ENGINEERING CHANGE NOTICE

Page 1 of 2

1. ECN No 624777

Proj.
ECN

2. ECN Category (mark one) Supplemental ☐ Direct Revision ☒ Change ECN ☐ Temporary ☐ Standby ☐ Supersedeure ☐ Cancel/Void ☐	3. Originator's Name, Organization, MSIN, and Telephone No. Norman Scott Cannon, 8A200 L6-38, 376-0562	3a. USD Required? ☐ Yes ☒ No	4. Date 06/24/96
	5. Project Title/No./Work Order No. Retained Gas Sampler System Acceptance Test Report/N2155	6. Bldg./Sys./Fac. No. 200-G	7. Approval Designator Q
	8. Document Numbers Changed by this ECN (includes sheet no. and rev.) WHC-SD-WM-ATR-137, Rev. 0	9. Related ECN No(s). N/A	10. Related PO No. N/A
11a. Modification Work ☐ Yes (fill out Blk. 11b) ☒ No (NA Blks. 11b, 11c, 11d)	11b. Work Package No. N/A	11c. Modification Work Complete N/A Cog. Engineer Signature & Date	11d. Restored to Original Condition (Temp. or Standby ECN only) N/A Cog. Engineer Signature & Date

12. Description of Change

The document WHC-SD-WM-ATP-137, Rev. 0 was revised to produce WHC-SD-WM-ATP-137, Rev. 1. Major changes resulting from the revision was the inclusion of an additional long term sampler aging test finished after the original document was complete; also the original Appendix F (Two Volume Method Ammonia Vapor Pressure Approximation) was changed to Appendix G, and a new Appendix F created dealing with hydrogen seal penetration.

13a. Justification (mark one)

Criteria Change ☐	Design Improvement ☐	Environmental ☐	Facility Deactivation ☐
As-Found ☐	Facilitate Const ☒	Const. Error/Omission ☐	Design Error/Omission ☐

13b. Justification Details

This revision adds addition sampler aging test data analysis and conclusions not available at the time the original document was completed due to the necessary long term nature of the test.

14. Distribution (include name, MSIN, and no. of copies)

Norman Scott Cannon, L6-38 (5)
 Ronald Craig Knight, L6-38 (3)
 See attached Distribution Sheet

JUL 18 1996		RELEASE STAMP
DATE:	HANFORD	20
STA: 37	RELEASE	
		ID:

ENGINEERING CHANGE NOTICE

Page 2 of 2

1. ECN (use no. from pg. 1)

624777

15. Design Verification Required

☐ Yes
☒ No

16. Cost Impact

ENGINEERING

Additional ☐ [N] \$
Savings ☐ [N] \$

CONSTRUCTION

Additional ☐ [N] \$
Savings ☐ [N] \$

17. Schedule Impact (days)

Improvement ☐ [N]
Delay ☐ [N]

18. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 12. Enter the affected document number in Block 19.

SDD/DD	☐	Seismic/Stress Analysis	☐	Tank Calibration Manual	☐
Functional Design Criteria	☐	Stress/Design Report	☐	Health Physics Procedure	☐
Operating Specification	☐	Interface Control Drawing	☐	Spares Multiple Unit Listing	☐
Criticality Specification	☐	Calibration Procedure	☐	Test Procedures/Specification	☐
Conceptual Design Report	☐	Installation Procedure	☐	Component Index	☐
Equipment Spec.	☐	Maintenance Procedure	☐	ASME Coded Item	☐
Const. Spec.	☐	Engineering Procedure	☐	Human Factor Consideration	☐
Procurement Spec.	☐	Operating Instruction	☐	Computer Software	☐
Vendor Information	☐	Operating Procedure	☐	Electric Circuit Schedule	☐
OM Manual	☐	Operational Safety Requirement	☐	ICRS Procedure	☐
FSAR/SAR	☐	IEFD Drawing	☐	Process Control Manual/Plan	☐
Safety Equipment List	☐	Cell Arrangement Drawing	☐	Process Flow Chart	☐
Radiation Work Permit	☐	Essential Material Specification	☐	Purchase Requisition	☐
Environmental Impact Statement	☐	Fac. Proc. Samp. Schedule	☐	Tickler File	☐
Environmental Report	☐	Inspection Plan	☐		☐
Environmental Permit	☐	Inventory Adjustment Request	☐		☐

19. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision

Document Number/Revision

Document Number/Revision

WHC-SD-WM-ATR-137

Revision 0

20. Approvals

Signature

Date

Signature

Date

OPERATIONS AND ENGINEERING

Cog. Eng. N. S. Cannon *N S Cannon*

7-2-96

Cog. Mgr. J. R. Thielges *J R Thielges*

7/3/96

QA B. J. Hug *B J Hug*

07/05/96

Safety

Environ.

Other R. C. Knight *R C Knight*

7/2/96

R. E. Bauer *R E Bauer*

7/2/96

ARCHITECT-ENGINEER

PE

QA

Safety

Design

Environ.

Other

DEPARTMENT OF ENERGY

Signature or a Control Number that tracks the Approval Signature

ADDITIONAL

RETAINED GAS SAMPLER SYSTEM ACCEPTANCE TEST REPORT

N. S. Cannon

Westinghouse Hanford, Richland, WA 99352

U.S. Department of Energy Contract DE-AC06-87RL10930

EDT/ECN: ECN 624777

UC: 510

Org Code: 8A200

Charge Code: N2155

B&R Code: EW3120072

Total Pages: 116

Key Words: Retained Gas Sampler, Extractor, Extruder, Gas Chromatograph, Calibration, Spectrometer, Pressure Transducer, Thermocouple

Abstract: Acceptance test results for the Retained Gas Sampler System (RGSS) obtained in the 306E laboratory are reported. The RGSS will be utilized to retrieve and analyze samples from the Hanford flammable gas watch-list tanks to determine the quantity and chemistry of gases confined within the waste.

TRADEMARK DISCLAIMER. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors.

Printed in the United States of America. To obtain copies of this document, contact: WHC/BCS Document Control Services, P.O. Box 1970, Mailstop H6-08, Richland WA 99352, Phone (509) 372-2420; Fax (509) 376-4989.

Jamie Bishop 7-18-96
Release Approval Date

Release Stamp

Approved for Public Release

**RETAINED GAS SAMPLER SYSTEM
ACCEPTANCE TEST REPORT**

N. S. Cannon
R. C. Knight

July 1996

CONTENTS

	Page
1.0 Introduction	5
1.1 Purpose	5
1.2 Scope	5
2.0 References	5
3.0 Responsibilities	6
4.0 Description of System	6
5.0 Test Conditions and Data Analysis	7
5.1 Test Facility	7
5.2 Data Acquisition and Control Software	7
5.3 Instrument Calibration	7
5.4 Preoperational Leak Test	8
5.5 Test Data	8
5.5.1 Gas Composition Analysis	8
5.5.1.1 <u>Insoluble Gas Analysis</u>	9
5.5.1.2 <u>Ammonia Solution Analysis (ASA)</u>	9
5.5.2 Gas Quantity Determination	9
5.5.2.1 <u>Back Fill Method</u>	9
5.5.2.2 <u>Hg Pump Method</u>	10
5.5.2.3 <u>Two Volume Method</u>	11
6.0 Safety	11
7.0 Acceptance Test Results	12
7.1 Hardware Setup/Volume Determinations	12
7.1.1 2nd Volume Measurement	12
7.1.1.1 <u>Test 1</u>	13
7.1.1.2 <u>Test 2</u>	13
7.1.1.3 <u>Test 3</u>	13
7.1.2 Extractor Volume Measurement	14
7.1.3 Sampler Volume Measurement	15
7.1.4 Extractor/Sampler Integration Test	15
7.2 Gas Injection Tests	16
7.2.1 Required Injection Tests	17
7.2.1.1 <u>Nitrous Oxide Gas Mix Analysis</u>	17
7.2.1.2 <u>Hydrogen/Ammonia Gas Mix Analysis</u>	17
7.2.1.3 <u>Results Summary for Required Injection Tests</u>	18
7.2.2 Additional Injection Tests	19
7.2.2.1 <u>Ammonia Solution Analysis (ASA)</u>	20
7.2.2.2 <u>RGSS Sampler Leakage</u>	21
7.2.2.3 <u>Water Degassing</u>	22
7.2.2.4 <u>Wet Gas Mix Injection Test Results</u>	22
7.2.2.5 <u>Wet Argon Gas Injection Test Results</u>	23
7.2.2.5.1 <u>Composition Results</u>	23
7.2.2.5.2 <u>Gas Quantity Results</u>	24

CONTENTS (Cont.)

	Page
7.3 Simulant Extrusion Tests	25
7.4 Sampler Aging Tests	26
7.4.1 Hydrogen Seal Permeation	27
7.4.2 Sampler In-Leakage	28
7.4.3 Sampler Out-Leakage	28
8.0 Exceptions to ATP	29
9.0 Summary	31
9.1 Testing and Analysis Description	31
9.2 Acceptance Test Results	32
9.3 Conclusions	33
10.0 Acknowledgements	34

Appendixes

A	RGSS Instrumentation Calibration	A-1
B	Ammonia Solution Analysis (ASA)	B-1
C	RGSS Raw Data Sheets	C-1
D	Adjusting Gas Composition for Leakage or Ammonia Washout	D-1
D.1	Leakage/Outgassing	D-1
D.1.1	Oxygen Tracer	D-1
D.1.2	Argon Tracer	D-1
D.2	Ammonia Washout	D-2
E	Acceptance Test Summary	E-1
F	Hydrogen Permeation Rate for Retained Gas Sampler	F-1
G	Two Volume Method Ammonia Vapor Pressure Approximation	G-1
G.1	Ammonia Vapor Pressure Variation on Volume Expansion	G-1
G.2	Ammonia Vapor Pressure Variation Effects on Two Volume Method Results	G-2
G.2.1	Insoluble Gas Quantity Error	G-3
G.2.2	Ammonia Vapor Pressure Error	G-5

Figures

1	Retained Gas Sampling System - 306E Layout	56
2	Extractor and 2nd Volume Details	57
3	Gas Composition Error Analysis	58
4	Gas Quantity Error Analysis	59
5	Gas Composition Difference (Measured-Expected) Is Plotted Versus Sampler Aging Time For Hydrogen, Argon and Nitrogen	60
G-1	Variation of P_{v1}/P_{v2} versus H/m_L	G-7

Tables

1	NDE Leak Test Procedure and Report: 6-26-95	35
2	NDE Leak Test Procedure and Report: 8-14-95	36
3	Extractor Volume Measurement Table	37

CONTENTS (Cont.)

	<u>Tables (cont.)</u>	Page
4	Cabinet Volume Measurements	38
5	Determination of New Line Volume (7-26-95)	39
6	Determination of New Extractor Volume (8-15-95)	39
7	Extractor/Sampler Integration Test	40
8	Gas MIX#1 Certificate of Analysis	41
9	Gas MIX#2 Certificate of Analysis	42
10	Injection Test RGS-001 Data Sheet	43
11	Injection Test RGS-003 Data Sheet	44
12	Summary of Required Injection Test Results	45
13	Injection Test RGS-005 Data Sheet	46
14	Injection Test RGS-006 Data Sheet	47
15	Ammonia Solution Analysis (ASA) on MIX#1 Control Samples	48
16	Injection Tests - MIX#1 Ammonia Analysis	49
17	Wet (Degassed) Mixed Gas Injection Test Composition Results	50
18	Wet (Degassed) Mixed Gas Injection Test Quantity Results	50
19	Wet (Degassed) Argon Gas Injection Test Composition Results	51
20	Wet (Degassed) Argon Gas Injection Test Quantity (@STP) Results	52
21	Simulant Extrusion Test Gas Quantity Results	53
22	Simulant Extrusion Test Gas Composition Results	54
23	Sampler Aging Test Gas Composition Results	55
A-1	Initial (9-1-94) Calibration of MKS 10 Torr Pressure Transducer	A-3
A-2	Final (9-15-95) Calibration of MKS 10 Torr Pressure Transducer	A-4
A-3	Initial (9-1-94) Calibration of MKS 100 Torr Pressure Transducer	A-5
A-4	Final (9-15-95) Calibration of MKS 100 Torr Pressure Transducer	A-6
A-5	Initial (9-1-94) Calibration of MKS 1000 Torr Pressure Transducer	A-7
A-6	Final (9-14-95) Calibration of MKS 1000 Torr Pressure Transducer	A-8
A-7	HP-3497A DACU Calibration (5-30-95)	A-9
A-8	RGSS Type K Thermocouple Data	A-10
A-9	Out-of-Specification Pressure Transducer Calibration Results	A-10
A-10	Potential Pressure Error Effect on Hg Pump Measured Gas Quantity	A-11
B-1	Preliminary MIX#1 Ammonia D-Series Test Results	B-3

RETAINED GAS SAMPLER SYSTEM ACCEPTANCE TEST REPORT

N. S. Cannon
R. C. Knight

1.0 INTRODUCTION

1.1 PURPOSE

This report documents the results of acceptance testing performed on and with the Retained Gas Sampler System (RGSS) in the 306E laboratory, prior to shipment of the system to the 222S laboratory for installation and field use. The RGSS shall be used in support of the mitigation of Hanford flammable gas watch-list (FGWL) waste tanks. Acceptance testing was performed in accordance with the Retained Gas Sampler System Acceptance Test Procedure (WHC-SD-WM-ATP-137).

1.2 SCOPE

The objective of the acceptance testing was to perform testing in accordance with WHC-SD-WM-ATP-137 (ATP) to verify that the features of the RGSS performed as designed and that the system is ready for field service. Testing was to verify that the system meets the functional design criteria specified in WHC-SD-WM-FRD-018. This testing addressed all components of the RGSS with the exception of the extruder, which is addressed in WHC-SD-WM-ATP-495 and WHC-SD-WM-ATR-495. Test results, including exceptions to the ATP are documented in this test report.

This report (WHC-SD-ATR-137) of the RGSS acceptance test results generally follows the ATP format, heading for heading, allowing for a direct comparison between acceptance test requirements and acceptance test results. However, *the authors note that a relatively concise summary of the test results is given in Section 9.0 of this report.*

2.0 REFERENCES

The following documents are listed as references for the acceptance testing activities:

1. WHC-SD-WM-ATP-137, "Retained Gas Sampler System Acceptance Test Procedure."

2. WHC-SD-WM-FRD-018, "Functional Design Criteria for the Retained Gas Sampler System."
3. WHC-SD-WM-TRP-225, "Test Results of the Phase I Test Plan."
4. WHC-SD-WM-ETP-144, "Retained Gas Sampler Engineering Task Plan."
5. WHC-SD-WM-ATP-495, "Acceptance Test Procedure for the Sample Extruder, H-2-85441."
6. WHC-SD-WM-ATR-495, "Acceptance Test Report for the Sample Extruder, H-2-85441."
7. WHC-SD-WM-TP-328, "Retained Gas Sampler System Operation."
8. WHC-SD-WM-TRP-232, "Retained Gas Sampler Program Proof Testing - 306E Building."
9. WHC-CM-6-1, "Standard Engineering Practices."
10. WHC-IP-0882, "306E Administration Manual."
11. Letter Report, "Hydrogen Permeation Rate for Retained Gas Sampler," A. L. Pitner to A. E. Bridges, April 28, 1994.
12. PNLFGP: 091595, "Retained Gas Sampler System Analysis," Letter Report.

3.0 RESPONSIBILITIES

Primary organizational responsibilities for the Retained Gas Sampler System are outlined in the engineering task plan for the project (WHC-SD-WM-ETP-144). Organization titles and cognizant personnel may differ from those identified in the ETP. Actual names of those performing, witnessing, and approving acceptance testing are identified in this test report document. R. C. Knight acted as overall test director.

4.0 DESCRIPTION OF SYSTEM

The Retained Gas Sampler System is designed to capture and analyze representative samples of Hanford tank waste to determine the quantity and composition of gases trapped within the waste. The RGSS consists of three primary components: 1) the retained gas sampler (RGS), 2) the extruder, and 3) the extractor. The sampler is designed to capture and retain a representative waste sample--including solids, liquids, and trapped gases. The extruder is designed to transfer the entire sample from the RGS to the

extractor. The extractor is designed to separate the gases from the liquids and solids, determine the amount of gases, remove the gas constituents for analysis, and also determine the volume of liquid and solid components of the waste sample.

A data acquisition and analysis system is also an integral part of the RGSS. This system displays, monitors, and records critical test parameters such as pressure and temperature, and did so during acceptance testing. The primary gas analytical technique involves transferring sample gas to analysis canisters for a determination of gas composition by Pacific Northwest Laboratories (PNL) utilizing a mass spectrometer (MS). An on-line gas analysis capability includes a gas chromatograph (GC) for analysis of insoluble gases; however, due to calibration time constraints, the GC was not used during acceptance testing. Ammonia gas analysis, utilizing a solution absorption technique, is also included in the RGSS.

A schematic of the RGSS is shown in Figure 1, and Extractor details including the 2nd Volume are sketched in Figure 2.

5.0 TEST CONDITIONS AND DATA ANALYSIS

5.1 TEST FACILITY

The RGSS equipment acceptance testing was performed in the 300 area, 306E building Fabrication, Examination, and Testing Laboratory. The RGSS equipment setup was prototypic of the final equipment arrangement in the 200W area, 222S laboratory, 1E2 hot cell. Testing in 306E did not simulate hot cell remote operations.

5.2 DATA ACQUISITION AND CONTROL SOFTWARE

Validation of the computer software used for data acquisition and control unit (DA-CU) functions was accomplished by testing the "off-the-shelf" National Instruments Labview program during RGSS system acceptance testing. The primary function that the software performs is to read data from the Hewlett Packard model 3497 data acquisition/control unit (DA/CU), convert and display the data in engineering units, and record the data to disk. These functions were tested by applying a known input to the DA/CU and verifying that the resulting parameter was displayed and recorded correctly for each data channel.

5.3 INSTRUMENT CALIBRATION

All instrumentation interfaced to the DA-CU were calibrated prior to use. Pressure and linear displacement transducers were calibrated by the WHC

Standards Laboratory. Thermocouples have traceability to vendor calibration results. Calibration documentation is presented in Appendix A, along with an evaluation of pressure transducer drift.

5.4 PREOPERATIONAL LEAK TEST

The ATP required that prior to acceptance testing, the entire gas extraction system be evacuated and helium leak tested to assure that the system leak rate did not exceed 10^{-6} atm/cc/sec. The leak rate was to be determined and documented with an RGS attached to the extractor vessel, alternately with the sampler valve in both the closed and open position. Also, an overall system vacuum of 1.33 Pascal (10^{-2} Torr) or better was to be demonstrated.

The pretest helium leak test was performed June 26, 1995 by W. D. Purdy, and the test report is given in Table 1. The leak rate was better than 10^{-9} atm/cc/sec (no detectable leaks were found). This leak test covered all of the extraction system identified above except in the condition with the sampler valve open. A second helium leak test was performed on August 14, 1995 (while acceptance testing was ongoing) by B. L. Hopkins; this test report is given in Table 2. This test covered the entire gas extraction system, including the condition of the sampler valve being open. The leak rate was better than 10^{-10} atm/cc/sec (no detectable leaks were found).

A pretest system vacuum as good or better than 0.001 KPa was identified in tests RGS-047 and RGS-049, fulfilling the requirement for overall system vacuum.

5.5 TEST DATA

Test data, pertinent observations and exceptions to the ATP are documented in this acceptance test report (ATR). In order to verify that acceptance testing has demonstrated compliance with the design criteria specified in WHC-SD-WM-FRD-018 (FRD), as required by the ATP, analysis of the test data is required. As previously mentioned, the primary function of the RGSS is to determine the quantity and composition of gases retained in a waste tank sample within the specifications set forth in the FRD. Once the raw test data was obtained, several methods of data analysis were employed to verify compliance of the RGSS with the FRD.

5.5.1 Gas Composition Analysis

Composition analysis of the sample gas was performed using a mass spectrometer to determine the insoluble gas component in the sample, and an ion probe technique to measure the ammonia content of the sample.

5.5.1.1 Insoluble Gas Analysis

Composition analysis of insoluble gas transferred from the RGSS extractor to analysis canisters was performed using PNL's 325 Building mass spectrometer, as previously described.

5.5.1.2 Ammonia Solution Analysis (ASA)

Analysis of the sample gas for ammonia was performed using a technique described in detail in the RGSS proof test report WHC-SD-WM-TRP-232 (TRP). Briefly, after sample gas has been dumped into the evacuated 2nd Volume (2ndV) of the extractor as part of gas quantification testing, the 2ndV is isolated, and a slightly acidic solution is injected into it. This solution absorbs the ammonia and retains it (even when exposed to air) while a transfer of the solution is made to the ion probe (Orion 9512BN Ammonia Gas Sensing Electrode) testing container. From the solution's measured ammonia concentration, solution volume, gas volume and pressure, the original percentage of ammonia in the sample gas can be calculated.

A more detailed description of this ammonia analysis technique is given in Appendix B.

5.5.2 Gas Quantity Determination

A determination of the quantity of sample gas, as well as its composition, is an important function of the RGSS. Three methods of analyzing test data will be used to determine the quantity of sample gas. They are the Back Fill method (BF), the Hg Pump transfer method, and the Two Volume (TV) method. These analysis methods will be discussed in more detail as follows.

5.5.2.1 Back Fill Method

The Back Fill method has been described in the RGSS proof test report (WHC-SD-TRP-232). This technique allows a determination of the volume of the sample's solids and liquid (V_{SL}) from the empty extractor volume (V_{EX}) as

$$V_{SL} = V_{EX} - V_x \quad (1)$$

where

$$V_x = \frac{(P_a - P_f)}{(P_f - P_{EO})} [V_2 + V_L] \quad (2)$$

The values of V_{EX} (extractor volume), V_2 (2nd Volume) and V_L (line volume) were measured before acceptance testing was initiated. After the insoluble gas has been transferred to analysis canisters (or after previous BT measurements), the initial pressure in the extractor is measured as P_{EO} , and the extractor is isolated from the rest of the system. The lines and the 2ndV are then charged with air or argon gas to a pressure of P_a . When the 2ndV and extractor are reconnected, the final pressure is recorded as P_f . These steps can be repeated several times to provide redundant data for determining V_{SL} .

It should be noted that a determination of V_{SL} allows a calculation of the volume of gas in the Sampler; since all Sampler volumes are premeasured (V_s), the gas volume in the Sampler (V_g) can be calculated as simply

$$V_g = V_s - V_{SL} \quad (3)$$

However, the BF method does not allow a direct determination of the quantity (number of moles) of sample gas. Using the value of V_{SL} determined here, either the Hg Pump Method or the Two Volume Method can provide independent value for the number of moles of insoluble gas in the extractor.

5.5.2.2 Hg Pump Method

The mercury transfer pump shown in Figure 1 transfers sample gas from the extractor to analysis canisters for MS composition analysis. In addition, the quantity of gas transferred can also be determined using the perfect gas law, since the volumes of the analysis canisters (V_c) and feed lines (V_{fl}) were measured before acceptance testing was initiated, and at the end of each gas transfer, pressure and temperature are recorded. It has been shown (WHC-SD-TRP-225) that when the Hg Pump is operated through 10 cycles, over 99 percent of the extractor gas is transferred to the collection side of the system. During acceptance testing, the Hg Pump was normally operated through at least 10 cycles, allowing a determination of the initial sample gas quantity from the transferred gas.

Depending on the completeness of the gas transfer, the remaining pressure in the extractor after pumping is finished is the vapor pressure of soluble gas and water (P_v). Measurement of this vapor pressure is also a requirement of the FRD.

Thus, from the perfect gas law,

$$n_g = \frac{(P_f - P_v)(V_c + V_{fl})}{RT} \quad (4)$$

where P_f is the final pressure measured in the analysis canister after gas transfer has been completed. P_v is assumed to be the same vapor pressure in the analysis canister as remains in the extractor. Of course, R is the universal gas constant and T is the temperature (in K).

5.5.2.3 Two Volume Method

The Two Volume method is a hybrid technique combining the Start Pressure Technique described in the proof testing report (WHC-SD-TRP-232) with the BF method to eliminate the need to know the initial pressure (potentially unavailable) in the Sampler. Once the value of V_{SL} has been determined from the BT method, the number of moles of insoluble gas can be calculated as

$$n_g = (P_{exL} - P_2) \left(\frac{V_x}{V_2} \right) \left[\frac{V_x + V_2}{RT} \right] \quad (5)$$

where P_{exL} is the initial pressure in the extractor and feed lines, after a gas releasing step (like sample extrusion, mixing or dilution) has been completed and before the isolated (and evacuated) 2ndV is combined with the extractor; P_2 is the pressure in the combined volumes.

The vapor pressure can then be calculated as

$$P_v = P_{exL} - \left[\frac{n_g RT}{V_{exL} - V_{SL}} \right] \quad (6)$$

where V_{exL} is the sum of the feed line and extractor volumes.

Thus, the Hg Pump and Two Volume methods provide two separate and independent techniques of determining insoluble gas quantity and soluble gas/water vapor pressure.

6.0 SAFETY

Only the test director, cognizant test engineers, and/or their approved personnel operated the RGSS equipment. A Hanford Job Hazard Analysis Checklist and 306E Specific Job Hazard Analysis Checklist were prepared and approved prior to acceptance testing. A prejob safety briefing was conducted with all test personnel. All of these steps were performed in accordance with the 306E Facility Administration Manual WHC-IP-0882.

7.0 ACCEPTANCE TEST RESULTS

The RGSS was fabricated and assembled essentially as shown in Figure 1. Although proof testing had verified RGSS operation principles, acceptance testing as outlined in WHC-SD-WM-ATR-137 was required to verify proper system design specific to the actual hardware and operating procedures.

The RGSS ATP directed that acceptance testing be conducted in three phases as described in the following sections. The raw data for each test was recorded in one of three logbooks; HEDL-N-2603, WHC-N-1111 I or WHC-N-96. Raw data sheets are included in Appendix C for all tests that provided data used in this ATR. In performing these tests, the operating procedures specified in WHC-SD-WM-TP-328 were used as a guideline for step-by-step equipment operation.

7.1 HARDWARE SETUP/VOLUME DETERMINATIONS

In order to obtain gas quantity and void fraction data, it was necessary to accurately know the volumes of many of the RGSS components, including the extractor, 2nd volume, feed lines and sampler.

7.1.1 2nd Volume Measurement

The 2nd volume was measured by weighing it and attached components (including valves U, Q, P, R, S and T) in the dry condition. Then it was filled with liquid of a known density, and reweighed. The difference in mass divided by the liquid density gives the volume; actual test details are given below.

