Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

WTP Waste Feed Qualification: Hydrogen Generation Rate Measurement Apparatus Testing Report

M. E. Stone

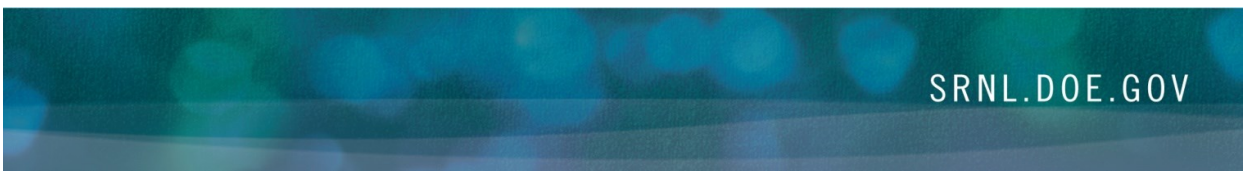
J. D. Newell

T. E. Smith

J. M. Pareizs

June 2016

SRNL-STI-2016-00247, Revision 0



DISCLAIMER

Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2016-00247
Revision 0

Keywords: *Hydrogen*
Mixing
Measurement
Qualification

Retention: *Permanent*

WTP Waste Feed Qualification: Hydrogen Generation Rate Measurement Apparatus Testing Report

M. E. Stone
J. D. Newell
T. E. Smith
J. M. Pareizs

June 2016

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

Savannah River National Laboratory™
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

REVIEWS AND APPROVALS

AUTHORS:

M. E. Stone, Hanford Missions Programs Date

TECHNICAL REVIEW:

C. L. Crawford, Engineering Process Development, Reviewed per E7 2.60 Date

APPROVAL:

C. C. Herman, Manager Date
Hanford Missions Programs

ACKNOWLEDGEMENTS

The authors would like to acknowledge the assistance of the following individuals in completion of this task: Whitney Riley, Courtney Burckhalter, and Beverly Wall for assistance with sample analysis, Holly Hall, Jonathon DuVall, Andy Foreman, Madison Caldwell, Phyllis Workman, and Vickie Williams for coordination and performance of simulant testing, Frances Williams for assistance with coordination and setup of experimental apparatus and calibration of the gas chromatograph, J. M. Pareizs for assistance with the gas chromatograph setup and calibration, Bridget Miller for coordination of M&TE calibrations, Curt Sexton and Gary Dobos for glass design consultation and fabrication of the glassware, Ron Blessing for design consultation, Jane Howard and Rita Sullivan for performing mockup testing, Babb Attaway and Maurice Lee for coordination of cells testing, Kevin Hera and Larry Feutral for assistance with drawing preparations, Don Pak for design and fabrication of the apparatus stand, Bruce Boulineau for photography, Duane Adamson and Connie Herman for test program coordination, and Rick Pelfrey for program management assistance.

EXECUTIVE SUMMARY

The generation rate of hydrogen gas in the Hanford tank waste will be measured during the qualification of the staged tank waste for processing in the Hanford Tank Waste Treatment and Immobilization Plant. Based on a review of past practices in measurement of the hydrogen generation, an apparatus to perform this measurement has been designed and tested for use during waste feed qualification.

The hydrogen generation rate measurement apparatus described in this document and shown in Figure 0-1 utilized a 100 milliliter sample in a continuously-purged, continuously-stirred vessel, with measurement of hydrogen concentration in the vent gas. The vessel and lid had a combined 220 milliliters of headspace. The vent gas system included a small condenser to prevent excessive evaporative losses from the sample during the test, as well as a demister and filter to prevent particle migration from the sample to the gas chromatography system. The gas chromatograph was an on line automated instrument with a large-volume sample-injection system to allow measurement of very low hydrogen concentrations. This instrument automatically sampled the vent gas from the hydrogen generation rate measurement apparatus every five minutes and performed data regression in real time.

The fabrication of the hydrogen generation rate measurement apparatus was in accordance with twenty-three (23) design requirements documented in the conceptual design package, as well as seven (7) required developmental activities documented in the task plan associated with this work scope.

The HGRMA was initially tested for proof of concept with physical simulants and a remote demonstration of the system was performed in the Savannah River National Laboratory Shielded Cells Mockup Facility. Final verification testing was performed using non-radioactive simulants of the Hanford tank waste. Three different simulants were tested to bound the expected rheological properties expected during waste feed qualification testing. These simulants were tested at different temperatures using purge gas spiked with varying amounts of hydrogen to provide verification that the system could accurately measure the hydrogen in the vent gas at steady state. Refer to Table 4-1 for the testing summary.

Conclusions

The HGRMA was designed, fabricated, and subjected to several iterations of testing. The fabricated apparatus has been demonstrated to meet all twenty-three (23) design requirements, except for slight variation in the temperature control, and ability to drain 100 percent of the introduced samples. Discussions of variations are provided below. Additionally, each of the seven (7) developmental activities identified in the task plan have been completed with the appropriate documentation that was reviewed and accepted by WTP. These seven (7) development requirements were associated with design and testing of the hydrogen generation rate measurement apparatus system.

The testing has shown that the fabricated HGRMA is capable of performing the measurements with the required accuracy to ensure that the action limits for hydrogen generation rate in the waste feeds will not exceed the waste acceptance criteria.

Krypton was successfully used as a tracer gas during the testing. A bottle of zero air with krypton spiked in at 0.5% should be used as the purge gas for the HGRMA during waste feed qualification.

After initial draining the hydrogen generation rate measurement apparatus required additional rinsing of the vessel in place and cleaning to remove the sample residue beyond the rinsing and cleaning originally

specified. The vessel should be removed and manually cleaned between runs after the rinsing is complete. A nitric acid rinse is not required if manually cleaning the vessel.

The temperature specification for the HGR measurement was to maintain the targeted temperature within one degree Celsius. However, the testing demonstrated the apparatus held the target temperature within two degrees Fahrenheit of the setpoint, which is 1.1 degree Celsius. The two degree Fahrenheit variation was monitored using RTD probe readouts. The temperature target during testing should be adjusted such that the measurement temperature is always above the required temperature. Since the apparatus was able to maintain the setpoint within two degrees Fahrenheit, the temperature target during testing should be two degrees above the required temperature. Therefore, if the required temperature for the HGR measurement is 120 degrees, the testing should be performed at 122 +/- 2 degrees Fahrenheit.

The time for the hydrogen measurement to reach steady-state was determined to be approximately 8 hours based on a model of the system as well as the results of the testing. The testing duration required to reach steady measurement needs to be verified during testing with actual radioactive waste samples to verify any changes due to radiolytic hydrogen generation. The use of a krypton tracer gas will allow for identification of steady-state conditions during tests below the detection limit for hydrogen.

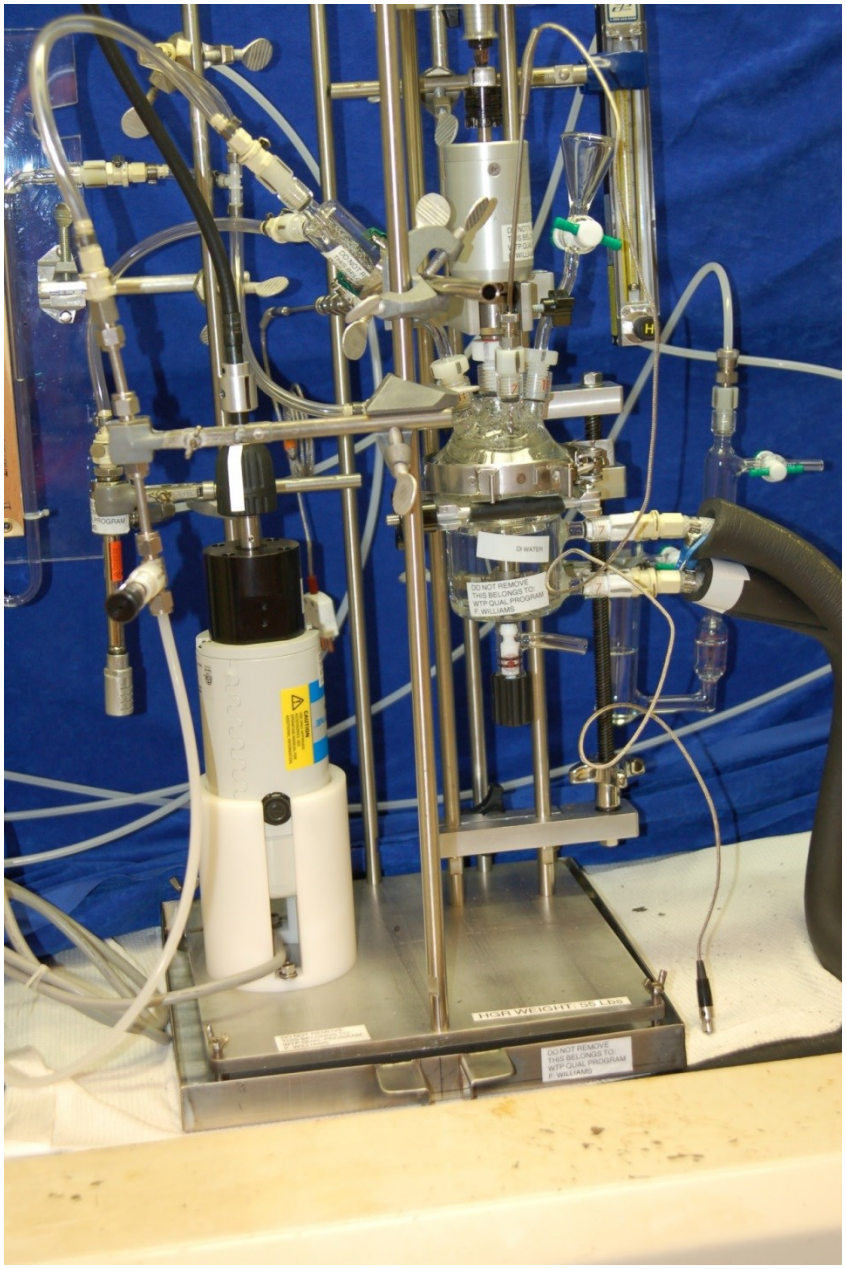


Figure 0-1. HGRMA Apparatus

TABLE OF CONTENTS

LIST OF TABLES xii
LIST OF FIGURES xii
LIST OF APPENDIX TABLES xiii
LIST OF APPENDIX FIGURES xiii
LIST OF ABBREVIATIONS xvii
1.0 Introduction 1
2.0 Experimental Procedure 8
2.1 Simulated Wastes 8
2.2 Calibration and Tracer Gases 8
2.3 Test Matrix 8
2.4 Test Apparatus 10
2.5 Test Procedure 11
2.6 Technical Review Process 11
3.0 Results and Discussion 12
3.1 HGR1a Testing 12
3.1.1 HGR1a Loading and Draining the Vessel 12
3.1.2 HGR1a Hydrogen Measurements 12
3.2 HGR2a Testing 13
3.2.1 HGR2a Loading and Draining the Vessel 13
3.2.2 HGR2a Hydrogen Measurement 13
3.3 HGR3a Testing 14
3.3.1 HGR3a Loading and Draining the Vessel 14
3.3.2 HGR3a Hydrogen Measurements 14
3.4 HGR3b Testing with Tracer Gas 15
3.4.1 HGR3b Loading and Unloading the Vessel 15
3.4.2 HGR3b Hydrogen and Krypton Measurements 17
3.5 Hydrogen Measurement Results Summary 18
3.5.1 HGRMA Vessel CSTR Model 18
3.5.2 Comparison of data to CSTR model 19
3.5.3 Air Only Tests 21
3.5.4 Detection Limits 21
3.5.5 Impact of Concentration on Measurement Error and Variability 21
3.5.5.1 Error/Uncertainty 21

3.5.5.2 Variability 22
3.5.6 Impact of Changes in Environmental Conditions Around the Apparatus 22
3.6 Other Testing 22
3.6.1 Water Runs 22
3.6.2 Initial Tracer Gas Testing 23
3.6.3 Load Test 23
3.7 General Observations and Lessons Learned 23
3.7.1 Flow Controller Failure 23
3.7.2 Water Bath Issues 23
3.7.2.1 Erosive Wear 24
3.7.3 Calibration Gas Consumption 25
3.7.3.1 Negative Pressure during Cooling 26
3.7.4 HGRMA System Performance 26
3.7.4.1 Temperature Control 26
3.7.4.2 Condenser/Vortex Chiller 27
3.7.4.3 Vessel Air Leak Checks 27
3.7.4.4 Addition Funnel 27
3.7.4.5 Manometer 27
3.7.4.6 Mixing System 27
3.7.4.7 Other Systems 27
3.8 Other Requirements 28
3.8.1 Work Instructions for HGRMA Operation 28
3.8.2 HGRMA Assembly and Tear Down Instructions 28
3.8.3 Work Instructions for GC Operation 28
3.8.4 Packaging Plan 28
3.8.5 Design Drawings 28
4.0 Testing Results Versus Design Requirements 29
5.0 TTQAP Documentation 41
6.0 RAMI Review 43
6.1 Reliability 43
6.2 Availability 44
6.3 Maintainability 47
6.4 Inspectibility 47
7.0 Conclusions 48

8.0 Recommendations 48

9.0 References 50

Appendix A . Hydrogen Measurement Method and Graphs A-1
Appendix B . HGR CSTR Model Results B-1
Appendix C . HGRMA Assembly Instructions C-1
Appendix D . HGRMA Operating Instructions D-1
Appendix E . GC Instructions E-1
Appendix F . RAMI Review F-1
Appendix G . Packaging Plan G-1
Appendix H . Design Drawings H-1
Appendix I . HGRMA Test Instructions I-1
Appendix J . Supplemental Temperature Testing J-1
Appendix K . Simulant Recipes K-1

LIST OF TABLES

Table 1-1. Staged Waste Feed Action Limits 1
Table 1-2 HGRMA Design Requirements 3
Table 1-3 HGRMA Task Technical and Quality Assurance Plan Activities 5
Table 2-1. HGRMA Verification Testing Simulants 8
Table 2-2 HGRMA Verification Test Matrix 9
Table 3-1. HGR1a Hydrogen Results 13
Table 3-2. HGR2a Hydrogen Results 14
Table 3-3. HGR3a Hydrogen Results 15
Table 3-4. Hydrogen Results from HGR3b-3 17
Table 3-5. Krypton Results from HGR3b-3 18
Table 3-6. Water Run Results 22
Table 4-1 HGRMA Design Requirements and Testing Summary 39
Table 5-1 HGRMA Technical Task Documentation Summary 41
Table 6-1. Failure Mode Assessment 44

LIST OF FIGURES

Figure 0-1. HGRMA Apparatus viii
Figure 2-1 HGRMA Apparatus 10
Figure 3-1. Splatter During HGR3b-1 16
Figure 3-2. Residual Solids after Draining and Rinsing 17
Figure 3-3. CSTR Model of HGRMA Vessel 19
Figure 3-4. Comparison of Data to HGRMA CSTR Model 20
Figure 3-5. Comparison of Data from HGR3b with CSTR Model 21
Figure 3-6. Impact of Concentration on Relative Standard Deviation of Steady State Data 22
Figure 3-7. Crack in Failed Water Fitting 24
Figure 3-8. Erosive Wear on Vessel 25
Figure 3-9. Erosive Wear on Impeller 25

Figure 3-10. Temperature Profile during HGR3a-4 26

LIST OF APPENDIX TABLES

Table B-1. Factors Used to Adjust HGRMA Gas Chromatography Data B-1
Table F-1. HGRMA RAMI Review Summary F-1
Table K-1. Recipe for LAW Simulant K-1
Table K-2. Recipe for HLW Low Bound Simulant K-2
Table K-3. Recipe for HLW High Bound Simulant K-3
Table K-4. Recipe for LAW Low Bound GFCs K-4
Table K-5. Recipe for LAW High Bound GFCs K-5
Table K-6. Recipe for HLW Low Bound GFCs K-6
Table K-7. Recipe for HLW High Bound GFCs K-7

LIST OF APPENDIX FIGURES

Figure A-1. GC Setup Parameters A-1
Figure A-2. Channel A Setup Parameters A-2
Figure A-3. Channel B Setup Parameters A-2
Figure A-4. GC Integration Settings A-2
Figure A-5. Gas Chromatography Method Peak A-3
Figure A-6. Chromatogram from Run with 20 parts per million Air and Krypton Tracer A-3
Figure A-7. 20 parts per million Hydrogen Chromatogram Enlarged to Show Hydrogen Peak A-4
Figure A-8. Chromatogram of ACTL Instrument Air A-4
Figure A-9. ACTL Instrument Air Chromatogram Enlarged to Show Helium and Hydrogen Peaks A-5
Figure A-10. HGR1a-1 Test Results A-6
Figure A-11. HGR1a-2 Test Results A-6
Figure A-12. HGR1a-3 Test Results A-7
Figure A-13. HGR1a-4 Test Results A-7
Figure A-14. HGR1a-Air Test Results A-8

Figure A-15. HGR2a-1 Test Results A-8
Figure A-16. HGR2a-2 Test Results A-9
Figure A-17. HGR2a-2 Test Results A-9
Figure A-18. HGR2a-4 Test Results A-10
Figure A-19. HGR2a-Air Test Results A-10
Figure A-20. HGR3a-1 Test Results A-11
Figure A-21. HGR3a-2 Test Results A-11
Figure A-22. HGR3a-3 Test Results A-12
Figure A-23. HGR3a-4 Test Results A-12
Figure A-24. HGR3a-Air Test Results A-13
Figure A-25. HGR3b-3 Test Results A-13
Figure B-1. HGR1a-1 Results Overlaid with CSTR Model B-2
Figure B-2. HGR1a-2 Results Overlaid with CSTR Model B-3
Figure B-3. HGR1a-3 Results Overlaid with CSTR Model B-4
Figure B-4. HGR1a-4 Results Overlaid with CSTR Model B-5
Figure B-5. HGR2a-1 Results Overlaid with CSTR Model B-6
Figure B-6. HGR2a-2 Results Overlaid with CSTR Model B-7
Figure B-7. HGR2a-3 Results Overlaid with CSTR Model B-8
Figure B-8. HGR2a-4 Results Overlaid with CSTR Model B-9
Figure B-9. HGR3a-1 Results Overlaid with CSTR Model B-10
Figure B-10. HGR3a-2 Results Overlaid with CSTR Mode B-11
Figure B-11. HGR3a-3 Results Overlaid with CSTR Model B-12
Figure B-12. HGR3a-4 Results Overlaid with CSTR Model B-13
Figure B-13. HGR3b Testing with Tracer Gas B-14
Figure B-14. Water Run 70-20 Results Overlaid with CSTR Model B-15
Figure B-15. Water Run 140-20 Results Overlaid with CSTR Model B-16
Figure B-16. Water Run 70-5 Results Overlaid with CSTR Model B-17
Figure B-17. Water Run 140-5 Results Overlaid with CSTR Model B-18
Figure C-1. HGR Stand C-5

Figure C-2. Containment Pan C-5
Figure C-3. Installation of Magnetic Coupling and Upper Agitator Shaft C-6
Figure C-4. Magnetic Coupling Support C-7
Figure C-5. Vessel Head C-8
Figure C-6. Vessel Head Installation Support C-9
Figure C-7. Lower Agitator Shaft and Detail of Threads C-10
Figure C-8. Vessel Installation C-11
Figure C-9. Vessel Support C-12
Figure C-10. Air Purge Line Connections C-13
Figure C-11. Air Purge Line Connection Mounted to Stand and CPC PMC12 Series Quick-Connect . C-13
Figure C-12. Condenser Installation C-14
Figure C-13. Condenser Chiller Air Outlet RTD Installation C-15
Figure C-14. Vortex Chiller C-16
Figure C-15. Vortex Chiller Rotameter C-17
Figure C-16. Manometer Assembly C-18
Figure C-17. Ace-Thred Diagram C-19
Figure C-18. Bubbler Support and Bubbler C-20
Figure C-19. Filter and Septum C-21
Figure C-20. Water Bath and Connections C-22
Figure C-21. Agitator Holder C-23
Figure C-22. Flexible Shaft with Cable End Collar Installed C-24
Figure C-23. Flexible Shaft Support C-25
Figure C-24. Agitator Wiring Amphenol Connectors, with clamp installed C-26
Figure C-25. O-ring Installation Tool and O-ring Removal Tool C-26
Figure C-26. Markings on Threaded Parts and Ace-Thred after Optional Machining C-27
Figure C-27. Assembled HGR Measurement Apparatus, Side View C-28
Figure C-28. Assembled HGR Measurement Apparatus, Front View C-29
Figure G-1. Packaged HGRMA G-1
Figure G-2. HGRMA Vessel in Case G-2

Figure G-3. HGRMA Miscellaneous Components in Case G-2

Figure G-4. HGRMA Water Bath and Controllers, RTD Readout, and Miscellaneous Components in Case G-3

Figure H-1. TECA Chiller Remoting Diagram H-1

Figure H-2. HGRMA Process Flow Diagram H-2

Figure H-3. HGRMA Assembly H-3

Figure H-4. HGRMA Vessel Assembly and Details H-4

Figure H-5. Vessel Assembly Turntable H-5

Figure H-6. HGRMA Glassware Details H-6

Figure H-7. HGRMA Stand Details Sheet One H-7

Figure H-8. HGRMA Stand Details Sheet 2 ..... H-8

Figure H-9. HGRMA Stand Details Sheet Three H-9

Figure J-1. Supplemental Temperature Test at 140 °F J-1

Figure J-2. Supplemental Temperature Test at 194 °F J-2

Figure J-3. HGRMA Neoprene Insulating Sleeve Dimensions J-0

LIST OF ABBREVIATIONS

ACTL	Aiken County Technology Laboratory
CPC	Colder Products Company
CSTR	Continuously Stirred Tank Reactor
DI	Deionized Water
DWPF	Defense Waste Processing Facility
GC	Gas Chromatograph
HGR	Hydrogen Generation Rate
HGRMA	hydrogen generation rate measurement apparatus
HLW	High-Level Waste
LAW	Low-Activity Waste
M&TE	Measurement & Testing Equipment
NPT	National Pipe Thread
MTBC	Mean Time Between Calibrations
MTBF	Mean Time Between Failures
MTBM	Mean Time Between Maintenance
MTTC	Mean Time To Calibrate
MTTM	Mean Time To Maintain
MTTR	Mean Time To Repair
PI	Principal Investigator
POC	Proof of Concept
R&D	Research and Development
RAMI	Reliability, Availability, Maintainability, and Inspectibility
RTD	Resistive Temperature Detector
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.0 Introduction

The Hanford Site is storing millions of gallons of high level liquid waste generated during production of plutonium and other nuclear materials in large underground storage tanks. The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is designed to immobilize the tank waste by conversion of the waste into glass products. Two types of glass will be produced: a high level waste (HLW) glass that contains the majority of the radionuclides and will eventually be sent to a federal repository, and a low activity waste (LAW) glass that is designed to meet Class C limits for land disposal.

Hydrogen is generated by the tank waste from radiolysis of water as well as from chemical reactions of the organics in the waste with oxidizers also contained in the waste. Correlations have been developed to predict the hydrogen generation rate (HGR) from the tank waste, but these correlations have a large amount of uncertainty (300%) [1]. Therefore, WTP embarked on a program to develop an apparatus and ancillary method to measure HGR[2].

The HGR action limits for staged LAW and HLW feeds[3] to the WTP pretreatment feed receipt vessels are provided in Table 1-1[1]. The detection limit for HGR measurement apparatus (HGRMA) was based on the limit for the staged LAW feed.

Table 1-1. Staged Waste Feed Action Limits

Waste Stream	Action Limit (gram-moles hydrogen per liter-hour)
Staged HLW Feed	2.1E-06 at 150 degrees Fahrenheit
Staged LAW Feed	3.7E-07 at 120 degrees Fahrenheit

Measurement of HGR from tank wastes during the qualification of tank waste feeds for processing in the WTP is planned [4], as well as measurement of HGR from process slurries generated during unit operations testing as part of the WTP waste feed qualification program.

Development of the HGRMA was performed in accordance with the scope of work SCT-M0SRV00028-00-012, *Inter-Entity Work Order M0SRV00028 Task 12* [2]. As part of this work scope the following activities were completed.

- A literature study was performed that recommended a continuously purged continuously stirred tank reactor (CSTR)[a] vessel with a gas chromatography measurement of the vent gas as the method to be used for the HGR measurement during qualification[1].
- A Task Technical and Quality Assurance Plan[5] was issued to provide additional guidance for development and testing of the HGRMA.
- A conceptual design[6] was performed that provided design criteria and preliminary design
- The apparatus was designed and fabricated with design reviews at the 30[7], 60[8] and 90% completion intervals.
- A final design package was prepared[9].
- Initial Proof of Concept (POC) testing was performed on the apparatus and individual components[10] included in the design.
- Mockup testing was performed to evaluate the remote operation and maintenance of the apparatus[11].

[a] CSTR is a standard term used in chemical engineering and does not necessarily imply that the vessel is a reaction vessel.

- Final verification testing of the apparatus was performed with non-radioactive simulants to evaluate the performance of the system for conditions that bound the expected rheological properties and temperature conditions expected during qualification[12].

The design requirements for the HGRMA were originally documented in the conceptual design document[6]. Those twenty-three (23) design requirement are re-summarized in Table 1-2 below.

Table 1-2 HGRMA Design Requirements

Req.	Applies to	Design Requirement Description
1.	Overall System	*The apparatus will be designed to perform HGR measurements with sufficient accuracy to allow a determination of whether the sample meets the HGR action limits. The most restrictive action limit is for the Low-Activity Waste receipt samples at 3.7E-7 gram mole of hydrogen per liter per hour at 120 degrees Fahrenheit (48.9 degrees Celsius).*
2.		*A leak check will be performed on the system prior to testing to ensure that 90% of the purge flow entering the vessel is measured in the vent gas.*
3.		*A tracer will be used in the purge gas to ensure that the analyzer is measuring gas from the vessel and not from room air leaking into the vent gas line.*
4.		*The sample will be held within one degree Celsius of the specified temperature during the HGR measurement.*
5.		*The sample will be well mixed during the HGR measurement.*
6.		*The individual components on the apparatus will be remoted to allow removal and replacement of degraded or failed components.*
7.		*As part of the final design process, all subcomponents of the apparatus that may contain software will be checked against the applicability of NQA-1-2000 Part II Subpart 2.7. Additionally, if a data collection software system is used, it will also be reviewed to determine if NQA-1-2000 Part II Subpart 2.7 is applicable during final design.*
8.	Apparatus Support	*The apparatus support will allow the HGR measurement apparatus to be safely installed in the shielded cells facility and will provide support for equipment during operation.*
9.		*The containment pan will be capable of holding the entire contents of the vessel.*
10.	Lid and Vessel	*The vessel must be able to contain 100 milliliters of sample with some vapor space at the top, allow complete draining of sample, provide a seal adequate to meet leak check requirements, and allow visual inspection of cleanliness and mixing.*
11.	Mixing System	*The mixing system must be able to maintain a small surface vortex without excessive splatter for samples with Bingham Plastic yield stresses of 0 to 30 Pascals while maintaining a seal sufficient to meet leak check requirements*
12.	Air Purge System	*The air purge system must deliver a purge gas with a tracer to the vessel with sufficient accuracy to allow the vent gas measurement to be sufficiently accurate to ensure that the HGR action limits are not exceeded*
13.		*The air purge system must include pressure relief to prevent over pressurization of the HGR apparatus*
14.		*The air purge system must be sufficiently leak tight to meet leak check requirements*
15.	Temperature Control	*The temperature control system must maintain the vessel contents at specified temperature to within one degree Celsius during the HGR measurement. Specified temperatures will be between 25 and 90 degrees Celsius.*

Req.	Applies to	Design Requirement Description
16.	Sample Gas System	*The sample gas system must be sufficiently leak tight to meet leak check requirements.*
17.		*The sample gas system must sufficiently treat the vent gas to prevent damage to the gas analyzer.*
18.		*The sample gas system must prevent sample loss through evaporation*
19.		*The sample gas system must sufficiently treat the vent gas to allow the gas to exit the shielded cells and be monitored by an analyzer in a fume hood*
20.		*The gas analyzer must be sufficiently accurate to perform HGR measurements to allow determination of whether the sample meets the specified action limits.*
21.		*The gas analyzer must be able to be calibrated in place.*
22.		*The sample gas system will include provisions for flow measurements during leak checks to allow the overall sealing of the HGR measurement apparatus to be assessed.*
23.	Operation of the Apparatus	*The measurement of HGR using the HGR apparatus will be performed using the steps / general actions identified in Section 11.0 of the conceptual design document.*[4]

The results of the various phases of testing of the HGRMA and whether or not the design criteria were met are documented in sections 3.0 and 4.0.

The Task Technical and Quality Assurance Plan[5] outlined the necessary activities to complete the development and testing of the HGRMA. Those activities are summarized in Table 1-3 below.

Table 1-3 HGRMA Task Technical and Quality Assurance Plan Activities

TTQAP Section	Activity Description	Activity Detail
4.1	Design of HGRMA	*The design of the HGR measurement apparatus will be performed based on the criteria developed during the literature review [1]. Design drawings will be prepared and submitted for review in two phases. Phase I will be considered Conceptual Design and will consist of the recommended equipment and a line diagram showing connections. Definitive design drawings (Phase II) will include final dimensions and a 3D rendering of the final assembly plan, list of equipment, and a Piping and Instrumentation drawing. These documents will be reviewed and accepted by WTP prior to fabrication of the apparatus. The design will take into consideration that the unit must be able to be assembled, operated and disassembled remotely in a laboratory hot cell, and will be shipped to the WTP at the completion of testing.*
4.2	Fabrication of the HGRMA	*This task will commence once the design documents are accepted by the WTP. Long lead time components may be procured prior to approval of the design documentation with concurrence from WTP. Fabrication will include procurement or fabrication of required components, assembly of components in the HGR measurement apparatus, and system checkout to include leak checks and water runs.* *The analytical instrument will likely utilize software supplied by the vendor. Controls applicable to Measurement and Sensing Equipment will be applied to the testing at SRNL, which specify a check with standards prior to and after each use to ensure the entire system (including software) is operating as intended.*
4.3	HGRMA POC Testing	*Proof of concept testing will be performed by assessing whether the analytical instrument for the measurement of generated gases can be calibrated for hydrogen in the range required. The calibration test will be performed with and without the mixing vessel attached to the system. If the desired detection limits are not achievable using air as the cover gas, an inert gas purge will be evaluated since an inert cover gas allows a greater sensitivity for hydrogen and other gas species during analysis. Purified bottled air supplies will be used to eliminate the background hydrogen if elevated hydrogen concentrations are noted in the instrument air utilized during the testing.* *In addition to the hydrogen measurements, the system will be evaluated to ensure that it is sufficiently leak tight to perform the HGR measurement with the required precision and accuracy, and that it can adequately mix process slurries. The leak check will be performed using a flow test. A known purge will be introduced into the system and a flow meter will be used to measure the flow out of the vent line. The out flow must match the flow into the system within 10%. As described in the literature review [1], the leak rate from a continuous system will not impact accuracy of the test results.*

TTQAP Section	Activity Description	Activity Detail
		Mixing will be evaluated with Xanthan gum solutions (or other physical simulant) and with chemical simulants of known rheological properties. Adequate mixing will be determined by visual inspection to ensure that no stagnant areas exist along the walls or on the bottom of the mixing vessel and that a slight vortex is noted at the surface. *SRNL will work with WTP representatives to appropriately specify the simulant used during proof of concept testing.*
4.4	Mockup and Final Design of HGRMA	*The HGR measurement apparatus will be tested for remote operation by installing the system in the SRNL Shielded Cells mockup facility. The operability of each component with manipulators will be demonstrated. Any required changes for remote operation will be made and documented. The rig will be assembled and disassembled in mock up. Leak checks will be performed after assembly in mockup to ensure that the apparatus can be remotely sealed.*
4.5	Verification Testing with Simulants	*Tests of the final design of the HGR measurement apparatus will be performed using simulants. A measured volume of the selected simulant will be added to the vessel and mixed while the appropriate calibration gas is passed through the vessel. Initially, the calibration gas will be passed through the vapor space. Subsequent tests will introduce the calibration gas subsurface to validate that the mixing system prevents holdup of hydrogen in the test fluid.* *The offgas analysis will be compared to the calibration gas known values to determine the accuracy and repeatability of the method over the required measurement range of the system.* *The verification testing will be performed using work instructions developed for final operation of the system and will include setup, operation, tear-down, and a cleaning of the system.* *This testing will also include verification that the cleaning methods developed for the system adequately clean the vessel without damaging the system, and can occur in a laboratory hot cell. Cleaning is expected to consist of chemical cleaning with fifty weight percent nitric acid followed by a deionized water rinse. A visual inspection of the system will be performed to ensure the components have been cleaned. If deemed necessary, a second chemical cleaning followed by analysis of the cleaning solution may be performed to verify the initial chemical cleaning was adequate.* *SRNL will work with WTP representatives to appropriately specify the simulant used during verification testing.*

TTQAP Section	Activity Description	Activity Detail
		Once the integrated tests are completed, the HGR measurement apparatus will be packaged and sent to WTP for use during qualification testing. The packaging will be performed as specified in Appendix B of the scope of work.
4.6	Development of Work Instructions for Operating the HGRMA	*The finalized detailed work instructions for setup, operation, tear-down, and cleaning the HGR measurement apparatus will be prepared based on the verification testing and included in the final report.*
4.7	Data Reporting and Submittals	*The results of the HGR measurement apparatus testing will be documented in a final report containing the following information:* • *Detection limits of the HGR measurement apparatus* • *Measurement uncertainty/precision of the method* • *Description of methods used to evaluate and determine the detection limits and measurement uncertainty* • *Description of system design and operation* • *Impact of changes in environmental conditions around the apparatus* *Design of the system will be documented in system drawings at two stages in the design:* • *Conceptual design* • *Definitive design* *Work instructions for final system operation will be documented and submitted to WTP.*

The documentation of completion of the necessary work activities associated with the development, fabrication, and testing of the HGRMA are found in sections 3.0 and 4.0

In addition to the design requirements summarized in Table 1-2, and the required work activities summarized in Table 1-3, the scope of work[2] also requires any apparatus developed for remote operations during waste feed qualification to have a Reliability, Availability, Maintainability, and Inspectibility (RAMI) review performed. Refer to section 6.0 for the results of the RAMI review.

2.0 Experimental Procedure

Task specific Research and Development (R&D) Directions were generated to govern the testing program. These R&D Directions provided specific guidance for experimental parameters to be used while following the HGRMA operating procedure developed for use at the Aiken County Technology Laboratory (ACTL). The testing was performed in fume hoods at ACTL using simulants specified for waste feed process program development by WTP. The testing included a number of scoping tests as well as tests with three different simulated waste feeds[13].

Each of the tests with the three simulated waste feeds had multiple test runs at different temperatures and hydrogen concentrations, including runs with air only to evaluate any hydrogen generation from the simulants from chemical reactions of nitrate with other components (such as acetate) in the waste simulants.

2.1 Simulated Wastes

The three simulants tested are shown in Table 2-1 and consisted of a LAW simulant and two types of melter feed simulants.

Table 2-1. HGRMA Verification Testing Simulants

Test ID	Description
HGR-1	LAW Simulant
HGR-2	LAW High Bound Melter Feed Simulant
HGR-3	HLW High Bound Melter Feed Simulant

The recipes for these simulants are shown in Appendix K.

2.2 Calibration and Tracer Gases

Two calibration gases were used during the testing. The first calibration gas was 20 parts per million (volume basis) hydrogen with the balance air purchased from Air Liquide,. The second was 5 parts per million (volume basis) hydrogen gas, 4.9 % oxygen, and the balance nitrogen also purchased from Air Liquide. The tolerance for each component was +/- 10%.

Krypton (UN1056) and xenon (UN2036) were evaluated for use as tracer gas during the final verification testing. Each gas was purchased as 100% pure gas from Air Liquide..

2.3 Test Matrix

The test matrix is outlined in

Table 2-2 below.

Table 2-2 HGRMA Verification Test Matrix

Test ID	Temperature (Fahrenheit)	Air Flow (milliliters per minute)	Calibration Gas Flow (milliliters per minute)	Expected HGR# (parts per million)
HGR 1a-1	70	1	1	10
HGR 1a-2	70	1.5	0.5	5
HGR 1a-Air	120	2	0	0
HGR 1a-3	120	1.75	0.25	2.5
HGR 1a-4	120	0	2	20
HGR 2a-1	70	1	1	10
HGR 2a-2	70	1.5	0.5	5
HGR 2a-Air	140	2	0	0
HGR 2a-3	140	1.75	0.25	2.5
HGR 2a-4	140	0	2	20
HGR 3a-1	70	0	2	20
HGR 3a-2	70	0	2 (sub)##	20
HGR 3a-3	140	0	2	20
HGR 3a-Air	194	2	0	0
HGR 3a-4	194	0	2	20

Assumes HGR of simulant is zero and that nominal 0.5 parts per million H_2 in air purge is negligible.
subsurface gas purge

2.4 Test Apparatus

A three dimensional representation of the HGRMA is shown in Figure 2-1.

Figure 2-1 HGRMA Apparatus

The detailed design drawings associated with the HGRMA are listed below and copies are provided in Appendix H.

- Hydrogen Gas Rate Measurement Apparatus (HGRMA) Vessel Installation Turntable Subassembly, R-R2-A-00028 Rev 0[14]
- Hydrogen Gas Rate Measurement Apparatus (HGRMA) TECA TLC-900 Liquid Chiller/ Controller Wiring Diagram, E-EV-A-00082 Rev 0[15]
- Hydrogen Generation Rate Measurement Apparatus: Main Assembly, R-R1-A-00092, Rev. 0[16].
- Hydrogen Generation Rate Measurement Apparatus: Vessel Assembly and Details, R-R1-A-00081, Rev. 0[17].
- Hydrogen Generation Rate Measurement Apparatus: Condenser, Funnel, Manometer, and Bubbler Details, R-R4-A-00112, Rev. 0[18].
- Hydrogen Generation Rate Measurement Apparatus: Details Sheet 1, R-R4-A-00114, Rev. 0[19].
- Hydrogen Generation Rate Measurement Apparatus: Details Sheet 2, R-R4-A-00123, Rev. 0[20].
- Hydrogen Generation Rate Measurement Apparatus: Details Sheet 3, R-R4-A-00126, Rev. 0[21].
- Hydrogen Generation Rate Measurement Apparatus: Process Flow Diagram, J-J-A-00007, Rev. 0[22].

2.5 Test Procedure

The testing consisted of loading a sample into the vessel and then performing a number of tests with varying temperature and hydrogen concentrations in the purge gas, then draining and rinsing the vessel.

Procedure ITS-0207[23] was used during the testing with additional details for each step provided in Appendix I. R&D Directions were documented in an electronic laboratory notebook[24] which also includes the run sheets.

2.6 Technical Review Process

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

While the testing was designed to test the performance of each part of the HGRMA, special focus was applied during the testing to evaluate items that could not be assessed during the POC[25,10] or mockup[11] testing previously documented. These items were the mixing and draining of simulants with solids, measurement of HGR with simulants in the vessel, and performance of the system with a tracer gas. A description of the method used by the gas chromatograph and example chromatograms is shown in Appendix A. Instructions for GC calibration and operation are shown in Appendix E.

3.1 HGR1a Testing

This testing was performed with a simulant of the LAW feed to the process. This simulant contained trace amounts of undissolved solids and was Newtonian with a viscosity of 6 centipoise at 25 degrees Celsius.

3.1.1 HGR1a Loading and Draining the Vessel

The LAW simulant was loaded into the vessel on 11/10/2015 and remained in the vessel during the different flow conditions until it was drained on 11/23/2015. No etching or other issues were noted with the glassware at the conclusion of the test. Mixing was held at 300 revolutions per minute during all test runs with the LAW simulant. During the initial drain, 95% of the material added was collected. Subsequent rinses did not recover any additional material; therefore, 5% of the material added was not recovered. Some condensation of material was noted on the vessel lid, this condensate was likely the reason for the discrepancy in weight of the material collected versus the amount added.

3.1.2 HGR1a Hydrogen Measurements

The test conditions and hydrogen results are shown in

Table 3-1. Hydrogen concentrations in the table were calculated from taking the average of hydrogen readings from the gas chromatography data after steady state conditions were determined for the offgas data. Steady-state conditions were determined for each test based on a review of the data and involved selecting data once the hydrogen readings had reached a steady value for 30 minutes. The difference between the expected value and the measured value was typically less than 20%, but higher differences (~30%) were noted at low concentrations. For a 100 milliliter sample and a purge flow of 2 milliliters per minute, a sample that emits hydrogen at the action limit for the LAW feed (3.7E-07 gram-moles hydrogen per liter-hour at 120 degrees Fahrenheit) would result in a hydrogen concentration of 7 parts per million [1] (Refer to Appendix D, for details on conversion of gas chromatography data to the reporting requirement). The error/uncertainty noted during the testing (~30%) was small enough that a detection limit of 2 parts per million would be able to protect the action limit for LAW. The data from the testing is shown graphically in Appendix A.

Table 3-1. HGR1a Hydrogen Results

Test ID	Date Performed	Test Temperature (Fahrenheit)	Expected Hydrogen Concentration at Steady State (parts per million)	Measured Hydrogen Concentration at Steady State (parts per million)	Delta (%)	Relative Standard Deviation (%)
HGR1a-1	11/10/2015	70	10	8.3	-17.5	4.3
HGR1a-2	11/11/2015	70	5	3.4	-32	5.7
HGR1a-Air	11/12/2015	120	0	<2	0	na
HGR1a-3	11/16/2015	120	2.5	2.0	-20	11.7
HGR1a-4	11/20/2015	120	20	22.6	12.9	3.3

3.2 HGR2a Testing

This testing was performed with the LAW High Bound melter feed simulant prepared during the waste qualification glass fabrication testing. The sample ID of the feed tested was MFP-2a-31. This material was non-Newtonian with a yield stress of 6.4 Pascals and a Plastic Viscosity of 43 centipoise at 25 degrees Celsius and contained 59 weight percent undissolved solids.

3.2.1 HGR2a Loading and Draining the Vessel

The MFP2a-31 sample was loaded into the vessel on 11/24/2015 and was drained from the vessel on 12/7/2015. Mixing speed was held at 450 revolutions per minute during the HGR2a testing. When the sample was drained from the vessel, 94% of the initial sample was drained. The initial water rinse increased the recovery of the sample to 98%. Subsequent acid and water rinses removed some additional material, but some solids were still noted around the vessel lid, in the drain plug, and on the mixer shaft. The vessel was removed from the holder and cleaned prior to the next set of tests.

3.2.2 HGR2a Hydrogen Measurement

The test conditions and steady-state hydrogen measurements are shown in

Table 3-2. Hydrogen concentrations in the table were calculated from taking the average of hydrogen readings from the gas chromatography data after steady state conditions were determined for the offgas data. The difference between the expected value and the measured value was typically less than 20%. Relative standard deviation was higher for the lower concentrations. The maximum HGR for the LAW melter feed is slightly above the limit for the LAW feed; therefore, the error/uncertainty was small enough that a detection limit of 2 parts per million would be sufficient for measurement of LAW melter feed samples . The data from the testing is shown graphically in Appendix A.

Table 3-2. HGR2a Hydrogen Results

Test ID	Date Performed	Test Temperature (Fahrenheit)	Expected Hydrogen Concentration at Steady State (parts per million)	Measured Hydrogen Concentration at Steady State (parts per million)	Delta (%)	Relative Standard Deviation (%)
HGR2a-1	11/24/2015	70	10	10.7	6.9	6.8
HGR2a-2	12/2/2015	70	5	6.1	21.9	6.2
HGR2a-Air	12/3/2015	140	0	<2	-	23.5
HGR2a-3	12/3/2015	140	2.5	2.9	16.2	14.8
HGR2a-4	12/4/2015	140	20	17.3	-13.4	2.4

3.3 HGR3a Testing

This testing was performed with the HLW High Bound melter feed simulant prepared during the waste qualification glass fabrication testing. The sample ID of the feed tested was MFP4a-31. This material was non-Newtonian with a yield stress of 33.3 Pascals and a Plastic Viscosity of 68.3 centipoise at 25 degrees Celsius and contained 54 weight percent undissolved solids.

3.3.1 HGR3a Loading and Draining the Vessel

The MFP4a-31 sample was loaded into the vessel on 12/8/2015 and was drained from the vessel on 12/15/2015. Mixing speed was held at 600 revolutions per minute during the HGR3a testing. The sample did not drain completely from the vessel when the drain valve was opened. The vessel was removed from the holder and cleaned prior to the next set of tests.

3.3.2 HGR3a Hydrogen Measurements

The test conditions and steady-state hydrogen measurements are shown in

Table 3-3. Hydrogen concentrations in the table were calculated from taking the average of hydrogen readings from the gas chromatography data after steady state conditions were determined. The difference between the expected value and the measured value was typically less than 20%. Relative standard deviation was higher for the lower concentrations. The maximum HGR for the HLW melter feed is well above the limit for the LAW feed, therefore the testing was performed at the upper end of the current calibration range. Note that HGR3a-2 used a subsurface feed line to determine the impact of hydrogen being emitted below the slurry surface. Selected tests, such as HGR3a-2, were stopped when hydrogen readings appeared to be at steady state but closer examination of the data reveals that the hydrogen values were still increasing slowly and the test could have benefitted from a longer run time.

Issues were noted in achieving the 194 degrees Fahrenheit target temperature during the testing. Insulation was used to wrap the vessel to achieve the desired setpoint. This issue is further discussed in Section 3.7 General Observations and Lessons Learned.

The data from the testing is shown graphically in Appendix A.

Table 3-3. HGR3a Hydrogen Results

Test ID	Date Performed	Test Temperature (Fahrenheit)	Expected Hydrogen Concentration at Steady State (parts per million)	Measured Hydrogen Concentration at Steady State (parts per million)	Delta (%)	Relative Standard Deviation (%)
HGR3a-1	12/8/2015	70	20	19	-4.9	2.9
HGR3a-Air	12/9/2015	194	0	2.4	-	11.7
HGR3a-3	12/9/2015	140	20	18.9	-5.5	2.6
HGR3a-4	12/10/2015	194	20	17.1	-14.3	3.1
HGR3a-2	12/15/2015	70	20	17.9	-10.7	4.6

3.4 HGR3b Testing with Tracer Gas

This testing was performed with the HLW High Bound melter feed simulant prepared during the waste qualification glass fabrication testing. The sample ID of the feed tested was MFP4b-31. The rheological properties of this sample were not measured, since it can be assumed that this material was the nearly the same as the MFP4a-31 sample which had a yield stress of 33.3 Pascals and a Plastic Viscosity of 68.3 centipoise at 25 degrees Celsius and contained 54 weight percent undissolved solids. The MFP4b-31 sample was prepared in an identical manner to the MFPa-31a sample: the same amount of SRNL prepared HLW-HB simulant was mixed with the same amount of HLW-HB Glass Forming Chemicals (GFCs).

3.4.1 HGR3b Loading and Unloading the Vessel

The sample was loaded on 3/1/2016 and was drained later that day. Mixing was held at 900 revolutions per minute during the testing, but splatter onto the lid was noted when the temperature was increased for the HGR measurement, as shown in Figure 3-1. A reduction in mixing speed as the sample temperature increased could have prevented the splatter.

The sample was cooled to 70 degrees Fahrenheit prior to draining. The sample drained very slowly from the vessel and only 74% of the sample was recovered during the initial draining and a large amount of material was left coating all interior surfaces. The first water rinse was 167 grams to fill the entire vessel. After this rinse, approximately 99% of the material added had been recovered. Subsequent acid and water rinses removed most of the remaining material, but solids residues were noted on the vessel and mixing shaft as shown in Figure 3-2. The rinses did not remove the splatter from the lid. The vessel was removed and cleaned with additional rinses and by wiping with a chem-wipe. The lid was cleaned in place with a water spray and a chem-wipe.

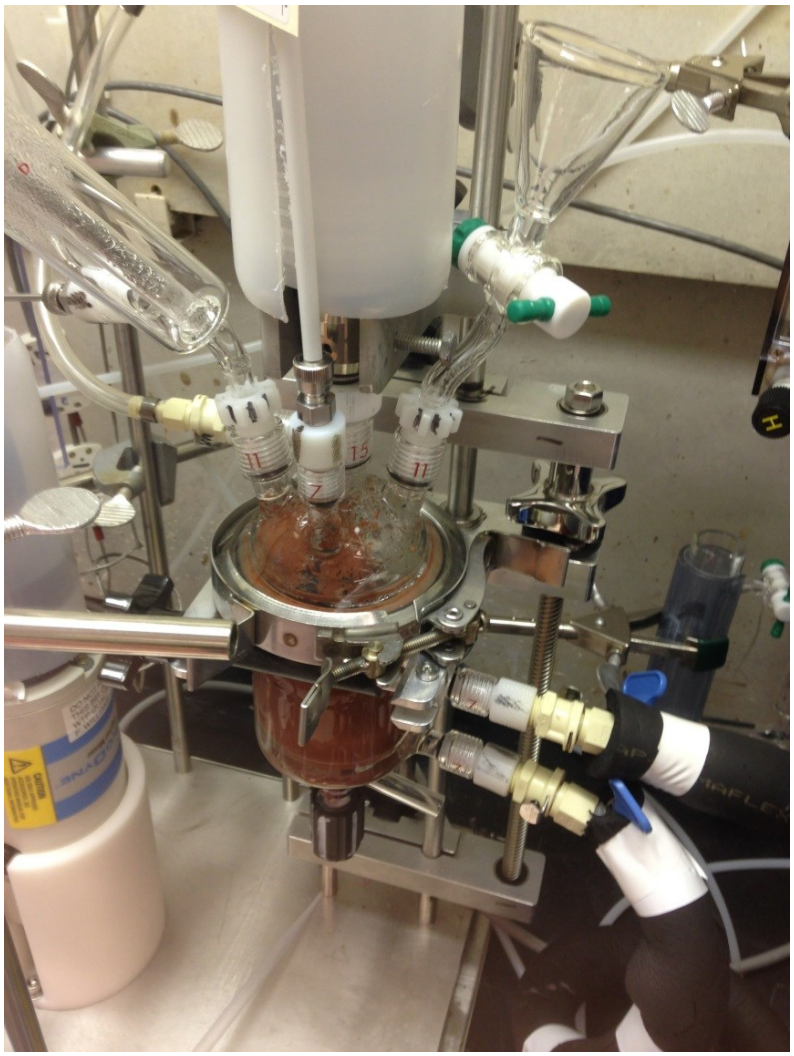


Figure 3-1. Splatter During HGR3b-1

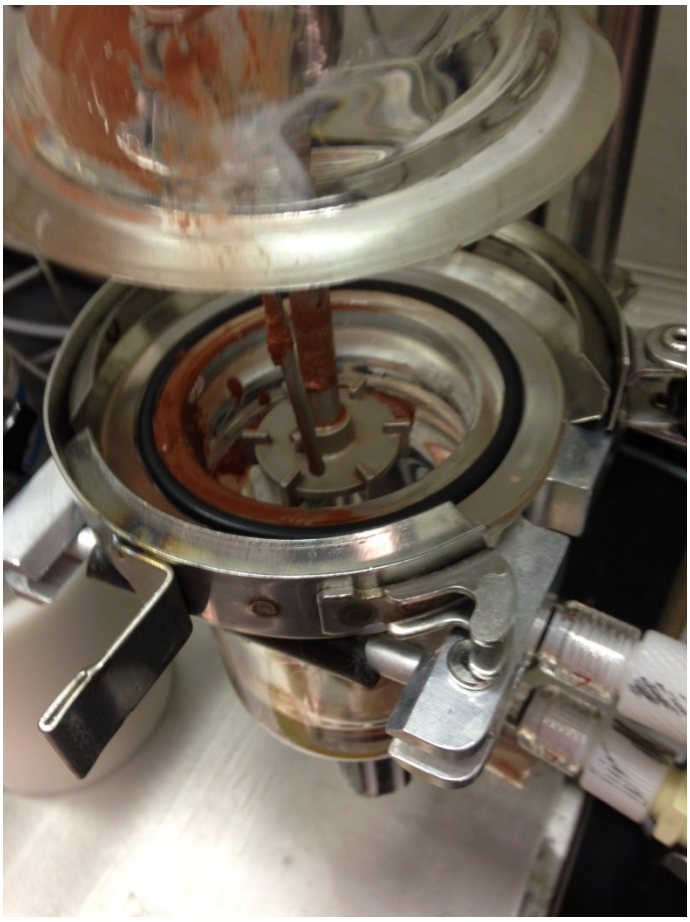


Figure 3-2. Residual Solids after Draining and Rinsing

3.4.2 HGR3b Hydrogen and Krypton Measurements

Once krypton was selected as the tracer gas, a test was performed with the HLW-HB melter feed simulant to evaluate the use of the tracer gas. The concentration of krypton was 12.5 weight percent during the test to allow the 0-5 milliliters per minute flow controllers to have enough flow to accurately blend the 100% krypton gas with the 20 parts per million purge gas as well as to bound any expected interferences of the krypton with the gas measurements. As shown in Table 3-4, the hydrogen measurement was not impacted by the presence of the tracer gas. The krypton measurement during the test is shown in

Table 3-5. Excellent agreement was noted between the expected concentration and the measured value.

Table 3-4. Hydrogen Results from HGR3b-3

Test ID	Date Performed	Test Temperature (Fahrenheit)	Expected Hydrogen Concentration at Steady State (parts per million)	Measured Hydrogen Concentration at Steady State (parts per million)	Delta (%)	Relative Standard Deviation (%)
HGR3b-3	3/1/16	140	17.5	16.5	-6.0	3.7

Table 3-5. Krypton Results from HGR3b-3

Test ID	Date Performed	Test Temperature (Fahrenheit)	Expected Krypton Concentration at Steady State (weight percent	Measured Krypton Concentration at Steady State (weight percent)	Delta (%)	Relative Standard Deviation (%)
HGR3b-3	3/1/16	140	12.5	12.1	-3.4	3.7

3.5 Hydrogen Measurement Results Summary

3.5.1 HGRMA Vessel CSTR Model

Although the HGRMA system is not a reaction tank, for the purposes of modelling it was evaluated as a CSTR. The model used the initial conditions, purge flow rates, vapor space volume, and hydrogen concentrations in the purge gas to predict the hydrogen concentration in the system at any given point in time. Starting at a hydrogen concentration in the vessel at zero, the concentration at time equal to one minute was calculated based on a mass balance assuming that the vent gas was equal to the purge air supplied and the concentration of the vent gas was equal to the concentration of the vessel. This calculation was repeated at a time equal to two minutes assuming the calculated concentration at one minute is used as the initial condition. The equations used in the model are shown below:

Hydrogen Concentration at Time T = (Hydrogen in Vessel at T-1 - Hydrogen lost in vent gas + Hydrogen added in Purge)/Vessel Headspace Volume
Where:

Hydrogen in the vessel at T-1 is equal to the hydrogen concentration at T-1 times the headspace volume

Hydrogen lost in vent gas is equal to the purge rate times the hydrogen concentration at T-1

And

Hydrogen in the air purge is equal to the air purge rate times the hydrogen concentration in the purge gas

Note that the model used does not have terms for hydrogen evolved from the simulant as this term is assumed to be zero during the simulant testing.

The model assumed a 225 milliliter vapor space in the HGRMA vessel, an initial concentration of 0 parts per million hydrogen in the vessel and a purge with hydrogen concentration of 20 parts per million, shown graphically in Figure 3-3. After development of the model, the vapor space of the HGRMA vessel and lid was estimated by filling the assembled vessel and lid with water, using the difference between the empty and full weight. After subtracting the 100 milliliter sample volume, the measured vapor space volume was 220 milliliters. A small amount of air remained in the lid after filling; the amount was estimated to be 5 to 10 milliliters visually. Therefore, the determination of 225 milliliters for the vapor space volume in the model was appropriate.

If plug flow is assumed in the vent line, the concentration measured at the gas chromatograph will be offset in time from the concentration in the HGRMA vessel by the time required for the gas exiting the vessel to flow through the tubing and reach the gas chromatograph. The time delay between the vessel and the gas chromatograph was estimated to be 50 minutes from the test data, but this delay could change depending on the size and length of the tubing between the HGRMA vessel and the gas chromatograph.

The model indicated that the time to reach steady state conditions (readings are within 2.5% of the projected concentration) was approximately 460 minutes. The run time estimate of 510 minutes (8.5 hours) to verify that steady state will be reached was significantly higher than the estimates of 300 minutes (5 hours) from the Proof of Concept tests [25] [10].

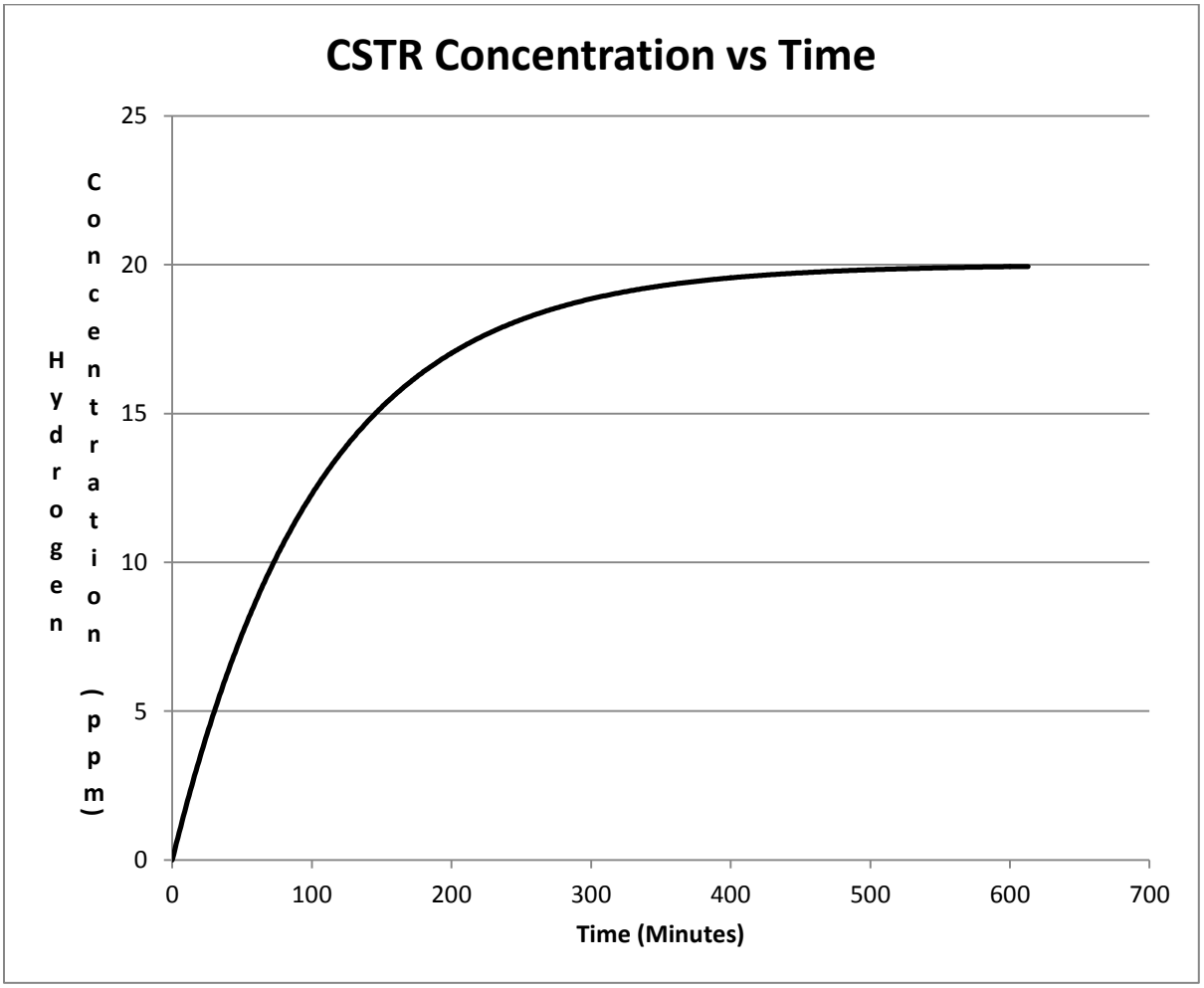


Figure 3-3. CSTR Model of HGRMA Vessel

3.5.2 Comparison of data to CSTR model

Adjustments were made to the data to account for the offset between the CSTR concentration and the gas chromatograph measurement time as well as to normalize the data to a concentration of 20 parts per million. The normalization to 20 parts per million involved multiplying each measurement during the test by 20 divided by the steady state value for hydrogen determined from the run as shown in sections 3.1 through 3.4. The time stamp for the data was then adjusted to place the data onto the model curve to account for the delay between the gas exiting the vessel and the gas chromatograph measurement. The time adjustment required was typically 35 to 50 minutes. A typical comparison of the data to the model is shown in Figure 3-4 with the remaining graphs shown in Appendix B. During the test with the krypton tracer, the tracer gas concentration was also fit to the model as shown in Figure 3-5. During tests where no hydrogen is generated, the concentration of the krypton tracer would allow a determination of when steady-state conditions are reached in the vessel.

