Controlled
Copy

CCP-TP-083

Revision 6

CCP
Gas Generation Testing

EFFECTIVE DATE: 03/27/2009

D. K Ploetz

PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	03/29/2005	Initial Issue.
1	06/13/2005	Address safety concerns by equipment Operators and change it to Type IV operations only.
2	07/28/2005	Revised to address Carlsbad Field Office (CBFO) Document Review Record (DRR) comments.
3	10/18/2005	Revise to update the equipment from using a 5 psi relief valve to a 10 psi relief valve.
4	03/16/2006	Revised to incorporate the requirements of Revision 2 of CCP-PO-016, *CCP Gas Generation Test Program Quality Assurance Project Plan*.
5	04/13/2006	Revised to address additional requirements of Revision 2 of CCP-PO-016, *CCP Gas Generation Test Program Quality Assurance Project Plan*.
6	03/27/2009	Combined CCP-TP-083, *CCP Heated Gas Test Canister Operations*, CCP-TP-089, *CCP Mobile Gas Generation Testing Sampling System (MGSS) Sampling Operation*, CCP-TP-092, *CCP Mobile Gas Generation Testing Sampling System (MGSS) Data Calculation*, and CCP-TP-094, *CCP GGTP Drum Screening and Batching*, to simplify and incorporate changes to CCP-PO-016, *CCP Gas Generation Testing Program Quality Assurance Project Plan*.

TABLE OF CONTENTS

LIST OF ATTACHMENTS

1.0 PURPOSE

This procedure provides instructions for implementing the Gas Generation Testing (GGT) for the Gas Generation Testing Program (GGTP). This procedure uses the Gas Generation Test Canister (GGTC) to measure the pressure change and to collect off-gases generated from a waste drum to analyze for hydrogen to determine the hydrogen generation rate, the hydrogen/methane (H_2/CH_4) generation rate, and the total gas generation rate of the drum. This procedure complies with the requirements of CCP-PO-016, *CCP Gas Generation Testing Program Quality Assurance Project Plan.*

1.1 Scope

The GGTC is a Bell Jar that encloses a waste drum and accumulates all the gases generated from this drum. Since all drums selected for the test are equipped with a filtered vent in the lid, opening the drum is unnecessary in order to collect the gases. Only 55-gallon drums are allowed. The GGTC isolates the waste drum for sampling to determine the hydrogen concentration and pressure change to calculate the hydrogen generation rate and total gas generation rate.

The system calculates the hydrogen generation rate and uses either actual CH_4/H_2 ratios from headspace of the drum or a correction factor, per CCP-PO-016 Table 4-3, Methane Determination, to determine the H_2/CH_4 generation rate.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- DOE/WIPP 01-3187, *Quality Assurance Program Plan for TRUPACT-II Gas Generation Test Program*
- CCP-PO-003, *CCP Transuranic Authorized Methods for Payload Control (CCP CH-TRAMPAC)*
- CCP-QP-011, *CCP Notebooks and Logbooks*
- CCP-TP-140, *CCP Equipment Maintenance*

Referenced Documents

- U. S. Department of Energy, *Contact Handled Transuranic Waste Authorized Methods for Payload Control (CH-TRAMPAC)*, Carlsbad Field Office, Carlsbad, NM
- CCP-HSP-012, *CCP Health and Safety Plan For GGTP*
- CCP-PO-016, *CCP Gas Generation Testing Program Quality Assurance Project Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*
- CCP-QP-008, *CCP Records Management*
- CCP-QP-022, *CCP Software Quality Assurance Plan*
- Internal Memo, M. Rivera to E. D'Amico, "Bounding Propagated Error Analysis for the CCP GGTP", February 15, 2006

2.2 Training Requirements

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

2.3 Software

2.3.1 All software used in this program is approved in accordance with CCP-QP-022, *CCP Software Quality Assurance Plan.*

[A] Shimadzu analytical software used to control the Gas Chromatograph (GC) to determine the concentration of hydrogen gas in the samples.

[B] GGTP Data Calculation Microsoft Excel spreadsheet(s)

2.4 Equipment List

2.4.1 Analytical System

[A] GC with Thermal Conductivity Detector (TCD) mounted in a movable cart called Mobile Gas Generation Testing Sampling System (MGSS)

[B] Calibrated vacuum/pressure gauge

[C] Vacuum pump

[D] Computer/Data System – A data system that allows the continuous acquisition and storage of TCD data obtained throughout the duration of the chromatographic program is interfaced to the GC/TCD System.

2.4.2 Supplies and Equipment

[A] Gases

[A.1] Hydrogen Gas standards shall be procured as certified gas standards.

[A.2] Argon, ultra high purity

[A.3] Nitrogen

[B] Sampling Equipment

[B.1] GGT Canister with calibrated 10 pound per square inch (lb/in^2) pressure relief valve(s), calibrated temperature controller(s), calibrated pressure gauge (capable of measuring up to 15 pounds per square inch gauge [psig]).

[C] Miscellaneous Equipment

[C.1] Calibrated torque wrench

[C.2] Lifting device

[C.3] Adjustable wrenches and/or open-end wrenches

[C.4] Leak-detection fluid

[C.5] Gas supply – Air or Nitrogen

[C.6] Spare O-ring for the GGTC (0.375-inch diameter, BUNA-N® O-ring, or equivalent, field cut to fit)

[C.7] Latex gloves, as required

[C.8] Nitrile gloves for liquids present in the GGTC

[C.9] O-ring tool

[C.10] Vacuum grease

[C.11] Anti-seize compound

[C.12] Windex® or equivalent

[C.13] Caution – Surface Hot to Touch signs.

2.5 Precautions and Limitations

2.5.1 Personnel SHALL keep their exposed body parts, particularly faces, away from the area above the relief valve.

2.5.2 The fittings and gauges on the GGTC are susceptible to damage and caution SHALL be taken to avoid any impact against the GGTC.

2.5.3 The inlet gas tube inside the GGTC is susceptible to damage while raising and lowering the top portion of the GGTC. Caution SHALL be taken to avoid contact between the drum and the inside of the GGTC.

2.5.4 Personnel SHALL comply with the personal protective equipment (PPE) as listed in CCP-HSP-012, *CCP Health and Safety Plan For GGTP*, and/or Host site requirements.

2.5.5 GGTCs SHALL be vented after GGT has been completed. During the testing period, the canisters may have a buildup of Volatile Organic Compounds (VOCs) and/or flammable gases including H_2, flammable VOCs, CH_4, and acid gases. The canisters SHALL **NOT** be vented to the room atmosphere.

2.5.6 Personnel must be aware of body positioning to avoid pinch and crush hazards when the GGTC top portion is suspended.

2.5.7 DO **NOT** set the GGTC top portion down on any surface other than the bottom portion or other approved surface as determined by the Lead Operator (LO).

