number (if applicable): _____

ICAL BDR Number: _____

MDL BDR Number: _____

TABLE OF CONTENTS

Report Content:

Content	Page Number

_____ _____ _____

Operator Printed Name Signature Date

Independent Technical Reviewer:

_____ _____ _____

Printed Name Signature Date

Attachment 8 – ITR Review Checklist (Example)

Batch Data Report Number: _____

REQUIREMENT	Yes	No	COMMENT
1. Did BFB Tune check meet acceptance criteria of Table 2?			
2. Did CCV meet acceptance criteria for each compound less than or equal to 30 %D?			
3. Were the TICs correctly identified and reported?			
4. Were there any NCRs and is a copy included in the BDR?			
5. Is determination correct for the Drum Age Criteria (DAC) in Attachment 1?			
6. Were the peaks integrated correctly?			
7. Was the correct ICAL used to quantitate the data?			

Independent Technical Reviewer:

_____ _____

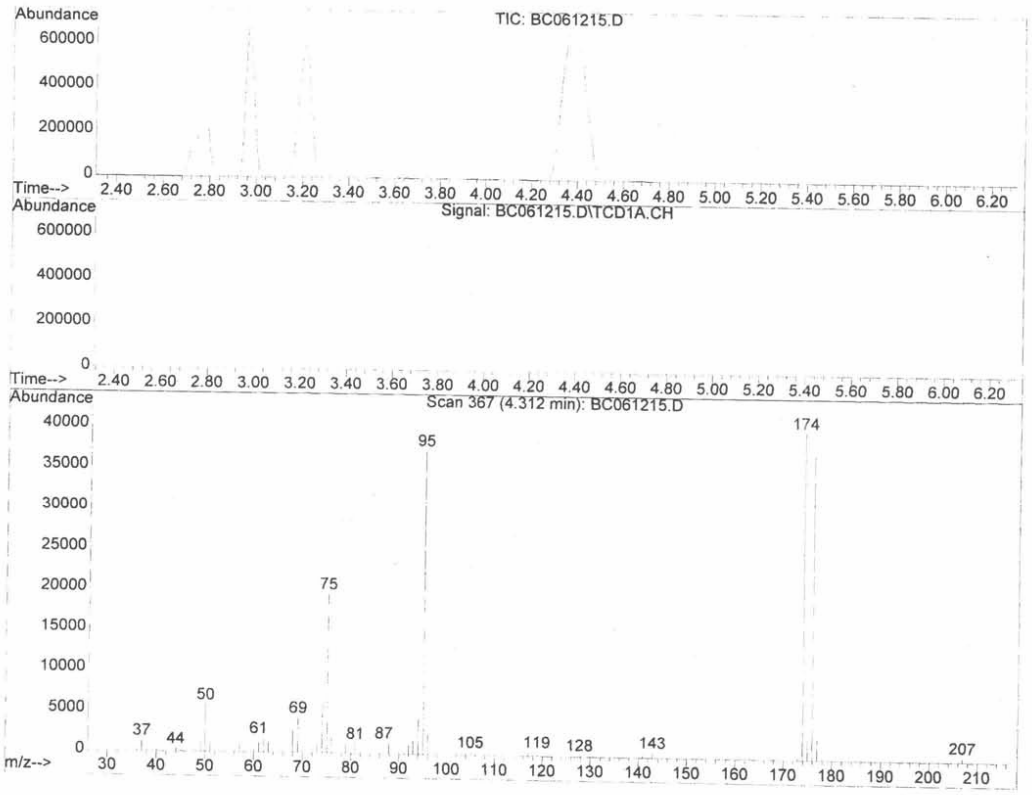
Printed Name Signature Date

Attachment 9 – BFB Tune Evaluation Report (Example)

BFB

Data File : C:\HPCHEM\1\DATA\061215C\BC061215.D
Acq On : 15 Dec 2006 6:17
Sample :
Misc : ALM019796 EXP 8/10/07
Method : C:\HPCHEM\1\METHODS\TUNE.M (RTE Integration)
Title :

Vial: 1
Operator: ec
Inst : UNIT-1
Multiplr: 1.00
Sample Amount: 0.00
Integration Params: rteint2.p