The data indicated excellent agreement between the data and the model and showed that the gas chromatograph was performing acceptably for both the krypton tracer and hydrogen measurements. The krypton data indicated a slightly longer lag time between the calculated HGRMA vessel concentration and the measured data at the gas chromatograph than the hydrogen measurements from this run. However, the delay in the krypton equaled the 50 minutes typically noted while the hydrogen values only required a 40-minute delay to fit on the curve.

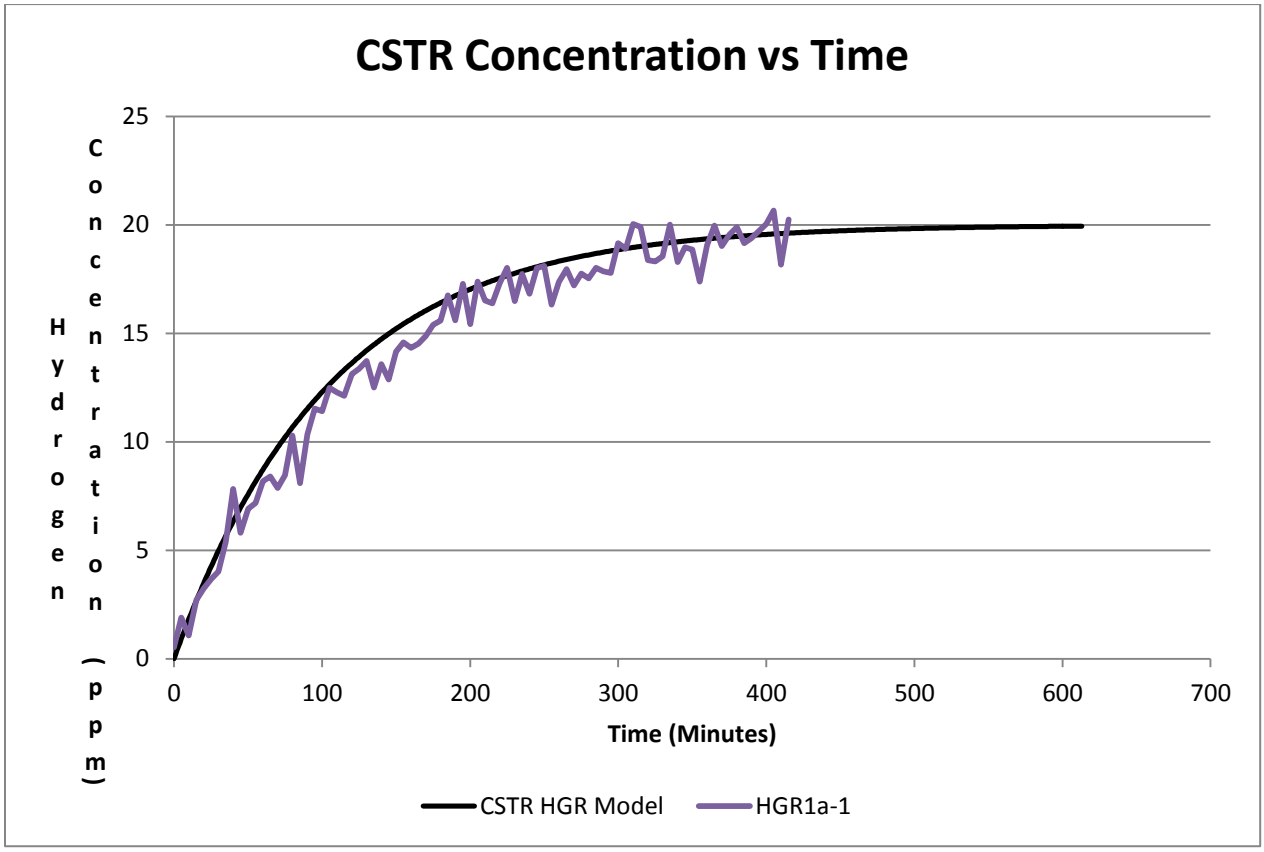


Figure 3-4. Comparison of Data to HGRMA CSTR Model

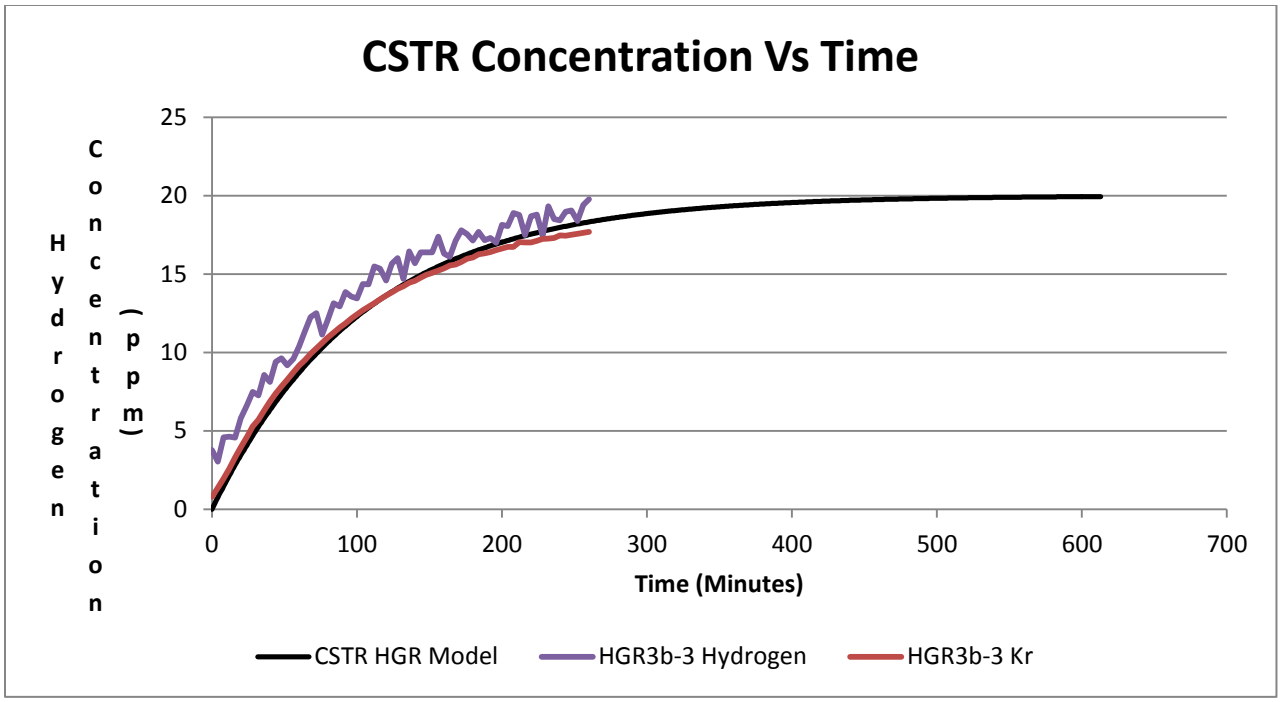


Figure 3-5. Comparison of Data from HGR3b with CSTR Model

3.5.3 Air Only Tests

Each simulant was tested with air purge only at the maximum temperature to be tested with each simulant. No hydrogen was noted in the test with LAW simulant at 120 degrees Fahrenheit, but hydrogen at 1.5 parts per million was noted in the LAW High Bound Melter Feed simulant at 140 degrees Fahrenheit. The HLW High Bound simulant at 194 degrees Fahrenheit resulted in hydrogen concentrations of 2.4 parts per million. The test data from runs with these simulants was not corrected for these values as most runs were not performed at the higher temperatures. In addition, higher than expected hydrogen measurements were generally not noted during the testing. Results of the air tests are shown graphically in Appendix A.

3.5.4 Detection Limits

The detection limit was determined to be 1 part per million during the testing, but linearity of the gas chromatograph response became an issue below 2 parts per million. Therefore, the detection limit for reporting results was 2 parts per million.

3.5.5 Impact of Concentration on Measurement Error and Variability

3.5.5.1 Error/Uncertainty

The measured error/uncertainty in the hydrogen concentration was generally less than 10% for runs at 20 parts per million. At this concentration, the air purge was off and no error was attributed to the gas flow rates. At lower concentrations, air was blended with the calibration gas using the MKS mass flow controllers. Any errors in flow rates would result in errors in the hydrogen concentration. Therefore, the errors in measured hydrogen concentration for these runs include the error in blending the calibration gas with air. The errors were higher at the lower concentrations, with some errors as high as 32% of the targeted value.

3.5.5.2 Variability

The HGR determination was performed using the average of hydrogen measurements once steady state conditions were achieved. The variability in the steady state data was determined by calculating the relative standard deviation of each data set used to determine the steady state hydrogen concentration. As the concentration of hydrogen decreased, the variability in the data also increased, as shown in Figure 3-6. At hydrogen concentrations of 2.5 parts per million, the relative standard deviation was 10-16% of the reading while at 20 parts per million the relative standard deviation was only 3-4% of the reading.

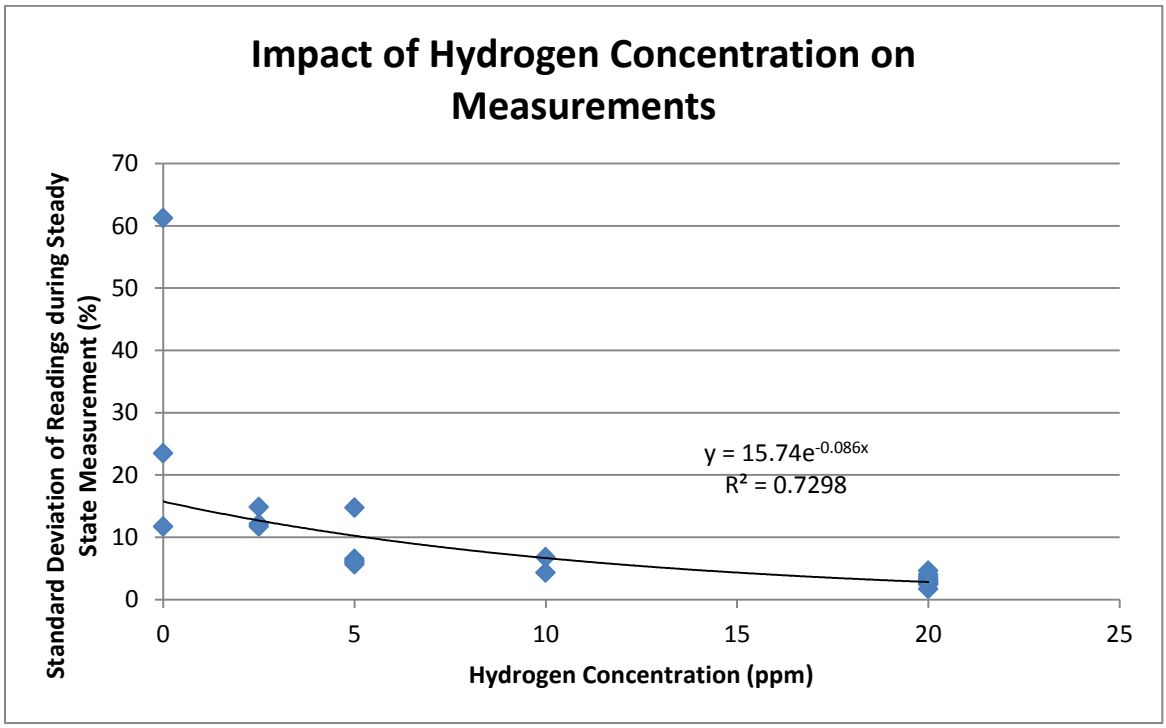


Figure 3-6. Impact of Concentration on Relative Standard Deviation of Steady State Data

3.5.6 Impact of Changes in Environmental Conditions Around the Apparatus

No impacts were noted during the testing with changing conditions in the hood or laboratory environment that impacted operation of the apparatus.

3.6 Other Testing

3.6.1 Water Runs

Water runs were conducted on 12/16/2015 to evaluate the performance of the HGRMA with water. Test conditions and results from these tests are shown in Table 3-6.

Table 3-6. Water Run Results

Test ID	Test Temperature (Fahrenheit)	Expected Hydrogen Concentration at Steady State (parts per million)	Measured Hydrogen Concentration at Steady State (parts per million)	Delta (%)	Relative Standard Deviation (%)

HGR-Water-70-20	70	20	17.1	-14.4	4.0
HGR-Water-140-20	140	20	18.7	-6.7	2.8
HGR-Water-70-5	70	5	5.4	7.0	5.9
HGR-Water-140-5	140	5	5.0	0.5	14.7

3.6.2 Initial Tracer Gas Testing

Helium is typically used as the tracer gas during SRNL testing for the Defense Waste Processing Facility (DWPF) chemical process cell. The tracer allows verification that the gas chromatograph is not sampling room air due to a leak as well as providing a means to calculate total offgas flow. During the HGRMA testing, helium was rejected as a tracer gas due to the proximity of the helium peak to hydrogen and the need for more sensitivity in the hydrogen measurement than for DWPF testing. After consultation with the gas chromatograph vendor, xenon, and krypton were selected as candidates for the tracer gas.

Initial testing indicated the xenon had significant overlap with the nitrogen peak while krypton had a peak sufficiently beyond nitrogen peak to avoid interference with any of the gases being measured. Tests with the calibration gases blended with the tracer gases confirmed that hydrogen measurements were not impacted by the tracer gases and that quantification of the krypton was feasible. The peak area of the krypton peaks noted during the testing indicates that operation with tracer gas concentrations of 0.5 weight percent would be sufficient during waste feed qualification testing.

3.6.3 Load Test

The support stand and lifting bail were load tested to ensure that a 100 pound load on the stand was acceptable per Savannah River Nuclear Solutions (SRNS) procedures. This load test was performed to verify the adequacy of the design. The lifting protocols of the laboratory chosen to perform waste feed qualification activities will need to be reviewed, and any additional testing to meet those protocols will need to be performed by the qualification lab. The HGRMA stand weight was 33 pounds, while the final weight of the assembled HGRMA apparatus was 55 pounds. Therefore, the weight of the equipment installed on the stand was 22 pounds, well below the working limit of 80 pounds applied to the stand after the load test at 100 pounds.

3.7 General Observations and Lessons Learned

3.7.1 Flow Controller Failure

The air purge flow controller failed during the testing with no flow indicated despite the valve being in the full open position and approximately 60 milliliters per minute of air flowing through the controller. This failure was noted by the high differential pressure noted at the manometer. The cause of this flow controller failure was determined by the manufacturer to be a failed circuit board.

These flow controllers are used frequently for control of gas flow rates in a wide variety of testing at ACTL and in the Shielded Cells and none have exhibited this type of failure in the past. While these flow controllers have been very reliable, some failures have been noted in past use. The past failures typically were the result of having the flow path plugged or the valve becoming stuck, but the flow indication has typically indicated when these failures occurred. It is important to understand the expected pressure differential, typical flow rates, etc. and to investigate when off-normal readings are obtained.

3.7.2 Water Bath Issues

The water bath was noted to leak during the initial testing. No failed fittings were noted during the investigation of the leak, but one of the fittings was found to be very loose. The fitting was tightened and no further leaks from the bath were noted. In addition, one of the fittings connecting the water hoses to

the HGRMA began leaking during the HGR3b testing. The fitting was found to be deformed and cracked, as shown in Figure 3-7. The fitting was replaced and the testing resumed.

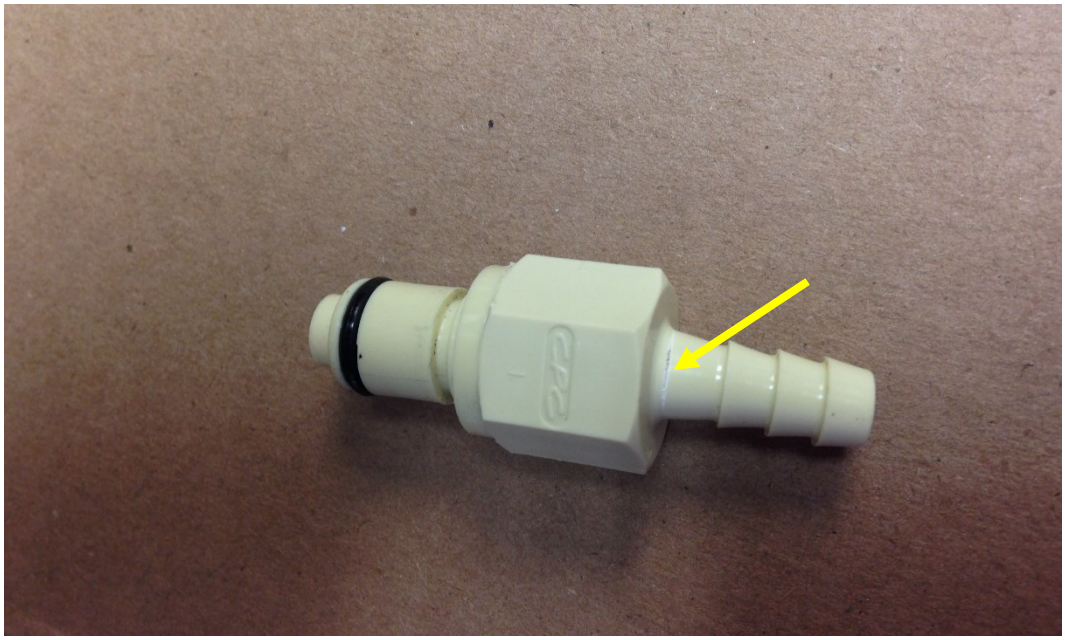


Figure 3-7. Crack in Failed Water Fitting

During HGR3a-4, issues were noted reaching the 194 degrees Fahrenheit temperature target for the vessel. The vessel was insulated to reach the desired temperature; this insulation was not required during the Proof of Concept testing[10]. During the POC testing, it was noted that insulating the vessel had only a marginal impact on the vessel temperature as the heat loss from the tubing between the water bath and the vessel was much higher than the heat loss from the vessel. The tubing was significantly shortened and insulated during the POC testing to achieve the setpoint. The shorter, insulated tubing was in place during the verification testing with simulants.

It was noted during the testing that tubing in the internals of the water bath were not insulated, and heat loss from the uninsulated tubing could account for significant heat loss between the bath and the vessel. Therefore, insulating the vessel may be avoided by insulating the tubing inside the water bath. Insulation of the vessel is not preferred, but a neoprene wrap is typically used on the 1 liter Shielded Cells DWPF chemical process cell apparatus. This wrap is installed and removed remotely, a similar wrap could be fashioned and used on the HGRMA if issues reaching temperature are noted in the waste feed qualification testing.

A test was performed to verify that a neoprene insulation wrap similar to those used on the one liter DWPF apparatus would be suitable for the HGRMA. The results of this testing indicated that the water bath could hold the vessel at 194 plus or minus 2 degrees Fahrenheit using the neoprene wrap. Details of this testing are discussed in Appendix J. Proper insulation of the vessel was important to reach the upper temperature ranges required for testing.

3.7.2.1 Erosive Wear

The glass forming chemicals added to the test simulants are abrasive and a slight amount of wear was noted on the impeller and vessel, as shown in Figure 3-8 and Figure 3-9. Note that lighting for the vessel photograph used was adjusted to allow the wear to be seen more easily and exaggerates the impact of the

wear noted. Although the amount of wear on the vessel and impeller was sufficient to allow it to be noticed, it was not deemed sufficient to warrant removing the vessel or impeller from service. This type of wear was expected when mixing abrasive mixtures like the melter feed slurries. Inspection of the vessel and other glassware should be performed prior to each use. The LAW simulant test (HGR-1a) had five HGR runs performed that were conducted over a 10 day period. The LAW-HB test (HGR-2a) also had five runs conducted over a 10 day period. The HLW-HB test (HGR-3a) had five runs conducted over a 7 day period and an additional test (HGR-3b) performed in one day. When initial testing and other evaluations are included, the HGR vessel contained caustic simulant for approximately 35 days and was used to perform over 20 HGR measurements at various temperatures.

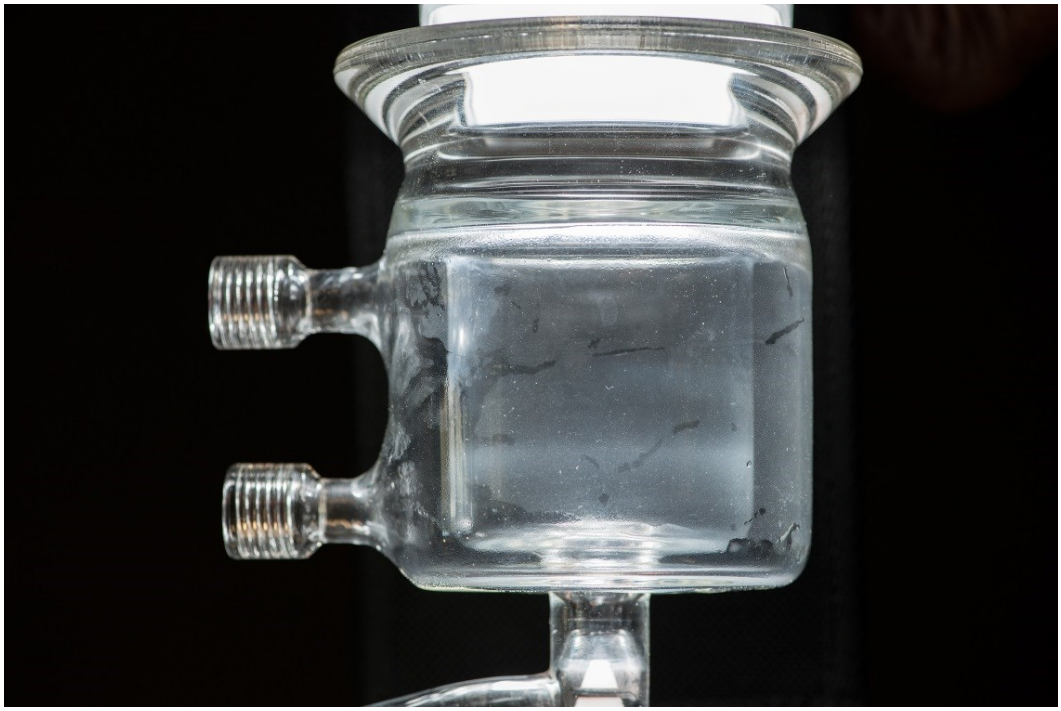


Figure 3-8. Erosive Wear on Vessel

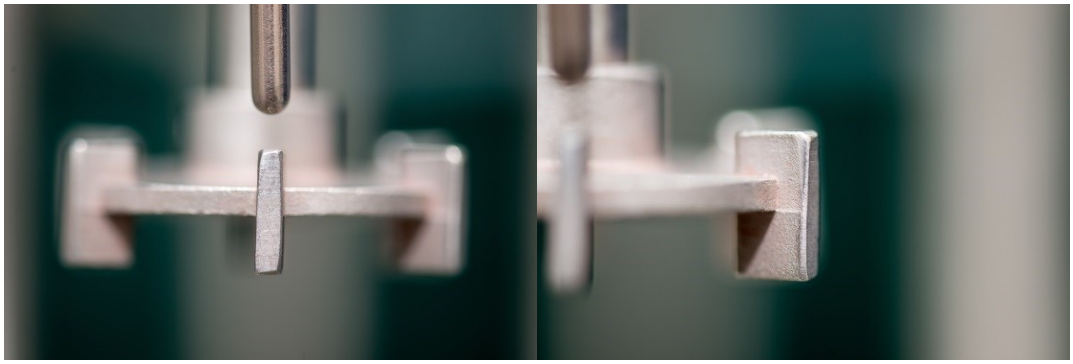


Figure 3-9. Erosive Wear on Impeller

3.7.3 Calibration Gas Consumption

The 20 parts per million calibration gas was used as the purge gas for most tests. A standard gas cylinder was procured for the testing and was delivered with approximately 2,000 pounds per square inch gage internal pressure. Despite the extensive use, the internal pressure had only decreased to 1,900 pounds per

square inch gauge during the testing due to the low flow rates used during the tests. As stated above, this gas was used for over 20 HGR measurements during final verification testing with simulants as well as calibrations before and after each test. In addition, a significant number of proof of concept runs, gas chromatography method development, and other tests (such as air only testing with required calibrations before and after use).

3.7.3.1 Negative Pressure during Cooling

It was noted that rapidly cooling the vessel would result in a negative pressure that led to backflow from the vent into the vessel even with the purge (2 milliliters per minute) on. This issue was resolved by opening the addition funnel valve during cooling.

3.7.4 HGRMA System Performance

3.7.4.1 Temperature Control

The temperature control during the runs was within 2 degrees Fahrenheit of the setpoint for all runs, although one or two points were noted outside this range. The temperature profile during HGR3a-4 is shown below in Figure 3-10 and shows the offset between the bath and vessel.

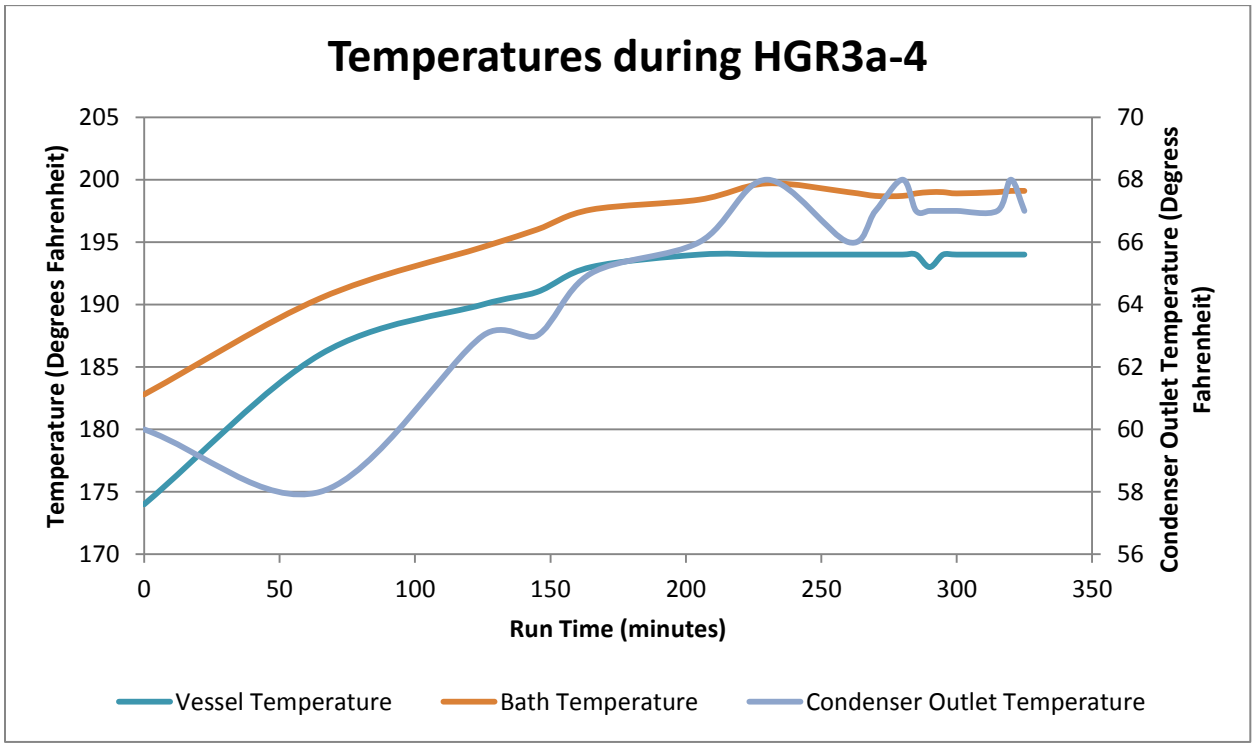


Figure 3-10. Temperature Profile during HGR3a-4

The RTD readout was set to read in Fahrenheit and did not include a decimal place. It was not possible during the testing to hold the temperature to +/- 1 degree Fahrenheit, therefore the temperature was held +/- 2 degrees Fahrenheit. Converted to Celsius, the 2 degrees Fahrenheit is 1.1 degrees Celsius and is not within the +/- 1 degree Celsius specified. The temperature specification was based on the literature review finding the HGR is a function of temperature and that a two degrees Celsius change in temperature

could result in a 20 percent change in the HGR while a 0.5 degree Celsius change could result in a 5 percent change. Therefore, maintaining tight control of the temperature during an HGR measurement is desirable and the temperature control specification was based on tightest control believed to be achievable.

Since higher temperatures increase the HGR, it is recommended that the temperature during the waste feed qualification measurements be set such that the specified temperature is the minimum allowed during the measurement. Measurement of the HGR at 122 degrees Fahrenheit when a value is needed for 120 degrees Fahrenheit would allow the system to maintain the required setpoint at all times.

3.7.4.2 Condenser/Vortex Chiller

No condensate or other indication of evaporative loss from the vessel was noted during the testing. The temperature of the cooling air stream exiting the condenser was recorded during the testing and was typically 50 to 65 degrees Fahrenheit. As shown above in Figure 3-10, the temperature of the condenser outlet approached 70 degrees Fahrenheit during tests at 194 degrees Fahrenheit. Condensation was frequently noted on the outside of the condenser where the cooling air enters the condenser, while condensation was typically not noted on the condenser closer to the vessel. A pressure of 50 -70 pounds per square inch gauge was required to maintain an air flow of 50-60 liters per minute through the vortex chiller.

3.7.4.3 Vessel Air Leak Checks

No issues were noted during the testing with meeting the leak check requirements prior to performing a test run.

3.7.4.4 Addition Funnel

The addition of rinses through the addition funnel had to be added slowly since the HGRMA vessel is sealed with very low inlet and outlet flowrates. No other available port to open the system and allow pressure equalization was available on the HGRMA. Material would slowly drain into the vessel, but air had to escape past the rinse solution in the funnel. This issue was not deemed severe enough to warrant pursuing a change in funnel design.

3.7.4.5 Manometer

The filters on the vent gas line can cause back pressure if they plug. No issues with back-pressure were noted during the testing except when the failure of the air flow controller led to 60 milliliters per minute of air purge on the vessel. The back-pressure on the vent gas filter at that flow rate was higher than the manometer was designed to accommodate. Typically, 1-3 inches of water column of backpressure was noted during the tests, but some runs had back pressures as high as 4 inches of water column. The back-pressure noted during the testing for normal operations was well within the range that allows the manometer to keep the vessel sealed and the vent gas routed to the gas chromatography measurement system.

3.7.4.6 Mixing System

The agitator mixing speed was checked before and after each test. No significant difference was noted between the mixing speed indicated by the agitator controller and the speed measured by the tachometer. The ability to mix each simulant was demonstrated, as described above.

3.7.4.7 Other Systems

No issues were noted with the stand, vessel lift, containment pan, or other HGRMA components during the testing.

3.8 Other Requirements

The development of the HGRMA included preparation of a number of documents in addition to the design, fabrication and testing of the HGRMA as described above. The documents include assembly instructions, instructions for operation of the apparatus, instructions for operation of the gas chromatograph, a RAMI review, and a packaging plan for shipment. The RAMI review is shown in Section 6.0, the other required documents are discussed below.

3.8.1 Work Instructions for HGRMA Operation

Work instructions were developed for operation of the HGRMA based on the POC testing, mockup testing, and final verification testing with simulants. The final work instructions are included as Appendix C.

3.8.2 HGRMA Assembly and Tear Down Instructions

The remote assembly and disassembly was demonstrated during mockup testing. The instructions used for that demonstration were revised to incorporate lessons learned from that testing and are included as Appendix D.

3.8.3 Work Instructions for GC Operation

The calibration of the gas chromatograph is required for operation of the HGRMA, but the work instructions were developed separately from the HGRMA operation for this task. These instructions are included as Appendix E.

3.8.4 Packaging Plan

A packaging plan was required to package the HGRMA for shipment to the Hanford site once the testing was completed as well as to allow storage of the apparatus until needed. This packaging plan is documented in Appendix G.

3.8.5 Design Drawings

Design drawings were prepared to document the design of the HGRMA and allow for fabrication of spares. These design drawings are separately approved documents, but the current versions of the documents are shown in Appendix H.

4.0 Testing Results Versus Design Requirements

The design requirements for the HGRMA were originally documented in the conceptual design document[6]. Those design requirement are re-summarized in Table 1-2.

The contents of this section examine each of these twenty-three (23) design requirements including discussions on each during the various phases of testing, and whether or not the design criteria was met during POC testing[10,25], Mockup Testing[11] and/or Verification Testing.

Overall System

Design Requirement 1 *The apparatus will be designed to perform HGR measurements with sufficient accuracy to allow a determination of whether the sample meets the HGR action limits. The most restrictive action limit is for the Low-Activity Waste receipt samples at 3.7E-7 gram mole of hydrogen per liter per hour at 120 degrees Fahrenheit (48.9 degrees Celsius).*

Results from POC Testing: The apparatus met the requirement outlined in the conceptual design.

Results from Mockup Testing: The apparatus met the requirement outlined in the conceptual design.

Results from Verification Testing with Simulants: The HGRMA demonstrated the ability to perform hydrogen gas measurements to verify that the HGR from a sample is less than the action limit for the LAW receipt samples.

In order to meet the overall design requirement for the system, design requirements for the HGR apparatus and individual systems or components of the HGR have been specified. The following requirements apply to the overall apparatus.

Design Requirement 2 *A leak check will be performed on the system prior to testing to ensure that 90% of the purge flow entering the vessel is measured in the vent gas.*

Results from POC Testing: The overall system was assembled and leak checked to the levels specified. Leak check testing was performed with 5 standard cubic centimeters per minute of nitrogen. An exit flow rate of 4.8 standard cubic centimeters per minute indicated that the system is capable of maintaining an adequate seal (exit flow at least 90% of inlet flow) at low flow rates. Testing with the HGR apparatus indicated that 2 standard cubic centimeters per minute of purge flow resulted in offgas flow at the flow verification bubbler, also indicating a good seal. The leak check is conducted as a mass balance where the pressure in the system is not measured.

Results from Mockup Testing: All leak checks performed passed.

Results from Verification Testing with Simulants: All leak checks passed.

Design Requirement 3 *A tracer will be used in the purge gas to ensure that the analyzer is measuring gas from the vessel and not from room air leaking into the vent gas line.*

Results from POC Testing: A tracer gas was not used. However, the apparatus was demonstrated to be leak tight.

Results from Mockup Testing: A tracer gas was not used. However, the apparatus was demonstrated to be leak tight.

Results from Verification Testing with Simulants: Use of krypton (Kr) as a tracer gas was successfully demonstrated. A recommendation was made to use cylinders of air spiked with 0.5% Kr as the purge gas during waste feed qualification testing.

Design Requirement 4 *The sample will be held within one degree Celsius of the specified temperature during the HGR measurement.*

Results from POC Testing: During the initial testing, the system was configured with a ThermoElectric Cooling America Corporation (TECA) TLC-700HC chiller. The initial testing revealed that the TECA-700HC chiller was not sufficient to maintain an elevated temperature set-point. Subsequently, a TECA TLC-900 series chiller was selected and installed. The TECA TLC-900 chiller was able to hold the vessel within 1 degree Celsius of the set point with set points up to 90 degrees Celsius.

Results from Mockup Testing: The TECA TLC-900 chiller was able to hold the vessel within 1 degree Celsius of the set point with set points up to 90 degrees Celsius.

Results from Verification Testing with Simulants: The HGRMA was able to achieve the targeted temperature and maintain temperature to within 2 degrees Fahrenheit. However, insulation was required during testing at 90 degrees Celsius. See discussion in Section 3.7.4.1 for resolution of the difference between maintaining sample to within two degrees Fahrenheit (1.1 degree Celsius) of temperature setpoint versus 1 degree Celsius.

Design Requirement 5 *The sample will be well mixed during the HGR measurement.*

Results from POC Testing: Mixing was demonstrated with water and several different xanthan gum solutions. The mixing demonstrations were visual in nature and are documented in POC test summary. These mixing tests included samples with a yield stress of up to 26 Pascals.

Results from Mockup Testing: Mixing was only demonstrated with water.

Results from Verification Testing with Simulants: The HGRMA was able to mix all of the simulants used during the testing. These tests included simulants with a yield stress as high as 33 Pascals.

Design Requirement 6 *The individual components on the apparatus will be remoted to allow removal and replacement of degraded or failed components.*

Results from POC Testing: Remoting of the apparatus was complete with the exception of the TECA chiller and the agitator. The agitator is the same design that has been remoted in the past at SRNL, while the TECA chiller is similar to the model that also has been remoted at SRNL.

Results from Mockup Testing: The remoting of these components was completed when the apparatus was moved into the mockup area of the SRNL shielded cells facility.

Results from Verification Testing with Simulants: No testing of remotability was performed.

Design Requirement 7 *As part of the final design process, all subcomponents of the apparatus that may contain software will be checked against the applicability of NQA-1-2000 Part II Subpart 2.7. Additionally, if a data collection software system is used, it will also be reviewed to determine if NQA-1-2000 Part II Subpart 2.7 is applicable during final design.*

Results from POC Testing: The only software planned for use is the EZ-IQ (version 3.3.2 SP2) control software for the gas chromatograph as well as the Diablo EZReporter (version 3.0.1.2) monitoring software used in conjunction with the EZ-IQ software. This software was and will be functionally checked each time the gas chromatograph calibration and calibration checks are performed in accordance with SRS 1Q QAP 2-7, QA Program Requirements for Analytical Systems as required, therefore no additional controls are needed to comply with NQA-1a-2009 Part II Subpart 2.7 as the software is considered exempt. SRNS implements NQA-1a-2009 which meets or exceeds NQA-1-2000 requirements.

Results from Mockup Testing: No additional software controls were identified.

Results from Verification Testing with Simulants: No additional software controls were identified.

Apparatus Support System

Design Requirement 8 *The apparatus support will allow the HGR measurement apparatus to be safely installed in the shielded cells facility and will provide support for equipment during operation.*

Results from POC Testing: The design of the apparatus stand was completed and the HGR apparatus was assembled on the stand.

Results from Mockup Testing: The apparatus was installed and operated with water in a simulated remote environment during mockup testing.

Results from Verification Testing with Simulants: A load test was performed and a working load limit of 80 pounds was established. This load limit applies to items installed on the stand, not the weight of the stand itself. The equipment installed on the stand weighs approximately 22 pounds.

Design Requirement 9 *The containment pan will be capable of holding the entire contents of the vessel.*

Results from POC Testing: This design requirement was not tested as part of this testing. However, the containment pan was designed and fabricated. The details of the containment pan are included in detail drawing R-R4-A-00126[21]. The approximate volume of the containment pan is 2,700 milliliters, which far exceeds the sample volume and the overall vessel volume (including headspace) of ~325 milliliters as noted in Section 3.5.1.

Results from Mockup Testing: This design requirement was not tested as part of this testing. No changes were made to the containment pan as part of this testing, the design of the containment pan was sufficient.

Results from Verification Testing with Simulants: No changes were made to the containment pan during the testing.

Lid and Vessel

Design Requirement 10 *The vessel must be able to contain 100 milliliters of sample with some vapor space at the top, allow complete draining of sample, provide a seal adequate to meet leak check requirements, and allow visual inspection of cleanliness and mixing.*

Results from POC Testing: As stated in the literature review[1], 100 milliliters was chosen as the sample size to allow for adequate mixing and to produce enough hydrogen during the testing to allow the gas chromatography measurement to meet the LAW action limit. The vessel and lid has been designed and fabricated. The details of the vessel and lid are contained in R-R1-A-00081[17]. The vessel has been shown to adequately contain 100 milliliters of sample without excessive splashing into vent gas line during mixing with xanthan gum and water. The vapor space was reduced as much as possible while using glass components. Reducing the size of the vapor space further would either require the vessel to be fabricated from metal or the water jacket to be reduced in size so that the entire sample is not covered by the water jacket. After consultation with the SRNL glass shop, a glass lid using Ace-Thred connections was used in place of a stainless steel lid with Swagelok® Ultra-Torr fittings. For the RTD, the Ace-Thred connector was adapted to a Swagelok® Ultra-Torr fitting. The overall volume of the vessel and lid is approximately 300 milliliters. Leak checks were successfully performed on the apparatus. The vessel and lid are manufactured of glass, allowing for visual inspection.

Results from Mockup Testing: The apparatus was successfully loaded with 100 milliliters of water during mockup testing and leaks checks were successfully performed after a complete disassembly/reassembly of the

apparatus in mockup. The vessel was successfully drained during the mockup testing.

Results from Verification Testing with Simulants: The vessel successfully contained 100 milliliters of each simulant testing. The total volume of the vessel and lid was measured to be 320 milliliters, therefore the vapor space volume with a 100 milliliters sample is 220 milliliters.

No issue with leak checks were noted during testing.

Although the simulant samples were successfully drained from the vessel during the testing, it was noted that the initial drain did not recover 100% of the samples (Refer to section 3.1.1, 3.2.1, and 3.3.1). As a result, rinsing and cleaning strategies were developed to remove the remaining materials from the vessel.

The glass vessel was easily inspected after draining and rinsing for cleanliness.

Mixing System

Design Requirement 11 *The mixing system must be able to maintain a small surface vortex without excessive splatter for samples with Bingham Plastic yield stresses of 0 to 30 Pascals while maintaining a seal sufficient to meet leak check requirements.*

Results from POC Testing: The mixing system was tested with two weight percent xanthan gum slurries, with a nominal yield stress of 26 Pascals during this testing. The vessel was leak checked with the mixer coupling in place and with the mixer running. The seal is a magnetic coupler and leak rate was not impacted by the mixer speed, torque, or particulate. The conceptual design[6] used off-center mixing, but this mounting positioned limited the size of the impeller that could be used. A larger impeller was shown to be needed during initial testing. The agitator was moved to a center mounted position and a two inch commercially available impeller was used for the testing.

Results from Mockup Testing: No issues were noted with installation or tear down of the mixing system during mockup testing. The mockup testing included connection of the wiring to the agitator controller located outside the mockup cell.

Results from Verification Testing with Simulants: The HGRMA was able to mix all of the simulants used during the testing. These tests included simulants with a yield stress as high as 33 Pascals. Some splatter was noted during one of the tests due to the sample thinning as the vessel was heated to temperature since the agitator speed was not reduced to compensate.

Air Purge System

Design Requirement 12 *The air purge system must deliver a purge gas with a tracer to the vessel with sufficient accuracy to allow the vent gas measurement to be sufficiently accurate to ensure that the HGR action limits are not exceeded.*

Results from POC Testing: The mass flow controllers from MKS Instruments have been procured and installed on the apparatus as shown in the assembly completion documentation[26]. The flow controllers are ranged from 0 to 5 standard cubic centimeters per minute to provide as high accuracy as possible in the flow ranges specified for testing (approximately 2 standard cubic centimeters per minute). Inclusion of a tracer gas was not performed as part of the POC testing.

Results from Mockup Testing: No issues were noted with remote installation of the air purge lines from the MKS flow controllers (outside the cell) to the vessel.

Results from Verification Testing with Simulants: The purge air system was able to accurately meter gas into the vessel during the testing. However, one of the flow controllers failed during the testing and was replaced with a unit on hand at SRNL.

Design Requirement 13 *The air purge system must include pressure relief to prevent over-pressurization of the HGR apparatus.*

Results from POC Testing: A manometer has been designed, fabricated, and installed on the system. The details of the manometer are contained in drawing R-R4-A-00112[18]. The manometer provides pressure indication of the vessel, as well as pressure relief in the event of over pressurization. The use of a manometer as a pressure relief device is common practice for apparatus designed, installed, and used within the SRNL Shielded Cells. The manometer allows for approximately 12 inches of water column of pressure prior to relieving; typical back pressure of 1-2 inches of water column is expected during typical operation with back pressure of 4-5 inches of water column during higher temperature tests.

Results from Mockup Testing: The manometer was easily installed and maintained during the remotabilty testing in mockup.

Results from Verification Testing with Simulants: The back-pressure during the verification testing was approximately 2 inches of water column during most tests, but reached 3.6 inches of water column during selected testing. When the flow controller failed and supplied 60 milliliters per minute instead of the 2 milliliters per minute planned, the manometer relieved as expected.

Design Requirement 14 *The air purge system must be sufficiently leak tight to meet leak check requirements.*

Results from POC Testing: The overall HGR apparatus was demonstrated to be leak-tight during POC testing.

Results from Mockup Testing: Leaks checks were successfully performed after a complete disassembly/reassembly of the apparatus in mockup.

Results from Verification Testing with Simulants: All leak checks performed during the verification testing passed, typically with 95-100% on the inlet flow measured on the bubbler.

Temperature Control

Design Requirement 15 *The temperature control system must maintain the vessel contents at specified temperature to within one degree Celsius during the HGR measurement. Specified temperatures will be between 25 and 90 degrees Celsius.*

Results from POC Testing: A TECA chiller (TLC-900) was selected for use on the HGR apparatus. This recirculator has a 650 watt heater.

An estimate for the heat loss was performed assuming that the vessel was at 90 degrees Celsius, ambient temperature in the cells was 10 degrees Celsius, and the heat transfer area was equal to the vessel area plus the water circulator tubing (10 feet of 5/8 inch outer diameter tubing). This estimate resulted in a heat loss of approximately 400 watts. An item of note from the estimate is that heat loss from the tubing was significant and that insulating the tubing would significantly reduce the heat loss.

The chiller was demonstrated to hold the vessel at the specified temperatures during POC testing. The vessel was held at 45, 60, and 90 degrees Celsius during the testing.

A 3 wire RTD was procured for testing outside the cells. The RTD has a diameter of 1/8 inch to allow smaller fittings to the utilized.

Results from Mockup Testing: The RTD was installed and removed easily during mockup testing. The mockup testing included connection of the RTD wiring to the RTD readout outside the mockup cell.

Results from Verification Testing with Simulants: The HGRMA was able to achieve the targeted temperature and maintain temperature to within 2 degrees Fahrenheit during the testing. However, insulation was required during testing at 90 degrees Celsius and a leak was noted from a loose fitting in the chiller during the testing. See discussion in Section 3.7.4.1 for resolution of the difference between maintaining sample to within two degrees Fahrenheit (1.1 degree Celsius) of temperature setpoint versus 1 degree Celsius.

Sample Gas System

Design Requirement 16 *The sample gas system must be sufficiently leak tight to meet leak check requirements.*

Results from POC Testing: The overall HGR apparatus was demonstrated to be leak-tight during this testing.

Results from Mockup Testing: Leaks checks were successfully performed after a complete disassembly/reassembly of the apparatus in mockup.

Results from Verification Testing with Simulants: All leak checks performed during the verification testing passed, typically with 95-100% on the inlet flow measured on the bubbler.

Design Requirement 17 *The sample gas system must sufficiently treat the vent gas to prevent damage to the gas analyzer.*

Results from POC Testing: The vent gas condenser, demister pad, and vent gas filter were installed and did not show excessive back pressure at the flowrates expected during testing. The temperature of the condenser cooling air vent (less than 5 degrees Celsius) and the amount of condensation on the outside of the condenser indicate the cooling system is able to condense water from the vent gas.

Results from Mockup Testing: The system was easily installed and removed during mockup testing.

Results from Verification Testing with Simulants: No indications of significant sample loss were noted during the testing. The temperature of the cooling air vented from the condenser did not exceed 70 degrees Fahrenheit during the testing. Typically, the condenser outlet temperature was 55-60 degrees Fahrenheit. These temperatures are adequate to prevent excessive moisture from reaching the gas chromatograph. No operational issues with the gas chromatograph were noted during the testing, a further indication that the amount of water reaching the gas analyzer was acceptable.

Design Requirement 18 *The sample gas system must prevent sample loss through evaporation.*

Results from POC Testing: Long duration tests were not performed during this testing to demonstrate that the system will prevent sample loss from evaporation, but the temperature of the condenser cooling air vent (less than 5 degrees Celsius) and the amount of condensation on the outside of the condenser indicate the cooling system will be able to condense water from the vent gas.

Results from Mockup Testing: The system was easily installed and removed during mockup testing.

Results from Verification Testing with Simulants: No indications of significant sample loss were noted during the testing. The temperature of the cooling air vented from the condenser did not exceed 70 degrees Fahrenheit during the testing. Typically, the condenser outlet temperature was 55-60 degrees Fahrenheit. These temperatures are adequate to prevent excessive moisture from reaching the gas chromatograph. No operational issues with the gas chromatograph were noted during the testing, a further indication that the amount of water reaching the gas analyzer was acceptable.

Design Requirement 19 *The sample gas system must sufficiently treat the vent gas to allow the gas to exit the shielded cells and be monitored by an analyzer in a fume hood.*

Results from POC Testing: The design incorporates a sintered metal filter to mitigate entrained solids from passing through the offgas line. Incorporation of similar filters has allowed vent gas from similar experiments to exit the SRNL shielded cells. Final evaluation is pending including determination of the required treatment for the facility where the system will be installed.

Results from Mockup Testing: The system was easily installed and removed during mockup testing.

Results from Verification Testing with Simulants: No changes were made to the filter. Once a laboratory a is chosen to perform the waste feed qualification testing, a review is needed to determine if the practices used by SRNL are adequate to meet the requirements needed to allow the vent gas to leave the containment of the shielded cells.

Design Requirement 20 *The gas analyzer must be sufficiently accurate to perform HGR measurements to allow determination of whether the sample meets the specified action limits.*

Results from POC Testing: The INFICON 3000 micro gas chromatograph with a large volume injector was capable of detecting hydrogen at 1 part per million during POC testing. The Low-Activity Waste (LAW) action limit would result in 7 parts per million hydrogen.

Results from Mockup Testing: The system was easily connected to the vessel and removed during mockup testing. The accuracy of the GC was not tested during mockup testing.

Results from Verification Testing with Simulants: No issues were noted with gas chromatograph performance. Errors and variability were higher at lower hydrogen concentrations, but the errors were small enough that the HGRMA can meet the lower action for the LAW waste feed. Instrument uncertainty is included in the error assessment; therefore, instrument uncertainty was acceptable for use in the HGR measurement.

Design Requirement 21 *The gas analyzer must be able to be calibrated in place.*

Results from POC Testing: This design requirement was not evaluated. However, the existing gas chromatograph in the SRNL Shielded Cells has been calibrated in place throughout its service life. The gas chromatograph selected for use with this newly designed HGRMA is identical in operation to the existing gas chromatograph except for the volume injected.

Results from Mockup Testing: Calibrations were not performed as part of the mockup testing, however SRNL has a nearly identical (only the injector is different) gas chromatograph installed at the SRNL Shielded Cells Facility. This gas chromatograph is frequently calibrated in place during use.

Results from Verification Testing with Simulants: The gas chromatograph was calibrated in place during the verification testing using 3-way valves to switch the source of the gas entering the gas chromatograph from the HGRMA to the calibration gas bottles.

Design Requirement 22 *The sample gas system will include provisions for flow measurements during leak checks to allow the overall sealing of the HGR measurement apparatus to be assessed.*

Results from POC Testing: The flow verification bubbler has been fabricated and tested. The details of the bubbler are contained in R-R4_A-00112[18]. The bubbler provides visual indication of gas flow during the testing as well as allowing a leak check to be performed.

Results from Mockup Testing: The system was easily installed and removed during mockup testing. Leaks checks were successfully performed in the mockup facility after remote assembly of the apparatus.

Results from Verification Testing with Simulants: All leak checks performed during the verification testing passed, typically with 95-100% on the inlet flow measured on the bubbler.

Operation of the Apparatus

Design Requirement 23 *The measurement of HGR using the HGR apparatus will be performed using the steps / general actions identified in Section 11.0 of the conceptual design document[6].*

Results from POC Testing: This design requirement was not evaluated as part of this testing.

Results from Mockup Testing: This design requirement was not evaluated as part of this testing.

Results from Verification Testing with Simulants: The HGRMA demonstrated the ability to perform hydrogen gas measurements to verify that the HGR from a sample is less than the action limit for the LAW receipt samples.

The twenty-three (23) design requirements and the testing phase where the requirement was demonstrated is summarized in Table 4-1.

Table 4-1 HGRMA Design Requirements and Testing Summary

Requirement	Applies to	Design Requirement Description	Proof-of-Concept Testing (1) [25]	Proof-of-Concept Testing (2) [10]	Mock-Up Testing [11]	Final Verification Testing	Design Requirement Met
1.	*Overall System*	*The apparatus will be designed to perform HGR measurements with sufficient accuracy to allow a determination of whether the sample meets the HGR action limits. The most restrictive action limit is for the Low-Activity Waste receipt samples at 3.7E-7 gram mole of hydrogen per liter per hour at 120 degrees Fahrenheit (48.9 degrees Celsius).*	✓	✓	☒	✓	Yes
2.		*A leak check will be performed on the system prior to testing to ensure that 90% of the purge flow entering the vessel is measured in the vent gas.*	✓	✓	✓	✓	Yes
3.		*A tracer will be used in the purge gas to ensure that the analyzer is measuring gas from the vessel and not from room air leaking into the vent gas line.*	☒	☒	☒	✓	Yes
4.		*The sample will be held within one degree Celsius of the specified temperature during the HGR measurement.*	✓	✓	✓	The sample was held to within 1.1 degree Celsius.	Yes See discussion in Section 3.7.4.1 for resolution of the difference between maintaining sample to within two degrees Fahrenheit (1.1 degree Celsius) of temperature setpoint versus 1 degree Celsius.
5.		*The sample will be well mixed during the HGR measurement.*	✓*	☒	☒	✓	Yes
6.		*The individual components on the apparatus will be remoted to allow removal and replacement of degraded or failed components.*	☒	☒	✓	✓	Yes
7.		*As part of the final design process, all subcomponents of the apparatus that may contain software will be checked against the applicability of NQA-1-2000 Part II Subpart 2.7. Additionally, if a data collection software system is used, it will also be reviewed to determine if NQA-1-2000 Part II Subpart 2.7 is applicable during final design.*	✓	✓	✓	✓	Yes
8.	*Apparatus Support*	*The apparatus support will allow the HGR measurement apparatus to be safely installed in the shielded cells facility and will provide support for equipment during operation.*	☒	☒	✓	✓	Yes
9.		*The containment pan will be capable of holding the entire contents of the vessel.*	✓	☒	☒	✓	Yes
10.	*Head and Vessel*	*The vessel must be able to contain 100 milliliters of sample with some vapor space at the top, allow complete draining of sample, provide a seal adequate to meet leak check requirements, and allow visual inspection of cleanliness and mixing.*	✓	☒	☒	100% of the simulant samples did not drain completely.	Yes Rinsing/Cleaning strategies were developed to remove the residue.
11.	*Mixing System*	*The mixing system must be able to maintain a small surface vortex without excessive splatter for samples with Bingham Plastic yield stresses of 0 to 30 Pascals while maintaining a seal sufficient to meet leak check requirements.*	✓*	☒	☒	✓	Yes
12.	*Air Purge System*	*The air purge system must deliver a purge gas with a tracer to the vessel with sufficient accuracy to allow the vent gas measurement to be sufficiently accurate to ensure that the HGR action limits are not exceeded.*	☒	☒	☒	✓	Yes

Requirement	Applies to	Design Requirement Description	Proof-of-Concept Testing (1) [25]	Proof-of-Concept Testing (2) [10]	Mock-Up Testing [11]	Final Verification Testing	Design Requirement Met
13.		*The air purge system must include pressure relief to prevent over pressurization of the HGR apparatus*	✓	☒	☒	✓	Yes
14.		*The air purge system must be sufficiently leak tight to meet leak check requirements.*	✓	☒	✓	✓	Yes
15.	*Temperature Control*	*The temperature control system must maintain the vessel contents at specified temperature to within one degree Celsius during the HGR measurement. Specified temperatures will be between 25 and 65 degrees Celsius.*	✓	✓	✓	✓	Yes
16.	*Sample Gas System*	*The sample gas system must be sufficiently leak tight to meet leak check requirements.*	✓	✓	✓	✓	Yes
17.		*The sample gas system must sufficiently treat the vent gas to prevent damage to the gas analyzer.*	✓	✓	✓	✓	Yes
18.		*The sample gas system must prevent sample loss through evaporation.*	✓	☒	☒	✓	Yes
19.		*The sample gas system must sufficiently treat the vent gas to allow the gas to exit the shielded cells and be monitored by an analyzer in a fume hood.*	✓	✓	✓	✓	Yes
20.		*The gas analyzer must be sufficiently accurate to perform HGR measurements to allow determination of whether the sample meets the specified action limits.*	✓	✓	☒	✓	Yes
21.		*The gas analyzer must be able to be calibrated in place.*	☒	☒	✓	✓	Yes
22.		*The sample gas system will include provisions for flow measurements during leak checks to allow the overall sealing of the HGR measurement apparatus to be assessed.*	✓	✓	✓	✓	Yes
23.	*Operation of the Apparatus*	*The measurement of HGR using the HGR apparatus will be performed using the steps / general actions identified in Section 11.0 of the conceptual design document [SRNL-RP-2013-0397].*	☒	☒	☒	✓	Yes

** Mixing was demonstrated with a 26 Pascal xanthan gum solution.*
✓ = requirement demonstrated
☒ = requirement not demonstrated

5.0 TTQAP Documentation

The TTQAP listed seven tests/evaluations to be performed on the HGRMA. This section describes the documentation of the completion of each test/evaluation required by the TTQAP.