2.6 Quality Assurance Objectives (QAO)

2.6.1 Precision and Accuracy

The QAO for precision of the measured or calculated H_2/CH_4 and total gas generation rates shall be less than or equal to a relative percent difference of 25 percent. The QAO for accuracy of the measured or calculated H_2/CH_4 and total gas generation rates shall be between 70 to 130 percent recovery (%R). In the case where a gas generation rate is not measured directly but calculated from knowledge of other measured or estimated parameters, the requirements of precision and accuracy listed above shall be met as demonstrated through an error propagation analysis. The Precision and Accuracy of the hydrogen generation rate and total gas generation rate calculations are derived from error propagation analysis. Central Characterization Project (CCP) uses the error propagation analysis for Hydrogen Generation Rate Testing which is documented in Internal memo, M. Rivera to E. D'Amico, "Bounding Propagated Error Analysis for the CCP GGTP", February 15, 2006.

CCP uses the PRQL of 6.90E-9 moles/second for the H_2/CH_4 generation rate. This PRQL is less than the lowest allowable generation rate of 3.03E-8 moles/second of Waste Type IV (55-gallon drum) waste expected to be shipped from Idaho National Laboratory (INL). CCP uses the PRQL for Total Gas Generation Rate of 6.36E-7 moles/second which is less than the rate allowed by U.S. Department of Energy (DOE), *Contact-Handled Transuranic Waste Authorized Methods for Payload Control (CH-TRAMPAC)*, Table 5.2-11 for 55-gallon drums.

2.6.2 Representativeness

CCP will perform a Leak Test of the GGTC upon loading the drum into GGTC and prior to starting the heating of the GGTC. A Vacuum Leak Check will be performed on the sampling manifold prior to each sampling batch to verify integrity of the sampling line connection.

2.6.3 Completeness

All waste containers in which the flammable VOC concentration cannot be shown through acceptable knowledge (AK) or process knowledge (PK) to be less than or equal to 500 parts per million (ppm) by volume shall have a valid gas sample collected and analyzed to establish the flammable VOC concentration.

Containers that undergo GGT shall have valid parameter measurements to establish the H_2/CH_4 gas generation rates in the container. All containers where compliance with the total gas generation limits is not shown by conservative theoretical analysis in accordance with the CH-TRAMPAC shall have valid pressure and temperature parameter values from calibrated pressure and temperature gauges to establish the total gas generation rate.

CCP will test containers to establish the H_2/CH_4 gas generation rate and total gas generation rate using the following measured parameters: Hydrogen concentration, sampling time, drum age, temperature, pressure, and effective resistance (R_{eff}).

2.6.4 Comparability

Comparability of data shall be ensured through the use of standardized, approved testing, sampling, and analytical techniques. Consistent use and application of uniform procedures, calibration of analysis instruments against nationally acceptable (traceable to National Institute of Standards and Technology [NIST]) or manufacturer-certified standards, and use of instruments that are of the required precision and accuracy should ensure data measurements on different waste containers are comparable.

2.7 Quality Control (QC) Requirements

2.7.1 **Initial Calibration (ICAL)** – A minimum three-point ICAL curve is generated to establish an ability to quantitate Hydrogen and serves to define the linear range of the method. 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 coefficient of determination (r^2) must be greater than or equal to 0.990.

2.7.2 **Continuing Calibration Verification (CCV) –** Each day of sampling operations, a CCV check will be performed. The CCV must be within the 70 – 130 %R of the certified value.

2.7.3 **Batch Data Report (BDR) Number** – A batch is defined by the number of canisters being heated collectively as a batch. The BDR is assigned a unique identification number in the format SSYYGGNXXX where SS is the site designation, YY are the last two digits of the year, GG is place holder, N is the instrument number, and xxx is a sequential number starting with 001 each year. The ICAL has the same unique identification number except with _ICAL added to the number.

3.0 RESPONSIBILITIES

3.1 Vendor Project Manager (VPM)

3.1.1 Responsible for the operation and maintenance of the GGT apparatus and the management of the data generated from GGT operations.

3.1.2 Performs the function of GGTP Manager.

3.2 Independent Technical Reviewer (ITR)

3.2.1 Responsible for performing technical reviews of GGT BDRs prior to forwarding to the Quality Assurance Officer (QAO).

3.3 Quality Assurance Officer (QAO)

3.3.1 Responsible for the review and validation of GGT GC Chromatogram(s), nonconformance, and BDRs.

3.3.2 When separately qualified as Operator/Technical Supervisor (TS), may perform Operator/TS review.

3.4 Operator/Technical Supervisor (TS)

3.4.1 Performs the loading and unloading of drums in the GGTCs.

3.4.2 Assists the manager in coordination of GGT operation.

3.4.3 Samples candidate drums in the GGTCs.

3.4.4 Compiles the GGT BDRs and forwards to the ITR.

3.4.5 Reviews the screening of candidate drums for GGT.

3.4.6 Documents any nonconformances in accordance with CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*.

3.5 Lead Operator (LO)

3.5.1 Provides technical supervision for the operation.

3.5.2 Provides guidance in the event of abnormal conditions.

3.6 Facility Records Custodian

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

4.0 PROCEDURE

Operator/TS

4.1 Container Selection

4.1.1 **IF** a waste container meets at least one of the criteria below, **THEN** the drum is considered a test category drum and can be tested using this procedure:

- The decay heat exceeds analytical limit
- The waste type has no bounding G value (Type IV)
- The flammable VOC concentration in container headspace cannot be demonstrated to be less than or equal to 500 ppm

4.2 Testing Determination

4.2.1 On the Drum Information sheet (see Attachment 1, Drum Information, for an example), enter the following:

NOTE

The closure date, number of layers of confinement, rigid liner, and %Fill information is obtained from real-time radiography (RTR) or visual examination (VE) data. The filter number is obtained from the drum. The Hydrogen Diffusivity of filter is obtained from the DOE approved filter list. The R_{eff} is looked up in a table of calculated values. The methane concentration is obtained from the headspace data and if detected used to calculate the CH4/H2 ratio.

- BDR Number (Page 1 and 2)
- Drum No. (Page 1 and 2)
- Answer question with Y or N as appropriate in Column 2
- Answer question with Y or N as appropriate in Column 3
- Answer question with Y or N as appropriate in Column 4
- Closure Date
- No. of Layers of Confinement
- Rigid Liner (Y or N)
- %Fill
- Calculate and enter void volume (220- [%Fill X 192])

AND,

- Filter Model Number(s)

- Hydrogen Diffusivity of Filter(s) mole per second per mole fraction (m/s/mf)
- Effective resistance to the release of Hydrogen (sec/mole) (R_{eff})
- Determine CH_4/H_2 Ratio, **OR** enter default value (.1)

4.2.2 Print name, sign, and date Drum Information.

4.2.3 Forward completed Drum Information sheet to ITR.

ITR

4.2.4 Verify technical accuracy and completeness of Drum Information sheet.

4.2.5 Print name, sign, and date Drum Information sheet, **AND** return to Operator/TS.

Operator/TS

4.3 Putting Drum in Empty GGTC

4.3.1 Before work begins on GGTC, ensure the following:

[A] Record BDR number and date on the GGTC Checklist (see Attachment 2, GGTC Checklist for an example),

[B] Obtain a torque wrench that is in calibration, **AND** record Torque Wrench ID and Torque Wrench Cal Due Date on the GGTC Checklist,

[C] The GGTC is unplugged,

[D] The outlet valve(s) are CLOSED, **AND** the exhaust hose is disconnected from the GGTC.