Spectrum Information: Scan 367

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	8	40	15.4	5752	PASS
75	95	30	66	52.5	19568	PASS
95	95	100	100	100.0	37272	PASS
96	95	5	9	7.0	2619	PASS
173	174	0.00	2	0.9	343	PASS
174	95	50	120	107.4	40032	PASS
175	174	5	9	6.3	2522	PASS
176	174	93	101	93.8	37552	PASS
177	176	5	9	6.6	2472	PASS

BC061215.D TUNE.M Fri Dec 15 06:22:49 2006

000003

Attachment 10 – CCV Evaluation Report (Example)

Evaluate Continuing Calibration Report

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

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

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

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

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

050604.M Wed Aug 09 15:31:47 2006

Attachment 11 – Flammable Gas Analysis MDL Spreadsheet (Example)

MDL date:04/25/05			Operator: HLP			c:\hpchem\1\data\050425c									aaaa	
Compound Name	Actual Conc. (ppmv)	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	avg % Rec.	max % Rec	min % Rec.	Std Dev	% RSD	MDL ppmv	Moles analyte	MW g/mol	MDL (ng)
Chloromethane	9.2	9.3	9.5	9.2	9.1	9.2	9.1	9.1	100.16	103.26	98.91	0.15	1.59	0.46	1.49E-12	50.5	0.08
methanol	92.01	91.60	83.78	92.70	87.98	110.80	68.60	92.17	97.45	120.42	74.56	12.56	14.01	39.49	1.28E-10	32	4.11
ethyl ether	9.2	8.40	7.88	7.98	8.12	9.93	6.58	8.10	88.49	107.93	71.52	0.98	12.08	3.09	1.00E-11	74	0.74
1,1-dichloroethylene	9.2	9.50	9.06	9.64	9.31	11.32	7.33	9.34	101.71	123.04	79.67	1.17	12.46	3.66	1.19E-11	97	1.15
1,1,2-trichloro-1,2,2-trifluoroethane	9.2	9.60	8.71	9.60	9.49	11.55	7.16	9.50	101.88	125.54	77.83	1.30	13.92	4.10	1.33E-11	187	2.49
acetone	92.01	104.70	97.51	108.75	105.80	123.72	81.46	104.72	112.82	134.46	88.53	12.69	12.22	39.87	1.30E-10	58	7.51
carbon disulfide	9.2	8.06	7.44	7.96	7.86	9.71	6.05	8.11	85.70	105.54	65.76	1.08	13.68	3.39	1.10E-11	76	0.84
methylene chloride	9.2	9.20	8.83	8.63	8.54	10.68	6.61	8.17	94.19	116.09	71.85	1.22	14.02	3.82	1.24E-11	85	1.05
trans-1,2-dichloroethene	9.2	8.85	7.94	9.07	8.65	10.52	6.69	8.83	94.02	114.35	72.72	1.16	13.43	3.65	1.19E-11	84	1.00
1,1-dichloroethane	9.2	9.43	8.77	9.52	9.43	11.03	7.13	9.47	100.59	119.89	77.50	1.16	12.54	3.65	1.19E-11	99	1.17
cis-1,2-dichloroethene	9.2	10.24	9.68	10.33	10.10	11.90	7.80	10.01	108.79	129.35	84.78	1.21	12.05	3.79	1.23E-11	97	1.19
methyl ethyl ketone	92.01	112.50	106.49	116.43	112.70	133.34	87.30	112.99	121.38	144.92	94.88	13.64	12.21	42.87	1.39E-10	72	10.03
chloroform	9.2	9.50	9.08	9.73	9.34	10.96	7.06	9.10	100.57	119.13	76.74	1.16	12.54	3.65	1.18E-11	119.5	1.42
1,1,1-trichloroethane	9.2	9.11	8.58	9.30	8.89	10.52	6.91	8.86	96.54	114.35	75.11	1.07	12.07	3.37	1.09E-11	133	1.46
