Waste Isolation Pilot Plant Flammable Gas Analysis

May 2009

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Flammable Gas Analysis

May 2009

Revision 3.1

Approved by: //signature on file// Date: 07 April 2009
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RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	11/6/2005	Initial Use.
1	12/28/2005	Added RRF equation and editorial changes.
1.1	1/10/2007	Editorial correction to step 4.2.4[T].
1.2	3/19/2007	Change to allow SRS & Hanford to perform procedure at sea level and other editorial corrections.
2	12/6/07	Added step to allow for dilution of analyte and made other various operational efficiencies.
2.1	2/21/08	Added use of Appendix C of the CH-TRUCON document for DAC selection.
3	03/26/09	Changed to allow for computer and software upgrade.
3.1	04/07/09	Changed to incorporate editorial corrections.

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1.0 PURPOSE

This document describes and implements the process to determine 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), HalfPACT, or RH-TRU 72-B packagings. An aliquot of headspace gas (HSG) is sampled from a waste container and analyzed using a gas chromatograph (GC) with two detectors: a mass spectrometer (MS), and a thermal conductivity detector (TCD). The sample analyzed in the GC is split and half is sent to the MS for volatile organic compound (VOC) analysis while the other half is sent to the TCD for hydrogen and methane analysis.

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) and the Remote-Handled Transuranic Waste Authorized Methods for Payload Control (RH-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 CH TRAMPAC Evaluation Software (CHTES) or the RH TRAMPAC Evaluation Software (RHTES). The target analytes are listed in Table 1, Gas Target Analyte List.

For contact-handled transuranic (CH-TRU) waste, 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 TRUPACT-II content (CH-TRUCON) codes document, then CHTES will accept the new compound.

For remote-handled transuranic (RH-TRU) waste, measured compounds are restricted in the gas generation limits established during the development and approval of the RH-TRUCON codes document describing the containers. The RHTES will accept only containers with approved RH-TRUCON codes assigned.

2.0 REQUIREMENTS

2.1 Training Requirements

2.1.1 Personnel performing this procedure will be trained and qualified in accordance with U.S. Department of Energy (DOE), *Quality Assurance Program Document*, DOE/CBFO 94-1012.

2.2 Equipment List

2.2.1 Analytical System

[A] GC with MS and TCD detector.

[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 MS and TCD data obtained throughout the duration of the chromatographic program is interfaced to the GC/MS-GC/TCD system. The data system software 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 searching the 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 standards shall be procured as certified gas standards. The gas standards shall contain the target analytes listed in Table 1.

[A.3] The internal standard (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] The tune check gas mixture contains 4-Bromofluorobenzene (BFB) in nitrogen. The tune check gas mixture will be procured at a concentration that will deliver approximately 50 nanograms (ng) of BFB to the MS.

[B] Sampling Equipment

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

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

[B.3] 50 milliliter (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 and the Lead Operator (LO) will be notified. 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 Certified gas standards must not be used past their expiration dates.

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 to 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 (r2) requirement for an ICAL and when meeting the %D requirement for CCV.

2.5.3 Representativeness – Representativeness of HSG sampling and analysis is ensured by U.S. Department of Energy (DOE) Carlsbad Field Office (CBFO) issuance of this technical standard operating procedure.

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 by CBFO issuance of this technical standard operating procedure.

2.6 Quality Control (QC) Requirements

2.6.1 BFB Tune Check – BFB is introduced into the 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%. 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 or equal to 0.990. The r^2 is calculated using the following equation:

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

Where:

r^2 = the coefficient of determination

x_i = is the concentration

y_i = is the area response

2.6.3 Continuing calibration verification (CCV) – The CCV standard is analyzed at the start of the operational shift, but after the BFB tune check. The CCV standard must be from a different supplier source or lot than the standard used for the ICAL. The IS responses must be within 50–200% of the IS response for the initial calibration mid-point standard. Internal standard retention time (RT) must not shift by ≥0.5 minute 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 30.0. 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% 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% t-statistic,

n = the number of observations

s = 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 parts per million by volume (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 1000 ppmv.

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:

[A] U = Target analyte was not detected and concentration reported as the MDL

[B] 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 10%.

3.0 RESPONSIBILITIES

3.1 Vendor Project Manager (VPM)/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 containers.

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)

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

Performs container evaluation using CHTES or RHTES.

4.0 PROCEDURE

NOTE

Meeting the drum age criteria (DAC) requirement as specified herein is only required when sampling and analyzing CH-TRU waste for VOCs only or for VOCs and hydrogen and methane together. The DAC requirement is not required for hydrogen and methane analysis alone. The DAC requirement specified herein is applicable only to CH-TRU waste.

4.1 Determining If Waste Container Can Be Sampled

NOTE

Reference Attachment 1 and collect necessary information as needed.