Table 5-1 HGRMA Technical Task Documentation Summary

Task Plan Section	Activity Description	Activity Detail	Documentation Summary
4.1	Design of HGRMA	*The design of the HGR measurement apparatus will be performed based on the criteria developed during the literature review [1](. Design drawings will be prepared and submitted for review in two phases. Phase I will be considered Conceptual Design and will consist of the recommended equipment and a line diagram showing connections. Definitive design drawings (Phase II) will include final dimensions and a 3D rendering of the final assembly plan, list of equipment, and a Piping and Instrumentation drawing. These documents will be reviewed and accepted by WTP prior to fabrication of the apparatus. The design will take into consideration that the unit must be able to be assembled, operated and disassembled remotely in a laboratory hot cell, and will be shipped to the WTP at the completion of testing.*	A Conceptual Design Package was completed and accepted by WTP[6]. Design packages were reviewed and accepted by WTP at the 30% [7,27], 60%[8,28], and Final Design stages.[9] An additional review was performed at 90% design complete,[29] but a separate design package was not completed for that review; all comments from the 90% review were included in the Final Design Package.
4.2	Fabrication of the HGRMA	*This task will commence once the design documents are accepted by the WTP. Long lead time components may be procured prior to approval of the design documentation with concurrence from WTP. Fabrication will include procurement or fabrication of required components, assembly of components in the HGR measurement apparatus, and system checkout to include leak checks and water runs.* *The analytical instrument will likely utilize software supplied by the vendor. Controls applicable to Measurement and Sensing Equipment will be applied to the testing at SRNL, which specify a check with standards prior to and after each use to ensure the entire system (including software) is operating as intended.*	Procurement of components for the HGRMA was completed[30,31] and the unit assembled at ACTL[26,32]. Information for each component supplied by the vendor (manuals, etc.) was forwarded to WTP[33].
4.3	HGRMA POC Testing	*Proof of concept testing will be performed by assessing whether the analytical instrument for the measurement of generated gases can be calibrated for hydrogen in the range required. The calibration test will be performed with and without the mixing vessel attached to the system. If the desired detection limits are not achievable using air as the cover gas, an inert gas purge will be evaluated since an inert cover gas allows a greater sensitivity for hydrogen and other gas species during analysis. Purified bottled air supplies will be used to eliminate the background hydrogen if elevated hydrogen concentrations are noted in the instrument air utilized during the testing.* *In addition to the hydrogen measurements, the system will be evaluated to ensure that it is sufficiently leak tight to perform the HGR measurement with the required precision and accuracy, and that it can adequately mix process slurries. The leak check will be performed using a flow test. A known purge will be introduced into the system and a flow meter will be used to measure the flow out of the vent line. The out flow must match the flow into the system within 10%. As described in the literature review, the leak rate from a continuous system will not impact accuracy of the test results (Stone, 2012).* *Mixing will be evaluated with Xanthan gum solutions (or other physical simulant) and with chemical simulants of known rheological properties. Adequate mixing will be determined by visual inspection to ensure that no stagnant areas exist along the walls or on the bottom of the mixing vessel and that a slight vortex is noted at the surface.* *SRNL will work with WTP representatives to appropriately specify the simulant used during proof of concept testing.*	Proof of concept testing on the HGRMA[25] and individual components[10] was performed during the design and fabrication process.
4.4	Mockup and Final Design of HGRMA	*The HGR measurement apparatus will be tested for remote operation by installing the system in the SRNL Shielded Cells mockup facility. The operability of each component with manipulators will be demonstrated. Any required changes for remote operation will be made and documented. The rig will be assembled and disassembled in mock up. Leak checks will be performed after assembly in mockup to ensure that the apparatus can be remotely sealed.*	The assembled HGRMA apparatus was transported to the SRNL Shielded Cells and tested in the Mock Up Facility to demonstrate the remotability of the apparatus[11].
4.5	Verification	*Tests of the final design of the HGR measurement apparatus will be performed using simulants. A measured volume of the*	This report documents the completion of this testing.

Task Plan Section	Activity Description	Activity Detail	Documentation Summary
	Testing with Simulants	*selected simulant will be added to the vessel and mixed while the appropriate calibration gas is passed through the vessel. Initially, the calibration gas will be passed through the vapor space. Subsequent tests will introduce the calibration gas subsurface to validate that the mixing system prevents holdup of hydrogen in the test fluid.* *The offgas analysis will be compared to the calibration gas known values to determine the accuracy and repeatability of the method over the required measurement range of the system.* *The verification testing will be performed using work instructions developed for final operation of the system and will include setup, operation, tear-down, and a cleaning of the system.* *This testing will also include verification that the cleaning methods developed for the system adequately clean the vessel without damaging the system, and can occur in a laboratory hot cell. Cleaning is expected to consist of chemical cleaning with fifty weight percent nitric acid followed by a deionized water rinse. A visual inspection of the system will be performed to ensure the components have been cleaned. If deemed necessary, a second chemical cleaning followed by analysis of the cleaning solution may be performed to verify the initial chemical cleaning was adequate.* *SRNL will work with WTP representatives to appropriately specify the simulant used during verification testing.* *Once the integrated tests are completed, the HGR measurement apparatus will be packaged and sent to WTP for use during qualification testing. The packaging will be performed as specified in Appendix B of the scope of work.*	
4.6	Development of Work Instructions for Operating the HGRMA	*The finalized detailed work instructions for setup, operation, tear-down, and cleaning the HGR measurement apparatus will be prepared based on the verification testing and included in the final report.*	A draft version of the setup instructions[34] as well as the operating procedure[23] were developed and tested during mockup[11] and the final verification testing. The final work instructions, setup and tear-down instructions, cleaning, are attachments to this document.
4.7	Data Reporting and Submittals	*The results of the HGR measurement apparatus testing will be documented in a final report containing the following information:* • *Detection limits of the HGR measurement apparatus* • *Measurement uncertainty/precision of the method* • *Description of methods used to evaluate and determine the detection limits and measurement uncertainty* • *Description of system design and operation* • *Impact of changes in environmental conditions around the apparatus* *Design of the system will be documented in system drawings at two stages in the design:* • *Conceptual design* • *Definitive design* *Work instructions for final system operation will be documented and submitted to WTP.*	This document is the final report for the HGRMA and contains the information listed. The conceptual design[6] and definitive design[9] documents were completed and issue. Work instructions for final system operation are included as an attachment.

6.0 RAMI Review

A RAMI review of the HGRMA was performed. This review was based on the RAMI design guide 24590-WTP-GPG-ENG-009 Rev. 2[35]. The support services for the HGRMA were not addressed in this review.

Several terms and the associated definitions related to the RAMI review are provided below.

- Mean Time Between Failures (MTBF) – A statistical value that is meant to be the mean over a long period of time and a large number of units.
- Mean Time Between Maintenance (MTBM) - A value that is the analogue of MTBF. The mean time between identified maintenance tasks can be used as an estimate of this parameter.
- Mean Time to Maintain (MTTM) – It is the arithmetic mean time to perform identified maintenance tasks.
- Mean Time to Repair (MTTR) – is the sum of corrective maintenance times as a specific level of repair, divided by the total number of failures, for an item required at that level, during a particular interval, under stated conditions. This time is all-inclusive and is intended to cover all downtime for completing the repair of replacement activity.

- Mean Time Between Calibrations (MTBC) - Similar to MTBM, but specific to analytical instruments to denote the time period between calibrations
- Mean Time to Calibration (MTTC) – It is the arithmetic mean time to perform identified calibrations.
- Mean Time Between Calibration Failures – Average time estimated between instruments failing to meet specified accuracy during calibrations.

6.1 Reliability

Nearly all of the components specified for use on the HGRMA have been utilized on similar apparatus in the SRNL Shielded Cells or other radioactive service at SRS. The agitator and controller, flexible mixing shaft, water recirculating bath, vortex chiller, gas filters, tube fittings, mass flow meters, manometers, and gas chromatograph have been used and shown reliable for extended service. A Cole-Parmer Servodyne mixer remoted in a similar manner to the mixer on the HGRMA has been in service for over 4 years without issues and the water bath similar to the one selected for the HGRMA has been in service in the cells for significantly longer.

The Parr magnetic seal was successfully used in a recent sludge batch qualification run. The RTDs have not been used in previous studies, but similar RTDs have been successfully used and they are not expected to be rapidly degraded by the shielded cells environment. Failure modes of each piece of equipment were reviewed by SRNL per section 5.0 *RAMI Guidance for Engineering Disciplines*[35]. The main components from the assembly and possible failures for each are shown in Table 6-1. Structural components from the detail sheets were reviewed but deemed to be overdesigned enough to not be included into the table

Table 6-1. Failure Mode Assessment

Component	Failure Mechanism	Drawing Number
Vessel Assembly Reaction Vessel	11 mm glass tubing which attaches to the bottom of the vessel or the ACE-Thread adapters could break off, erosive wear from glass forming material slurries.	R-R1-A-00081-B
Vessel Assembly Head	ACE-Thread adapter fused to the top of the Head could break off. The funnel which attaches into the front of the Vessel assembly adapter would be the easiest to break since it is in the front and not supported.	R-R1-A-00081-C
Vessel Assembly Clamp	Clamp is made from stainless steel and should not have any failures based on the use in the design.	R-R1-A-00081-D
Bubbler Assembly	Stopcocks will wear out from use, seals can degrade with radiation and chemical exposure, discoloration of the glass which limits it use, fracturing of the glass tube adapters.	R-R4-A-00112-A
Condenser Assembly	10mm glass tubing attached to the bottom could break off. O-ring of the ACE fitting could degrade with exposure.	R-R4-A-00112-B
Addition Funnel Assembly	Long length of the funnel could lead to breaking once attached into the Reaction Vessel. Stopcock and O-ring on ACE fitting could degrade from exposure.	R-R4-A-00112-C
Septum Connect	Unsupported in main assembly could lead to the glass breaking at the connections.	R-R4-A-00112-D

Component	Electrical Failure Mechanism	Drawing Number
Agitator Head and Controller	Radiation exposure, chemical exposure, damage wiring cables	Commercial
Water recirculating bath	Radiation exposure, chemical exposure, damage wiring cables	Commercial
Vortex chiller	Radiation exposure, chemical exposure	Commercial
mass flow controller	Contaminants in gas stream shorting heater wire, Contaminants causing control valve to stick	Commercial
gas chromatograph	Radiation exposure, chemical exposure, damage wiring cables	Commercial
RTD	Radiation exposure, chemical exposure, damage wiring cables	Commercial

Other parts listed on the Main Assembly drawing[16] include hoses which could burst from wear or damage from other cell operations. The Swagelok fitting for the hoses sometime have a long lead time, it would be beneficial to have a set of spare fittings available to replace any damaged fittings.

6.2 Availability

The major components of the HGRMA include:

- Servodyne Mixer
- MKS Mass Flow Meters and Controller
- Vortex Chiller and Rotameter
- Reaction Vessel and Condenser RTDs
- TECA Water Recirculator
- Condenser Assembly with Mist Eliminator Mesh
- Micro GC 3000
- Manometer
- Reaction Vessel and Attachments

The HGRMA Servodyne Mixer has not had a failure in over four years of operation in SRNL Shielded Cells[36] resulting in a MTBF of 35,040 hours. As the mixer has been remoted, the MTTR is estimated to be 24 hours to replace the failed module. The mixer has not failed in over four years of operation in SRNL Shielded Cells. During non-radioactive use at ACTL, no failures other than electrical cord failures or issues with a collet that is not on the remoted mixer. Therefore, it is anticipated that the mixer will be run to failure. The MTTM and MTBM are not applicable for the mixer.

The MKS Mass Flow Meters and Controller have been used at SRNL for both non-rad and Shielded Cells testing. Typically, no issues are noted with the flow controllers over many years of operation, but some failures have been noted if solids or water are allowed to get in the gas streams being metered by the flow controllers. A MTBF of 43,800 hours has been set for these components as they are external to the Shielded Cell, but it was noted that one of the flow controllers on the HGRMA failed during simulant testing. This failure is not characteristic of the service typically seen by SRNL for these flow controllers. The failure mode is that the electronics of the MKS flow meters could fail over time in service or solids/liquids in the gas stream cause issue with plugging of the flow or solids causing the control valve to stick in place. MKS provides a two-year warranty on their mass flow meters and they have been demonstrated to be very reliable in a wide range of service in SRNL. The MTTR of a flow meter which involves calibration of a new unit and replacement of the failed unit associated with the HGRMA Shielded Cell, if necessary, is 2 hours. The MTTM is 120 hours which involves a calibration check of the mass flow meter. The MTBM for the mass flow meter is 17,520 hours based on the two-year warranty. It is anticipated that the MKS mass flow meter and controller will be run to failure. The MTBC of the MKS mass flow meter and controller is not applicable as a calibration by the laboratory is recommended before and after use to see if performance has degraded.

No failures have been noted with the vortex chiller and rotameters during Shielded Cells operations, but the longest run time for any of the experiments was 90 days. The failure mode for the vortex chiller and rotameter is that they can become clogged with solids degrading their performance. As the vortex chiller and rotameter are connected with quick connects to the HGRMA, the MTTR is estimated to be 4 hours to replace the failed module. The vortex chiller and rotameter will be run to failure. The MTTM and MTBM are not applicable for the vortex chiller and rotameter except that they will be replaced if performance has degraded.
Based on SRNL Shielded Cells experience, the failure mode for the reaction vessel and condenser RTDs is that they will degrade over time and eventually short circuit, resulting in a MTBF of 43,800 hours. There is no warranty on RTDs in nuclear service within SRNL Shielded Cells. Therefore, SRNL recommends their replacement every two years. The MTTR of an RTD which involves calibration of a new RTD and replacement of the failed unit associated with the HGRMA Shielded Cell, if necessary, is 24 hours. The RTD MTTM is 120 hours which involves a calibration check of the RTD. The MTBM for the RTDs is 17,520 hours based on their replacement every two years. The MTBC of the Omega reaction vessel and condenser RTDs is 17,520 hours as a 48 hour MTTC by the laboratory is recommended before installation in the cells. After two years, new RTDs should be installed on the HGRMA and used to perform a check of the RTDs being removed.

The TECA water recirculator has not had a significant failure in over five years of operation for SRNL resulting in a MTBF of 43,800 hours as the controller has been remoted external to the Shielded Cell to reduce the probability of controller failure. Two failure modes have been postulated in SRNL for the TECA water recirculator and they include pump failure and water line fitting leakage. The fittings can be tightened or replaced, if necessary. The MTTR of a TECA water recirculator is 4 hours for HGRMA components in the Shielded Cells. The MTTM, the MTBM, and the MTBM are not applicable for the TECA water recirculator because it will be run to failure.

The condenser assembly with mist eliminator mesh is not expected to fail as it has no moving components and the MTBF was set to 43,800 hours. The only failure mode postulated for the condenser assembly is that the mesh for eliminating mist could become clogged. Replacement of the entire condenser would be required if the mesh became clogged. The MTTR of the condenser assembly with mist eliminator mesh is 24 hours for HGRMA components in the Shielded Cells. The MTTM, the MTBM, and the MTBM are not applicable for the condenser assembly with mist eliminator because it will be run to failure.

The Micro Gas Chromatograph 3000 failure mode is degradation of performance over time and eventually hydrogen peaks will not be discernable during their operation inside SRNL Shielded Cells resulting in a MTBF of 43,800 hours. The failure method for the gas chromatographs is the electronic components fail and need to be replaced. To improve reliability of the gas chromatography system, the micro gas chromatograph contains two 10 meter Molsieve columns which are fed the sample in parallel. The MTTR of the gas chromatograph associated with the Shielded Cell is 168 hours as it includes time to calibrate the gas chromatograph Molsieve columns. The MTTM the gas chromatograph is 120 hours which involves a calibration check of the gas chromatograph. The MTBM for the gas chromatographs is 8,760 hours based on calibration checks before and after use. The MTBC of the gas chromatograph is 730 hours and a MTTC with a gas chromatograph calibration check gas is 8 hours. The MTBC and time to calibrate generally do not apply as it is recommended the gas chromatograph calibration be checked before and after use.

The 7 millimeter inner-diameter manometer has not had a failure in over three years of operation in SRNL Shielded Cells resulting in a MTBF of 26,280 hours. The normal failure mode for the manometer is that it becomes plugged with solids. The MTTR is estimated to be 4 hours to rod out the solids or replace the U-tube. As the manometer has not failed in over three years of operation in SRNL Shielded Cells, it is anticipated that the manometer will be run to failure. The MTTM and MTBM are not applicable for the manometer except that it will be replaced if performance has degraded.

The glass reaction vessel and attachments such as the addition funnel assembly have not had a failure in over three years of operation in SRNL resulting in a MTBF of 126280 hours. The failure modes for the reaction vessel involves leakage from the O-ring/seals or breaking of the glass attachments as well as erosive wear from the abrasive melter feed slurries. The MTTR of reaction vessel O-rings/ seals which involves tightening seals or their replacement and replacement of broken attachments associated with the HGRMA in Shielded Cell, if necessary, is 4 hours. The MTTM is 2 hours which involves a leak check of the reaction vessel and attachments. The MTBM for the reaction vessel and attachments is not applicable as the leak check will be performed each time the HGRMA is used to perform a test. It is anticipated that the reaction vessel and attachments will be run to failure. The MTBC of the reaction vessel and attachments is not applicable as there is nothing to calibrate.

An availability evaluation of the 222-S Laboratory Equipment has previously been performed [37]. The Availability Summary of 222-S Laboratory Equipment determined that the gas chromatograph availability was 87% and the Master Slave Manipulator (MSM) had an availability of only 72%. This low availability makes these HGRMA required instruments and equipment the least reliable. Experience from operation of the HGRMA in mockup and Shielded Cells should present opportunities to improve availability of the HGRMA.

6.3 Maintainability

The system was designed to allow replacement of a failed component (e.g. a failed agitator motor) and included provisions to allow remote disassembly and reassembly. Similar systems have been assembled remotely at SRNL. The primary concern with assembly in the remote environment is the ability to achieve and demonstrate a leak-tight system. An evaluation of the practicality of disassembly and reassembly in a remote cell was performed as part of the system testing.[9]

The gas chromatograph specified has been operated and maintained for extended periods of time in a fume hood while connected to an apparatus in the Shielded Cells at SRNL. The gas chromatograph was routinely calibrated and "baked out" in place prior to each use and the calibration was checked after each use. An argon purge must be maintained on the gas chromatograph anytime the instrument is powered on. The gas chromatograph specified for the HGRMA has two columns, providing redundancy in case of column failure. Column and sample pump failures are the two types of failures noted with gas chromatograph operation at SRNL, but long term reliability has been noted in systems without large mono-nitrogen oxide emissions.

The mass flow controllers require periodic calibration; at SRNL these components are installed outside the cells to allow removal and calibrations. A one to two-year calibration frequency is specified for the MKS flow controllers at SRNL.

Calibration of the RTD temperature probes is required on some frequency. At SRNL, this type of calibration is performed by installation of a new probe. The new probe is used in a side-by-side evaluation with the old probe to perform the close out calibration check of the old probe. The old probe is then discarded. The RTD readout is sent for calibration since it remains outside the cell. A 2-5 year calibration frequency is used at SRNL for temperature probes and 2-3 years for temperature readouts.

The rotameter for the vortex chiller air should be considered an information only instrument and does not require calibration. Verification that the outlet temperature is maintained below the desired target is sufficient to indicate proper chiller function. Therefore, measurement of the air flow is a secondary indicator that allows troubleshooting to be performed but is not essential for operation.

Similarly, visual indication of mixing performance is the primary indication that the mixing is adequate with mixer speed a secondary indicator. Use of a tachometer to verify the rotational speed of the mixer is not required for successful operation of the HGRMA.

The temperature of the water bath does not impact the operation of the HGRMA and calibration of the internal temperature readings of the bath is not required.

6.4 Inspectibility

The vessel and condenser are fabricated from glass, allowing direct viewing of the cleanliness of the vessel prior to testing as well as allowing inspection of the sample and mixing during tests. In addition, the system is designed to allow remote assembly and disassembly to allow inspection of components. It is expected that some components (e.g. vessel O-ring) could be single use while others are expected to be replaced on some frequency (e.g. flexible agitator shaft).

A visual inspection of the HGRMA is to be performed in order to evaluate the following conditions prior to each use:

1. Broken, chipped or etched glassware
2. Frayed or damaged electrical connectors/cords
3. Discolored or cracked air/water/offgas hoses or tubing

The apparatus is mounted on a stand that allows the entire assembly to be positioned in order to allow inspection of any part of the system.

7.0 Conclusions

The simulant testing demonstrated the ability of the HGRMA to perform the required steady-state measurements of HGR during waste feed qualification.

The use of a krypton tracer gas did not interfere with any of the other gases measured during the test and allows a determination of steady state conditions to be made when no hydrogen is detected. Krypton should be used as the tracer gas during the waste feed qualification testing.

The drain valve on the HGRMA was used to recover the sample and perform initial rinsing of the vessel. However, the rinse protocols testing showed some material remained in the vessel and lid. It is required that the vessel be removed and manually cleaned between runs and that the lid is wiped clean in place.

The temperature target for HGR measurements should be adjusted to account for the ability of the HGRMA to hold the temperature setpoint within two degrees Fahrenheit. Thus, a measurement with a required temperature of 120 degrees Fahrenheit should be performed at 122 degrees Fahrenheit.

The time for the hydrogen measurement to reach steady-state was determined to be approximately 8 hours based on a model of the system as well as the results of the testing. The testing duration required to reach steady measurement needs to be verified during testing with actual radioactive waste samples to verify any changes due to radiolytic hydrogen generation. The use of a krypton tracer gas will allow for identification of steady-state conditions during tests below the detection limit for hydrogen.

8.0 Recommendations

It is recommended that a cylinder of zero air (synthetic air made from blending high purity nitrogen and oxygen) spiked with 0.5 weight percent krypton be used for the purge gas during HGR measurements. The use of zero air will prevent the nominal 5.2 parts per million helium from ambient air from interfering with the hydrogen measurement and the incorporation of the tracer gas prevents needing two flow controllers to provide the purge as well as preventing any errors in the flowrates from impacting the measurement of the tracer gas concentration. The low purge flowrates would allow a standard cylinder of gas to provide the needed purge gas for a large number of tests.

The mixing in the vessel should be closely monitored as the sample is heated to avoid splatter, particularly if the rheological properties are significantly reduced as the temperature is increased. The changing rheological properties will change the mixing characteristics of the sample and mixing should be adjusted accordingly.

It is recommended that a complete spare apparatus be maintained assembled on the stand in a clean area. When a component is needed for the apparatus installed in the shielded cell environment, it would be removed from the spare apparatus. A replacement for the removed component would be purchased/fabricated and installed on the spare skid. This approach allows for a check of the fit of the

new component on the apparatus and provides a readily available apparatus that would also support training and/or mockup testing.

9.0 References

1. M.E. Stone, "Evaluation of Methods to Measure Hydrogen Generation Rate in a Shielded Cell Environment and a Method Recommendation," Savannah River National Laboratory, Aiken, South Carolina, SRNL-STI-2012-00511, SCT- M0SRV00028-00-012-01-00001, 2012.

2. P.A. Benson, "Hanford Tank Waste Treatment and Immobilization Plant Iewo M0srv00028, Amendment 21 Waste Qualification Program Development Scope of Work and Technical Requirements Rev 3," River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Wa, SCT-M0SRV00028-00-012 Rev 3.,

3. A.V. Arakali and e. al, "Initial Data Quality Objectives for Wtp Feed Acceptance Criteria," River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Washington, 24590-WTP-RPT-MGT-11-014, Rev. 0, 2011.

4. P.A. Benson and T.G. Halverson, "Waste Feed Qualification Program Plan," River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Washington, 24590-WTP-PL-OP-12-0004, Rev. 0, 2013.

5. M.E. Stone, "Task Technical and Quality Assurance Plan for Measurement of Hydrogen Generation Rate During Waste Feed Qualification at the Hanford Tank Waste Treatment and Immobilization Plant," Savannah River National Laboratory, Aiken, South Carolina, SRNL-RP-2012-00858, SCT-M0SRV00028-00-012-01-00003, 2012.

6. M.E. Stone, J.D. Newell, and M.P. Jones, "Conceptual Design for Hydrogen Generation Rate Measurement Apparatus," Savannah River National Laboratory, Aiken, South Carolina, SRNL-RP-2013-00397, SCT-M0SRV00028-00-012-01-00005, 2013.

7. M.E. Stone, J.D. Newell, and D.J. Adamson, "Hydrogen Generation Rate Apparatus 30% Design Package," Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2013-00230, SCT-M0SRV00028-00-012-01-00007, 2013.

8. M.E. Stone, J.D. Newell, and D.J. Adamson, "Hydrogen Generation Rate Apparatus 60% Design Complete Package," Savannah River National Laboratory, Aiken, Sc, SRNL-L3100-2014-00059, SCT-M0SRV00028-00-012-01-00009, 2014.

9. M.E. Stone, D.J. Adamson, D.J. Pak, and J.M. Pareizs, "Hydrogen Generation Rate Measurement Apparatus: Final Design Package," Savannah River National Laboratory, Aiken, South Carolina, SRNL-RP-2014-00866, 2014.

10. M.E. Stone, J.M. Pareizs, and D.J. Adamson, "Hydrogen Generation Rate Measurement Apparatus Temperature Stability and Gas Chromatograph Proof of Concept Testing," Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2014-00217, 2014.

11. M.E. Stone and D.J. Adamson, "Hydrogen Generation Rate Measurement Apparatus Remote Operation and Maintenance Evaluation," Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2014-00264, 2014.

12. M.E. Stone and F.C. Johnson, "Glass Fabrication Unit Operations Tasks and Hydrogen Generation Rate Measurement Appartus: Final Verification Testing with Simulants," Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2015-00012, 2015.

13. D. Burbank, "Simulant Selection and Specification for Verification Testing of Waste Feed Qualification Apparatus," River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Washington, CCN 271283, 2014.

14. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Vessel Installation Turntable Subassembly Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R2-A-00028 Rev 0, 2016.

15. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Teca Tlc-900 Liquid Chiller/ Controller Wiring Diagram Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, E-EV-A-00082 Rev 0, 2016.

16. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Main Assembly Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R1-A-00092 Rev 0, 2016.

17. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Vessel Assembly and Details Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R1-A-00081 Rev 0, 2016.

18. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Condenser, Funnel, Manometer and Bubbler Details Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R4-A-00112 Rev 0, 2016.

19. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Details Sheet 1 Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R4-A-00114 Rev 0, 2016.

20. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Details Sheet 2 Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R4-A-00123 Rev 0, 2016.

21. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Details Sheet 3 Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, R-R4-A-00126 Rev 0, 2016.

22. "Hydrogen Gas Rate Measurement Apparatus (Hgrma) Process Flow Diagram Unclassified (U)," Savannah River National Laboratory, Aiken, South Carolina, J-JJ-A-00007 Rev 0, 2016.

23. M.E. Stone, "Operation of the Hydrogen Generation Rate Appatus for Hanford Waste Treatment Plant Waste Qualification Testing," Savannah River National Laboratory, Aiken, South Carolina, L29-ITS-0207, 2015.

24. M.E. Stone, "Electronic Notebook for Hgrma Experimentation," Savannah River National Laboratory, Aiken, South Carolina, Y4968-00031, 2016.

25. M.E. Stone, J.D. Newell, and D.J. Adamson, "Hydrogen Generation Rate Apparatus Proof of Concept Testing Summary," Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2013-00229, SCT-M0SRV00028-00-012-01-00006, 2013.

26. M.E. Stone and D.J. Adamson, "Assembly of the Hydrogen Generation Rate Apparatus," Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2014-00110, 2014.

27. P.A. Benson and M.W. Vanatta, “Wtp/Srnl – Hgr Apparatus Definitive Design Update,” River Protection Project, Waste Treatment Plant, Bechtel National Inc, Richland, Washington, CCN-257306, Rev.0, 2013.

28. P.A. Benson and M.W. Vanatta, “Wtp/Srnl - Hgr Apparatus Definitive Design Update,” River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Washington, CCN-257311, Rev 0, 2014.

29. P.A. Benson and M.W. Vanatta, “Wtp/Srnl - Hgr Apparatus Definitive Design Update,” River Protection Project, Waste Treatment Plant, Bechtel National Inc., Richland, Washington, CCN-268476, Rev 0, 2014.

30. M.E. Stone and D.J. Adamson, “Srnl Procurements and Receipts in Support of the Wtp Hgr Apparatus Task,” Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2014-00147, SCT-M0SRV00028-00-012-02-00004, 2014.

31. M.E. Stone and D.J. Adamson, “Srnl Procurement of Gas Chromatograph for Wtp Hgr Apparatus Task,” Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2014-00072, Rev 0, SCT-M0SRV00028-00-012-02-00001); , 2014.

32. M.E. Stone and D.J. Adamson, “Hgr Apparatus Assembly Completion,” Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2014-00172, Rev 0, SCT-M0SRV00028-00-012-02-00005, 2014.

33. M.E. Stone and D.J. Adamson, “Vendor Documentation for Hydrogen Generation Rate Measurement Apparatus,” Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2014-00214, 2014.

34. M.E. Stone, “Draft Hgr Measurement Apparatus Assembly Instructions,” Savannah River National Laboratory, Aiken, South Carolina, SRNL-L3100-2014-00233, 2014.

35. “Design Guide: Reliability, Availability, Maintainability, and Inspectability (Rami),” Bechtel National Inc., Richland, WA, 24590-WTP-GPG-ENG-009 Rev2, 2006.

36. J.M. Pareizs and C.L. Crawford, “Sludge Batch Washing and Demonstration of the Dwpf Flowsheet in the Srnl Shielded Cells for Sludge Batch 8 Qualification,” Savannah River National Laboratory, Aiken, South Carolina, SRNL-STI-2013-00116, 2013.

37. J. Berry, “One System 222s Laboratory Operational Research Model Assessment Report,” Washington River Protection Solutions, Richland, Washington, RPP-RPT-51533, Rev. 1 2014.

Appendix A. Hydrogen Measurement Method and Graphs

Gas Chromatography Method

Presented below are screen shots of the gas chromatography method for measuring hydrogen with a krypton tracer. The method is designed to identify hydrogen, oxygen, nitrogen and krypton utilizing Channel A of the gas chromatograph. The intent is to utilize Channel B as a backup, or it may be desirable to occasionally switch between channels A and B. Values in these screen shots may need to be adjusted, particularly retention times, as the gas chromatograph is used.

Instrument Set Up

Figure A-1 details the gas chromatograph setup – column temperatures, pressures, inject time, sample pump run time, etc. Figure A-2 and Figure A-3 show the state of each channel's filament, that data is to be acquired, etc. Note that with the krypton tracer, run time must be at least 1.5 min, not the 1.25 min given in the screen shots.

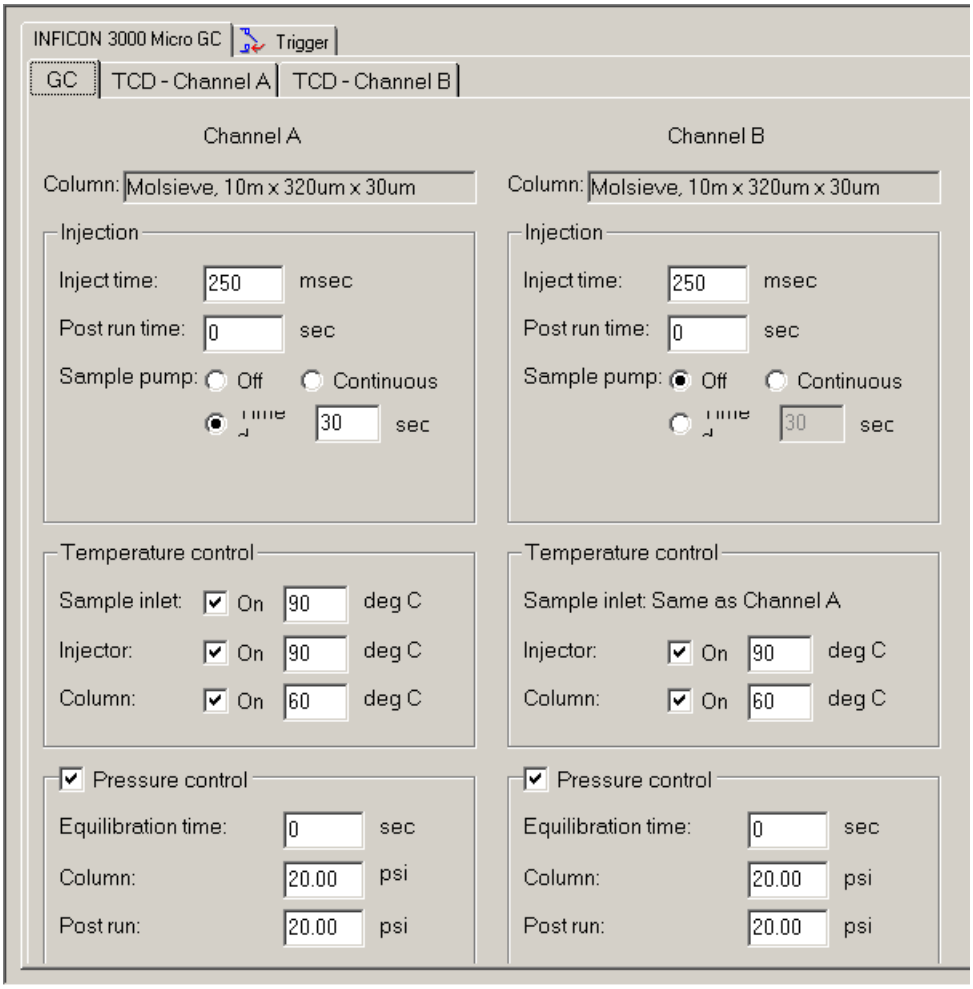


Figure A-1. GC Setup Parameters

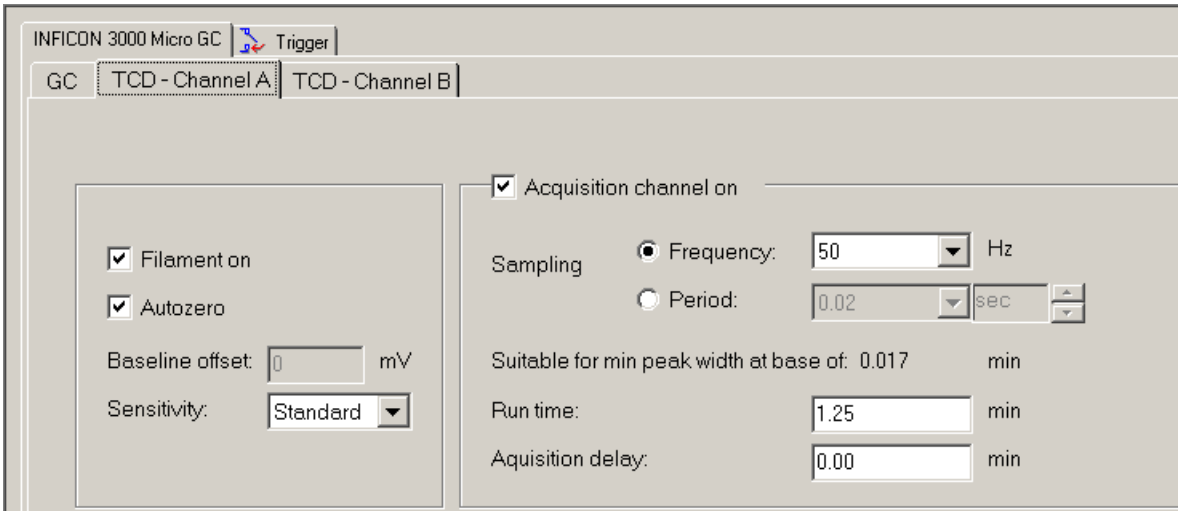


Figure A-2. Channel A Setup Parameters

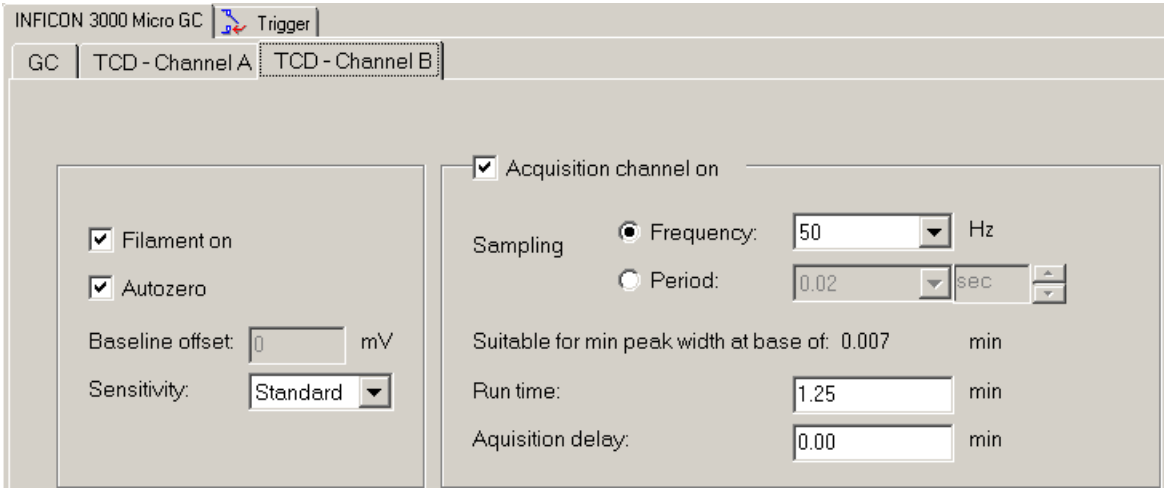
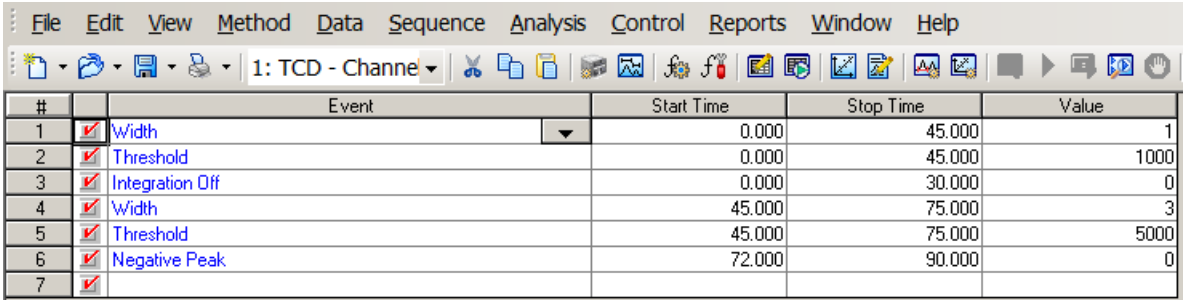


Figure A-3. Channel B Setup Parameters

Integration Settings

Figure A-4 shows the integration settings used to quantify the desired gas peaks of hydrogen, oxygen, nitrogen, and krypton.



#	Event	Start Time	Stop Time	Value
1	Width	0.000	45.000	1
2	Threshold	0.000	45.000	1000
3	Integration Off	0.000	30.000	0
4	Width	45.000	75.000	3
5	Threshold	45.000	75.000	5000
6	Negative Peak	72.000	90.000	0
7				

Figure A-4. GC Integration Settings

Peak Table

Figure A-5 shows the method peak table. This table lists the peaks to be identified, expected retention time, window, and concentration of the calibration gas (Level 1), among other parameters.

Named Peaks | Groups

#	Name	ID	Ret. Time	Window	Ref. ID #	ISTD. ID #	Resolution ID #	Units	RT Update
1	H2	1	39.1	2.3	0	0	0		Calib
2	O2	2	50.1	4	0	0	0		Calib
3	N2	3	63.72	4.46151	0	0	0		Calib
4	Kr	4	79	4.5	0	0	0		None
5									

#	Name	ID	LOD	LOQ	Quantitate	Fit Type	Zero	Calib Flag
1	H2	1	0.000000	0.000000	Area	Point-to-Point		Replace
2	O2	2	0.000000	0.000000	Area	Point-to-Point		Replace
3	N2	3	0.000000	0.000000	Area	Point-to-Point		Replace
4	Kr	4	0.000000	0.000000	Area	Point-to-Point		Replace
5								

#	Name	ID	Calib Weight	% Calib Margin	Scale	Weighting Method
1	H2	1	100	0	None	None
2	O2	2	100	0	None	None
3	N2	3	100	0	None	None
4	Kr	4	100	0	None	None
5						

#	Name	ID	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1	H2	1	20.1						
2	O2	2	20.9						
3	N2	3	78						
4	Kr	4	1						
5									

#	Name	ID	STD ID #	STD Mult.	Manual RF	Low Conc	High Conc	Check Std 1 Conc	Check Std 1 %RD	Check Std 2 Co
1	H2	1	0	1		0	0	0	0	
2	O2	2	0	1		0	0	0	0	
3	N2	3	0	1		0	0	0	0	
4	Kr	4	0	1		0	0	0	0	
5										

Figure A-5. Gas Chromatography Method Peak

Example chromatograms are shown in Figure A-6 through Figure A-9. Hydrogen in air was indicated by a small hydrogen peak in Figure A-9. Note the absence of the helium peak in Figure A-7 as this run used zero air with hydrogen added at 20 parts per million which was blended with a krypton tracer while a large helium peak (relative to hydrogen) is shown in Figure A-9.

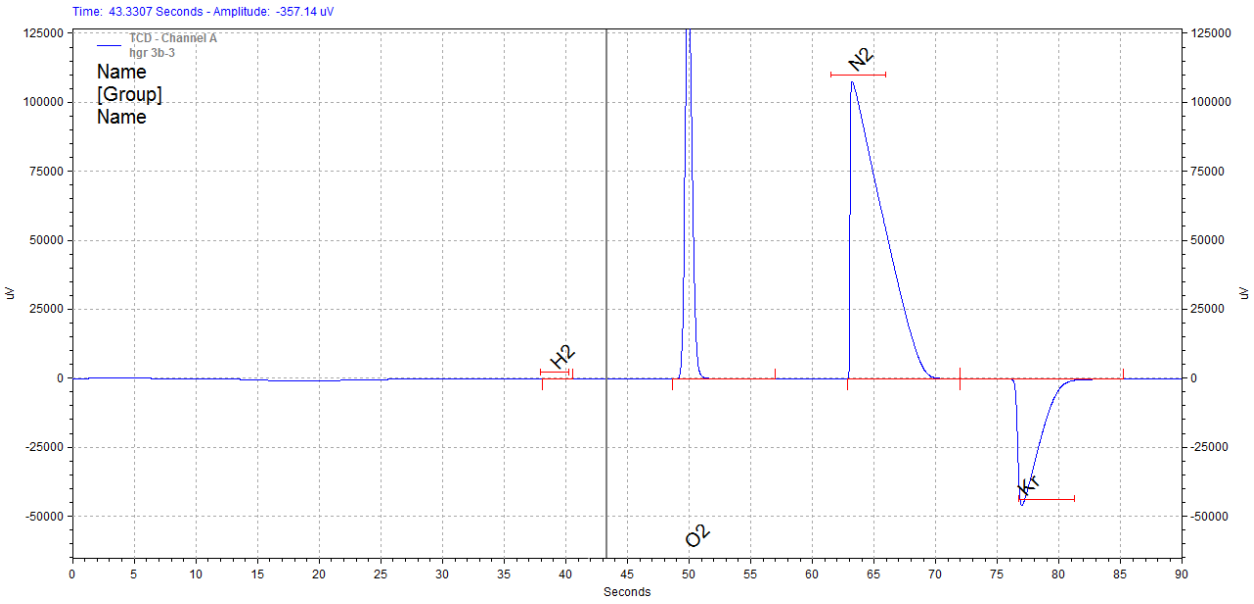


Figure A-6. Chromatogram from Run with 20 parts per million Air and Krypton Tracer

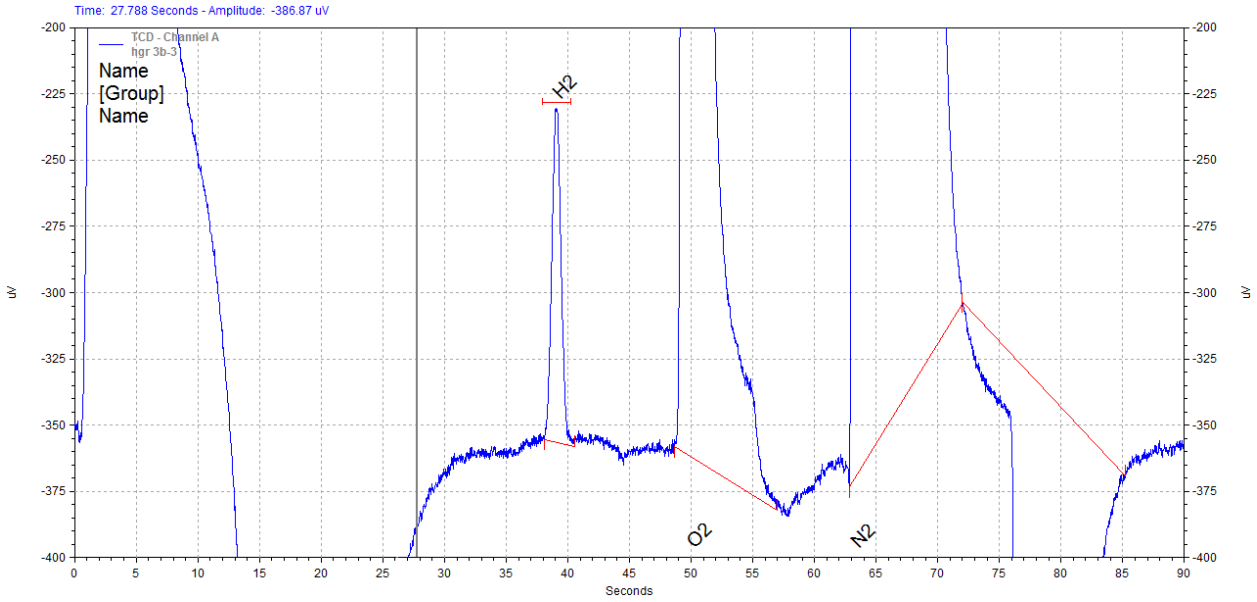


Figure A-7. 20 parts per million Hydrogen Chromatogram Enlarged to Show Hydrogen Peak

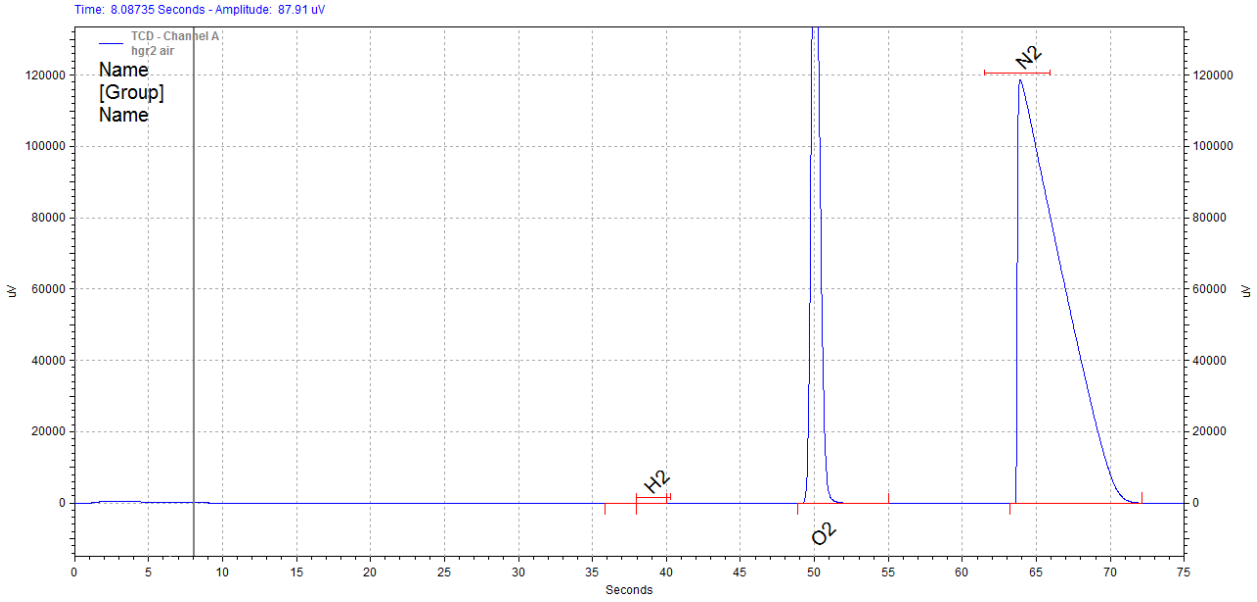


Figure A-8. Chromatogram of ACTL Instrument Air

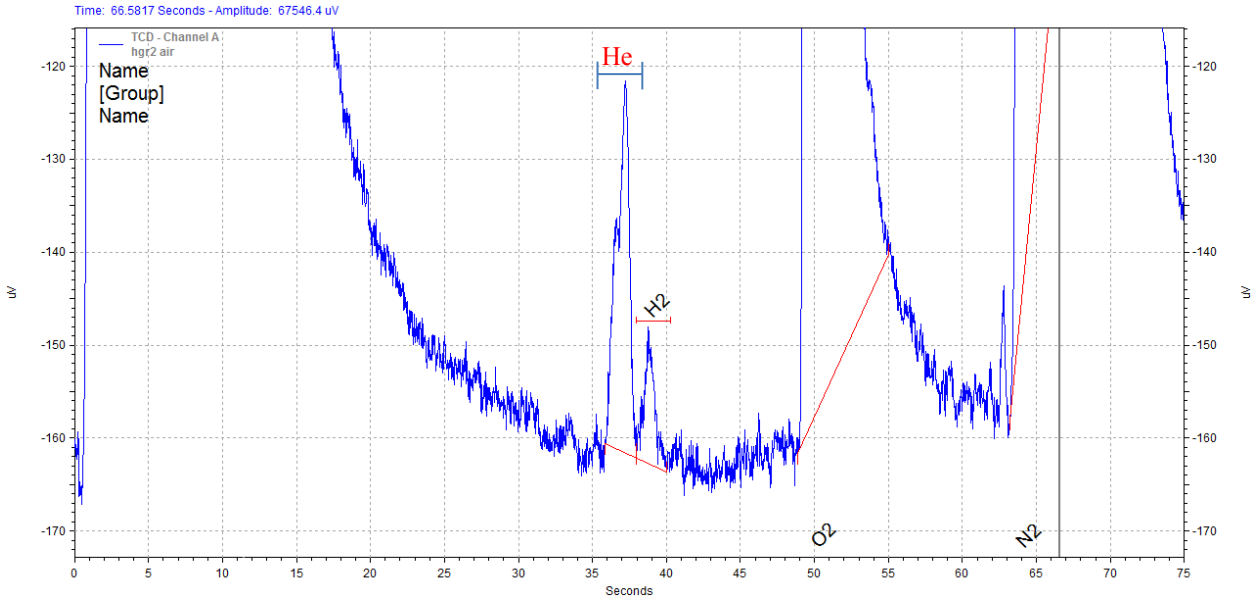


Figure A-9. ACTL Instrument Air Chromatogram Enlarged to Show Helium and Hydrogen Peaks

The results of the HGRMA tests are shown in Figure A-10 through Figure A-25. These figures show the hydrogen data in parts per million during each test run. These charts only show the hydrogen data during the test run; the data from the gas chromatograph during calibration is not shown.