4.3.2 Position drum and GGTC, as necessary, for GGTC operations.

NOTE
Information for GGTC Checklist may be entered at any time before isolation of GGTC.

4.3.3 Record the GGTC No., Temperature Controller ID, Temperature Controller Cal. Due Date, Pressure Gauge ID, Pressure Gauge Cal. Due Date, and Drum No. on GGTC Checklist.

4.3.4 Remove the Empty Tag from the GGTC to be loaded, as necessary.

NOTE
The flange bolts can be removed from all of the GGTCs rather than individually at the discretion of the Operator/TS.

4.3.5 After verifying the pressure gauge reads 0 pounds per square inch (psi), remove the flange bolts.

4.3.6 Slowly lift the top portion of GGTC and move away from base plate.

4.3.7 Inspect the O-ring, **AND** ensure it is free of defects, such as nicks, cracks, or gouges.

4.3.8 **IF** a nick, crack, or gouge is found,
THEN replace the O-ring.

4.3.9 Clean any dirt or debris from the bottom plate seating surface that may have accumulated during drum movement.

4.3.10 Grease the O-ring, if necessary, **AND** ensure it is seated properly.

4.3.11 Position and center a selected drum onto the bottom plate.

4.3.12 Lift the GGTC top portion over the selected drum, **AND** lower until it rests onto the bottom plate, covering the drum.

4.3.13 Ensure the GGTC is aligned and seated properly.

NOTE
Steps 4.3.14 through 4.3.30 may be performed individually or in groups to be determined by the Operator/TS.

4.3.14 Apply anti-seize compound, if necessary, **AND** thread each flange bolt into the bottom plate.

4.3.15 Tighten and torque the flange bolts to 44 foot-pounds (ft-lbs.) (± 5 ft-lbs.)

4.3.16 **IF** one of the bolts CAN **NOT** be tightened or torqued,
THEN contact LO for instructions.

4.3.17 Record initials and date on GGTC Checklist for bolt torque.

4.3.18 Pressurize the GGTC with air or nitrogen to approximately 4 – 7 psig.

4.3.19 Perform leak detection on the GGTC mechanical joints using leak-detection fluid.

4.3.20 **IF** a leak is detected,
THEN contact LO for instructions.

4.3.21 Record Leak Test Pass (P) on GGTC Checklist.

4.3.22 Ensure the GGTC vent tubing is connected to the pressure relief valve, **AND** the exhaust hose is connected.

4.3.23 OPEN the outlet valve until 0 psi on pressure gauge.

4.3.24 CLOSE inlet and outlet valve.

4.3.25 Ensure that the thermocouple leads are attached to the thermocouple(s), **AND** the thermocouple(s) is installed in the top of the GGTC.

4.3.26 Ensure that the temperature display cabinet is energized.

4.3.27 Ensure that the temperature controller(s) is reading in degrees celsius (°C) and that the controller set point temperature is set at 57.3 °C.

4.3.28 Place a sign reading, Caution – Surface Hot to Touch, or equivalent on the affected GGTC.

4.3.29 Put drum documentation in plastic sleeve on GGTC as necessary.

4.3.30 Position GGTC in proper location, if applicable.

4.3.31 Plug the GGTC heater power cord into the heater/power supply cabinet, **AND** turn the heater ON, if applicable.

4.3.32 Record Initials, Date, and Time of Isolation on GGTC Checklist.

4.3.33 Notify VPM and Host site that heating has been initiated.

4.4 Unloading and Loading a Drum in a GGTC

4.4.1 Before work begins on GGTC ensure the following:

[A] Record BDR number and date on GGTC Checklist.

[B] Obtain a torque wrench that is in calibration, **AND** record Torque Wrench ID and Torque Wrench Cal Due Date on GGTC Checklist,

[C] The GGTC is unplugged,

[D] The outlet valve and inlet valve are CLOSED, **AND** the exhaust hose is disconnected from the GGTC.

4.4.2 Position drum and GGTC, as necessary, for GGTC operations.

4.4.3 Record the GGTC No., Temperature Controller ID, Temperature Controller Cal. Due Date, Pressure Gauge ID, Pressure Gauge Cal. Due Date, and Drum No. on GGTC Checklist.

4.4.4 Remove the drum documentation from the GGTC to be loaded, as necessary, and contact LO for disposition of drum documentation.

4.4.5 Verify the pressure gauge reads 0 psi, and then remove flange bolts.

4.4.6 Slowly lift the top portion of GGTC over the drum and move away from base plate.

4.4.7 **IF** any liquid is found,
THEN STOP WORK, **AND** notify the VPM or Host site personnel.

4.4.8 Carefully lift the drum from the GGTC bottom plate, **AND** visually inspect the drum bottom for container integrity.

4.4.9 **IF** container integrity is compromised,
THEN STOP WORK, **AND** contact the VPM or Host site for instructions.

4.4.10 Place drum in designated location.

4.4.11 Inspect the O-ring, **AND** ensure it is free of defects, such as nicks, cracks, or gouges.

4.4.12 **IF** a nick, crack, or gouge is found,
THEN replace the O-ring.

4.4.13 Clean any dirt or debris from the bottom plate seating surface that may have accumulated during drum movement.

4.4.14 Grease the O-ring, if necessary, **AND** ensure it is seated properly.

4.4.15 Position and center a selected drum onto the bottom plate.

4.4.16 Lift the GGTC top portion over the selected drum, **AND** lower it until it rests onto the bottom plate, covering the drum.

4.4.17 Ensure the GGTC is aligned and seated properly.

NOTE
Steps 4.4.18 through 4.4.35 may be performed individually or in groups to be determined by the Operator/TS.

4.4.18 Apply anti-seize compound, if necessary, **AND** thread each flange bolt into the bottom plate.

4.4.19 Torque the flange bolts to 44 ft-lbs. (± 5 ft-lbs.).

4.4.20 **IF** one of the bolts CAN **NOT** be tightened or torque,
THEN contact LO for instructions.

4.4.21 Record initials and date on GGTC Checklist for bolt torqued.

4.4.22 Pressurize the GGTC with air or nitrogen to approximately 4 – 7 psig.

4.4.23 Perform leak detection on the GGTC mechanical joints using leak-detection fluid.

4.4.24 **IF** a leak is detected,
THEN contact LO for instructions.

4.4.25 Record Leak Test Pass (P) on GGTC Checklist.

4.4.26 Ensure the GGTC vent tubing is connected to the pressure relief valve, **AND** connect the exhaust hose to the GGTC outlet valve.

4.4.27 OPEN outlet valve until pressure gauge reads 0 psi.

4.4.28 CLOSE inlet and outlet valves.

4.4.29 Ensure that the thermocouple leads are attached to the thermocouple(s), **AND** the thermocouple(s) is installed in the top of the GGTC.

4.4.30 Ensure that the temperature display cabinet is energized.

4.4.31 Ensure that the temperature controller(s) is reading in °C and that the controller set point temperature (green LED) is set at 57.3°C.