NOTE

This section may be completed simultaneously with Section 4.4, Sampling and Analysis, so long as the waste container's DAC is completed prior to sampling of an individual container of CH-TRU waste.

4.1.1 If sampling for hydrogen and methane only or RH-TRU waste then GO TO step 4.2.

4.1.2 Determine the correct sampling scenario to establish the CH-TRU DAC by performing the following analysis:

- CH-TRU waste DACs are defined for three unique venting and sampling scenarios. These 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 t_1 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]; or Appendix C of the CH-TRUCON document). Under Scenario 3, DAC values also may be established and published in Appendix C of the CH-TRUCON document. For containers assigned to approved codes with specified DAC values, the CH-TRUCON Appendix C DAC values are used.

NOTE

CH-TRU waste containers with Scenarios 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 CH-TRU DAC 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, II, III, or 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 CH-TRU DAC Scenario 2,
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 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 Rigid Liner Lid Hole Diameter as 0.30, 0.375, 0.75, or 1.0 inch, or enter 1.0 inch if no rigid liner is present.

[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 CH-TRU DAC 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, II, III, or 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 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, or enter NA if using approved CH-TRUCON code-specific DAC value.

[M] Closure Date.

[N] Vent Date.

[O] Required DAC Value (in days) from Table 6 or Table 7, or enter applicable DAC value from Appendix C of the CH-TRUCON document.

[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 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:\msdchem\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 METHOD.

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

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR NAME field.

[H] In the DATA PATH field enter c:\msdchem\1\data\YYMMDDI, where YY is year, MM is month, and DD is day. In the DATA FILE NAME field enter BIYYMMDD.d, **AND** in the miscellaneous box enter the cylinder number and expiration date.

[I] Click OK AND RUN METHOD button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** when GC/MS is ready, 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:\MSDCHEM\1\Methods, select Tune.m, **AND** click OK.

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

[S] From the directory c:\MSDCHEM\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 BFB, select EVALUATE TO PRINTER, **AND** click OK.

[V] **IF** the key ion abundance criteria in Table 2 are met,
THEN proceed to 4.2.2.

[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.

4.2.2 VOCGAS.m

[A] OPEN Analysis.

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

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

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

[E] From pull down menu INITIAL CALIBRATION, select EDIT COMPOUNDS.

[F] Select FLUOROBENZENE.

[G] 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.

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

[I] Click CALIBRATION.

[J] In the Conc column, check to see if the IS concentrations in Levels 1 through 5 are the same as on the Identification page.

[K] **IF** the Concentration is not the same,
THEN make the correction.

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

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

[N] Click IDENTIFICATION.

[O] Repeat steps 4.2.2[F] 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.5] for each target compound:

[P.1] Select COMPOUND.

[P.2] Click CALIBRATION.

[P.3] In the column CONCENTRATION, for each LVL ID row used, compare the different entered levels of ICAL concentrations to the ICAL concentrations to be used, **AND** ensure that CC entered values are the same as level 3.

[P.4] For hydrogen and methane only, in column CONCENTRATION for LVL ID CC , ensure the CC concentration is the same as the LVL ID 3 concentration.

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

[Q] Click OK.

[R] Click OK.

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

[T] Save method as VOCGAS.m.

[U] Click OK.

[V] 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 formula:

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] **IF** VOCGAS.m is already loaded,
THEN go to step 4.2.3[J]

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

[H] From the directory c:\MSDCHEM\1\Methods, select VOCGAS.m.

[I] Click OK.

[J] Click on GREEN ARROW.

[K] Enter initials in OPERATOR NAME field.

[L] In the DATA PATH field enter c:\msdchem\1\data\YYMMDDI, where YY is year, MM is month, and DD is day; in the DATA FILE NAME field enter ICALX.d, where X is a standard level; **AND** in the miscellaneous box enter the cylinder number and expiration date.

[M] Click OK AND RUN METHOD Button.

[N] Using a suitable syringe, flush sample loop with approximately 30 to 50 milliliter (ml) of nitrogen or air.

[O] Attach standard syringe to inlet.

[P] OPEN IS valve.

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

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

[S] START GC/MS when GC/MS is ready.

[T] CLOSE IS valve.

[U] Remove standard syringe.

[V] Enter the following on Attachment 2:

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

[V.2] Laboratory File Name.

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

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

[V.5] Sample/Standard Pressure (Torr or mmHg).

[V.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:\MSDCHEM\1\Data\YYMMDDI\, select ICAL1.d, **AND** click OK.

[D] From the pull down menu QUANTITATE, select CALCULATE/GENERATE Report, select SUMMARY TO SCREEN, deselect PRINTER, if applicable, **AND** click OK.

[E] Exit MULTIVU.

[F] From pull down menu QUANTITATE, select QEdit QUANT Results.

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

[H] Select EXIT.