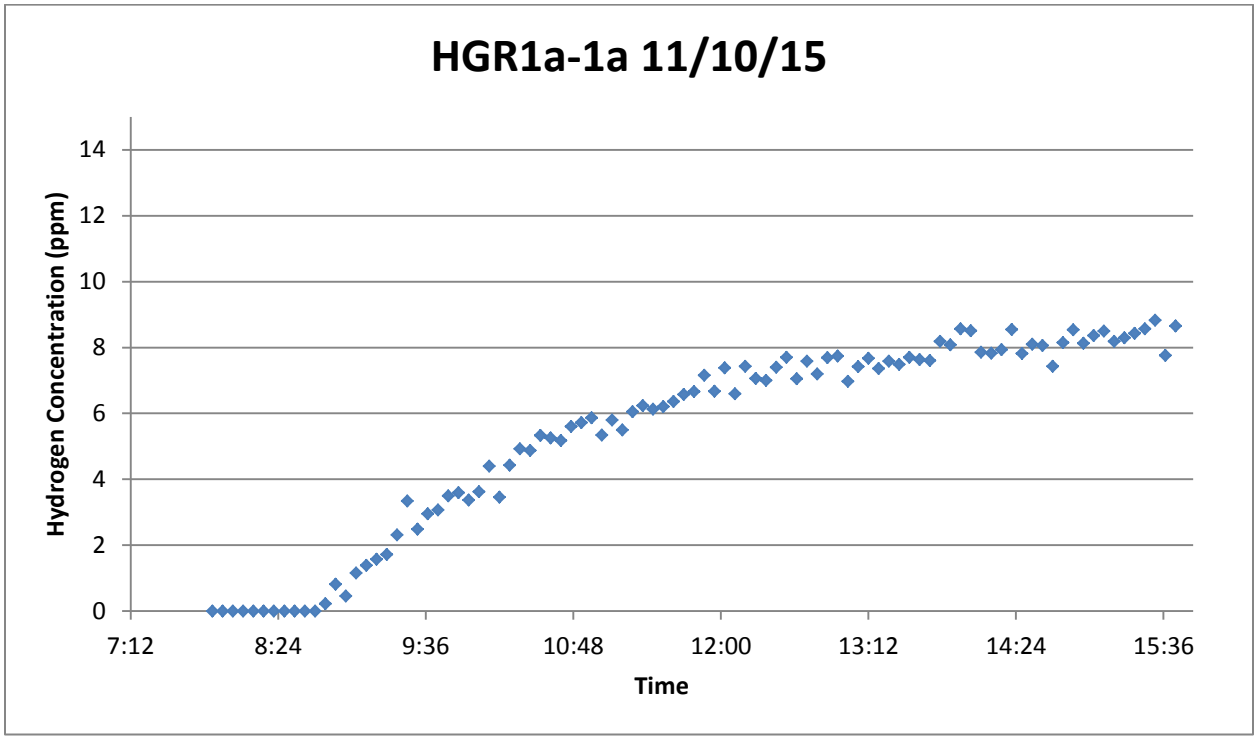


Figure A-10. HGR1a-1 Test Results

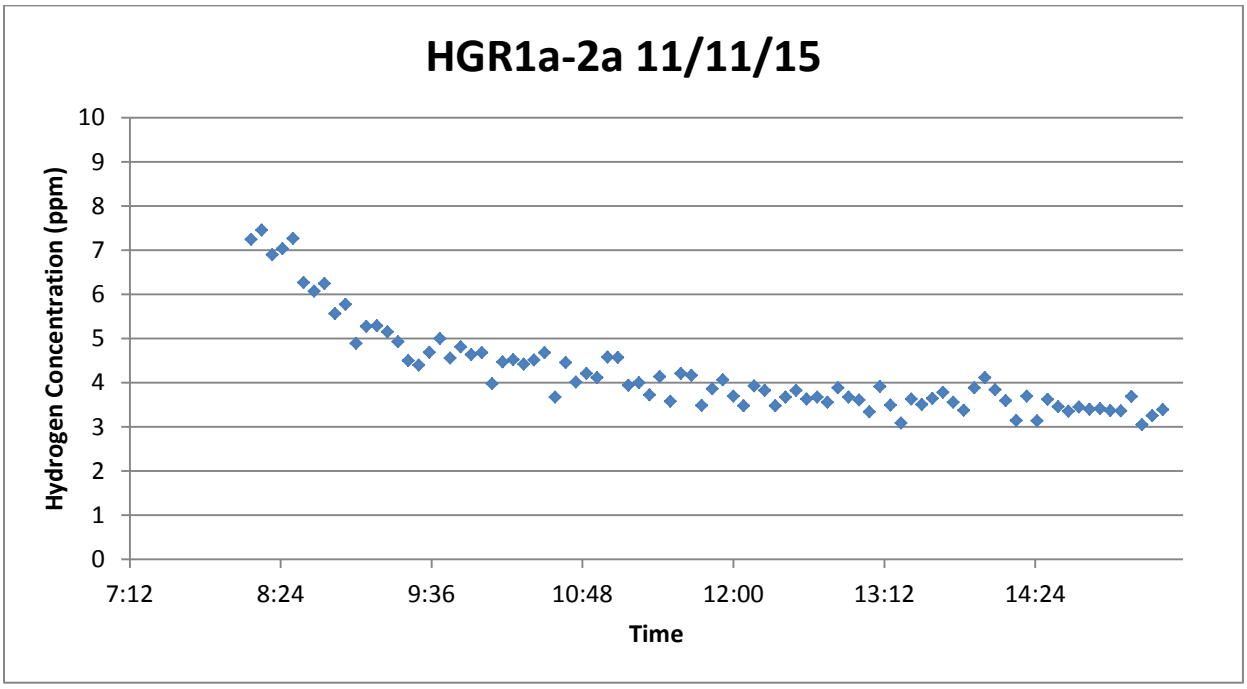


Figure A-11. HGR1a-2 Test Results

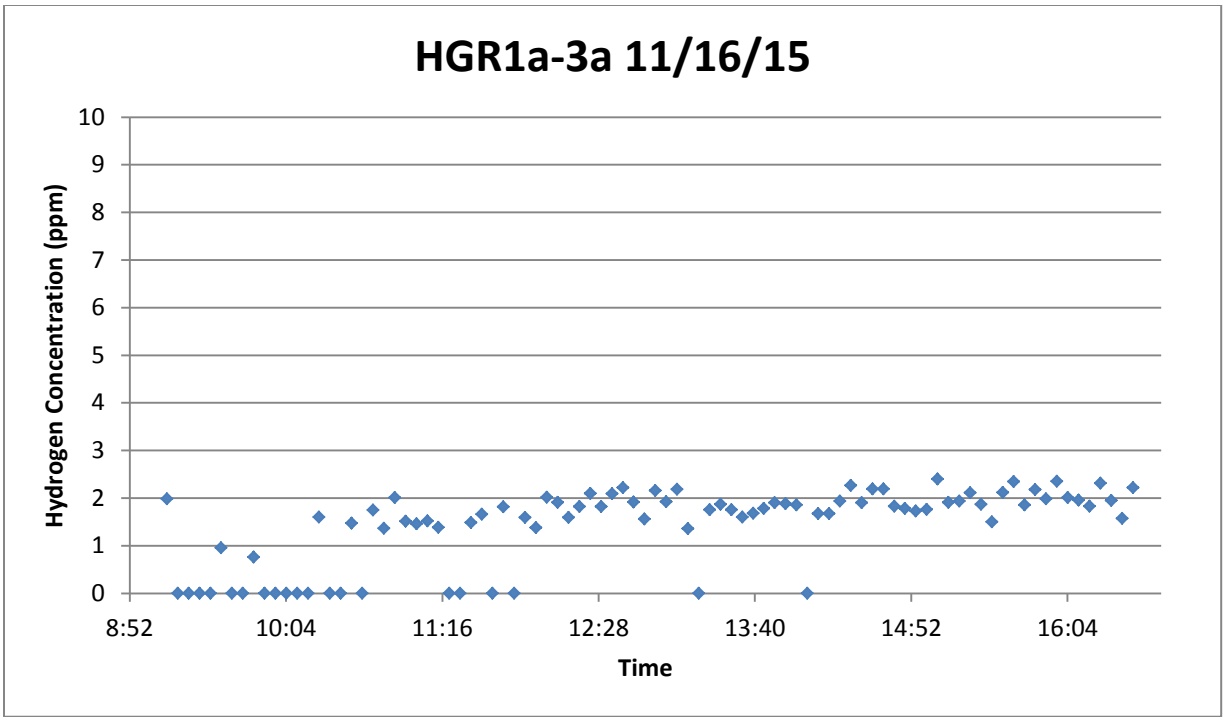


Figure A-12. HGR1a-3 Test Results

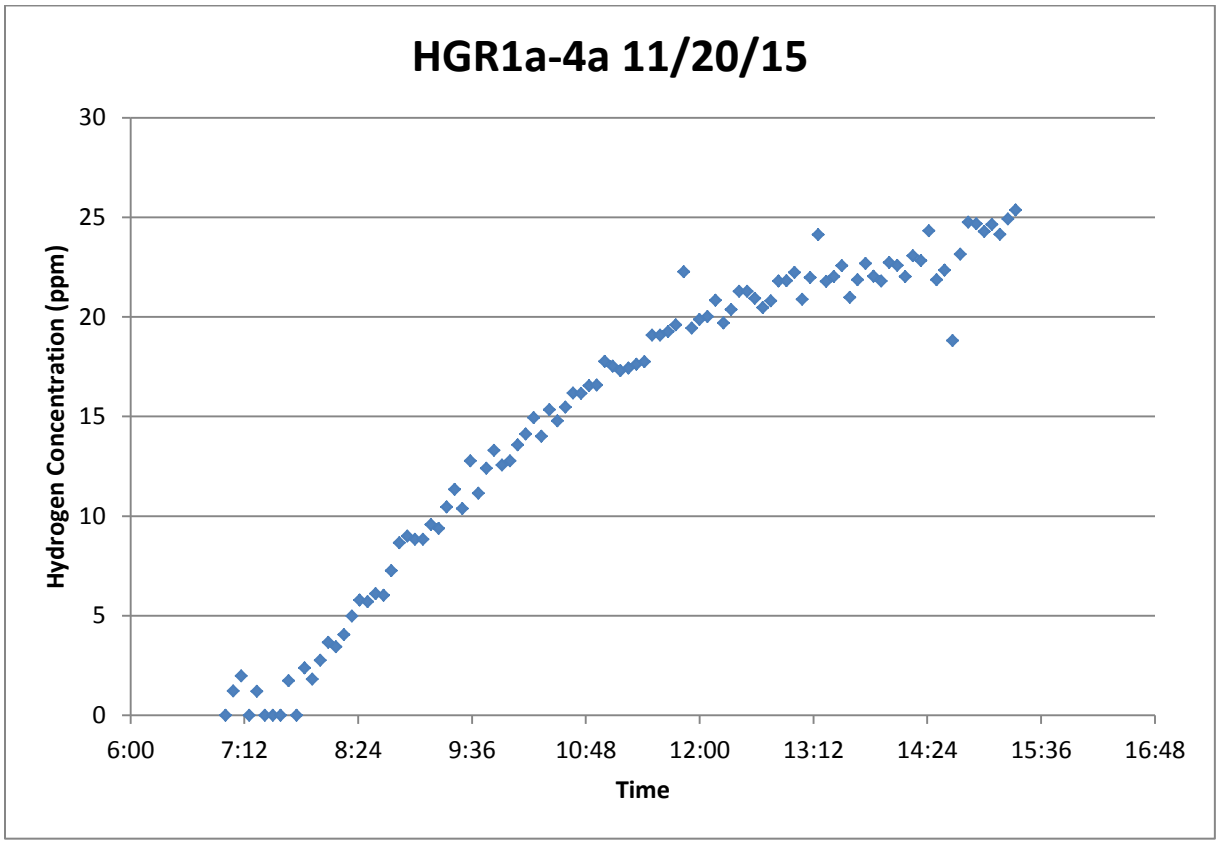


Figure A-13. HGR1a-4 Test Results

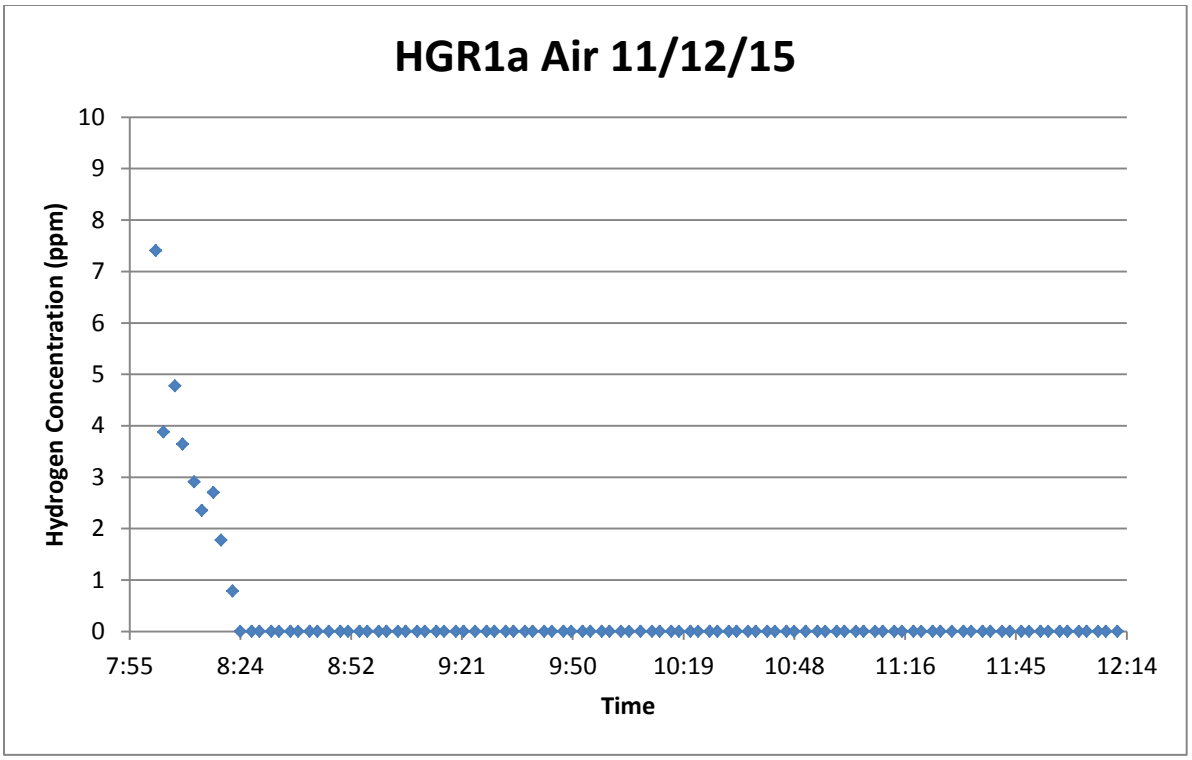


Figure A-14. HGR1a-Air Test Results

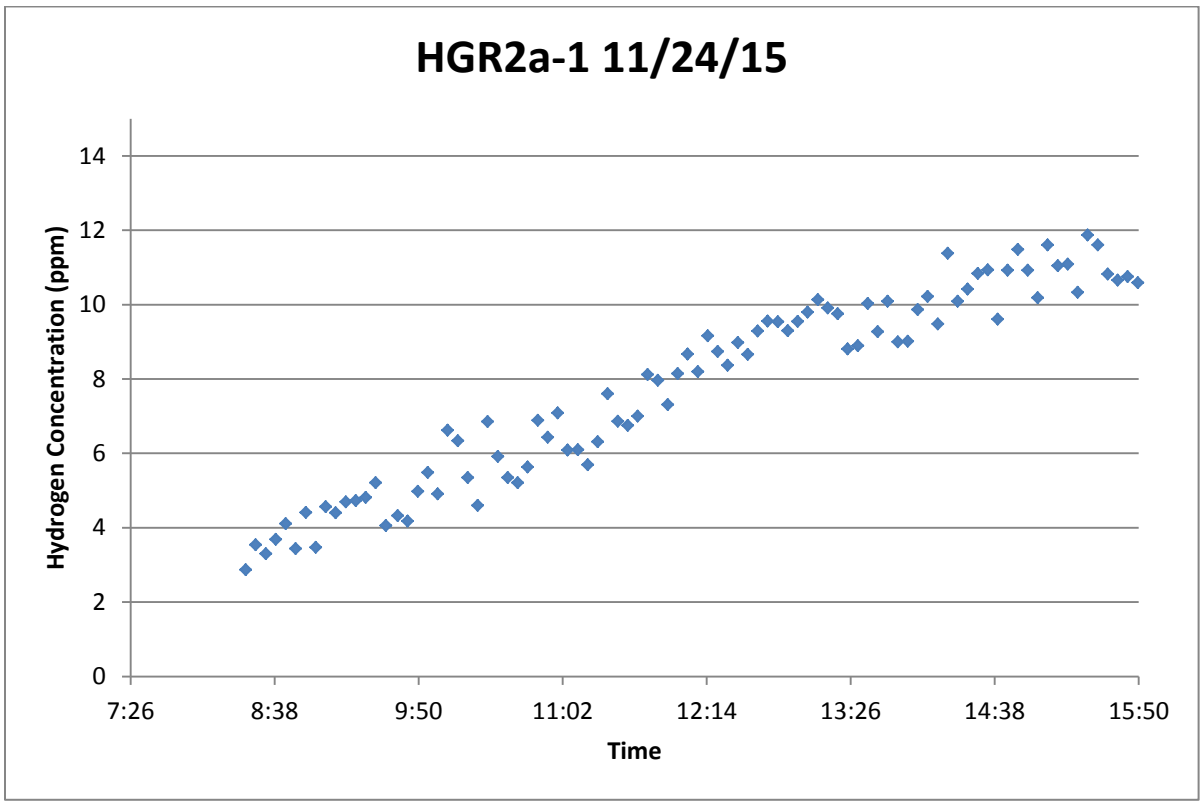


Figure A-15. HGR2a-1 Test Results

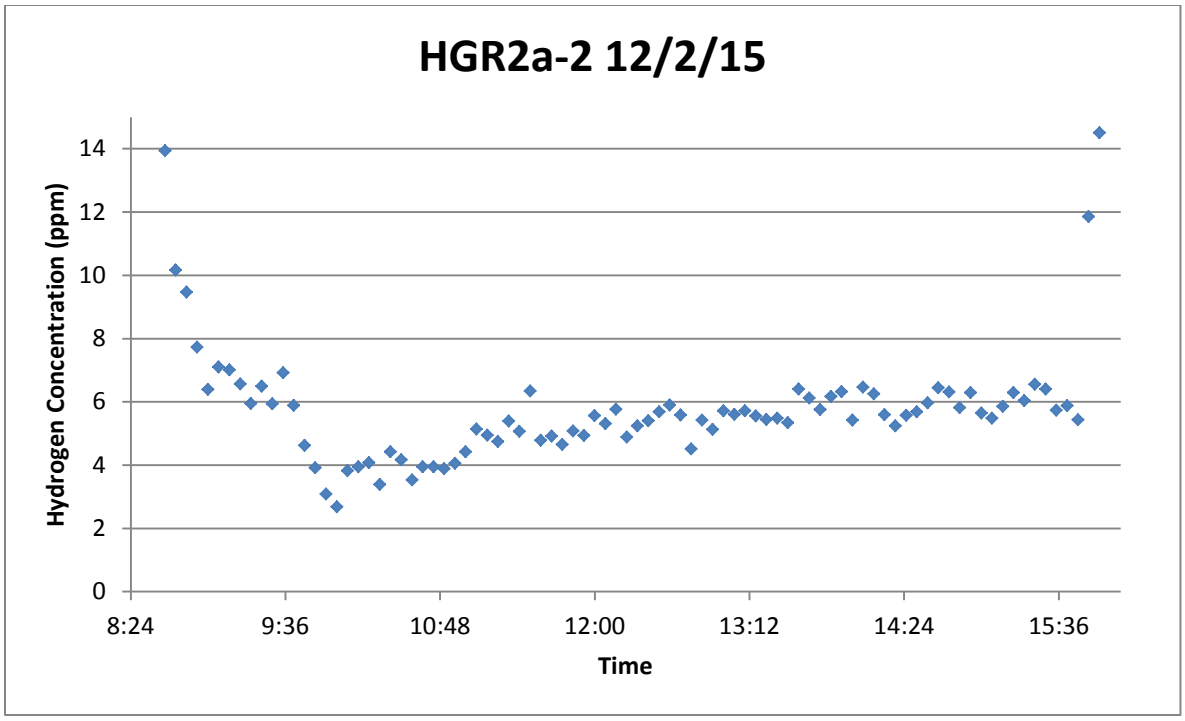


Figure A-16. HGR2a-2 Test Results

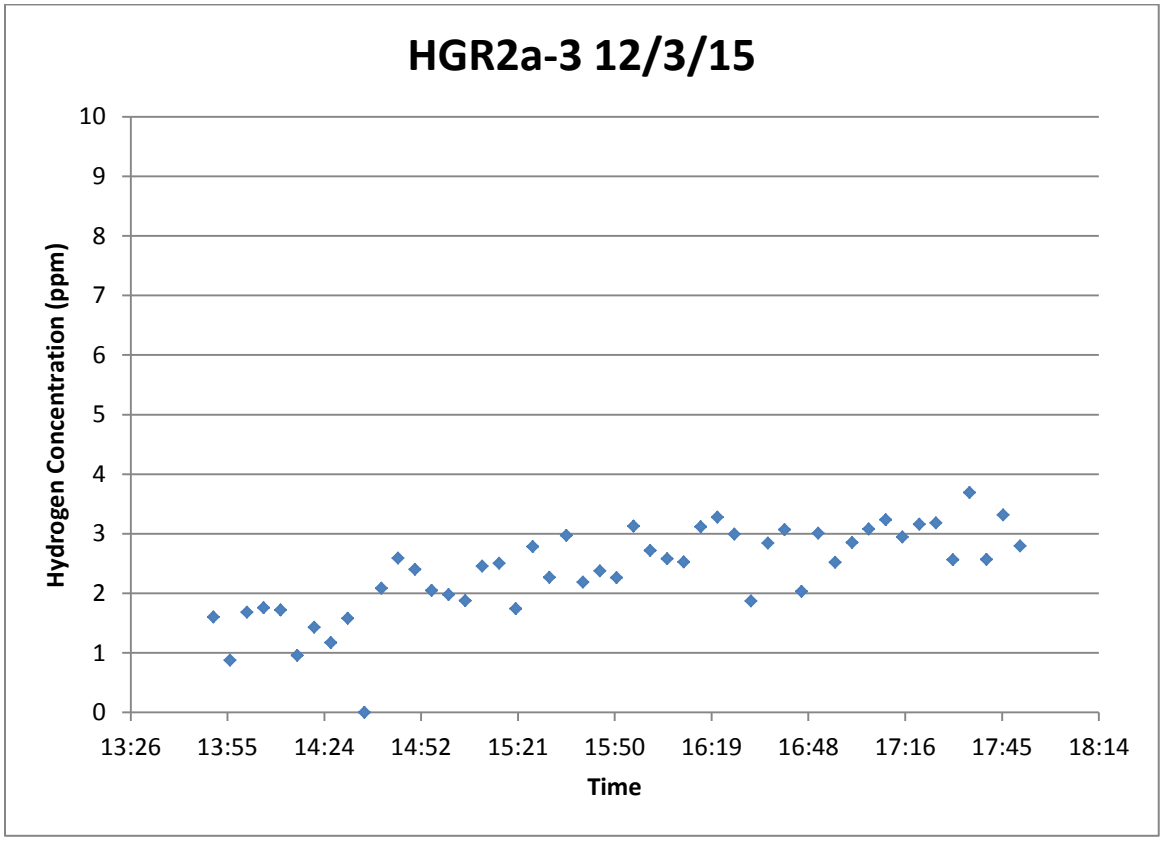


Figure A-17. HGR2a-2 Test Results

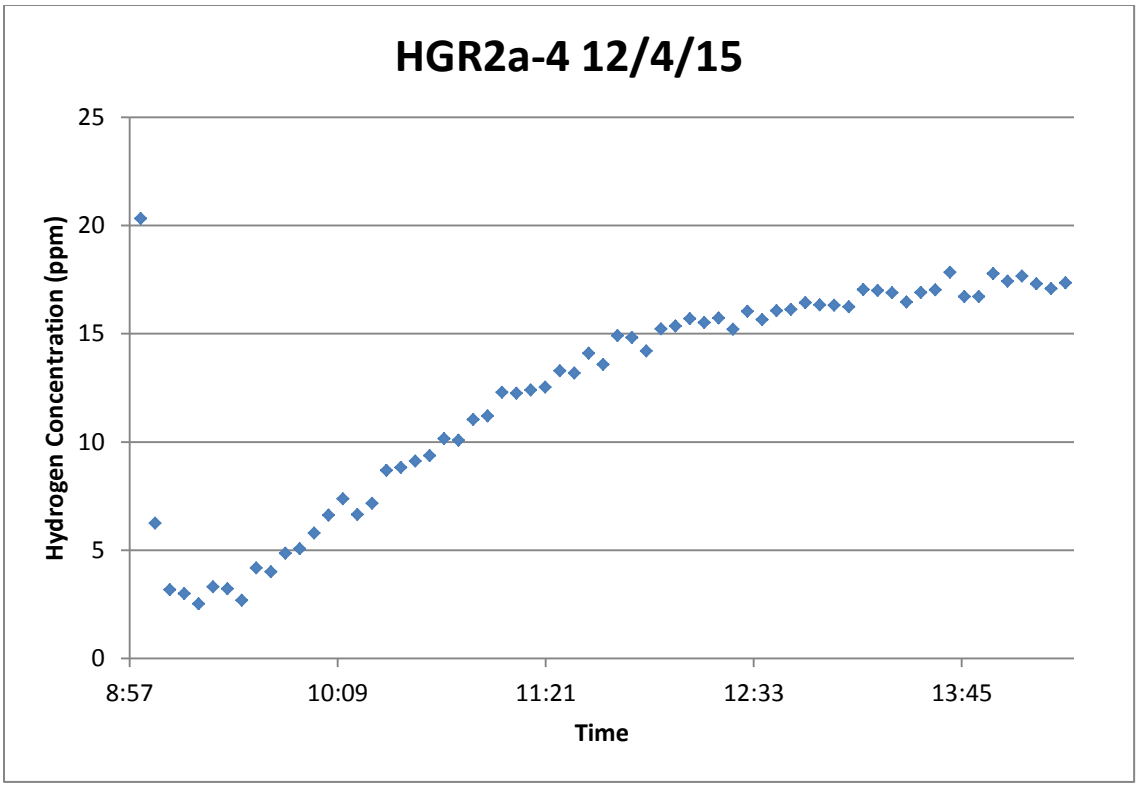


Figure A-18. HGR2a-4 Test Results

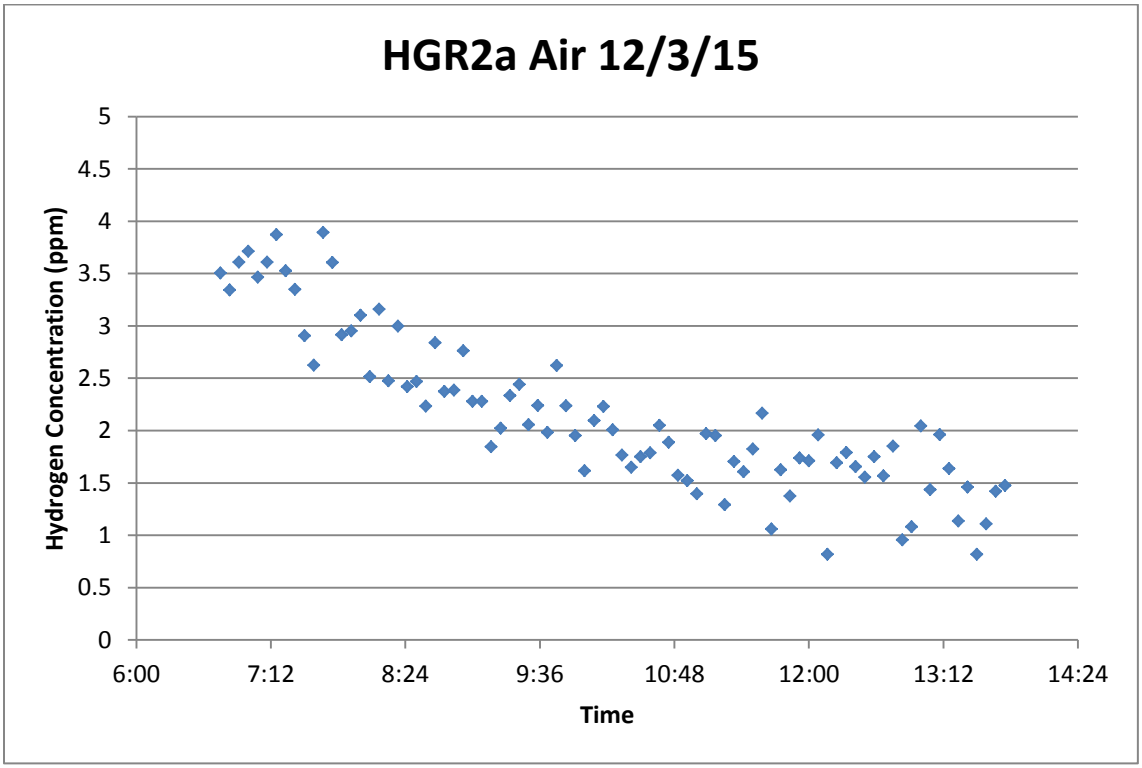


Figure A-19. HGR2a-Air Test Results

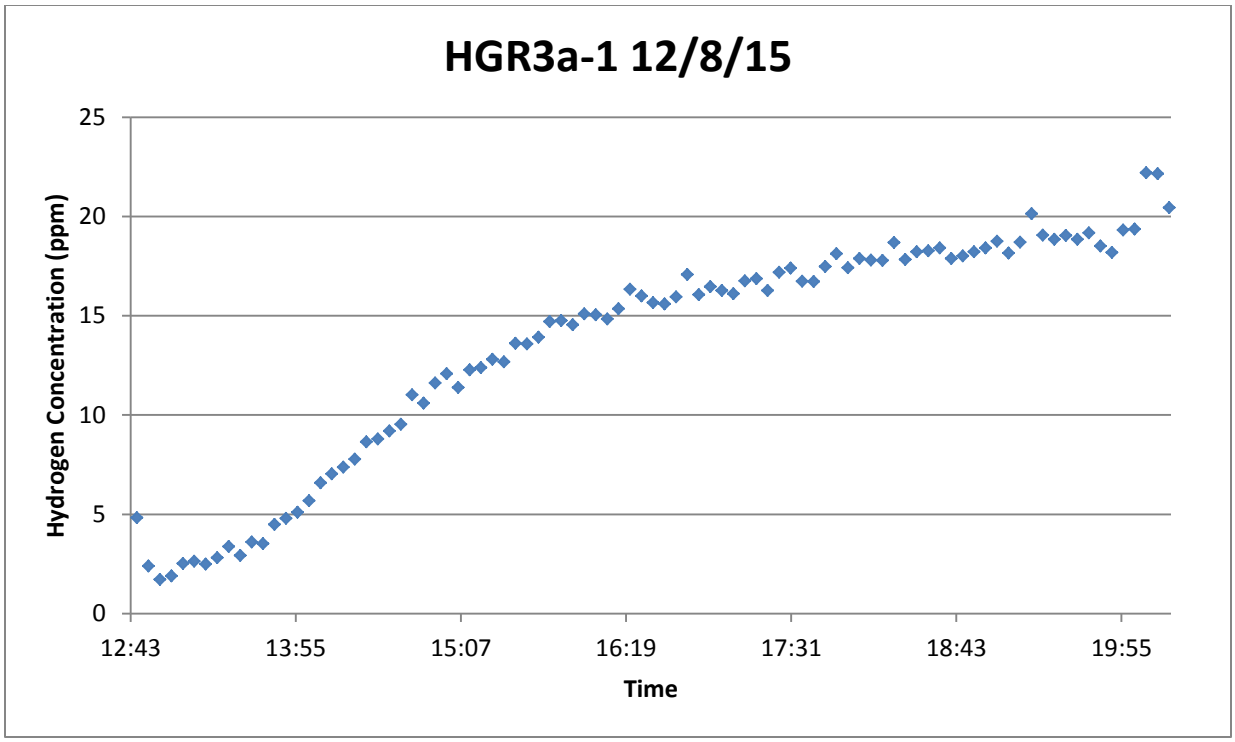


Figure A-20. HGR3a-1 Test Results

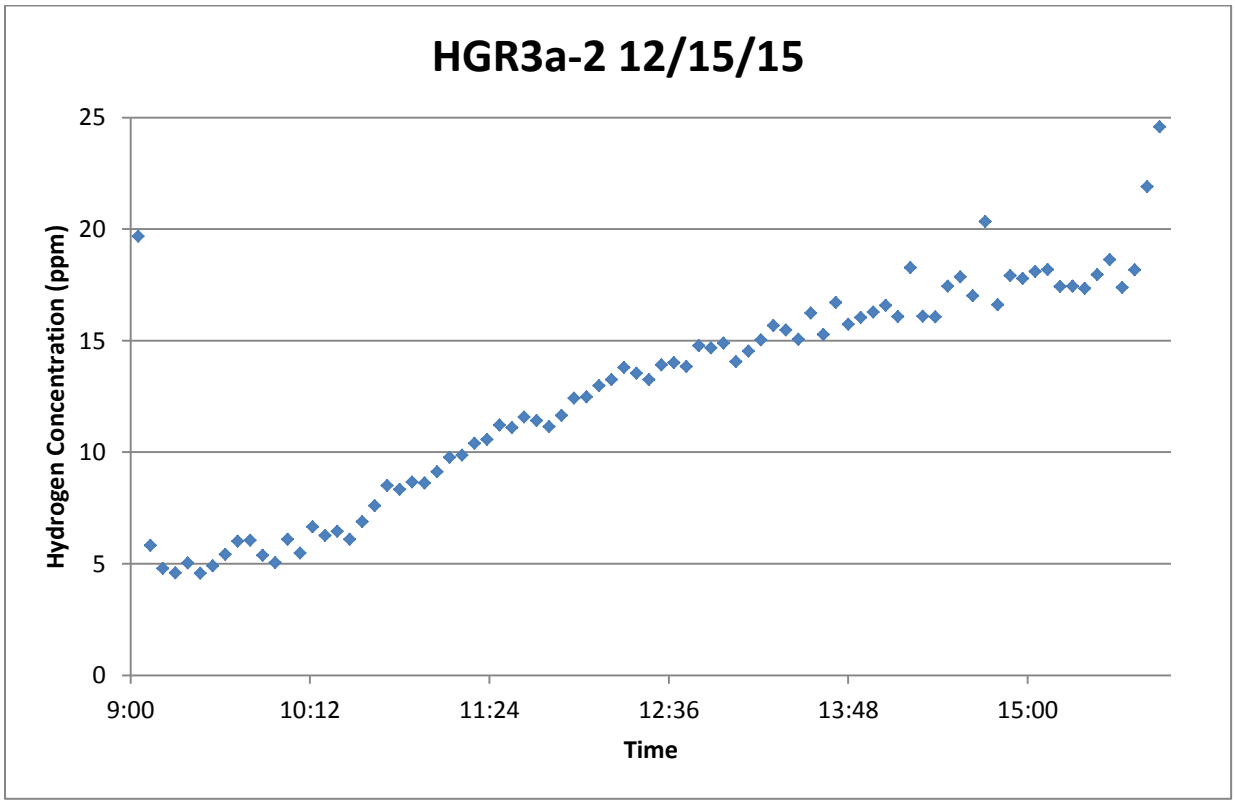


Figure A-21. HGR3a-2 Test Results

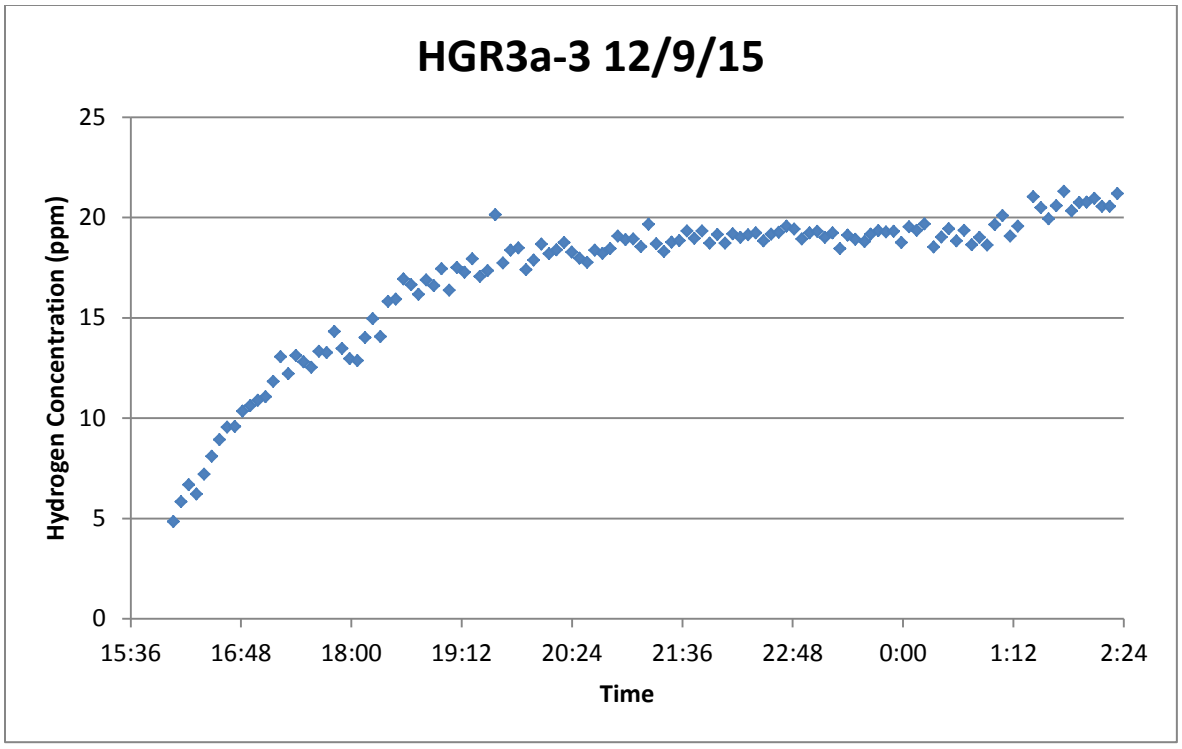


Figure A-22. HGR3a-3 Test Results

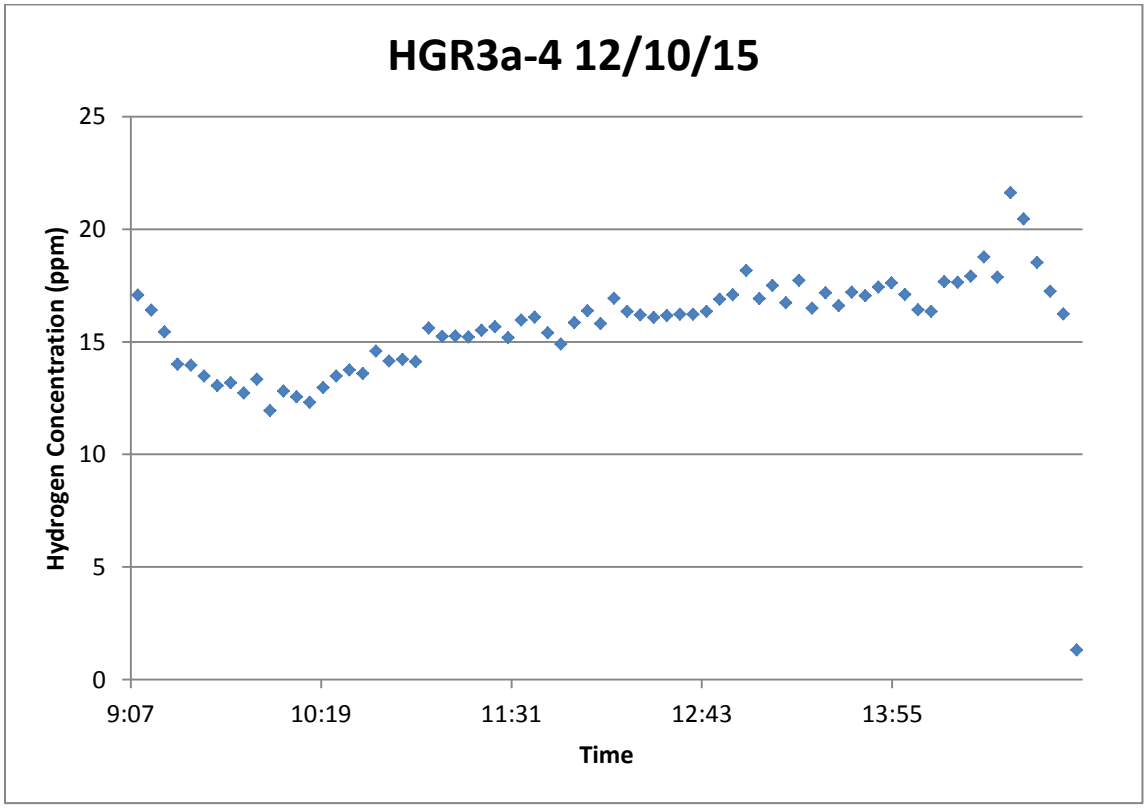


Figure A-23. HGR3a-4 Test Results

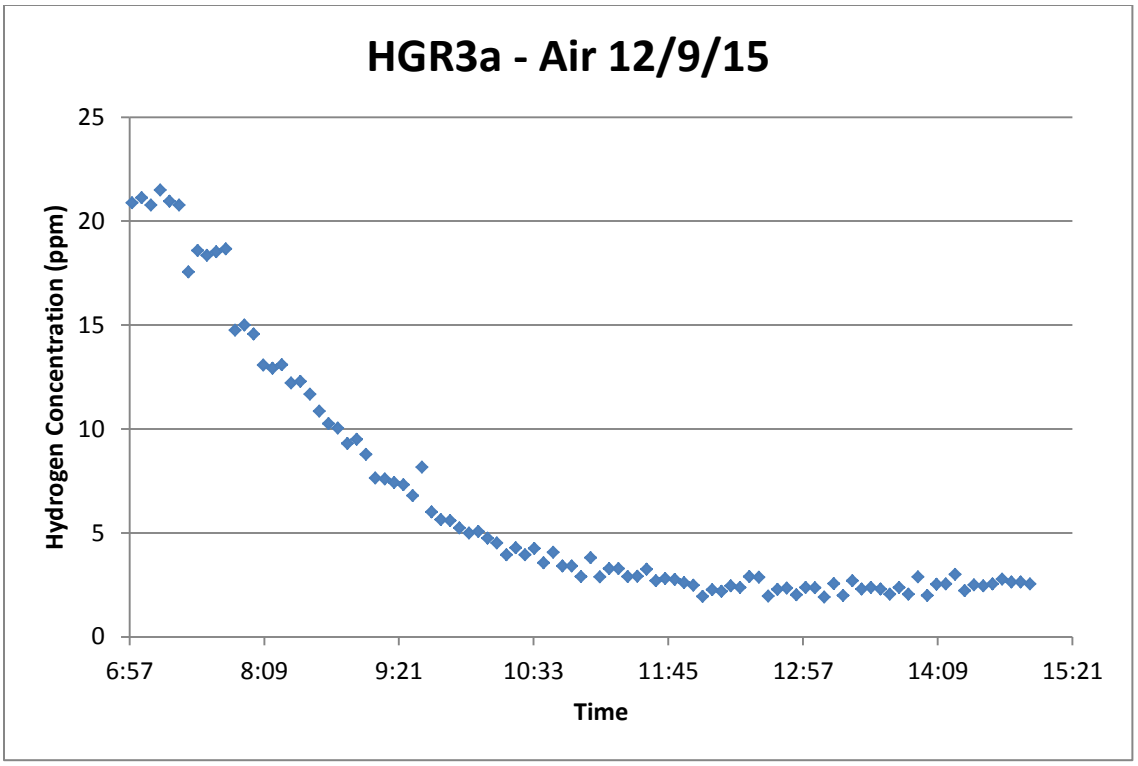


Figure A-24. HGR3a-Air Test Results

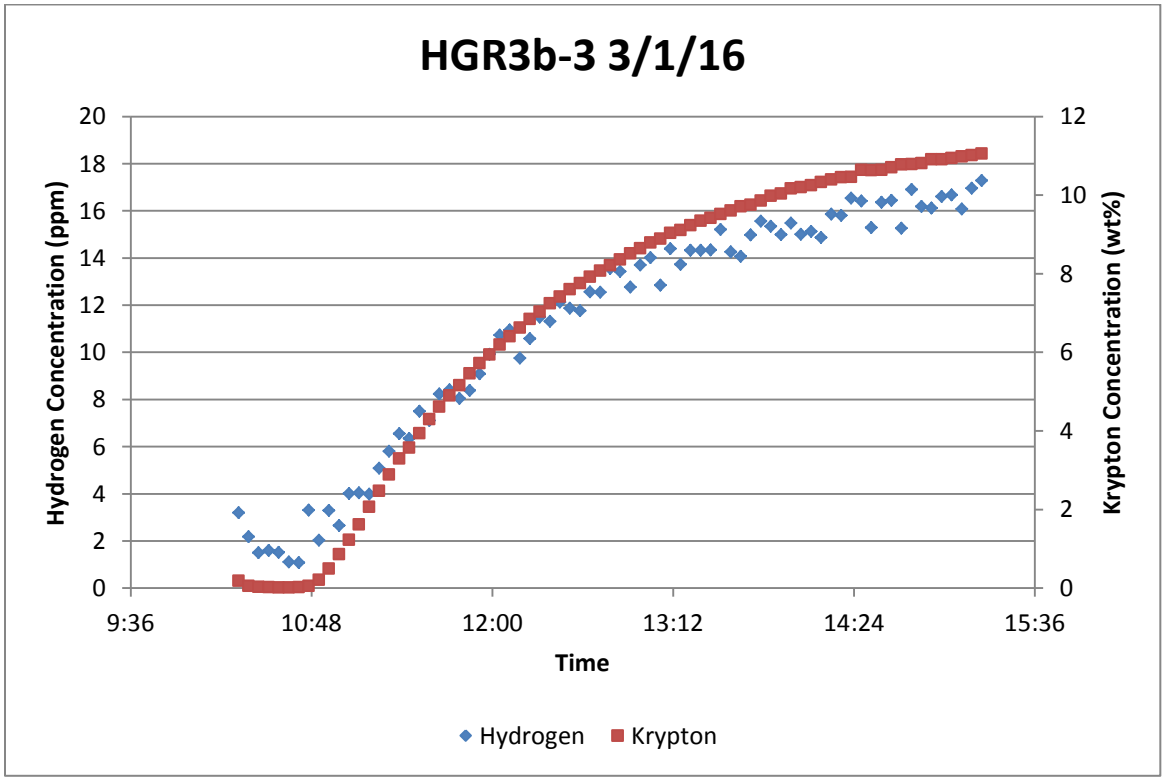


Figure A-25. HGR3b-3 Test Results

Appendix B. HGR CSTR Model Results

The factors used to normalize the hydrogen values to a value of 20 parts per million are shown in Table B-1. These values are frequently the same as the error noted in the steady state values, but some were adjusted to better fit the model curve. As shown, at 20 parts per million the average adjustment factor was less than 10% different from the expected value of 1.0 with only one run over 10 percent off. At 10 parts per million, the expected adjustment factor was 2.0, and both runs had values within 15 percent of that value. At 5 and 2.5 parts per million, the differences between the expected adjustments and the actual adjustment were 20 to 32 percent. It should be noted that sources of error for values other than 20 parts per million include any errors in flowrates of the calibration gas and air; separation of these errors from errors in measurement is not possible.

The delay between the predicted value and the measured value was typically 50 minutes. The delay for HGR1a-3 was somewhat indeterminate as the required adjustments made determining what delay fit the data difficult.

Table B-1. Factors Used to Adjust HGRMA Gas Chromatography Data

Run ID	Expected Factor	Factor Used	Delta (%)	Delay between Model and Gas Chromatograph Measurement (minutes)
HGR1a-1	2.0	2.3	14.5	50
HGR1a-2	4.0	5.9	32.0	63
HGR1a-3	8.0	10.0	20.0	25
HGR1a-4	1.0	0.9	-15.0	50
HGR2a-1	2.0	1.9	-7.0	50
HGR2a-2	4.0	3.3	-22.0	40
HGR2a-3	8.0	6.3	-28.0	50
HGR2a-4	1.0	1.1	7.5	15
HGR3a-1	1.0	1.0	3.8	50
HGR3a-2	1.0	1.1	8.5	35
HGR3a-3	1.0	1.0	3.0	30
HGR3a-4	1.0	1.1	8.5	65
Water 70-20	1.0	1.1	10.0	40
Water 140-20	1.0	1.1	6.5	25
Water 70-5	4.0	3.7	-8.0	110
Water 140-5	4.0	4.0	0.0	40
HGR3b-3	1.14	1.14	0	40
HGR3b-3 (Kr)	1.6	1.6	0	50

Most data was an extremely good fit for the model. The small adjustments needed in the 20 parts per million data for the curves to fit the model indicate that the error in the flow rates is a significant source of the error in the tests at low hydrogen concentrations during the testing. However, quantification of this source of error was not attempted. As shown in Table B-1, the error in the measured hydrogen concentration during the water runs using the 5 parts per million calibration gas had errors less than 10 percent from the expected correction factor.