4.4.32 Place a sign reading, CAUTION – Surface Hot to Touch, or equivalent on the affected GGTC.

4.4.33 Place drum documentation in a plastic sleeve on GGTC.

4.4.34 Position GGTC in proper location, if applicable.

4.4.35 Plug the GGTC heater power cord into the heater/power supply cabinet, **AND** turn the heater ON, if applicable.

4.4.36 Record Initials, Date, and Time of Isolation on GGTC Checklist.

4.4.37 Notify VPM and Host site that heating has been initiated.

4.5 Unloading GGTC and Putting GGTC in Standby State

4.5.1 Before work begins on GGTC ensure the following:

[A] The GGTC is unplugged,

[B] The outlet valve(s) are CLOSED, **AND** the exhaust hose is disconnected from the GGTC.

4.5.2 Position drum and GGTC, as necessary, for GGTC operations.

4.5.3 Remove the drum documentation from the GGTC to be unloaded, as necessary, and contact LO for disposition of drum documentation.

4.5.4 Verify the pressure gauge reads 0 psi, and then remove flange bolts.

4.5.5 Slowly lift the top portion of GGTC over the drum and move away from base plate.

4.5.6 **IF** any liquid is found,
THEN STOP WORK, **AND** notify the VPM or Host site personnel.

4.5.7 Carefully lift the drum from the GGTC bottom plate, **AND** visually inspect the drum bottom for container integrity.

4.5.8 **IF** integrity is compromised,
THEN STOP WORK, **AND** contact the VPM or Host site for instructions.

4.5.9 Place drum in designated location.

4.5.10 Inspect the O-ring, **AND** ensure it is free of defects, such as nicks, cracks, or gouges.

4.5.11 **IF** a nick, crack, or gouge is found,
THEN replace the O-ring.

4.5.12 Clean any dirt or debris from the bottom plate seating surface that may have accumulated during drum movement.

4.5.13 Lift the GGTC top portion, **AND** lower it until it rests onto the bottom plate.

4.5.14 Ensure the GGTC is aligned and seated properly.

NOTE
Steps 4.5.15 through 4.5.17 may be performed individually or in groups to be determined by the Operator/TS.

4.5.15 Apply anti-seize compound, if necessary, **AND** thread each flange bolt into the bottom plate.

4.5.16 Place Empty tag on GGTC.

4.5.17 Position GGTC in proper location, if applicable.

4.6 Initial Calibration

NOTE
An ICAL is performed at startup, after major repairs, or when the quality controls can no longer be met.

4.6.1 Turn ON the Argon gas bottle, **AND** check that there is greater than 100 psi.

4.6.2 Check GC is energized, **AND** vacuum pump is ON.

4.6.3 Ensure Valves V-6, V-7, V-8, V-9, V-10, V-11, and V-13 are SHUT.

4.6.4 Ensure Valves V-1, V-2, V-3, V-4, and V-12 (V-4 and V-12 fully counter clockwise) are OPEN.

4.6.5 Check vacuum is less than 10 Torr.

4.6.6 Turn computer ON.

4.6.7 Open the software by clicking the icon.

4.6.8 Select GC (i.e., GC-14B).

4.6.9 Load Method by clicking on File, Method, Open.

4.6.10 Click on Method.

4.6.11 Click on Instrument Setup.

4.6.12 Click on Download Method.

4.6.13 Unlock keypad (if necessary), **AND** press the Start key on the GC keypad.

4.6.14 Exit window.

4.6.15 OPEN Windows Explorer.

4.6.16 Click Class VP.

4.6.17 Click Data.

4.6.18 Create a new directory (C:\Class-VP\data\BDR number), **AND** minimize Windows Explorer.

4.6.19 Click Method.

4.6.20 Click Peaks/Groups.

4.6.21 Scroll right to the column entitled Level 1.

4.6.22 **IF** the value is **NOT** the Certificate of Accuracy (COA) concentration,
THEN type in the correct ppm.

4.6.23 Repeat step 4.6.21 and step 4.6.22 for each level of calibration.

4.6.24 Open file, click Method, and Save As GGTP MMDDYY where MMDDYY is the date of the new ICAL.

4.6.25 CLOSE the Peak/Group Table.

4.6.26 Click the Single Run button on the toolbar at the top of the screen.

4.6.27 Enter Data path C:\class-vp\data\ICALmmddyy.

4.6.28 Enter Method: GGTP MMDDYY.

4.6.29 Enter Pressure (Torr)_Temperature (C): 630_22 in Sample ID box.

4.6.30 In Data file, enter Level n where n is the ICAL level.

4.6.31 Click Description button, enter the calibration standard cylinder number and the expiration date, **AND** click OK.

4.6.32 Click in the box labeled Calibrate, click on the Calibrate Level option box, **AND** enter either, 1, 2, 3, 4, or 5 in the adjacent text box, corresponding to the calibration level.

4.6.33 Click on the option box labeled Clear Calibration For Level, **AND** for highest level, Print Calibration Report (see Attachment 10, Calibration Report, for an example).

4.6.34 Click the Start command button.

4.6.35 Ensure the manifold vacuum is less than or equal to 10 Torr.

4.6.36 Ensure that cylinder valves on the calibration gases are OPEN, cylinder pressures are greater than 100 psig, **AND** the line pressure is set at approximately 5 psig.

4.6.37 SHUT V-2 and V-3.

4.6.38 OPEN V-8 (V-9, V-10, or V-7) until the pressure on the pressure gauge starts to rise.

4.6.39 Adjust pressure using V-4, **AND/OR** V12 to 630 ±3 Torr.

4.6.40 SHUT V-8 (V-9, V-10, or V-7) to purge the line.

4.6.41 OPEN V-2 and V-3, allow the pressure to decrease to less than or equal to 10 Torr, **AND** ensure the reading maintains below 10 Torr for approximately 15 seconds.

4.6.42 SHUT V-2 and V-3.

4.6.43 OPEN V-8 (V-9, V-10, V- 7).

4.6.44 Adjust the pressure using V-12 **AND/OR** V-4, if necessary, to 630 ± 3 Torr.

4.6.45 **WHEN** the pressure gauge equals 630 ± 3 Torr,
THEN push the Start button on the GC keypad, starting the calibration run.

4.6.46 SHUT V-8 (V-9, V-10, or V-7), **AND** CLOSE the cylinder valve.

4.6.47 OPEN V-2 and V-3, **AND** allow the manifold to pump down to less than 10 Torr.

4.6.48 For the other calibration levels, repeat step 4.6.26 through step 4.6.47.

4.6.49 **IF** it is necessary to replace a single point on the calibration curve, with a single run, which was analyzed as an unknown (but is of known ppm),
THEN perform the following:

[A] After completion of the run or retrieval of the run from Data files, click Analysis…Single Level Calibration.

[B] Load appropriate data file.

[C] Click Calibration.

[D] Enter appropriate calibration level.

[E] Click Clear Calibration For Level.

[F] Confirm appropriate method and data path.

[G] Click Start.

NOTE
The calibration report containing the r^2 value is printed automatically after the last calibration standard is analyzed.

4.6.50 Verify that the r^2 Value is greater than or equal to 0.990.

4.6.51 **IF** r^2 Value is <0.990,
THEN repeat steps 4.6.25 to 4.6.48.

NOTE
The GC chromatograms are printed automatically after each level of a calibration standard is analyzed and the calibration report is printed automatically after the last calibration standard is analyzed.