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

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

[K] In the QUANTITATE REPORT OPTIONS dialog box, select SUMMARY and DESTINATION PRINTER, deselect SCREEN, if applicable, **AND** click OK.

[L] From pull down menu INITIAL CALIBRATION, select UPDATE LEVELS.

[M] Select UPDATE LEVEL in UPDATE CALIBRATION dialog box.

[N] Select UPDATE LEVEL RESPONSES, REPLACE, **AND** level 1.

[O] **IF** doing ICAL3,
THEN select RETENTION TIMES, REPLACE, **AND** REPLACE QUALIFIER ION RELATIVE RESPONSES.

[P] **IF NOT** doing ICAL3
THEN unselect RETENTION TIMES and REPLACE QUALIFIER ION RELATIVE RESPONSES.

[Q] Click DO UPDATE.

[R] Click OK.

[S] Click OK.

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

[U] From the pull down menu INITIAL CALIBRATION, select RESPONSE FACTORS TO PRINTER.

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

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

[X] From the pull down menu INITIAL CALIBRATION, select EDIT COMPOUNDS.

[Y] Select HYDROGEN.

[Z] Select CALIBRATION.

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

[BB] **IF** r^2 is less than 0.990 for hydrogen
THEN contact LO,
AND repeat the individual standard level or entire ICAL, as appropriate.

[CC] If the r^2 is greater than or equal to 0.990 for hydrogen, then proceed.

[DD] Click ALT-Print Screen, paste into a word document program, and print image from word document program.

[EE] Repeat steps 4.2.4[Y] through 4.2.4[DD], substituting methane for hydrogen.

[FF] Select OK.

[GG] Select OK.

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

[II] Save method as VOCGAS.m.

[JJ] Click OK.

4.2.5 ICAL Report

[A] Assemble the 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.

[A.9] Certificate of Analysis (COA) for ICAL standard.

[A.10] COA for IS.

[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.

4.2.6 Data Review

ITR

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

[A.1] BFB tune check met the acceptance criteria of Table 2.

[A.2] ICAL standards for target VOCs, hydrogen and methane, and IS were updated correctly into VOCGAS.m and, for target VOCs and ISs, there are no concentrations or area responses entered in the cc fields.

[A.3] Concentrations of ISs and target analytes for all levels used for ICAL standard analysis are entered correctly.

[A.4] The %RSD for all VOCs is less that 35%.

[A.5] The r^2 is greater than or equal to 0.990 for hydrogen and methane.

[B] Notify the operator of all errors or omissions found during the review, if applicable.

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

[D] Print name, sign, and date Data Report cover page.

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

NOTE

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

4.3 MDL

Operator

4.3.1 Tune Check

[A] On the computer, create new folder called c:\msdchem\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 METHOD.

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

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR NAME field.

[H] In the DATA PATH field, enter c:\msdchem\1\data\YYMMDDM, where YY is year, MM is month, and DD is day, in the DATA FILE NAME field, enter BMYYMMDD.d, **AND** in the miscellaneous box, enter the cylinder number and expiration date.

[I] Click OK and RUN METHOD button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** when GC/MS is ready, 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:\MSDCHEM\1\Methods, select Tune.m, **AND** click OK.

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

[S] From the directory c:\MSDCHEM\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 BFB, SELECT Evaluate to Printer, **AND** click OK.

[V] **IF** the key ion abundance criteria in Table 2 are met, **THEN** proceed to 4.3.2.

[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.

4.3.2 Analyze MDL

[A] OPEN Analysis.

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

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

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

[E] **IF** it is necessary to prepare an MDL standard less than the stock calibration standard,
THEN calculate the DF using the following formula:

DF = initial volume / final volume

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

[G] Perform serial dilutions, if necessary.

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

[H.1] Date.

[H.2] Standard Cylinder Number.

[H.3] Stock Standard Expiration Date.

[H.4] Initial Standard Concentration.

[H.5] Initial Volume.

[H.6] Final Volume.

[H.7] Dilution Factor.

[H.8] Final Standard Concentration.

[H.9] Operator Initials.

[I] OPEN HGAS 05.

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

[K] From the directory c:\MSDCHEM\1\Methods, select VOCGAS.m.

[L] Click OK.

[M] Click on GREEN ARROW.

[N] Enter initials in OPERATOR NAME field.

[O] In the DATA PATH field, enter c:\msdchem\1\data\YYMMDDM, where YY is year, MM is month, and DD is day, in the DATA FILE NAME field, enter MDLX.d, where X is the MDL standard, **AND** in the miscellaneous box, enter the cylinder number and expiration date.

[P] Click OK and RUN METHOD button.

[Q] Using a suitable syringe, flush sample loop with approximately 30 to 50 ml of nitrogen or air.

[R] Attach standard syringe to inlet.

[S] OPEN IS valve.

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

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

[V] START GC/MS when GC/MS is ready.

[W] CLOSE IS valve.

[X] Remove standard syringe.