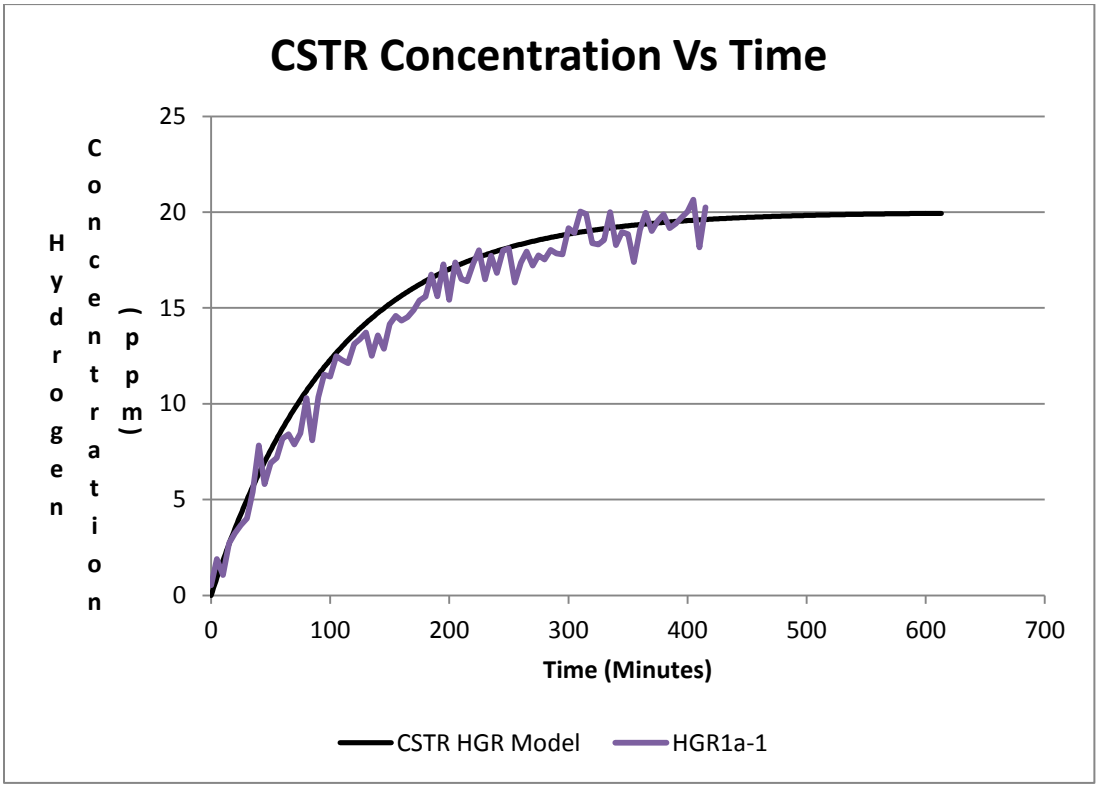


Figure B-1. HGR1a-1 Results Overlaid with CSTR Model

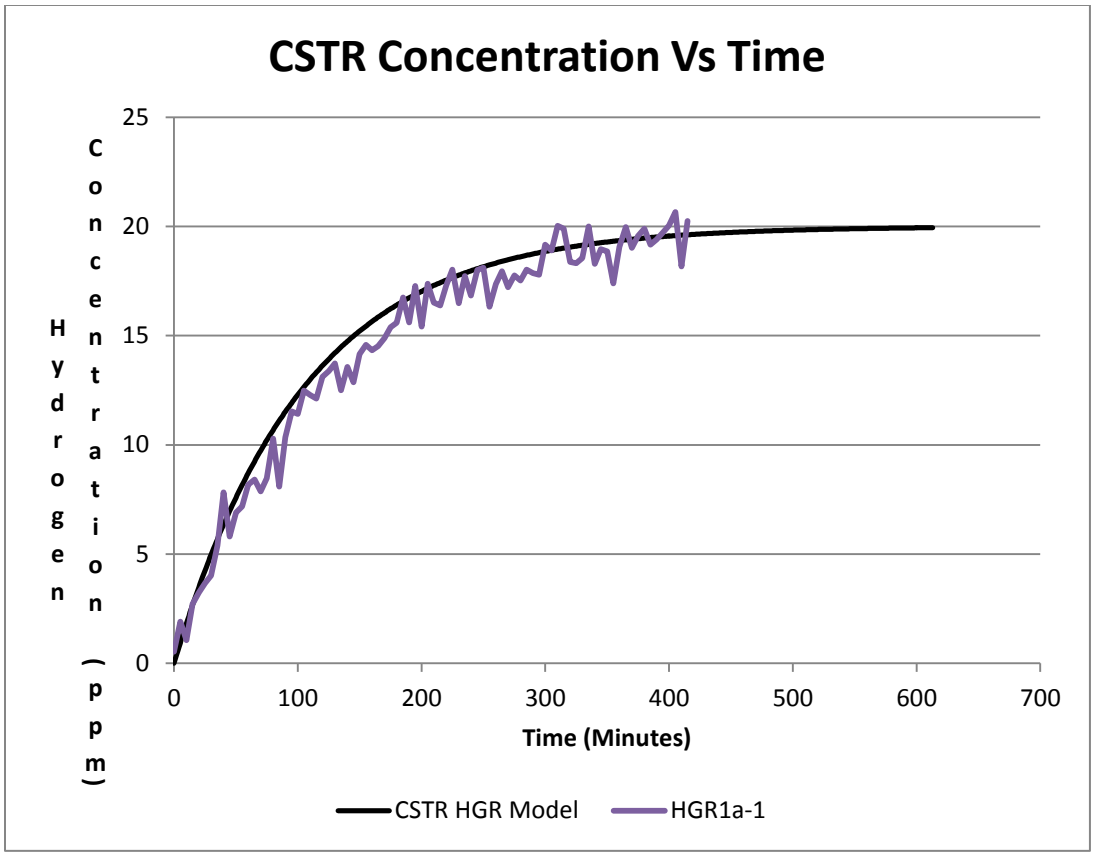


Figure B-2. HGR1a-2 Results Overlaid with CSTR Model

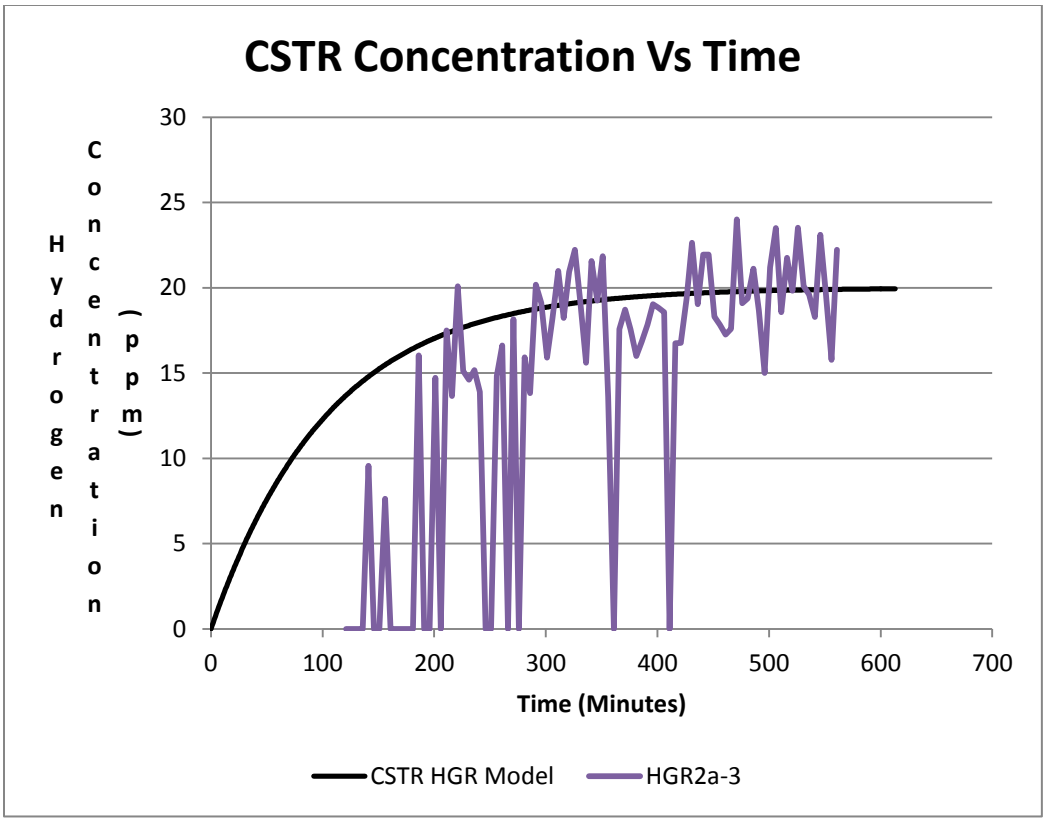


Figure B-3. HGR1a-3 Results Overlaid with CSTR Model

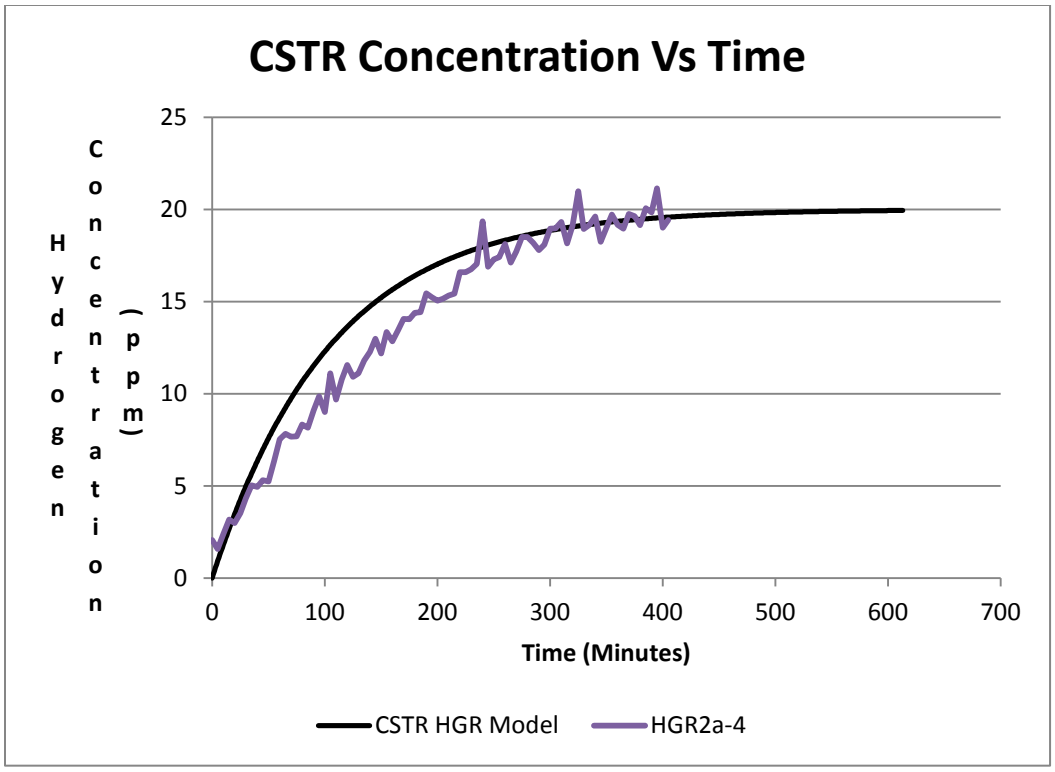


Figure B-4. HGR1a-4 Results Overlaid with CSTR Model

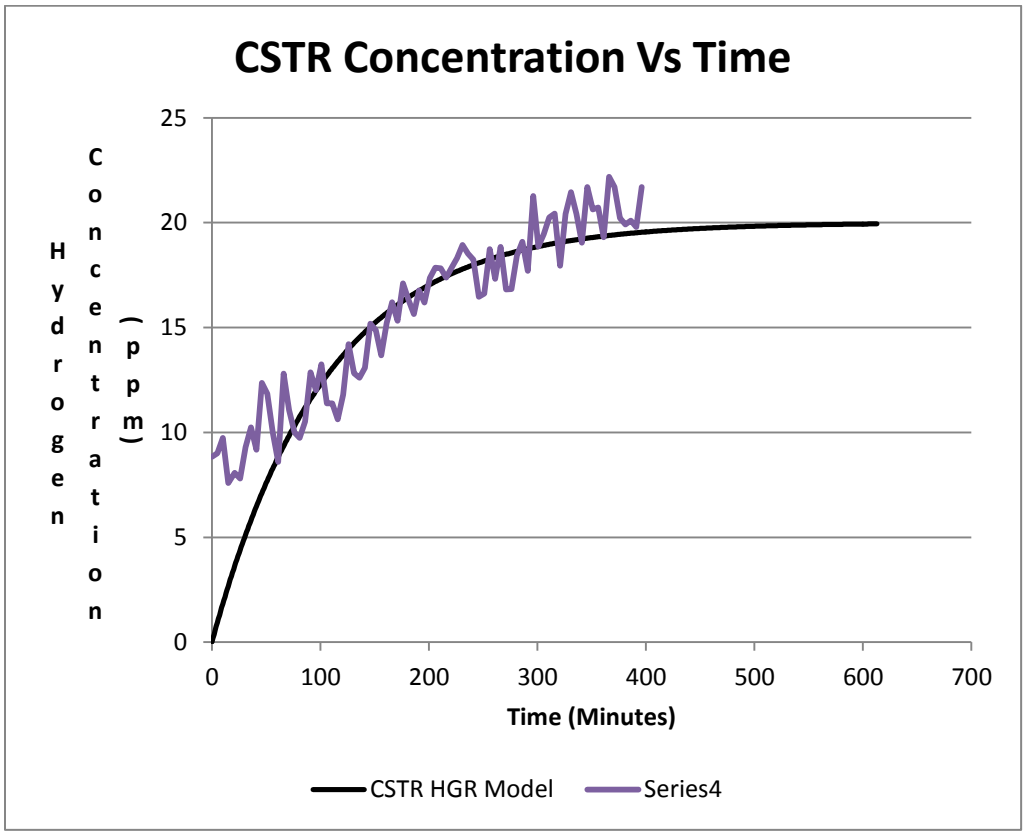


Figure B-5. HGR2a-1 Results Overlaid with CSTR Model

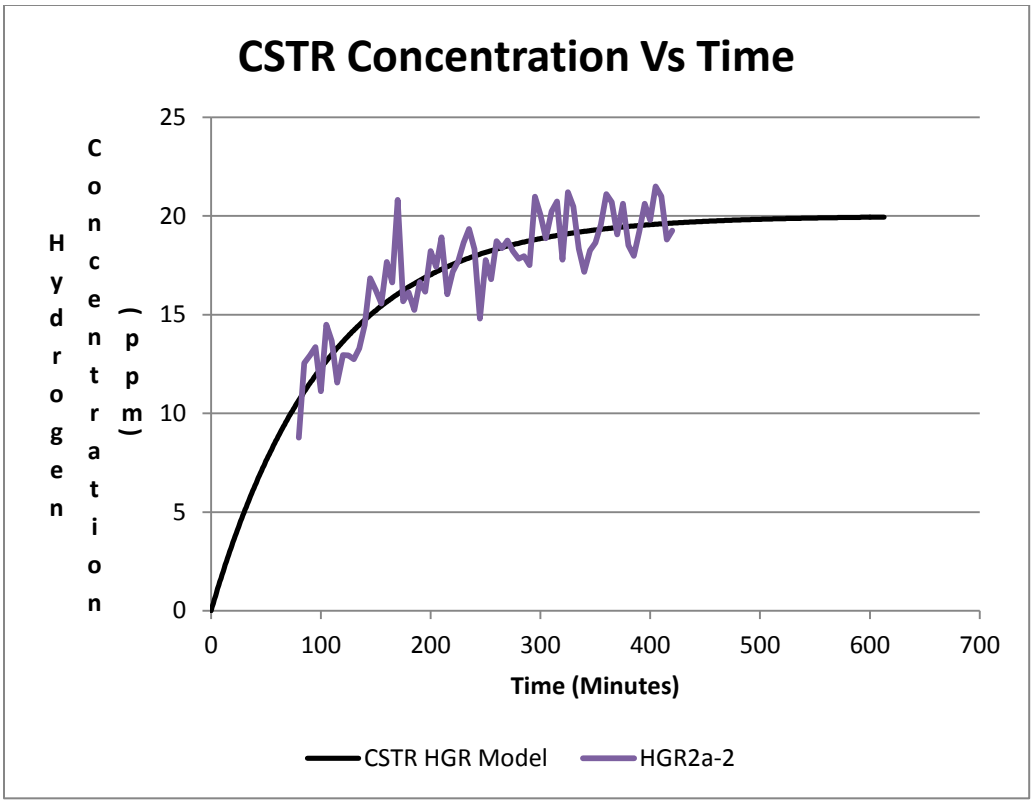


Figure B-6. HGR2a-2 Results Overlaid with CSTR Model

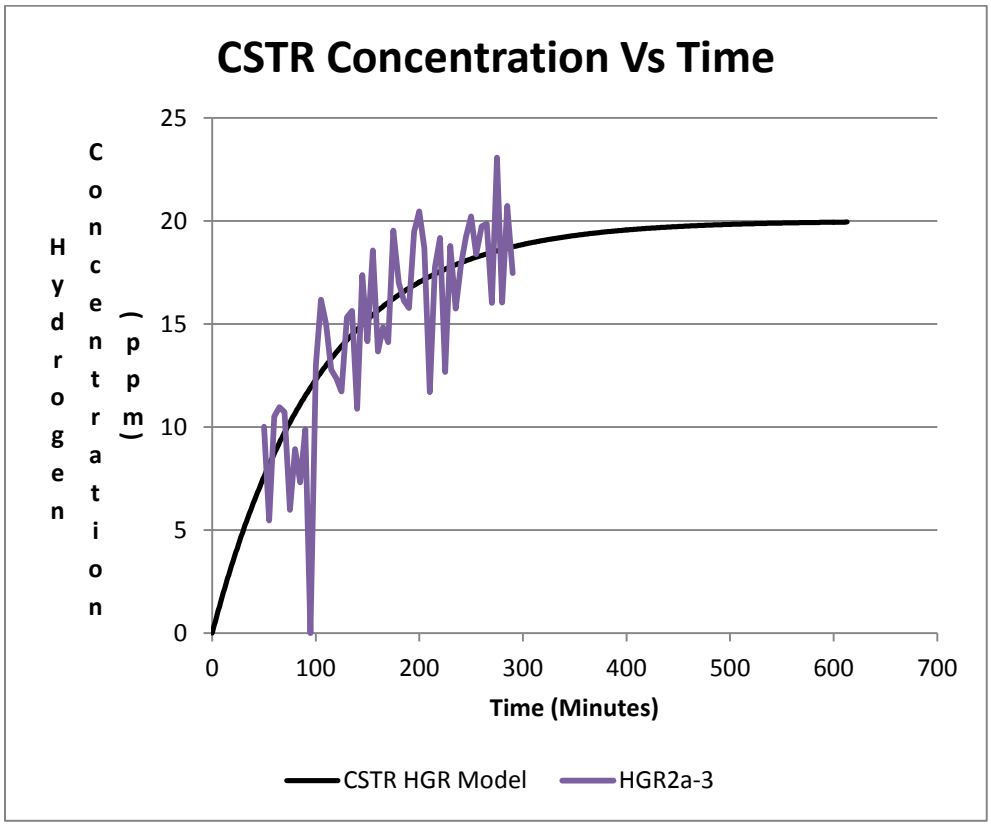


Figure B-7. HGR2a-3 Results Overlaid with CSTR Model

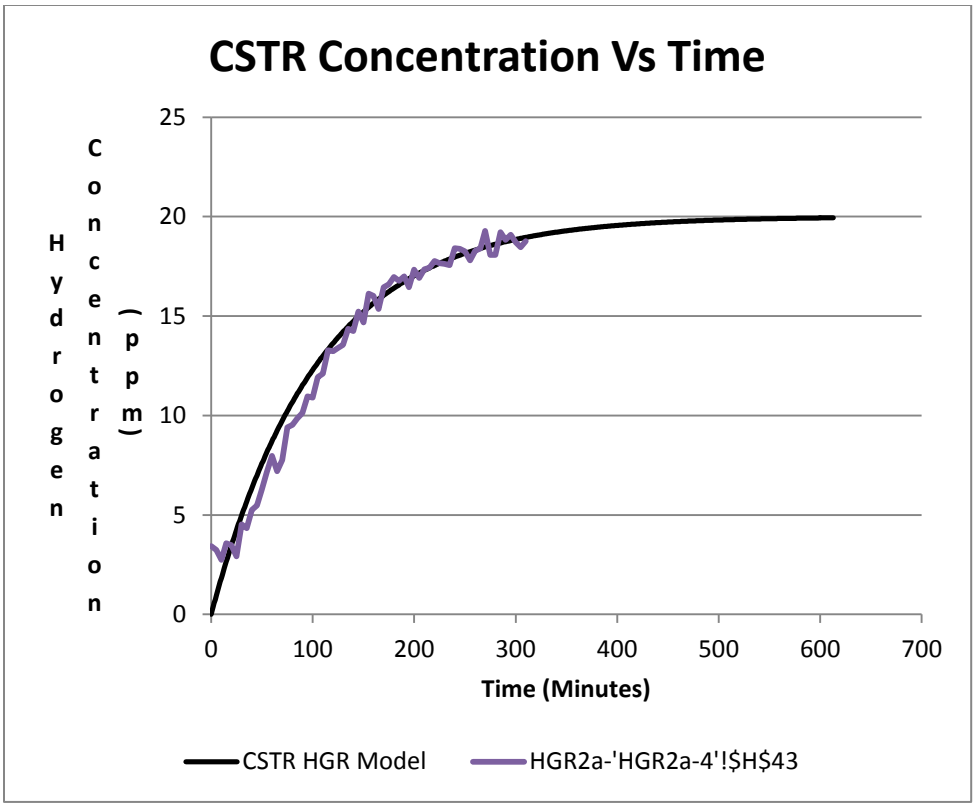


Figure B-8. HGR2a-4 Results Overlaid with CSTR Model

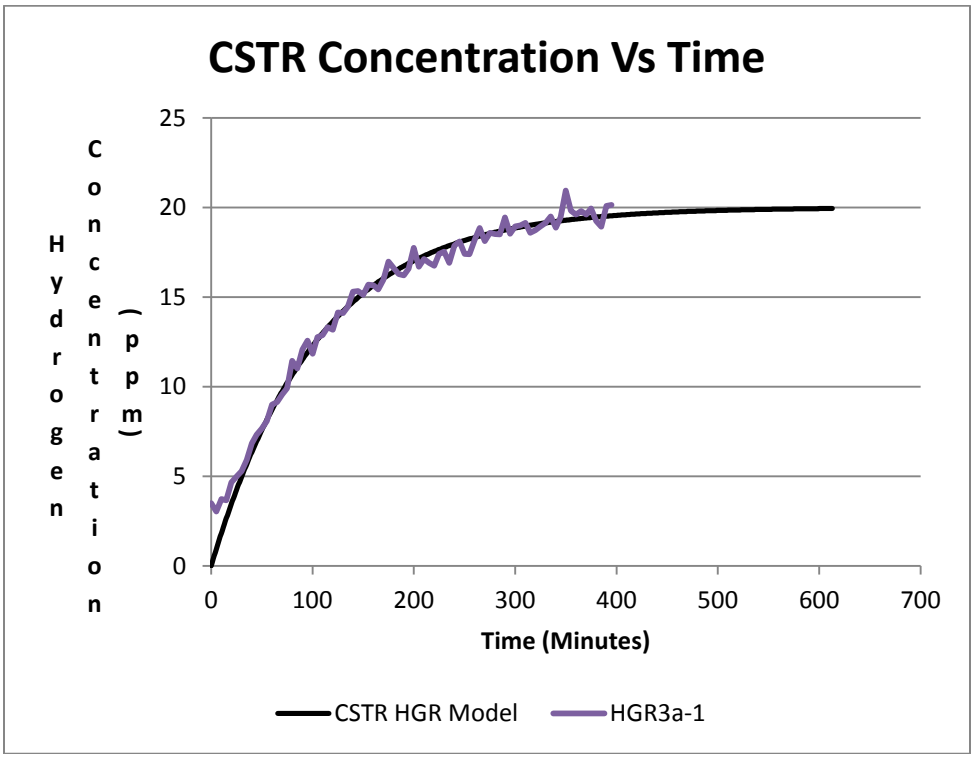


Figure B-9. HGR3a-1 Results Overlaid with CSTR Model

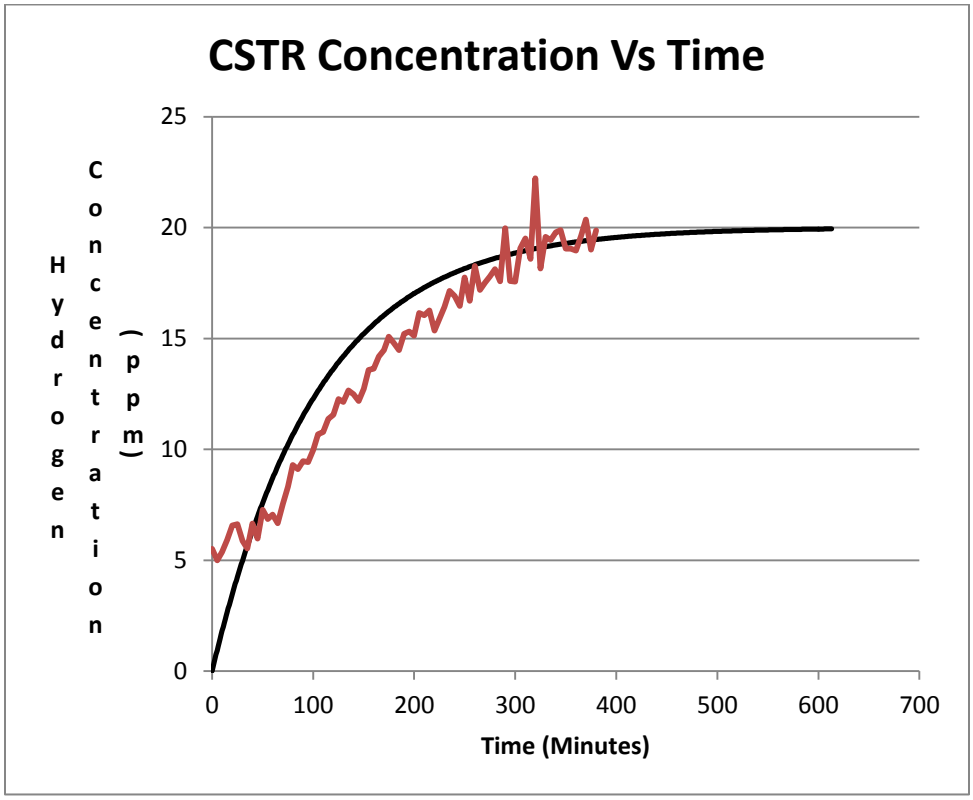


Figure B-10. HGR3a-2 Results Overlaid with CSTR Mode

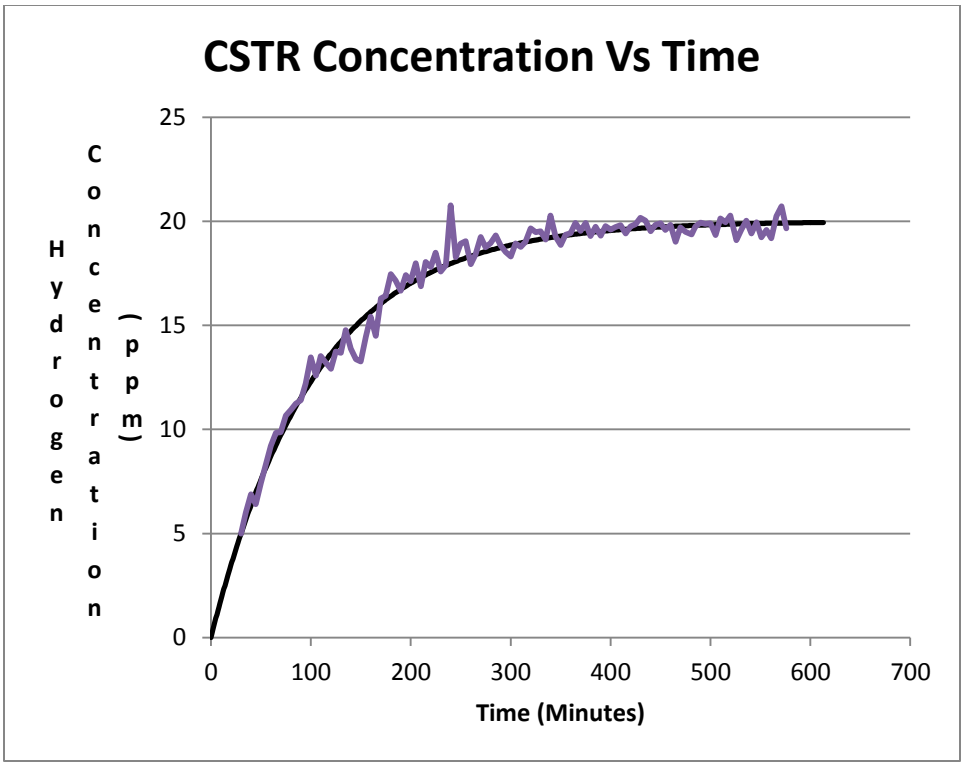


Figure B-11. HGR3a-3 Results Overlaid with CSTR Model

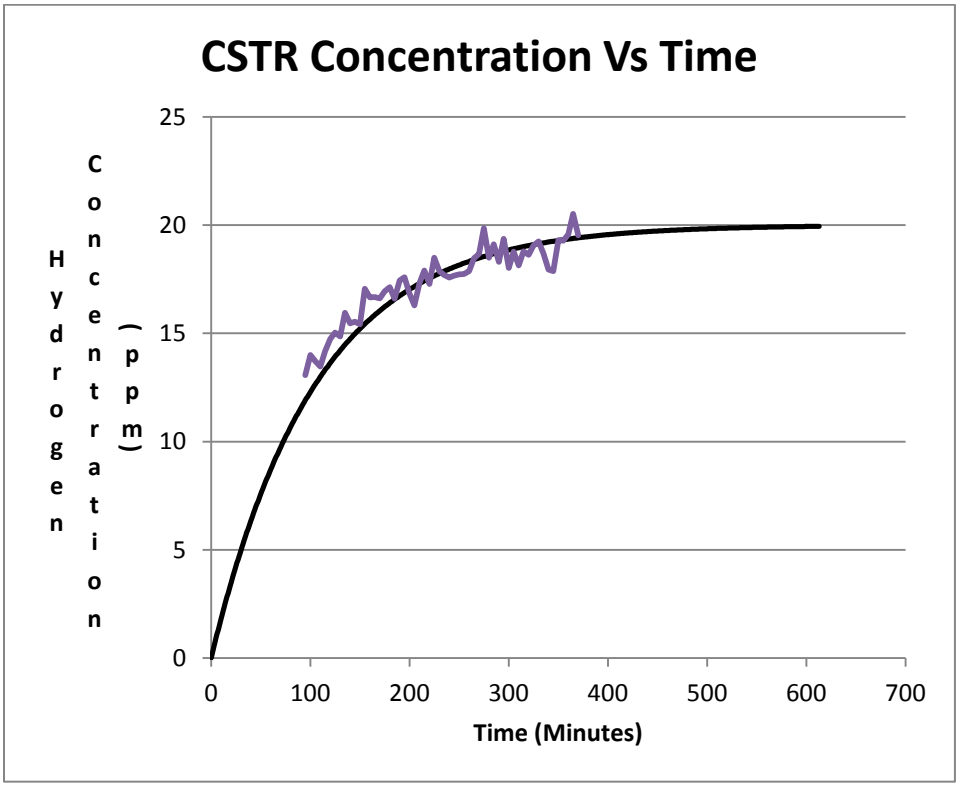


Figure B-12. HGR3a-4 Results Overlaid with CSTR Model

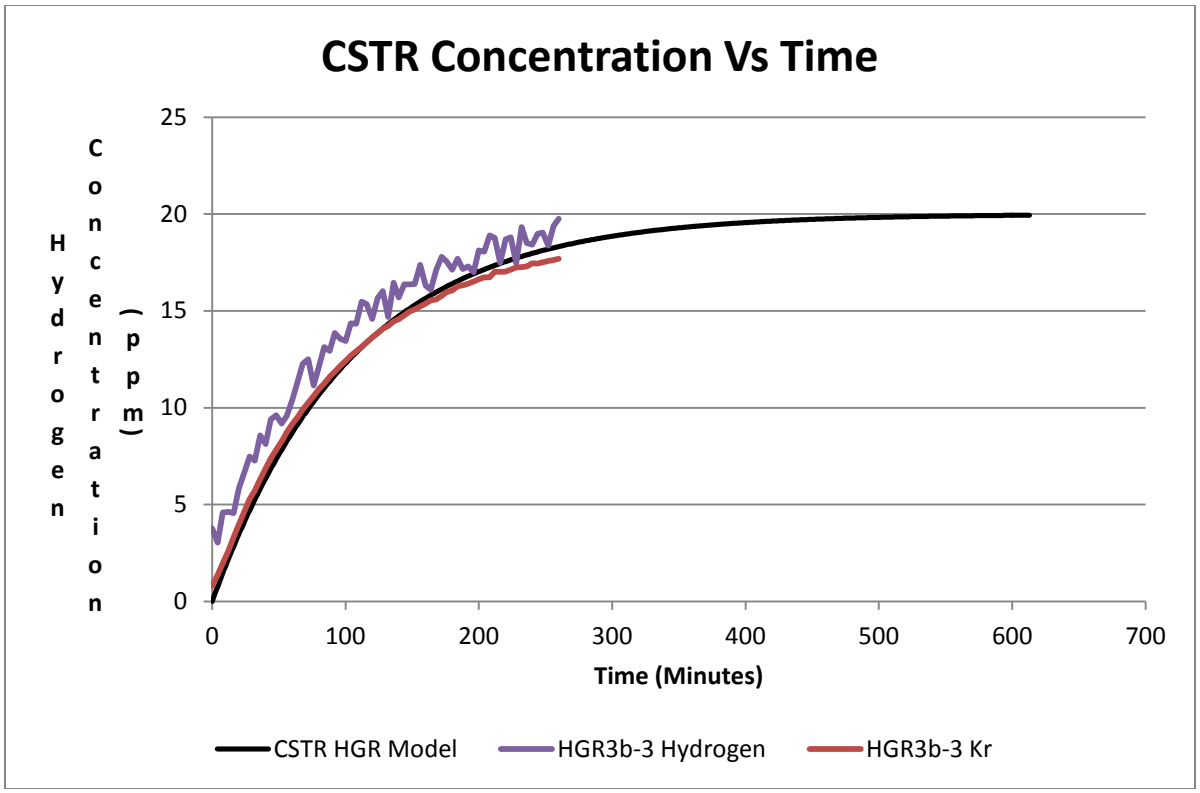


Figure B-13. HGR3b Testing with Tracer Gas

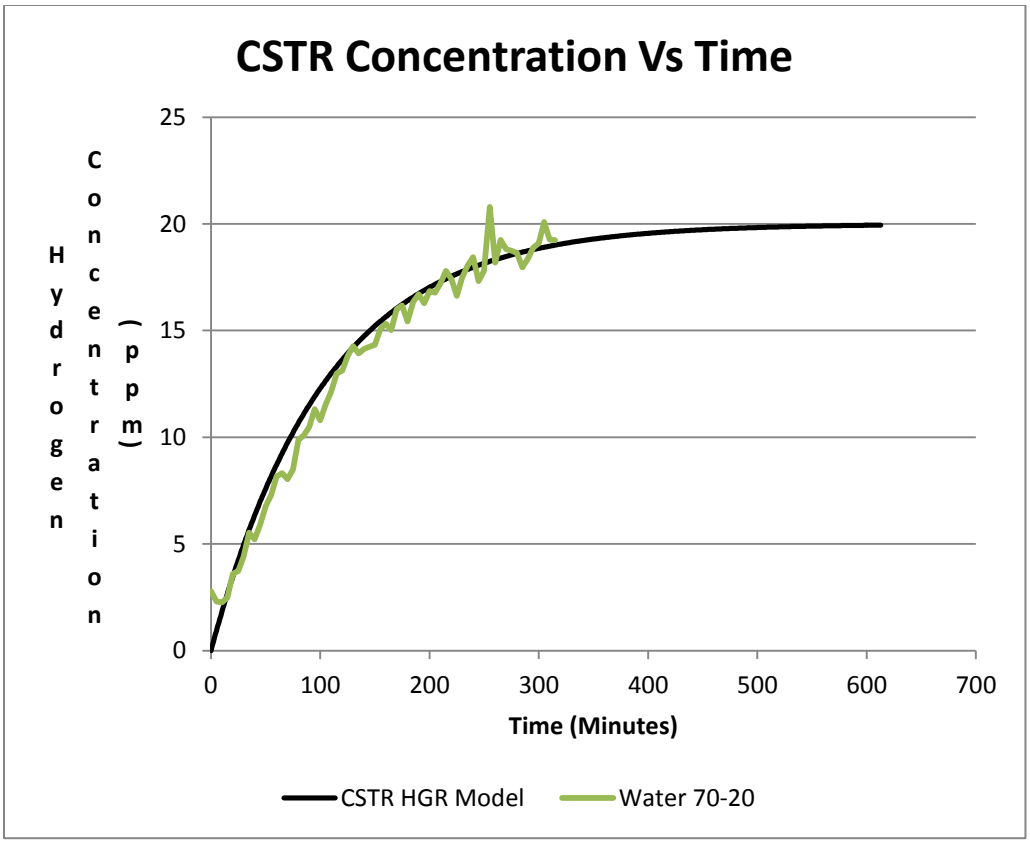


Figure B-14. Water Run 70-20 Results Overlaid with CSTR Model

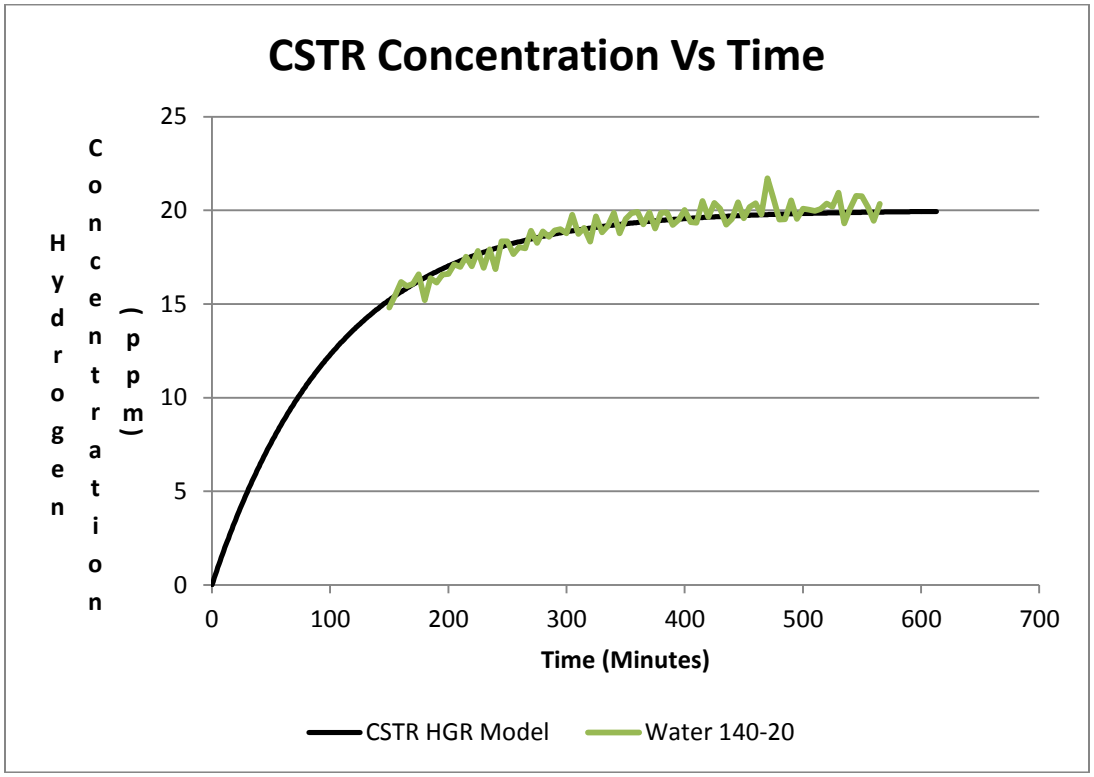


Figure B-15. Water Run 140-20 Results Overlaid with CSTR Model

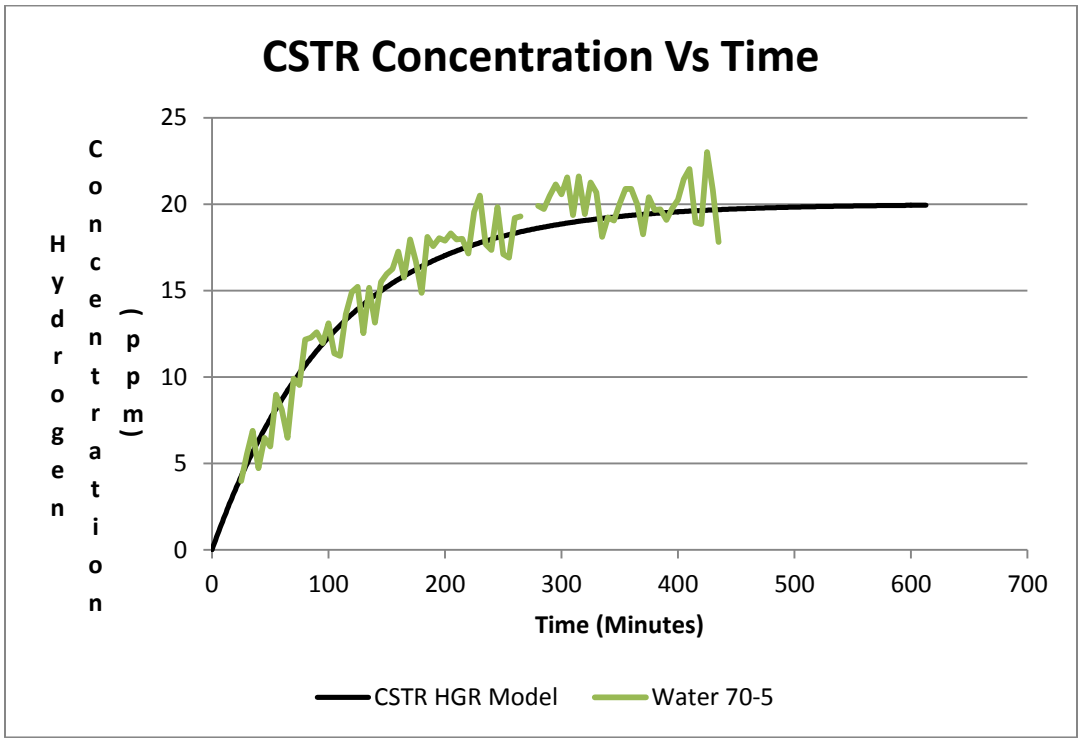


Figure B-16. Water Run 70-5 Results Overlaid with CSTR Model

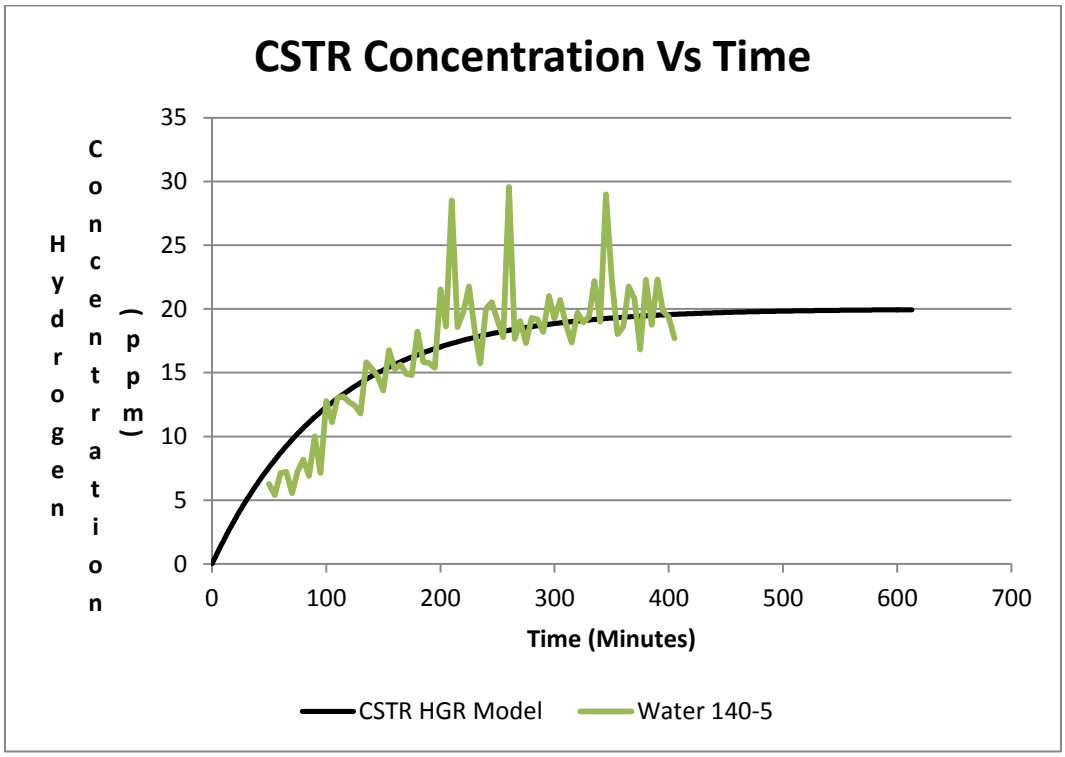


Figure B-17. Water Run 140-5 Results Overlaid with CSTR Model

Appendix C. HGRMA Assembly Instructions

The HGRMA is a small reaction vessel and offgas system designed to remotely measure the hydrogen generation rate of samples of Hanford tank waste. Guidance for assembling the apparatus includes instructions and photographs of the prototype apparatus. The instructions were demonstrated in the SRNL Shielded Cells during mockup of the apparatus in the SRNL Shielded Cells Mockup facility.

APPENDIX C – CONTENTS

C.1 Introduction C-4

C-1.2 Magnetic Coupling C-6

C-1.3 Vessel Head Installation C-8

C-1.4 Lower Agitator Shaft C-10

C-1.5 Vessel Installation C-10

C-1.6 Vessel Head Clamp Installation C-12

C-1.7 Air Purge Line Connections C-12

C-1.8 Condenser Installation C-14

C-1.9 Condenser Chilled Air RTD Installation C-15

C-1.10 Vortex Chiller Installation C-16

C-1.11 Vortex Chiller Rotameter C-17

C-1.12 Manometer C-18

C-1.13 Addition Funnel C-19

C-1.14 Vessel RTD C-19

C-1.15 Bubbler C-19

C-1.16 Vent Gas Filter and Septum C-21

C-1.17 Water Bath Connections C-22

C-1.18 Mixer Head Installation C-23

C-1.19 Mixer Shaft Installation C-24

C-1.20 Wiring Connections C-26

C-1.21 O-ring Installation Tool C-26

C-1.22 Markings C-27

C-1.23 Completion C-27

APPENDIX C – LIST OF FIGURES

Figure C-1. HGR Stand C-5
Figure C-2. Containment Pan C-5
Figure C-3. Installation of Magnetic Coupling and Upper Agitator Shaft C-6
Figure C-4. Magnetic Coupling Support C-7
Figure C-5. Vessel Head C-8
Figure C-6. Vessel Head Installation Support C-9
Figure C-7. Lower Agitator Shaft and Detail of Threads C-10
Figure C-8. Vessel Installation C-11
Figure C-9. Vessel Support C-12
Figure C-10. Air Purge Line Connections C-13
Figure C-11. Air Purge Line Connection Mounted to Stand and CPC PMC12 Series Quick-Connect C-13
Figure C-12. Condenser Installation C-14
Figure C-13. Condenser Chiller Air Outlet RTD Installation C-15
Figure C-14. Vortex Chiller C-16
Figure C-15. Vortex Chiller Rotameter C-17
Figure C-16. Manometer Assembly C-18
Figure C-17. Ace-Thred Diagram C-19
Figure C-18. Bubbler Support and Bubbler C-20
Figure C-19. Filter and Septum C-21
Figure C-20. Water Bath and Connections C-22
Figure C-21. Agitator Holder C-23
Figure C-22. Flexible Shaft with Cable End Collar Installed C-24
Figure C-23. Flexible Shaft Support C-25
Figure C-24. Agitator Wiring Amphenol Connectors, with clamp installed C-26
Figure C-25. O-ring Installation Tool and O-ring Removal Tool C-26
Figure C-26. Markings on Threaded Parts and Ace-Thred after Optional Machining C-27
Figure C-27. Assembled HGR Measurement Apparatus, Side View C-28
Figure C-28. Assembled HGR Measurement Apparatus, Front View C-29

C.1 Introduction

The HGR measurement apparatus is a small, water jacketed vessel with internal mixing, air purge, and an offgas measurement system to detect radiolytic and thermal hydrogen generation from samples. The apparatus has been designed for remote operation in a shielded cell system, with selected components outside the cell. The assembly of this apparatus should be performed as specified to ensure that the apparatus is assembled in the cell in the same manner each time to prevent variability in the results from variations in the apparatus assembly.

The assembly instructions were developed based on initial assembly of the prototype, then verified by testing in the mockup cell in the SRNL Shielded Cells facility. This testing demonstrated that the equipment could be installed and removed in a shielded cell as described in this document.

The order of the steps performed does not typically impact the assembly; specific steps will include order-specific instructions when the order of steps is important to the assembly process. Heavy components, such as the agitator head and magnetic coupling should be installed first to minimize the risk of breaking glassware.

Tear-down of the HGRMA follows the same steps listed here, but in reverse order and by performing the opposite actions listed (e.g. disconnect versus connect). As during assembly, the order of the steps performed is not required to match the order listed in these instructions unless specifically stated in the instructions. However, heavy components should typically be removed last to minimize the risk of breaking glassware.

Once assembled, the HGR measurement apparatus weighs approximately 55 pounds while the water bath weighs approximately 42 pounds. Both of these values represent the empty weight.

C-1.1.1 Support Stand Inspection and Adjustment

The support stand is shown in Figure C-1and typically does not need adjustment. The stand should be inspected before use to ensure that the adjustment knobs are tight and that the lift screw operates smoothly. Adjustments to the stand can be made by loosening the appropriate knob and moving the piece needing adjustment; this adjustment is best performed with the glassware removed. It is recommended that the stand support rods be marked with the positions of the stand prior to moving any piece to allow the piece to be returned to its prior position if needed.

Removal of the stand components is possible in a remote setting; the supports for the vessel, magnetic coupling, and flexible shaft will lift off if the adjustment knobs are loosened. Support rods simply unscrew from the base, but a wrench will be required as the rods are installed tightly. A t-handled hex tool (3/16 inch) can be used to remove and reinstall the mixer head support.

Clamps are pre-installed on the stand to support or brace selected components, as shown in Figure C-1. Typically, the exact location of these clamps is not as important as flexible lines are used to connect the components.

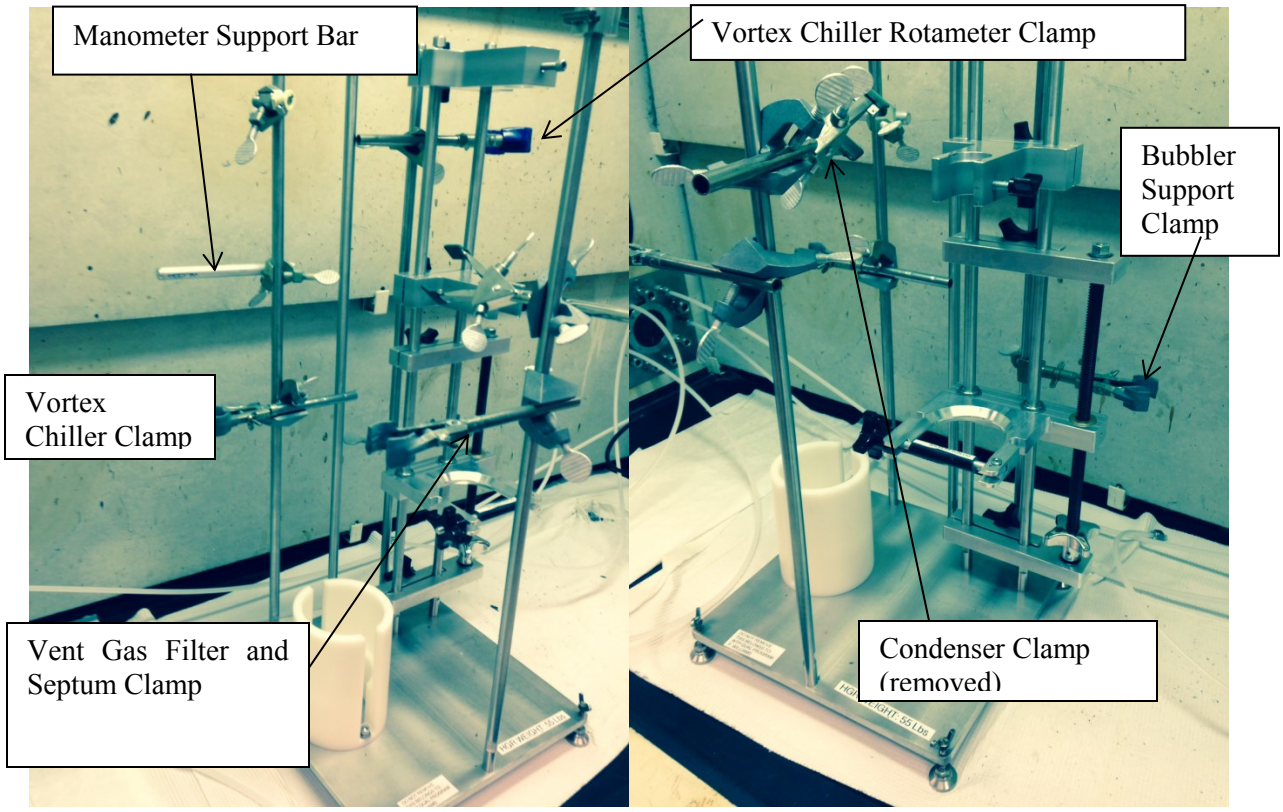


Figure C-1. HGR Stand

C-1.1.2 Containment Pan

The containment pan, shown in Figure C-2,is custom fit to the support stand and should be placed under the stand prior to assembly of any components on the stand. The manipulator grip tabs on the containment pan should face the front of the apparatus. Once the stand is place in the pan, the entire apparatus can be moved into different positions using the grip tabs on the front of the stand.

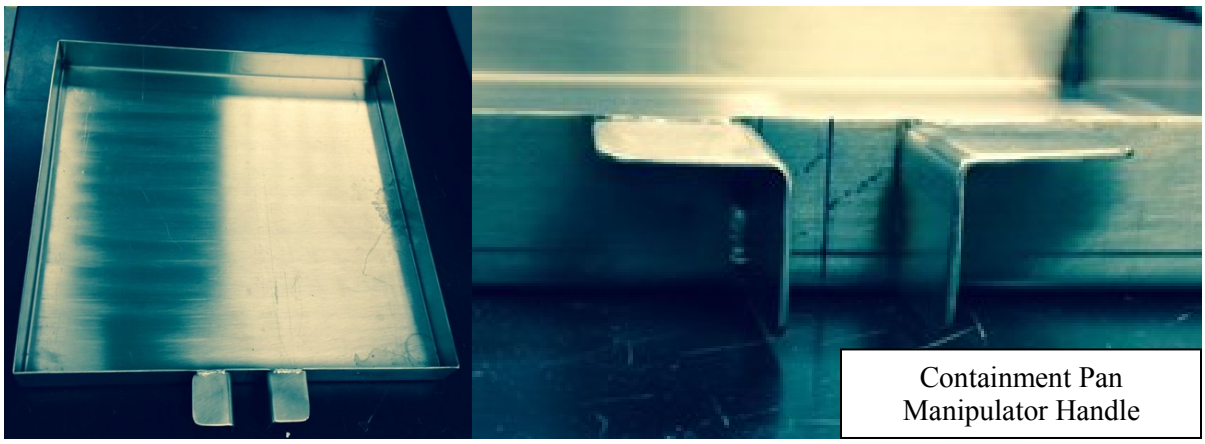


Figure C-2. Containment Pan

C-1.2 Magnetic Coupling

The Parr magnetic coupling must be installed prior to installing the lid or vessel. The coupling should have the upper agitator shaft assembly installed prior to placing the coupling in the stand. This assembly is shown in Figure C-3and consists of a short 3/8 inch shaft screwed into the top of the coupling. A flexible coupler is installed on the top of this shaft. This coupler should have the socket head installed prior to placing the coupler on the magnetic coupling upper shaft. The magnetic coupler installs into the support shown in Figure C-3 and the adjustment knob is tightened to lock the coupling in place.

If not pre-installed, then the Ace-Thread Parr Coupling Adapter should be installed on the bottom threads of the magnetic coupler and o-rings installed as shown in Figure C-4.

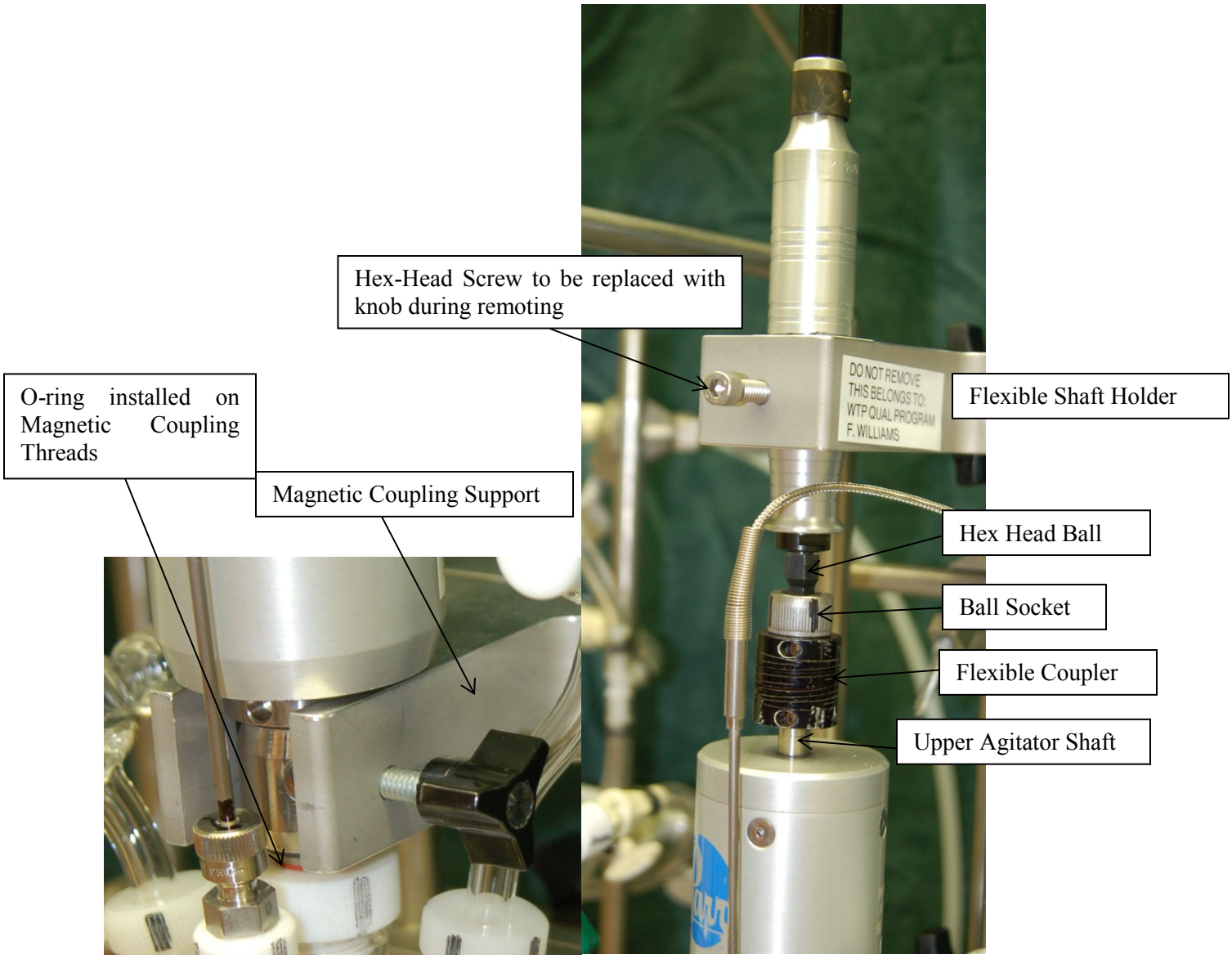


Figure C-3. Installation of Magnetic Coupling and Upper Agitator Shaft

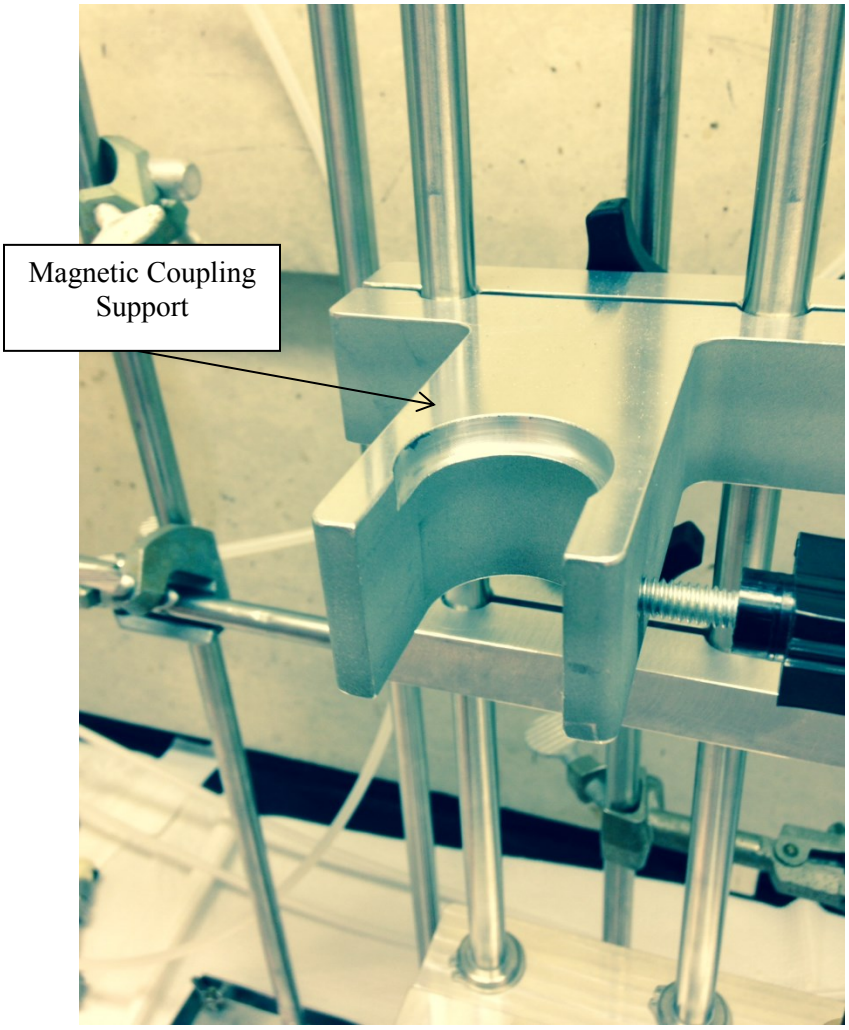


Figure C-4. Magnetic Coupling Support

C-1.3 Vessel Head Installation

The vessel head should be inspected for cracks or chips prior to use. The vessel head is custom fabricated glassware containing 5 ports. The center port threads onto the Parr Coupling adapter as shown in Figure C-5. The coupler/head assembly should be turned so that the RTD port is facing the front of the apparatus. The addition funnel will contact the knob on the magnetic coupling support unless the vessel head is positioned correctly. The head can be rotated after installation by loosening the magnetic coupling support knob and rotating the head.

A temporary support has been fabricated that installs onto the vessel support to hold the head during installation, as shown in Figure C-6. This support aligns the lid with the magnetic coupling and greatly simplifies installation of the vessel head. This holder should also be used when removing the head to minimize the risk of dropping the head.

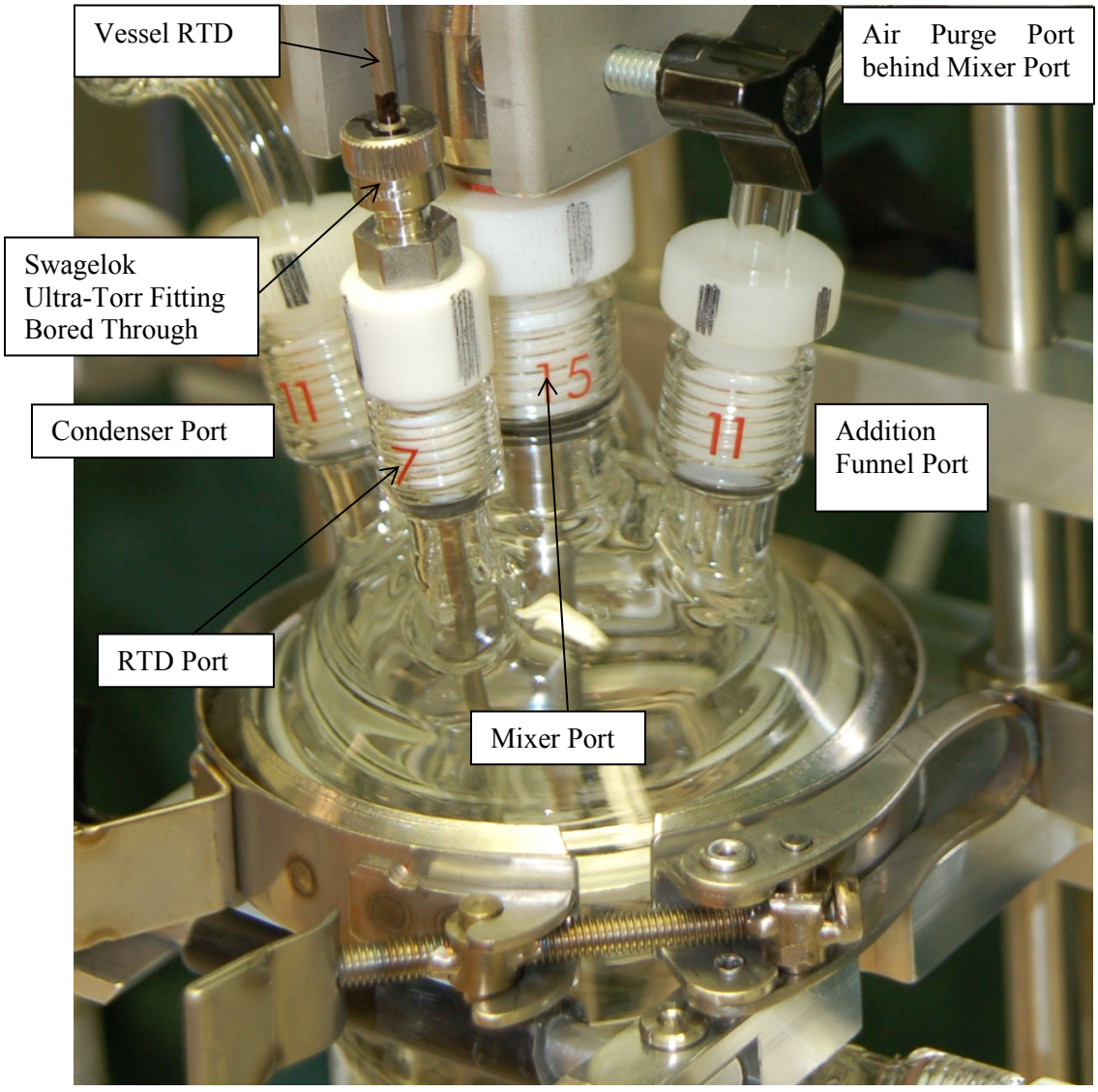


Figure C-5. Vessel Head

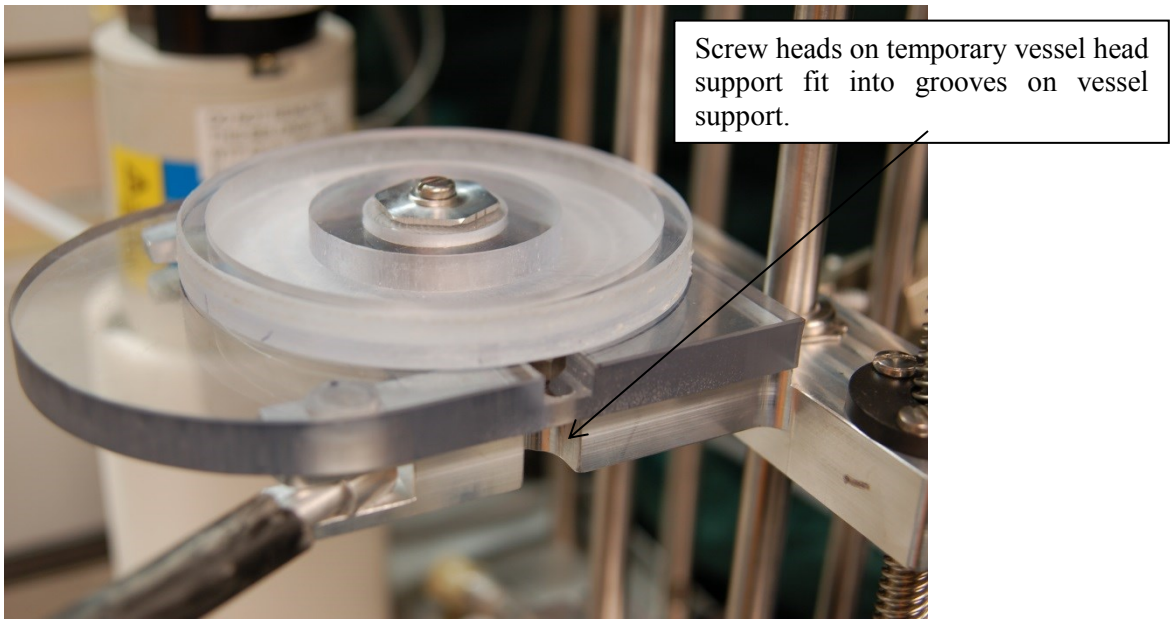


Figure C-6. Vessel Head Installation Support

C-1.4 Lower Agitator Shaft

The lower shaft with impeller must be installed prior to installing the vessel. The shaft threads into the center of the magnetic coupling through the vessel head as shown in Figure C-7, but it should be noted that the threads are left-handed threads to prevent loosening during operation. The shaft must be securely tightened to prevent loosening during the test run. The impeller is installed on the end of the shaft with a set screw; this screw must also be securely tightened to prevent loosening during the test. The shaft can be installed by holding the shaft in place and rotating the Parr magnetic coupling and then secured by holding the coupling and using the impeller as a handle to turn the shaft.

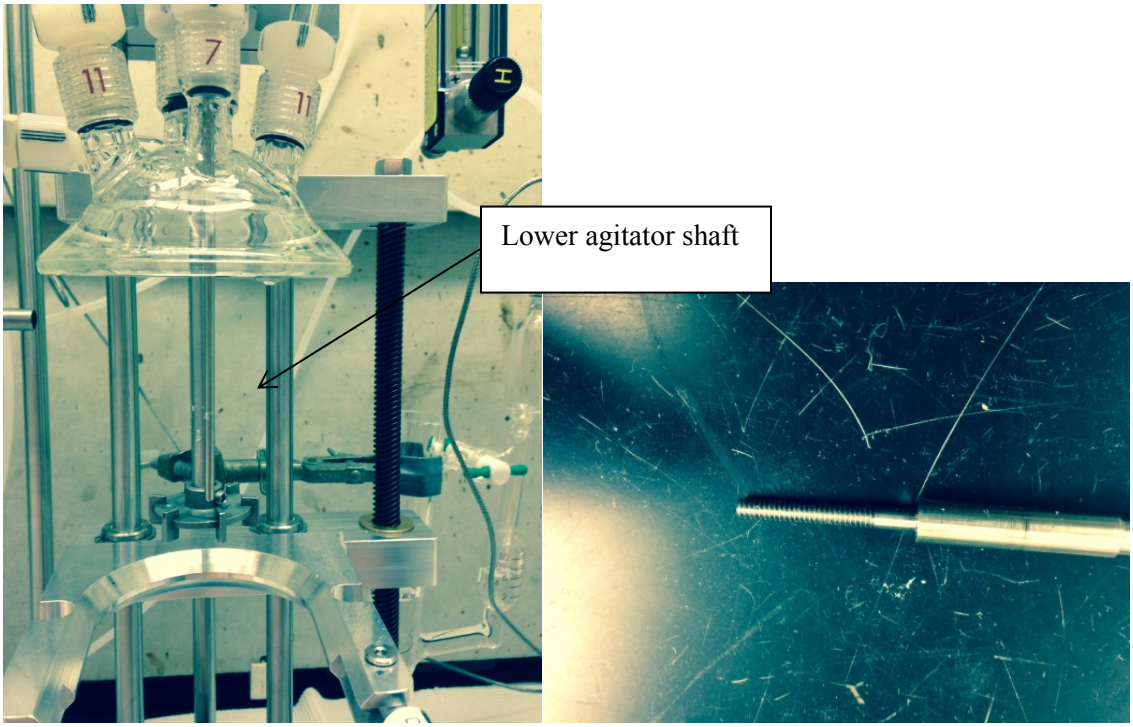


Figure C-7. Lower Agitator Shaft and Detail of Threads

C-1.5 Vessel Installation

The vessel should be inspected for cracks or chips prior to use. The vessel o-ring should be inspected and replaced if worn. A light coating of vacuum grease should be applied to the new o-ring prior to entry into the cells if the o-ring is replaced. The grease can be applied in the cells using a Q-tip (or similar) to first spread grease on the vessel groove prior to installing the o-ring. After the o-ring is installed, the Q-tip is used to spread grease on the upper surface of the o-ring. Dow Corning high vacuum grease is a silicone based grease typically used at SRNL for this purpose.

The vessel lift should be lowered to allow the vessel to clear the lower agitator shaft during installation, as shown above in Figure C-7. The vessel must be installed with the water bath connections facing to the right as viewed from the front of the apparatus, as shown in Figure C-8. The vessel support arm is loosened with the adjustment knob and the arm swings to the right to allow the vessel to be placed on the stand, as shown in Figure C-8. The vessel is placed in the support and the arm retightened to securely hold the vessel in place. The support arm has a rubber tube protecting the glass ware from metal to glass contact. Some of the ridges on the vessel drain valve handle may be scraped off to allow the manipulator to grip the handle.