4.6.52 Assemble the ICAL BDR with the following information:

[A] Print Attachment 7, Data Report Cover Page, to include in ICAL BDR.

[B] Calibration Report (see Attachment 10, Calibration Report, for an example)

[C] GC Chromatogram(s)

[D] Copy of COA(s)

4.6.53 Paginate ICAL BDR.

4.6.54 Complete Attachment 7.

4.6.55 Print name, sign, and date Attachment 7 and Attachment 10.

4.6.56 Forward to the ITR.

ITR

4.6.57 Review ICAL BDR and ensure the following:

[A] That the r^2 is greater than or equal to 0.990 for hydrogen.

[B] Notify the Operator if there are any errors or omissions.

[C] Recheck the data after the errors or omissions have been corrected.

4.6.58 Print name, sign, and date Attachment 7.

4.6.59 Forward to QAO.

QAO

4.6.60 Review ICAL BDR and ensure the following:

[A] That the r^2 is greater than or equal to 0.990 for hydrogen.

[B] Notify the ITR if there are any errors or omissions.

[C] Recheck the data after the errors or omissions have been corrected.

4.6.61 Print name, sign, and date Attachment 7.

4.6.62 Submit ICAL BDR to the Facility Records Custodian.

Facility Records Custodian

4.6.63 Receive, process, and transmit ICAL BDR in accordance with CCP-QP-008.

4.7 GGTC Sampling

Operator/TS

4.7.1 Initial GGT Sampling

[A] After a minimum of 120 hours from the isolation date and time, **OR** at the discretion of the LO, reset the Peak and Valley temperatures on the temperature controllers.

[B] Move the MGSS cart to the GGTC to be tested.

[C] Begin the CCV by first clicking the Single Run button on the toolbar at the top of the screen.

[D] Enter Data path C:\class-vp\data\BDR number-1.

[E] Ensure the GGTP MMDDYY.met method is listed at the top of the screen where MMDDYY is the date of the ICAL.

[F] Enter Pressure (Torr)_Temperature (°C): 630_22 in Sample ID box.

[G] Enter Data file VCAL01.

[H] Click Description button, enter the CCV cylinder number and expiration date, **AND** click OK.

[I] Click the Start command button on the screen.

[J] Ensure the manifold vacuum is less than or equal to 10 Torr.

[K] Ensure that cylinder valve for CCV is OPEN, cylinder pressure is greater than 100 psig, **AND** line pressure is set at approximately 5 psig.

[L] SHUT V-2 and V-3.

[M] OPEN V-9.

[N] Adjust the pressure using V-12, **AND/OR** V-4 to 630 ± 3 Torr.

[O] SHUT V-9.

[P] OPEN V-2 and V-3 to purge the line.

[Q] Allow the pressure to decrease to less than 10 Torr, **AND** ensure the reading maintains below 10 Torr for approximately 15 seconds.

[R] SHUT V-2 and V-3.

[S] OPEN V-9.

[T] Adjust the pressure using V-12, **AND/OR** V-4, if necessary, to 630 ± 3 Torr.

[U] **WHEN** the pressure reads 630 ± 3 Torr,
THEN press the Start button on the keypad, starting the run.

[V] SHUT V-9, **AND** CLOSE the cylinder valve.

[W] OPEN V-2 and V-3, **AND** allow the manifold to pump down to less than 10 Torr.

NOTE

The GC chromatogram is printed automatically after the CCV is analyzed. The GC chromatogram contains the measured hydrogen concentration used to calculate the %R.

[X] Verify the %R (Measured ppm ÷ Certified ppm X 100% = %R) is 70 – 130%.

[Y] **IF** %R is **NOT** between 70 – 130%,
THEN repeat CCV.

[Z] **IF** a CCV will **NOT** pass,
THEN notify the LO for instructions.

[AA] Attach the sample hose to GGTC quick-disconnect.

[BB] OPEN V-6, **AND** allow the pressure to pump down to less than 10 Torr.

[CC] SHUT V-6.

[DD] OPEN the GGTC Outlet Valve, **AND** SHUT the GGTC Outlet valve to fill the sample line.

[EE] To purge the sample line, OPEN V-6, allow the pressure to pump down to less than 10 Torr, **AND** ensure the reading maintains below 10 Torr for approximately 15 seconds.

[FF] Select the Single Run button on the Class-VP toolbar.

[GG] SHUT V-2 and V-3.

[HH] OPEN the GGTC outlet valve (Note the pressure increase to GGTC pressure).

[II] Enter the following in the box labeled Sample ID Pressure (Torr)_Temperature (C): 630_22.

[JJ] Enter selected Drum number in data file box.

[KK] Enter Data path C:\class-vp\data\BDR number-1.

[LL] Click the Start Command button on the screen.

[MM] **WHEN** the “Waiting for Trigger…” message flashes at the bottom of the screen,
THEN push the Start button on the GC keypad.

[NN] SHUT V-6.

[OO] SHUT the GGTC Outlet Valve, **AND** detach sample hose.

[PP] OPEN V-2 and V-3.

[QQ] Repeat steps 4.7.1[AA] through 4.7.1[PP] for all of the GGTC to be sampled.

4.7.2 Final GGT Sampling

[A] Final sampling can be performed after a minimum of 48 hours from initial sampling and minimum of 240 hours from isolation.

[B] Move the MGSS cart to the GGTC to be tested.

[C] Begin the CCV by first clicking the Single Run button on the toolbar at the top of the screen.

[D] Enter Data path C:\class-vp\data\BDR number-2.

[E] Ensure the GGTP MMDDYY.met method is listed at the top of the screen where MMDDYY is the date of the ICAL.

[F] Enter Pressure (Torr)_Temperature (°C): 630_22 in Sample ID box.

[G] Enter Data file VCAL01.

[H] In the Description box, enter the CCV cylinder number and the expiration date, **AND** click OK.

[I] Click the Start Command button on the screen.

[J] Ensure the manifold vacuum is less than or equal to 10 Torr.

[K] Ensure that cylinder valve for CCV is OPEN, cylinder pressure is greater than 100 psig, **AND** line pressure is set at approximately 5 psig.

[L] SHUT V-2 and V-3.

[M] OPEN V-9.

[N] Adjust the pressure using V-12 **AND/OR** V-4 to 630 ± 3 Torr.

[O] SHUT V-9.

[P] OPEN V-2 and V-3 to purge the line.

[Q] Allow the pressure to decrease to less than 10 Torr, **AND** ensure the reading maintains below 10 Torr for approximately 15 seconds.

[R] SHUT V-2 and V-3.

[S] OPEN V-9.

[T] Adjust the pressure using V-12, **AND/OR** V-4, if necessary, to 630 ± 3 Torr.

[U] **WHEN** the pressure reads 630 ± 3 Torr,
THEN press the Start button on the keypad, starting the run.

[V] SHUT V-9, **AND** CLOSE the cylinder valve.

[W] OPEN V-2 and V-3, **AND** allow the manifold to pump down to less than 10 Torr.

NOTE

The GC chromatogram is printed automatically after the CCV is analyzed. The GC chromatogram contains the measured hydrogen concentration used to calculate the %R.

[X] Verify the %R (Measured ppm ÷ Cert. ppm X 100% = %R) is 70 – 130%.