[Y] Enter the following on Attachment 2:

[Y.1] MDL for Analysis Type.

[Y.2] Laboratory File Name.

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

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

[Y.5] Sample/Standard Pressure (Torr or mmHg).

[Y.6] Operator Initials.

[Z] After analysis is finished, OPEN ANALYSIS.

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

[BB] From the directory c:\MSDCHEM\1\Data\YYMMDDM\, select MDL1.d, **AND** click OK.

[CC] From the pull down menu QUANTITATE, select CALCULATE/GENERATE REPORT, SELECT SUMMARY TO SCREEN, deselect PRINTER, if applicable, **AND** click OK.

[DD] Exit MULTIVU.

[EE] From pull down menu QUANTITATE, 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 QUANTITATION 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 and destination printer, deselect SCREEN, if applicable, **AND** click OK.

[KK] Repeat steps 4.3.2[E] 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 **AND** repeat the individual MDL or entire MDL, as appropriate.

[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:\MSDCHEM\1\Methods, select VOCGAS.m and click OK.

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

[J] Starting with Target Compound 1, repeat steps 4.3.4[J.1] through 4.3.4[J.3] for each target compound:

[J.1] Select COMPOUND.

[J.2] Click REPORTING.

[J.3] In the water (conc) row and MDL column, enter MDL value **OR** verify MDL value is correct.

[K] Click OK.

[L] Click OK.

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

[N] Save method as VOCGAS.m

[O] Click OK.

[P] Print name, sign, and date the Data Report cover page.

[Q] Submit to operator.

Operator

[R] Repeat steps 4.3.4[E] through 4.3.4[P], **THEN** return to step 4.3.4[S].

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

4.4 Sampling and Analysis

4.4.1 Tune Check

[A] On the computer, create new folder called c:\msdchem\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 METHOD.

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

[E] Click OK.

[F] Click on GREEN ARROW.

[G] Enter initials in OPERATOR NAME field.

[H] In the DATA PATH field enter c:\msdchem\1\data\YYMMDDC, where YY is year, MM is month, and DD is day, in the DATA FILE NAME field enter BCYYMMDD.d, **AND** in the miscellaneous box enter the cylinder number and expiration date.

[I] Click OK and RUN METHOD button.

[J] OPEN BFB valve.

[K] Wait approximately 15 seconds, **AND** when GC/MS is ready, 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:\MSDCHEM\1\Methods, select Tune.m, **AND** click OK.

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

[S] From the directory c:\MSDCHEM\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 BFB SELECT EVALUATE TO PRINTER, **AND** click OK.

[V] **IF** the key ion abundance criteria in Table 2 are met, **THEN** proceed to 4.4.2.

[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, **AND** rerun BFB Tune Check.

4.4.2 CCV

[A] OPEN HGAS 05.

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

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

[D] Click OK.

[E] Click on GREEN ARROW.

[F] Enter initials in OPERATOR NAME field.

[G] In the DATA PATH field enter c:\msdchem\1\data\YYMMDDC, where YY is year, MM is month, and DD is day, and in the DATA FILE NAME field enter CCYYMMDD.d.

[H] **IF** using a CCV cylinder, **THEN** enter the cylinder number and expiration date in the miscellaneous box.

[I] **IF** using an ICAL cylinder for the CCV, **THEN** in the miscellaneous box enter the cylinder number, expiration date, project number or lot number, and DF.

[J] **IF** using an ICAL cylinder for the CCV, **ENSURE** the project number or lot number for the current ICAL cylinder is different than the project number or lot number for ICAL cylinder used as the CCV.

[K] Click OK and RUN METHOD button.

[L] Using a suitable syringe, flush sample loop with approximately 30 to 50 ml of nitrogen or air.

[M] Attach standard syringe to inlet.

[N] OPEN IS valve.

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

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

[Q] START GC/MS when GC/MS is ready.

[R] CLOSE IS valve.

[S] Remove standard syringe.

[T] Enter the following on Attachment 2:

[T.1] CCV for Analysis Type.

[T.2] Laboratory File Name.

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

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

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

[T.6] Operator Initials.

[U] After analysis is finished, OPEN ANALYSIS.

[V] From the pull down menu METHOD, select LOAD METHOD **AND** click YES

[W] From the directory c:\MSDCHEM\1\Methods, select VOCGAS.m.

[X] Click OK.

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

[Z] From the directory c:\MSDCHEM\1\Data\YYMMDDC\, select CCYYMMDD.d **AND** click OK.

[AA] From the pull down menu QUANTITATE, select CALCULATE/GENERATE REPORT, select SUMMARY TO SCREEN, deselect PRINTER, if applicable, **AND** click OK.

[BB] Exit MULTIVU.

[CC] From pull down menu QUANTITATE, select QEdit QUANT Results.

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

[EE] Select EXIT.