Three tests were performed on 6-22-95. Weighing of the entire 2nd Volume hardware was performed at the WHC Standards Laboratory using scale # 002-06-01-013. The dry mass was determined at 6816.8 grams. The 50 mL canister (between Valves Q and P) was also weighed dry at 835.1 grams, on a different scale (Standards Lab # 677-06-01-024, located in 306E Building).

Then a fill canister was attached to the 2nd Volume through Valve U using a Tygon tube. The fill canister was filled with test liquid, and the canister was raised above the 2nd Volume. Valves P, Q, R, S and T were in the open position until liquid started to drain through Valve S. Then Valve S was closed until liquid again started to drain through Valve T, at which point, Valve R was closed. Liquid continued to fill the 50 mL canister until it started to drain out Valve P; Valves Q and P were then closed. (Note: the 50 mL canister was used in these tests to make certain that the volume through Valve Q was completely filled with test liquid.)

The outside of the 2nd Volume and its accessible tubes were then dried; the 2ndV was reweighed (now filled with test liquid). Then the 50 mL canister

was separated from the 2ndV, and weighed on the 306E Building scale. Using the data obtained from these scales, the volume of the 2ndV was determined, since the density of the test liquid was known. These tests were performed at nominally 25 °C, using water and alcohol as the test liquids; at this temperature, the density of water is $\rho_w = 0.997$ g/mL, and of alcohol is $\rho_a = 0.787$ g/mL.

Three different tests were performed to measure this volume.

7.1.1.1 Test 1

The 2nd Volume was filled with deionized water and reweighed at 7182.8 grams. Then the 50 mL canister was removed and weighed at 886.7 grams. Thus, the total mass of water in the 2ndV and 50 mL canister was 7182.8 g - 6816.8 g = 366.0 grams. The mass of water in the 50 mL canister was 886.7 g - 835.1 g = 51.6 grams. This leaves the second volume water mass at 314.4 grams, and corresponds to a volume of 314.4 g/ ρ_w = 315.3 mL in the 2nd Volume.

7.1.1.2 Test 2

One of the concerns in using water as the liquid to fill the 2ndV was that bubbles might form, causing the volume to be determined at a lower value than it actually was. For this reason, the next two volume measurement tests were performed using an alcohol/water mix; alcohol will "wet" better than pure water, helping to minimize bubble formation.

For this test, the mix was 1600 mL of deionized water to 200 mL of ethyl alcohol. The full weight of the 2ndV (including canister) was measured at 7179.3 grams; subsequently, the full 50 mL canister was measured at 885.9 grams. Thus, the mass of liquid in the 2nd Volume was 311.7 grams.

The density of the combined alcohol and water is a weighted average of the individual densities. Thus, $\rho_T = (\rho_w * 1600 \text{ mL} + \rho_a * 200 \text{ mL}) / (1800 \text{ mL}) = 0.9737$ g/mL. This yields a volume of 320.1 mL for the 2nd Volume.

7.1.1.3 Test 3

The third test used a different alcohol/water mix; 1600 mL of water with 10 mL of alcohol added. The full assembly was measured at 7188.5 grams; the full 50 mL canister was weighed at 887.0 grams. The mass of liquid in the 2ndV would then be 319.8 grams.

The density of the combined alcohol and water is $\rho_T = (\rho_w * 1600 \text{ mL} + \rho_a * 10 \text{ mL}) / (1610 \text{ mL}) = 0.9957$ g/mL. This yields a volume of 321.2 for the 2nd Volume.

The addition of alcohol to water to form the test liquid increased the measured volume, as expected. Only the two alcohol/water tests were used in

the final determination of the 2nd Volume. The average of these two test results gives $V_2 = 320.64$ mL.

7.1.2 Extractor Volume Measurement

The ATP requires that the volume of the extractor will be measured using a known quantity of gas introduced into the previously evacuated extractor. The extractor volume can then be calculated using the perfect gas law, based on the measured pressure changes (at constant temperature).

The ATP requires that these measurements be repeated at least 10 times. This test was first performed on 6-27-95, and Table 3 gives the results. This data table is slightly modified from the one described in the ATP to allow the determination of the line volume (from Valves B and Z to Valves R and T, see Figure 1). The 3 and the 2 are transposed in Table 3 for the Measured 2nd Volume value; the intended number was 320.0 mL. For this value, the average extractor volume was $V_{EX} = 1349.7$ mL, while the average line volume was $V_L = 220.9$ mL. Subsequent reevaluation of the 2nd Volume measurement indicates that $V_2 = 320.64$ mL is a better value; then $V_{EX} = 1352.4$ mL and $V_L = 221.3$ mL.

The RGSS ATP requires that the standard deviation for the extractor volume results be 0.3 percent (or less) of the average volume determined (see WHC-SD-WM-FRD-018). The standard deviation for the above extractor volume measurements was 0.15 percent of the average value, well within the requirement.

Although not included in the ATP, it is important (particularly for the Hg Pump Method) to know volumes within the RGSS cabinet on the analysis canister side of the Hg transfer pump. These volumes were measured by using a charge of argon gas in the line volume, and dumping the gas sequentially through the previously evacuated volumes as defined in Table 4. The average values of these determined volumes are also given in Table 4.

A change was made on 7-26-95 to the feed lines coming to the extractor which resulted in a change in the line volume. A new line volume measurement was made by charging the 2nd Volume to a given starting pressure, evacuating the line volume, and then dumping the 2nd Volume into the line. These measurements are given in Table 5, and result in a new line volume of $V_L = 199.95$ mL. This is the line volume value that is used for analysis of all RGSS acceptance tests performed after 7-26-95.

Also, a change was made on 8-15-95 to the extractor. A new piston stop was welded into the extractor for the extractor/sampler interface; although not expected to change significantly, the extractor volume was remeasured as a precaution. The results are given in Table 6, and the average over ten tests was $V_{EX} = 1354.0$ mL, with a 0.04 percent standard deviation. This is the value of the extractor volume that was used for analysis of all RGSS acceptance tests performed after 8-15-95.

It should be noted that in all cases, the volume of the extractor measured in this Section included the stirrer and all internal extractor apparatus that will be used with actual tank waste samples.

7.1.3 Sampler Volume Measurement

The three prototypic Samplers that were used for essentially all of the acceptance testing each had a volume measurement made using the water fill (dry and full weighing) technique. After dry weighing, the Samplers were completely filled with deionized water; when the ball valve was closed, the valve remained completely submerged in water so that no air bubbles were trapped in the closing process. Since the Sampler barrel was a simple cylinder, visual examination was made for bubbles before the ball valve was closed. After external drying, the following volume measurements were made for the respective Samplers indicated:

Sampler	Date	Measured Volume (mL)
#1	7-12-95	314.63
#2	7-18-95	315.26
#3	7-18-95	315.08

These three Samplers were fully prototypic of production units (including passing a 3 atmosphere helium leak-check criteria specified in the FRD), once leakage problems to be discussed in Section 7.2.2.2 were solved.

7.1.4 Extractor/Sampler Integration Test

To verify the proper operation of the extractor and sampler together (including verification of measured volumes for extractor and 2nd volume), the ATP requires the performance of a start pressure test (SPT) in which the sampler is filled with air at a measured pressure (atmospheric) and temperature (ambient). The SPT, described in WHC-SD-WM-TRP-232, was to be performed with a resulting determination of the starting gas volume/quantity within the FRD specifications.

This SPT test (RGS-007) was performed (7-14-95), and the raw data results are given in Table 7. Sampler #1 was loaded with 754.8 Torr (100.64 kPa) of air at 21 °C, which corresponds to $n_g = 0.01295$ moles of air at STP.

From WHC-SD-WM-TRP-232, the following SPT equations are reproduced for convenience:

$$V_{SL} = \frac{[(V_{EXL} - V_s)(V_{EXL} + V_2) - kV_{EXL}(V_{EXL} + V_2 - V_s)]}{[V_{EXL} - V_s - k(V_{EXL} + V_2 - V_s)]} \quad (7)$$

and

$$n_g = \frac{[(P_s - P_2)(V_s - V_{SL})(V_{EXL} - V_{SL} + V_2)]}{[(V_{EXL} + V_2 - V_s)RT]} \quad (8)$$

where $k = (P_s - P_{EX})/(P_s - P_2)$, P_s is the "start" pressure in the Sampler, P_{EX} is the pressure in the extractor after the Sampler ball valve has been opened and the piston extended, and P_2 is the pressure in the combined extractor and 2ndV. These two equations are slightly modified in appearance from the way they appear in the TRP in that $V_{EXL} = V_{EX} + V_L$ has been substituted for V_{EX} , as is correct for the present RGSS hardware configuration.

V_s is the volume of the Sampler (314.63 mL), and $P_s = 102.04$ KPa (compensated for the change from 21 to 25.1 °C), $P_{EX} = 20.38$ KPa and $P_2 = 16.96$ KPa. $V_{EXL} = 1352.4 + 221.3 = 1573.7$ mL (note the corrected difference from the value reported in Table 7). With these values, the calculated results for this test are $V_{SL} = 3.72$ (should be 0), and $n_g = 0.01277$ moles; this represents a -1.4 percent error when compared to the expected value of 0.01295 moles. This error is well within the allowable limits of the FRD (2% absolute error is the specification).

7.2 GAS INJECTION TESTS

Gas injection tests were performed to verify the ability of the RGSS to provide the correct (within specifications) gas composition analysis and quantity determination of a known gas mix injected directly into the evacuated extractor through the dilution port, without the use of a Sampler. Tests were conducted with an RGS mated to the extractor with the ball valve open and piston in the forward position. The ATP required four injection tests; however, many additional injection tests were performed and will be described after reporting the results of the required tests.

The ATP specified that for each injection test, two samples of the known gas were to be obtained simultaneously from the same gas source. One sample was to be used as a control while the second was to be injected directly into the extractor through the dilution port. Both SPT and backfill test (BT) procedures (see WHC-SD-WM-TRP-232) were to be performed to measure the quantity of the injected gas, allowing a comparison of the result with the known value. However, in an ATP change, only one control sample was obtained for each type of gas used in injection testing because of the reliability of the mass spectrometer composition analysis. Also, only the Hg Pump method was used to determine gas quantity for the four ATP required injection tests.

7.2.1 Required Injection Tests

Injection tests were performed at both ambient and 50°C for each of the two control gas mixes, giving a total of four required injection tests. According to vendor certification, gas MIX#1 contains ammonia (5.0%), argon (24.9%), hydrogen (30.3%) and nitrogen (39.8%); MIX#2 contains nitrogen (30.0%), nitrous oxide (30.8%) and helium (39.2%). Vendor certification for these gas mixes are given in Tables 8 and 9, respectively. These control gas mixes were chosen to include gas components in roughly the same relative percentages expected from a "typical" flammable gas waste tank (101-SY); two mixes were used in order to separate the potentially explosive combination of hydrogen and nitrous oxide.

7.2.1.1 Nitrous Oxide Gas Mix Analysis

Gas MIX#2 was used in the first injection tests. A control gas sample (labeled RGS-002) was obtained in a 40 mL canister at the same time another 40 mL canister was filled for injection test RGS-001, performed at ambient temperature. The raw data results from test RGS-001 are given in Table 10; Hg transfer pumping of the gas to the collection canister (J1) resulted in a final pressure reading of 63.48 KPa at a cabinet temperature of 32.3 °C.

The second injection test (RGS-003) for this gas mix was performed at an extractor temperature of nominally 52.7 °C. The raw data results from this test are given in Table 11. After gas transfer, the final pressure in the analysis canister J2 was 59.06 KPa at a cabinet temperature of 31.7 °C.

Table 12 gives both the gas composition analysis (including the control sample) and gas quantity results for these two tests.

7.2.1.2 Hydrogen/Ammonia Gas Mix Analysis

Gas MIX#1 was used in the remaining two required injection tests. A control sample (labeled RGS-004) was obtained at the same time as the injection test sample RGS-005. The raw data results from test RGS-005 (performed at ambient temperature) are given in Table 13; the Hg pump transfer of gas to the analysis canister resulted in a final pressure reading of 59.57 KPa at a cabinet temperature of 32.3 °C.

Test RGS-006 was performed (using MIX#1 gas) at nominally 52.4 °C; raw data from this test are given in Table 14. The final pressure in the analysis canister after Hg pump gas transfer was 56.43 KPa, with a cabinet temperature of 29.5 °C.

Gas composition analysis (MS) and gas quantity results for RGS-005 and RGS-006 are also reported in Table 12.

7.2.1.3 Results Summary for Required Injection Tests

As previously mentioned, a results summary for the required injection tests using two gas mixes is given in Table 12.

For the simpler nitrous oxide gas mix (MIX#2), the composition test results are straight forward. The vendor and MS control sample (RGS-002) results are within 0.4 percent agreement for all three gas components in this mix. Both the ambient and 50 °C injection tests had composition results that were within 2.7 percent of the vendor (or control) compositions; the FRD requires only a 6 percent agreement for these gas components at these percentage levels.

When adjusted for "leakage", using oxygen as a tracer (see Appendix D), the MIX#2 injection test agreement is within 0.4 percent of the vendor or control sample composition for both test conditions. It should be noted that the "leakage" noticed (particularly for test RGS-001) is the result of off-gassing from the chamber walls rather than from actual system leakage. Whenever, the system is exposed to air, an extensive vacuum pump down over a long period of time (overnight is recommended) is required to reduce this off-gassing effect.

The hydrogen/ammonia gas mix (MIX#1) composition analysis results are more complicated because of the "washout" of ammonia experienced in processing the gas through system tubing and canisters. A comparison of the vendors composition with the control sample's (RGS-004) shows a significant difference in ammonia levels. This washout also affects the remaining gas components' relative percentages.

A calculational method for compensating ammonia washout by using the "known" value for ammonia, and adjusting the relative percentages of the remaining gas components for ammonia loss, is given in Appendix D. Ammonia loss compensation was used for the control sample results as shown in Table 12; this improved the agreement between the vendor and control sample results, with the largest difference being 0.9 percent for nitrogen.

The control sample's MS composition was adjusted for the change from 2.9 percent ammonia to 1.4 percent for comparison with the RGS-005 results after testing at 28.0 °C. The 1.5 percent difference for hydrogen between the control and test results is the largest for the measured gas components, but still within the FRD requirement of an absolute difference of 2 percent for hydrogen. (The FRD allowance of ± 20 percent relative difference for other gas components is much more lenient than for hydrogen, allowing an absolute difference of ± 6 percent for an actual level of 30 percent.) When the "leakage" adjustment is performed, the largest difference between control and test values shrinks to 0.4 percent for hydrogen.

Similarly, the control sample's MS composition was adjusted for the change from 2.9 percent ammonia to 1.8 percent for comparison with the RGS-006

results after testing at 52.4 °C. The 0.3 percent difference for argon between the control and test samples was the largest noted. When the "leakage" adjustment was performed, the maximum difference between control and test compositions increased slightly to 0.4 percent for nitrogen. These test results are clearly within the specifications of the FRD.

Also presented in Table 12 are the analysis results for gas quantity for these four injection tests, based on the Hg Pump Method. These results are presented in terms of gas volumes at Standard Temperature and Pressure (STP) conditions. The "adjusted (2ndV) gas" volume is a adjustment to compensate for the gas dumped into the 2nd Volume that was not transferred to the analysis canister portion of the RGSS. This adjustment was accomplished using a simple ratio of the affected system volumes multiplied by the "final canister gas" volume. Errors between the initial starting gas volume and the volume determined from the Hg Pump Method for these four tests were 3 percent or less. The FRD allows a relative error of ± 20 percent for these gas quantities.

The ammonia solution absorption (ASA) technique for ammonia analysis determined the ammonia concentrations at 2.1 percent and 2.7 percent for tests RGS-005 and RGS-006, respectively. These values for ammonia concentration are better than those determined from the mass spectrometer as reported in Table 12, but significantly lower than the expected 5.0 percent. They do not meet the FRD requirement for ammonia (which allows an absolute error of ± 2 percent for these conditions); a result of at least 3 percent is required for an actual 5 percent ammonia gas mix.

MIX#1 gas had been previously tested (directly from the gas bottle to 40 mL canisters, followed by absorption into solution) using the ASA technique, with the average result over seven tests of 5.05 percent, verifying the vendor certification (although a large standard deviation of about 1 percent was observed). These test results are reported in Table 15.

Under these test conditions, the results for the four required injection tests verified compliance of the RGSS with the FRD for gas quantity analysis and composition analysis, with the exception of ASA ammonia gas analysis.

7.2.2 Additional Injection Tests

Many more gas injection tests (than were required in the ATP) were performed because these tests provided a valuable tool in trouble shooting problems that developed during the RGSS acceptance testing. In addition, injection tests were performed to gather more data under combined gas and water simulate test conditions to supplement (at lower cost) the limited number of full scale simulant extrusion test results.

Three types of problems were encountered during acceptance testing; ammonia analysis accuracy, leaking samplers and degassing water simulant. Additional injection tests were used to evaluate all three of these problems.

Ammonia analysis accuracy remains outside the FRD specification. The leakage of samplers was identified, and a change in sampler assembly procedure was made to eliminate this problem. Degassing of water during a simulant test altered the gas composition, producing an apparent error; this problem was solved by degassing the water prior to adding the simulant gas to the sample. It should be noted that the water degassing problem was not caused by an RGSS hardware or procedure failure; it was caused by initially failing to recognize that ordinary aerated water would create a simulation problem.

A complete list of all acceptance tests performed (including problem tests) is given in Appendix E. Only those tests that provide useful data will be analyzed in the remaining report that follows.

7.2.2.1 Ammonia Solution Analysis (ASA)

A series of injection tests were performed to more fully evaluate the analysis of ammonia gas concentration using the acid solution absorption technique. The test results are reported in Table 16, and include the two required injection tests RGS-005 and RGS-006 previously described.

For tests RGS-005 and RGS-006, during ASA ammonia analysis a brown precipitate formed in the test solution when its pH was adjusted (to between 12 and 14), releasing the trapped ammonia to the ion probe. It was thought that welding contaminants remaining inside the 2nd Volume might have caused the precipitate, so the 2ndV was recleaned with acid. Informal tests A-1 and A-2 were performed to verify the success of the cleaning process; no precipitates were observed, and the ammonia results were much better (at 4.6 percent). However, low ammonia concentrations were later measured for the three subsequent tests, also without the appearance of the precipitate. Thus, it appears likely that the precipitate was not related to the low ammonia readings.

Schedule considerations prevented further study of this ammonia analysis problem. It was decided to finish other aspects of the RGSS acceptance testing and move towards installation and operation of the system, recognizing that the ammonia analysis method failed to pass the requirements of the FRD.

Any future evaluation of the ammonia analysis problem should start by looking at the stirring motor splash shield and sleeve (Drawing H-2-821610, Rev 0, Sheet 9, Item 35). The Drawing specifies that this sleeve be made with ultra high density polyethylene; tests should be made to see if replacing the polyethylene with 304L stainless steel improves ammonia analysis performance, since many materials absorb ammonia. Other than the essential viton vacuum seals (copper seals are known to absorb ammonia), the polyethylene is the only non stainless steel material inside the RGSS extractor and 2nd Volume.

There are other possible explanations for the low ammonia readings. The stainless steel walls of the extractor and 2nd Volume may pull some of the ammonia out of a gas mix to coat the wall surfaces. Mockup tests should be

performed with different sized stainless steel containers using a constant quantity of gas to see if the ammonia concentration is reduced with increasing container size. Also, the wide variation of results noted in Table 16 may be related to how well the system has been dried out prior to testing; residual water (even in small quantities) would absorb ammonia, and may account for the wide variation in results.

The injection tests performed provide a limited amount of ammonia (only what is in the gas); actual waste samples may contain a significantly larger reservoir of ammonia "in solution". In such a case, walls would become saturated with ammonia with a minimal decrease in the ammonia concentration in the gas. A more appropriate simulation of this condition (than gas injection tests) would be to use a aqueous ammonia solution with a known ammonia vapor pressure as the simulant.

All of the above steps should be taken to characterize the response of the RGSS system to different ammonia gas source conditions. Multiple ammonia tests for the same tank sample could be used to investigate the ammonia "reservoir" content of the waste sample.

7.2.2.2 RGSS Sampler Leakage

All RGSS samplers were leak tested prior to use, and found to be leak tight. However, as a result of the low resistance provided by the water or Carbopol® simulants, the sampler piston often "slammed" forward with high impact during extrusion because of the pressure difference between the evacuated extractor and the external atmosphere on the other side of the piston. During most of the early simulant extrusion tests (to be discussed later), the system began leaking immediately after the extrusion step. This effect was traced to leakage through the threads of the sampler O-Ring Insert Assembly (H-2-821608, Sheet 2, Item 23) mated to the Valve Housing (same drawing, Item 9).

This sampler leakage path had been sealed using Varian Associates' "Torr Seal" vacuum epoxy per the manufacturer's specifications. The low vapor pressure Torr Seal hardened to a fairly stiff bead on the interface between Items 23 and 9 and in addition to providing leak protection, also prevented the insert from turning during sampler handling. However, this bead was found to have cracked for those samplers where a post extrusion leak had developed.

This leakage problem was solved by changing the sampler assembly procedure; Torr Seal was put on the threads of Item 23 prior to sampler assembly as well as being used to form a bead between the insert and valve housing after assembly. Once this step was added to the sampler assembly

* Carbopol® is a trademark of the B. F. Goodrich Company

procedure, no sampler was found to have leaked during the remaining thirty-five acceptance tests (including the final six extrusion tests).

7.2.2.3 Water Degassing

Deionized water was used as a simulant for the solid/liquid portion of a sample in many acceptance tests. Initially, this water was used in its ordinary aerated form. In this form, the water ruined composition analysis tests by discharging dissolved air into the vapor space, thus changing the expected composition of the gas. At the same time, total gas quantity increased, particularly during Hg Pump gas transfer, since air was sucked out of the water during this low pressure operation.

Starting with acceptance test RGS-024 and on through RGS-053, when a water simulant was used, it was degassed prior to test performance. This was done by evacuating the container for the water (extractor or sampler) several times below the vapor pressure of water, then allowing it to sit overnight (usually), and re-evacuating the container again. Composition analysis results improved significantly for degassed water tests. Gas quantity results were reduced below expected levels (although within FRD requirements) as measured by the Hg Pump analysis method; this was to be expected as some of the injected test gas would be absorbed by the degassed water.

7.2.2.4 Wet Gas Mix Injection Test Results

Five additional gas MIX#2 "over" water injection tests were performed and also one MIX#1 injection test. The water simulant for these tests was left in the extractor from previous tests, and degassed in-situ before the initiation of each test. Initially, about 300 mL of water had been added to the extractor; at the end of testing, over 250 mL remained (as crudely remeasured in a beaker - not including some residual water left in the extractor and elsewhere). Since water was evaporated during each degassing cycle, the precise V_{SL} was not known, and the Back Fill Technique was not used to measure it, as the BT measurement accuracy could not be cross verified.

The composition results for these tests are reported in Table 17, and excellent agreement was achieved as compared with the control samples, except in the case of RGS-029. This test was performed with only 2.5 mL (at STP) of gas, which appears to define a minimum gas volume limit for RGSS composition analysis accuracy. Except for RGS-029, all the Table 17 test results, as compared with control sample compositions, are within the specifications required by the FRD.

Gas quantity determinations based on the Hg Pump method for these tests are given in Table 18. All of the determinations underestimate the initial starting injection quantity, which is consistent with some of the injection gas being absorbed by the degassed water simulant. However, all of these test results (except RGS-037) are within the FRD requirement that the ratio of gas volume to bulk waste volume have an absolute accuracy of ± 2 percent. The

bulk waste volume (assuming complete filling of a sampler) would be 315 mL; 2 percent of this bulk volume corresponds to 6.3 mL of gas. Only the RGS-037 difference between start quantity and Hg Pump transferred quantity of 6.41 mL slightly exceeds this FRD specification. But if the fact that upon injection, all of the ammonia was immediately absorbed in the water is accounted for, the gas start quantity is reduced to 35.36 mL and the transfer difference is only 4.68 mL, well within the FRD requirement.

Thus, it is concluded that for the six gas MIX#1 and MIX#2 injection tests performed in the presence of degassed water simulant, all RGSS composition and quantity analyses produced results within the FRD requirements for accuracy (with the exception of test RGS-029, for which the initial 2.5 mL gas volume is presumed too small for accurate RGSS composition analysis).

7.2.2.5 Wet Argon Gas Injection Test Results

Tables 19 and 20 list analysis results for gas composition and quantity, respectively, of RGSS injection tests using argon gas and degassed water simulants. A single component gas (argon) was used as the simulant gas in these tests to simplify the analysis interpretation of leakage and outgassing effects. The primary purpose of the argon gas injection test series was to correlate composition analysis accuracy with the initial gas quantity injected; however, the Hg Pump Method was also used to determine gas quantity.

Three of these argon injections tests included an elevated temperature and "mixing" excursion to nominally 50 °C; these are tests RGS-047, RGS-049 and RGS-050. Previously, it had been found that gas transfer at these elevated temperatures resulted in water condensing and plugging the filter (located as shown in Figure 1). Thus, the extractor and second volume valves (R and T) were closed while the temperature was above ambient during these tests. Gas transfer to the analysis canister took place after the temperature had been reduced to ambient.

While above 50 °C, the mixer motor (with blades attached) was operated for 10 minutes at about 15 RPM to simulate stirring waste; this length of time should be long enough to drive out most actual waste gas bubbles in the presence of a near vacuum. It is recommended that the mixing motor operation be limited, since the vacuum feedthrough bellows failed and had to be replaced after only a few hours operation in prior testing.

7.2.2.5.1 Composition Results

Two control gas sample analyses were performed and both produced a 99.95 percent argon result. The composition results from the remaining argon injection tests were compared with these control sample values in Table 19; an error value was recorded for each test. A plot of this error versus initial gas quantity is made in Figure 3. Also included in Figure 3 is error data from the mixed gas injection tests; error for mixed gas tests was defined as

the maximum difference between control and test gas composition for any of the major components (excluding ammonia).

Again, with the exception of RGS-029 (MIX#2 at 2.5 mL), all of the data given in Figure 3 satisfies the FRD requirements.

It should be noted that the FRD composition error requirement is 20 percent (relative) for each gas species. Thus, for single component argon gas, a 20 percent absolute error is acceptable; however, for nitrous oxide (for example) at 30 percent of the total, only a 6 percent absolute error is allowed. Regardless of actual composition, a 2 percent absolute error is within the FRD requirement. Hydrogen has a special requirement that for all composition levels, the hydrogen concentration must be measured to within 2 percent absolute accuracy.