[Y] **IF** %R is **NOT** between 70 – 130%,
THEN repeat CCV.

[Z] **IF** a CCV will **NOT** pass,
THEN notify the LO for instructions.

[AA] Display the Peak and Valley temperatures on the temperature controllers in accordance with the Temperature Controller User's Manual, **AND** record Peak/Valley temperature (°C) on GGTC Checklist.

[BB] Print name, sign, and date GGTC Checklist.

[CC] Place GGTC Checklist in BDR Holding File.

[DD] **IF** either Peak or Valley temperature is **NOT** 57.3 ± 3°C,
THEN initiate a Nonconformance Report (NCR) in accordance with CCP-QP-005.

[EE] Attach the sample hose to GGTC quick-disconnect.

[FF] OPEN V-6, **AND** allow the pressure to pump down to less than 10 Torr.

[GG] SHUT V-6.

[HH] OPEN the GGTC Outlet Valve, then SHUT the GGTC Outlet valve to fill the sample line.

[II] To purge sample line, OPEN V-6, allow the pressure to pump down to less than 10 Torr, **AND** ensure the reading maintains below 10 Torr for approximately 15 seconds.

[JJ] Select the Single Run button on the Class-VP toolbar.

[KK] SHUT V-2 and V-3.

[LL] OPEN the GGTC outlet valve (Note the pressure increase to GGTC pressure).

[MM] Enter the following in the box labeled Sample ID Pressure (Torr) _Temperature (C): 630_22.

[NN] Enter selected Drum Number in data file box.

[OO] Enter Data path C:\class-vp\data\BDR number-2.

[PP] Click the Start Command button on the screen.

[QQ] **WHEN** the "Waiting for Trigger…" message flashes at the bottom of the screen,
THEN push the Start button on the GC keypad.

[RR] SHUT V-6.

[SS] SHUT the GGTC Outlet Valve, **AND** detach sample hose.

[TT] OPEN V-2 and V-3.

[UU] Repeat steps 4.7.2[EE] through 4.7.2[TT] for each GGTC to be sampled.

4.8 MGSS Shutdown

4.8.1 Ensure Valves V-6, V-7, V-8, V-9, V-10, V-11, and V-13 are SHUT.

4.8.2 Ensure Valves V-1, V-2, V-3, V-4, and V-12 are OPEN.

4.8.3 Shutdown the computer.

4.8.4 Shut OFF the GC and pump.

4.8.5 Shut OFF the Argon gas cylinder.

4.9 GGTC Shutdown

4.9.1 Switch the heater OFF or unplug the heater power cord(s).

4.9.2 Ensure that the controller remains energized to maintain temperature indication.

4.9.3 Connect the air or nitrogen to the inlet valve of the GGTC.

4.9.4 Connect the exhaust hose to the outlet of the GGTC.

4.9.5 OPEN the inlet and outlet valves, **AND** allow the GGTC to purge for 10 minutes or at the discretion of the LO.

4.9.6 CLOSE the inlet valve, **AND** disconnect the air or nitrogen from the GGTC.

4.9.7 Allow the drum to cool until it is below 37 °C (100 °F).

4.10 GGTC Data Calculation

4.10.1 Open Data calculation software, **AND** verify the GGTP Data Calculation software is the same version on the ftp site, ftp://q.wipp.ws/.

4.10.2 In the GGTP Data calculation software, enter BDR number, drum number, R_{eff}, drum void volume, CH_4/H_2 ratio, closure date, GGTC number, and isolation date.

4.10.3 Upload raw data or manually input data acquired from MGSS into GGTP Data Calculation software spreadsheet.

4.10.4 Review all the data entered into the GGTP calculation software spreadsheet and ensure it is correct.

NOTE

In some drums the Total Gas Generation Rate may be less than the -2.00X10-6 negative rate limit. This can be explained by the depletion of oxygen with the formation of rust (iron oxide) and is indicated by the reduction of the concentration of oxygen from the initial sampling event to the final sampling event. The GC is not calibrated for oxygen but the peaks in the sample results may be used in a qualitative manner to determine if oxygen depletion is occurring. This indicates a chemical reaction is occurring and would provide the basis for a "use-as-is" disposition on a nonconformance report.

4.10.5 **IF** the Total Gas Generation Rate is less than -2.00X10-6 negative limit,
THEN initiate an NCR in accordance with CCP-QP-005.

4.10.6 **IF** the Negative Hydrogen Gas Generation Rate is a result of low concentrations (high uncertainty) of hydrogen,
THEN the drum is acceptable.

4.10.7 **IF** the Negative Hydrogen Gas Generation Rate is **NOT** a result of high uncertainty,
THEN NCR drum to be retested in accordance with CCP-QP-005.

4.10.8 **IF** the hydrogen concentration on the initial sample is zero, **AND NOT** due to instrument problems,
THEN the Hydrogen Gas Generation Rate is acceptable because this is the most conservative rate.

4.10.9 Print the following Attachments from the GGTP Calculation Software spreadsheet for the BDR:

[A] Attachment 7

[B] CCV report

[C] Drum Data Sheet(s)

[D] ITR Checklist

[E] QAO Checklist

4.10.10 Add the following to the BDR:

[A] GC Chromatogram(s)

[B] Drum Information (Attachment 1)

[C] GGTC Checklist (Attachment 2)

[D] Copies of NCRs, if applicable

4.10.11 Paginate BDR and complete Table of Contents.

4.10.12 Review all documents, **AND** print, sign, and date the report forms.

4.10.13 Forward BDR to ITR.

4.11 Data Review

ITR

4.11.1 Review BDR using Attachment 5.

4.11.2 Reconcile discrepancies with Operator/TS.

4.11.3 Complete, print name, sign, and date Attachment 5.

4.11.4 Forward BDR to QAO.

QAO

4.11.5 Review BDR using Attachment 6.

4.11.6 Reconcile discrepancies with ITR and Operator/TS.

4.11.7 Complete, print name, sign, and date Attachment 6.

4.11.8 Submit BDR to Facility Records Custodian.

Facility Records Custodian

4.11.9 Receive, process, and transmit BDR in accordance with CCP-QP-008.

5.0 RECORDS

5.1 Records generated during the performance of this procedure are maintained as quality assurance (QA) records in accordance with CCP-QP-008.