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

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

[HH] In the QUANTITATE REPORT OPTIONS dialog box, select SUMMARY and destination printer, deselect SCREEN, if applicable, **AND** click OK.

[II] From pull down menu CONTINUING CALIBRATION, select EVALUATE DATA FILE AS CONTINUING CAL TO PRINTER.

[JJ] **IF** the %D for each of the target compounds in the CCV is less than or equal to 30.0%, **IF** the AvgRF listed on CCV report is the same (within ±0.001) of the Average listed on the current Response Factor Report of the ICAL, **IF** the internal standard responses are within 50-200% of the internal standard response of current ICAL3, **AND** **IF** the internal standard RT has not shifted by >0.5 minute from the RT of the current ICAL3,
THEN proceed.

[KK] **IF** the %D for any of the target compounds in the CCV is greater than 30.0%, **IF** the AvgRF listed on CCV report is not the same (within ±0.001) of the Average listed on the current Response Factor Report of the ICAL, **IF** the internal standard responses are not within 50-200% of the internal standard response for the current ICAL3,
OR IF the internal standard RT has shifted by >0.5 minute from the RT of the current ICAL3,
THEN contact the LO, **AND** repeat CCV.

4.4.3 Sampling

[A] Flush a 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, disposable filter(s) 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.

[I] Discard the disposable filter(s).

NOTE

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

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

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

NOTE

Appropriate steps in 4.4.3 may be repeated in order to take multiple samples before analysis. Multiple samples may be taken if the samples are taken by the operator who will be performing the analysis or if the operator is under control (visual) of the operator who will be performing analysis.

[L] Take sample to analytical instrument.

[M] Open HGAS 05.

[N] Click on GREEN ARROW.

[O] Enter initials in OPERATOR NAME field.

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

[P.1] In the DATA FILE NAME field, enter X.d, where X is the Waste Container ID.

[P.2] 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 analysis is always performed the same day as sampling.

[Q] **IF** this is a sample of a waste container which has been diluted,
THEN in the DATA FILE NAME field, enter diluteN.d, where N is the dilution number, and in the miscellaneous box, enter waste container ID and DF.

[R] Click OK and RUN METHOD button.

[S] Using a suitable syringe, flush with nitrogen or air to clear sample loop.

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

[U] OPEN IS valve.

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

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

[X] START GC/MS when GC/MS is ready.

[Y] CLOSE IS valve.

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

[AA] Enter the following on Attachment 2:

[AA.1] Sample for Analysis Type.

[AA.2] Laboratory File Name.

[AA.3] Waste Container ID.

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

[AA.5] Sample introduction pressure for Sample/Standard Pressure (Torr or mmHg).

[AA.6] Operator Initials.

[BB] After analysis is finished, OPEN ANALYSIS.

[CC] **IF** VOCGAS.m is already loaded,
THEN GO TO step 4.4.3[GG].

[DD] From the pull down menu METHOD, select LOAD METHOD and click Yes.

[EE] From the directory c:\MSDCHEM\1\Methods, select VOCGAS.m.

[FF] Click OK.

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

[HH] From the directory c:\MSDCHEM\1\Data\YYMMDDC\, select X.d or diluteN.d, **AND** click OK.

[II] From the pull down menu QUANTITATE, select CALCULATE/GENERATE REPORT, SELECT SUMMARY TO SCREEN, deselect PRINTER, if applicable, **AND** click OK.

[JJ] Exit MULTIVU.

[KK] From pull down menu QUANTITATE, select QEdit QUANT Results.

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

[MM] Select EXIT.

[NN] Select YES or NO in the SAVE CHANGES TO QUANTITATION RESULTS? dialog box, as appropriate.

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

[PP] In the QUANT REPORT OPTIONS dialog box, select DETAILED, DESTINATION PRINTER, **AND** click OK.

[QQ] **IF** the IS responses are not within 50–200% of the IS responses for current CCV, or the IS RT has shifted by >0.5 minute from the RT of the current CCV and are due to Matrix effects,
THEN document on the waste container quantitation report that the IS and/or RT does not meet the requirement due to Matrix effects,
ELSE contact LO.

NOTE

When a reportable tentatively identified compound (TIC) is present at a concentration causing the detector to be saturated, then a dilution must be performed to establish the estimated TIC concentration.

[RR] **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 formula so the analyte on reanalysis will be approximately in the mid range of the calibration curve.

DF = initial volume / final volume.

[SS] Perform serial dilutions, if necessary.

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

[TT.1] BDR Number (only during first sample).

[TT.2] Date (only during first sample).

[TT.3] Waste Container ID.

[TT.4] Initial Volume.

[TT.5] Final Volume.

[TT.6] Dilution Factor.

[TT.7] Operator Initials.

[UU] **IF** the waste container sample was diluted, repeat steps 4.4.3[M] through 4.4.3[TT],
ELSE repeat steps 4.4.3[A] through 4.4.3[TT] 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

TIC is equivalent to 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 % of the nearest IS.