Based on the data presented in Figure 3 and Tables 17 and 19, adequate composition analysis accuracy has been demonstrated for initial gas volumes (at STP) as low as 5 mL. This quantity of gas would, for example, correspond to a sample void fraction of about 0.7 percent if the sampler were full and at a pressure of 2.5 atmospheres (a pressure level consistent with lower 101-SY tank levels).

7.2.2.5.2 Gas Quantity Results

The argon injection test gas quantity results given in Table 20 are plotted in Figure 4; i.e., the absolute value of the gas quantity error (based on the worst case results of $P_v \neq 0$) was plotted versus initial injection gas volume (expected V_g). Also plotted in Figure 4 are the gas quantity results for the mixed gas compositions given in Table 18. All of these gas quantity results in Tables 18 and 20 were obtained using the Hg Pump Method.

Only the RGS-047 argon injection test result exceeds the FRD error limit for gas quantification (by roughly a factor of two). However, this particular large error can be explained as the result of a lower than normal temperature during gas transfer to the analysis canister. At the end RGS-047, the extractor temperature was lowered to 15.6 °C to investigate the resulting lower water vapor pressure effects on gas transfer. Thus, gas was transferred at a temperature 5 °C lower than for any of the other RGSS acceptance tests; however, the reduced temperature also caused more of the test gas to be dissolved in the water than for the higher temperature tests. The Hg transfer pump was not able to drag this extra dissolved gas from solution in just 10 cycles.

In fact, for all of the tests covered in Figure 4, the test results underpredict the actual quantity of injected gas. The major reason for this error is believed to be absorption of the test gas in the degassed water. Degassing the water was necessary to perform valid composition analysis testing, but this step also affected the quantity results because the Hg pump was unable to remove all of the dissolved gas from the water. However, this

was not a significant problem, since the quantity results were still within the requirements of the FRD when the gas transfer was performed at ambient temperature.

A second potential reason for the underprediction of gas quantity using the Hg Pump Method is the difficulty in properly accounting for the water vapor pressure. The vapor pressure of water in the analysis canister can range from zero to the full P_v expected in the presence of liquid water, depending on how much water is transferred with the Hg pump. In Table 20, the gas quantity results are calculated using Equation (4) both with P_v equal to that measured in the extractor, and also with $P_v = 0$ (for which the error is reduced). The results plotted in Figure 4 assume the larger error, in order to be conservative.

7.3 SIMULANT EXTRUSION TESTS

The purpose of these extrusion tests was to verify the RGSS capability (including the extruder) to transfer the simulant from the sampler to the extractor, to mix the simulant in the extractor to drive out gas bubbles, and to transport the simulant gas from the extractor to the collection canisters.

The ATP specified that extrusion tests be performed using water or Carbopol simulants for the liquid/solid component of the waste, and air or helium to simulate the waste's gas content. The requirements of the ATP were exceeded in that a gas composition analysis was also performed for each test. Tests were to be performed at both ambient and 50°C for each simulant condition identified below. A minimum of one simulant test was to include a 2:1 dilution of the sample with water as a proof-of-principal test of the method proposed to release bound gases from the waste.

Argon was substituted for air in the above ATP requirements. Carbopol tests were performed (RGS-018 and RGS-019), but Sampler leakage and Carbopol degassing invalidated these results. It was not possible to degas Carbopol, so that these tests were not repeated. Without the Carbopol simulant, the dilution (2:1) of the simulant was not performed, since the simulant was already water.

Three simulant extrusion tests were performed; results are given in Table 21 for gas quantity analysis, and in Table 22 for gas composition analysis. Only water was used as the solid/liquid simulant for these extrusion tests; argon was used as the simulation gas for the first two tests, and helium in the last test. The first test (RGS-051) was performed entirely at ambient temperature. The last two tests (RGS-052 and RGS-053) both included a high temperature (nominally 50 °C) excursion to allow a ten minute operation of the mixer at that temperature.

Examining Table 21, it can be noted that all gas quantity results were within the FRD accuracy requirements except for the RGS-052 Two Volume

analysis; this TV analysis overpredicted the gas quantity by 7.7 mL where only 6.3 mL is allowed by the FRD. The TV analysis overpredicted gas quantity, whereas, the Hg Pump technique underpredicted gas quantity as it did for the previously discussed injection tests. The vapor pressure predictions were significantly better for the TV analysis method than for the Hg Pump method.

The gas composition results in Table 22 are well within the FRD requirements for all three tests. It is interesting to note that test RGS-052 had a significant nitrogen/oxygen component (air) of about 8.6 percent, indicating either leakage or (more likely) further degassing of the water during testing. This extra air would account for about 2.5 mL of the TV gas quantity overprediction for this test, placing that test result within the FRD requirement (if the air is the result of further degassing).

7.4 SAMPLER AGING TESTS

Although not required by the ATP, four "aging" tests were performed to investigate the ability of a sampler to retain its gas with minimal leakage over an extended period of time. (After a sample is obtained from a waste tank, it may not be possible to test it immediately.) Also of interest was the possible loss of hydrogen into sampler seals.

The maximum internal pressure in samplers is expected to be in the 2.5 atmosphere (absolute) range; however, these aging tests were performed at roughly one atmosphere internal pressure (and in one case, at 0.33 atmospheres). There was no need to perform these tests at higher pressure, as the in-leakage rate for oxygen (a tracer for air) would be essentially independent of internal pressure; thus, a lower pressure test would provide greater leakage sensitivity based on the relative oxygen composition. Hydrogen absorption rate in seals is proportional to pressure (Pitner letter report), resulting in the same gas composition ratio changes, independent of pressure.

These tests cover an aging time of from 24 to 1829 hours. Three different samplers were pressurized with a 30 percent hydrogen gas (MIX#1; see Table 8) at approximately atmospheric pressure, with the fourth sampler pressurized at about a third of an atmosphere (absolute). The results are reported in Table 23.

The differences between the gas concentrations measured from the four aged samplers and the concentrations expected (assuming no leakage or hydrogen absorption) are plotted in Figure 5 for the primary test gases (hydrogen, nitrogen and argon). It should be noted that ammonia loss is expected during PNNL's Mass Spectrometer analysis, and this loss will alter the relative composition of other gas components. Thus, calculations were performed (as described in Appendix D) to compensate for the decreased ammonia concentrations actually measured; these calculations (based on RGS-004 control data) produced the "expected" compositions (COMP 1, 2, and 3) shown in

Table 23 for comparison with test results. The RGS-004 control data was used for the expected composition calculations because this allows these comparisons to be made with all gas composition analysis performed by the same high accuracy mass spectrometer.

Measured hydrogen levels were slightly higher than expected (+0.4 to +1.2 percent, absolute) in all four aged samplers, but these results are still well within the (most stringent) FRD requirement of ± 2 percent (absolute) error for hydrogen, as can be seen from Figure 5. The argon and nitrogen measurements are in even better agreement with the expected concentration values, whereas the FRD is more lenient with these gases, allowing more than ± 5 percent (absolute) error at these levels (20 percent relative error).

7.4.1 Hydrogen Seal Permeation

The surplus of hydrogen suggests that seal permeation losses were not significant over the time period examined, and although there appears to be a general decrease in hydrogen over time, this effect is likely due to data scatter. Note that the MS measurements have an error bars of ± 0.6 and ± 0.3 percent for hydrogen and ammonia, respectively. Accounting for the ammonia uncertainty, the hydrogen error bars are increased to ± 0.7 percent for expected concentration calculations.

Thus, assuming maximum errors, the expected composition for the RGS-058 test might be as high as 30.8 percent hydrogen, whereas the actual hydrogen concentration might be as low as 29.9 percent. The worst case hydrogen loss during the RGS-058 sampler aging test would thus be 0.9 percent absolute (2.9 percent, relative) which is acceptable over this 1829 hour (76 day) aging time. This assessment of acceptability is based on FRD compliance; a real hydrogen loss of 0.9 percent combined with the MS measurement error of 0.6 percent would produce a maximum error of 1.5 percent (conservatively), well within the 2.0 percent FRD requirement. There was no evidence during ATR testing that other sources of error such as processing the gas sample through the extractor produces errors exceeding the MS measurement error bars of ± 0.6 percent (see hydrogen composition results for injection tests RGS-005, RGS-006 in Table 12 and RGS-037 in Table 17).

A conservative estimate of hydrogen permeation loss rates has been predicted by A. L. Pitner (see Appendix F) as less than 0.044 percent of the hydrogen inventory in an RGSS sampler per day (at 100 °F). This loss rate would decrease at lower temperatures. For the 76 day RGS-058 aging test reported in Table 23, Pitner would predict the hydrogen loss at less than 3.3 percent (resulting in a change in the hydrogen concentration from the expected value of 30.1 percent to 29.1 percent). This estimate is consistent with the maximum hydrogen loss of 2.9 percent determined above from the RGS-058 data, although the actual loss is probably significantly lower.

7.4.2 Sampler In-Leakage

The maximum oxygen concentration measured was 0.054 percent for aging test RGS-058, and subtracting the 0.007 percent intrinsic oxygen measured for gas MIX#1 (determined from control sample RGS-004) results in a 0.047 percent level of leaked oxygen. This oxygen should correspond to a leakage of 0.23 mL of air (at STP) over the 1829 hour test.

The significance of this amount of leakage depends on the quantity of sample gas; as the total quantity of sample gas decreases, the error percentage due to leakage increases. The FRD specifies a required sample "void fraction" (ratio of STP gas volume to solid/liquid volume) measurement accuracy of 2 percent (absolute). A 2 percent sample void fraction (SVF) would correspond to an STP gas volume of about 6 mL for a nearly full sampler; thus, this is the minimum quantity of retained gas that the FRD requires the RGSS to analyze with full accuracy.

A 0.23 mL quantity of leaked air has negligible effect on the sample void fraction determination for a nearly full sampler; however, it would comprise 3.8 percent of a 6 mL gas sample and would affect composition results. For example, if the sample gas is 30 percent hydrogen, then the hydrogen composition measurement error would be -1.2 percent - which is still within the FRD requirements (not including hydrogen seal permeation and MS measurement errors).

The previous description of the permeation (0.9%) and measurement ($\pm 0.7\%$) errors assumed a worse case combination of these errors; however, statistically, the error band for the combination of these errors can be reduced from 1.6 percent to a ± 1.1 percent error band for hydrogen. This leaves a 0.9 percent allowable error for air leakage, while still meeting the stringent 2 percent FRD accuracy requirement for hydrogen. Assuming a maximum of 50 percent hydrogen in tank gas samples, and an air leak rate constant with time, then this limit is reached in about 36 days.

7.4.3 Sampler Out-Leakage

To date, helium leak test measurements performed on fabricated retained gas samplers have averaged about 5×10^{-7} atm/cc/sec at an internal pressure of 3 atmospheres. The specified maximum leak rate allowed for a retained gas sampler is 1×10^{-6} atm/cc/sec at an internal pressure of 3 atmospheres; this would correspond to an out-leakage of about 0.086 mL/day (at STP).

Assuming the maximum allowed out-leakage of 0.086 mL/day and a 36 day hold time (see in-leakage considerations), the total volume of sample gas lost would be 3.1 mL, or more than half the sample if the original gas volume was 6 mL. This loss of sample gas might affect both sample void fraction and composition determinations, as will be discussed.

The effect of sample gas out-leakage on sample void fraction determination will be examined first. For a given gas sample loss and V_{SL} error, the SVF error increases with smaller initial gas sample and solid/liquid volume. The maximum error in the measurement of V_{SL} during both RGSS proof testing and acceptance testing was less than 20 mL. Starting with the minimum sample gas quantity of 6 mL (as previously discussed) and assuming that V_{SL} can be measured to within ± 20 mL and that out-leakage is at the maximum rate for 36 days, then the 2 percent SVF accuracy requirement (FRD) would be met provided that V_{SL} is greater than 170 mL.

Out-leakage of the sample gas itself would generally not seriously affect composition analysis unless there were a preferential leakage of certain constituent gases; there was no evidence of this effect observed in the long term RGS-058 test. However, the combination of out-leakage of sample gas and the in-leakage of air can seriously alter the sample gas composition measurement. Using spreadsheet software, the maximum hold time for an RGS sampler before testing was determined by assuming the worst case out-leakage rate of 0.086 mL/day and using the "measured" (assuming constant leak rate) air in-leakage rate of 0.23 mL/76 days = 3.03×10^{-3} mL/day. Making these assumptions, then the FRD requirements for both sample void fraction and gas composition accuracy are met provided the maximum hold time for the sampler is 23 days or less, and the V_{SL} is measured at 150 mL or more. (The minimum V_{SL} value is reduced from the previous determination because of the reduction in allowed hold time.)

It should be noted that the air in-leakage rate used in the above hold time determination is based on only two different sampler aging tests, RGS-57 and RGS-58 (assuming that aging for 1 week or longer provides more valid data than the remaining two shorter term tests). As funding priorities allow, it is recommended that additional long term aging tests be performed.

Based on this analysis, the authors recommend that the maximum storage time for samples be less than 23 calendar days at temperatures less than 100 °F. However, if it is not possible to test a stored sampler within this time period, accuracy validation can be performed on a case by case basis depending on the measured gas concentrations and volume determinations.

8.0 EXCEPTIONS TO ATP

The ATP specified that exceptions and deviations from the ATP testing requirements should be documented in this ATR report. Allowed exceptions could include deletion of tests or specification of additional tests as agreed to by the cognizant test director. Throughout this ATR, changes from the ATP description have been identified as the specific tests were discussed. In this section, these exceptions and deviations from the ATP are summarized.

The in situ gas chromatograph was not used in acceptance testing, which is relevant to Section 5.3 of the ATP.

For the Extractor Volume Measurement called out in Section 7.1.2 of both the ATP and ATR, Table 1 of the ATP was modified slightly changing the test procedure and reporting format for the ATR (see Table 3); the primary change was decreasing the "settling time" from 10 minutes to 3 minutes. Also, the RGSS line volume and cabinet volumes were measured using pressure drops and the perfect gas law.

In Section 7.2 of the ATP, four injection tests using two gas mixes were described and required. These four tests were performed as described, although minor changes were made in the test data sheet ("Sampler" changed to "Canister" for volume, temperature and pressure of the injection gas). Also, one control sample analysis for each type of gas used was determined to be adequate, so that (contrary to the ATP) control samples were not obtained for each injection test.

Many (24) additional injection tests were also performed that were not identified or described in the ATP. These additional tests were very helpful in demonstrating the capability of the RGSS to meet the requirements of the FRD.

For all injection tests only the Hg Pump method was used to determine gas quantity, instead of both the SPT and BT methods as suggested in the ATP.

In Section 7.3 of the ATP, simulant extrusion tests are described; a number of changes were made in the tests actually performed from those described in the ATP. Gas composition analysis WAS performed, even though the ATP stated that it would not be. Although the ATP implies that six of these tests would be performed, two (ambient and 50 °C) for each of the three conditions identified, only three tests were performed. Air and Carbopol were not used as simulants; air was not used because gas composition analysis was performed, and Carbopol was eliminated because of the outgassing problems it presented. Since Carbopol simulant was not used, the 2:1 dilution test was deleted. The three tests actually performed demonstrated proper operation and function of the complete RGSS, including sampler attachment and sample extrusion. The data sheet identified in the ATP was modified to eliminate the ASA and change several other features to better match the tests performed. Please compare the ATP data sheet with those given in Appendix C.

Additionally, four sampler aging tests were added to the acceptance test list which were not identified in the ATP. These tests allowed a determination that under normal conditions a sampler could be expected to retain its sample gas without compromising its composition or quantity for up to 76 days after sampling.

9.0 SUMMARY

The RGSS was developed to provide an analysis of both gas quantity and gas composition of samples obtained from certain flammable gas waste tanks at Hanford. The acceptance tests and results described herein were performed to verify the compliance of the RGSS design and fabricated system with the functions and requirements specified in the FRD.

9.1 Testing and Analysis Description

Analysis of the data obtained during acceptance testing was performed using several different techniques. Gas composition analysis was performed with a mass spectrometer (MS) for insoluble gas composition, and with the ammonia solution analysis (ASA) technique to determine the ammonia content. Gas quantity analysis used three different techniques; the Backfill Technique (BT), the Hg Pump method and the Two Volume (TV) method.

Ammonia analysis with the ASA technique was performed by isolating a portion of the test gas in the RGSS 2nd Volume, and fully absorbing ammonia into a slightly acidic solution. The solution was removed and tested with an ion probe to determine the solution's ammonia concentration. The original concentration of ammonia gas in the test gas could then be calculated.

The Backfill Technique was performed after the test sample gas had been transferred to collection canisters for MS composition analysis. Using argon or air, the extractor was repressurized in steps; the data gathered was used with the perfect gas law to determine the bulk volume (V_{SL}) of the solid and liquid portion of the test sample. The test sample's solid/liquid volume is essential for determining the ratio of sample gas to bulk sample volume, as required by the FRD. Also, V_{SL} is essential for the Two Volume analysis determination of gas quantity and soluble gas vapor pressure.

The Hg Pump method simply transfers the test gas from the extractor to the known volumes on the collection side of the transfer pump. The quantity of gas transferred can be calculated using the perfect gas law. However, only insoluble gas is fully transferred, and soluble gas vapor pressure can complicate the analysis.

The Two Volume method consists of measuring the pressure and temperature of the sample gas, first in the extractor, and then again as the previously evacuated 2nd Volume is opened up to the extractor. The data gathered in these two volume conditions, along with V_{SL} measured later allows the determination of the gas quantity and the soluble gas vapor pressure. It should be noted that the TV method relies on the approximation that the soluble gas vapor pressure doesn't change significantly when the system volume expands to include the 2nd Volume. This may not be a good approximation in the case of ammonia, as has been pointed out by Shekarritz and Norton (PNLFGP: 091595). This subject is discussed in Appendix G.

9.2 Acceptance Test Results

Twenty eight gas injection tests were performed, with the results used for both gas composition analysis and gas quantity analysis (limited to the Hg Pump Method). A gas injection test was conducted by putting gas directly into the extractor through the RGSS dilution port, rather than by using a sampler; this was done either with the system dry, or with a prototypic quantity of degassed water (to simulate bulk waste) already in the extractor. Injection tests were simpler and less costly to run than the full simulation tests performed at the end of acceptance testing.

One of three types of gas was used in each injection test; these gas types included two mixes and relatively pure (99.95%) argon. Gas MIX#1 contained ammonia (5.0%), argon (24.9%), hydrogen (30.3%) and nitrogen (39.8%); MIX#2 contained nitrogen (30.0%), nitrous oxide (30.8%) and helium (39.2%). Using two separate gas mixes to simulate expected waste gas components allows the separation of hydrogen and nitrous oxide, which together would be an explosive mixture.

All MS gas composition analyses performed for these test conditions were accurate within the FRD requirements, except for ammonia composition. Ammonia measurement by the ASA method (performed for seven tests) also failed to meet the FRD requirement; although the average of the ASA results was 3.17 percent (where 5.02 percent was expected), four of the seven tests had ammonia compositions below 3 percent and failed to meet the FRD requirement of ± 2 percent absolute accuracy. However, the ASA results were more accurate than the MS ammonia results, which averaged 1.6 percent ammonia.

All injection test gas quantity determinations using the Hg Pump method were within the requirements of the FRD.

Three full simulation tests were performed. For each test, a sampler was filled with a simulation gas (argon or helium) and degassed water. The sampler was attached to the RGSS extractor, and the simulants extruded into the previously evacuated extractor. The 2nd Volume was then connected with the extractor, and isolated (however, no ASA was performed). The simulant gas was transferred to the collection canister and the gas analyzed for composition. The BT procedure was then performed.

The MS composition results for these three tests were all within the FRD requirements. The BT determination of the solid/liquid volume was correct to within 3.5 mL for V_{SL} nominally equal to 285 mL (equivalent to approximately a 1 percent error). The gas quantities determined with the Hg Pump method were all within the FRD requirement, although the vapor pressure determination was too high by roughly 25 percent. Also, the gas quantities determined with the Two Volume method were all within the FRD requirement, except that one test required correction for air outgassing (using oxygen as a tracer, see Appendix D). The vapor pressure determination using the Two Volume method was quite accurate (average error = 1.6 percent).

Sampler "aging" tests were performed in which four different samplers were pressurized with a 30 percent hydrogen gas (MIX#1) at or less than atmospheric pressure. The samplers were allowed to sit for aging times of between 24 to 1829 hours (76 days); then the composition of the sampler gases was determined.

A conservative estimate of hydrogen loss rates has been made by A. L. Pitner as less than 0.044 percent of the hydrogen inventory in an RGSS sampler per day (at 100 °F). This loss rate would decrease at lower temperatures. For the 76 day RGS-058 aging test reported in Table 23, Pitner would predict the hydrogen loss at less than 3.3 percent, resulting in a change in the hydrogen concentration from the expected value of 30.1 percent to 29.1 percent. The measured hydrogen concentration for RGS-058 was 30.5 percent, with an error band of ± 0.6 percent; this result suggests minimal hydrogen loss and some data scatter. In addition, a worst case hydrogen loss (accounting for error bars) was determined from the RGS-058 data as 2.9 percent, which is also within the Pitner estimate.

An analysis of the measured oxygen level of 0.054 percent for the long term RGS-058 aging test was made. It appears that air in-leakage and sample out-leakage may pose a problem for lengthy storage of RGS samplers prior to testing. Combining sampler leakage with possible hydrogen permeation and measurement error considerations, it is concluded that the FRD accuracy requirements might be compromised for sampler storage exceeding 23 calendar days, or at temperatures higher than 100 °F.

9.3 Conclusions

Acceptance testing of the RGSS demonstrated that the system meets all of the functions and requirements specified in the FRD except for ammonia analysis accuracy. It is recommended that this first generation system be deployed to the 222-S hot cell facility and that testing of authentic waste samples be initiated. All of the testing capability of the system should be used, including ASA ammonia analysis; although the ASA didn't meet the FRD requirements, it still provides the best data available at this time for ammonia composition.

The analysis techniques that produce data with the greatest confidence are:

• Insoluble Gas Quantity:	Hg Pump Method
• Bulk Volume (V_{SL}):	Backfill Technique
• Soluble Gas Vapor Pressure:	Hg Pump Method
• Ammonia Concentration:	ASA

Additional study of the ASA ammonia measurement technique will be performed in the future. Efforts will center on determining if the ammonia loss is due to extra stainless steel surface area not present during proof testing, or due to the extractor plastic splash shield (or other potential ammonia absorbers). It is likely that the ASA method can be improved to produce results within the FRD requirements using essentially the same hardware. Until this goal is achieved, reported ammonia concentration data obtained with the RGSS will be footnoted to indicate the RGSS noncompliance with the FRD requirements reported here.

The Two Volume method provided the best data for measuring water vapor pressure during acceptance testing. However, as discussed in Appendix G, when ammonia is present in the waste, the error associated with the TV method of measurement of the ammonia gas pressure may be quite large (up to a factor of two). Thus, the Hg Pump method is recommended for authentic waste sample measurements of soluble gas pressure despite the approximate 25 percent errors encountered in acceptance testing. This recommendation is conditional on the quantity of ammonia measured; when ammonia vapor pressure is small compared to water vapor pressure (less than 25 percent), it is expected that the TV method would provide better results, based on acceptance test data.

As also discussed in Appendix G, the Two Volume method may provide an excellent method for characterizing ammonia properties in waste samples, provided that ammonia vapor pressure can be accurately measured using improved ASA or Hg Pump techniques, and there are no other soluble gases present in significant quantity.

The sampler aging tests performed demonstrated that a delay in RGSS testing of up to 18 calendar days after a waste sample is obtained can be tolerated without significant adverse affects on the accuracy of the gas composition results - provided that the sampler is stored at temperatures of less than 100 °F.

10.0 ACKNOWLEDGEMENTS

The authors wish to express their appreciation to their management for the support and guidance received throughout RGSS acceptance test preparation and performance. Also greatly appreciated was the dedicated and insightful system fabrication and maintenance support provided by M. E. Dahl and many other Equipment Development personnel.

In order to allow familiarization with operation of the RGSS by the receiving group (Process and Development), B. E. Hey performed several of the acceptance tests (see Appendix E); we gratefully acknowledge this contribution.

Page 1 of 1

A-6001-492 (11/94)

TABLE 3
EXTRACTOR VOLUME MEASUREMENT TABLE

- Use argon as working medium. Evacuate lines, 2nd Volume and Extractor. Fill lines to known pressure (recommend about 750 Torr). Record pressure & temperature data; then open valve to 2nd Volume, record data; then open valve to Extractor and record data. Allow 3 minutes for "settling" after each valve opening before taking pressure measurement. Temperature at pressure measurement time should be recorded.
- P1 is starting pressure in Lines; P2 is pressure 2nd Volume + Lines; P3 is pressure in Extractor + Lines + 2nd Volume.