cyclohexane	9.2	9.39	8.93	9.47	9.46	11.14	7.29	9.43	101.10	121.09	79.24	1.13	2.65	3.55	1.15E-11	84	0.97
carbon tetrachloride	9.2	9.57	8.96	9.76	9.40	11.10	7.09	9.15	100.98	120.65	77.07	1.19	2.80	3.75	1.22E-11	154	1.88
benzene	9.2	9.86	9.33	10.30	9.84	11.32	7.47	9.51	105.02	123.04	81.20	1.17	12.07	3.67	1.19E-11	78	0.93
1,2-dichloroethane	9.2	10.42	9.66	10.65	10.01	11.56	7.66	9.81	108.34	125.65	83.26	1.20	12.05	3.77	1.23E-11	99	1.21
trichloroethene	9.2	9.63	9.10	9.83	9.65	11.39	7.67	9.68	103.96	123.80	83.37	1.10	11.48	3.45	1.12E-11	131	1.47
1,2-Dichloropropane	9.2	9.30	9.50	9.48	9.60	9.20	9.10	9.50	101.99	104.35	98.91	0.18	1.97	0.58	1.88E-12	113	0.21
butanol	92.01	119.05	114.38	125.76	115.18	131.34	104.51	117.06	128.45	142.75	113.59	8.59	7.27	27.01	8.77E-11	74	6.49
methyl isobutyl ketone	92.01	120.80	108.34	118.90	116.50	126.90	95.01	119.57	125.14	137.92	103.26	10.47	9.09	32.90	1.07E-10	100	10.69
toluene	9.2	9.50	8.81	9.69	9.41	10.69	7.12	9.14	99.94	116.20	77.39	1.09	11.81	3.41	1.11E-11	92	1.02
tetrachloroethene	9.2	10.53	9.59	10.27	10.23	11.99	8.07	10.26	110.16	130.33	87.72	1.17	11.54	3.67	1.19E-11	166	1.98
chlorobenzene	9.2	8.88	7.85	8.69	8.81	10.16	6.83	8.67	93.00	110.43	74.24	1.02	11.93	3.21	1.04E-11	113	1.18
ethylbenzene	9.2	9.37	8.34	9.61	9.57	10.78	7.04	9.05	99.01	117.17	76.52	1.17	12.83	3.67	1.19E-11	106	1.27
m and p xylene	18.4	20.24	18.20	20.09	20.14	23.19	15.20	19.91	106.34	126.03	82.61	2.42	12.39	7.62	2.47E-11	106	2.62
o-xylene	9.2	9.38	8.24	9.51	9.30	10.70	7.08	9.13	98.35	116.30	76.96	1.13	12.48	3.55	1.15E-11	106	1.22
bromoform	9.2	10.26	8.95	10.10	9.93	11.47	7.49	9.60	105.28	124.67	81.41	1.23	12.73	3.87	1.26E-11	253	3.18
1,1,2,2-tetrachloroethane	9.2	9.56	8.15	9.40	9.29	10.69	6.89	9.19	98.09	116.20	74.89	1.20	13.28	3.77	1.22E-11	168	2.06
1,3,5-trimethylbenzene	9.2	9.59	8.59	9.74	9.34	10.61	6.97	9.44	99.81	115.33	75.76	1.14	12.46	3.60	1.17E-11	120	1.40
1,2,4-trimethylbenzene	9.2	9.77	8.40	9.78	9.50	10.68	7.27	9.36	100.56	116.09	79.02	1.10	11.93	3.47	1.13E-11	120	1.35
		vol%	vol%	vol%	vol%	vol%	vol%	vol%	avg %	max %	min %	Std Dev	% RSD	MDLvol			
hydrogen	0.075	0.07705	0.0716	0.0748	0.0711	0.071	0.0675	0.0683	95.50	102.73	90.00	0.00	4.71	0.01			
methane	0.075	0.0776	0.0737	0.0764	0.0755	0.0763	0.0727	0.078	100.99	104.00	96.93	0.00	2.57	0.01			

Ambient Pressure (Torr)	626.50
Correction Factor/Pressure	1.21
Bottle Temperature (Celcius)	36.00
Bottle Temperature (Kelvin)	309.00
Correction Factor/Temp	1.13
Molar Gas Volume (mls)	30775.68

Attachment 12 – ICAL Response Factor Report (Example)

Response Factor Report GB-01

Method : C:\HPCHEM\1\METHODS\VOCGAS.M (RTE Integrator; Aligned)
Title : TRU HGAS VOA Compounds
Last Update : Thu Mar 24 10:25:42 2005
Response via : Initial Calibration