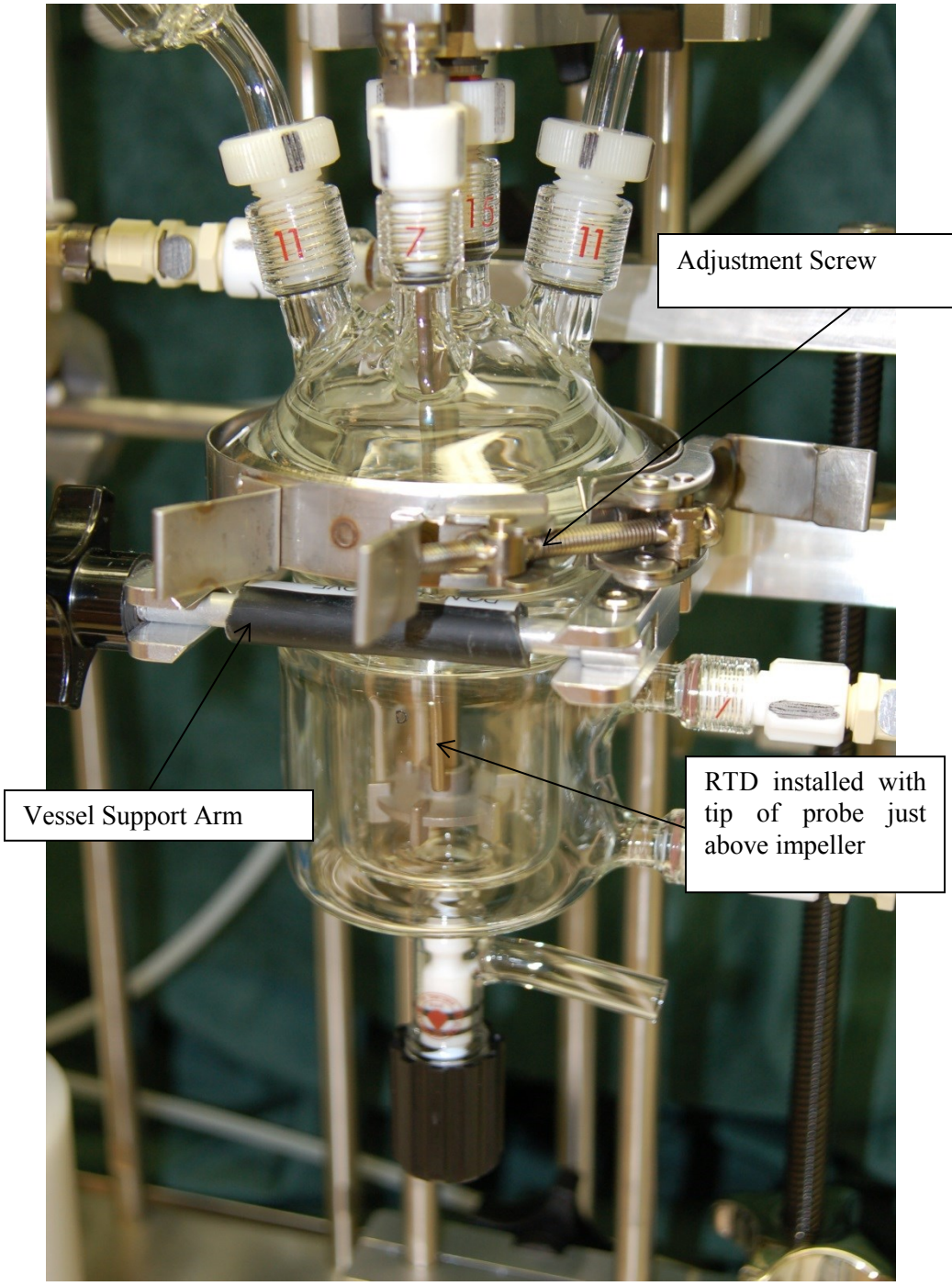


Figure C-8. Vessel Installation

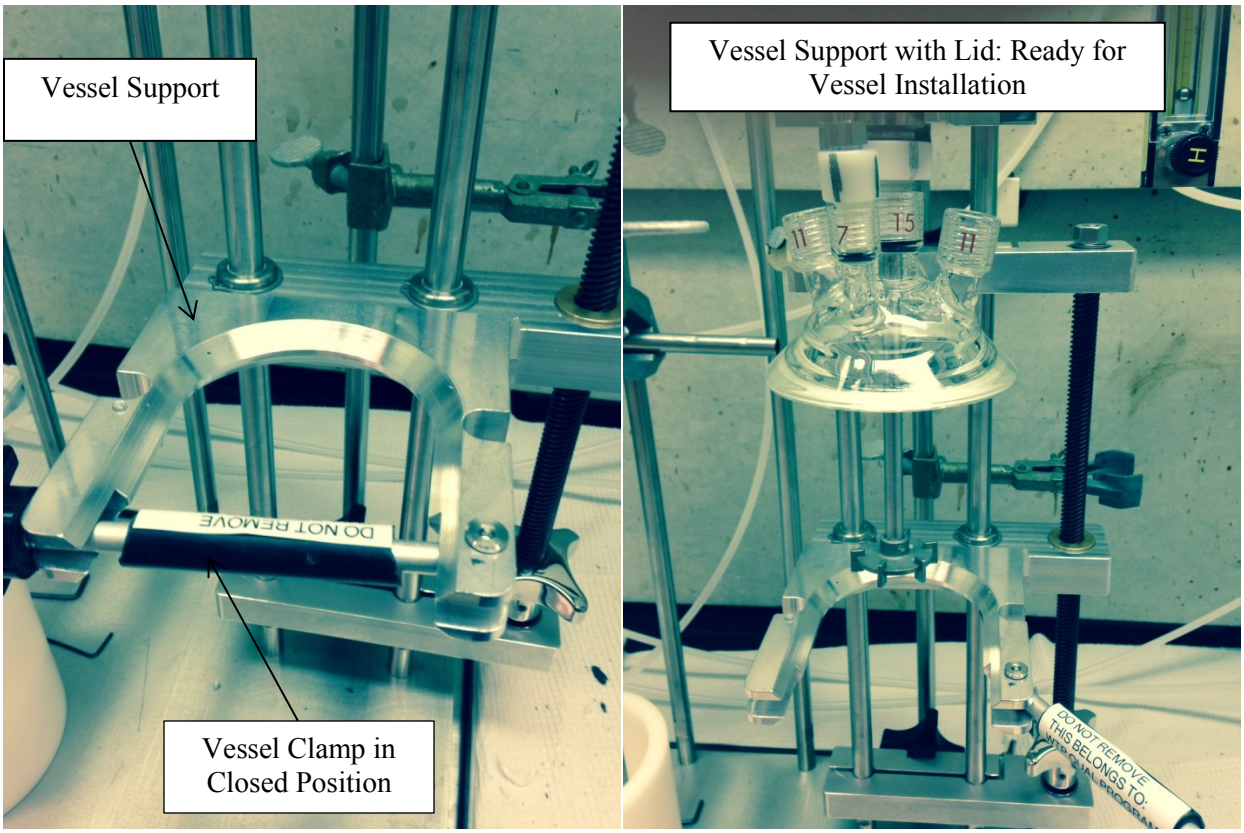


Figure C-9. Vessel Support

C-1.6 Vessel Head Clamp Installation

The vessel head clamp has three tabs to allow remote manipulation, as shown above in Figure C-9. The clamp should be placed on top of the vessel, then the vessel lift is used to raise the vessel to the vessel head. The clamp may need to be adjusted to allow the vessel to be completely raised to the head. Once the vessel is raised, the clamp is adjusted to fit over the lid and vessel, then the clamp is closed to seal the vessel to the head. The clamp closes by first securing the adjustment screw onto the clamp, then pushing the clamp lever towards the back of the clamp. The adjustment screw has been tack welded in position and does not turn.

C-1.7 Air Purge Line Connections

The air purge line is in the rear of the apparatus, therefore it will be easier to reach and make connections if these lines are connected prior to installing components in the front of the apparatus. The air purge is connected to the vessel using an #7 Ace-Thread to 1/8" NPT adaptor, then a Colder Products Company (CPC) 1/8 inch quick connect, as shown in Figure C-10. A Tygon tubing harness consisting of a CPC tubing connector, ~12 inch of Tygon tubing, a polyethylene tubing tee, and two ~6 inch pieces of tubing also with CPC quick-connects is used to connect the vessel and manometer to the incoming purge air line. The Ace-thread adaptor with CPC quick connect should be installed on the vessel purge port, then the air line connection. The air line connection is simply a CPC quick connect mounted on the stand to allow the incoming purge air line to be connected, as shown in Figure C-11. Oetiker hose clamps are typically used at SRNL, as shown in Figure C-11, to secure the tubing to the fitting, but other types of hose clamps could also be used.

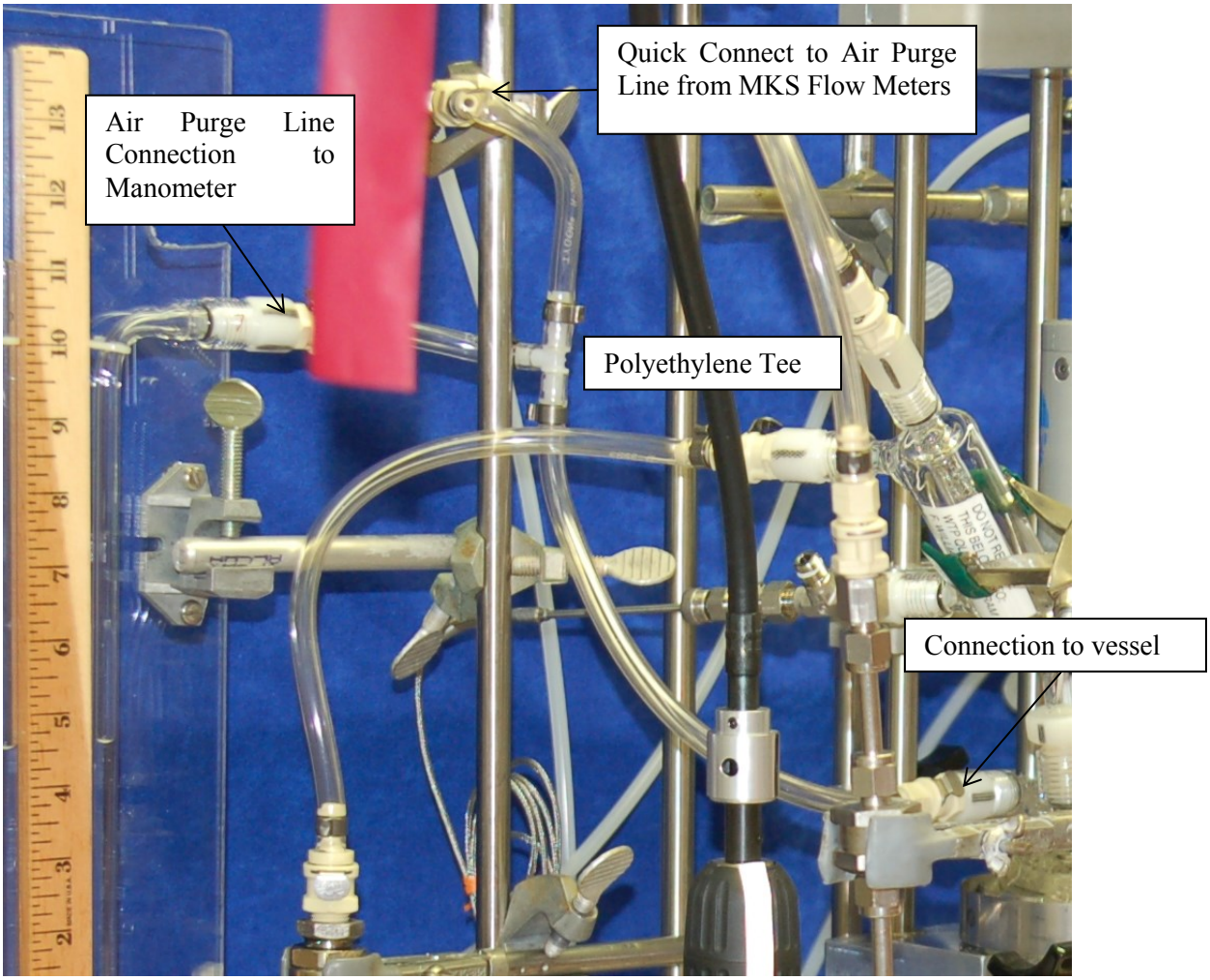


Figure C-10. Air Purge Line Connections

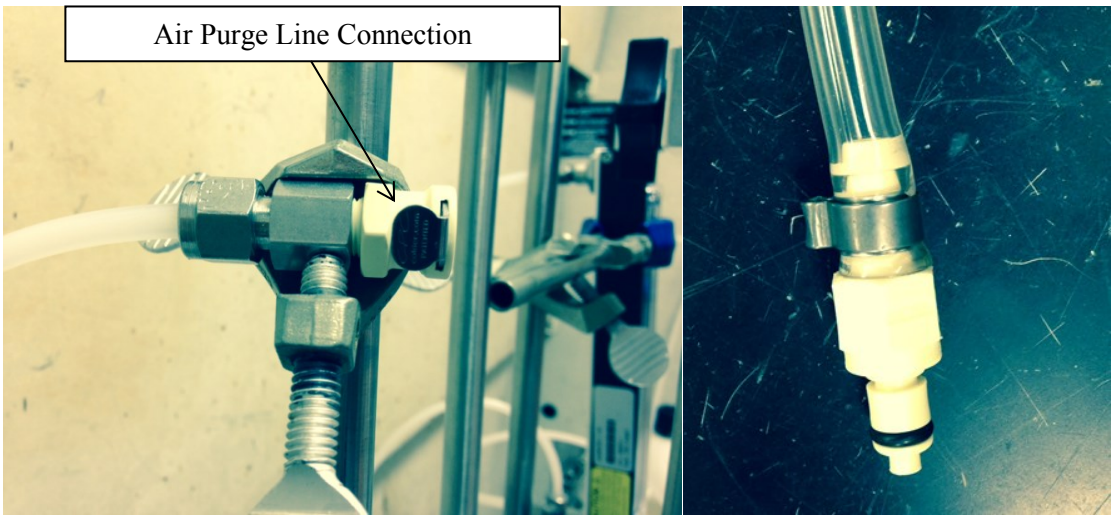


Figure C-11. Air Purge Line Connection Mounted to Stand and CPC PMC12 Series Quick-Connect

C-1.8 Condenser Installation

The custom fabricated glass condenser should be inspected for cracks or chips prior to use and should have demister mesh preinstalled prior to placing the condenser in the cells. The condenser connects to the vessel with an #11 Ace-thred and o-ring as shown in Figure C-12. The o-ring should be inspected and replaced if worn using the o-ring installation instructions below. The condenser is installed on the vessel by tightening the #11 Ace-thred until snug. The condenser is not clamped to the stand to prevent breaking the inlet tubing; the clamp shown in Figure C-12 has been removed. The condenser chilled air outlet RTD is not yet installed in this photograph.

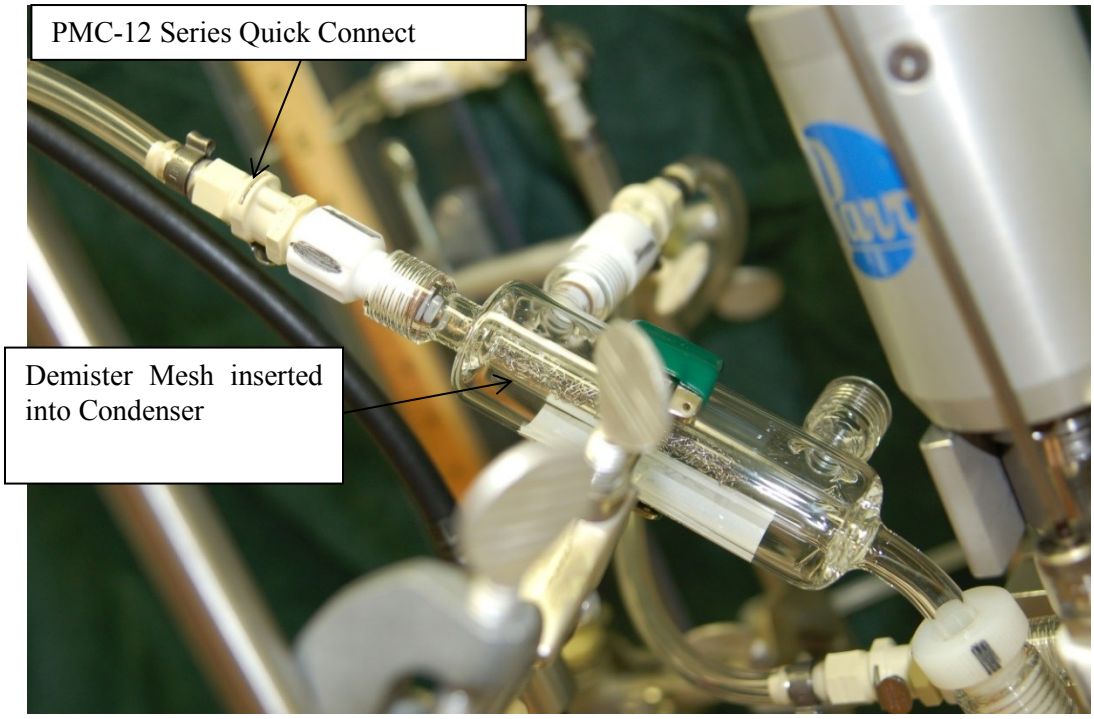


Figure C-12. Condenser Installation

C-1.9 Condenser Chilled Air RTD Installation

This RTD is installed using and Ace-Thred adaptor and a Swagelok Ultra-Torr fitting as shown in Figure C-13. The RTD should be inserted until the tip is just inside the condenser; avoid touching the inner tube with the RTD.

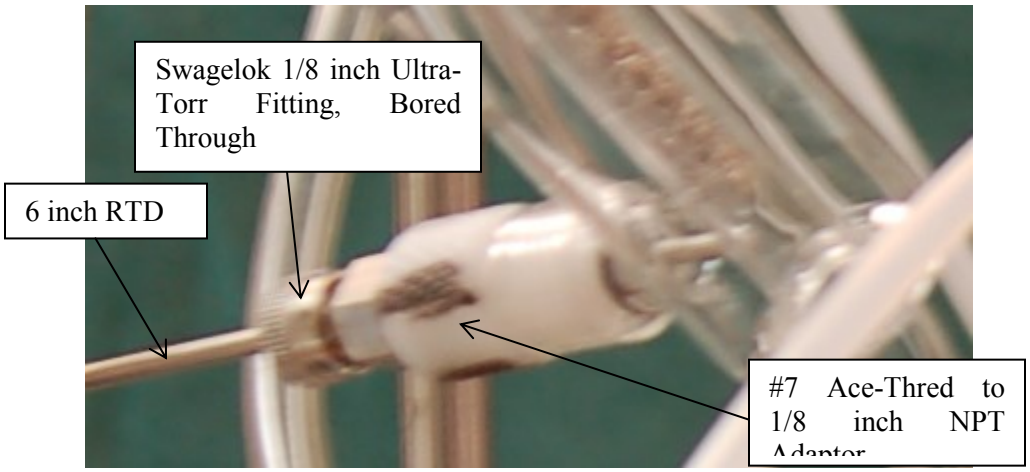


Figure C-13. Condenser Chiller Air Outlet RTD Installation

C-1.10 Vortex Chiller Installation

The vortex chiller has the required fittings pre-installed prior to entry into the cells. These fittings convert the National Pipe Thread (NPT) pipe threads on the air inlet to ¼ inch Swagelok and the cold air outlet to a ¼ inch CPC fitting. The threaded fittings should be sealed with NEOLUBE thread lubricant. The inlet air line is connected using a quick connect. The cold air outlet line is a 12 inch piece of Tygon tubing with CPC fittings and simply snaps into place. Installation of the vortex chiller is shown in Figure C-14, the air inlet is hidden behind the support clamp.

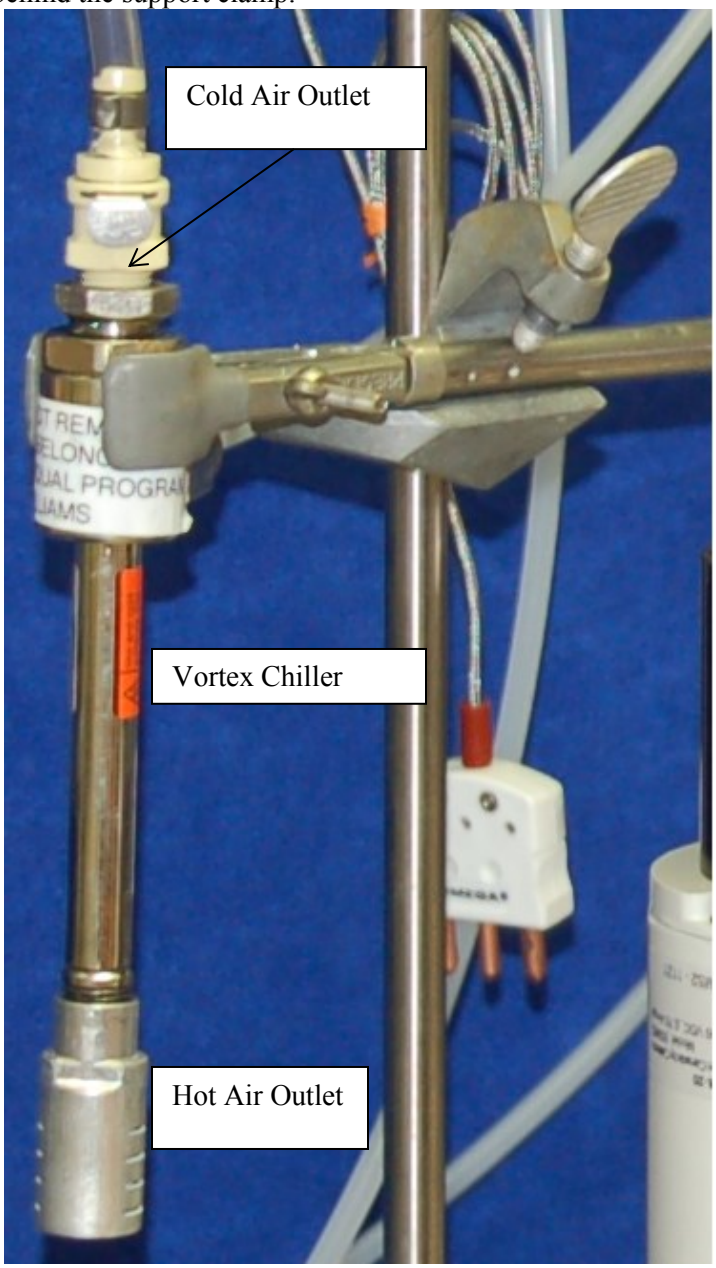


Figure C-14. Vortex Chiller

C-1.11 Vortex Chiller Rotameter

The rotameter is clamped to the stand being careful to ensure that the rotameter is installed vertically, as shown in Figure C-15. ¼ inch Swagelok fittings should be pre-installed on the rotameter using NEOLUBE thread lubricant prior to placing the rotameter in the cells. The inlet and outlet air lines are then connected with ¼ inch Swagelok fittings.

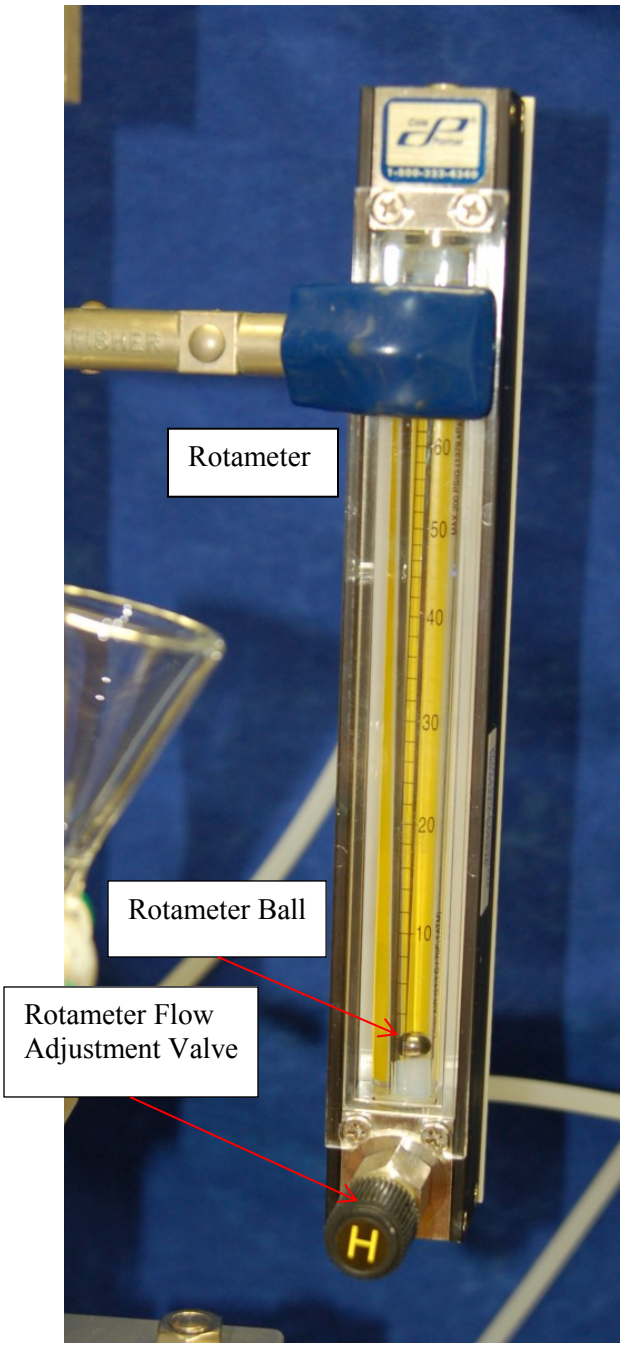


Figure C-15. Vortex Chiller Rotameter

C-1.12 Manometer

The manometer should be preassembled prior to entry into the cell to consist of the glass manometer, Plexiglass support, ruler, and clamp as shown in Figure C-16. The #7 Ace-Thred fitting utilizes an Ace-Thred to 1/8 inch NPT adapter with a CPC quick connect. The inlet air line simply snaps into the CPC fitting. The manometer should be filled with DI water to the 5 inch mark. Red food dye may be added to aid in visibility.

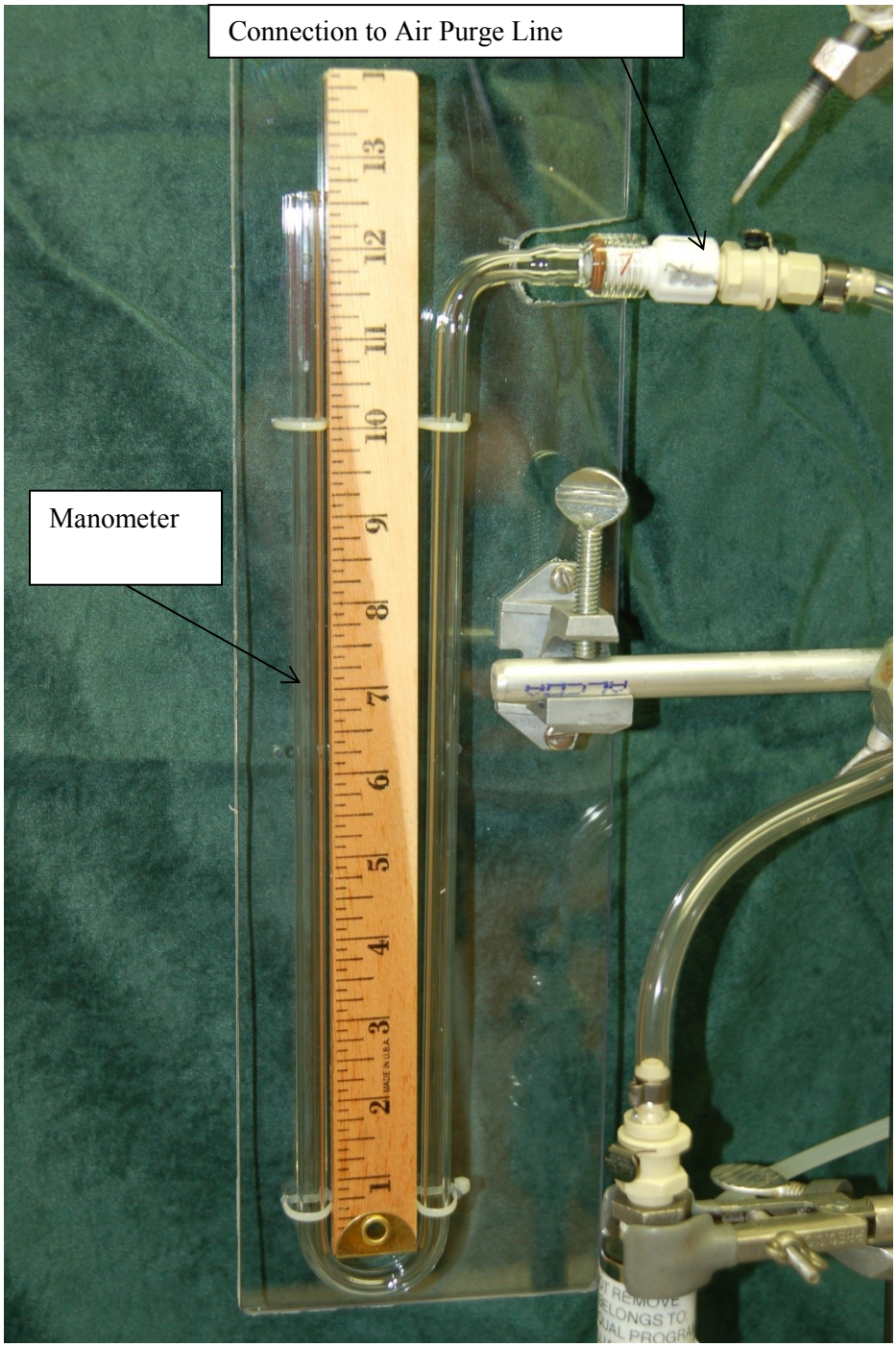


Figure C-16. Manometer Assembly

C-1.13 Addition Funnel

The addition funnel (shown in Figure C-28) simply mounts on the lid using a #11 Ace-Thred connection like the condenser.

The basic configuration of an Ace-Thred is shown below in Figure C-17, but the adapter is integral to the vessel lid on the HGR measurement apparatus and the adaptors on the HGR are polypropylene instead of Teflon. A retaining ring on the glass tube helps hold the O-ring in place during installation.

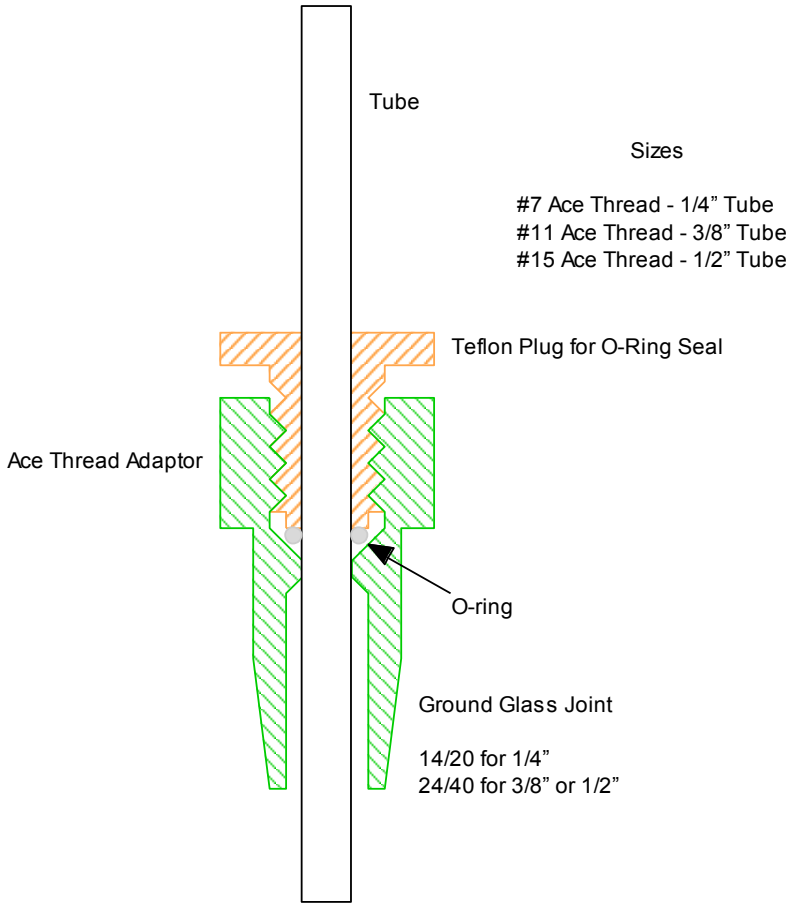


Figure C-17. Ace-Thred Diagram

C-1.14 Vessel RTD

The vessel RTD is mounted using a #7 Ace-Thred adapted to a 1/8 inch Swagelok Ultra-Torr fitting. The RTD slides through the fitting and is secured by tightening the Ultra-torr fitting. The RTD should be inserted to just above the impeller, typically ~7 inches as measured to the top of the Swagelok Ultra-Torr fitting. Once installed, the RTD should be marked to allow re-insertion to the same position. Installation of the RTD is shown above in Figure C-5 and Figure C-8. A length of tubing can be installed on the RTD to prevent insertion past the specified length, this tubing is optional.

C-1.15 Bubbler

A clamp is used to hold the bubbler support in place and the bubbler simply slides into the support, as shown in Figure C-18. The vent gas return line is installed using a quick connect.

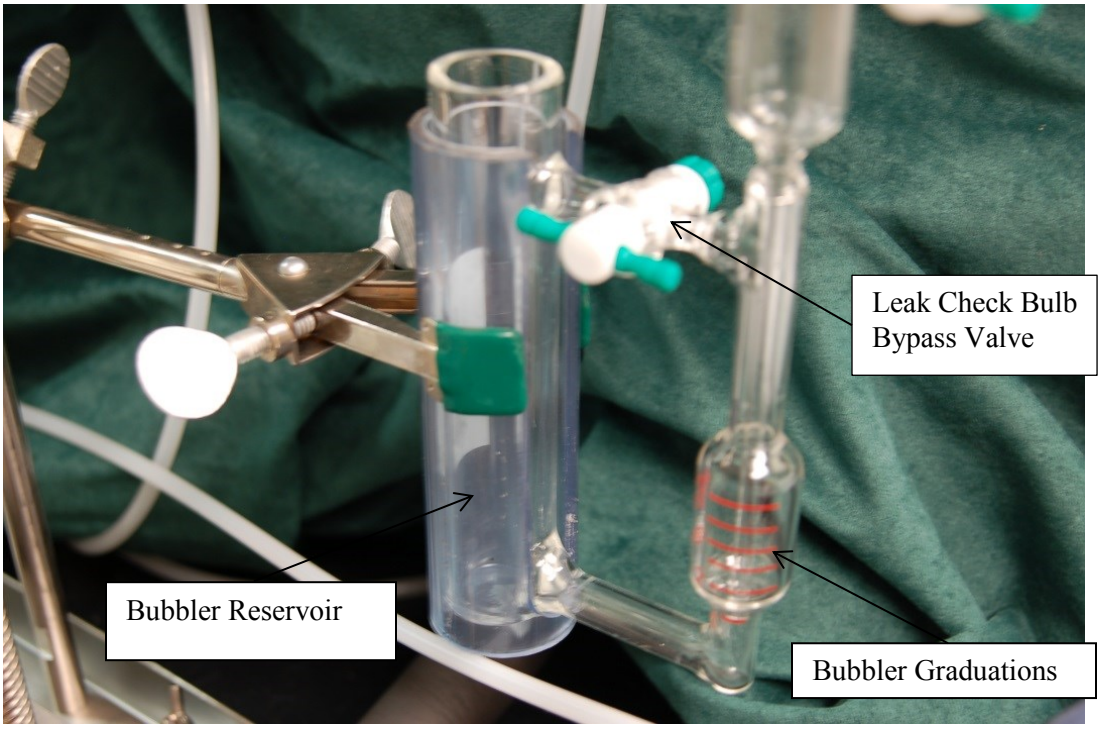


Figure C-18. Bubbler Support and Bubbler

C-1.16 Vent Gas Filter and Septum

The vent gas filter and septum assembly is shown in Figure C-19 and should be preassembled outside the cells. A clamp is used to hold the filter, and then 12 inch Tygon tubing with CPC connectors is installed to connect the condenser to the filter. The offgas line to the gas chromatograph is connected using Swagelok fittings. Note that a ¼ inch vent line to the gas chromatograph is shown, this line may be reduced to 1/8 inch during installation in the cells.

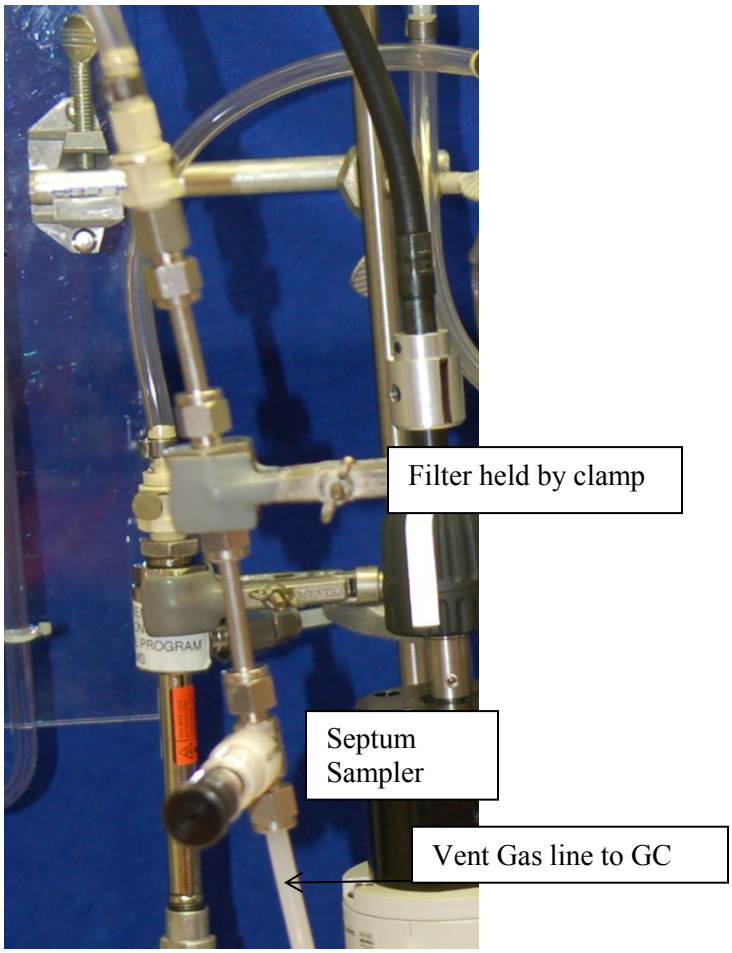


Figure C-19. Filter and Septum

C-1.17 Water Bath Connections

The water hoses should be preassembled outside the cells and consist of 2 3-4 foot insulated hoses with CPC male fittings on each end. The quick connects on the bath side are CPC HFC12 Series fittings, while the connections to the vessel are CPC PMC-12 Series fittings. The Ace-Thred to NPT adapters on the vessel allow the CPC PMC12 quick connects to be installed on the vessel. The hoses simply snap into place at the vessel and bath connections. The connections on the bath are shown in Figure C-20, while the connections on the vessel are shown above in Figure C-8.

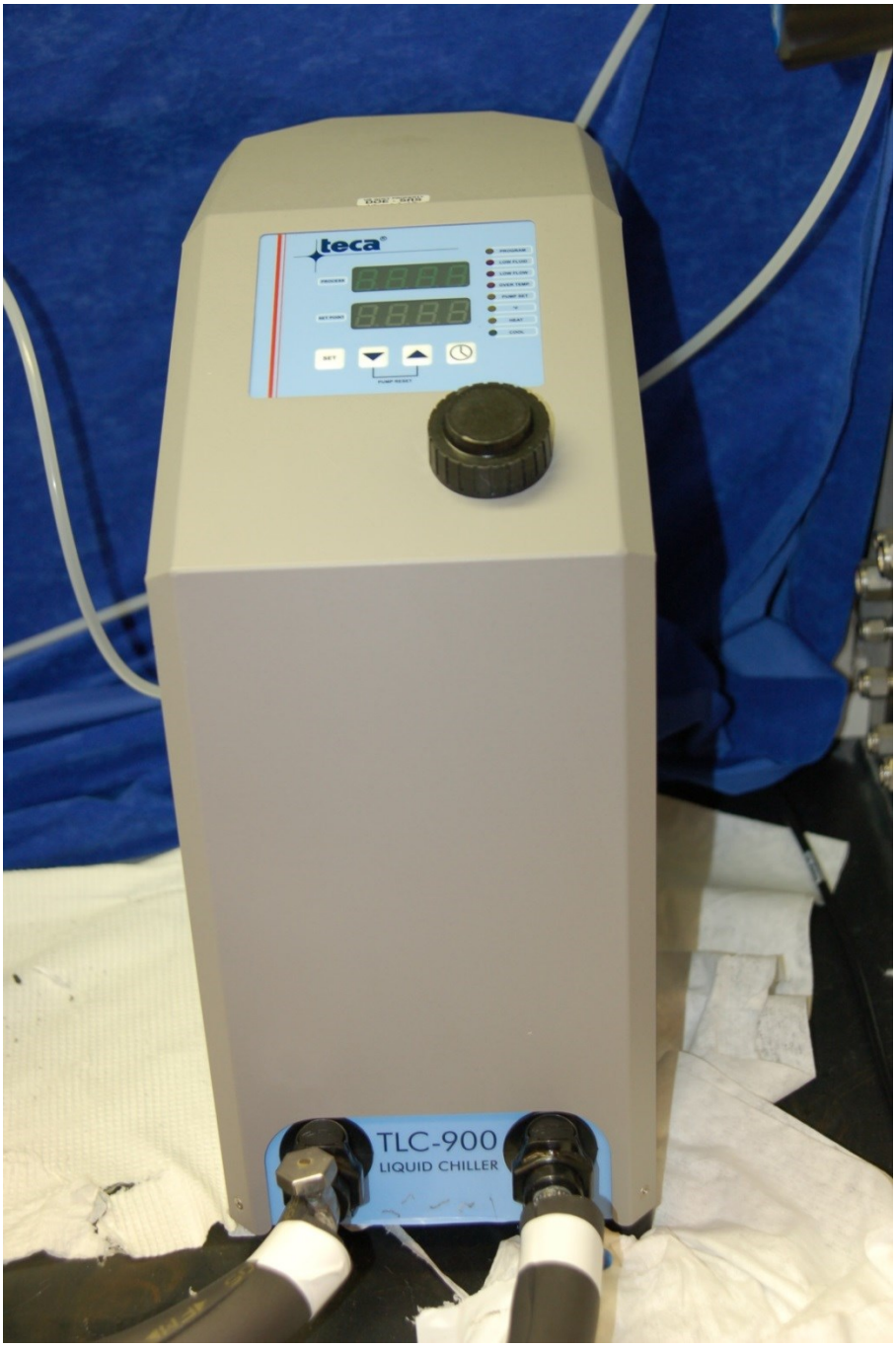


Figure C-20. Water Bath and Connections

C-1.18 Mixer Head Installation

The Cole-Parmer Servodyne mixer head simply slides into the Delrin holder, shown in Figure C-21. This holder is typically pre-installed on the stand, but the holder can be removed or reinstalled with t-handled Allen wrenches. The mixer has a local shutoff on the top of the head, care should be taken to ensure that this switch is not turned off during installation. The photograph shows the initial holder; a replacement holder with an inch of height added to the bottom ring has been fabricated.

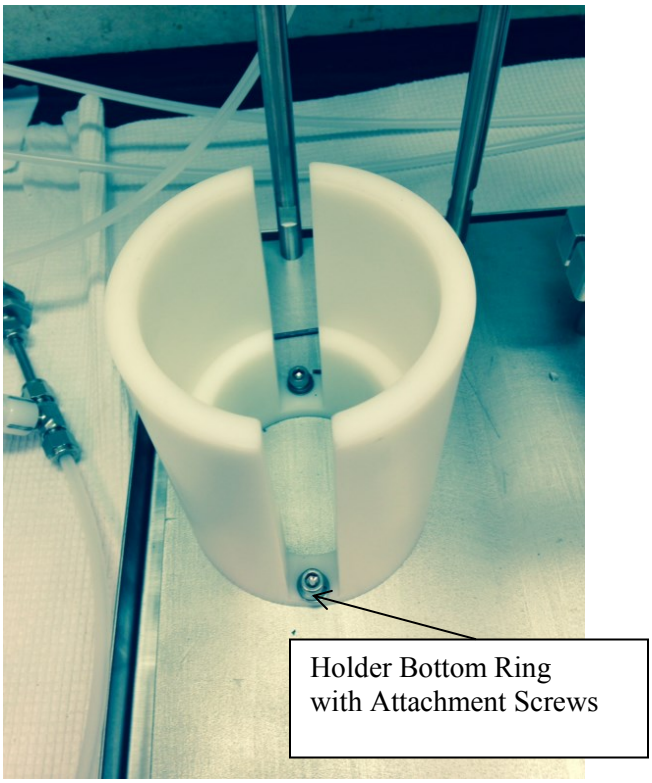


Figure C-21. Agitator Holder

C-1.19 Mixer Shaft Installation

The flexible shaft should be preassembled outside the cells to include the converter to allow the shaft to be chucked into the motor (this converter comes with the shaft) as well as having the ball head and cable end collar installed on the other end (as shown in Figure C-22 to allow coupling with the upper agitator shaft socket. The cable end collar can be adjusted with a set screw to allow the ball to fit securely in the socket with the support fully engaged in the support, shown in Figure C-3. A knob (Allen screw shown in Figure C-23 was replaced) tightens the support in the holder and prevents excessive vibration during operation. The keyless chuck holds one end of the shaft and the support hold the other.

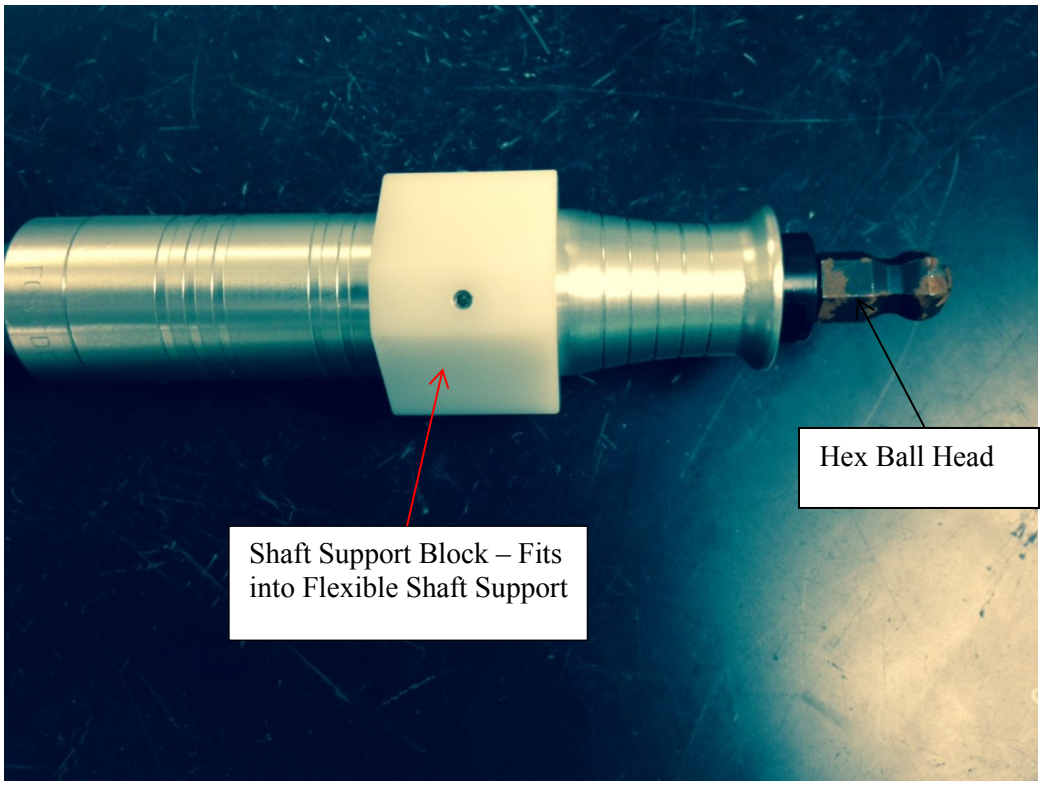


Figure C-22. Flexible Shaft with Cable End Collar Installed

Figure C-23. Flexible Shaft Support

C-1.20 Wiring Connections

The agitator motor, water bath, and RTD wiring connections to the remote control units outside the cells should be made last to preclude inadvertent operation during assembly. The remote control units should be unplugged during connection of the wires. Each wire will have a quick connect that is mated to a wire from the remote units. The connections inside the cell should be made first, and then the connections to the remote units outside the cell, then the remote control boxes may be plugged in. The remote boxes should be unplugged during any maintenance or adjustment of the apparatus.

The Amphenol connector on the agitator head may be installed in a clamp to allow it to be held more easily when connecting the wire from the agitator controller, as shown in Figure C-24.

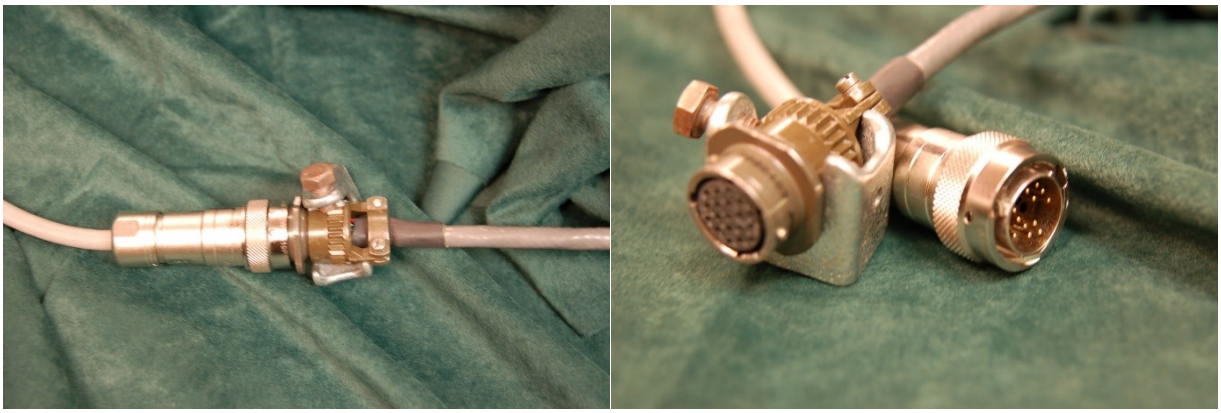


Figure C-24. Agitator Wiring Amphenol Connectors, with clamp installed

C-1.21 O-ring Installation Tool

A tool has been fabricated to aid in the installation of o-rings on the addition funnel and condenser as shown in Figure C-25. The tool allows an o-ring to be easily transferred from a piece of 3/8 inch tubing to the 3/8 inch tubing on the addition funnel and condenser. A #11 Ace-Thred adaptor is used to slide the o-ring from the 3/8 inch tubing onto the tool. The adapter is then placed on the addition funnel or condenser and the o-ring "pusher" is used to push the o-ring onto the piece. A modified spatula can be used to remove o-rings if they stick in the Ace-threads during removal. Tape or tubing can be wrapped around the middle of the spatula to make it easier to grip as well as preventing the spatula from falling into the vessel.

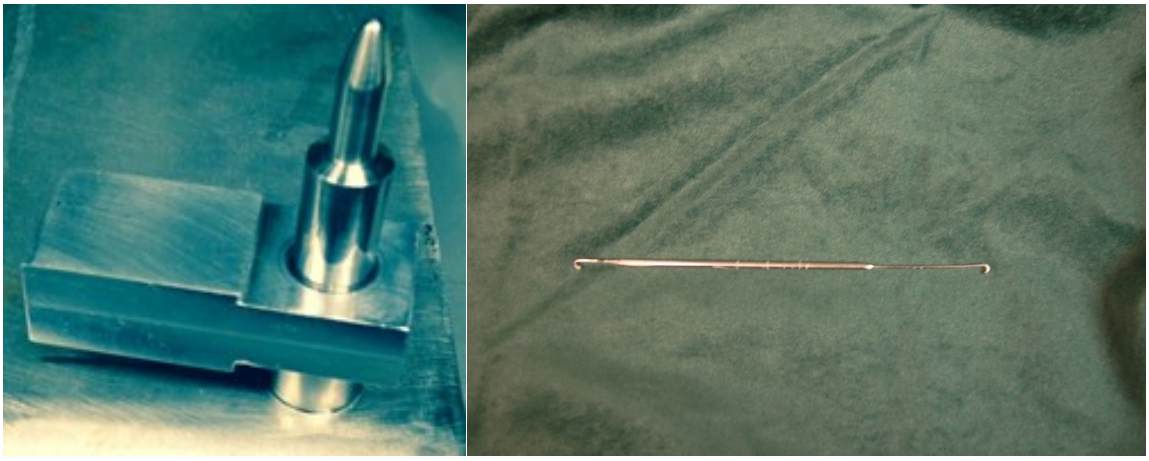


Figure C-25. O-ring Installation Tool and O-ring Removal Tool

C-1.22 Markings

All threaded connections are marked to allow the rotation of the thread part to be observed, as shown on the Ace-Thred adaptors in Figure C-26. The markings are made with a permanent marker prior to entry of the part into the cells. In addition, the vessel RTD is marked, again as shown in Figure C-26, to allow the insertion of the RTD to be monitored during the run as well as allow re-insertion of the RTD to the same position if removed. Components, such as knobs or Swagelok compression fittings, which are shaped in a way that allows the rotation to be visible do not require marking. Note that the Swagelok Ultra-Torr fitting in Figure C-26 has not yet been marked.

Figure C-26. Markings on Threaded Parts and Ace-Thred after Optional Machining

C-1.23 Completion

Once the HGR measurement apparatus assembly is complete, a visual inspection should be performed to verify that all components are installed per these instructions and the design drawings. This inspection should be performed by individuals who were not involved in the assembly process. The assembled apparatus is shown in Figure C-27 and Figure C-28 for reference. Note that the O-ring inspection tool has not been installed when these photographs were taken.

Once the visual inspection is completed, the following actions should be performed to verify proper operation:

1) The mixer and RTD readout should be turned on.
2) Both RTD probes should be reading ambient temperature.
3) The mixer should be started at low speed (~ 10 revolutions per minute) to verify the impeller spins freely without hitting the vessel walls or the RTD probe.
4) The mixer speed should slowly be increased to ensure excessive vibrations do not occur. The flexible shaft can be secured to a support with a tie wrap (or similar) if needed.
5) The water bath should be started and the connections on the water lines inspected for leaks.
    a. Add water to bath reservoir as needed.
6) Ensure the HGR vessel drain valve and addition funnel valves are CLOSED.
7) Air flow to the vortex chiller should be started at 60 liters per minute and the Condenser Chiller Air Outlet RTD temperature verified to be decreasing.
8) The vessel and lid can be insulated using ¼ inch Neoprene foam, as shown in Appendix J for operation at temperatures above 190 degrees Fahrenheit.

It is expected that leak checks and other pre-run testing are to be performed by the operating procedure.

Figure C-27. Assembled HGR Measurement Apparatus, Side View

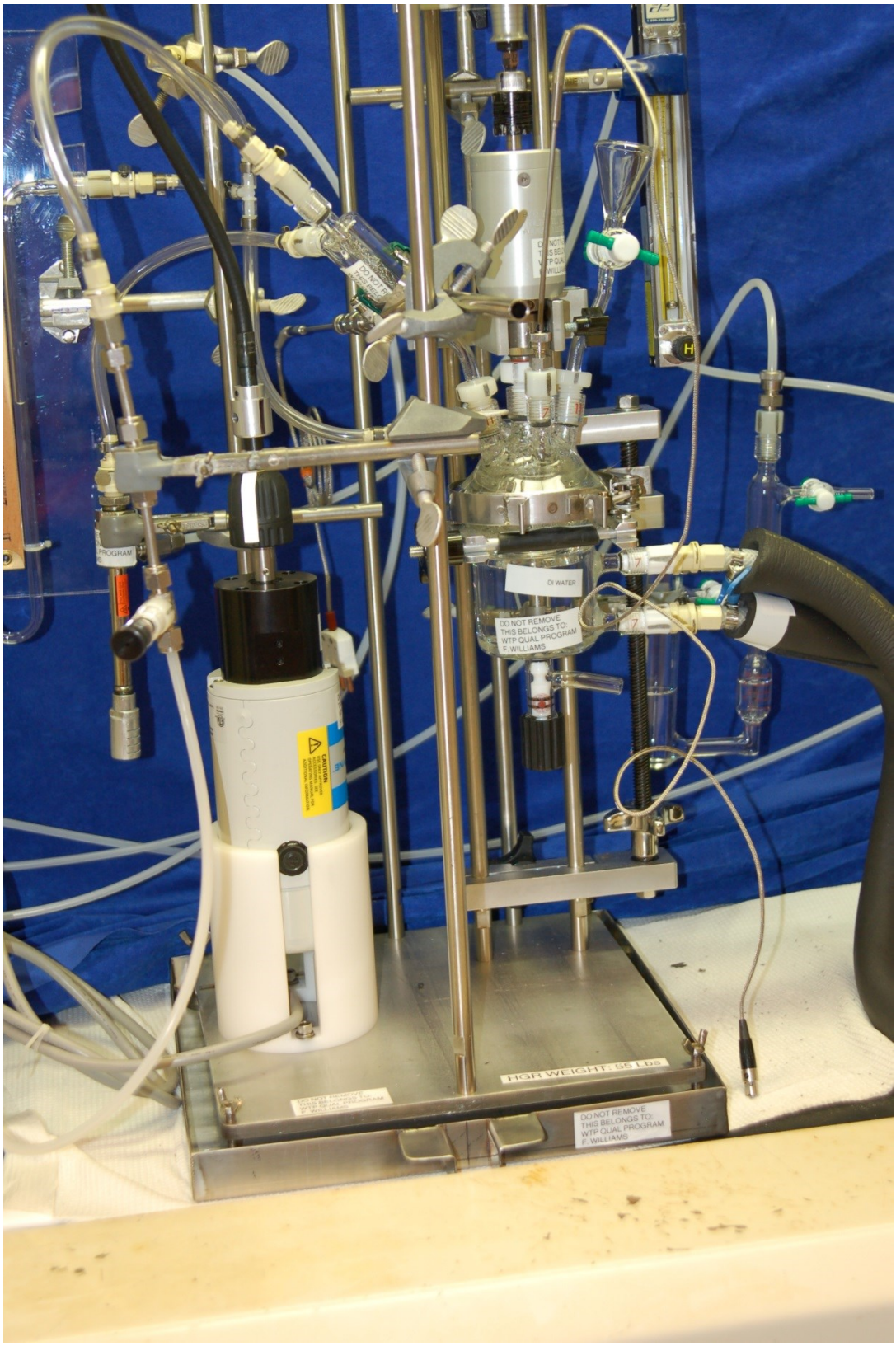


Figure C-28. Assembled HGR Measurement Apparatus, Front View

Appendix D. HGRMA Operating Instructions

The following instructions for operation of the HGRMA were developed based on the methods used at SRNL to test the apparatus. These instructions will need to be adapted based on the standard format for work instruction at the performing laboratory as well as to incorporate facility specific requirements and practices.