TEST DATE 6-27-95 TEST DESCRIPTION: Acceptance Test -

Test Performer N.S. Cannon

Initial Volume
Measurements

Measured 2nd Volume = $\frac{320.0}{230.0} \times 220.4$ mL

TEST #	P1 Press. (Torr)	Temp. (°C) @ P1	P2 Press. (Torr)	Temp. (°C) @ P2	P3 Press. (Torr)	Temp. (°C) @ P3	Line * Volume (cc)	Extractor * Volume (cc)
1	872.4	20.9	294.2	20.9	84.1	21.0	220.4	1350.0
2	706.0	21.2	288.3	21.2	82.4	21.3	220.9	1351.5
3	754.4	21.8	307.8	21.8	87.8	21.8	220.5	1354.4
4	749.4	22.5	306.0	22.5	87.5	22.6	220.8	1350.6
5	726.0	23.0	296.8	23.1	85.0	23.1	221.3	1348.8
6	724.4	23.2	295.9	23.2	84.8	23.2	221.0	1346.2
7	730.6	23.3	296.6	23.3	85.5	23.3	221.2	1348.0
8	723.2	23.4	295.4	23.4	84.5	23.4	221.0	1350.2
9	736.4	23.5	300.8	23.5	86.1	23.5	221.0	1349.0
10	748.8	23.5	305.8	23.5	87.6	23.5	220.9	1347.3

AVERAGE EXT. VOLUME (mL) 1349.7 STANDARD DEV. (mL) 2.1

Test Witnesses: M. Scott Cannon
Cognizant Engineer

6-27-95
Date

B.J. Hery
Quality Assurance

06-28-95
Date

* Calculated

TABLE 4

CABINET VOLUME MEASUREMENTS

Uses Line Volume (221.3 mL) as starting volume

VE = Volume between Valves B and E VF = Volume between Valves E and F
VH = Volume between Valves F and H
VH1 = Volume between Valves H1 and J1 canister
VH2 = Volume between Valves H2 and J2 canister
VH3 = Volume between Valves H3 and J3 canister
VJ1 = Volume of J1 Canister
VJ2 = Volume of J2 Canister
VJ3 = Volume of J3 Canister
Vsamp = VH + VH1 + VJ1

General Calculation: $V_x = (P1-P2)/P2 * V_s$

VL = 221.3 cc

Test #	Date	Start Press. (Torr)	Dump to E (Torr)	Cal. VE (cc)	Dump to F (Torr)	Cal. VF (cc)	Dump to H1 (Torr)	Cal. VH (cc)	Dump to Through H1 (Torr)	Cal. VH1 (cc)
1	06-29-95	686.1	645.4	13.96	NA	NA	NA	NA	NA	NA
2	06-29-95	648.1	609.7	13.94	591.8	7.12	549.4	18.70	544.2	2.49
3	06-29-95	417.7	393.0	13.91	381.4	7.15	354.0	18.76	350.6	2.53
4	06-29-95	792.5	745.9	13.83	724.0	7.11	672.3	18.63	665.8	2.55
AVERAGE =				13.91		7.13		18.70		2.52

Dump to Through H2 (Torr)	Cal. VH2 (cc)	Dump to Through H3 (Torr)	Cal. VH3 (cc)	Dump in to Can. J1 (cc)	Cal. J1 (cc)	Dump in to Can. J2 (cc)	Cal. J2 (cc)	Dump in to Can. J3 (cc)	Cal. J3 (cc)	Vsmp (J1) (cc)
NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
539.0	2.54	533.9	2.54	465.4	39.54	412.0	39.94	370.3	39.20	60.7
347.3	2.51	344.1	2.48	299.9	39.59	265.4	40.07	238.5	39.28	60.9
659.5	2.52	653.4	2.48	569.6	39.49	504.3	39.87	453.2	39.21	60.7
AVERAGE =		2.52	2.50		39.54		39.96		39.23	60.76

TABLE 5
DETERMINATION OF NEW LINE VOLUME (7-26-95)

2nd Volume = 320.64 cc

Gas	2nd Vol Press. (KPa)	Start Line P. (KPa)	Combined Press. (KPa)	Temp. (C)	Calcul. Line Vol. (cc)
Air	99.67	0.024	61.40	21.6	199.85
Argon	99.26	0.022	61.18	21.7	199.57
Argon	61.18	0.021	37.71	21.7	199.56
Air	99.65	0.027	61.33	21.8	200.34
Air	61.33	0.018	37.77	21.7	200.01
Argon	100.10	0.020	61.70	21.7	199.56
Argon	61.70	0.025	38.01	21.7	199.84
Air	99.46	0.030	61.21	21.8	200.37
Air	61.21	0.030	37.67	21.8	200.37
Air	37.67	0.023	23.20	21.6	199.99
AVERAGE =					199.95
STD =					0.31

TABLE 6
DETERMINATION OF NEW EXTRACTOR VOLUME (8-15-95)

Line + 2nd Volume = 520.6 cc

Gas	Line + 2nd Vol Press. (KPa)	Start Ext. P (KPa)	Combined Press. (KPa)	Temp. (C)	Calcul. V Extractor (cc)
Air	99.33	0.250	27.76	21.9	1354.37
Air	99.37	0.690	28.10	21.9	1353.61
Air	99.40	0.700	28.12	21.9	1353.31
Air	99.43	0.820	28.21	21.9	1353.65
Air	99.41	0.790	28.19	21.9	1353.15
Air	99.44	0.820	28.20	21.9	1354.52
Air	99.38	0.830	28.19	21.9	1354.56
Air	99.40	0.820	28.19	21.9	1354.45
Air	99.41	0.790	28.18	21.9	1353.84
Air	99.40	0.810	28.18	21.9	1354.64
AVERAGE =					1354.01
STD =					0.53
%STD =					0.04

TABLE 7

EXTRACTOR/SAMPLER INTEGRATION TEST

- Using air as working medium, fill Sampler to known pressure and evacuate the Extractor/2nd Volume. Perform SPT testing to verify proper functioning of the RGSS, and that volumes have been correctly measured. Allow 10 minutes for "settling" after each valve opening before taking pressure measurement. Temperature at pressure measurement time should be recorded.
- P1 is starting pressure in 2nd Volume, P2 is resulting pressure in Extractor.

TEST DATE 7-14-95 TEST DESCRIPTION: Integration Test
Test Performer C Knight No. 10

SAMPLER ID <u>#1</u>		SAMPLER VOLUME <u>314.63</u> (mL)				
SAMPLER PRESSURE <u>754.8</u> (Torr)		TEMPERATURE <u>between 21 - 25.1</u> (°C)				
(WORKING GAS = AMBIENT AIR)						
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000] (Torr)	Sensor 2 [100] (Torr)	Sensor 3 [10] (Torr)
SPT	Zero	9:31 ^{pm}	25.1	.05	.006	.006
	Leak Test Start	2:32 ^{pm}	25.1	.05	.006	.006
	Leak Test End	2:42	25.1	.06	.017	.017
	Extractor Start	2:46	25.1	20.37	—	—
	Extractor End	2:56	25.1	20.38	—	—
	2nd Vol. Start	2:57	25.1	16.95	16.942	—
	2nd Vol. End	3:07	25.1	16.96	16.956	—

AVERAGE EXT. VOLUME (mL) 1570.6 2ND VOLUME (mL) 320.0

Test Witnesses: M. J. Connor
Cognizant Engineer
B. J. Hugg
Quality Assurance

7-14-95
Date
07-14-95
Date

TABLE 8

Scott Specialty Gases, Inc.

Shipped 2330 HAMILTON BOULEVARD
From: SOUTH PLAINFIELD NJ 07080 Fax: 908-754-7303
Phone: 908-754-7700

CERTIFICATE OF ANALYSIS

WESTINGHOUSE HANFORD PROJECT #: 07-23919-001
MAY PRETORIUS PGM: WAP-YVV40795
BLDG 1163 ITEM #: 0702A4023434A
2355 STEVENS DRIVE DATE: 4/07/95
RICHLAND WA 93352

CYLINDER #: 1A010613

ANALYTICAL ACCURACY: SEE BELOW

ELEND TYPE : CERTIFIED MASTER GAS

COMPONENT	REQUESTED GAS	ANALYSIS
	CONC. MOLES	(MOLES)
AMMONIA	5. %	5.02 %
ARGON	25. %	24.9 %
HYDROGEN	30. %	30.3 %
NITROGEN	BALANCE	BALANCE

Arbitrarily designated as MIX # 1 (M/C)

ANALYTICAL ACCURACY +/- 1% FOR
ANALYTICAL ACCURACY +/- 2% FOR

ARGON & HYDROGEN
AMMONIA

ANALYST:

Thomas De Long
THOMAS DE LONG

BEST AVAILABLE COPY

PLUMSTEADVILLE, PENNSYLVANIA / TROY, MICHIGAN / HOUSTON, TEXAS / DURHAM, NORTH CAROLINA
SOUTH PLAINFIELD, NEW JERSEY / FREMONT, CALIFORNIA / WAKEFIELD, MASSACHUSETTS / LONGMONT, COLORADO
BATON ROUGE, LOUISIANA

TABLE 9

RECEIVED

PAGE 1

APR 11 1995

ACCOUNTS PAYABLE

Scott Specialty Gases, Inc.

2600 CAJON BOULEVARD, SAN BERNARDINO, CA 92411

(909) 887-2571

Fax: (909) 887-0549

CERTIFICATE OF ANALYSIS

WESTINGHOUSE HANFORD

PROJECT #: 02-37224-001

BLDG 1163

PO#: WAP-YVV407950

2355 STEVENS DRIVE

ITEM #: 0202N3016714A

RICHLAND

WA 99352

DATE: 4/03/95

CYLINDER #: X94572J

ANALYTICAL ACCURACY: +/-2%

FILL PRESSURE: 1900PSIG

BLEND TYPE : CERTIFIED MASTER GAS

COMPONENT

NITROGEN

NITROUS OXIDE

HELIUM

REQUESTED GAS

CONC MOLES

30.0 %

30.0 %

BALANCE

ANALYSIS

(MOLES)

30.0 %

30.8 %

BALANCE

Arbitrarily designated as MIX #2 (gdc)

1900PSIG BIN#8 MB/RG 04/10

ANALYST:

BEST AVAILABLE COPY

FREMONT, CALIFORNIA SAN BERNARDINO, CALIFORNIA LONGMONT, COLORADO CHICAGO, ILLINOIS BATON ROUGE, LOUISIANA
WAKEFIELD, MASSACHUSETTS TROY, MICHIGAN DURHAM, NORTH CAROLINA SOUTH PLAINFIELD, NEW JERSEY
AVON LAKE, OHIO SARINIA, ONTARIO HOUSTON, TEXAS PLUMSTEADVILLE, PENNSYLVANIA BREDA, THE NETHERLANDS

INJECTION TEST DATA SHEET

WMC-SD-WM-ATR-137
Rev. 1
Page 43TEST DATE 6-29-95Test Performer N. S. CannonGAS MIX ID MIX #2 (N₂O+)CANISTER TEMP. 29 °CCANISTER VOL. (mL) 41.3CANISTER PRESS. 102.6 KPa (769.2 Torr)

TEST TYPE	TEST STEP	TIME (PM)	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	4:29	26.7	0.04	0.013	0.0120
	Leak Test Start	4:29	26.7	0.04	0.013	0.0120
	Leak Test End	4:35	26.7	0.06	0.028	0.0279
	Extractor Start	4:36:20	26.7	2.64	2.666	OFF S.
	Extractor End	4:43	26.7	2.64	2.667	OFF S.
	2nd Vol. Start	4:43:30	26.7	2.21	2.239	OFF S.
	2nd Vol. End	4:52	26.6	2.22	2.244	OFF S.

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃							

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							

ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	0	0	30.8	30.0	39.2	0	0
CONTROL	*	*	30.4	30.3	39.2	*	*
TEST	*	*	29.0	32.7	37.1	0.1	O ₂ = 1.0 %

Test Witnesses: N. Scott Cannon
Cognizant Engineer6-30-95
DateQuality Assurance7-12-95
Date

* Less than 0.1%

PWL Report #

95-06959

95-06958

RGS-002

RGS-001

INJECTION TEST DATA SHEET

 WHC-SD-WM-ATR-137
 Rev. 1
 Page 44
TEST DATE 6-30-95Test Performer N.S. CannonGAS MIX ID MIX #2CANISTER TEMP. 26 °CCANISTER VOL. (mL) 41.3CANISTER PRESS. 103.97 KPa (779.8 Torr)

TEST TYPE	TEST STEP	TIME AM	TEMP. (°C) TC # 2	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	10:10	52.7	0.05	0.002	0.002
	Leak Test Start	10:14	52.7	0.05	0.003	0.003
	Leak Test End	10:22	52.7	0.05	0.009	0.009
	Extractor Start	10:23	52.7	2.76	2.777	OFF S.
	Extractor End	10:34	52.7	2.77	2.782	OFF S.
	2nd Vol. Start	10:34:15	52.7	2.33	2.340	OFF S.
	2nd Vol. End	10:45	52.6	2.34	2.347	OFF S.

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃							

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							

ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	0	0	30.8	30.0	39.2	0	0
CONTROL	*	*	30.4	30.3	39.2	*	*
TEST	*	*	30.2	30.7	38.7	*	O ₂ = 0.3 %

PNL Report #

95-06959

95-07005

Test Witnesses: M. Scott Cannon
Cognizant Engineer6-30-95
DateQuality Assurance7-10-95
Date

* Less than 0.1 %

TABLE 12
Summary of Required Injection Test Results

GAS COMPOSITION ANALYSIS								
Test ID	Type	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	O ₂ (%)
MIX#2	Vendor	--	--	30.8	30.0	39.2	--	--
RGS-002	Control	--	--	30.4	30.3	39.2	--	0.02
RGS-001	26.7 °C	--	--	29.0	32.7	37.1	0.1	1.04
RGS-001	Leak Adj.	--	--	30.5	30.3	39.0	0	0
RGS-003	52.7 °C	--	--	30.2	30.7	38.7	--	0.27
RGS-003	Leak Adj.	--	--	30.6	30.1	39.2	0	0
MIX#1	Vendor	5.0	30.3	--	39.8	--	24.9	--
RGS-004	Control	2.9	29.2	--	41.7	--	25.9	--
MIX#1 Vendor	NH3 Loss Compensated	<u>2.9</u>	31.0	--	40.7	--	25.4	--
RGS-005	28.0 °C	1.4	28.2	--	43.9	--	25.6	0.78
MIX#1 Control	NH3 Loss Compensated	<u>1.4</u>	29.7	--	42.5	--	26.4	--
RGS-005	Leak Adj.	1.45	29.3	--	42.5	--	26.6	0
RGS-006	52.4 °C	1.8	29.4	--	42.4	--	26.0	0.30
MIX#1 Control	NH3 Loss Compensated	<u>1.8</u>	29.6	--	42.3	--	26.3	--
RGS-006	Leak Adj.	1.83	29.8	--	41.9	--	26.4	0

GAS QUANTITY ANALYSIS - HG PUMP METHOD							
Test ID	Gas Mix	Starting Inj. Gas at STP (mL)	Final Canister Pressure (KPa)	Final Canister Temp. (°C)	Final Canister Gas @STP (mL)	Adjusted (2ndV) Gas @STP (mL)	Gas Transf. Error (mL)
RGS-001	MIX#2	37.80	63.61	32.3	33.86	40.76	3.0
RGS-003	MIX#2	38.69	59.09	31.9	31.49	37.91	-0.8
RGS-005	MIX#1	36.99	59.57	32.3	31.71	38.17	1.2
RGS-006	MIX#1	37.46	56.43	29.5	30.32	36.49	-1.0

INJECTION TEST DATA SHEET

WHC-SD-WM-ATR-137
Rev. 1
Page 46TEST DATE 7-11-95Test Performer N.S. CannonGAS MIX ID MIX#1 (NH₃ +)CANISTER TEMP. 28 °CCANISTER VOL. (mL) 41.3CANISTER PRESS. 100.07 KPa (750.5 Torr)

TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	2:50 pm	28.0	0.05	0.014	0.0138
	Leak Test Start	2:55	28.0	0.05	0.014	0.0138
	Leak Test End	3:05	28.0	0.09	0.067	0.0662
	Extractor Start	3:07:30	28.1	2.62	2.637	OFF S
	Extractor End	3:18	28.0	2.59	2.621	OFF S
	2nd Vol. Start	3:19	28.0	2.21	2.237	OFF S
	2nd Vol. End	3:29	28.0	2.24	2.267	OFF S

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	52.3	3:31	28.0	2.267	7-13-95	1.07x10 ⁻⁷	2.1 *

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

* Adjusted for leak rate

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	5.02	30.3	0	39.8	0	24.9	0
CONTROL	2.9	29.2	0.0	41.7	0	25.9	methoxy 0.2
TEST	1.4	28.2	0.0	43.9	0	25.6	O ₂ = 0.8 N ₂ = 0.1

Test Witnesses: N. Scott Cannon
Cognizant Engineer7-17-95
Date[Signature]
Quality Assurance7-17-95
Date

* Computer time - about 19 minutes fast.

Data file RGS-005. INT

INJECTION TEST DATA SHEET

WMC-SD-WM-ATR-137
Rev. 1
Page 47TEST DATE 7-12-95Test Performer N.S. CannonGAS MIX ID MIX #1 (NH₃ +)CANISTER TEMP. 25°C (e 11:27AM - Filled @ 11:00AM)CANISTER VOL. (mL) 41.3CANISTER PRESS. 752.5 Torr

TEST TYPE	TEST STEP	TIME	TEMP. (°C) Chen 2	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	1:11 PM	52.4	0.03	.004	.0043
	Leak Test Start	1:12	52.4	0.03	.004	.0043
	Leak Test End	1:22	52.4	0.04	.019	0.0197
	Extractor Start	1:23	52.4	2.27 2.71	6.021	6.0210
	Extractor End	1:34	52.4	2.71	2.743	OFF Scale
	2nd Vol. Start	1:35	52.4	2.27	2.301	OFF Scale
	2nd Vol. End	1:46	52.3	2.27	2.310	OFF Scale

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V TEMP. (°C) Chen 2	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	51.9	1:47	51.1	2.31	7-13-95	1.37x10 ⁻⁷	2.7 % *

RGS-006

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

* Adjusted for Leak Rate

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							

ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	5.02	30.3	0	39.8	0	24.9	0
CONTROL	2.9	29.2	0.0	41.7	0	25.9	Methane 0.2
TEST	1.8	29.4	0.0	42.4	0.0	26	O ₂ = 0.3 Methane = 0.1

RGS-004

RGS-006

Test Witnesses: N. Scott Cannon
Cognizant Engineer7-17-95
Date[Signature]
Quality Assurance7-17-95
Date

Data file RGS-006.1NT

TABLE 15
Ammonia Solution Analysis (ASA) on MIX#1 Control Samples

MIX#1 Sample Acquisition					First Solution			Second Solution		
6-13-95: D-1 through D-2 6-14-95: D-3 through D-7					Analysis 6-15-95 Start Cal(100)= 98.1 End Cal(100) = 117			Analysis 6-16-95 Start Cal(100)= 98.1 End Cal(100) = 117		
Test Name	Fill Canister Vol. (mL)	Fill Pressure (Torr)	Fill Temp. (°C)	Solution Volume (mL)	Analysis Order	Raw NH ₃ (%)	Adj. NH ₃ (%)	Analysis Order	Raw NH ₃ (%)	Adj. NH ₃ (%)
D-1	42.0	12.0	22	40.36	1	6.59	6.72	7	9.23	7.27
D-2	40.9	48.3	22	38.55	2	5.82	5.75	6	7.54	6.13
D-3	42.0	99.4	22	43.34	3	4.26	4.08	5	5.16	4.34
D-4	40.9	188.2	21	41.08	4	6.39	5.94	4	6.23	5.42
D-5	42.0	250.2	21	43.29	5	3.65	3.30	3	3.85	3.47
D-6	40.9	399.7	21	47.47	6	5.29	4.65	2	5.29	4.94
D-7	42.0	750.1	22	43.07	7	4.96	4.24	1	4.53	4.40
Note: Solution Volume was divided approximately in two, allowing two separate analyzes to be performed.					AVERAGE=	5.28	4.95	AVERAGE=	5.98	5.14
					STD =	1.00	1.12	STD =	1.73	1.17

The Starting Calibration was performed before the first sample analysis was performed, and the Ending Calibration was performed after the last analysis was performed for a given analysis sequence. Some calibration drift was noted.

$$\text{ADJUSTED NH}_3 \% = \text{RAW NH}_3 \% * (100 / [\text{START} + (\text{END} - \text{START}) / 6 * (\text{ORDER} - 1)])$$

Here, START is the Starting Calibration value, END is the Ending Calibration value, and ORDER is the test order number. Also, the standard deviation for the Adjusted NH₃ difference between the two analysis sequences was only 0.31%, suggesting that the major source of error was in ammonia absorption into solution, rather than from the ion probe.

TABLE 16

Injection Tests - MIX#1 Ammonia Analysis

Test ID	Test Date	Fill Canister Volume (mL)	Fill Pressure (KPa)	Fill Temp. (°C)	Solution Volume (mL)	Gas NH ₃ (%)	QA Overs. (Y/N)
RGS-005	7-11-95	320.64	2.267	28.0	52.3	2.1	Y
RGS-006	7-12-95	320.64	2.31	51.1	51.9	2.7	Y
A-1	7-17-95	320.64	NA	Amb.	NA	4.6	N
A-2	7-17-95	320.64	NA	≈ 50	NA	4.7	N
RGS-008	7-17-95	320.64	2.16	25.4	40.3	3.5	Y
RGS-009	7-18-95	320.64	2.368	52.2	40.4	2.7	Y
RGS-020	8-08-95	320.64	2.205	21.9	38.8	2.0	N
Expected NH ₃ Concentration = 5.02 %						Average =	
						Standard Deviation =	

TABLE 17
Wet (Degassed) Mixed Gas Injection Test Composition Results

GAS COMPOSITION ANALYSIS								
Test ID	Comment	NH₃ (%)	H₂ (%)	N₂O (%)	N₂ (%)	He (%)	Ar (%)	O₂ (%)
MIX#2	Vendor	--	--	30.8	30.0	39.2	--	--
RGS-002	Control	--	--	30.4	30.3	39.2	--	0.02
RGS-025	37.6mL @STP	--	--	29.6	31.4	38.3	--	0.43
RGS-026	37.7mL @STP	--	--	30.0	30.6	39.1	--	0.13
RGS-027	20.1mL @STP	--	--	29.9	30.5	39.3	--	0.14
RGS-028	10.1mL @STP	--	--	29.0	31.7	38.1	--	0.95
RGS-029	2.5mL @STP	--	--	15.7	46.3	26.0	0.6	11.0
MIX#1	Vendor	5.0	30.3	--	39.8	--	24.9	--
RGS-004	Control	2.9	29.2	--	41.7	--	25.9	--
MIX#1 Control	NH3 Loss Compensated	0	30.2	--	43.1	--	26.8	--
RGS-037	37.2mL @STP	0	29.6	--	43.6	--	26.4	0.39

TABLE 18
Wet (Degassed) Mixed Gas Injection Test Quantity Results

GAS QUANTITY - AT STP				
Test ID	Start Quantity (mL)	Hg Pump Transfer (mL)	Hg Pump Error (%)	Hg Pump Error (mL)
RGS-025	37.62	33.67	-10.51	-3.95
RGS-026	37.70	33.80	-10.33	-3.90
RGS-027	20.12	18.85	- 6.32	-1.27
RGS-028	10.06	7.96	-20.91	-2.10
RGS-029	2.52	0.68	-73.01	-1.84
RGS-037	35.36*	30.81	-12.86	-4.55

* Assumes total washout of ammonia, as expected and demonstrated in Table 17.

TABLE 19
Wet (Degassed) Argon Gas Injection Test Composition Results

Test ID	COMMENTS (Volumes given in mL are at STP)	Ar (%)	O ₂ (%)	N ₂ (%)	Error (%)
RGS-032	Control	99.95	0	0.02	NA
RGS-044	Control	99.95	0.01	0.03	NA
RGS-033	BH#2 Pumped; Start 37.11 mL	98.37	0.35	1.17	1.6
RGS-034	BH#3 Dumped; Same as RGS-035	98.28	0.22	0.6	1.7
RGS-035	BH#3 Pumped; Start 20.11 mL	99.68	0.08	0.21	0.3
RGS-036	BH#4 Pumped; Start 20.09 mL	99.6	0.08	0.26	0.4
RGS-038	Pumped; Start 2.55 mL	82.8	2.7	13.5	17.2
RGS-039	Pumped; Start 5.11 mL	96.32	0.76	2.71	3.6
RGS-043 B	BH#5 Pumped; Start 5.04 mL	94.4	1.73	3.44	5.6
RGS-043 C	BH#5 Dumped	94.5	1.49	2.48	5.5
RGS-045 A	BH#6 Dumped	85.9	2.73	2.22	14.1
RGS-045 B	BH#6 Pumped; Start 5.04 mL	97	0.98	1.78	2.6
RGS-046 A	BH#7 Dumped	31.2	17.2	18.9	68.8
RGS-046 B	BH#7 Pumped; Start 3.77 mL	95.65	1.4	2.48	4.3
RGS-047	50.6 °C; Pumped; Start 20.14 mL	99.25	0.09	0.29	0.7
RGS-048 A	Dumped	92.5	1.55	5.33	7.5
RGS-048 B	Pumped; Start 10.06 mL	93.7	1.37	4.84	6.3
RGS-049	50.1 °C; Pumped; Start 10.14 mL	98.58	0.15	0.85	1.4
RGS-050	50.1 °C; Pumped; Start 5.04 mL	97.38	0.32	1.34	2.6

"Dumped" means the analysis canister was opened directly to the extractor; "Pumped" refers to use of the Hg transfer pump to move most (99%) of the gas from the extractor to the analysis canister.