5.1.1 QA/Nonpermanent

[A] BDR

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

[A.2] CCV Report

[A.3] Drum Data Sheet(s)

[A.4] ITR Checklist – Attachment 5

[A.5] QAO Checklist – Attachment 6

[A.6] GC Chromatogram(s)

[A.7] Drum Information

[A.8] GGTC Checklist

[A.9] Copies of NCRs, if applicable

[B] ICAL BDR

[B.1] Data Report Cover Page – Attachment 7

[B.2] Calibration Report

[B.3] GC Chromatogram(s)

[B.4] COA (s)

Attachment 1 – Drum Information – Example

BDR Number:_____

1. Drum No.	2. Was Drum Lid Removed <14 days after HSG? (Y or N)	3. Was closure date >38 days from initial sampling: (Y or N)	4. Acceptable to test? (Y or N)	5. Closure date?	6. No. Layers of Confinement	8. Rigid Liner? (Y or N)	9. %Fill	10. Calculated void volume (220-[%Fill X 192])

Attachment 1 – Drum Information – Example (Continued)

BDR Number:_____

1. Drum No.	2. Filter Model Number(s)	3. Hydrogen Diffusivity of Filter(s) (md/s/mf)	4. R_{eff}	5. Determine CH_4/H_2 Ratio, or enter default value (.1)

Operator/TS			ITR		
Print Name	Signature	Date	Print Name	Signature	Date

Attachment 2 – GGTC Checklist – Example

BDR Number: Date: Torque Wrench ID: Torque Wrench Cal Due Date:

GGTC No.	Temperature Controller ID	Temperature Controller Cal. Due Date	Pressure Gauge ID	Pressure Gauge Cal Due Date	Drum No.	Bolts @ 44 ft-lb (±5) Torque Initials/Date	Leak Test P – Pass	Isolation Initials/Date/Time	Peak/Valley (°C)

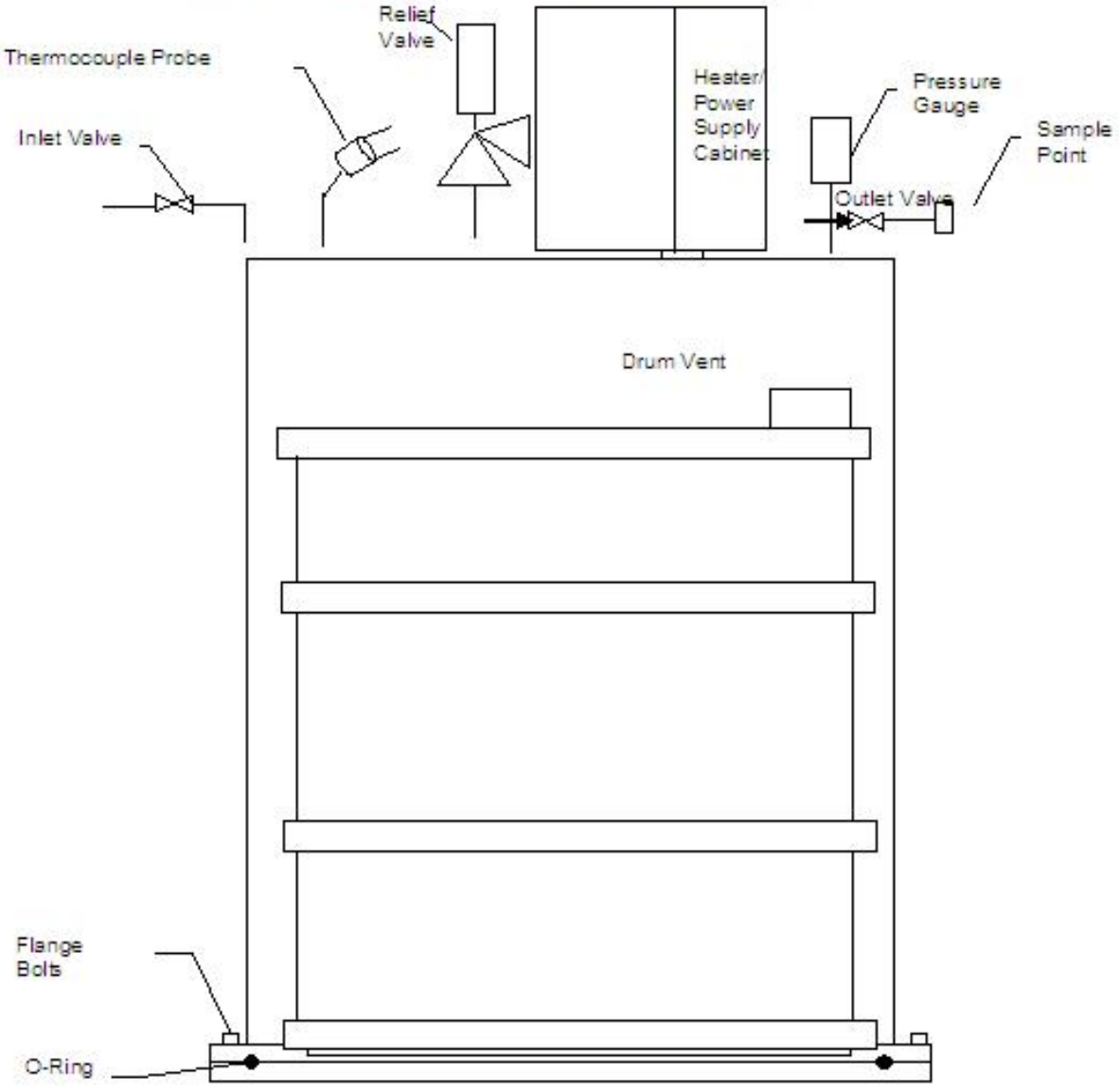
Operator/TS

Print Name _____ Signature _____ Date _____

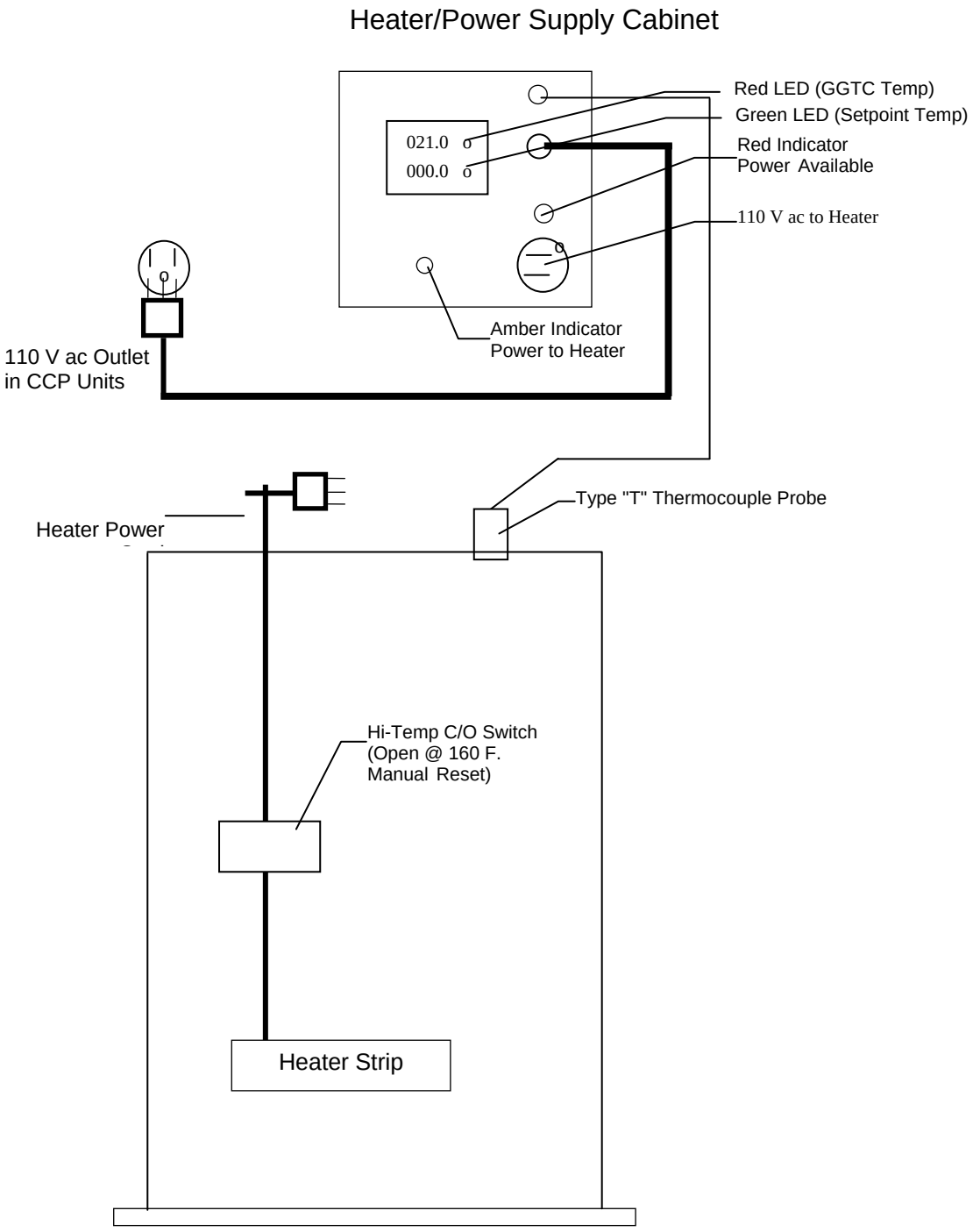
Attachment 3 – Gas Generation Test Canister