NOTE

IF the unknown has a total ion area greater than 10% 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% 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% 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% 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 of co-eluting compounds,
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks.

NOTE

The concentration of the reportable TIC compound shall be estimated using the IS 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 the IS and then dividing by the area count of the IS.

[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 a National Fire Protection Association (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

NOTE

Steps 4.6.1 and 4.6.2 can be performed any time (e.g., after each analysis is completed or after all the analyses are complete).

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] Measured ppmv or MDL, whichever is appropriate, and reporting "Flag."

4.6.2 **IF** TICs are reportable,
THEN report on the 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.

[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 nonconformance report (NCR), 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** verify data evaluation (Step 4.5).

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 CHTES or RHTES.

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 the 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 the 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 CHTES or RHTES 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 CHTES or RHTES container evaluation is run successfully, the user will receive a summary of the results of the evaluation as shown in Figure 9-3, CHTES or RHTES 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

[C] BDR

5.1.2 QA/Lifetime Records

[A] None.

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.\

6.4 U.S. Department of Energy (DOE). *Remote-Handled Transuranic Waste Authorized Methods for Payload Control (RH-TRAMPAC)*, NRC Docket No. 71-9212, 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. CH-TRU DAC_1 Values (in days)

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

Note: CH-TRU Waste 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. CH-TRU 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. CH-TRU 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. CH-TRU 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/mole fraction
SWB = Standard Waste Box
TDOP = Ten-Drum Overpack

Table 6. CH-TRU 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/mole 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 6. CH-TRU 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/mole fraction
SWB = Standard Waste Box
TDOP = Ten-Drum Overpack

Table 7. CH-TRU 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. CH-TRU 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 = moles/second/mole fraction
SWB = Standard Waste Box
TDOP = Ten-Drum Overpack

Table 7. CH-TRU 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

[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.

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 = moles/second/mole fraction
SWB = Standard Waste Box
TDOP = Ten-Drum Overpack

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

NOTE: *The DAC requirement specified herein is applicable to CH-TRU waste only. The equivalent requirement for RH-TRU waste is determined and compliance is ensured outside this procedure. “NA – RH” may be entered in these fields to indicate that these data are not applicable for RH-TRU waste containers.

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:___________________________

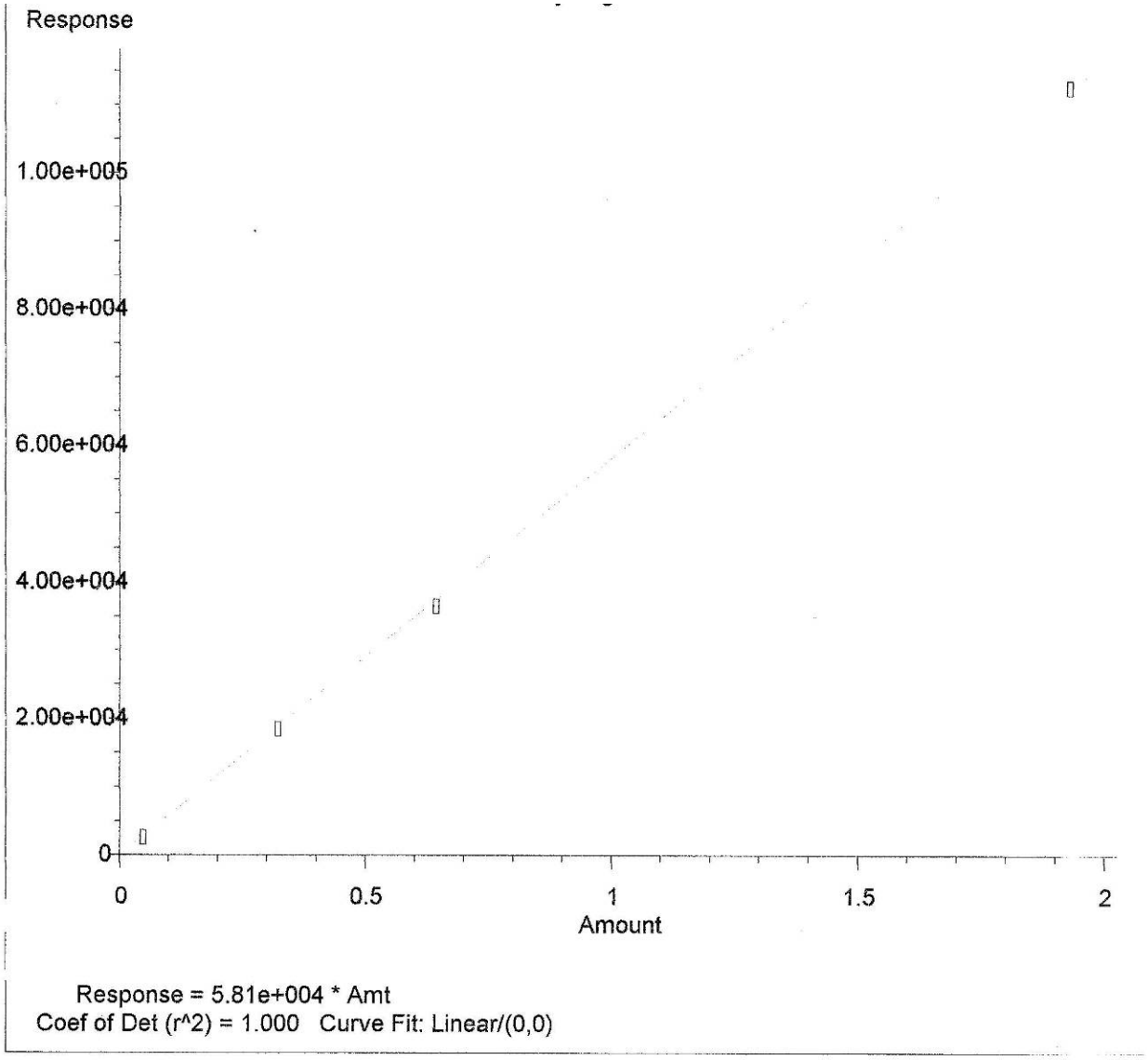
Waste Container ID	Initial Volume	Final Volume	Dilution Factor	Operator Initials