TABLE 20
Wet (Degassed) Argon Injection Test Gas Quantity (@STP) Results

Test ID	Comment	Expected V_g (mL)	RESULT: Hg Pump with P_v (mL)	Error (mL)	RESULT: Hg Pump $P_v = 0$ (mL)	Error (mL)
RGS-033	Single	37.11	34.79	- 2.32	37.02	- 0.25
RGS-035	Dual: RGS-034	20.11	16.70	- 3.41	18.94	- 1.17
RGS-036	Single	20.09	14.88	- 5.21	17.11	- 2.98
RGS-038	Single	2.55	1.51	- 1.04	3.51	0.96
RGS-039	Single	5.11	2.73	- 2.39	4.60	- 0.52
RGS-043C	Dual with B	5.04	2.43	- 2.61	4.35	- 0.69
RGS-045B	Dual with A	5.04	2.05	- 2.99	3.98	- 1.07
RGS-046B	Dual with A	3.77	1.85	- 1.92	3.74	- 0.03
RGS-047	Single @ 50.6 °C	20.14	7.64	-12.51	9.55	-10.60
RGS-048B	Dual with A	10.06	6.50	- 3.56	8.42	- 1.64
RGS-049	Single @ 50.1 °C	10.14	6.10	- 4.04	8.02	- 2.13
RGS-050	Single @ 50.1 °C	5.04	2.27	- 2.77	4.22	- 0.82
Excluding RGS-047 (extra ammonia absorption):				Average =		- 1.11
				STD =		0.81

TABLE 21
Simulant Extrusion Test Gas Quantity Results

GAS QUANTITY BT/TV ANALYSIS - @STP											
Test ID	Max. Temp. (°C)	Gas Type	Expected V_{SL} (mL)	BT Meas. V_{SL} (mL)	Expected V_g (mL)	TV Meas. V_g (mL)	V_g Error (mL)	V_g Error (%)	Expect. P_v (KPa)	TV Meas. P_v (KPa)	Error (%)
RGS-051	21.0	Argon	287.0	288.36	25.7	26.8	1.1	4.3	2.487	2.53	1.7
RGS-052	50.5	Argon	284.2	280.63	28.1	35.8	7.7	27.2	2.502	2.49	-0.4
RGS-053	51.3	Helium	287.5	284.06	25.3	28.7	3.4	13.4	2.487	2.53	1.9

GAS QUANTITY Hg PUMP ANALYSIS - @STP											
Test ID	Max. Temp. (°C)	Gas Type	BT Meas. V_{SL} (mL)	Expected V_g (mL)	RESULT: Hg Pump with P_v (mL)	Error (mL)	RESULT: Hg Pump $P_v = 0$ (mL)	Error (mL)	Exp. P_v (KPa)	Hg P. P_v (KPa)	Error (%)
RGS-051	21.0	Argon	288.36	25.7	21.0	-4.7	23.0	-2.7	2.487	3.00	20.6
RGS-052	50.5	Argon	280.63	28.1	25.5	-2.6	27.6	-0.5	2.502	3.13	25.1
RGS-053	51.3	Helium	284.06	25.3	20.8	-4.5	23.0	-2.3	2.487	3.21	29.1

TABLE 22

Simulant Extrusion Test Gas Composition Results

GAS COMPOSITION ANALYSIS						
Test ID	Maximum Temperature (°C)	Expected Gas Type	Argon (%)	Helium (%)	Oxygen (%)	Nitrogen (%)
RGS-032 Control	NA	Argon	99.95	0.02	0	0.02
RGS-044 Control	NA	Argon	99.95	0	0.01	0.03
RGS-051	21.0	Argon	97.98	0	0.18	1.8
RGS-052	50.5	Argon	91.3	0	1.6	7.0
RGS-054 Control	NA	Helium	0	99.78	0.02	0.19
RGS-053	51.3	Helium	0.07	96.89	0.47	2.46

TABLE 23
Sampler Aging Test Gas Composition Results

Test ID	Description/Comment	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	O ₂ (%)
MIX#1	Vendor Certification (see Table 8)	5.0	30.3	--	39.8	--	24.9	--
RGS-004	Control: PNNL IMS Lab Analysis (7-12-95 Log-in No. 95-07111)	2.9	29.2	--	41.7	--	25.9	0.007
RGS-055	Aged 24 hrs (1 d) IMS Lab Analysis (9-13-95 Log-in No. 95-08647)	1.3	31.0	--	41.6	--	26.1	0.022
COMP 1	<u>Expected</u> Composition based on RGS-004 (Control) Compensation Calculated for NH ₃ Loss.	<u>1.3</u>	29.8	--	42.5	--	26.4	0.01
RGS-056	Aged 74 hrs (3.1 d) IMS Lab Analysis (9-15-95 Log-in No. 95-08719) [See COMP 1]	1.3	30.5	--	41.9	--	26.2	0.022
RGS-057	Aged 187 hr (7.8 d) IMS Lab Analysis (9-21-95 Log-in No. 95-08778)	0.04	31.3	--	42.2	--	26.4	0.010
COMP 2	<u>Expected</u> Composition based on RGS-004 (Control) Compensation Calculated for NH ₃ Loss.	<u>0.04</u>	30.2	--	43.1	--	26.7	0.01
RGS-058	Aged 1829 hrs (76 d) IMS Lab Analysis (11-30-95 Log-in No. 96-01643) Sampler gas pressure was 0.33 atm.	0.23	30.5	--	42.7	--	26.5	0.054
COMP 3	<u>Expected</u> Composition based on RGS-004 (Control) Compensation Calculated for NH ₃ Loss.	<u>0.23</u>	30.1	--	43.0	--	26.7	0.01

* Ammonia loss during PNNL's Mass Spectrometer analysis will alter the relative composition of other gas components. Calculations were performed (as described in Appendix D) to compensate for the decreased ammonia concentrations actually measured; these calculations (based on RGS-004 control data) produced "Expected" Compositions (COMP 1, 2, and 3) for comparison with test results. The RGS-004 control data was used for the Expected Composition calculations because this allows these comparisons to all be made with gas composition analysis performed by the same high accuracy mass spectrometer.

FIGURE 1. RETAINED GAS SAMPLING SYSTEM

306E LAYOUT

BEST AVAILABLE COPY

FIGURE 2. EXTRACTOR AND 2nd VOLUME DETAILS.

FIGURE 3. GAS COMPOSITION ERROR ANALYSIS.

FIGURE 4. GAS QUANTITY ERROR ANALYSIS

FIGURE 5. GAS COMPOSITION DIFFERENCE (MEASURED - EXPECTED) IS PLOTTED VERSUS SAMPLER AGING TIME FOR HYDROGEN, ARGON AND NITROGEN.

APPENDIX A

RGSS Instrumentation Calibration

All of the RGSS instrumentation requiring calibration for Acceptance Testing are listed below; this list includes the location of calibration data for each instrument or transducer.

INSTRUMENT	WHC STANDARDS LAB #	CALIBRATION TABLE #	CALIBRATION DATES
MKS Baratron 622 Pressure Transducer (10 Torr)	559-80-02-012	A-1 A-2	9-01-94 to 9-01-95 9-15-95 to 9-15-96
MKS Baratron 622 Pressure Transducer (100 Torr)	559-80-02-013	A-3 A-4	9-01-94 to 9-01-95 9-15-95 to 9-15-96
MKS Baratron 622 Pressure Transducer (1000 Torr)	559-80-02-011	A-5 A-6	9-01-94 to 9-01-95 9-14-95 to 9-14-96
HP3497A DACU	835-67-11-001	A-7	5-30-95 to 5-30-96
Type K Thermocouples, PO WfJ-XV- 257890 from ARI.	NA	A-8	NA

The MKS pressure transducers were recalibrated at the end of the acceptance testing; as noted in Tables A-4 and A-6, the 100 and 1000 Torr transducers, respectively, were found to be out of specification. Table A-9 contains a summary of the calibration error measured for these two transducers. Pressure data errors affect only the BT, TV and Hg Pump method gas quantity/void fraction results. The consequences of these potential errors on the acceptance test results will be evaluated in the following.

For the BT method, during acceptance testing, only the 1000 Torr transducer was used. The BT analysis uses only a difference in pressure measurements to determine sample solid/liquid volume (see Equation 2); thus, the transducer error is nearly eliminated in the V_{sl} calculation since the pressure error is relatively constant (between 1.7 and 2.6 Torr) over the transducer's range (see Table A-9). For example, in the case of RGS-053, the pressure transducer error for the measured pressures would be expected to produce a high value for V_{sl} by about 0.2 percent.

The Two Volume method calculates gas quantity based on the difference between pressure measurements (see Equation 5), also. However, the 100 Torr MKS transducer used for the TV measurements has more variability in the error over the range of the transducer than for the 1000 Torr transducer. The actual pressure measurements for the three tests of interest (RGS-051, RGS-052 and RGS-053) were between 4 and 5.3 KPa (30 to 40 Torr). Based on the

recalibration data given in Table A-9, the worst case pressure difference error might conservatively be as large as 0.18 Torr, or 0.024 KPa. This would correspond to overpredictions of gas quantity of 5.3 percent (RGS-051), 4.0 percent (RGS-052), and 4.9 percent (RGS-053). Compensation for these pressure measurement errors would actually improve the results presented in Table 21.

The Hg Pump method for determining total gas quantity again uses a difference in pressure readings (see Equation 4). The P_f pressure measurements were based entirely on the 1000 Torr transducer; however, the P_v pressure measurements were most often obtained using the 100 Torr transducer, and always ranged between 20 and 30 Torr. In cases where both pressure measurements were based on the 1000 Torr transducer, the gas quantity would have been overestimated (due to pressure measurement error) by no more than 0.3 percent, a small effect. In the cases where P_v was determined with the 100 Torr transducer, the error in the pressure difference $P_f - P_v$ might have been as large as 2.4 Torr.

The effect of this potential pressure measurement error on gas quantity determination is summarized in Table A-10 for all reported Hg Pump results, assuming that a worst case pressure difference error of 2.6 Torr (0.35 KPa) occurred. The "corrected" error in gas quantity measurement given in Table A-10 in units of volume (mL) is essentially the same as that given in Tables 12, 18, 20, and 21; all errors (except for test RGS-047) remain within the range allowed by the FRD of 6.3 mL for a full sampler. (The RGS-047 result continues to be out of specification, presumably because the abnormally low extractor temperature of 15.6 °C during gas transfer allowed more gas to remain dissolved in solution, as previously discussed.)

Thus, acceptance test results for gas quantity and void fraction were only slightly altered by assuming worst case pressure measurement errors resulting from the calibration drift in the two MKS pressure transducers. The error analysis made using worst case pressure data assumptions demonstrated that the RGSS continued to meet the FRD requirements under these conditions.

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS L6-38		STANDARDS CODE NUMBER 559-80-02-012		NEW MODIFY ☒ 384156		REFERENCE NUMBER 384156	
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 10 TORR		SERIAL NUMBER 94153108A		PROPERTY NUMBER N/A		RECALL STATUS 1. ACTIVE 2. NONRECALL 3. SUSPENDED 2 4. DELETED 5. PH 6. NONDATA MLT	
SENDER JEFF 6-3988		ROOM 267		BUILDING 306E		DATE RECEIVED 360	
INSTRUMENT SPECIFICATIONS ±.5% of RGS ± 2 DIGIT, ASS 16450 WSL		COMMENTS W/RECAL MKS SN# 94202107A		SERVICE DEPARTMENT 5		TOLERANCE HISTORY TOLERANCE AS RECEIVED 1 IN 2 OUT 3 NA 4 FAILED	
STANDARD(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS 102-31-017		EXPIRATION DATE 4-27-95		EXPIRATION DATE		TRAINING HOURS	
REMARKS		REPAIR HOURS		OTHER HOURS		CALIBRATION HOURS 20	
PROCEDURE NUMBER WMC-IHP-PRESSA-REV-2 WM-5-MKS 622 R		TOTAL CHARGE = (3120 * SUM OF HOURS) * MATERIAL		DATE CALIBRATED 9-1-94		DATE DUE 9-1-95	
TEST POINTS		AS FOUND		FINDING		TOLERANCE	
mm Hg		mm Hg		mm Hg		mm Hg	
2.00		2.000		same		±.011	
3.970		3.990		↓		±.021	
5.978		6.162				±.031	
7.987		8.011				±.0241	
9.985		10.007				±.057	
<div style="border: 1px solid black; padding: 10px; text-align: center;"> <p>TABLE A-1 MKS 10 TORR TRANSDUCER RGSS Initial Calibration</p> </div>							
APPROVED BY JLBell 9-6-94		CALIBRATED BY 46		Banford Operations and Engineering Contractor for the United States Department of Energy		Westinghouse Banford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352	
						PAGE 1 OF 1	

BEST AVAILABLE COPY

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS L6-38		STANDARDS CODE NUMBER 559-80-02-012		NEW MODIFY ORGANIZATION CODE WOM530		REFERENCE NUMBER 394002 WORK ORDER N2D2A	
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 10 TORR		SERIAL NUMBER 94153108A	PROPERTY NUMBER N/A	RECALL STATUS 1 ACTIVE 2 NONRECALL 3 SUSPENDED 4 DELETED 5 PM 6 NONDATA MATE	RECALL CYCLE 360	TOLERANCE HISTORY N	
SENDER JEFF 6-3988		ROOM 267	BUILDING 306E	SERVICE DEPARTMENT 5	DATE RECEIVED 950914	TOLERANCE AS RECEIVED 1 IN 2 OUT 3 NA 4 FAILED	
INSTRUMENT SPECIFICATIONS		COMMENTS W/MKS SN#94202107A ASS. +/- .5%RDG+/-1DIG				SHIPPING DAY WE	
STANDARD(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS				TRAINING HOURS			
A:1 RATIO Y ☐ N ☒				CALIBRATION HOURS 2.0			
EXPIRATION DATE 6-1-31-04-02-6 10-2-95				REPAIR HOURS			
				OTHER HOURS			
REMARKS 11560 BEST DEVICE POSSIBLE				MATERIALS			
PROCEDURE NUMBER WHC-5-9-PRESSGA REV 3a Vm-5-mks 6227				TOTAL CHARGE (\$122 x SUM OF HOURS) + MATERIAL			
				DATE CALIBRATED 9-15-95			
				DATE DUE 9-18-96			
				AMBIENT TEMPERATURE 24.18			
TEST POINTS		AS FOUND		FINDING		TOLERANCE	
PSEA mmHg		mmHg		mmHg		mmHg	
0.034 2.017		2.008		98mm		± 0.011	
0.077 3.982		3.963				0.02	
0.116 6.000		5.990				0.031	
0.155 8.006		8.001				0.041	
0.193 9.981		9.958				0.050	
<div style="border: 1px solid black; padding: 10px; margin: 10px auto; width: 80%;"> <p>TABLE A-2</p> <p>MKS 10 TORR TRANSDUCER</p> <p>RGSS Final Calibration</p> </div>							
APPROVED BY D. J. Wilson		CALIBRATED BY [Signature]		Hanford Operations and Engineering Contractor for the United States Department of Energy		Westinghouse Hanford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352	
						PAGE 1 OF 1	

BEST AVAILABLE COPY

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS		STANDARDS CODE NUMBER 559-80-02-013		NEW ☒	REFERENCE NUMBER 384157																												
L6-38				MODIFY ☐	WORK ORDER N2D2B																												
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 100 TORR		SERIAL NUMBER 94195211A	PROPERTY NUMBER N/A	RECALL STATUS 1 ACTIVE 2 NONRECALL 3 SUSPENDED 4 DELETED 5 PM 6 NONDATA MATR	RECALL CYCLE 360																												
ROOM 267		BUILDING 306E	SERVICE DEPARTMENT 5	DATE RECEIVED 940819	TOLERANCE AS RECEIVED ± 1%																												
SENDER JEFF 6-3988		COMMENTS W/REPORT MKS SN# 94202107A		SHIPPING DATE WE	TOLERANCE HISTORY 2 OUT 3 NA 4 FAILED																												
INSTRUMENT SPECIFICATIONS F.24% OF RSG ± 1.0% DT																																	
STANDARD(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS 4:1 RATIO Y ☒ N ☐																																	
EXPIRATION DATE 6-1-31-04-02-4			EXPIRATION DATE 11-5-94																														
REMARKS																																	
PROCEDURE NUMBER WHC-IHP-PRESGA REV.2 Vm S-MKS 622A				TRAINING HOURS																													
				CALIBRATION HOURS 20																													
				REPAIR HOURS																													
				OTHER HOURS																													
				MATERIALS																													
				TOTAL CHARGE = (\$120 x SUM OF HOURS) + MATERIAL DATE CALIBRATED DATE DUE																													
				9-1-94 9-1-95																													
				AMBIENT TEMPERATURE 24.52																													
<table border="1"> <thead> <tr> <th>TEST POINTS</th> <th>PS FOUND</th> <th>FINISH</th> <th>TOLERANCE</th> </tr> <tr> <th>PS</th> <th>mm Hg</th> <th>mm Hg</th> <th>mm Hg</th> </tr> </thead> <tbody> <tr> <td>.4</td> <td>20.19</td> <td>20.64</td> <td>±.05</td> </tr> <tr> <td>.799</td> <td>41.67</td> <td>41.35</td> <td>±.11</td> </tr> <tr> <td>1.098</td> <td>62.05</td> <td>62.10</td> <td>±.16</td> </tr> <tr> <td>1.596</td> <td>82.72</td> <td>82.78</td> <td>±.22</td> </tr> <tr> <td>1.995</td> <td>103.40</td> <td>103.37</td> <td>±.27</td> </tr> </tbody> </table>						TEST POINTS	PS FOUND	FINISH	TOLERANCE	PS	mm Hg	mm Hg	mm Hg	.4	20.19	20.64	±.05	.799	41.67	41.35	±.11	1.098	62.05	62.10	±.16	1.596	82.72	82.78	±.22	1.995	103.40	103.37	±.27
TEST POINTS	PS FOUND	FINISH	TOLERANCE																														
PS	mm Hg	mm Hg	mm Hg																														
.4	20.19	20.64	±.05																														
.799	41.67	41.35	±.11																														
1.098	62.05	62.10	±.16																														
1.596	82.72	82.78	±.22																														
1.995	103.40	103.37	±.27																														
<div style="border: 1px solid black; padding: 10px; width: fit-content; margin: 0 auto;"> <p>TABLE A-3 MKS 100 TORR TRANSDUCER RGSS Initial Calibration</p> </div>																																	
APPROVED BY DL 9-6-94																																	
CALIBRATED BY WMC Sanford Operations and Engineering Contractor for the United States Department of Energy																																	
Westinghouse Sanford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352																																	
PAGE 1 OF 1																																	

BEST AVAILABLE COPY

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS L6-38		STANDARDS CODE NUMBER 559-80-02-013		NEW MODIFY ☒ 394003		REFERENCE NUMBER 394003	
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 100 TORR		SERIAL NUMBER 94195211A	PROPERTY NUMBER N/A	RECALL STATUS 1 ACTIVE 2 NONRECALL 3 SUSPENDED 4 DELETED 5 PM 6 NONDATA MLT	RECALL CYCLE 360	TOLERANCE HISTORY I AS RECEIVED 1 IN 2 OUT 3 NA 4 FAILED	
SENDER JEFF 6-3988		ROOM 267	BUILDING 306E	SERVICE DEPARTMENT 5	DATE RECEIVED 950914	SHIPPING DAY WE	
INSTRUMENT SPECIFICATIONS		W/MKS SN# 94202107A MS+/- .26%RDG+/-1DIG					
STANDARDS(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS		EXPIRATION DATE 6-1-31-04-02-6		EXPIRATION DATE 10-25-95		TRAINING HOURS	
						CALIBRATION HOURS 20	
						REPAIR HOURS	
						OTHER HOURS	
						MATERIALS	
REMARKS U80-RSS						TOTAL CHARGE = (\$120 x SUM OF HOURS) + MATERIAL	
						DATE CALIBRATED 9-15-95	
						DATE DUE 9-15-96	
PROCEDURE NUMBER WHC-5-9-PRESGA-REV-3A		VM-5-MKS-622A				AMBIENT TEMPERATURE 21.119	
TEST POINTS	AS FOUND	FOUND	TOLERANCE				
PSCA mantle	mmHg	mmHg	mmHg				
0.4	20.67	20.87	20.67				
0.8	41.37	41.73	41.48				
1.2	62.06	62.57	62.21				
1.6	82.11	82.37	82.46				
2.0	103.13	103.59	103.37				
<div style="border: 1px solid black; padding: 10px; text-align: center;"> <p>TABLE A-4</p> <p>MKS 100 TORR TRANSDUCER</p> <p>RGSS Final Calibration</p> </div>							
APPROVED BY 9-15-95 W. Nelson		CALIBRATED BY W. Nelson		Westinghouse Hanford Operations and Engineering Contractor for the United States Department of Energy		Westinghouse Hanford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352	
						PAGE -L OF 1-	

BEST AVAILABLE COPY

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS L6-38		STANDARDS CODE NUMBER 559-80-02-011		NEW MODIFY ☒	REFERENCE NUMBER 384155
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 1000 TORR		SERIAL NUMBER 94181110A	PROPERTY NUMBER N/A	RECALL STATUS 1 ACTIVE 2 NONRECALL 3 SUSPENDED 4 DELETED 5 PH 6 NONDATA MATE	ORGANIZATION CODE WSD620 WORK ORDER N2D2B
SENDER JEFF 6-3988		ROOM 267	BUILDING 306E	SERVICE DEPARTMENT 5	DATE RECEIVED 360 940819
INSTRUMENT SPECIFICATIONS #26% OF R06 ± 1 DIGIT		COMMENTS W/RODOUT MKS SN# 94202107A MS ± 26% R06 ± 1 DIG WE		TOLERANCE HISTORY 1. OUT 2. OK 3. NA 4. FAILED	
STANDARD(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS 4:1 RATIO Y ☒ N ☐				CALIBRATION HOURS 2.0	
EXPIRATION DATE 641-31-04-02-6 11-5-94				REPAIR HOURS	
OTHER HOURS				MATERIALS	
TOTAL CHARGE - (\$120 x SUM OF HOURS) + MATERIAL				DATE CALIBRATED 9-1-94	
DATE CALIBRATED 9-1-94				DATE DUE 9-1-95	
PROCEDURE NUMBER 08-MHC-IHF-PRESGA-REV-2-VM-5-MKS 622 A				AMBIENT TEMPERATURE - 24.54	
TEST POINTS	AS FOUND	FINISH	TOLERANCE		
PSE mm Hg	mm Hg	mm Hg	mm Hg		
4 206.8	206.9	5mm	± 5		
8 413.7	414.4		± 11		
12 620.5	621.6		± 1.6		
16 827.4	828.4		± 22		
19 982.5	983.2		± 2.6		

TABLE A-5
MKS 1000 TORR TRANSDUCER
RGSS Initial Calibration

APPROVED BY JLB 109-6-94	CALIBRATED BY 48	Sanford Operations and Engineering Contractor for the United States Department of Energy	Westinghouse Sanford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352	PAGE 1 OF 1
-----------------------------	---------------------	--	---	----------------

BEST AVAILABLE COPY

WESTINGHOUSE STANDARDS LABORATORY PHYSICAL AND ELECTRICAL REPORT

CUSTODIAN/ADDRESS CANNON NS		STANDARDS CODE NUMBER 559-80-02-011		NEW ☒	REFERENCE NUMBER 394001
L6-38				MODIFY ☒	WORK ORDER N2D2A
INSTRUMENT PRESSURE TRANSDUCER MKS BARATRON 622 1000 TORR		SERIAL NUMBER 94181110A	PROPERTY NUMBER N/A	ORGANIZATION CODE WOM530	TOLERANCE HISTORY AS I RECEIVED 2
SENDER JEFF 6-3988		ROOM 267	BUILDING 306E	DATE RECEIVED 9-15-95	TOLERANCE 2
		SERVICE DEPARTMENT 5	RECALL STATUS 1 ACTIVE 2 NONRECALL 3 SUSPENDED 4 DELETED 5 PW 6 NONDATA MITE	RECALL CYCLE 360	SHIPPING DATE 9-14-95
INSTRUMENT SPECIFICATIONS		W/MKS SN# 94202107A MS+/- .26*RDG+/-1DIG		WE	3 NA 4 FAILED

STANDARD(S) USED IN CALIBRATION TRACEABLE TO NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY OR NATIONALLY RECOGNIZED STANDARDS		EXPIRATION DATE 6-1-31-04-02-6		EXPIRATION DATE 11-25-95	
REMARKS		TRAINING HOURS		CALIBRATION HOURS 20	
		REPAIR HOURS		OTHER HOURS	
		MATERIALS		TOTAL CHARGE = (\$120 x SUM OF HOURS) + MATERIAL	
		DATE CALIBRATED 9-14-95		DATE DUE 9-11-96	
		AMBIENT TEMPERATURE = 21.12			

PROCEDURE NUMBER WMC-5-9-PRESGA-REV 3# VM-5-MKS 622A	
--	--

TEST POINTS	AS FOUND	EXACT	TOLERANCE
PSA ^{820.5} 1000 mmHg	mmHg	mmHg	mmHg
1 206.8	± 208.5	206.8	± .5
8 413.7	± 415.8	414.1	1.1
12 620.5	± 623.1	621.3	1.6
16 827.4	± 829.9	828.0	2.2
19 982.5	984.7	982.5	2.6

TABLE A-6
MKS 1000 TORR TRANSDUCER
RGSS Final Calibration

APPROVED BY D. J. Nelson	DATE 9-15-95	CALIBRATED BY WMS	DATE 9/15	Hanford Operations and Engineering Contractor for the United States Department of Energy	Westinghouse Hanford Company Subsidiary of Westinghouse Electric Corporation Box 1970, Richland, WA 99352	PAGE 1 OF 1
------------------------------------	------------------------	-----------------------------	---------------------	--	---	------------------------------

BEST AVAILABLE COPY

[illegible]

BEST AVAILABLE COPY

米

TABLE A-8

RGSS Thermocouples

All thermocouples (except Channel 10) were from PO WFJ-XVV-257890 (Vendor:ARI)

TC Assy. No.	Probe SN	Description	RT Loop Res. (ohms)	RT Ins Res.* (ohms)
TC Channel 1	24961	Ext. Bottom	15.5	4×10^{10}
TC Channel 2	24967	Ext. Bottom	15.3	3×10^9
TC Channel 3	24950	Ext. Side	15.4	7×10^{11}
TC Channel 4	24962	Ext. Side	15.5	3×10^{10}
TC Channel 5	24969	Ext. Top	15.7	6×10^9
TC Channel 6	24977	Ext. Top	15.3	1×10^{11}
TC Channel 7	24940	2nd Vol.	15.5	6×10^{11}
TC Channel 8	24959	2nd Vol.	15.5	4×10^9
TC Channel 9	24960	Cross Line	15.3	9×10^{10}
TC Channel 10	Stores Standard Type K	Cabinet	NA	NA

*At 500 volts.

TABLE A-9

Out-of-Specification Pressure Transducer Calibration Results

MKS Recalibration on 9-15-95							
MKS 100 Torr Transducer				MKS 1000 Torr Transducer			
Test Value (Torr)	Trans. Result (Torr)	Error (Torr)	Error (%)	Test Value (Torr)	Trans. Result (Torr)	Error (Torr)	Error (%)
20.69	20.87	0.18	0.87	206.8	208.5	1.7	0.82
41.37	41.73	0.36	0.87	413.7	415.8	2.1	0.51
62.06	62.57	0.51	0.82	620.5	623.1	2.6	0.42
82.74	83.27	0.53	0.64	827.4	829.9	2.5	0.3
103.43	103.99	0.56	0.54	982.5	984.7	2.2	0.22

TABLE A-10

Potential Pressure Error Effect on Hg Pump Measured Gas Quantity

This Table is only for RGSS tests where P_v was measured using the 100 Torr Transducer. It is assumed the maximum error of +2.6 Torr (0.35 KPa) occurred in the calculation of the pressure difference $P_f - P_v$ (see Equation 4). The effect of this error on the measured gas quantity is evaluated in this Table.