1.0 PURPOSE

These instructions describe the steps necessary to perform testing to assess the Hydrogen Generation Rate (HGR) of samples during qualification of feed for the Hanford Waste Treatment and Immobilization Plant (WTP).

1.1 Terms and Abbreviations

GC	Gas Chromatograph
HGR	Hydrogen Generation Rate
HGRMA	Hydrogen Generation Rate Measurement Apparatus
ID	Identification
PI	Principal Investigator or designee
RTD	Resistance Temperature Detector
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.2 Responsibilities

None.

2.0 PRECAUTIONS/LIMITATIONS

2.1 Unusual conditions are to be reported immediately to the PI or the immediate supervisor.

2.2 Assembly of the apparatus can involve heavy objects, pinch points, and sharp edges if glassware is broken.

2.3 The experiments can involve various hazards. The potential hazards include hot liquids and surfaces, contact with hazardous substances, gases, acids and bases, mechanical and electrical equipment, and broken glass.

2.4 The experiments are performed under a slight positive pressure with a manometer used to monitor vessel pressure. The manometer also provides a pressure relief leg to prevent high pressure in the case of an offgas flow blockage. If the pressure differential increases during the run, the equipment should be monitored for any blockage or pluggage. If the pressure relief leg relieves due to high pressure, the leg can be refilled and the PI should be notified.

2.5 The sludge can be extremely viscous and difficult to mix. Heating viscous materials without adequate mixing can lead to a buildup of superheated slurry near the heated areas. This superheated material can erupt from the vessel once mixing resumes or when the

pressure is sufficient to overcome the viscous nature of the sludge. Prior to heating the vessel, verify that mixing is adequate. Mixing should be periodically monitored during the run to ensure that stagnant areas are minimized.

2.6 The assembled HGR apparatus weighs approximately 55 pounds.

2.7 Manipulator operation can lead to fatigue and stress, particularly on the hands and wrists. Frequent rest periods should be taken to minimize the stress on the wrist and hands.

3.0 PREREQUISITES

3.1 The hazards associated with operation of the HGRMA should be reviewed and understood by all individuals involved in the task prior to performing work.

3.2 Outstanding questions/issues should be resolved before the run commences through either a pre-job meeting or direct communications.

3.3 Appropriate procedures for recording the measuring and test equipment IDs shall be performed.

3.4 Any changes or deviations from the procedure or run plan should be approved by the PI and documented in notebook.

4.0 PERFORMANCE

4.1 General Information

The WTP has placed limits on the amount of hydrogen that can be released from feeds. The limit for the LAW feed stream is the most restrictive with an Action Limit of 3.7E-07 gram moles hydrogen per liter per hour at 45 degrees Celsius. The HGRMA is designed to measure the HGR from feed samples to determine if the Action Limits for the sample have been exceeded.

The hydrogen release from the WTP feeds result from 3 sources: radiolytic reactions, thermal (chemical) degradation reactions, and corrosion processes. Temperature impacts the rate of these processes, therefore precise control of temperature is required to accurately assess the generation rate of the sample.

Retention of hydrogen in settled solids has been documented in a number of lab-scale studies and during full-scale operations in the tank farms. Mixing has been shown to release the retained hydrogen. Thorough mixing during sample measurement is necessary to ensure that the hydrogen released from the sample is not retained by a settled bed of solids.

This procedure addresses performing the measurements necessary to determine the HGR measurement as well as conversion of the measured value to a gram mole/Liter-hr value.

4.2 Preparation

4.2.1 **ENSURE** the equipment is assembled per J-J-A-00007and assembly instructions.

4.2.1.1 **ENSURE** vessel leak check is performed as shown in Attachment 1 or as specified by PI.

4.2.2 **RECORD** sample number, sample date, sample description, PI, technician, and test date in notebook.

4.2.3 **ENSURE** total solids and density measurements are available for sample.

4.2.4 **ENSURE** GC is calibrated as specified by PI.

4.2.5 **RELEASE** vessel head clamp and **LOWER** vessel.

4.2.6 **ADD** specified amount of sample to vessel.

4.2.6.1 **RECORD** amount added.

4.2.7 **RAISE** vessel and **REINSTALL** vessel head clamp.

4.2.8 **PERFORM** vessel leak check as shown in Attachment 1 or as specified by PI.

4.2.8.1 **RECORD** leak check results on datasheet.

4.3 Performance

4.3.1 **RECORD** data during the testing on the run datasheet (Attachment 2).

4.3.2 **START** mixer and **ADJUST** speed to obtain a slight surface vortex.

4.3.2.1 **IF** a vortex cannot be achieved, **CONSULT** PI.

4.3.3 **START** air flow to vortex chiller at 50 to 60 liters per minute or as specified by PI.

4.3.3.1 **ENSURE** air supply valve is OPEN.

4.3.3.2 **ADJUST** needle valve on vortex air supply rotameter to achieve targeted air flow.

4.3.4 **START** purge flow at 2.0 standard cubic centimeters or as specified by PI.

4.3.5 **START** tracer gas flow as specified by PI if tracer is not integrated into air purge.

4.3.6 **START** GC measurements at interval specified by PI.

4.3.7 **START** heater recirculator and inspect water lines/connections for any signs of leaking.

NOTE: Open bubbler bypass valve or addition funnel valve if vessel setpoint is below current vessel temperature until vessel cools to desired setpoint.

4.3.8 **SET** temperature as specified by PI.

4.3.9 **MONITOR** experiment and **RECORD** when temperature targets for sample are met.

4.3.10 **RECORD** data specified on datasheet during the test run.

4.3.11 **MONITOR** hydrogen readings and **NOTE** when steady state values are achieved.

4.3.12 **RECORD** hydrogen data for at least seven GC readings during steady state operation.

NOTE: Steady State can also be determined by the concentration of the krypton tracer gas.

4.3.13 **If** no hydrogen is noted after 1,100 milliliters of purge air flow through vessel since reaching the temperature specified, **RECORD** the steady state hydrogen generation as < 2 parts per million.

4.3.14 **REPEAT** steps 4.3.8 through 4.3.12 as needed to obtain hydrogen measurements at all specified temperatures.

4.3.15 **STOP** GC once GC readings are complete.

NOTE: Open bubbler bypass valve or addition funnel valve until vessel cools to desired setpoint.

4.3.16 **SET** water bath to 70 °Fahrenheit and **ALLOW** vessel to cool below 100 °Fahrenheit.

4.3.17 **STOP** water bath, purge and tracer gas flow, mixer, and air flow to vortex chiller.

4.4 Shutdown / Cleanup

4.4.1 **CLEAN** all equipment in preparation for the next run.

4.4.1.1 **DRAIN** sample from vessel by opening the drain valve and addition funnel valve.

4.4.1.2 **RINSE** vessel with DI water as specified by PI.

4.4.1.3 **RINSE** vessel with nitric acid as specified by PI.

4.4.1.4 **RINSE** vessel with DI water as specified by PI.

4.4.1.5 **INSPECT** vessel to ensure that the sample has been drained and rinsed and no residue remains in vessel.

4.4.1.6 **IF** vessel or lid is not clean **OR IF** specified by PI, **REMOVE** vessel to allow vessel and lid to be cleaned.

4.4.1.7 **IF** removed, **CLEAN** vessel and lid using rinses and wipes as needed.

4.4.2 **DISPOSITION** all rinse residues and excess samples as directed by the PI**.**

4.4.3 **CONVERT** hydrogen concentrations from the GC per Attachment 3.

5.0 RECORDS

Observations and data from testing should be recorded in the laboratory notebook as directed by the PI. No additional permanent records are generated as a result of this procedure.

6.0 REFERENCES

Stone, M.E.; Adamson, D. J.; Pak, D.J.; Pareizs, J.M.,"Hydrogen Generation Rate Measurement Apparatus: Final Design Package", SRNL-RP-2014-00866, September 2014, Savannah River National Laboratory

Hydrogen Generation Rate Measurement Apparatus: Process Flow Diagram, J-J-A-00007, Rev. 0, May 2016, Savannah River National Laboratory.

7.0 ATTACHMENTS

8.1 Attachment 1 – Leak Check Instructions

8.3 Attachment 2 – Run Datasheet

8.4 Attachment 3 – Conversion of HGR Units

Attachment 1. HGRMA Vessel Air Leak Check Instructions

ENSURE flow controller is zeroed as specified by PI.

START flow through purge inlet at 4.5 sccm or as specified by PI.

CLOSE stopcock between reservoir and gas measurement leg on leak check apparatus.

When air reaches 10 ml mark on gas measurement leg, **START** stopwatch and **RECORD** system pressure.

When air reaches 2 ml mark on gas measurement leg, **STOP** stopwatch and **RECORD** system pressure and run time.

CALCULATE vent gas flow as specified by PI.

IF vent gas flow measurement is < 90% of inlet flow:

- **ENSURE** the drain and addition valves are CLOSED.
- **ENSURE** lid clamp is installed correctly.
- **ENSURE** inlet purge and vent gas valving is set correctly.
- **ADJUST** temperature probe fittings, vent gas condenser fittings, and other process connections.
- **CONSULT** PI if leak check still unacceptable after adjustments.

Attachment 2. Datasheet Example

HGR Apparatus Datasheet									Initial Leak Check					
									Flow in		sccm		Date	
Sample ID					Sample Amount		grams		Flow Out		sccm		Technician	
Sample Description									Pressure		cm water		PI	

Date	Time	Gas Flows		Pressure	Agitator Speed	Temperatures			Gas Concentrations					Comments (vortex description, other)
		Purge Flow	Tracer			Vessel	Vent Gas	Recirculator	Hydrogen	Nitrogen	Oxygen	NO2	Tracer	
		sccm	sccm	cm water	rpm	°C	°C	°C	%	%	%	%	%	

Attachment 3. Conversion of GC Measurements

RECORD sample number _____

RECORD run date and time _____

RECORD sample temperature during measurement _____ °Fahrenheit

CONVERT sample temperature to Celius _____ °Celsius
(Sample Temperature – 32) X 5 / 9

RECORD sample amount: _____ grams

RECORD density _____ g/ml

RECORD temperature for density measurement _____ °Celsius

VERIFY density measurement temperature is equal to or less than sample temperature during measurement

CALCULATE sample volume by dividing amount by density _____ milliliters
RECORD purge flow _____ sccm

RECORD average of at least 5 hydrogen concentration readings _____ ppm
Utilize minimum detection limit if all readings were zero

CALCULATE amount of hydrogen released _____ gram mole/hr
(purge flow X hydrogen concentration/1,000,000) X 60/22,000)

CALCULATE HGR for sample _____gmole/hr-L
(hydrogen released *1000 / sample volume)
Include less than sign (<) if all readings were zero

Assumptions: Ideal gas law
Gas released << purge air flow
Non-correction of density for temperature difference is bounding

Example Calculation:

RECORD sample number	Example
RECORD run date and time	5/25/16 9:15
RECORD sample temperature during measurement	140 °Fahrenheit
CONVERT sample temperature to Celius (140 – 32) X 5 / 9	60 °Celsius
RECORD sample amount:	119 grams
RECORD density	1.19 g/ml
RECORD temperature for density measurement	25 °Celsius

VERIFY density measurement temperature is equal to or less than sample temperature during measurement

CALCULATE sample volume by dividing amount by density (119 / 1.19)	100 milliliters
RECORD purge flow	2 sccm
RECORD average of at least 5 hydrogen concentration readings Utilize minimum detection limit if all readings were zero	3.5 ppm
CALCULATE amount of hydrogen released (2*3.5/1,000,000)*60/22,000	1.9E-08 gram mole/hr
CALCULATE HGR for sample (1.9E-08 * 1000/100)	1.9E-07 gmole/hr-L

Appendix E. GC Instructions

The following instructions for operation of the HGRMA gas chromatograph were developed based on the methods used at SRNL to test the apparatus. These instructions will need to be adapted based on the standard format for work instruction at the performing laboratory as well as to incorporate facility specific requirements and practices.

1.0 PURPOSE/SCOPE

The purpose of these instructions are to describe the operation and use of the HGRMA micro GC (GC).

2.0 PERFORMANCE

NOTE: This work instruction is divided into several sections listed below. The GC must be periodically baked out, calibrated prior to use, and the calibration must be checked after use. Other sections of the procedure need only be completed as necessary.

General

The micro GC should be baked out (columns regenerated) between experiments or after prolonged use. The micro GC should be calibrated prior to an experiment and checked at the conclusion of the experiment. During long experiments, it is beneficial to check calibration periodically.

2.1 Bake-out

The GC should be baked out prior to a run and periodically during a long run (e.g., weekly for a multi-week run). Ideally, the GC should be baked out overnight. However, there is benefit for times as short as four hours.

2.1.1 **ENSURE** EZ IQ is running. **RUN** the following steps from EZ IQ.

2.1.1.1 **CHOOSE** File>>Method>>Open.

2.1.1.2 **OPEN** the method BakeoutClassic.net.

2.1.1.3 **ENSURE** that Column A temperature is set at 180 °C, Column B temperature is set at 180 °C, column pressures are set at 20 psi, and both detector filaments are ON.

2.1.1.4 **CHOOSE** Control>>Download Method to send the method to the GC.

2.1.1.5 **CHOOSE** Control>>Instrument Status and **ENSURE** method was downloaded. Detector filaments should be on and column temperatures should be increasing.

2.1.2 To return the GC columns to their normal operating temperatures,

2.1.2.1 **CHOOSE** File>>Method>>Open.

2.1.2.2 **OPEN** the “HGRClassic.met” method.

2.1.2.3 **CHOOSE** Control>>Download Method to send the method to the GC.

2.1.2.4 **CHOOSE** Control>>Instrument Status and **ENSURE** method was downloaded. Column temperatures should be dropping.

2.1.3 The GC must be calibrated (Section 2.2) following a bake-out and prior to use for sampling.

2.1.4 RECORD on the data sheet (Attachment 1) the date and time bake-out began and ended.

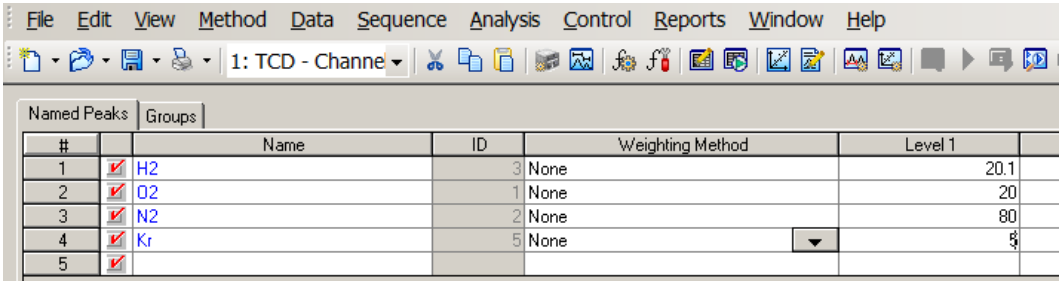
2.2 Calibration

2.2.1 ENSURE Diablo EZReporter and EZ IQ are running. Run the following steps from EZ IQ.

2.2.2 ENSURE the 3-way valve for the GC inlet is positioned to flow calibration gas through the GC.

2.2.3 ENSURE the HGRClassic.net is loaded.

2.2.4 ENSURE the level 1 gas concentrations are correct via Method>>Peaks/Groups… Note: values in screen shot are examples only.



#		Name	ID	Weighting Method	Level 1
1	☑	H2	3	None	20.1
2	☑	O2	1	None	20
3	☑	N2	2	None	80
4	☑	Kr	5	None	8
5	☑				

2.2.5 CHOOSE File>>Sequence/Open…

2.2.6 OPEN the "HGR.seq" file.

2.2.7 CHANGE the sample ID field to describe the sample. For example, CalGas.

2.2.8 CHOOSE Control>>Sequence Run.

2.2.9 Under Run range, **SELECT** "All".

2.2.10 CLICK on the clock icon in the lower left of the dialog box.

2.2.11 SET "Perform run every" to 2 minutes.

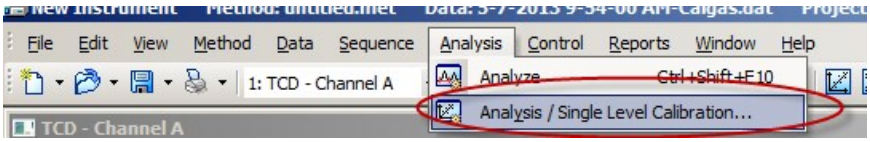
2.2.12 CLICK OK.

2.2.13 CLICK Start.

2.2.14 MONITOR the area of the gas of interest using EZReporter.

2.2.15 When gas concentation is constant, or there is no discernable upward or downward trend, stop sampling by choosing Control>>Stop run. Choose "Stop sequence after current run completes", and click OK.

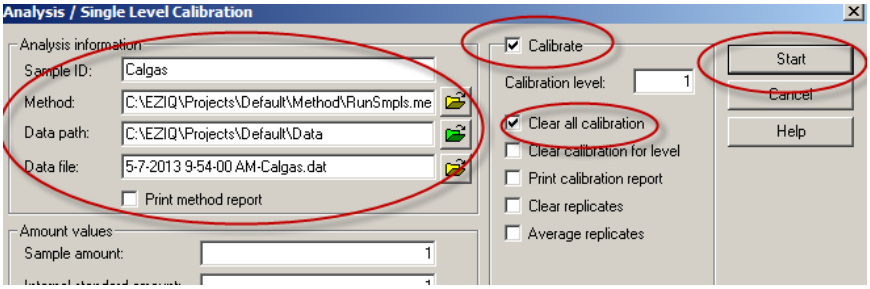
2.2.16 CHOOSE Analysis>>Analysis/Single Level Calibration.



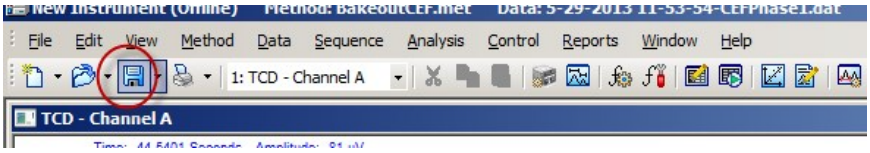
2.2.17ENSURE sample ID, data file, etc., are populated with the last run sample.

2.2.18CHECK Calibrate and Clear all Calibration.

2.2.19CLICK Start.



2.2.20CLICK the diskette icon to save the method.



2.2.21ENSURE 3-way valve for the GC inlet is positioned to sample the vessel. **CLOSE** the valve on calibration gas if used.

2.2.22RECORD on the data sheet (Attachment 1) the date and time calibration was completed, the concentration of the gas of interest, and, if applicable, manufacturer and lot number of the calibration gas, and the expiration date of the gas.

2.3 Running Samples

2.3.1 ENSURE 3-way valve for the GC inlet is positioned to sample the vessel. **CHOOSE** File>>Sequence/Open…

2.3.2 OPEN the "HGR.seq" file,

2.3.3 CHANGE the sample ID to describe the run or experiment.

2.3.4 CHOOSE Control>>Sequence Run.

2.3.5 Under Run range, **ENSURE** "All" is selected.

2.3.6 CLICK on the clock icon in the lower left of the dialog box.

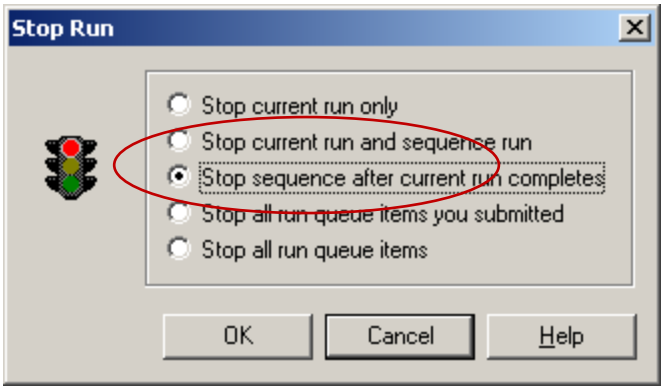
2.3.7 SET "Perform run every" to the desired sampling interval.

2.3.8 CLICK OK.

2.3.9 CLICK Start.

2.3.10 If prompted to save the method, do so.

2.3.11 To stop sampling, **CHOOSE** Control>>Stop run. **CHOOSE** "Stop sequence after current run completes", and **CLICK** OK.



2.4 Shut Down

If the GC will not be used for a period of weeks, or the GC needs to be turned off to move it to another location or to change carrier gas, columns should be cooled prior to turning off the instrument using a Stand By method.

2.4.1 **CHOOSE** File>>Method>>Open.

2.4.2 **OPEN** the StandbyClassic.met method.

2.4.3 **CHOOSE** Control>>Download Method to send the method to the GC.

2.4.4 **CHOOSE** Control>>Instrument Status.

2.4.5 When column temperatures reach 50 °C, the GC may be turned off.

3.0 Location of Chromatograms and Data

This section provides the location of chromatograms and output files.

3.1 Chromatograms are located in the folder C:\EZIQ\Projects\Default\Data.

3.2 The GC output file is a comma delimited file located at:
c:\Program Files\Diablo EZReporter\GC Output\HGRResults.csv.

Attachment 1 – GC User Log Information

Run Description_____

GC ID (MS&E number or ELI number)_____

Date(s) of Run_____

Baked out (Date bake-out began and date ended)_____

Calibration Gas manufacturer and lot number if applicable_____

Calibration Gas Expiration Date if applicable_____

Calibration completed on (date/time)_____

Calibration gas concentration(s) and other notes_____

Appendix F. RAMI Review

Table F-1. HGRMA RAMI Review Summary

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
Mixer head	Cole-Parmer Servodyne	WU-50008-20	0 to 900 RPM Remoted	Motor brush wear, wiring damage	35,040	4	N/A	N/A	N/A	N/A	N/A	N/A
Mixer Controller	Cole-Parmer Servodyne	WU-50008-00		No failure expected	35,040	2	N/A	N/A	N/A	N/A	N/A	N/A
Flexible Shaft	Ace	8081-30	Supplied with wrench to tighten collet	Breakdown of lubricating grease	17,520	4	N/A	N/A	N/A	N/A	N/A	N/A
Flexible Shaft Adapter	Ace	8081-27	Adapter to 3/8" motor	No failure expected	35,040	4	N/A	N/A	N/A	N/A	N/A	N/A
Mixer Coupling	Parr	A1120HC		No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
Impeller	Cole-Parmer	WU-04560-57	1.5" Lightnin R100 Series stainless steel impeller	Erosive wear	17,520	24	N/A	N/A	N/A	N/A	N/A	N/A
Impeller Shaft	SRNL		Custom Fab	No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
Aluminum Helical Flexible Shaft Coupling	McMaster-Carr	9861T619	3/8" shaft coupling	No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
Ball Socket	SRNL		Custom Fab	No failure expected	35,040	4	N/A	N/A	N/A	N/A	N/A	N/A
Ball	SRNL		Custom Fab	No failure expected	35,040	4	N/A	N/A	N/A	N/A	N/A	N/A
Mass Flow Controller	MKS	GM50A013500RBM010	0-5 ml/min Two needed	Failed electronics	43,800	2	120	8,760	17,520	120	N/A	0
Mass Flow Control Box	MKS	PR4000B		No failure expected	43,800	2	120	8,760	17,520	120	N/A	0
Cables for MKS Flow Controller	MKS	CB147-1-10	Two needed	No failure expected	43,800	2	N/A	N/A	N/A	N/A	N/A	N/A
Vortex Chiller	Exair	HT-3210		Plugged with solids	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Vortex Chiller Rotameter	Cole-Parmer	WU-32035-16	20-100 LPM	Plugged with solids	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Vessel RTD	Omega	PRTF-11-2-100-1/8-12-E-SB-OTP	Replace every two years	short circuit	43,800	8	120	17,520	17,520	48	17,520	0
Condenser RTD	Omega	PRTF-11-2-100-1/8-6-E-SB-OTP	Replace every two years	short circuit	43,800	8	120	17,520	17,520	48	17,520	0
RTD Readout	Omega	CN606RTD3	Replace every two years	short circuit	43,800	4	120	17,520	17,520	48	17,520	0
RTD Readout Case				No failure expected								
RTD Cable	Omega	GECU25-OTP-U-F-OTP-U-F	25' RTD extension cable with Female OTP connector on	short circuit	43,800	168	120	17,520	17,520	48	17,520	0

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
			one end									
Mist Eliminater Mesh	Koch-Glitsch	Knitted Yorkmesh	or equal - SRNL will provide spare material	Plugged with solids	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Vent Gas Filter	Swagelok	SS-4FWS-05	FW series 0. 5micron sintered metal filter	Plugged with solids	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Bubbler Assembly	SRNL	Glass		Stopcocks wear out	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
				Seals degrade from radiation/chemicals								
				Glass discoloration over time limit use								
				Fracturing of glass tube adapters								
Water Recirculator	TECA	TLC-900 6-E5EB-1-0A2	Remoted	impeller pump failure	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Tubing for Water Lines	Cole-Parmer	WU-95623-04	5/16" ID Peroxide Cured Reinforced Silicon tubing		43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Connections for Air and Water Lines	CRC		Operable to 90 degrees C per vendor info.	TECA email 3/2/16	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
			SRNL experience at higher temperature	water line leakage at fittings								
GC	Inficon	Micro GC 3000	2-Channels, 2 Carrier Gas Connections, 2 Sample Inlets Channel A = 10m Molsieve, Variable Large Volume Injector (GCMOD-GJ) Channel B = 10m Molsieve, Variable Large Volume Injector (GCMOD-GJ)	Electronic board failure	26,280	24	120	8,760	730	8	N/A	0
GC Software	Inficon	EZIQ		No failure expected	26,280	4	4	8760	N/A	N/A	N/A	N/A
GC Software	Diablo	EZReporter 1.0	Standard Ed.	No failure expected	26,280	4	4	8760	N/A	N/A	N/A	N/A
GC Hydrocarbon/Moisture	Inficon	G2870A-01	Carrier gas conditioner	No failure expected	26,280	4	N/A	N/A	N/A	N/A	N/A	N/A

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
Trap												
Connections on Vessel	Swagelok Ultra-Torr	SS-2-UT-1-2-BT SS-4-UT-1-2-BT SS-6-UT-1-4-BT	1/8" Diameter UT fitting adapter to 1/8" NPT, Bored Through 1/4" Diameter UT fitting adapter to 1/8" NPT, Bored Through 3/8" Diameter UT fitting adapter to 1/4" NPT, Bored Through	leakage	87,600	4	N/A	N/A	N/A	N/A	N/A	N/A
	Ace Ace-Thred	5844-16 5844-72 5844-34 5846-06	Nylon, #7 Ace Thread to 1/8" NPT PTFE, #7 Ace Thread to 1/4" NPT Nylon, #15 Ace Thread to 1/4" NPT Nylon, #11 Ace Thread stopper (3) (2 drilled through and threaded for 1/4" NPT)	leakage	87,600	24	N/A	N/A	N/A	N/A	N/A	N/A
				ACE-Thread adapter could break								
				Funnel attached to vessel front								
				could break off								
Condenser Assembly	SRNL Glass Shop	Glass		10 mm glass tubing on bottom		24						
				could break off								
				O-ring on ACE fitting degrade radiation								
Funnel Assembly	SRNL Glass Shop	Glass		Long tube to funnel could break		24						
				O-ring on ACE fitting degrade radiation								
				Stopcock on ACE fitting degrade radiation								
Septum Connect	SRNL Glass Shop	Glass		Unsupported and could break connection		4						
60 mm Vessel Lid Clamp	Ace	6517-22	Remoted and made of	No failure expected	87,600	24	8,760	24	N/A	N/A	N/A	0

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
			stainless steel									
Cole-Parmer	www.coleparmer.com											
Description	Quantity	Part/Cat Number										
0-900 RPM Servodyne Mixer Head	1	WU-50008-20		No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
Servodyne Mixer Controller	1	WU-50008-00		No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
1.5" Lightnin R100 Series stainless steel impeller	2	WU-04560-57		No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
5/16" ID Peroxide Cured Reinforced Silicon tubing	1	WU-95623-04		No failure expected	43,800	8	N/A	N/A	N/A	N/A	N/A	N/A
Variable Area Flowmeter, 20 to 100 LPM	1	WU-32035-16		No failure expected	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Omega	www.omega.com											
Description	Quantity	Part/Cat Number										
1/8" Diameter RTD, 12" long	2	PRTF-11-2-100-1/8-12-E-SB-OTP		short circuit	43,800	24	120	17,520	17,520	48	17,520	0
1/8" Diameter RTD, 6" long	2	PRTF-11-2-100-1/8-6-E-SB-OTP		short circuit	43,800	24	120	17,520	17,520	48	17,520	0
6 channel RTD Scanner with RS232 communication	1	CN606RTD3		short circuit	43,800	24	120	17,520	17,520	48	17,520	0
25' RTD Extension Cable with OTP ends	1	GECU25-OTP-U-F-OTP-U-F		short circuit	43,800	24	120	17,520	17,520	48	17,520	0
Swagelok	www.swagelok.com/columbiaSC											
Description	Quantity	Part/Cat Number										
1/8" Diameter UT fitting adapter to 1/8" NPT, Bored Through	2	SS-2-UT-1-2-BT		leakage	87,600	4	N/A	N/A	N/A	N/A	N/A	N/A
1/4" Diameter UT fitting adapter to 1/8" NPT, Bored Through	2	SS-4-UT-1-2-BT		leakage	87,600	4	N/A	N/A	N/A	N/A	N/A	N/A
3/8" Diameter UT fitting adapter to 1/4" NPT, Bored Through	2	SS-6-UT-1-4-BT		leakage	87,600	4	N/A	N/A	N/A	N/A	N/A	N/A

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
Nupro Filter, FWS Series, 0.5 micron	1	SS-4FWS-05		plugged with solids	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
MKS	www.mksinst.com											
Description	Quantity	Part/Cat Number										
Dual Channel Readout for MKS Flow Controllers	1	PR4000B		No failure expected	43,800	2	120	17,520	17,520	120	N/A	0
MKS Cables	2	CB147-1-10		No failure expected	43,800	2	N/A	N/A	N/A	N/A	N/A	N/A
Exair	www.exair.com											
Description	Quantity	Part/Cat Number										
Vortex Chiller with metal internals	1	HT-3210		solids pluggage from dirty air	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Parr	www.parrinst.com											
Description	Quantity	Part/Cat Number										
Magnetic Drive	1	A1120HC		No failure expected	35,040	24	N/A	N/A	N/A	N/A	N/A	N/A
Mixer head	*Cole-Parmer Servodyne*	*WU-50008-20*	*0 to 900 RPM Remoted*	*Checked with tach before/after use*	35,040	4	N/A	N/A	N/A	N/A	N/A	N/A
Mixer Controller	*Cole-Parmer Servodyne*	*WU-50008-00*		*Checked with tach before/after use*	35,040	2	N/A	N/A	N/A	N/A	N/A	N/A
Mass Flow Controller	MKS	GM50A013500RBM010	0-5 ml/min (2) Two year Warranty	Calibration by cal lab before/after	43,800	4	120	17,520	17,520	120	N/A	0
Mass Flow Control Box	MKS	PR4000B		Calibration by cal lab before/after	43,800	2	120	17,520	17,520	120	N/A	0
Vortex Chiller Rotameter	Cole-Parmer	WU-32035-16	20-100 LPM	Calibration by cal lab before/after	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Vessel RTD	Omega	PRTF-11-2-100-1/8-12-E-SB-OTP		Calibration by cal lab before/after	43,800	24	17,520	168	17,520	48	17,520	48
Condenser RTD	Omega	PRTF-11-2-100-1/8-6-E-SB-OTP		Calibration by cal lab before/after	43,800	24	17,520	168	17,520	48	17,520	48
RTD Readout	Omega	CN606RTD3		Calibration by cal lab before/after	43,800	2	17,520	168	17,520	48	17,520	48

HGRMA Component	Make	Model	Notes	Failure Mechanisms	Non-Calibration Check Failures				Calibration Check Failures		Scheduled Calibrations	
					Mean Time Between Failure	Mean Time to Repair	Mean Time to Maintain	Mean Time Between Maintenance	Mean Time Between Calibration Failures	Mean Time to Calibrate	Time Between Calibrations	Time to Calibration
					MTBF (hours)	MTTR (hours)	MTTM (hours)	MTBM (hours)	MTBCF (hours)	MTTC (hours)	(hours)	(hours)
GC	*Inficon*	*Micro GC 3000*	*2-Channels, 2 Carrier Gas Connections, 2 Sample Inlets Channel A = 10m Molsieve, Variable Large Volume Injector (GCMOD-GJ) Channel B = 10m Molsieve, Variable Large Volume Injector (GCMOD-GJ)*	*Calibration checked with cal gas before/after each use*	26,280	24	120	8,760	730	8	N/A	0
Manometer		7 mm ID glass tube		plugged with solids	26,280	4	N/A	N/A	N/A	N/A	N/A	N/A
Reaction Vessel	SRNL Glass Shop	Glass		Erosive wear/etching	17,520	4	2	N/A	N/A	N/A	N/A	N/A
				ACE thread adapters could break off								
				11 mm glass tubing attachments to								
				bottom of vessel could break off								
Spill Pan	SRNL Machine Shop	Stainless steel		No failure expected	43,800	4	N/A	N/A	N/A	N/A	N/A	N/A
Support Plate	SRNL Machine Shop	Stainless steel		No failure expected	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Support Rods	SRNL Machine Shop	Stainless steel		No failure expected	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Lifting Bale	SRNL Machine Shop	Stainless steel		No failure expected	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A
Vessel Lift	SRNL Machine Shop	Stainless steel		Wear on lift screw, corrosion of lift screw and support shafts	43,800	24	N/A	N/A	N/A	N/A	N/A	N/A

Appendix G. Packaging Plan

The plan for packaging the HGRMA for storage and shipment to WTP is shown below. These cases include precut foam that allows customization of the foam in the cases to hold and support the glassware and other components during shipment. The original equipment manufacturer (OEM) boxes were retained for the agitator head, MKS control box, gas chromatograph, and water bath. These boxes will be used for storage and shipment of those components, as shown in Figure G-1 through Figure G-4.

- Two Pelican 1600 cases w/ foam - B&H # PE1600FB
  - All glassware
- Pelican 1750 case w/ foam- B&H # PE1750FB
  - Support stand
- Two Pelican 1660 Case w/ foam - B&H # PE1660FB
  - Water bath controller, RTD readout, MKS controllers, Agitator controller, tubing and cables
- OEM boxes
  - Agitator head, MKS controller, GC, Water bath

Figure G-1. Packaged HGRMA

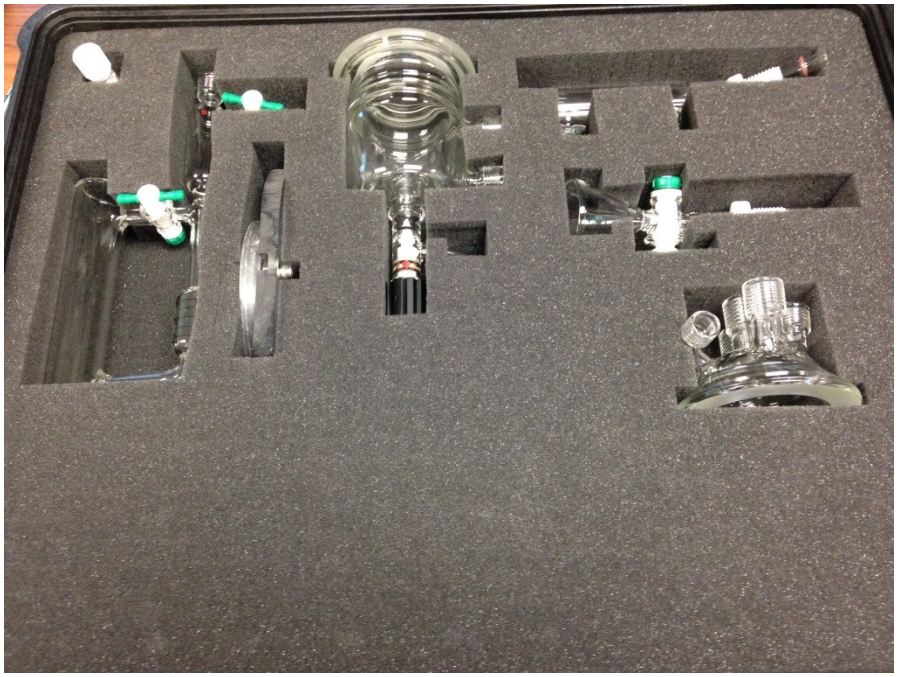


Figure G-2. HGRMA Vessel in Case

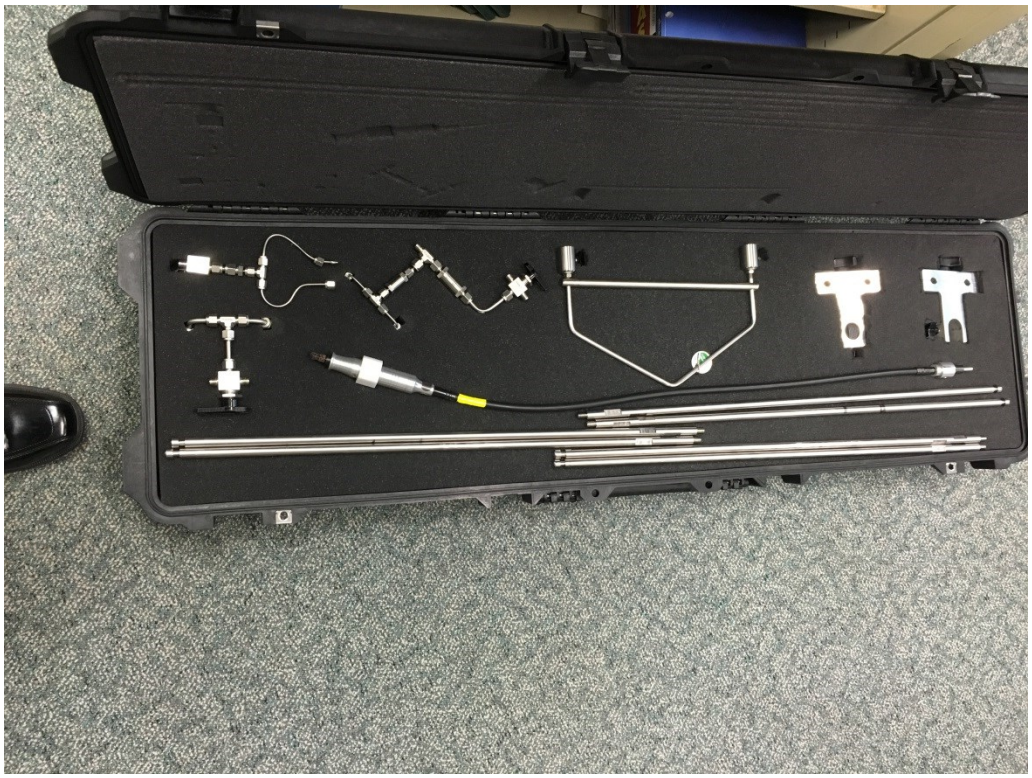


Figure G-3. HGRMA Miscellaneous Components in Case

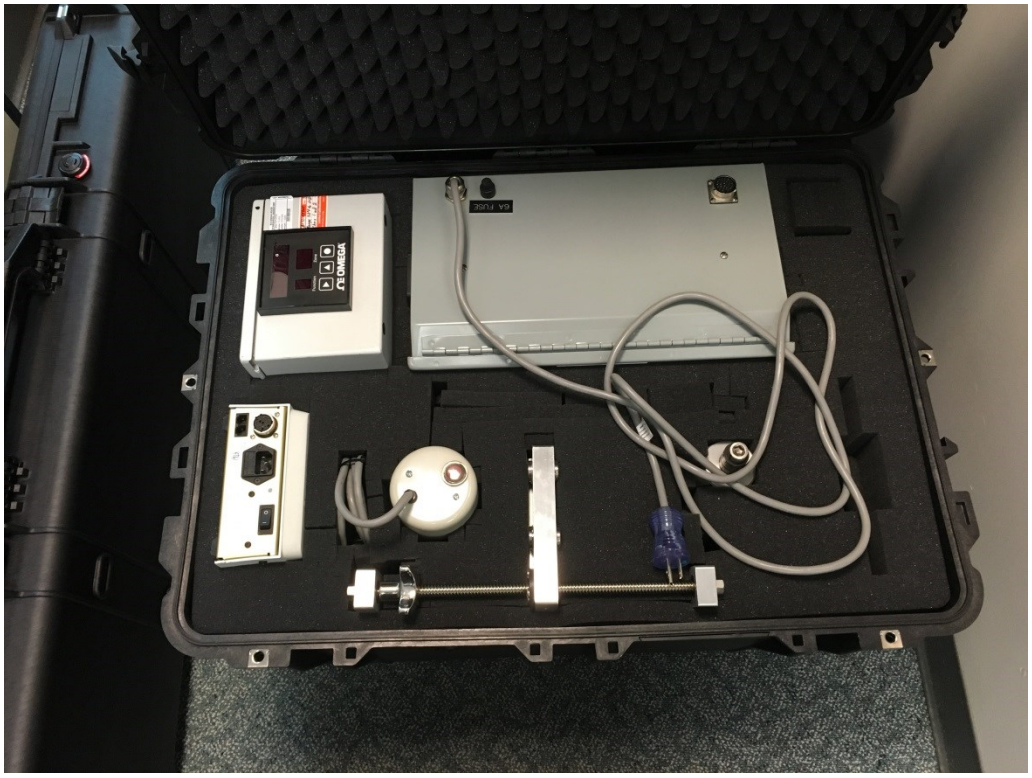


Figure G-4. HGRMA Water Bath and Controllers, RTD Readout, and Miscellaneous Components in Case

Appendix H. Design Drawings

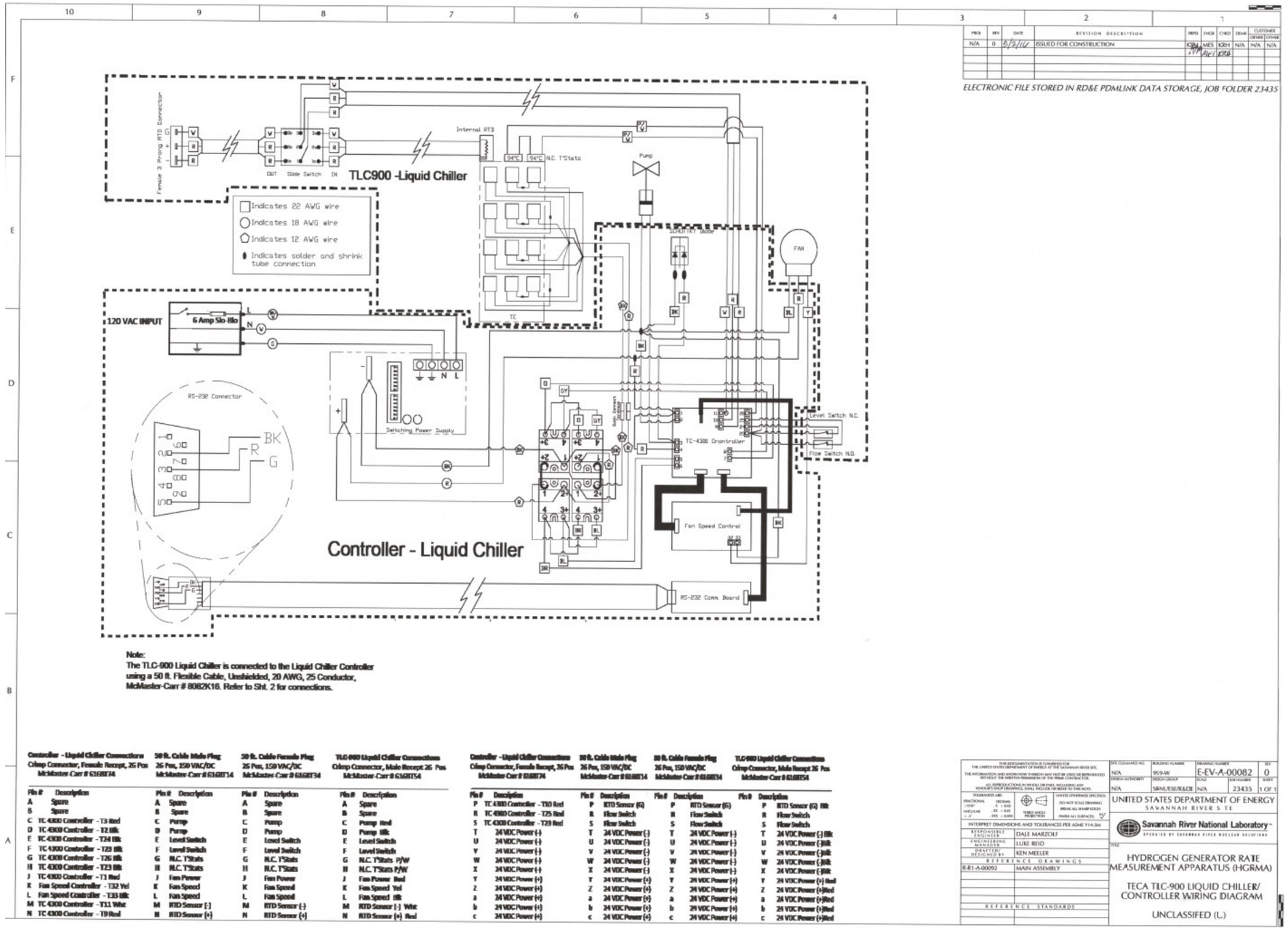
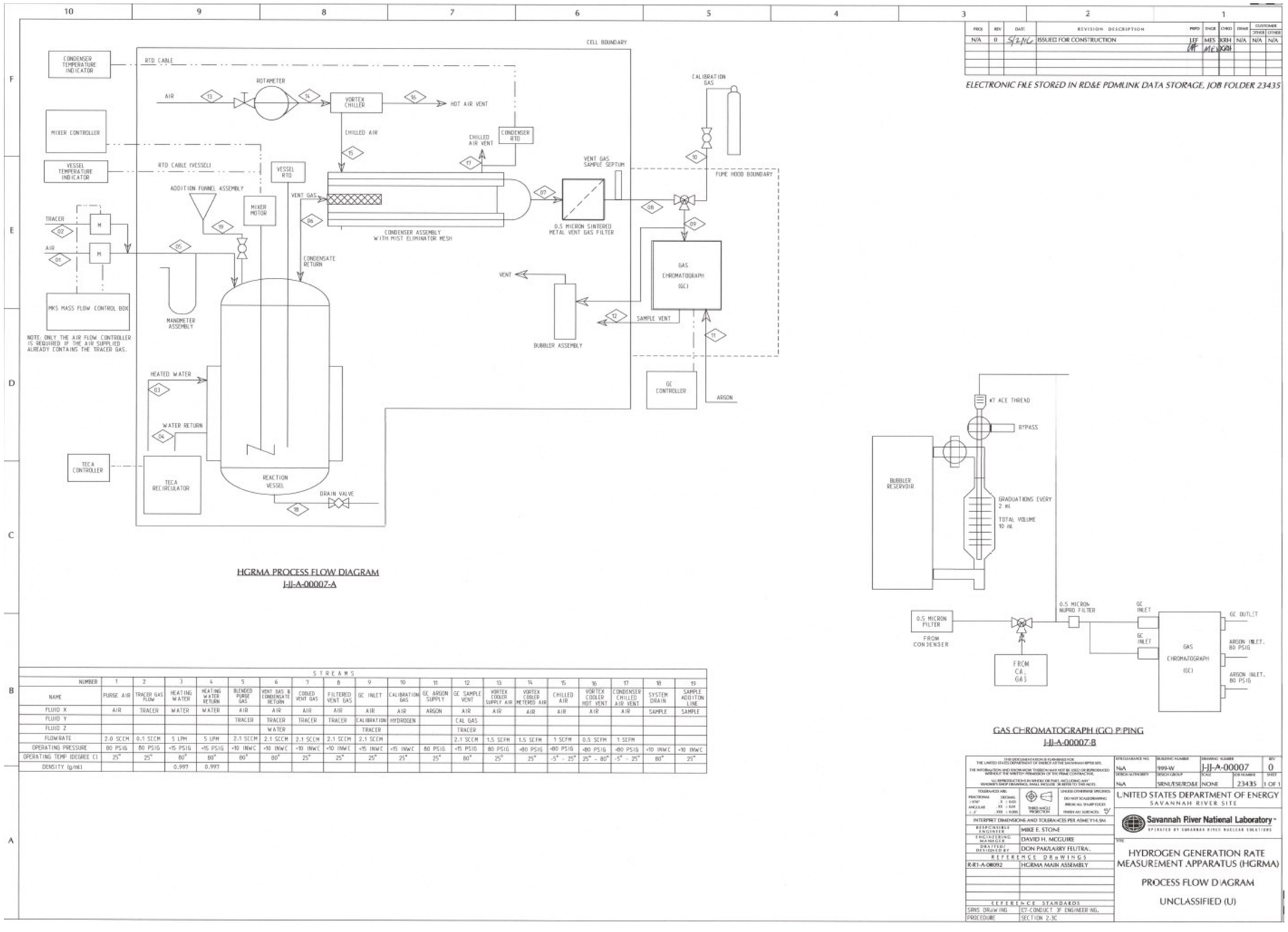


Figure H-1. TECA Chiller Remoting Diagram



HGRMA PROCESS FLOW DIAGRAM
I-JJ-A-00007-A

PROJ	REV	DATE	REVISION DESCRIPTION	PREP'D	ENGR	CHK'D	DRAW	CUSTOMER APVD	CUSTOMER OTHER
N/A	0	5/2/16	ISSUED FOR CONSTRUCTION	LEF	MES	KRH	N/A	N/A	N/A

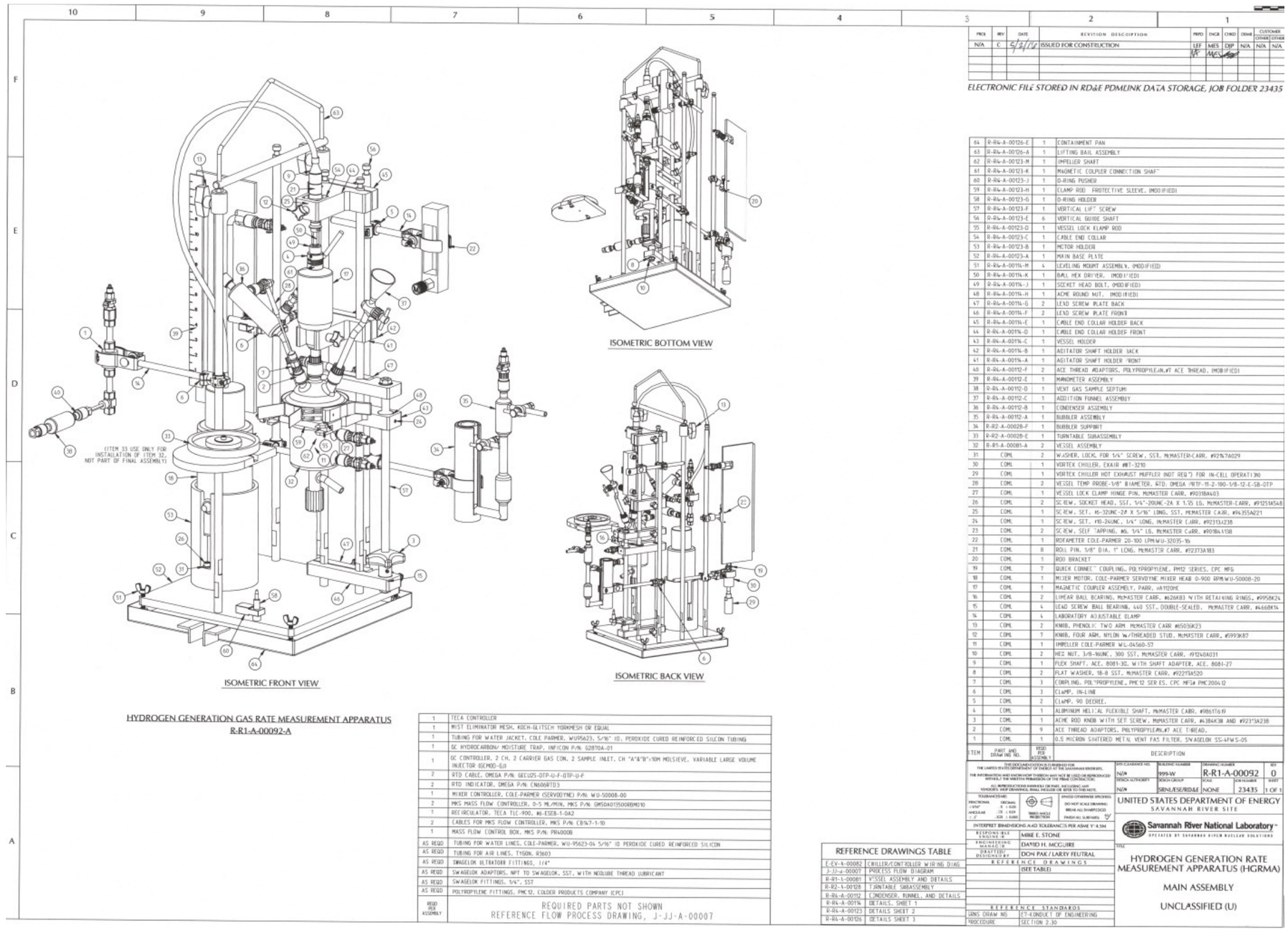
ELECTRONIC FILE STORED IN RD&E PDMLINK DATA STORAGE, JOB FOLDER 23435

STREAMS																			
NUMBER	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
NAME	PURGE AIR	TRACER GAS FLOW	HEATING WATER	HEATING WATER RETURN	BLENDED PURGE GAS	VENT GAS & CONDENSATE RETURN	COOLED VENT GAS	FILTERED VENT GAS	GC INLET	CALIBRATION GAS	GC ARGON SUPPLY	GC SAMPLE VENT	VORTEX COOLER SUPPLY AIR	VORTEX COOLER METERED AIR	CHILLED AIR	VORTEX COOLER HOT VENT	CONDENSER CHILLED AIR VENT	SYSTEM DRAIN	SAMPLE ADDITION LINE
FLUID X	AIR	TRACER	WATER	WATER	AIR	AIR	AIR	AIR	AIR	AIR	ARGON	AIR	AIR	AIR	AIR	AIR	AIR	SAMPLE	SAMPLE
FLUID Y					TRACER	TRACER	TRACER	TRACER	CALIBRATION	HYDROGEN		CAL GAS							
FLUID Z						WATER			TRACER			TRACER							
FLOWRATE	2.0 SCCM	0.1 SCCM	5 LPH	5 LPM	2.1 SCCM	2.1 SCCM	2.1 SCCM	2.1 SCCM	2.1 SCCM			2.1 SCCM	1.5 SCFM	1.5 SCFM	1 SCFM	0.5 SCFM	1 SCFM		
OPERATING PRESSURE	80 PSIG	80 PSIG	<15 PSIG	<15 PSIG	<10 INWC	<10 INWC	<10 INWC	<10 INWC	<15 INWC	<15 INWC	80 PSIG	<15 PSIG	80 PSIG	<80 PSIG	<80 PSIG	<80 PSIG	<80 PSIG	<10 INWC	<10 INWC
OPERATING TEMP (DEGREE C)	25°	25°	80°	80°	60°	80°	25°	25°	25°	25°	25°	60°	25°	25°	-5° - 25°	25° - 80°	-5° - 25°	80°	25°
DENSITY (g/ml)			0.997	0.997															

GAS CHROMATOGRAPH (GC) PIPING
I-JJ-A-00007-B

THIS DOCUMENT/DESIGN IS FURNISHED FOR THE UNITED STATES DEPARTMENT OF ENERGY AT THE SAVANNAH RIVER SITE. THE INFORMATION AND KNOW-HOW THEREIN MAY NOT BE USED OR REPRODUCED WITHOUT THE WRITTEN PERMISSION OF THE PRIME CONTRACTOR. ALL REPRODUCTIONS IN WHOLE OR PART, INCLUDING ANY VENDOR'S SHOP DRAWINGS, SHALL INCLUDE SIMILAR NOTE AS REFER TO THIS NOTE.	SITE/CLEARANCE NO.	BUILDING NUMBER	DRAWING NUMBER	REV
	N/A	999-W	I-JJ-A-00007	0
	DESIGN AUTHORITY	DESIGN GROUP	JOB	JOB NUMBER	SHEET
	N/A	SRNL/ESER/D&E	NONE	23435	1 OF 1
TOLERANCES ARE: FRACTIONAL ± 1/16" DECIMAL .X ± 0.03 ANGULAR .XX ± 0.01 .XXX ± 0.005 THIRD ANGLE PROJECTION UNLESS OTHERWISE SPECIFIED: DO NOT SCALE DRAWING BREAK ALL SHARP EDGES FINISH ALL SURFACES	UNITED STATES DEPARTMENT OF ENERGY SAVANNAH RIVER SITE			
INTERPRET DIMENSIONS AND TOLERANCES PER ASME Y14.5M	Savannah River National Laboratory™ OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS			
RESPONSIBLE ENGINEER	MIKE E. STONE	TITLE		
ENGINEERING MANAGER	DAVID H. MCGUIRE	HYDROGEN GENERATION RATE MEASUREMENT APPARATUS (HGRMA)		
DRAFTED/DESIGNED BY	DON PAKULA/BRY FEUTRA.	PROCESS FLOW DIAGRAM		
REFERENCE DRAWINGS		UNCLASSIFIED (U)		
R-R1-A-08092	HGRMA MAIN ASSEMBLY			
REFERENCE STANDARDS				
SRNS DRAWING	E7-CONDUCT OF ENGINEERING			
PROCEDURE	SECTION 2.3C			

Figure H-2. HGRMA Process Flow Diagram



HYDROGEN GENERATION GAS RATE MEASUREMENT APPARATUS
R-R1-A-00092-A

REV	DATE	REVISION DESCRIPTION	PRPD	ENGR	CHKD	DRWG	CUSTOMER OTHER	CUSTOMER OTHER
C	5/2/16	ISSUED FOR CONSTRUCTION	LEF	MES	DXP	N/A	N/A	N/A