Attachment 4 – Flammable Gas Analysis Form (Example)
Batch Data Report No.:_____
Waste Container ID: _____

CAS NUMBER	COMPOUND	Measured ppmv	Reporting Flag
1333-74-0	Hydrogen		
74-82-8	Methane		
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 Checklist (Example)

Batch Data Report Number: _____

REQUIREMENT	Yes	No	N/A	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?				
8. For CCV are the IS responses within 50–200 percent of the IS response for current mid ICAL standard?				
9. For each sample are the IS responses within 50–200 percent of the IS response for current CCV?				
10. For each sample are the IS RT <0.5 min from the RT of the current CCV?				
11. Are all Flammable Gas Analysis Form(s) attached for all drums reported on Attachment 2?				
12. Are the appropriate data qualifier flags assigned to each drum sample?				
13. Does the concentration reported on the Quantitation Report (excluding diluted samples) match what is reported on Attachment 4?				
14. If a sample is diluted was the dilution calculated correctly?				
15. Were the correct MDL used on Attachment 4?				

Independent Technical Reviewer:

_____ _____

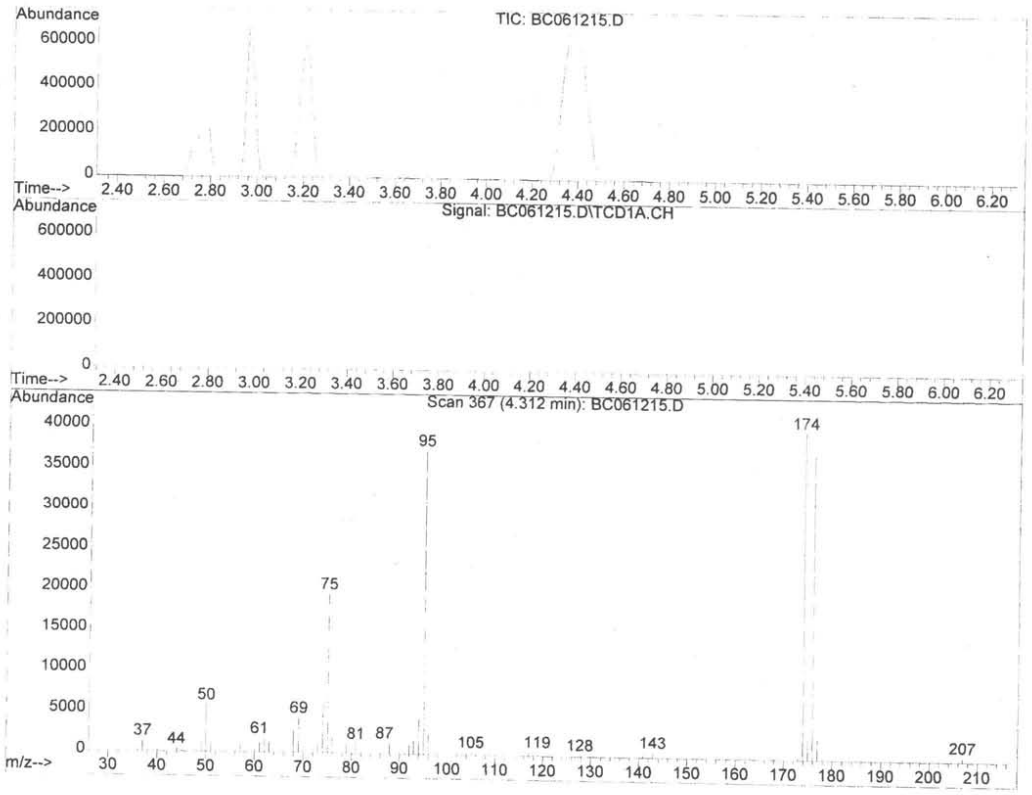
Printed Name Signature Date

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 Integrator)
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