Test ID	$P_f - P_v$ (KPa)	Error $0.35/(P_f - P_v)$ (%)	Starting Inj. Gas @ STP (mL)	Initial Hg Pump Measured Gas @STP (mL)	Corrected Hg Pump Measured Gas @STP (mL)	Corrected Error (mL)
RGS-001	63.61	0.55	37.80	40.76	40.54	2.74
RGS-003	59.09	0.59	38.69	37.91	37.69	-1.00
RGS-005	59.57	0.59	36.99	38.17	37.95	0.96
RGS-006	56.43	0.62	37.46	36.49	36.26	-1.20
RGS-025	62.21	0.56	37.62	33.67	33.48	-4.14
RGS-026	49.49	0.71	37.70	33.80	33.56	-4.14
RGS-027	27.61	1.27	20.12	18.85	18.61	-1.51
RGS-028	11.68	3.00	10.02	7.85	7.61	-2.41
RGS-029	1.0	35.00	2.52	0.68	0.44	-2.08
RGS-033	51.04	0.69	37.11	34.79	34.55	-2.56
RGS-035	22.05	1.59	20.11	16.70	16.43	-3.68
RGS-036	21.35	1.64	20.09	14.88	14.64	-5.45
RGS-037	45.16	0.78	35.36	30.81	30.57	-4.79
RGS-038	2.22	15.77	2.55	1.51	1.27	-1.28
RGS-039	3.44	10.17	5.11	2.73	2.45	-2.66
RGS-043	2.03	17.24	5.04	2.43	2.01	-3.03
RGS-045	1.53	22.88	5.04	2.05	1.58	-3.46
RGS-046	1.27	27.56	3.77	1.85	1.34	-2.43
RGS-047	11.17	3.13	20.14	7.46	7.23	-12.91
RGS-048	9.49	3.69	10.06	6.50	6.26	-3.80
RGS-049	8.91	3.93	10.14	6.10	5.86	-4.28
RGS-050	3.32	10.54	5.04	2.27	2.03	-3.01
RGS-051	30.58	1.14	25.7	21.0	20.76	-4.94
RGS-052	37.53	0.93	28.1	25.5	25.26	-2.84
RGS-053	30.19	1.16	25.3	20.8	20.56	-4.74

APPENDIX B

Ammonia Solution Analysis (ASA)

Analysis of the sample gas for ammonia was performed using a technique described in detail in the RGSS proof test report WHC-SD-WM-TRP-232 (TRP). The actual technique used during acceptance testing was modified to match the new RGSS hardware.

Briefly, after sample gas has been dumped into the evacuated 2nd Volume (2ndV) of the extractor as part of gas quantification testing, the 2ndV is isolated, and 30 to 50 mL of a slightly acidic solution is injected into it. This solution absorbs the ammonia and retains it (even when exposed to air) while a transfer of the solution is made to the ion probe (Orion 9512BN Ammonia Gas Sensing Electrode) testing container. From the solution's measured ammonia concentration, solution volume, gas volume, gas temperature and pressure, the original percentage of ammonia in the sample gas can be calculated.

By way of example, the ammonia test for RGS-008 will be described step by step. This test was performed 7-17-95, and at 4:07 PM the final pressure in the 2nd Volume (320.64 mL) was measured at 2.16 KPa with its temperature at 25.7 °C. Then the 2ndV was isolated by closing valves R and S (see Figure 2); valve Q was opened allowing 40.3 mL of 0.02 M H₂SO₄ to be drawn into the 2ndV. After about a half hour, this solution was drawn out through valve U and placed in a glass container for subsequent analysis.

The ammonia analysis was performed on 7-19-95 starting at 8:30 AM. First the ion probe was calibrated using two NH₄Cl solutions of concentrations 10⁻⁸ moles/mL and 10⁻⁵ moles/mL as standards. With the ion probe immersed in a standard, a pH adjusting chemical was added to drive the solution basic (pH between 12 and 14), releasing the ammonia to the probe. After the calibration of the probe was set with the two standards, a third calibration solution of 10⁻⁶ moles/mL was tested which gave a result of 1.02 x 10⁻⁶ moles/mL, verifying the ion probe calibration. The RGS-008 sample solution was then pH adjusted with the ion probe in place, giving a result of 2.35 x 10⁻⁷ moles/mL.

The total number of ammonia moles in the sample solution would then be the solution volume multiplied by the concentration; i.e. (40.3 mL x 2.35 x 10⁻⁷ moles/mL), or 9.47 x 10⁻⁶ moles of ammonia. The original number of moles of gas in the 2nd Volume before ammonia absorption can be calculated from the pressure and temperature using the perfect gas law:

$$n = [(2.16 \text{ KPa}) * (320.64 \text{ mL})] / [(8.31441 \text{ J}/\{\text{mol} * \text{K}\}) * (273 + 25.4) \text{ K}]$$
$$= 2.7915 \times 10^{-4} \text{ moles}$$

Thus, the ammonia percentage (NH3%) of the original gas is

$$\text{NH}_3\% = 9.47 \times 10^{-6} / 2.7915 \times 10^{-4} \times 100\% = 3.39 \text{ percent}$$

In the RGSS proof test report (WHC-SD-WM-TRP-232), ammonia proof testing had produced excellent results. The average of 17 tests of the same gas (5.14% ammonia) was 5.07 percent ammonia, with a standard deviation of 0.3 percent. For RGSS acceptance testing, a new gas supply (vendor specified at 5.02 percent ammonia) was procured, and another series of tests were performed using the same methods as were done for the TRP. The results from these tests are given here in Table B-1. For each of seven tests, the ammoniated test solution was divided into two samples, A and B.

The testing order was reversed for the B samples, helping to reduce a possible testing order bias. However, it can be noted that slightly higher results were obtained as testing progressed. There was a reassuring correlation between the A and B series tests; for example, D-1 produced the highest results in both test series, and D-5 the lowest.

The combined average result of 5.05 percent ammonia for both test series compares favorably with the expected value of 5.02 percent. The combined average standard deviation of 1.15 percent is much higher than that experienced in the proof testing, but well within the requirements of the FRD. Only the individual test D-1B exceeded the FRD requirement of ± 2 percent, absolute; this test was for the lowest quantity of gas tested (0.6 mL).

However, as previously discussed in Ammonia Solution Analysis Section (7.2.2.1), the RGSS acceptance test results for ammonia averaged 3.17 percent (see Table 16) where 5.02 percent was expected. A number of possible reasons are discussed there for this apparent degradation in accuracy when moving from proof testing to the RGSS acceptance testing.

TABLE B-1
Preliminary MIX#1 Ammonia D-Series Test Results

Test ID	Test Order	Gas Start Pressure (Torr)	STP Gas Volume (mL)	Measured NH ₃ Molar Percentage	Expected NH ₃ Molar Percentage
D-1A	1	12.0	0.61	6.72	5.02
D-2A	2	48.3	2.41	5.75	5.02
D-3A	3	99.4	5.08	4.08	5.02
D-4A	4	188.2	9.41	5.94	5.02
D-5A	5	250.2	12.84	3.30	5.02
D-6A	6	399.7	19.74	4.65	5.02
D-7A	7	750.1	38.36	4.24	5.02
Average =				4.95	
Standard Deviation =				1.12	
D-1B	14	12.0	0.61	7.27	5.02
D-2B	13	48.3	2.41	6.13	5.02
D-3B	12	99.4	5.08	4.34	5.02
D-4B	11	188.2	9.41	5.42	5.02
D-5B	10	250.2	12.84	3.47	5.02
D-6B	9	399.7	19.74	4.94	5.02
D-7B	8	750.1	38.36	4.40	5.02
Average =				5.14	
Standard Deviation =				1.17	

APPENDIX C

RGSS Raw Data Sheets

Included in this appendix are the raw data sheets for all RGSS acceptance tests that were successfully performed, and which provided data used in this report. These tests are summarized by test type in the following table

Hardware	Dry Injection	Wet Injection	Full Simulation	Sampler Aging
Extractor Volume 6-27-95	RGS-001	RGS-025	RGS-051	RGS-055
	RGS-003	RGS-026	RGS-052	RGS-056
Extractor-Sampler Intergration Test 7-14-95	RGS-005	RGS-027	RGS-053	RGS-057
	RGS-006	RGS-028		RGS-058
	A-1	RGS-029		
	A-2	RGS-033		
	RGS-008	RGS-034		
	RGS-009	RGS-035		
	RGS-020	RGS-036		
	RGS-022	RGS-037		
		RGS-038		
		RGS-039		
		RGS-043		
		RGS-045		
		RGS-046		
		RGS-047		
		RGS-048		
		RGS-049		
		RGS-050		

EXTRACTOR VOLUME MEASUREMENT TABLE

- P1 is starting pressure in lines; P2 is pressure 2nd Volume + lines; P3 is pressure in Extractor + lines + 2nd Volume.

Test Performer N.S. Gannon

Measured 2nd Volume = 297.0 ml

TEST #	P1 Press. (Torr)	Temp. (°C) P1	P2 Press. (Torr)	Temp. (°C) P2	P3 Press. (Torr)	Temp. (°C) P3	Line Volume (cc)	Extractor Volume (cc)
1	721.4	20.9	244.2	20.9	84.1	21.0	220.4	1350.0
2	706.0	21.2	289.3	21.2	82.4	21.3	220.4	1351.5
3	754.4	21.8	307.8	21.8	87.8	21.8	220.5	1354.4
4	749.4	22.5	346.0	22.5	85.5	22.6	220.8	1350.6
5	726.0	22.0	294.6	23.1	80.0	24.1	221.3	1348.4
6	704.4	23.2	295.9	23.2	94.8	23.2	221.0	1346.7
7	730.6	23.3	288.6	23.3	85.5	23.3	221.2	1348.8
8	723.2	23.4	293.4	23.4	84.5	23.4	221.0	1350.2
9	736.4	23.5	300.8	23.5	86.1	23.5	221.0	1349.0
10	748.8	23.5	305.8	23.5	87.6	23.5	220.9	1347.3

AVERAGE EXT. VOLUME (mL) 1349.7 STANDARD DEV. (mL) 2.1

Test Witnesses: M. Scott Cannon 6-27-95
Cognizant Engineer Date

B. J. Heng 06-28-95
Quality Assurance Date

* Calculated

EXTRACTOR/SAMPLER INTEGRATION TEST

- Using air as working medium, fill sampler to known pressure and evacuate the Extractor/2nd Volume. Perform GPT testing to verify proper functioning of the GSE, and that volumes have been correctly measured. Allow 10 minutes for "settling" after each valve opening before taking pressure measurement. Temperature at pressure measurement time should be recorded.

P1 is starting pressure in 2nd valve, P2 is resulting pressure in Extractor.

TEST DATE: 7-14-95 TEST DESCRIPTION: Infection Test

Test Performer C. Knut

SAMPLER ID. # <u>1</u>			SAMPLER VOLUME <u>314.63</u> (mL)			
SAMPLER PRESSURE <u>754.6</u> (Torr)			TEMPERATURE <u>21-25.1</u> (°C)			
(WORKING GAS - AMBIENT AIR)						
TEST TYPE	TEST STEP	TIME (min)	Temp. (°C)	Sensor 1 (100%) (Torr)	Sensor 2 (100%) (Torr)	Sensor 3 (10%) (Torr)
SPT	Zero	9:34	25.1	.05	.006	.006
	Leak Test Start	9:35	25.1	.05	.006	.006
	Leak Test End	9:42	25.1	.06	.017	.017
	Extractor Start	9:48	25.1	20.37	—	—
	Extractor End	2:56	25.1	30.38	—	—
	2nd Vol. Start	2:57	25.1	16.95	16.94	—
	2nd Vol. End	3:07	25.1	16.91	16.95	—

AVERAGE EXT. VOLUME (mL) 1570.6 2ND VOLUME (mL) 320.0

Test Witnesses: M. J. Connor 7-14-95
Cognizant Engineer Date
B. J. King 07-14-95
Quality Assurance Date

DEL FBI R63-007. INC.

BEST AVAILABLE COPY

RGS-001

TABLE 3
INJECTION TEST DATA SHEETTEST DATE 6-29-95 Test Performer N.S. Cannon

GAS MIX TO <u>MIX #2 (N₂O₂)</u>		CANISTER TEMP. <u>29 °C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>102.6 kPa (760.2 mmHg)</u>				
TEST TYPE	TEST STEP	TIME (PM)	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
SPT	Zero	4:29	26.7	0.04	0.013	0.0120
	Leak Test Start	4:29	26.7	0.04	0.013	0.0120
	Leak Test End	4:35	26.7	0.06	0.028	0.0279
	Extractor Start	4:38:20	26.7	2.64	2.666	OFF S.
	Extractor End	4:43	26.7	2.64	2.667	OFF S.
	2nd Vol. Start	4:43:30	26.7	2.21	2.239	OFF S.
	2nd Vol. End	4:52	26.6	2.22	2.244	OFF S.

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V. TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% H ₂
NA							

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	H ₂ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	0	0	30.8	30.0	39.2	0	0
CONTROL	*	*	30.4	30.3	39.2	*	*
TEST	*	*	29.0	32.7	37.1	0.1	Q+1.0%

RGS-002
RGS-001

Test Witnesses: M. Scott Cannon 6-30-95
 Logizant Engineer Date
7-12-95
 Quality Assurance Date

* Less than 0.1%

RGS-003

TABLE 3
INJECTION TEST DATA SHEETTEST DATE 6-30-95 Test Performer N.S. Cannon

GAS MIX TO <u>MIX #2</u>		CANISTER TEMP. <u>26 °C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>103.97 kPa (770.8 mmHg)</u>				
TEST TYPE	TEST STEP	TIME AM	TEMP. (°C) Tce 2	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
SPT	Zero	8:10	52.7	0.05	0.002	0.002
	Leak Test Start	10:14	52.7	0.05	0.003	0.003
	Leak Test End	10:22	52.7	0.05	0.009	0.009
	Extractor Start	10:23	52.7	2.76	2.777	OFF S.
	Extractor End	10:34	52.7	2.77	2.782	OFF S.
	2nd Vol. Start	10:36:15	52.7	2.33	2.340	OFF S.
	2nd Vol. End	10:45	52.6	2.34	2.347	OFF S.

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V. TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% H ₂
NA							

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	H ₂ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	0	0	30.9	30.0	39.2	0	0
CONTROL	*	*	30.4	30.3	39.2	*	*
TEST	*	*	30.2	30.7	38.7	*	O ₂ +0.3%

RGS-002
RGS-003

Test Witnesses: M. Scott Cannon 6-30-95
 Logizant Engineer Date
7-12-95
 Quality Assurance Date

* Less than 0.1%

BEST AVAILABLE COPY

TABLE 3
INJECTION TEST DATA SHEET

TEST DATE 7-11-95 Test Performer N.S. Cannon

GAS MIX ID <u>MIX #1 (NH₃)</u>		CANISTER TEMP. <u>28 °C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>10007 KPa (780.9 Torr)</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	2:50 PM	28.0	0.05	0.04	0.0138
	Leak Test Start	2:55	28.0	0.05	0.014	0.0138
	Leak Test End	3:05	28.0	0.09	0.067	0.0662
	Extractor Start	3:07.30	29.1	2.62	2.637	OFF S
	Extractor End	3:18	28.0	2.59	2.621	OFF S
	2nd Vol. Start	3:19	28.0	2.21	2.137	OFF S
	2nd Vol. End	3:29	28.0	2.24	2.267	OFF S

R65-005

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V Temp. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	52.3	3:31	28.0	2.267	7-11-95	LOT 1027	2.1 %

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

* Adjusted for leak rate.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	NH ₃ (g)	H ₂ (g)	H ₂ O (g)	N ₂ (g)	He (g)	Ar (g)	OTHER (g)
VENDOR	5.02	30.3	0	39.8	0	24.9	0
CONTROL	2.9	27.2	0.0	41.7	0	25.9	
TEST	1.4	28.2	0.0	43.9	0	26.6	

R65-004

R65-005

Test Witness: N.S. Cannon
Logistic Engineer

Quality Assurance

7-17-95
Date

7-17-95
Date

* Computer time - about 19 minutes fast.

Data file R65-005. INT

TABLE 3
INJECTION TEST DATA SHEET

TEST DATE 7-12-95 Test Performer N.S. Cannon

GAS MIX ID <u>MIX #1 (NH₃)</u>		CANISTER TEMP. <u>25 °C (p. 112741) - R65-006</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>785.5 Torr</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
SPT	Zero	1:11 PM	52.4	0.03	.004	.0043
	Leak Test Start	1:12	52.4	0.03	.004	.0043
	Leak Test End	1:22	52.4	0.04	.019	0.0177
	Extractor Start	1:23	52.4	2.23	2.22	2.221
	Extractor End	1:24	52.4	2.71	2.743	OFF S
	2nd Vol. Start	1:25	52.4	2.27	2.301	OFF S
	2nd Vol. End	1:46	52.4	2.27	2.310	OFF S

R65-006

TEST TYPE	Solution Volume (mL)	Mix Time	2nd V Temp. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	51.9	1:47	51.1	2.31	7-12-95	LOT 1027	2.7 %

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

* Adjusted for leak rate.

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	NH ₃ (g)	H ₂ (g)	H ₂ O (g)	N ₂ (g)	He (g)	Ar (g)	OTHER (g)
VENDOR	5.02	30.3	0	39.8	0	24.9	0
CONTROL	2.9	27.2	0.0	41.7	0	25.9	
TEST	1.4	28.2	0.0	43.9	0	26.6	

R65-004

R65-006

Test Witness: N.S. Cannon
Logistic Engineer

Quality Assurance

7-17-95
Date

7-17-95
Date

Data file R65-006. INT

BEST AVAILABLE COPY

TEST AVAILABLE COPY

$\times$ $\mu\text{g}/\text{mL}$ $(\frac{100}{100})$ $\mu\text{g}/\text{mL}$

Test Name	Date	Time	Standard (Fresh)	Standard E Value	Standard Real Val	Sample E Value	Sample Concentration
A-2	7-14-95	11:34	100	111	100	30.0	2.39 $\times 10^{-2}$
							3.4 $\times 10^{-2}$

$\% \text{NH}_3 = 4.21\%$

Test Name	Date	Time	Standard (Fresh)	Standard E Value	Standard Real Val	Sample E Value	Sample Concentration
A-1	7-14-95	10:56	100	100	100	32.9	2.52 $\times 10^{-2}$
							3.25 $\times 10^{-2}$

$\% \text{NH}_3 = 4.22\%$

AMMONIA WET CHEMISTRY TEST

validity = -6.9 $\mu\text{g}/\text{mL}$

7-17-95

These results are acceptable and within specification. No persulfate was observed during the ammonia analysis.

147

Two "informal" short injection tests were performed at ambient and ~50°C after the cleaning (on 7-14-95). Informal means no QA oversight and short means no Hg pumping or MS analysis. The results are given on the next page:

Cleaning of the second working cell 13K using an overnight solution of the second volume in 0.02M H_2SO_4 . A complete rinsing and dry out was performed the morning of the test prior to the injection test.

TABLE 3
INJECTION TEST DATA SHEET

TEST DATE 7-17-95 Test Performer N.S. Cannon

GAS MIX ID <u>MIX # 1</u>			CANISTER TEMP. <u>24°C</u>				
CANISTER VOL. (mL) <u>41.3</u>			CANISTER PRESS. <u>753.1 Torr</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)	
SPT	Zero	3:35	25.4	0.06	0.010	0.010	
	Leak Test Start	3:35	25.4	0.06	0.01	0.01	
	Leak Test End	3:45	25.4	0.07	0.027	0.028	
	Extractor Start	3:46	25.4	2.59	2.60	OFF SCALE	
	Extractor End	3:56	25.4	2.57	2.58	OFF SCALE	
	2nd Vol. Start	3:57	25.4	2.16	2.16	OFF SCALE	
	2nd Vol. End	4:07	25.4	2.16	2.16	OFF SCALE	
TEST TYPE	Solution Volume (mL)	Mix Time	2nd V. TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	40.3 g	2:50	25.4	2.16	7-19-95	2.85e ⁻⁷	3.48

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS. Data file = R65-008

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	H ₂ O (%)	Ar (%)	He (%)	Other (%)	
VENDOR							
CONTROL							
TEST							

Test Witnesses: N.S. Cannon 7-19-95
 Cognizant Engineer Date
Quality Assurance 7-19-95
 Date

TABLE 3
INJECTION TEST DATA SHEET

TEST DATE 7-18-95 Test Performer N.S. Cannon

GAS MIX ID <u>MIX # 1</u>			CANISTER TEMP. <u>25°C</u>				
CANISTER VOL. (mL) <u>41.3</u>			CANISTER PRESS. <u>753.2 Torr</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)	
SPT	Zero	9:06	52.4	0.06	0.014	0.015	
	Leak Test Start	9:07	52.4	0.06	0.013	0.014	
	Leak Test End	9:17	52.5	0.09	0.044	0.045	
	Extractor Start	9:18	52.5	2.79	2.79	OFF SCALE	
	Extractor End	9:28	52.4	2.77	2.787	OFF SCALE	
	2nd Vol. Start	9:27	52.4	2.35	2.353	OFF SCALE	
	2nd Vol. End	9:39	52.5	2.37	2.368	OFF SCALE	
TEST TYPE	Solution Volume (mL)	Mix Time	2nd V. TEMP. (°C)	2nd VOL. Pressure (KPa)	ANALYSIS DATE	Probe Conc. (m/cc)	% NH ₃
NH ₃	40.4	8:50	52.2	2.368	7-18-95	1.85e-7	2.74

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS. Data file = R65-009

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							
ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	H ₂ O (%)	Ar (%)	He (%)	Other (%)	
VENDOR							
CONTROL							
TEST							

Test Witnesses: N.S. Cannon 7-19-95
 Cognizant Engineer Date
Quality Assurance 7-18-95
 Date

NOT AVAILABLE COPY

SIMULANT EXTRUSION TEST DATA SHEET

TEST ID RG5-002 TEST DATE 8-8-95 Test Performer N.S. Cannon

SIMULANT: UNKNOWN
 GAS MIX #2 EXPECTED VOL (mL) 41.3 SOLID/LIQUID Water (3rd. exp.)
 SAMPLER: ID 2 VOLUME (mL) _____ PRESSURE (KPa) _____ TEMP (°C) 23°C

TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
SPT	Zero	12:19	21.9	0.24	0.014	0.012
	Leak Test Start	12:22	21.9	0.24	0.020	0.017
	Leak Test End	12:27	21.9	0.24	0.021	0.018
	Extractor Start	12:31	21.9	3.74	3.637	OFF S.
	Extractor End					
	2nd Vol. Start					
	2nd Vol. End					

70:15 min
 40-40-40
 23°C
 SPECIAL TEST

TEST TYPE	Solution Volume (mL)	2nd Vol. TEMP. (°C)	2nd VOL. Pressure (KPa)	Time at Mixing
NA				

Hg PUMP EXTRACTOR GASES TO COLLECTION CANISTERS.

Max. PT4 _____ Cab. Temp. _____ Finish Time _____

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)
BT						

Test Witnesses: N.S. Cannon 8-8-95
 Cognizant Engineer Date
NA
 Quality Assurance Date

TABLE 3

INJECTION TEST DATA SHEET

TEST DATE 8-16-95 Test Performer N.S. Cannon

RG5-022-INV
 GAS MIX ID MIX #2 CANISTER TEMP. 22°C
 CANISTER VOL. (mL) 41.3 CANISTER PRESS. 753.2 mm

TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
SPT	Zero	10:18	21.9	0.24	0.003	0.001
	Leak Test Start	10:20	21.9	0.24	0.004	0.001
	Leak Test End	10:30	21.9	0.24	0.004	0.002
	Extractor Start	10:31	21.9	2.55	2.371	OFF S.
	Extractor End	10:34	21.8	2.54	2.372	OFF S.
	2nd Vol. Start	10:35	21.8	2.19	2.007	OFF S.
	2nd Vol. End	10:39	21.8	2.19	2.007	OFF S.

TEST TYPE Solution Volume (mL) Mix Time 2nd V. TEMP. (°C) 2nd VOL. Pressure (KPa) ANALYSIS DATE Probe Conc. (m/cc) % H₂

NA

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS. Extra J1
 7203 cab. temp. PT4 23.60 KPa Cab. Temp. 26.1°C Time 11:13 AM

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL. PRESS. (KPa)	2nd V. TEMP. (°C)	COMB. PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP							

ANALYSIS RESULTS	H ₂ (%)	N ₂ (%)	H ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	30.8	30.0	39.2	—	—
CONTROL	—	—	30.4	30.3	39.2	—	—
TEST	—	—	30.3	30.5	39.1	—	—

Test Witnesses: N.S. Cannon 8-18-95
 Cognizant Engineer Date
NA
 Quality Assurance Date

BEST AVAILABLE COPY

INJECTION/WATER TEST DATA SHEET

TEST DATE 8-17-95 Test Performer N. S. Cannon

GAS MIX ID <u>MIX #2 (N₂O +)</u>		CANISTER TEMP. <u>23°C</u>	
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>750.7 Torr</u>	

TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 [1000 T] (KPa)	Sensor 2 [100 T] (KPa)	Sensor 3 [10 T] (KPa)
	WATER INJECT 250. (cc)	1:22	21.8	26.4	<u>See RGS-024</u>	
	VAC. PUMP START	3:00				
	VAC. PUMP END	3:17	21.6	2.93	2.740	OFFS
	GAS INJECTION	3:20	21.7	6.09	5.939	OFFS
	Leak Test Start	3:24	21.8	6.11	5.959	OFFS
	Leak Test End	3:44	21.7	6.15	6.004	OFFS
	2nd Vol. Start	NA				
	2nd Vol. End	NA				

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 65.52 (KPa) CAB. TEMP(°C) 26.5

FINISH TIME 4:20 PM

RGS-025.INW

RGS-002

ANALYSIS RESULTS	NH ₃ (%)	H ₂ (%)	N ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	30.8	30.0	39.2	—	—
CONTROL	—	—	30.4	30.3	39.2	—	—
TEST	—	—	29.6	31.4	38.3	—	O ₂ = 0.4

Test Witnesses: N. S. Cannon
Cognizant Engineer

8-17-95
Date

NA
Quality Assurance

Date

BEST AVAILABLE COPY

INJECTION/WATER TEST DATA SHEET
TEST DATE 8-19-95 Test Performer A.S. Gansland
TEST NAME RGS-026

GAS MIX TO <u>MIXER</u> (400)		CANISTER TEMP. <u>25 °C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>722.2 Torr</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor-1 (1000 T) (°C)	Sensor-2 (100 T) (°C)	Sensor-3 (10 T) (°C)
	WATER INJECT (vol. 250)	<u>8:40</u>	<u>21.7</u>	<u>2.92</u>	<u>2.748</u>	<u>OFF S</u>
	Leak Test Start	<u>8:50</u>	<u>21.7</u>	<u>2.93</u>	<u>2.752</u>	<u>OFF S</u>
	Leak Test End	<u>8:50</u>	<u>21.7</u>	<u>6.00</u>	<u>5.842</u>	<u>OFF S</u>
	GAS INJECTION	<u>8:53</u>	<u>21.7</u>	<u>6.00</u>	<u>5.763</u>	<u>OFF S</u>
	Leak Test End	<u>9:03</u>	<u>21.8</u>	<u>6.05</u>	<u>5.910</u>	<u>OFF S</u>
	End Vol. Start	<u>9:04</u>	<u>21.8</u>	<u>5.37</u>	<u>5.229</u>	<u>OFF S</u>
	End Vol. End	<u>9:14</u>	<u>21.7</u>	<u>5.34</u>	<u>5.241</u>	<u>OFF S</u>

NO PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINISH TIME 9:49.4A CAN. TEMP(°C) 25.9

ANALYSIS RESULTS	NH ₃ (S)	H ₂ (S)	H ₂ (S)	Ar (S)	OTHER (S)
VENDOR	—	—	30.8	31.2	—
CONTROL	—	—	30.4	31.2	—
TEST	—	—	30.0	31.1	9.0 ± 1

Test Witnesses: M. J. Gansland Captain Engineer 8-24-95
B. J. Gansland Quality Assurance 08-24-95

Q1: 8.4 K-methyl 235 cc of water in bottom

TEST AVAILABLE COPY

INJECTION/WATER TEST DATA SHEET
TEST DATE 8-28-95 Test Performer A.S. Gansland
TEST NAME Leak Pressure Test-1 (RGS-007)

GAS MIX TO <u>MIXER</u>		CANISTER TEMP. <u>22 °C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>400.1 (Torr)</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor-1 (1000 T) (°C)	Sensor-2 (100 T) (°C)	Sensor-3 (10 T) (°C)
	WATER INJECT	<u>8:30</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	Leak Test Start	<u>8:30</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	Leak Test End	<u>8:45</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	GAS INJECTION	<u>8:45</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	Leak Test End	<u>8:54</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	End Vol. Start	<u>8:54</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>
	End Vol. End	<u>9:08</u>	<u>21.8</u>	<u>2.752</u>	<u>2.752</u>	<u>OFF S</u>

NO PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINISH TIME 9:35.0 CAN. TEMP(°C) 26.0

ANALYSIS RESULTS	NH ₃ (S)	H ₂ (S)	H ₂ (S)	Ar (S)	OTHER (S)
VENDOR	—	—	30.0	31.2	—
CONTROL	—	—	30.4	31.2	—
TEST	—	—	30.0	31.3	—

Test Witnesses: R. J. Gansland Captain Engineer 8-28-95
A. S. Gansland Quality Assurance 8-28-95

BEST AVAILABLE COPY

Test Witnesses: AC Singh Cognizant Engineer 08/24/95 Date

ANALYSIS	IN	(%)	N	(%)	HE	AF	OTHER
MISCELLANEOUS	-	-	-	-	-	-	-
TEST	0.1	0.1	15.7	4.3	76.0	89.2	0.1
CONTROL	-	-	0.4	0.3	89.2	-	-
VENDOR	-	-	8.0	3.0	39.2	-	-

HYDRO PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANNISTERS.