ELECTRONIC FILE STORED IN RD&E PDMLINK DATA STORAGE, JOB FOLDER 23435

ITEM	QUAN. AND DRAWING NO.	REQD PER ASSEMBLY	DESCRIPTION
64	R-R4-A-00126-E	1	CONTAINMENT PAN
63	R-R4-A-00126-A	1	LIFTING BAIL ASSEMBLY
62	R-R4-A-00123-M	1	IMPELLER SHAFT
61	R-R4-A-00123-K	1	MAGNETIC COUPLER CONNECTION SHAFT
60	R-R4-A-00123-J	1	O-RING PUSHER
59	R-R4-A-00123-H	1	CLAMP ROD (PROTECTIVE SLEEVE, (MODIFIED)
58	R-R4-A-00123-G	1	O-RING HOLDER
57	R-R4-A-00123-F	1	VERTICAL LIFT SCREW
56	R-R4-A-00123-E	6	VERTICAL GUIDE SHAFT
55	R-R4-A-00123-D	1	VESSEL LOCK CLAMP ROD
54	R-R4-A-00123-C	1	CABLE END COLLAR
53	R-R4-A-00123-B	1	MOTOR HOLDER
52	R-R4-A-00123-A	1	MAIN BASE PLATE
51	R-R4-A-00114-M	4	LEVELING MOUNT ASSEMBLY, (MODIFIED)
50	R-R4-A-00114-K	1	BALL HEX DRIVER, (MODIFIED)
49	R-R4-A-00114-J	1	SOCKET HEAD BOLT, (MODIFIED)
48	R-R4-A-00114-H	1	ACME ROUND NUT, (MODIFIED)
47	R-R4-A-00114-G	2	LEAD SCREW PLATE BACK
46	R-R4-A-00114-F	2	LEAD SCREW PLATE FRONT
45	R-R4-A-00114-E	1	CABLE END COLLAR HOLDER BACK
44	R-R4-A-00114-D	1	CABLE END COLLAR HOLDER FRONT
43	R-R4-A-00114-C	1	VESSEL HOLDER
42	R-R4-A-00114-B	1	AGITATOR SHAFT HOLDER BACK
41	R-R4-A-00114-A	1	AGITATOR SHAFT HOLDER FRONT
40	R-R4-A-00112-F	2	ACE THREAD ADAPTORS, POLYPROPYLEN,#7 ACE THREAD, (MODIFIED)
39	R-R4-A-00112-E	1	MANOMETER ASSEMBLY
38	R-R4-A-00112-D	1	VENT GAS SAMPLE SEPTUM
37	R-R4-A-00112-C	1	ADDITION FUNNEL ASSEMBLY
36	R-R4-A-00112-B	1	CONDENSER ASSEMBLY
35	R-R4-A-00112-A	1	BUBBLER ASSEMBLY
34	R-R2-A-00028-F	1	BUBBLER SUPPORT
33	R-R2-A-00028-E	1	TURNTABLE SUBASSEMBLY
32	R-R5-A-00081-A	2	VESSEL ASSEMBLY
31	COML	2	WASHER, LOCK, FOR 1/4" SCREW, SST, McMASTER-CARR, #92146029
30	COML	1	VORTEX CHILLER, EXAIR #MT-3210
29	COML	1	VORTEX CHILLER HOT EXHAUST MUFFLER (NOT REQ'D FOR IN-CELL OPERATION)
28	COML	2	VESSEL TEMP PROBE-1/8" DIAMETER, RTD: OMEGA PRTF-11-2-100-1/8-12-E-SB-DTP
27	COML	1	VESSEL LOCK CLAMP HINGE PIN, McMASTER CARR, #90318A403
26	COML	2	SCREW, SOCKET HEAD, SST, 1/4"-20UNC-2A X 1.25 LG, McMASTER-CARR, #91251A548
25	COML	1	SCREW, SET, M5-32UNC-2A X 5/16" LONG, SST, McMASTER CARR, #94355A221
24	COML	1	SCREW, SET, #10-24UNC, 1/4" LONG, McMASTER CARR, #92313A238
23	COML	2	SCREW, SELF TAPPING, #6, 1/4" LG, McMASTER CARR, #90184A158
22	COML	1	ROTAMETER COLE-PARMER 20-100 LPM W U-32035-16
21	COML	8	ROLL PIN, 3/8" DIA, 1" LONG, McMASTER CARR, #92373A183
20	COML	1	RDD BRACKET
19	COML	7	QUICK CONNECT COUPLING, POLYPROPYLENE, PM12 SERIES, CPC MFG
18	COML	1	MIXER MOTOR, COLE-PARMER SERVODYNE MIXER HEAD 0-900 RPM W U-50008-20
17	COML	1	MAGNETIC COUPLER ASSEMBLY, PARR, #A1120HC
16	COML	2	LINEAR BALL BEARING, McMASTER CARR, #6264K83 WITH RETAINING RINGS, #9958K24
15	COML	4	LEAD SCREW BALL BEARING, 440 SST, DOUBLE-SEALED, McMASTER CARR, #6668K14
14	COML	4	LABORATORY ADJUSTABLE CLAMP
13	COML	2	KNOB, PHENOLIC TWO ARM McMASTER CARR #6503K23
12	COML	7	KNOB, FOUR ARM, NYLON W/THREADED STUD, McMASTER CARR, #5993K87
11	COML	1	IMPELLER COLE-PARMER WU-04560-57
10	COML	2	HEX NUT, 3/8-16UNC, 300 SST, McMASTER CARR, #91240A031
9	COML	1	FLEX SHAFT, ACE, 8081-3D, WITH SHAFT ADAPTER, ACE, 8081-27
8	COML	2	FLAT WASHER, 18-8 SST, McMASTER CARR, #92215A520
7	COML	3	COUPLING, POLYPROPYLENE, PME12 SERIES, CPC MFG# PME20412
6	COML	3	CLAMP, IN-LINE
5	COML	2	CLAMP, 90 DEGREE
4	COML	1	ALUMINUM HELICAL FLEXIBLE SHAFT, McMASTER CARR, #9861T69
3	COML	1	ACME ROD KNOB WITH SET SCREW, McMASTER CARR, #6384K38 AND #92313A238
2	COML	9	ACE THREAD ADAPTORS, POLYPROPYLEN,#7 ACE THREAD,
1	COML	1	0.5 MICRON SINTERED METAL VENT GAS FILTER, SWAGELOK SS-4FW5-05

1	TECA CONTROLLER		
1	MIST ELIMINATOR MESH, KOCH-GLITSCH YORKMESH OR EQUAL		
1	TUBING FOR WATER JACKET, COLE PARMER, WU95623, 5/16" ID, PEROXIDE CURED REINFORCED SILICON TUBING		
1	GC HYDROCARBON/ MOISTURE TRAP, INFICON P/N: 520T0A-01		
1	GC CONTROLLER, 2 CH, 2 CARRIER GAS CON, 2 SAMPLE INLET, CH "A"&"B":10M MOLSIEVE, VARIABLE LARGE VOLUME INJECTOR (GEM00-GJ)		
2	RTD CABLE, OMEGA P/N: GEC125-DTP-U-F-DTP-U-F		
2	RTD INDICATOR, OMEGA P/N: CN6068TD3		
1	MIXER CONTROLLER, COLE-PARMER (SERVODYNE) P/N: WU-50008-00		
2	MKS MASS FLOW CONTROLLER, 0-5 ML/MIN, MKS P/N: GMS0A0T35008M010		
1	RECIRCULATOR, TECA TLC-900, #6-ESEB-1-0A2		
2	CABLES FOR MKS FLOW CONTROLLER, MKS P/N: CB1A7-1-10		
1	MASS FLOW CONTROL BOX, MKS P/N: PR4000B		
AS REQD	TUBING FOR WATER LINES, COLE-PARMER, WU-95623-04 5/16" ID PEROXIDE CURED REINFORCED SILICON		
AS REQD	TUBING FOR AIR LINES, TYGON, R3603		
AS REQD	SWAGELOK ULTRATORR FITTINGS, 1/4"		
AS REQD	SWAGELOK ADAPTERS, NPT TO SWAGELOK, SST, WITH NEOLUBE THREAD LUBRICANT		
AS REQD	SWAGELOK FITTINGS, 1/4", SST		
AS REQD	POLYPROPYLENE FITTINGS, PM12, COLDER PRODUCTS COMPANY (CPC)		
REQD PER ASSEMBLY	REQUIRED PARTS NOT SHOWN REFERENCE FLOW PROCESS DRAWING, J-JJ-A-00007		

REFERENCE DRAWINGS TABLE	
E-EV-A-00082	CHILLER/CONTROLLER WIRING DIAG
J-JJ-A-00007	PROCESS FLOW DIAGRAM
R-R1-A-00081	VESSEL ASSEMBLY AND DETAILS
R-R2-A-00128	TURNTABLE SUBASSEMBLY
R-R4-A-00112	CONDENSER, RUNNEL, AND DETAILS
R-R4-A-00114	DETAILS, SHEET 1
R-R4-A-00123	DETAILS SHEET 2
R-R4-A-00126	DETAILS SHEET 3

RESPONSIBLE ENGINEER	MIKE E. STONE
ENGINEERING MANAGER	DAVID H. MCGUIRE
DRAFTED/ DESIGNED BY	DON PAK / LARRY FEUTRAL
REFERENCE DRAWINGS	(SEE TABLE)
REFERENCE STANDARDS	
SANS DRAW NO.	EY-4 CONDUCT OF ENGINEERING
PROCEDURE	SECTION 2.30

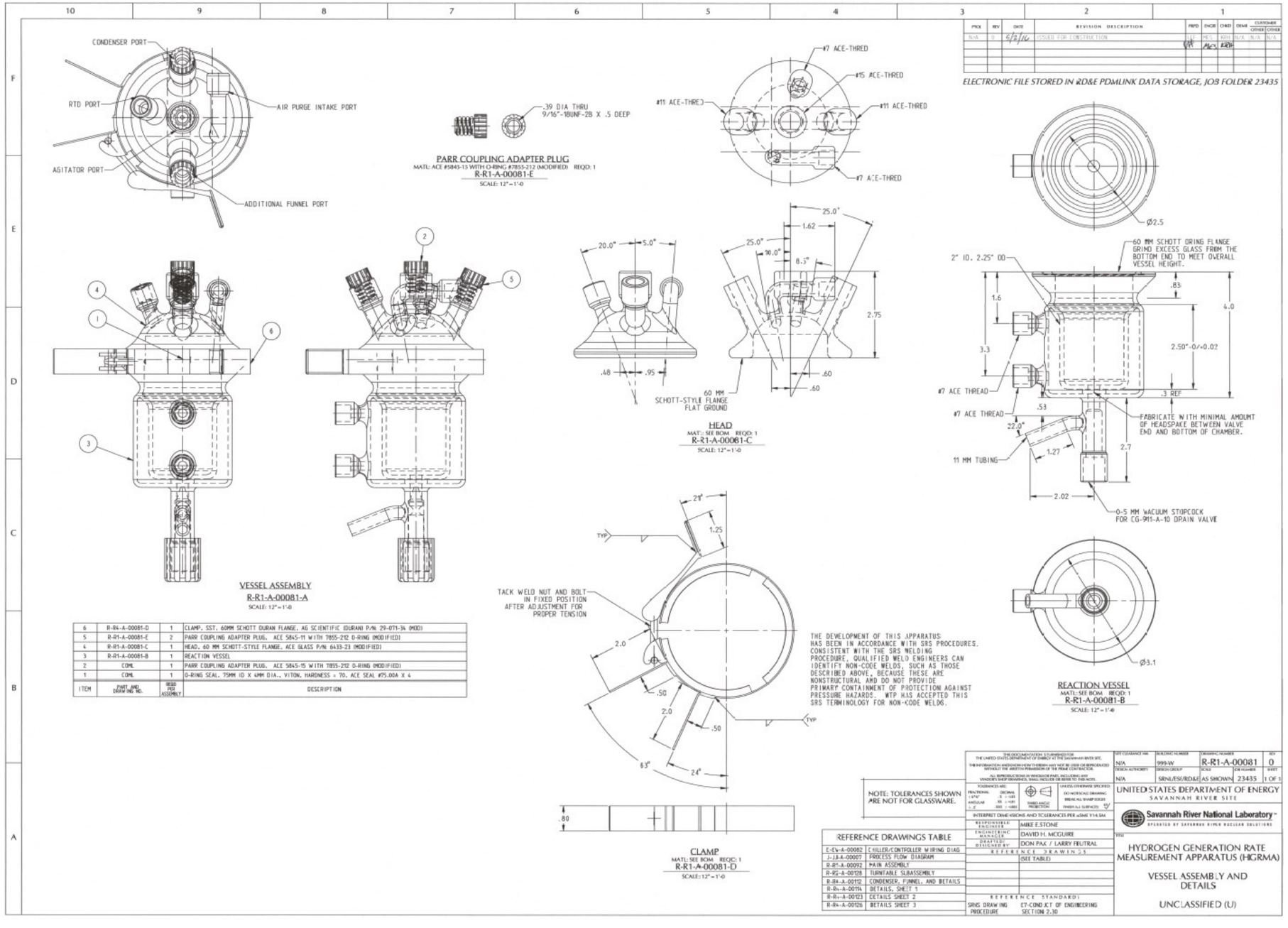
SITE CLEARANCE NO.	BUILDING NUMBER	DRAWING NUMBER	REV.
N/A	999-W	R-R1-A-00092	0
DESIGN AUTHORITY	WORK GROUP	SCALE	SHEET
N/A	SRNL/SERD/E&E	NONE	1 OF 1
	JOB NUMBER	23435	

UNITED STATES DEPARTMENT OF ENERGY
SAVANNAH RIVER SITE

Savannah River National Laboratory
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

TITLE
HYDROGEN GENERATION RATE MEASUREMENT APPARATUS (HGRMA)
MAIN ASSEMBLY
UNCLASSIFIED (U)

Figure H-3. HGRMA Assembly



ITEM	PART AND DRAWING NO.	REQD PER ASSEMBLY	DESCRIPTION
6	R-R4-A-00081-D	1	CLAMP, SST, 60MM SCHOTT DURAN FLANGE, AG SCIENTIFIC (DURAN) P/N 29-071-34 (MOD)
5	R-R1-A-00081-E	2	PARR COUPLING ADAPTER PLUG. ACE 5845-11 WITH 7855-212 O-RING (MODIFIED)
4	R-R1-A-00081-C	1	HEAD, 60 MM SCHOTT-STYLE FLANGE, ACE GLASS P/N 6433-23 (MODIFIED)
3	R-R1-A-00081-B	1	REACTION VESSEL
2	COML	1	PARR COUPLING ADAPTER PLUG. ACE 5845-15 WITH 7855-212 O-RING (MODIFIED)
1	COML	1	O-RING SEAL, 75MM ID X 6MM DIA., VITON, HARDNESS = 70, ACE SEAL #75.00A X 6

REFERENCE DRAWINGS TABLE	
E-EV-A-00082	CHILLER/CONTROLLER WIRING DIAG
J-JJ-A-00007	PROCESS FLOW DIAGRAM
R-R*-A-00092	MAIN ASSEMBLY
R-RG-A-00128	TURNTABLE SUBASSEMBLY
R-R4-A-00112	CONDENSER, FUNNEL, AND BETAILS
R-R4-A-00114	BETAILS, SHEET 1
R-R4-A-00123	BETAILS SHEET 2
R-R4-A-00126	BETAILS SHEET 3

RESPONSIBLE ENGINEER	MIKE E.STONE
ENGINEERING MANAGER	DAVID H. MCGUIRE
DRAFTED BY/ENGINEER	DON PAC / LARRY FEUTRAL
REFERENCE DRAWINGS	(SEE TABLE)
REFERENCE STANDARDS	SRNS DRAWING PROCEDURE E7-COND JCT OF ENGINEERING SECTION 2.30

SITE CLEARANCE NO.	BUILDING NUMBER	DRAWING NUMBER	REV
N/A	999-W	R-R1-A-00081	0
DESIGN AUTHORITY	DESIGN GROUP	SCALE	JOB NUMBER	SHEET
N/A	SRNL/ESE/RED&E	AS SHOWN	23435	1 OF 1

Figure H-4. HGRMA Vessel Assembly and Details

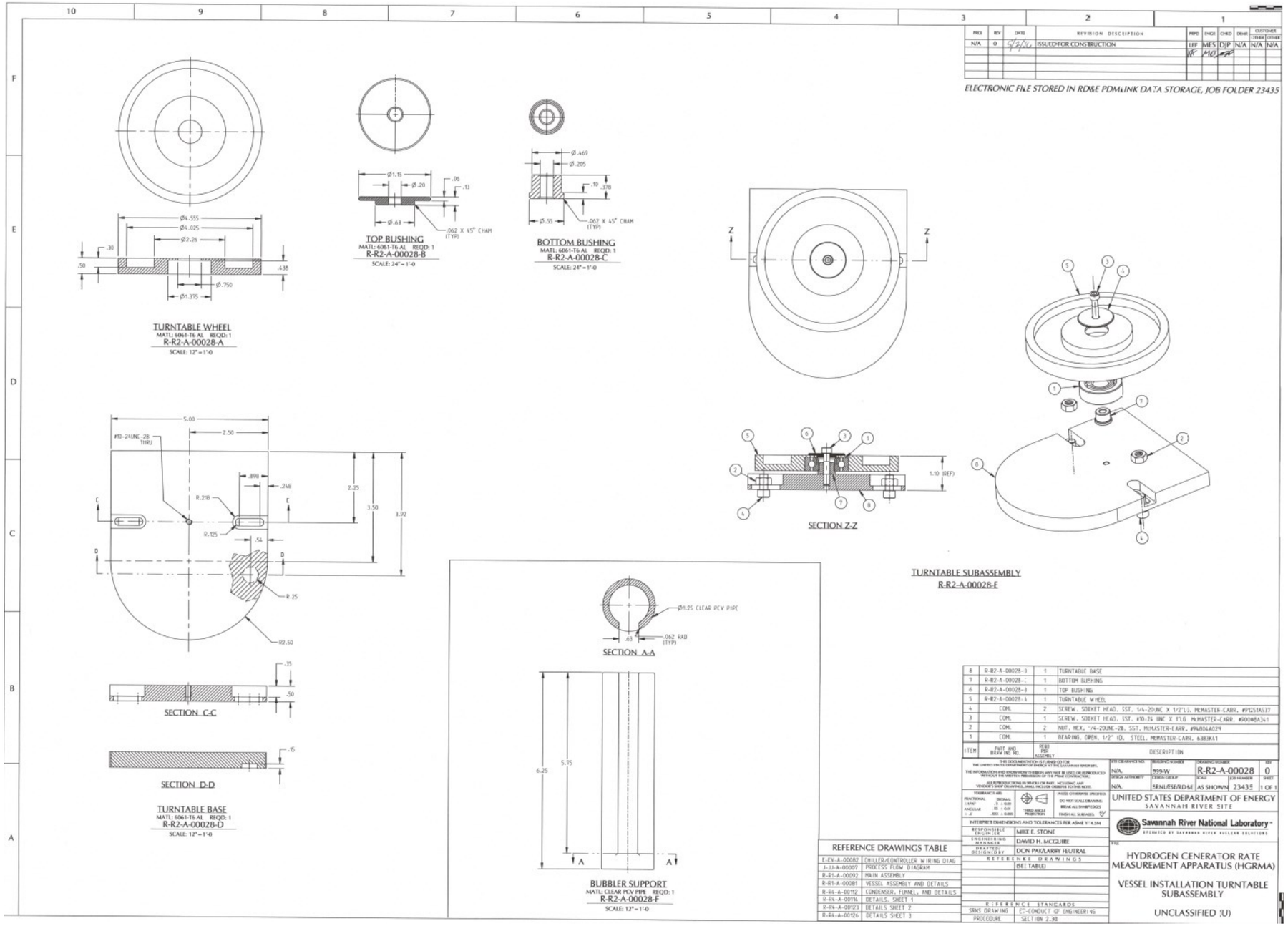


Figure H-5. Vessel Assembly Turntable

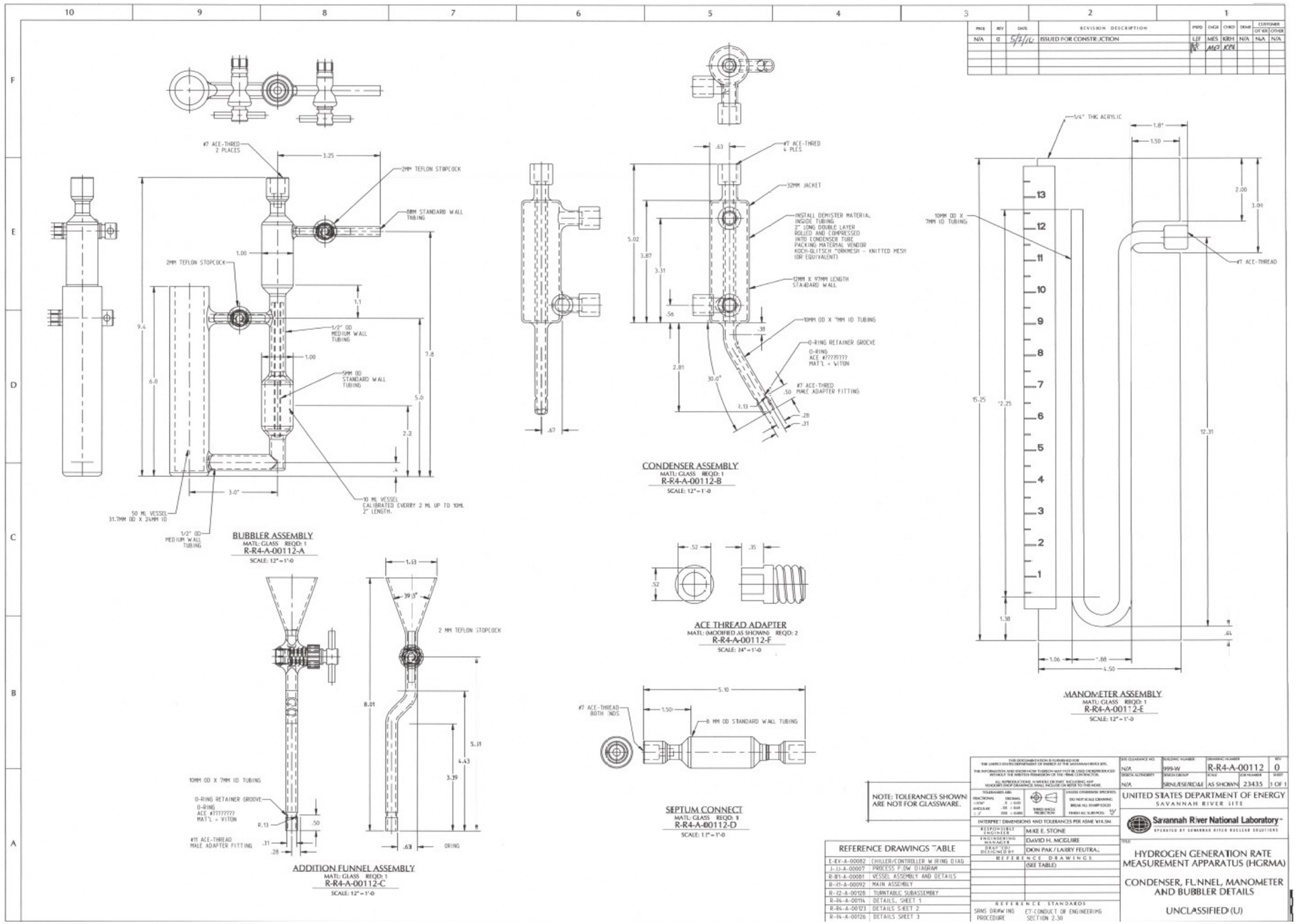


Figure H-6. HGRMA Glassware Details

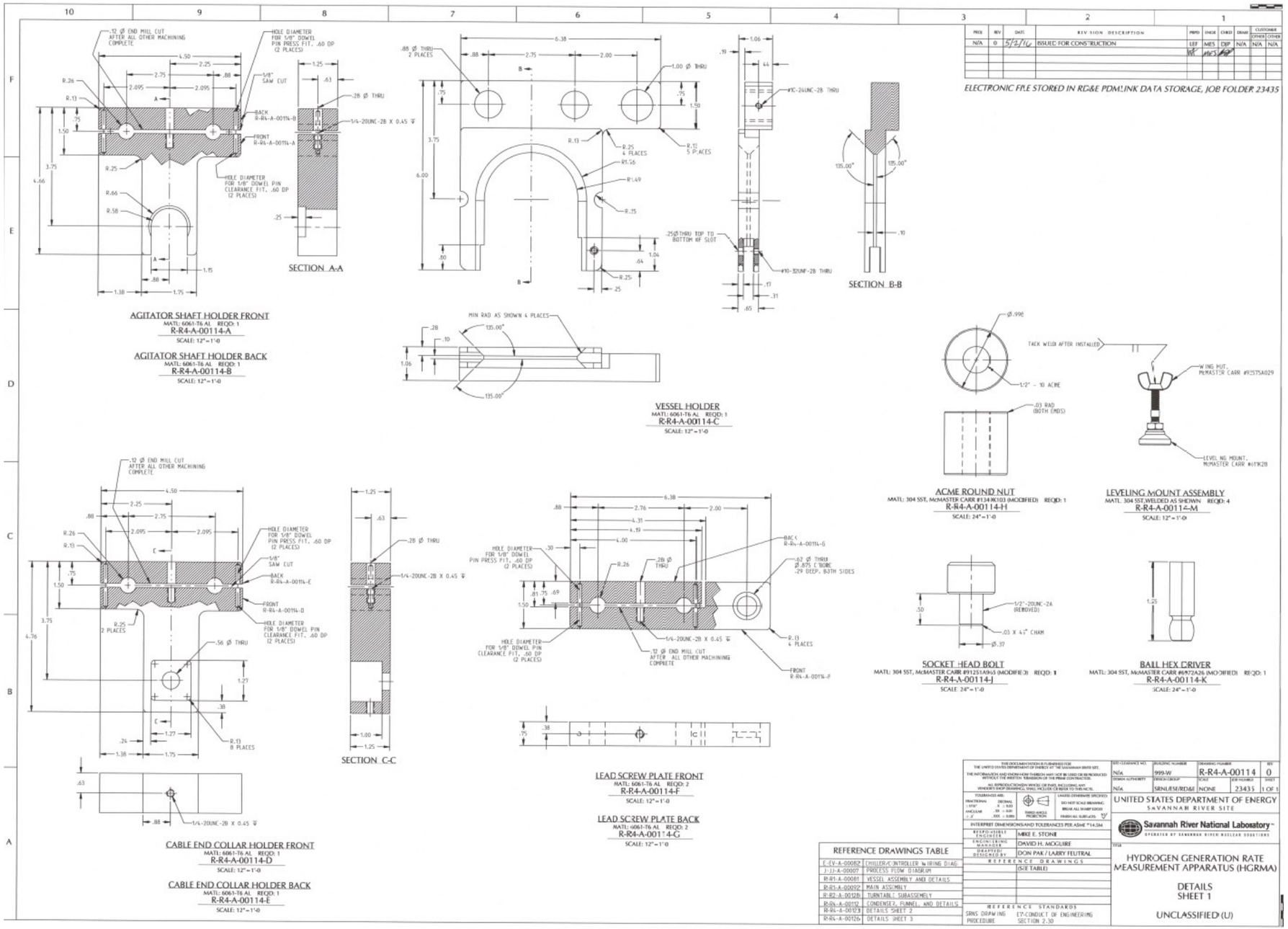


Figure H-7. HGRMA Stand Details Sheet One

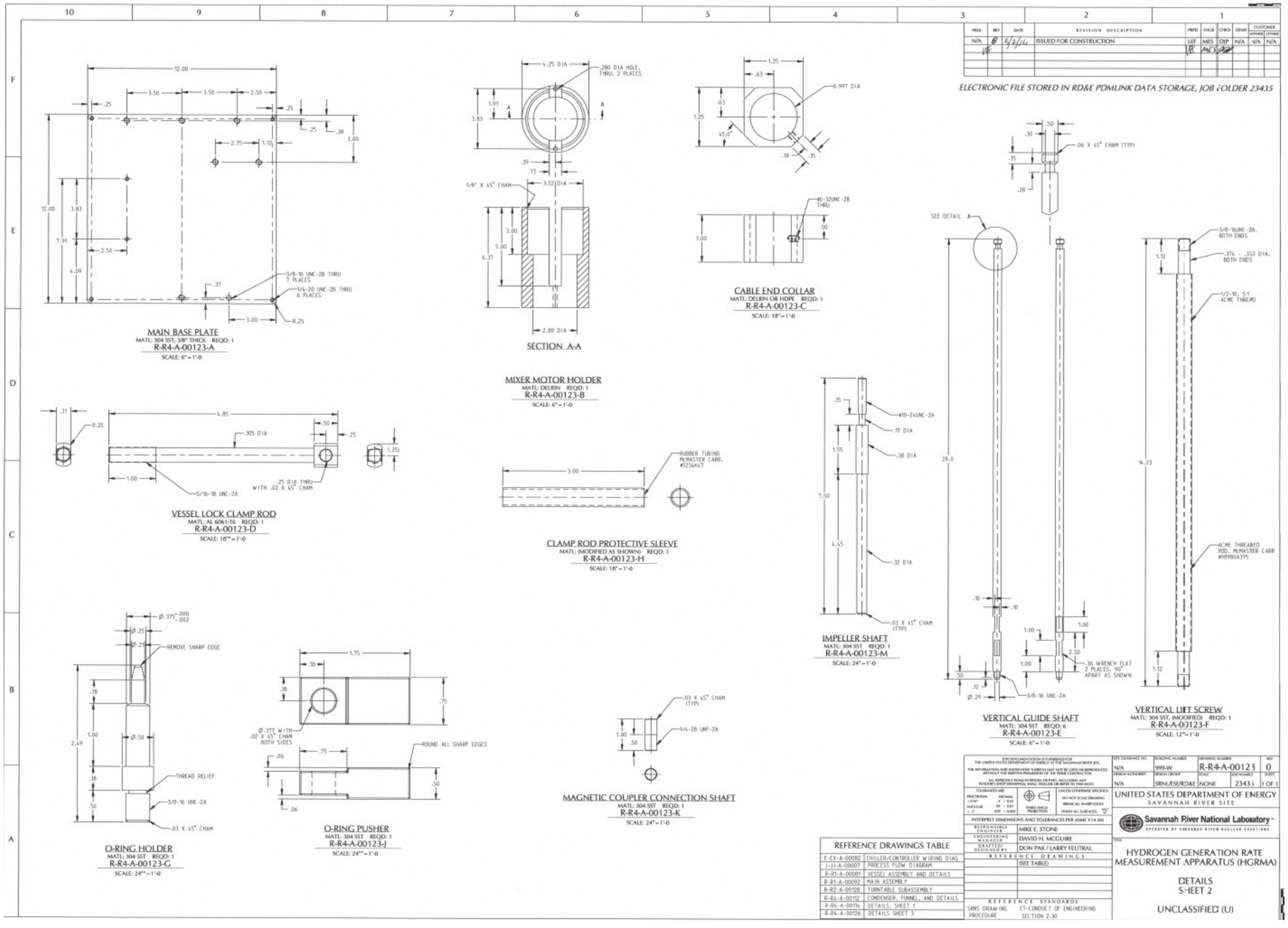
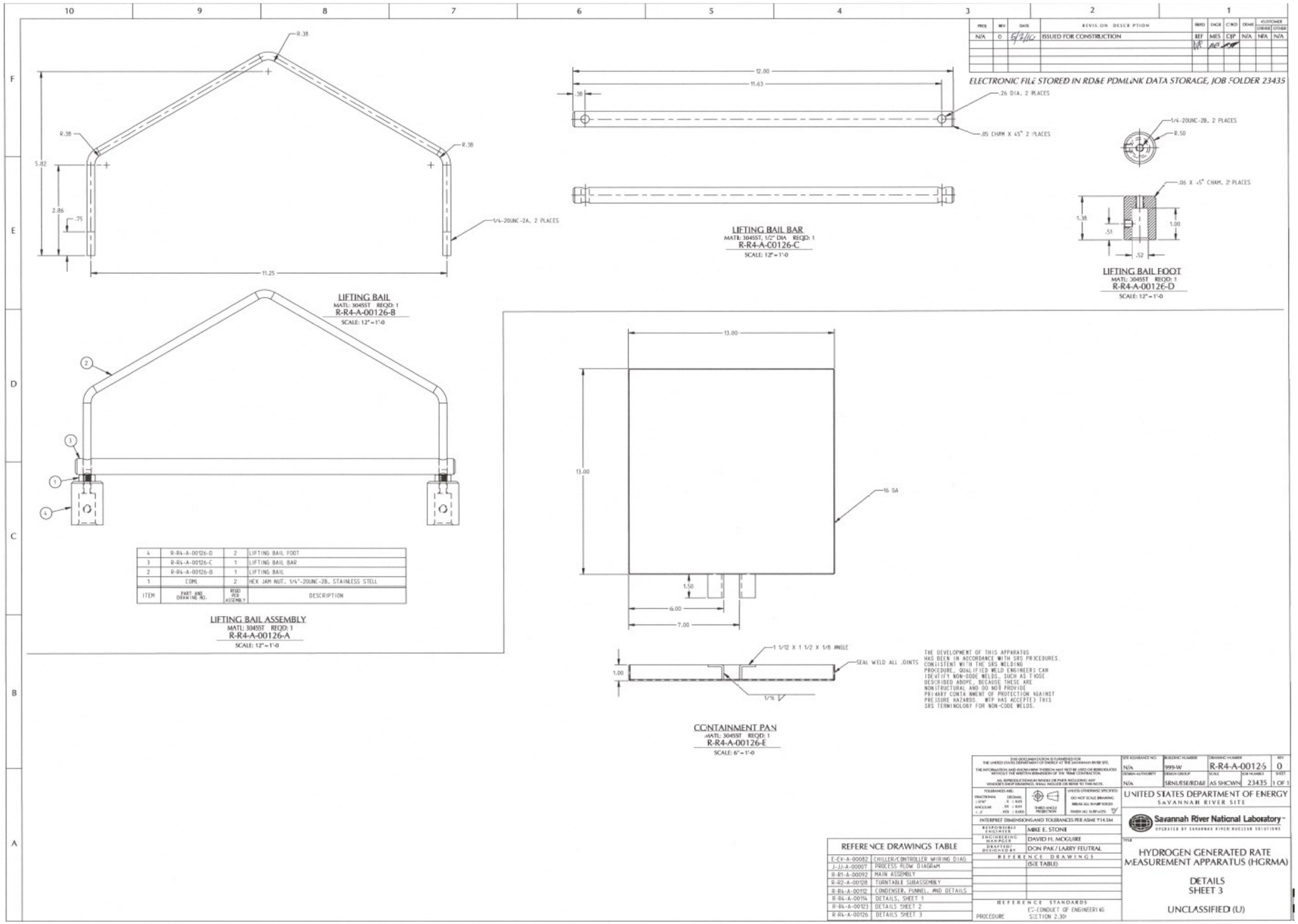


Figure H-8. HGRMA Stand Details Sheet 2



ITEM	PART AND DRAWING NO.	REQD PER ASSEMBLY	DESCRIPTION
4	R-R4-A-00126-D	2	LIFTING BAIL FOOT
3	R-R4-A-00126-C	1	LIFTING BAIL BAR
2	R-R4-A-00126-B	1	LIFTING BAIL
1	COML	2	HEX JAM NUT, 1/4"-20UNC-2B, STAINLESS STELL

REFERENCE DRAWINGS TABLE	
E-EV-A-00082	CHILLER/CONTROLLER WIRING DIAG
J-JJ-A-00007	PROCESS FLOW DIAGRAM
R-R1-A-00092	MAIN ASSEMBLY
R-R2-A-00128	TURNTABLE SUBASSEMBLY
R-R4-A-00112	CONDENSER, FUNNEL, AND DETAILS
R-R4-A-00114	DETAILS, SHEET 1
R-R4-A-00123	DETAILS SHEET 2
R-R4-A-00126	DETAILS SHEET 3

DES ASSURANCE NO.	N/A
BUILDING NUMBER	999-W
DRAWING NUMBER	R-R4-A-00125
REV	0
DESIGN AUTHORITY	N/A
DESIGN GROUP	SRNL/ESE/RD&E
SCALE	AS SHOWN
JOB NUMBER	23435
SHEET	1 OF 1

Figure H-9. HGRMA Stand Details Sheet Three

Appendix I. HGRMA Test Instructions

The instructions shown below are the detailed instructions used when performing the HGRMA tests at ACTL. These instructions were used as a guide when generating the Operating Instuctions shown in Appendix D.

1. This testing assumes that the required blending of simulants to prepare the melter feed test slurries has been performed as part of the Glass Fabrication Testing.
2. Ensure apparatus is assembled and leak checked per Assembly Instructions[34] and procedure ITS-0207[23].
3. Ensure M&TE calibrations are within date for the RTDs, flow controllers, and rotameter. Record M&TE information in "Run observations" section of data sheet.
4. Ensure mixer speed is checked with an M&TE tachometer before and after the testing each day.
5. Ensure Gas Chromatograph (GC) is calibrated as directed by Principal Investigator (PI). Calibration will be performed with hydrogen concentrations of 20 ppm and 5 ppm in the calibration gas.
6. Add sample to vessel
    a. Remove vessel head clamp
    b. Lower vessel as needed to allow sample to be added
    c. Add ~100 ml of sample to vessel as specified by PI
        i. Tare the addition bottle
        ii. Obtain ~ 125 ml of sample in bottle
        iii. Ensure sample in addition bottle is well mixed
        iv. Re-weigh addition bottle
        v. Add ~100 ml of sample to vessel
        vi. Re-weigh addition bottle
        vii. Calculate amount added
        viii. Ensure amount added is at least 95 ml and not greater than 125 ml. Note that the targeted amount is between 95 and 105 ml, but run will continue as long as amount does not exceed 125 ml.
        ix. If needed, allow solids to settle and collect supernate from vessel with a pipette to rinse solids from sides of vessel.
    d. Raise vessel and re-install head clamp
    e. Leak check vessel as specified by PI
7. Start purge on vessel as directed by PI
    a. Set air flowrate to 2± 0.1 sccm or as directed by PI
    b. Set calibration gas to 0± 0.1 sccm or as directed by PI
        i. If (sub) is specified, the calibration gas inlet will be adjusted to add the gas sub-surface as directed by PI.
    c. Turn on air and calibration gas flow
8. Turn on mixer to maintain a slight vortex or as directed by PI.
    a. Note any difficulty in keeping sample mixed during testing.
9. Start flow to vortex chiller
    a. Open air supply valve to rotameter
    b. Adjust vortex chiller rotameter to achieve 50± 5 lpm or as directed by PI
10. Heat sample to target temperature ± 1 degree Celsius (± 1.8 degree Fahrenheit)

a. Turn on water bath
b. Turn on mixer and adjust mixing speed to maintain a slight vortex
c. Set bath temperature to the maximum vessel temperature setpoint specified for the simulant being tested or as directed by PI
d. Adjust bath temperature as needed to achieve the required vessel temperature ± 1 degree Celsius (± 1.8 degree Fahrenheit) or as directed by PI

11. Measurement of offgas
    a. Begin automated reading of gas composition when directed by PI
    b. Monitor gas composition until steady state is reached as directed by PI
    c. Stop automated gas composition measurement when directed by PI
12. Repeat steps 7 through 10 at each of the temperatures and gas flow rates specified or as directed by PI.
13. Cool vessel to room temperature
    a. Set bath temperature to 70 degrees Fahrenheit
    b. Allow bath and vessel temperature to reach setpoint ± 5 degrees Celsius (± 9 degrees Fahrenheit)
14. Drain sample from vessel
    a. Open addition funnel valve to allow air inlet for draining
    b. Open drain valve and drain vessel contents into tared bottle
    c. Close drain valve and stop mixer
    d. Weigh bottle
    e. Record amount collected and label bottle with sample ID
15. Rinse vessel with water
    a. Open addition funnel and add 50 grams (± 3 grams) of DI water
        i. Tare addition bottle
        ii. Open bypass on offgas flow bubbler.
        iii. Add 50 grams of water to bottle and reweigh
        iv. Add water to vessel
        v. Close bypass on offgas flow bubbler
        vi. Reweigh addition bottle
    b. Start mixer at 300 rpm ± 10 rpm or as directed by PI
    c. Allow rinse to mix for at least one minute
    d. Open addition funnel valve to allow air inlet for draining
    e. Drain rinse into a tared bottle using instructions in step “14”
    f. Record amount collected and label bottle with sample ID
16. Rinse vessel with 50% Nitric Acid
    a. Open addition funnel and add 50 grams (± 3 grams) of 50% nitric acid, as directed by PI
        i. Tare addition bottle
        ii. Add 50 grams of nitric acid to bottle and reweigh
        iii. Open bypass on offgas flow bubbler
        iv. Add acid to vessel
        v. Close bypass on offgas flow bubbler
        vi. Reweigh addition bottle
    b. Start mixer at 300 rpm ± 10 rpm or as directed by PI
    c. Allow acid to mix for at least 10 minutes

   d. Open addition funnel valve to allow air inlet for draining
   e. Drain rinse into a tared bottle using instructions in step “14”
   f. Record amount collected and label bottle with sample ID
17. Rinse vessel with water per step “15”
18. Inspect glassware for cleanliness, etching, or cracks
19. If specified by PI, repeat step “16” to collect samples to determine amount of residual material in vessel. Label and submit samples as specified by PI.
20. Ensure a post calibration is performed on the GC at the conclusion of each work shift.
21. Once the run is complete, the PI will compile all test results, ensure the GC data is reviewed and corrected as needed, the rate of hydrogen generation is calculated and compared to action limits, and perform an evaluation of the performance of the HGRMA.

Appendix J. Supplemental Temperature Testing

After the simulant verification testing was completed, tests were conducted with water to show that the HGRMA could maintain the required temperatures using a neoprene insulating wrap. Two tests were completed: an insulated test at 140 degrees Fahrenheit and uninsulated and insulated tests targeting 194 degrees Fahrenheit. The results of the tests at 140 degrees Fahrenheit are shown in Figure J-1, with the uninsulated vessel reaching and holding the temperature target in approximately 25 minutes.

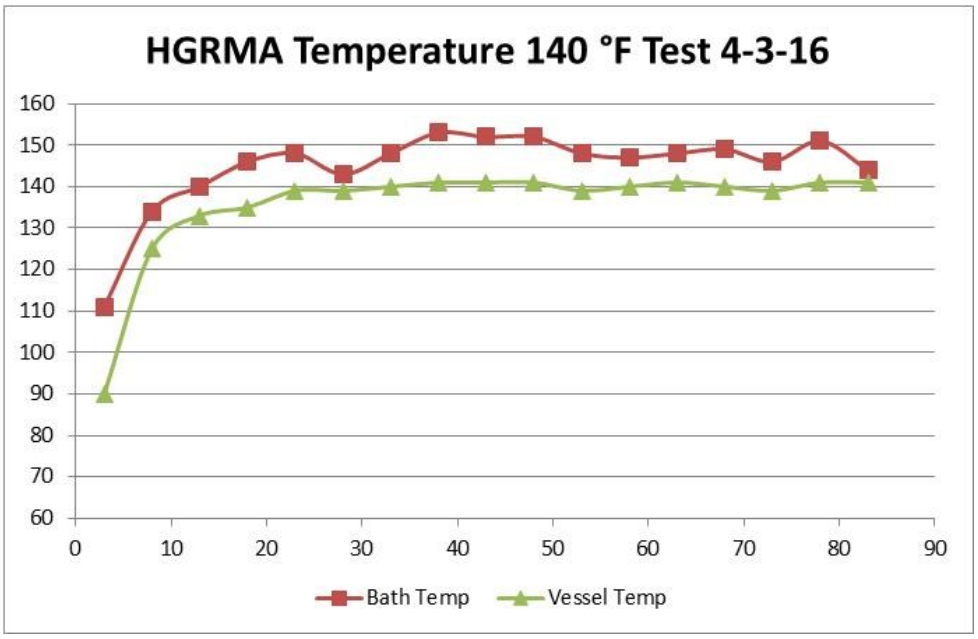


Figure J-1. Supplemental Temperature Test at 140 °F

The water bath set-point was changed to 206 degrees Fahrenheit, but the bath could not reach set-point without insulation on the vessel. After insulating the vessel at run time of 55 minutes and lid at run time of 70 minutes, the bath set-point was increased to 211 degrees Fahrenheit and the vessel reached the targeted temperature and maintained the target temperature plus or minus 2 degrees Fahrenheit, as shown in Figure J-2.

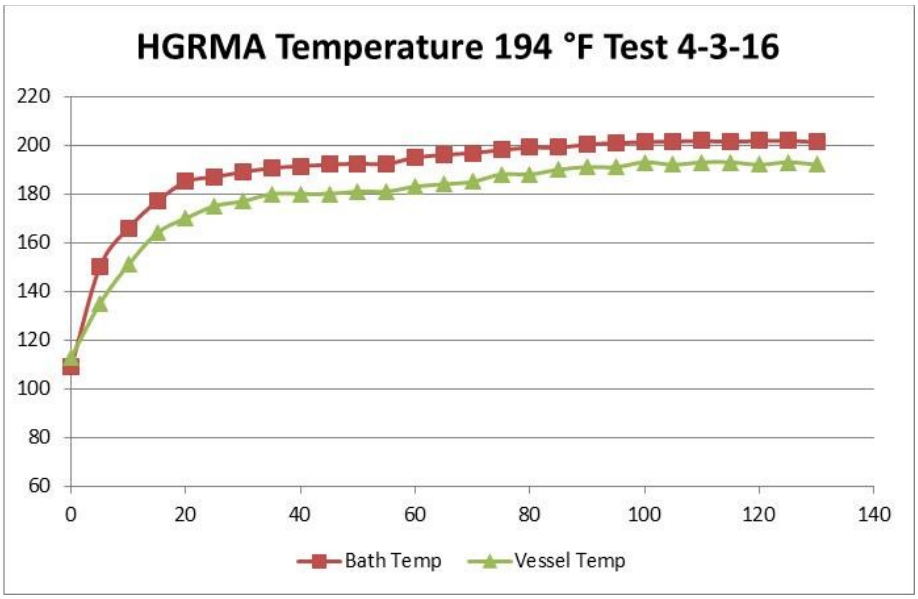


Figure J-2. Supplemental Temperature Test at 194 °F

The insulating sleeves used are shown in Figure J-3, and are similar to sleeves used on the 1 Liter DWPF chemical process vessel in the SRNL Shielded Cells. Tabs were not added or shown in the Figure, but remote removal and installation of similar sleeves has been performed with little difficulty by adding tabs to the sleeves to facilitate remote handling.

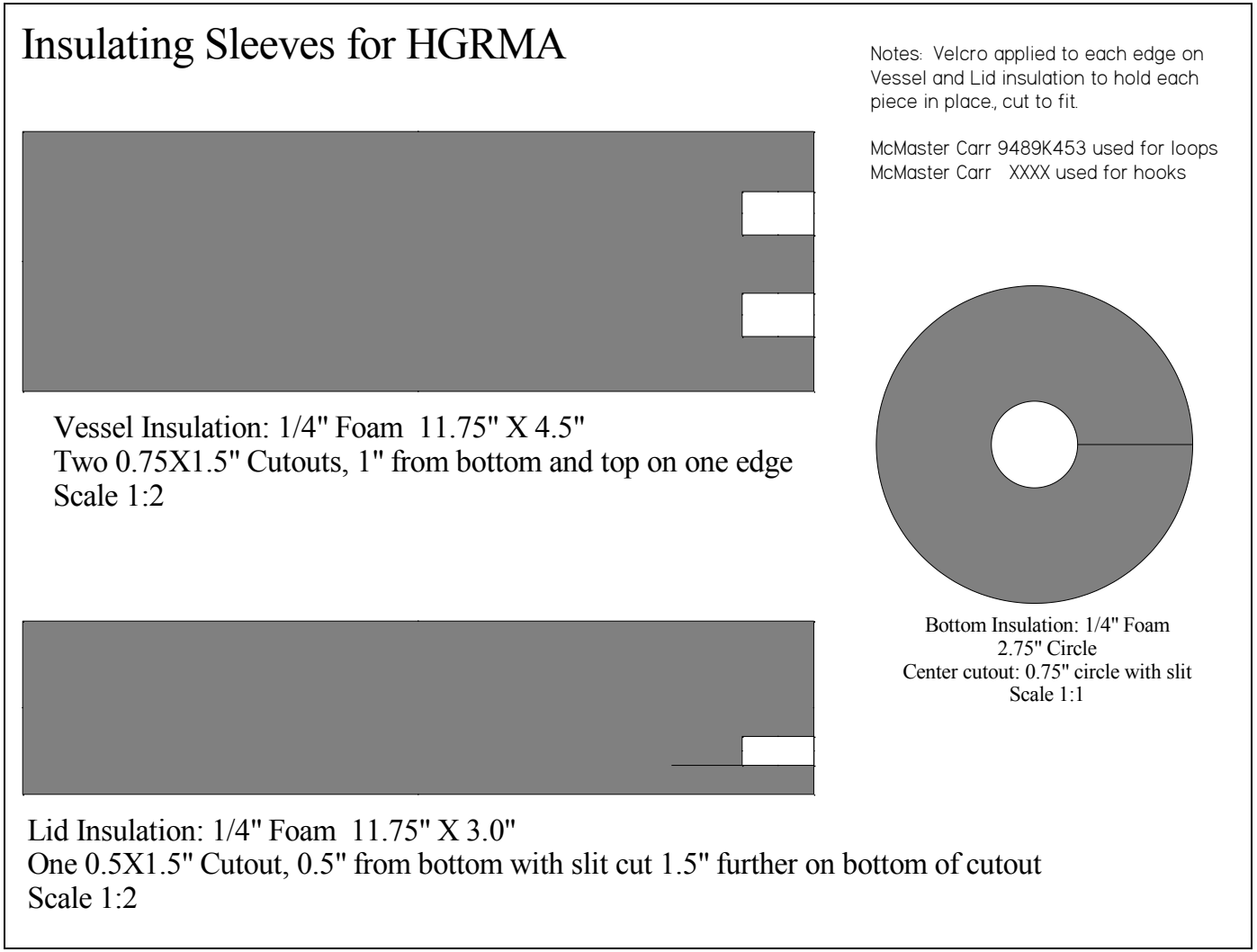


Figure J-3. HGRMA Neoprene Insulating Sleeve Dimensions

Appendix K. Simulant Recipes

LAW Simulant

The LAW simulant is a ~ 3 molar (M) sodium (Na) simulant that will be used to prepare both the low bound and high bound LAW melter feeds. This recipe was specified by WTP and prepared by NOAH Technologies Corporation. Note that preparation steps (such as addition order, wait times, etc.) and specific item number have been removed from the recipe as shown in Table K-1.

Table K-1. Recipe for LAW Simulant

Formula	Molecular Weight	grams/Liter
H_2O	18.0153	111.94
$NaCH_3COO\ (H_2O)_3$	136.0796	2.02
$Na_2C_2O_4$	133.9985	1.43
$Al(NO_3)_3$ (59.86% Solution)	212.9962	97.28
NaCl	58.44277	1.43
$NaH_2PO_4 \cdot H_2O$	137.9923	1.03
Na_2SO_4	142.0421	3.18
$NaNO_3$	84.99467	36
KNO_3	101.1032	12.01
NaOH (50.20% Solution)	39.99711	142.47
Na_2SiO_3	122.0632	0.32
HCOONa	68.00721	0.97
$NaNO_2$	68.99527	29.27
Na_2CO_3	105.9884	12.02
K_2CO_3	138.2055	21.31
H_2O	18.0153	684.9
Total		1157.58

HLW Low Bound Simulant

The HLW Low Bound simulant was specified by WTP and prepared by NOAH Technologies Corporation. Note that preparation steps (such as addition order, wait times, etc.) have been removed from the recipe and that the specific form of the compound (particle size, etc.) has been omitted from Table K-2.

Table K-2. Recipe for HLW Low Bound Simulant

Formula	Molecular Weight	grams/Liter
H_3BO_3	61.83302	0.084
NaCl	58.44277	0.187
NaF	41.98817	0.139
Na_2SO_4	142.0421	0.576
NaOH (50.2 % Solution)	39.99711	12.266
$Na_3PO_4 \cdot (H_2O)_{12}$	380.1241	8.942
$Na_2C_2O_4$	133.9985	0.128
Na_2CO_3	105.9884	6.377
$NaNO_3$	84.99467	0.483
KNO_2	85.1038	0.704
$NaNO_2$	68.99527	1.192
Fe_2O_3	159.6882	63.41
$Al(OH)_3$	78.00356	63.27
SiO_2	60.0843	6.12
$Zr(OH)_4$	159.2534	24.89

To the above, 26.025 grams/liter of the Cesium ion exchange (IX) Concentrate[13] was added to complete the recipe.

HLW High Bound Simulant

The HLW High Bound simulant was specified by WTP and prepared by NOAH Technologies Corporation. Note that preparation steps (such as addition order, wait times, etc.) have been removed from the recipe and that the specific form of the compound (particle size, etc.) has been omitted from Table K-3.

Table K-3. Recipe for HLW High Bound Simulant

Formula	Molecular Weight	grams/Liter
H_3BO_3	61.83302	0.073
NaCl	58.44277	0.162
NaF	41.98817	0.121
Na_2SO_4	142.0421	0.498
NaOH	39.99711	10.604
$Na_3PO_4 \cdot (H_2O)_{12}$	380.1241	7.73
$Na_2C_2O_4$	133.9985	0.11
Na_2CO_3	105.9884	5.513
$NaNO_3$	84.99467	0.417
KNO_2	85.1038	0.609
$NaNO_2$	68.99527	1.031
Fe_2O_3	159.6882	192.79
$Al(OH)_3$	78.00356	192.38
SiO_2	60.0843	18.61
$Zr(OH)_4$	159.2534	75.66

To the above, 72.55 grams/liter of the Cesium ion exchange (IX) Concentrate[13] was added to complete the recipe.

The rheological properties of the NOAH provided HLW High Bound simulant did not meet the targeted yield stress for the HLW-HB melter feed. As a result, SRNL prepared this simulant recipe using chemicals remaining from the development program for the recipes used by WTP to prepare the HLW simulants using the same recipe as above.

LAW Low Bound GFCs

The recipe for the LAW Low Bound Melter Feed specifies that the following GFCs be added to one liter of LAW simulant. Note that the GFCs were blended in the ratio below by NOAH Technologies Corporation. Therefore, 419 grams of the blended GFCs were added to a liter of LAW simulant during the testing, as shown in Table K-4.

Table K-4. Recipe for LAW Low Bound GFCs

Formula	Molecular Weight	grams/Liter
Al_2SiO_5	162.0456	35.67
H_3BO_3	61.83302	87.65
$CaSiO_3$	116.1617	21.16
Fe_2O_3	159.6882	27.21
Mg_2SiO_4	140.6931	15.5
SiO_2	60.0843	183.63
TiO_2	79.8658	10.52
ZnO	81.40839	14.99
$ZrSiO_4$	183.3071	22.67
Total		419

LAW High Bound GFCs

The recipe for the LAW High Bound Melter Feed specifies that the following GFCs be added to one liter of LAW simulant. Note that the GFCs were blended in the ratio below by NOAH Technologies Corporation. Therefore, 1,949.62 grams of the blended GFCs were added to a liter of LAW simulant during the testing, as shown in Table K-5.

Table K-5. Recipe for LAW High Bound GFCs

Formula	Molecular Weight	grams/Liter
Al_2SiO_5	162.0456	200.45
H_3BO_3	61.83302	87.65
$CaSiO_3$	116.1617	118.89
Fe_2O_3	159.6882	152.88
Mg_2SiO_4	140.6931	87.09
SiO_2	60.0843	1031.93
TiO_2	79.8658	59.11
ZnO	81.40839	84.23
$ZrSiO_4$	183.3071	127.39
Total		1,949.62

HLW Low Bound GFCs

The recipe for the HLW Low Bound Melter Feed specifies that the following GFCs be added to one liter of HLW Low Bound simulant. Note that the GFCs were blended in the ratio below by NOAH Technologies Corporation. Therefore, 429.74 grams of the blended GFCs were added to a liter of HLW Low Bound simulant during the testing, as shown in Table K-6.

Table K-6. Recipe for HLW Low Bound GFCs

Formula	MW (grams)	g/L
$Na_2B_4O_7 \cdot (H_2O)_{10}$	381.3721	136.98
Li_2CO_3	73.8909	44.86
Na_2CO_3	105.9884	31.17
SiO_2	60.0843	207.2
ZnO	81.40839	9.53
Total		429.74

HLW High Bound GFCs

The recipe for the HLW High Bound Melter Feed specifies that the following GFCs be added to one liter of HLW High Bound simulant. Note that the GFCs were blended in the ratio below by NOAH Technologies Corporation. Therefore, 1,202.7 grams of the blended GFCs were added to a liter of HLW High Bound simulant during the testing. Note that the GFC total amount is changed versus the Low Bound HLW melter feed, but the GFC formula is otherwise unchanged. Thus, the same blend of GFCs from NOAH Technologies Corporation was used for both HLW melter feeds, as shown in Table K-7.

Table K-7. Recipe for HLW High Bound GFCs

Formula	MW (grams)	g/L
$Na_2B_4O_7 \cdot (H_2O)_{10}$	381.3721	383.38
Li_2CO_3	73.8909	125.54
Na_2CO_3	105.9884	87.22
SiO_2	60.0843	579.9
ZnO	81.40839	26.66
Total		1202.7

Distribution:

T. B. Brown, 773-A
M. E. Cercy, 773-42A
D. A. Crowley, 773-43A
D. E. Dooley, 773-A
A. P. Fellinger. 773-42A
S. D. Fink, 773-A
C. C. Herman, 773-A
D. T. Hobbs, 773-A
E. N. Hoffman, 999-W
J. E. Hyatt, 773-A
K. M. Kostelnik, 773-42A
B. B. Looney, 773-42A
D. A. McGuire, 773-42A
T. O. Oliver, 773-42A
F. M. Pennebaker, 773-42A
G. N. Smoland, 773-42A
B. J. Wiedenman, 773-42A
W. R. Wilmarth, 773-A
J. D. Newell, 999-W
J. M. Pareizs, 773-A
T. E. Smith, 999-W
H. H. Burns, 773-41A
A. D. Cozzi, 999-W
D. J. McCabe, 773-42A
E. K. Hansen, 999-W
J. E. Hyatt, 773-A
K. M. Fox, 999-W
M. R. Poirier, 773-42A
S. D. Fink, 773-A
M. A. Reigel, 773-42A

V. Jain, 766-H
R. E. Edwards, 766-H
E. J. Freed, 704-S
J. F. Iaukea, 704-S
J. S. Contardi, 704-56H
T. L. Fellinger, 766-H
R. T. McNew, 766-H

Records Administration (EDWS)