000303

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)

	A	B	C	D	E	F	G	H	I	J	K	L	M
1		**Flammable Gas Analysis MDL Spreadsheet**											
2													
3	MDL date: 11-28-07												
4	Operator: TG												
5	c:\hpchem\1\data\		Measured Concentration										
6	Compound Name	Actual Conc. (ppmv)	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	avg % Rec.	Standard Deviation	% RSD	MDL ppmv
7	methanol	83.0	75.0	87.0	81.0	85.0	80.0	79.0	81.0	97.76	3.93	4.85	12.35
8	ethyl ether	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
9	1,1-dichloroethylene	20.4	22.0	23.0	24.0	20.0	21.0	22.0	22.0	107.84	1.29	5.87	4.05
10	acetone	82.0	75.0	87.0	81.0	85.0	80.0	79.0	81.0	98.95	3.93	4.85	12.35
11	1,1-dichloroethane	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
12	cis-1,2-dichloroethene	20.2	22.0	23.0	24.0	20.0	21.0	22.0	22.0	108.91	1.29	5.87	4.05
13	methyl ethyl ketone	82.2	75.0	87.0	81.0	85.0	80.0	79.0	81.0	98.71	3.93	4.85	12.35
14	cyclohexane	20.4	22.0	23.0	24.0	20.0	21.0	22.0	22.0	107.84	1.29	5.87	4.05
15	benzene	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
16	1,2-dichloroethane	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
17	butanol	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
18	methyl isobutyl ketone	82.6	75.0	87.0	81.0	85.0	80.0	79.0	81.0	98.24	3.93	4.85	12.35
19	toluene	82.4	75.0	87.0	81.0	85.0	80.0	79.0	81.0	98.47	3.93	4.85	12.35
20	chlorobenzene	21.0	22.0	23.0	24.0	20.0	21.0	22.0	22.0	104.76	1.29	5.87	4.05
21	ethylbenzene	20.4	22.0	23.0	24.0	20.0	21.0	22.0	22.0	107.84	1.29	5.87	4.05
22	m and p xylene	20.4	22.0	23.0	24.0	20.0	21.0	22.0	22.0	107.84	1.29	5.87	4.05
23	o-xylene	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
24	1,3,5-trimethylbenzene	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
25	1,2,4-trimethylbenzene	20.6	22.0	23.0	24.0	20.0	21.0	22.0	22.0	106.80	1.29	5.87	4.05
26													
27													
28		Actual Conc. (ppmv)	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	ppmv	avg % Rec.	Standard Deviation	% RSD	MDL ppmv
29	hydrogen	1500.0	1567.0	1588.0	1590.0	1500.0	1567.0	1590.0	1589.0	104.68	32.67	2.08	102.57
30	methane	1500.0	1567.0	1588.0	1590.0	1500.0	1567.0	1590.0	1589.0	104.68	32.67	2.08	102.57

SCO 0690 R0

Flammable Gas Analysis
MDL Spreadsheet

Microsoft Windows XP Professional
Excel 2002

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

Attachment 13 – Quantitation Report (Example)

Quantitation Report (Not Reviewed)

Data File : C:\HPCHEM\1\DATA\071112C\10181621.D Vial: 1
Acq On : 12 Nov 2007 8:45 Operator: AdeBK
Sample : Inst : UNIT-1
Misc : 55-gallon drum, thru filter vent Multiplr: 1.00
Sample Amount: 0.00
MS Integration Params: LSCINT.P GC Integration Params: RTEINT2.P
Quant Time: Nov 12 08:59:57 2007 Quant Results File: VOCGAS.RES

Quant Method : C:\HPCHEM\1\METHODS\VOCGAS.M (RTE Integrator)
Title : Flammable Gas Analysis for Transportation
Last Update : Mon Nov 12 08:52:07 2007
Response via : Initial Calibration
DataAcq Meth : VOCGAS

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
3) Fluorobenzene	7.07	96	375855	215.50	ppmv	0.00
15) Chlorobenzene-d5	10.50	117	395347	188.25	ppmv	-0.03
Target Compounds						Qvalue
1) Hydrogen	3.92	GC1	2373	0.0198	ppmv	100
7) Acetone	4.31	59	4534	35.9904	ppmv	70

(#) = qualifier out of range (m) = manual integration
10181621.D VOCGAS.M Mon Nov 12 09:59:59 2007