FINAL PTA	4.212	(KPA)	CAB. TEMP(°C)	26.6
FINISH TIME	3:32			

TEST	TEST	TIME	TEMP.	Sensor 1	Sensor 2	Sensor 3
TYPE	512P	(°C)	(°C)	1000 (°C)	100 (°C)	100 (°C)
TEST	TEST	TIME	TEMP.	Sensor 1	Sensor 2	Sensor 3
4/13	4/13	4/13	4/13	4/13	4/13	4/13
CMISTEN VOL. (m)	CMISTEN PRESS.	CMISTEN TEMP.	CMISTEN	CMISTEN	CMISTEN	CMISTEN

INJECTION/WATER TEST DATA SHEET

TEST DATE	TEST PERFORMED BY	TEST NAME
8/22/95	MED	P.T.I. Injection Test (R65-02c)

Test Witnesses: W. C. [Signature] Cognizant Engineer 11/2/56
11/2/56 Date

NA Quality Assurance 11/2/56
11/2/56 Date

ANALYSIS	YENDOR	CONTROL	TEST
NH ₄ N (%)	—	—	—
H (%)	2.8	30.4	1.2
H ₂ O (%)	2.0	30.3	1.8
N (%)	2.2	30.2	1.8
AP	—	—	—
OTHER (%)	—	—	—

FINAL PTA 14.715 (KPA) CAB. TEMP (°C) 26.54
FINISH TIME 1.80

[illegible]

INJECTION/WATER TEST DATA SHEET

TEST DATE	8/23/95	TEST PERFORMER	MFD
TEST NAME	Leak Detection Test - 2 (245-023)		

August 24, 1995

Argon test

RG-S-033

File RG-SS236A

Test gas sample 41.3 cc
140.4 torr Ar gas @ 23°C

300 ml water placed in extractor - pumped down to < 3 kPa
three different times.

Test start 0902 Value K ^(closed) off, value T opened

0902 Value K closed, value T opened 2.829 kPa 22.8°C ³⁰⁶
0911 Value K closed, value T opened ~~2.829~~ 2.827 kPa 22.4°C
0915 Gas vessel opened to extractor 5.336 kPa 22.4°C
0920 Value K closed (2nd volume indicated)
0929 Pump cycle start (Value F closed)
1003 Pump cycle end collector pressure 54.3 kPa 26.7°C

Collection sample removed J3 RG-S-033

B. B. B.

August 24, 1995

Argon Test

RG-S-034, RG-S-035

File RG-SS236B

Test gas: 41.3 cc, 400.0 Torr Ar @ 22°C
5 kPa

300 ml H₂O in extractor, pumped down to < 3 kPa 3 different times.

Test Start: 11:45 Value K closed, Value T opened

1146 Value K ^{30 closed} opened, Value T opened 2.804 kPa 22.1°C
1148 gas introduced to extractor 4.076 kPa 22.1°C
1150 2nd Volume closed, J3 closed RG-S-034 captured
Value F closed

Start of pump cycle

End of pump cycle 12:22

1222 Value J3 and H₂ closed collector pressure ^{RG-S-035} 25.24 kPa
extractor pressure 3.234 kPa
extractor temp. 22.1°C
collector temp. 26.1°C

NOT AVAILABLE COPY

B. B. B.

August 24, 1995

Argon Test RGS-036

File: RG-SS236C

Test gas: 41.3 cc 400.9 Torr Ar @ 23°C

300 ml H₂O in Extractor, pumped down to ^{3rd} 3.6 kPa 3 different times

Tests Start: 0:00 Valve K closed, Valve T opened

0:00 Valve K closed, Valve T opened 2.794 kPa 22.0°C

0:02 Gas injected into extractor 4.091 kPa 22.0°C

0:04 Valve R closed, Valve F closed

0:05 Pump Cycle start

30:19 Pump Cycle end 25.6 kPa 26.3°C

→ RGS-036 acquired

BETTER

INJECTION/WATER TEST DATA SHEET

TEST DATE: 8-28-95 Test Performer: M.S. Gannon

TEST NAME: RGS-037-JNW

GAS MIX ID: <u>MIX #1 (H₂O)</u>		CANISTER TEMP.: <u>22</u>	
CANISTER VOL. (mL): <u>41.3</u>		CANISTER PRESS.: <u>240.1</u>	

TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (kPa)	Sensor 2 (100 T) (kPa)	Sensor 3 (10 T) (kPa)
WATER INJECT	Injection (cc)	—	—	—	—	—
	Leak Test Start	12:15	21.8	2.44	2.768	OFF S.
	Leak Test End	12:28	21.7	2.47	2.779	OFF S.
	GAS INJECTION	12:28 PM	21.7	2.46	2.803	OFF S.
	Leak Test Start	12:30	21.7	2.49	2.801	OFF S.
	Leak Test End	12:43	21.3	2.53	2.841	OFF S.
	2nd Test Start	12:44	20.2.9	2.52	2.844	OFF S.
	2nd Test End	12:53	21.4	2.53	2.875	OFF S.

by PUMP EXTRACTABLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL P14: 48.87 (kPa) CAN. TEMP(°C): 26.7

FINISH TIME: 1:23 PM

ANALYSIS RESULTS	mg (S)	% (S)	H ₂ O (S)	N ₂ (S)	He (S)	Ar (S)	OTHER (S)
SPURDOR	5.02	30.3	—	29.8	—	24.9	—
CONTROL	2.9	29.2	—	41.7	—	25.9	—
TEST	—	29.6	—	43.6	—	26.4	—

*Exposed: 31.9 — 41.9 — 26.3 —

Test Witnessed: M.S. Gannon 8-28-95
BQU 10-1-95
 QUALITY ASSURANCE

BEST AVAILABLE COPY

* With H₂O reacted out by under absorption

INJECTION/WATER TEST DATA SHEET

TEST DATE 8-28-95 Test Performer N.S. Cannon
 TEST NAME RCS-032, INW

GAS MIX ID <u>ARGON</u>		CANISTER TEMP. <u>22</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>10.7</u>				
TEST TYPE	TEST STEP	TIME (PM)	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	WATER INJECT Previous (cc)	—	—	—	—	—
	Leak Test START	2:37	21.9	2.99	2.900	OFF S.
	Leak Test END	2:47	21.9	3.00	2.815	OFF S.
	GAS INJECTION	2:48	21.9	3.16	2.970	OFF S.
	Leak Test Start	—	—	—	—	—
	Leak Test End	2:52	22.0	3.18	2.997	OFF S.
	2nd Vol. Start	2:53	22.0	3.12	2.933	OFF S.
	2nd Vol. End	2:58	22.0	3.14	2.958	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 5.149 (KPa) CAB. TEMP(°C) 26.8
 FINISH TIME 3:11 PM

RCS-032

ANALYSIS RESULTS	H ₂ (%)	H ₂ (%)	H ₂ O (%)	H ₂ (%)	H ₂ (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—	—
CONTROL	—	—	—	—	—	99.95	—
TEST	0.1	0.2	0.7	13.5	—	82.8	0.4 ± 2.7

Test Witnesses: N.S. Cannon 8-28-95
 Cognizant Engineer Date
A.S. Hays 08/29/95
 Quality Assurance Date

INJECTION/WATER TEST DATA SHEET

TEST DATE 8-28-95 Test Performer N.S. Cannon
 TEST NAME RCS-039, INW

GAS MIX ID <u>ARGON</u>		CANISTER TEMP. <u>23°C</u>				
CANISTER VOL. (mL) <u>41.3</u>		CANISTER PRESS. <u>102.0 Tm</u>				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	WATER INJECT Previous (cc)	—	—	—	—	—
	Leak Test START	9:38	20.4	2.71	2.632	OFF S.
	Leak Test END	9:45	20.4	2.72	2.542	OFF S.
	GAS INJECTION	9:47	20.4	3.10	2.937	OFF S.
	Leak Test Start	9:50	20.4	3.12	2.953	OFF S.
	Leak Test End	10:00	20.4	3.16	2.991	OFF S.
	2nd Vol. Start	10:01	20.4	3.05	2.876	OFF S.
	2nd Vol. End	10:11	20.5	3.07	2.895	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 6.706 (KPa) CAB. TEMP(°C) 24.8
 FINISH TIME 10:21 AM

RCS-032

ANALYSIS RESULTS	H ₂ (%)	H ₂ (%)	H ₂ O (%)	H ₂ (%)	H ₂ (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—	—
CONTROL	—	—	—	—	—	97.95	—
TEST	—	—	0.1	2.71	—	96.32	0.4 ± 0.8

Test Witnesses: N.S. Cannon 8-28-95
 Cognizant Engineer Date
B.O. Hays 08-28-95
 Quality Assurance Date

BEST AVAILABLE COPY

August 29, 1995

Argon Test RGS-043A, RGS-043B

File: RGS-043.1.W

Test gas 41.3 cc Ar, 100.1 Torr @ 22°C

300 ml H₂O in the extractor - pumped down once for 2 minutes to < 3 kPa

Test Start 0826

0826 Valve K closed 2.464 kPa 20.1°C

0827 Gas injected into extractor 2.478 kPa

0836 Valve J1 closed, RGS-043A captured 2.910 kPa 20.1°C

0837 Valves T & S opened to 2nd volume 2

0848 Pump Cycle Start - 2.217 kPa 20°C

Ops - Valve B closed - probably nothing in J1 - 50
J3 opened 8:57 closed 9:00 2.784 kPa, 24.5°C
collected (43C)

901 Pump resume - Valve F reclosed
Pump cycle stopped after 5 strokes 4.7 kPa 24.5°C
J2 - collected - pumped gas (43B)

Betty

Aug. 29, 1995

Argon Test RGS-045

Test gas 41.3 cc Ar, 100.0 @ 21°C pumped down three

times 300 ml H₂O in extractor - pumped down to VP 3 times

Test 11:12

11:12 Valve K, A, T opened 34 7.433 kPa 19.9°C

11:15 Gas injected into extractor 2.740 kPa 19.9°C

11:18 Valve R closed, Valve F closed
J1 closed, H1 closed - canister isolated RGS-045A

11:20 Pump Cycle Started
11:40 J2 closed, H2 closed canister isolated -

BEST AVAILABLE COPY

Ops - forgot to data log.
no file for this one.

RGS-045B

collector 4.306 kPa 25°C
extractor 2.776 kPa 20.0°C

Betty

Aug. 29, 1995

Argon Test RG-5-D46

File: RGSS241A.H1EY

Test gas: 41.3 cc Ar, 75.0 Torr @ 22°C
300 ml H₂O in extractor pumped down to VP three times

Test Start 12:30

12:30 Valve K closed, Valve T opened 2.447 kPa 19.96°C
12:35 Gas injected into extractor 2.115 kPa 19.96°C
12:38 Valve R closed, Valve F closed

12:19 Valve K closed, Valve T opened 2.488 kPa 20.1°C
12:24 Gas injected into extractor 2.716 kPa 20.1°C
12:29 Valve R closed, Valve F closed
Ji closed, H₂ closed RG-5-D46A acquired

12:30 Pump Cycle Start
12:48 Pump Cycle End RG-5-D46B acquired 4.0 kPa 25.5°C
2.754 kPa 20.22°C

B. E. Hay

INJECTION/ANALYSIS TEST DATA SHEET

TEST DATE 8-30-95 Test Performer M.S. Cameron
TEST NAME RG-5-D47-HOT

ANALYZED @ 8:00 AM - 8:28 PM						
GAS MIX ID ARGON		CANISTER TEMP. 22 °C				
CANISTER VOL. (ml) 41.3		CANISTER PRESS. 499.6 kPa				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (100 T) (kPa)	Sensor 2 (100 T) (kPa)	Sensor 3 (10 T) (kPa)
WATER INJECT	Argon (cc)					
	Load Test Start	7:58	20.0	2.48	2.49	OFF S.
	Load Test End	8:05	20.2	2.67	2.50	OFF S.
	Gas Injection	8:06	20.2	4.29	4.14	OFF S.
	Hot Gas Load	8:17	20.6	NA	NA	NA
	Load Test Start	11:05	15.6	3.88	3.78	OFF S.
	Load Test End	11:08	15.6	3.86	3.70	OFF S.
	2nd Hot. Start	11:16	15.6	3.46	3.30	OFF S.
	2nd Hot. End	11:26	15.6	3.45	3.29	OFF S.

By PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PTA 19.86 (kPa) CAN. TEMP (°C) 25.5°
FINISH TIME 11:57

ANALYSIS RESULTS	H ₂ (S)	H ₂ (S)	H ₂ (S)	H ₂ (S)	He (S)	Ar (S)	OTHER (S)
VENDOR							
CONTROL						99.0%	
TEST				0.3		99.2%	0.3

RG-5-D47

Test Witnessed: M.S. Cameron
Logistics Engineer
Date 8-30-95
Quality Assurance
Date 8-31-95

BEST AVAILABLE COPY

INJECTION/WATER TEST DATA SHEET

TEST DATE 8-30-95 Test Performer N.S. Cannon
 TEST NAME RCS-048. INW

GAS MIX TO <u>ARGON</u>			CANISTER TEMP. <u>22°C</u>			
CANISTER VOL. (mL) <u>41.3</u>			CANISTER PRESS. <u>200.1</u>			
			Temp			
TEST TYPE	TEST STEP	TIME PM	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	WATER INJECT					
	<u>Pressure</u> (cc)	<u>1</u>				
	Leak Test START	9:17	20.4	2.70	2.525	OFF S.
	Leak Test END	9:27	20.4	2.72	2.543	OFF S.
	GAS INJECTION	9:28	20.4	2.55	3.385	OFF S.
	Leak Test Start	9:31	20.4	9.57	3.404	OFF S.
	Leak Test End	9:41	20.4	3.61	3.444	OFF S.
	2nd Vol. Start	9:42	20.4	3.39	3.224	OFF S.
	2nd Vol. End	9:52	20.4	3.40	3.282	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 12.302 (KPa) CAB. TEMP(°C) 25.1
 FINISH TIME 4:23

ANALYSIS RESULTS	N ₂ (%)	H ₂ (%)	H ₂ O (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—
CONTROL	—	—	—	—	99.95	—
TEST	—	—	—	4.84	1.37	93.7

RCS-032

Test Witnesses: N.S. Cannon
 Cognizant Engineer
R.O. Miller
 Quality Assurance

8-31-95
 Date
05-21-95
 Date

BEST AVAILABLE COPY

INJECTION/WATER(HOT) TEST DATA SHEET

TEST DATE 8-31-95 Test Performer N.S. Cannon
 TEST NAME RCS-049. HOT

GAS MIX ID <u>ARGON</u>			CANISTER TEMP. <u>22 °C</u>			
CANISTER VOL. (mL) <u>41.3</u>			CANISTER PRESS. <u>201.7</u> (PSIA)			
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	WATER INJECT					
	<u>Baseline</u> (cc)					
	Leak Test START	8:19	20.2	2.68	2.505	OFF S.
	Leak Test END	8:29	20.0	2.64	2.472	OFF S.
	GAS INJECTION	8:30	20.0	3.45	3.288	OFF S.
	HOT CYCLE END	10:05	50.1	NA	NA	NA
	Leak Test Start	11:46	19.7	2.49	3.381	OFF S.
	Leak Test End	11:56	19.7	3.51	3.353	OFF S.
	2nd Vol. Start	11:57	19.9	3.32	3.154	OFF S.
	2nd Vol. End	12:07	19.9	3.32	3.161	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 _____ (KPa) CAB. TEMP(°C) _____
 FINISH TIME _____

ANALYSIS RESULTS	N ₂ (%)	H ₂ (%)	H ₂ O (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—
CONTROL	—	—	—	—	99.95	—
TEST	—	—	—	0.75	99.6	C ₂ H ₆ 0.16

RCS-032

Test Witnesses: N.S. Cannon
 Cognizant Engineer
A.J. Hugg
 Quality Assurance

8-31-95
 Date
08-01-95
 Date

[illegible]

TEST TYPE	EXT. PRESS.	INT. PRESS.	TEMP. (°C)	COMB. TEMP. (°C)	COMB. VOL. (cm ³)	COMB. CORR. (cm ³)	CALCULATED VOLUME (cm ³)
1	1.0	1.0	25.0	25.0	10.0	10.0	10.0
2	1.0	1.0	25.0	25.0	10.0	10.0	10.0
3	1.0	1.0	25.0	25.0	10.0	10.0	10.0
4	1.0	1.0	25.0	25.0	10.0	10.0	10.0
5	1.0	1.0	25.0	25.0	10.0	10.0	10.0
6	1.0	1.0	25.0	25.0	10.0	10.0	10.0
7	1.0	1.0	25.0	25.0	10.0	10.0	10.0
8	1.0	1.0	25.0	25.0	10.0	10.0	10.0
9	1.0	1.0	25.0	25.0	10.0	10.0	10.0
10	1.0	1.0	25.0	25.0	10.0	10.0	10.0
11	1.0	1.0	25.0	25.0	10.0	10.0	10.0
12	1.0	1.0	25.0	25.0	10.0	10.0	10.0
13	1.0	1.0	25.0	25.0	10.0	10.0	10.0
14	1.0	1.0	25.0	25.0	10.0	10.0	10.0
15	1.0	1.0	25.0	25.0	10.0	10.0	10.0
16	1.0	1.0	25.0	25.0	10.0	10.0	10.0
17	1.0	1.0	25.0	25.0	10.0	10.0	10.0
18	1.0	1.0	25.0	25.0	10.0	10.0	10.0
19	1.0	1.0	25.0	25.0	10.0	10.0	10.0
20	1.0	1.0	25.0	25.0	10.0	10.0	10.0
21	1.0	1.0	25.0	25.0	10.0	10.0	10.0
22	1.0	1.0	25.0	25.0	10.0	10.0	10.0
23	1.0	1.0	25.0	25.0	10.0	10.0	10.0
24	1.0	1.0	25.0	25.0	10.0	10.0	10.0
25	1.0	1.0	25.0	25.0	10.0	10.0	10.0
26	1.0	1.0	25.0	25.0	10.0	10.0	10.0
27	1.0	1.0	25.0	25.0	10.0	10.0	10.0
28	1.0	1.0	25.0	25.0	10.0	10.0	10.0
29	1.0	1.0	25.0	25.0	10.0	10.0	10.0
30	1.0	1.0	25.0	25.0	10.0	10.0	10.0
31	1.0	1.0	25.0	25.0	10.0	10.0	10.0
32	1.0	1.0	25.0	25.0	10.0	10.0	10.0
33	1.0	1.0	25.0	25.0	10.0	10.0	10.0
34	1.0	1.0	25.0	25.0	10.0	10.0	10.0
35	1.0	1.0	25.0	25.0	10.0	10.0	10.0
36	1.0	1.0	25.0	25.0	10.0	10.0	10.0
37	1.0	1.0	25.0	25.0	10.0	10.0	10.0
38	1.0	1.0	25.0	25.0	10.0	10.0	10.0
39	1.0	1.0	25.0	25.0	10.0	10.0	10.0
40	1.0	1.0	25.0	25.0	10.0	10.0	10.0
41	1.0	1.0	25.0	25.0	10.0	10.0	10.0
42	1.0	1.0	25.0	25.0	10.0	10.0	10.0
43	1.0	1.0	25.0	25.0	10.0	10.0	10.0
44	1.0	1.0	25.0	25.0	10.0	10.0	10.0
45	1.0	1.0	25.0	25.0	10.0	10.0	10.0
46	1.0	1.0	25.0	25.0	10.0	10.0	10.0
47	1.0	1.0	25.0	25.0	10.0	10.0	10.0
48	1.0	1.0	25.0	25.0	10.0	10.0	

TEST TYPE	STEP	TIME	TEMP (°C)	Sensor 1 (Pa)	Sensor 2 (Pa)	Q ₂ (Pa)
GAS MIX ID	1	2.00	20.9	2.80	2.47	2.50
		2.02	20.9	2.80	2.47	2.50
GAS MIX ID	2	2.02	20.9	2.80	2.47	2.50
		2.04	20.9	2.80	2.47	2.50
GAS MIX ID	3	2.04	20.9	2.80	2.47	2.50
		2.06	20.9	2.80	2.47	2.50
GAS MIX ID	4	2.06	20.9	2.80	2.47	2.50
		2.08	20.9	2.80	2.47	2.50
GAS MIX ID	5	2.08	20.9	2.80	2.47	2.50
		2.10	20.9	2.80	2.47	2.50
GAS MIX ID	6	2.10	20.9	2.80	2.47	2.50
		2.12	20.9	2.80	2.47	2.50
GAS MIX ID	7	2.12	20.9	2.80	2.47	2.50
		2.14	20.9	2.80	2.47	2.50
GAS MIX ID	8	2.14	20.9	2.80	2.47	2.50
		2.16	20.9	2.80	2.47	2.50
GAS MIX ID	9	2.16	20.9	2.80	2.47	2.50
		2.18	20.9	2.80	2.47	2.50
GAS MIX ID	10	2.18	20.9	2.80	2.47	2.50
		2.20	20.9	2.80	2.47	2.50
GAS MIX ID	11	2.20	20.9	2.80	2.47	2.50
		2.22	20.9	2.80	2.47	2.50
GAS MIX ID	12	2.22	20.9	2.80	2.47	2.50
		2.24	20.9	2.80	2.47	2.50
GAS MIX ID	13	2.24	20.9	2.80	2.47	2.50
		2.26	20.9	2.80	2.47	2.50
GAS MIX ID	14	2.26	20.9	2.80	2.47	2.50
		2.28	20.9	2.80	2.47	2.50
GAS MIX ID	15	2.28	20.9	2.80	2.47	2.50
		2.30	20.9	2.80	2.47	2.50
GAS MIX ID	16	2.30	20.9	2.80	2.47	2.50
		2.32	20.9	2.80	2.47	2.50
GAS MIX ID	17	2.32	20.9	2.80	2.47	2.50
		2.34	20.9	2.80	2.47	2.50
GAS MIX ID	18	2.34	20.9	2.80	2.47	2.50
		2.36	20.9	2.80	2.47	2.50
GAS MIX ID	19	2.36	20.9	2.80	2.47	2.50
		2.38	20.9	2.80	2.47	2.50
GAS MIX ID	20	2.38	20.9	2.80	2.47	2.50
		2.40	20.9	2.80	2.47	2.50
GAS MIX ID	21	2.40	20.9	2.80	2.47	2.50
		2.42	20.9	2.80	2.47	2.50
GAS MIX ID	22	2.42	20.9	2.80	2.47	2.50
		2.44	20.9	2.80	2.47	2.50
GAS MIX ID	23	2.44	20.9	2.80	2.47	2.50
		2.46	20.9	2.80	2.47	2.50
GAS MIX ID	24	2.46	20.9	2.80	2.47	2.50
		2.48	20.9	2.80	2.47	2.50
GAS MIX ID	25	2.48	20.9	2.80	2.47	2.50
		2.50	20.9	2.80	2.47	2.50
GAS MIX ID	26	2.50	20.9	2.80	2.47	2.50
		2.52	20.9	2.80	2.47	2.50
GAS MIX ID	27	2.52	20.9	2.80	2.47	2.50
		2.54	20.9	2.80	2.47	2.50
GAS MIX ID	28	2.54	20.9	2.80	2.47	2.50
		2.56	20.9	2.80	2.47	2.50
GAS MIX ID	29	2.56	20.9	2.80	2.47	2.50
		2.58	20.9	2.80	2.47	2.50
GAS						

ANALYSIS	REH	H	H ₂ O	N	HE	AV	OTHER
TEST	—	—	—	—	—	—	8826
CONTROL	—	—	—	—	—	—	56bb
VENDOR							

Test Witnesses: M. J. [Signature] Cognizant Engineer B. J. [Signature] Quality Assurance
Date 9-5-95 Date 01-05-95

RESS SIMULATION TEST DATA SHEET

TEST DATE 9-6-95 Test Performer N.S. Cameron
TEST NAME RGS-052.SIM GAS SIMULANT: ARGON

TEST NAME: <u>MSD-USE 2011</u>		WGS SYMBOLS: <u>NR000</u>				
SAMPLER # <u>3</u>		SAMPLER VOL. (mL) <u>315.08</u>		WATER VOL. (mL) <u>284.2</u>		
GAS VOL. (mL) <u>30.9</u>		GAS PRESS. <u>100.0</u>		GAS TEMP(°C) <u>23</u>		
		(°F)				
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	Leak Test START	7:12	22.8	0.25	0.006	0.003
	Leak Test END	8:22	22.7	0.25	0.007	0.004
	EXTRUDE START	8:28	22.8	5.40	5.246	OFF S.
	EXTRUDE END	NA				
	MOI CYCLE END	7:53	50.5	NA	NA	NA
	Post Mix Start	8:21	20.7	5.55	5.396	OFF S.
	Post Mix End	11:31	21.0	5.57	5.414	OFF S.
	2nd Vol. Start	11:32	21.0	4.93	4.765	OFF S.
	2nd Vol. End	11:42	21.1	4.95	4.785	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 40.66 (KPa) CAB. TEMP(°C) 26.4
FINISH TIME 12:14 PM

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP	7.133 (unp)	21.3	100.76	21.4	35.20	21.3	
	35.20	21.3	100.84	21.4	56.65	21.3	
ANALYSIS RESULTS	N ₂ (%)	H ₂ (%)	H ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—	—
CONTROL	—	—	—	—	—	99.95	—
TEST	—	—	—	7	—	91.3	0.16

RGS-032

Test Witness: N.S. Cameron 9-6-95
Logizant Engineer Date
B.D. Hing 09-06-95
Quality Assurance Date

RESS SIMULATION TEST DATA SHEET

TEST DATE 9-9-95 Test Performer N.S. Cameron
TEST NAME RGS-053.SIM GAS SIMULANT: ARGON Helium

TEST NAME: (U-2) GDS-571						
SAMPLER # <u>2</u>		SAMPLER VOL. (mL) <u>315.26</u>		WATER VOL. (mL) <u>297.5</u>		
GAS VOL. (mL) <u>27.8</u>		GAS PRESS. <u>100.2</u>		GAS TEMP(°C) <u>23</u>		
TEST TYPE	TEST STEP	TIME	TEMP. (°C)	Sensor 1 (1000 T) (KPa)	Sensor 2 (100 T) (KPa)	Sensor 3 (10 T) (KPa)
	Leak Test START	7:56	22.1	0.25	0.003	0.001
	Leak Test END	8:06	21.0	0.24	0.003	0.002
	EXTRUDE START	9:09	22.0	5.07	4.922	OFF S.
	EXTRUDE END					
	MOI CYCLE END	9:37	51.3	NA	NA	NA
	Post Mix Start	11:04	20.7	4.99	4.146	OFF S.
	Post Mix End	11:14	20.9	5.01	4.869	OFF S.
	2nd Vol. Start	11:16	20.9	4.48	4.329	OFF S.
	2nd Vol. End	11:26	21.0	4.51	4.358	OFF S.