Calibration Files
1 =002I1002.D 2 =003I2003.D 3 =004I3004.D
4 =005I4005.D 5 =006I5006.D

		Compound	1	2	3	4	5	Avg	%RSD
1)	T	Hydrogen	5.491	5.744	5.659	5.829		5.681 E4	2.53
2)	T	Methane	2.502	2.686	2.633	2.647		2.617 E4	3.05
3)	I	Fluorobenzene	----------ISTD----------						
4)	T	Methanol	0.176	0.155	0.161	0.151	0.156	0.160	5.97
5)	T	Ethyl ether	0.615	0.552	0.571	0.546	0.569	0.571	4.72
6)	T	1,1-Dichloroethene	0.375	0.319	0.318	0.308	0.306	0.325	8.68
7)	T	1,1,2-Trichloro-1,2	0.911	0.763	0.705	0.665	0.620	0.733	15.39
8)	T	Acetone	0.218	0.178	0.170	0.161	0.141	0.174	16.24
9)	T	Carbon Disulfide	1.114	0.925	0.891	0.850	0.874	0.931	11.37
10)	TL	Methylene chloride	0.359	0.295	0.293	0.272	0.279	0.300	11.54
11)	T	trans-1,2-Dichloroe	0.381	0.295	0.306	0.293	0.316	0.318	11.42
12)	T	1,1-Dichloroethane	0.787	0.632	0.624	0.585	0.627	0.651	12.00
13)	T	cis-1,2-Dichloroeth	0.403	0.339	0.334	0.326	0.327	0.346	9.36
14)	T	Methyl ethyl ketone	0.203	0.165	0.155	0.147	0.133	0.160	16.33
15)	TL	Chloroform	0.897	0.750	0.732	0.685	0.726	0.758	10.75
16)	T	1,1,1-Trichloroetha	0.904	0.731	0.739	0.701	0.768	0.768	10.34
17)	T	Cyclohexane	0.620	0.520	0.554	0.549	0.578	0.564	6.63
18)	TL	Carbon Tetrachlorid	1.022	0.838	0.847	0.791	0.871	0.874	10.08
19)	TL	Benzene	1.187	0.970	0.973	0.955	0.962	1.010	9.87
20)	TL	1,2-Dichloroethane	0.631	0.529	0.524	0.492	0.531	0.541	9.71
21)	T	Trichloroethene	0.534	0.439	0.447	0.435	0.441	0.459	9.18
22)	T	Butanol	0.393	0.319	0.333	0.314	0.263	0.324	14.39
23)	I	Chlorobenzene-d5	----------ISTD----------						
24)	T	Methyl isobutyl ket	0.163	0.132	0.122	0.117	0.110	0.129	16.13
25)	TL	Toluene	0.972	0.838	0.861	0.846	0.850	0.873	6.37
26)	T	Tetrachloroethene	0.811	0.651	0.696	0.678	0.728	0.713	8.65
27)	T	Chlorobenzene	1.474	1.152	1.160	1.128	1.119	1.207	12.47
28)	T	Ethylbenzene	0.631	0.528	0.565	0.569	0.583	0.575	6.50
29)	T	m and p-xylene	0.793	0.669	0.706	0.702	0.699	0.714	6.52
30)	T	o-xylene	0.642	0.585	0.664	0.668	0.706	0.653	6.78
31)	T	Bromoform	0.610	0.505	0.545	0.532	0.546	0.548	7.04
32)	T	1,1,2,2-Tetrachloro	0.885	0.708	0.734	0.709	0.685	0.744	10.86
33)	T	1,3,5-Trimethylbenz	0.780	0.601	0.800	0.819	0.857	0.771	12.87
34)	T	1,2,4-Trimethylbenz	0.773	0.563	0.770	0.782	0.813	0.740	13.60

(#) = Out of Range

VOCGAS.M Fri Aug 18 12:32:05 2006

Operator
Printed Name _____ Signature _____ Date _____

ITR
Printed Name_____ Signature _____ Date _____

Attachment 13 – Quantitation Report (Example)

Quantitation Report (Not Reviewed)

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

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

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

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