GAS GENERATION TEST CANISTER

NOTE: *The actual box may be mounted differently than displayed here.*



Attachment 4 – Heating System Schematic



Attachment 5 – ITR Checklist

BDR Number: _____

	Yes	No	N/A	
1				Was the generation and reduction of data conducted in a technically correct manner in accordance with the method used?
2				Was the data reported in the proper units and significant figures?
3				Were the calculations performed with a valid calculation program?
4				Is the QA document, CCV, complete?
5				Does the CCV meet the %R requirement?
6				Was the correct Operating Software used?
7				Was the correct Application Software used?
8				Was the correct Application Software version used?
9				For each drum and QC, do the hydrogen concentrations, pressure and temperature on Sheet 2 match the Raw Data?
10				Are the Drum Numbers correct on data sheets?
11				If applicable are the NCRs attached?
12				Was the data reviewed for transcription errors?

Print Name _____ Signature _____ Date _____

Attachment 6 – QAO checklist

BDR Number: _____

	Yes	No	N/A	
1				Is the BDR complete?
2				Was precision and accuracy demonstrated through an error propagation analysis?
3				Was a Leak Test performed (Representativeness)?
4				Were all of the GGTC samples collected and analyzed (Completeness) for all of the drums listed on Drum Information?
5				Was the procedure approved by CBFO?
6				Were calibration standards NIST traceable or manufacturer-certified (Comparability)?
7				Did the CCV meet the %R requirement (Comparability-Instrument Accuracy)?
8				Did Hydrogen meet the coefficient of determination (r^2) requirement for the ICAL (Comparability-Instrument Precision)?

Print Name _____ Signature _____ Date _____

Attachment 7 – Data Report Cover Page – Example

COVER PAGE

BDR Number: _____

NCR Number (if applicable): _____

ICAL BDR Number: _____

TABLE OF CONTENTS

Report Content:

Content	Page Number

_____ _____ _____
Operator Printed Name Signature Date

_____ _____ _____
ITR Printed Name Signature Date

_____ _____ _____
QAO Printed Name Signature Date

Attachment 8 – Gas Generation Testing Continuing Calibration Verification – Example

GGT Batch Number: _____

CCV FOR INITIAL SAMPLING EVENT

Certified Concentration (ppm): _____

Initial CCV Date/Time: _____

Hydrogen Concentration (ppm): _____

Initial CCV % R _____

Pass/Fail _____

CCV FOR FINAL SAMPLING EVENT

Certified Concentration _____

Initial CCV Date/Time: _____

Hydrogen Concentration (ppm): _____

Initial CCV %R _____

Pass/Fail _____

Operator/TS: _____
Sign Date

Attachment 9 – Gas Generation Testing Drum 2 Data Sheet – Example

GGT Batch Number: _____ GGTC Number: _____

Drum Number: _____

Hydrogen Generation Rate: _____

Hydrogen/Methane Generation Rate: _____

Drum Gas Volume: _____

Initial Testing Date/Time: _____

Final Testing Date/Time: _____

Total Gas Generation: _____

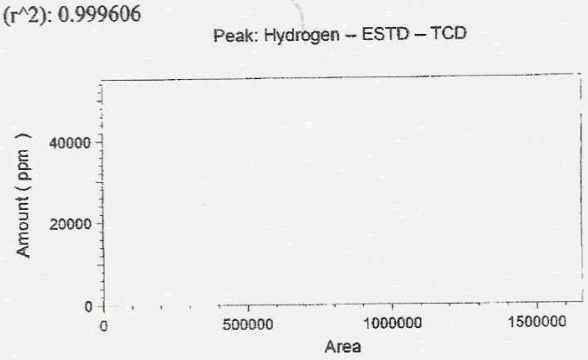
Operator/TS: _____
Sign Date

Attachment 10 – Calibration Report – Example

Calibration Report

Method: C:\CLASS-VP\Enterprise\Projects\Default\Method\GGTP092806.met
Print Time: 9/28/2006 10:08:00 AM
User: System
Instrument: GC-14B

Hydrogen (TCD)
Average RF: 0.0344514 RF StDev: 0.00141975 RF %RSD: 4.12102
Scaling: None LSQ Weighting: None Force Through Zero: Off
Replicate Mode: Replace
Fit Type: Linear
y = 0.0333788x + 140.694
Goodness of fit (r^2): 0.999606



	Level 1	Level 2	Level 3	Level 4	Level 5
Amount	100	999	10000	30100	50000
Area	2714	29180	297581	875820	1505873
RF	0.036845983	0.03423577793	0.033604295973	0.034367792468	0.0332033312
	7877671	00891	1972	7721	23815
Last Area					
Residual	-131.284	-115.688	-73.5904	725.487	-404.925
Rep StDev					
Rep %RSD					
Rep 1 Area	2714	29180	297581	875820	1505873
Rep 1 User	System	System	System	System	System
Rep 1 Data File	C:\CLASS-V P\Data\ICAL 092806\LEV EL 1.dat	C:\CLASS-VP\ Data\ICAL0928 06\LEVEL 2.dat	C:\CLASS-VP\ Data\ICAL0928 06\LEVEL 3.dat	C:\CLASS-VP\ Data\ICAL0928 06\LEVEL 4.dat	C:\CLASS-VP\ Data\ICAL092 806\LEVEL 5.dat
Rep 1 Sample ID	630 22	630 22	630 22	630 22	630 22
Rep 1 Calib. Time	9/28/2006 9:41:29 AM	9/28/2006 9:47:15 AM	9/28/2006 9:53:02 AM	9/28/2006 9:58:53 AM	9/28/2006 10:08:00 AM

Aaron deBruyn Kops Aaron deBruyn Kops 9-28-06

000021