Hg PUMP INSOLUBLE EXTRACTOR GASES TO COLLECTION CANISTERS.

FINAL PT4 33.40 (KPa) CAB. TEMP(°C) 26.0
FINISH TIME 11:59

TEST TYPE	EXTRACTOR PRESS. (KPa)	EXT. TEMP. (°C)	2nd VOL PRESS. (KPa)	2nd V TEMP. (°C)	COMBINED PRESS. (KPa)	COMB. TEMP. (°C)	CALCULATED SOLID/LIQUID VOLUME (mL)
BP	3.21	21.2	100.63	21.5	35.16	21.2	
	35.16	21.2	100.65	21.4	56.60	21.2	
ANALYSIS RESULTS	N ₂ (%)	H ₂ (%)	H ₂ O (%)	N ₂ (%)	He (%)	Ar (%)	OTHER (%)
VENDOR	—	—	—	—	—	—	—
CONTROL	—	—	—	0.19	99.78	—	—
TEST	—	—	—	2.46	96.69	—	0.87

RGS-054

Test Witness: N.S. Cameron 9-11-95
Logizant Engineer Date
B.J. Nuy 09-11-95
Quality Assurance Date

BEST AVAILABLE COPY

Sampler # 2 Sampler Volume 315.3 cc RGS-055
Fill Gas Hydrogen(30.3%) / Argon(24.9%) / Nitrogen(39.8%) / Ammonia(5.0%)
Fill Pressure 750.2 Fill Temperature 22°C
Fill Date 9-12-95 Fill Time 10:08
Sample Recovery (to test canister): Date 9-13-95 Time 9:57 AM
Sample Recovery Dump Pressure 177.1 Temperature 23°C
Mass Spec. Analysis Date 9-13-95 Approximate Analysis Time _____
MS Ammonia Result: 1.3 %
MS Results: H₂ 31 %, Ar 26.1 %, Nitrogen 41.6 %
Expected* H₂ 31.5 %, Ar 25.9 %, Nitrogen 41.4 %
* After compensation for ammonia wash out. $\Delta t = 24$ h

Sampler # 1 Sampler Volume 314.6 RGS-056
Fill Gas Hydrogen(30.3%) / Argon(24.9%) / Nitrogen(39.8%) / Ammonia(5.0%)
Fill Pressure 749.5 Fill Temperature 22°C
Fill Date 9-12-95 Fill Time 7:00 am
Sample Recovery (to test canister): Date ~~9-13-95~~⁹⁻¹⁵⁻⁹⁵ Time 8:50 AM
Sample Recovery Dump Pressure ~~177.1~~^{174.6} Temperature ~~23°C~~^{23°C}
Mass Spec. Analysis Date 9-15-95 Approximate Analysis Time before noon
MS Ammonia Result: 1.3 %
MS Results: H₂ 30.5 %, Ar 26.2 %, Nitrogen 41.9 %
Expected* H₂ 31.5 %, Ar 25.9 %, Nitrogen 41.4 %
* After compensation for ammonia wash out. $\Delta t = 74$ h

Sampler # 3 Sampler Volume 315.1 cc RGS-057
Fill Gas Hydrogen(30.3%) / Argon(24.9%) / Nitrogen(39.8%) / Ammonia(5.0%)
Fill Pressure 750.0 Fill Temperature 23 °C
Fill Date 9-12-95 Fill Time 103
Sample Recovery (to test canister): Date 9-20-95 Time 8:07 AM
Sample Recovery Dump Pressure 174.3 Temperature 22 °C
Mass Spec. Analysis Date 9-21-95 Approximate Analysis Time 9:00 AM
MS Ammonia Result: 0.04 %
MS Results: H₂ 31.3 %, Ar 26.4 %, Nitrogen 42.2 %
Expected* H₂ 31.9 %, Ar 26.2 %, Nitrogen 41.9 %
* After compensation for ammonia wash out. $\Delta t = 187 \text{ h}$

Sampler # 2 Sampler Volume 315.3 cc RGS-058
Fill Gas Hydrogen(30.3%) / Argon(24.9%) / Nitrogen(39.8%) / Ammonia(5.0%)
Fill Pressure 251.5 Fill Temperature 22 °C
Fill Date 9-15-95 Fill Time 8:32 AM
Sample Recovery (to test canister): Date 11/30/95 Time 1:50 PM
Sample Recovery Dump Pressure 60.5 Temperature 27 °C
Mass Spec. Analysis Date 12-1-95 Approximate Analysis Time AM
MS Ammonia Result: 0.23
MS Results: H₂ 30.5 %, Ar 26.5 %, Nitrogen 42.7 %
Expected* H₂ 31.8 %, Ar 26.2 %, Nitrogen 41.8 %
* After compensation for ammonia wash out. $\Delta t = 77 \text{ days}$

APPENDIX D

Adjusting Gas Composition for Leakage or Ammonia Washout

The result of a gas analysis may indicate the presence of potential air leakage or outgassing due to the presence of its constituents (oxygen, argon). Also, when MIX#1 gas was used, some ammonia "washout" was usually experienced with the MS analysis. These composition analyses can be compensated for this gain or loss of gas, as will be discussed.

D.1 Leakage/Outgassing

Since the RGSS has been carefully leak tested, actual leakage during a test normally would not exceed 1×10^{-6} atm/cc/sec unless a defective sampler or poor attachment of a sampler to the RGSS occurs. Outgassing from the RGSS chamber walls will always occur, although the effect can be minimized by extended vacuum pumpdowns prior to performing a test. Compensation of composition analysis results for extra air can be made provided it is known that the tracking constituent (oxygen, argon) were not originally in the sample gas. It is assumed that the leaked or outgassed air has a normal composition; i.e., nitrogen at 78.084%, oxygen at 20.946% and argon at 0.934%.

D.1.1 Oxygen Tracer

Using oxygen as the tracer element for air, the adjusted (A_i) fraction (of the i th gas component) of the original sample gas is given by

$$A_i = \frac{Q_i - f_i}{(1 - f_{ar} - f_{nit} - f_{O_2})}$$

where Q_i is the fraction actually measured for the i th gas component. If the i th gas component is not nitrogen, oxygen or argon, then $f_i = 0$. For nitrogen or argon, then f_i equals the appropriate value of either

$$f_{nit} = 0.78084/0.20946 * f_{O_2},$$

$$\text{or} \quad f_{ar} = 0.00934/0.20946 * f_{O_2},$$

$$\text{where} \quad f_{O_2} = \text{measured fraction of oxygen.}$$

D.1.2 Argon Tracer

Using argon as the tracer element for air, the same equation applies as for oxygen, except that

$$f_{nit} = 0.78084/0.00934 * f_{ar},$$

$$\text{and} \quad f_{O_2} = 0.20946/0.00934 * f_{ar},$$

$$\text{where} \quad f_{ar} = \text{measured fraction of argon.}$$

As an example, compensation for air outgassing during test RGS-001 was made in the following steps. The measured composition (see Table 12) was $Q_{N_2O} = 0.290$, $Q_{nit} = 0.327$, $Q_{he} = 0.371$. Oxygen (unexpected) was used as the tracer, so that $f_{O_2} = 0.0104$. Thus, $f_{nit} = 0.03877$ and $f_{ar} = 4.6374 \times 10^{-4}$. Let $f_{sum} = f_{O_2} + f_{nit} + f_{ar} = 0.04963$, then

$$A_{N_2O} = .290/(1 - 0.04963) = 0.290/0.950366 = 0.3051,$$

$$A_{nit} = (0.327 - 0.03877)/(0.950366) = 0.3033,$$

$$\text{and} \quad A_{he} = 0.371/0.950366 = 0.3904,$$

which are in excellent agreement with the control sample (RGS-002) results.

D.2 Ammonia Washout

It was difficult to transfer a gas mix containing ammonia from a collection vessel into the 325 Building mass spectrometer without losing some of the ammonia to wall surfaces and other moisture traps. Thus even the control samples taken directly from the vendor's gas bottle into a collection canister and to the MS resulted in low ammonia concentration results. Whenever gas MIX#1 was used in a test with water simulant, all of the ammonia washed out of the gas into the water, as would be expected.

An algorithm to compensate the known composition of MIX#1 gas for ammonia washout, thus allowing a comparison with the MS gas analysis results was made as follows. The new fraction of a MIX#1 gas component (AC_i) remaining after ammonia washout is given by

$$AC_i = C_i * Z$$

where $Z = (1 - NC_{amm})/C_{sum}$, with NC_{amm} being the measured fraction of ammonia from the MS data, and $C_{sum} = C_{H_2} + C_{ar} + C_{nit}$ where C_i is the known starting fraction of a given component of gas MIX#1.

As an example, compensation of the MIX#1 composition is made for test RGS-005 step by step; RGS-004 control test values are used for the known composition of MIX#1, rather than vendor values. The MS value for ammonia was $NC_{amm} = 0.014$; the original measured fractions for the rest of MIX#1 components are $C_{H_2} = 0.292$, $C_{ar} = 0.259$ and $C_{nit} = 0.417$; thus, $C_{sum} = 0.968$ when the ammonia concentration was measured as 2.9 percent. Then

$$AC_{H_2} = 0.292 * (1 - 0.014)/.968 = 0.292 * 1.0186 = 0.2974,$$

$$AC_{ar} = 0.259 * 1.0186 = 0.2638,$$

$$AC_{nit} = 0.417 * 1.0186 = 0.4248,$$

which are in good agreement with the actual test results (see Table 12).

APPENDIX E ACCEPTANCE TEST SUMMARY

Test Name	Description of Test	Status	Comments
RGS_001	Injection MIX2 - 26.7 °C Dry PNL IMS Anal.(6-30-95 Log-in No. 95-06958)	MIX2 Dry Tests are Completed	Excellent Results
RGS_002	MIX2 Control (001) - Ambient PNL IMS Anal.(6-30-95 Log-in No. 95-06959)		Taken with 001
RGS_003	Injection MIX2 - 52.7 °C Dry PNL IMS Anal.(6-30-95 Log-in No. 95-07005)		Excellent Results
RGS_004	MIX1 Control (005) - Ambient PNL IMS Anal.(7-12-95 Log-in No. 95-07111)	NH3 Low.	MS NH3 2.9%, Rest OK
RGS_005	Injection MIX1 - 28.0 °C Dry PNL IMS Anal.(7-12-95 Log-in No. 95-07112)		MS NH3 1.4%, Rest OK WC NH3 2.1%
RGS_006	Injection MIX1 - 52.4 °C Dry PNL IMS Anal.(7-12-95 Log-in No. 95-07123)		MS NH3 1.8%, Rest OK WC NH3 2.7%
RGS_007	Sampler Air Test	Complete	Volume Verif. - OK
RGS_008	Ammonia (MIX1) - 25.4 °C Dry	NH3 Still Low	WC NH3 3.48%
RGS_009	Ammonia (MIX1) - 52.5 °C Dry		WC NH3 2.74%
RGS_010	Air/Water 25.7 °C - No 2nd V.	Air/H2O & Sampler Tests: General Problems	Sampler - Leak!
RGS_011	Air(39.2 cc)/Water 26.4 °C		Sampler - SPT, Hg OK; BT High
RGS_012	Air(39.9 cc)/Water 52.1 °C		Sampler - SPT Too High; Plugged filter inval. BT, Hg
RGS_013	Air(39.9 cc)/Water 21.8 °C	Gen. Prob. New Line Volume	Sampler - SPT high; BT, Hg OK
RGS_014	Air(40.2 cc)/Water 53.4 °C		Sampler - SPT, BT bad; Hg OK but filter plugged
RGS_015	Injected(N2O+)/Water 22.0 °C PNL IMS Anal.(7-31-95 Log-in No. 95-07533)	Inj. N2O+ Problems	Obvious Leak! MS comp. awful
RGS_016	Injected(N2O+)/Water 22.2 °C PNL IMS Anal.(7-31-95 Log-in No. 95-07534)		Hg & BT OK; SPT & MS comp. poor
RGS_017	Injected(N2O+)/Water 53.3 °C PNL IMS Anal.(7-31-95 Log-in No. 95-07545)		Filter Plugged. MS comp. awful
RGS_018	Air(19.7 cc)/Carbopol 22 °C	Carbopol May Not Work -NSC-	Sampler - Aborted-Massive Leak

Test Name	Description of Test	Status	Comments
RGS_019	Air(22.7cc)/Carbopol 22 °C	No Good	Sampler - Massive Outgassing/Leak??
RGS_020	Ammonia (MIX1) - 21.9 °C Dry	NH3 Low	WC NH3 2.0%
RGS_021	Injected(N2O+)/Water @ 22°C Note: Water not degassed. PNL IMS Anal.(8-15-95 Log-in No. 95-07848) PNL IMS Anal.(8-16-95 Log-in No. 95-07849)	Not Good	Pumped and unpumped - not good enough
RGS_022	Dry gas injection/22°C (N2O+) PNL IMS Anal.(8-16-95 Log-in No. 95-07857)	Still works!	Excellent MS results
RGS_023	Injected(N2O+)/Water @ 22°C	Not Good	LEAKED! Aborted.
RGS_024	Injected(N2O+)/Water @ 22°C (New, degassed water)	Degassed Water	ABORTED! 2nd Vol. not pumped down.
RGS_025	Injected(N2O+)/Water @ 22°C (Reused water) PNL IMS Anal.(8-18-95 Log-in No. 95-07888)	Injection Tests	Good MS results
RGS_026	Injected(N2O+)/Water @ 22°C 752.2 Torr (Reused Water) PNL IMS Anal.(8-18-95 Log-in No. 95-07894)	Works!!!!	Excellent MS results
RGS_027	RCK Inj.(N2O+)/H2O @ 22°C 400.1 Torr (Reused Water) PNL IMS Anal.(8-22-95 Log-in No. 95-08090)	RCK Injection Tests	Excellent MS results
RGS_028	RCK Inj.(N2O+)/H2O @ 22°C 200.1 Torr (Reused Water) PNL IMS Anal.(8-22-95 Log-in No. 95-08091)		Excellent MS results
RGS_029	RCK Inj.(N2O+)/H2O @ 22°C 50.1 Torr (Reused Water) PNL IMS Anal.(8-23-95 Log-in No. 95-08094)		Poor MS results
RGS_030	BH Inj. (Argon)/H2O @ 22°C: 741.1 Torr (Reused Water) PNL IMS Anal.(8-23-95 Log-in No. 95-08037)	Argon Only is	4.5% MS Error (Unpumped)
RGS_031	BH Inj. (Argon)/H2O @ 22°C: Same test as RGS_030 (pumped) PNL IMS Anal.(8-23-95 Log-in No. 95-08038)	Simpler	4.5% MS Error (Hg Pumped)
RGS_032	BH Control Sample (Argon) PNL IMS Anal.(8-24-95 Log-in No. 95-08039)	Within Specs.	99.95% Ar (MS result)
RGS_033	BH Inj. Argon(740.4T)/H2O (New Water, degassed) PNL IMS Anal.(8-24-95 Log-in No. 95-08048)	B. Hey Ar/H2O Injection Tests	1.6% MS Error
RGS_034	BH Inj. 400 Torr Argon/Water (Reused water) PNL IMS Anal.(8-25-95 Log-in No. 95-08057)		98.3% Ar (Unpumped)
RGS_035	Same test as RGS_034 (pumped) PNL IMS Anal.(8-25-95 Log-in No. 95-08058)		99.7% Ar (Hg Pumped)

Test Name	Description of Test	Status	Comments
RGS_036	BH Inj. 400.9T Ar/Water-Reused PNL IMS Anal.(8-25-95 Log-in No. 95-08059)	B. Hey	99.6% Ar (Hg Pumped)
RGS_037	Injected(H ₂ +) @ 740.1T/Water (Reused Water) PNL IMS Anal.(8-25-95 Log-in No. 95-08070)	H ₂ !!!!!	Good MS Results
RGS_038	Inj. Ar (50.7T)/Water-Reused PNL IMS Anal.(8-28-95 Log-in No. 95-08239)	Ar Tests	82.8% Ar
RGS_039	Inj. Ar(102.0T)/Water-Reused PNL IMS Anal.(8-28-95 Log-in No. 95-08239)	Ar	96.3% Ar
RGS_040	BH Inj. Ar(201.0T)/Water-Reused. "Dumped" Sample PNL IMS Anal.(8-29-95 Log-in No. 95-08258)	B Hey Ar/H ₂ O Injection Tests	ERROR - No Ar originally obtained in injection can.
RGS_041	BH - Same as RGS_040, but Hg Pumped. PNL IMS Anal.(8-29-95 Log-in No. 95-08259)		ERROR - No Ar originally obtained in injection can.
RGS_042	BH Inj. 200.0T Ar/Water - was Hg pumped. Reused Water PNL IMS Anal.(8-29-95 Log-in No. 95-08260)		ERROR - No Ar originally obtained in injection can.
RGS_043 B	BH Inj. 100.1T Ar/Water - Hg pumped. Reused Water PNL IMS Anal.(8-29-95 Log-in No. 95-08267)		94.4% Ar MS Result
RGS_043 C	BH Inj. 100.1T Ar/Water - "Dumped". Reused Water PNL IMS Anal.(8-29-95 Log-in No. 95-08268)		94.5% Ar MS Result
RGS_044	Argon Control Sample PNL IMS Anal.(8-29-95 Log-in No. 95-08269)		99.949% Ar MS Result
RGS_045 A	BH Inj. Ar(100.0T)/H2O-Reused "Dumped" PNL IMS Anal.(8-30-95 Log-in No. 95-08285)	B Hey Ar/H2O Injection Tests	85.9% Ar MS Result
RGS_045 B	BH Inj. Ar(100.0T)/H2O-Reused "Hg Pumped" PNL IMS Anal.(8-30-95 Log-in No. 95-08286)		97% Ar MS Result
RGS_046 A	BH Inj. Ar(75.0T)/H2O-Reused "Dumped" PNL IMS Anal.(8-30-95 Log-in No. 95-08287)		31.2% Ar MS Result
RGS_046 B	BH Inj. Ar(75.0T)/H2O-Reused "Hg Pumped" PNL IMS Anal.(8-30-95 Log-in No. 95-08288)		95.65% Ar MS Result
RGS_047	Inj. Ar (400.6T)/Water-Reused Hot Cycle to 50.6°C PNL IMS Anal.(8-30-95 Log-in No. 95-08377)	Injected Ar/H2O- Degassed Tests	99.25% Ar MS Result Hg Pumped
RGS_048 A	Inj. Ar (200.1T)/Water-Reused "Dumped" PNL IMS Anal.(8-31-95 Log-in No. 95-08382)		92.5% Ar MS Result Dumped Sample

Test Name	Description of Test	Status	Comments
RGS_048 B	Inj. Ar (200.1T)/Water-Reused "Hg Pumped" PNL IMS Anal.(8-31-95 Log-in No. 95-08383)	Injected Ar/H2O- Degassed Tests	93.7% Ar MS Result Hg Pumped Sample
RGS_049	Inj. Ar (201.7)/Water-Reused Hot Cycle to 50.1°C PNL IMS Anal.(8-31-95 Log-in No. 95-08415)		98.59% Ar MS Result Hg Pumped Sample
RGS_050	Inj. Ar (100.2)/Water-Reused Hot Cycle to 50.1°C PNL IMS Anal.(9-1-95 Log-in No. 95-08424)		97.38 Ar MS Result Hg Pumped Sample
RGS_051	Complete Sampler Argon/Water RGSS Test: Ambient Temp. only PNL IMS Anal.(9-5-95 Log-in No. 95-08493)	Complete RGSS Gas/Water Sampler Tests GOOD	98.0% Ar MS Results Overnight degas.
RGS_052	Complete Sampler Argon/Water RGSS Test: Hot Cycle to 50.5 °C PNL IMS Anal.(9-6-95 Log-in No. 95-08507)		91.3% Ar MS Results Only 5 hr degas.
RGS_053	Complete Sampler HELIUM /Water RGSS Test: Hot Cycle to 51.3 °C PNL IMS Anal.(9-8-95 Log-in No. 95-08523)		96.89% He MS Results Overnight Degas - (Sampler & RGSS)
RGS_054	Helium Control Sample PNL IMS Anal.(9-11-95 Log-in No. 95-08554)		99.78% He MS Results
RGS-55	H ₂ Sampler Leak Test (24 hr) PNL IMS Anal.(9-13-95 Log-in No. 95-08647)	MIX#1 H ₂ + Sampler "Aging" Tests	Maximum Error 0.5 %
RGS-56	H ₂ Sampler Leak Test (74 hr) PNL IMS Anal.(9-15-95 Log-in No. 95-08719)		Maximum Error 1.0 %
RGS-57	H ₂ Sampler Leak Test (187 hr) PNL IMS Anal.(9-21-95 Log-in No. 95-08778)		Maximum Error 0.6 %
RGS-58	H ₂ Sampler Leak Test (1829 hr) PNL IMS Anal.(11-30-95 Log-in No.96-01643)		Maximum Error 0.4 %

APPENDIX F
Letter Report: Hydrogen Permeation Rate

Westinghouse
Hanford Company

Internal
Memo

From: Nuclear Fuel Evaluations
Phone: 376-9539
Date: April 28, 1994
Subject: HYDROGEN PERMEATION RATE FOR RETAINED GAS SAMPLER

To:	A. E. Bridges	L5-01		
cc:	R. E. Bauer	L6-37	R. P. Omberg	HO-40
	K. R. Birney	HO-40	A. L. Pitner	L5-01
	G. J. Bogen	R1-17	D. J. Sherwood	R2-78
	R. C. Bolden, Jr.	S2-48	B. J. Webb	S2-48
	S. A. Chastain	L5-01	D. W. Wootan	HO-40
	G. D. Johnson	R2-78		

An evaluation was performed to estimate the hydrogen permeation rate for the Retained Gas Sampler. The evaluations were done in a conservative fashion and should represent an upper bound for hydrogen permeation. Calculations were performed for two temperatures: a nominal value of 100 °F, and the upper design limit of 200 °F. At 100 °F the total permeation rate is calculated to be <0.1% of the hydrogen inventory per day, while at 200 °F the calculated rate is 0.4% per day.

Individual calculations were performed for the sampler housing, the O-ring seal on the piston, and the Viton seal for the rotary valve. Conservative approaches were applied throughout to obtain upper bound estimates. For example, the retrieved sample was assumed to be 100% hydrogen to provide the maximum partial pressure for permeation, and permeability values selected for the various materials were the highest reported. In reality, most of the hydrogen in the recovered samples would be expected to remain entrained in the viscous waste material and not have direct access to the containment barriers.

Most of the hydrogen permeation was calculated to occur through the O-ring seal on the piston, which accounted for about 2/3 of the total hydrogen permeation rate. Accordingly, application of multiple O-ring seals in this region could substantially reduce the calculated permeation rate.

Details of the hydrogen permeation calculations for the Retained Gas Sampler are presented in the following pages.

A. L. Pitner

Hydrogen Sample Inventory

As a bounding case, assume that the sample obtained in the 19-inch piston stroke volume is 100% hydrogen at 150 °F under a hydrostatic head of 3 atmospheres (Wootan 1993).

$$n = \frac{PV}{RT}$$

where n = moles

P = pressure = 3 atm

$$V = \text{volume} = \frac{\pi d^2 \ell}{4} = (0.7854) (1.126 \text{ in.})^2 (19 \text{ in.})$$

$$= 18.92 \text{ in}^3 = 0.310 \text{ liter}$$

R = gas constant = 0.08205 l·atm/mol·K

T = temperature = 150 °F = 339 K

$$n = \frac{(3 \text{ atm}) (0.310 \text{ l})}{(0.08205 \text{ l·atm/mol·K}) (339 \text{ K})} = \boxed{0.03344 \text{ mol}}$$

Permeation Through Sample Housing

The permeation rate of hydrogen through metal is given by (Steward 1983)

$$Q/t = K \frac{A}{\Delta x} \Delta P^{1/2}$$

where Q/t = mass permeation rate (mol/s)

K = permeability (mol/m·s·Pa^{1/2})

A = surface area (m²)

Δx = wall thickness (m)

ΔP = differential hydrogen partial pressure (Pa)

From (Steward 1983) for 304 stainless steel,

$$K @ 100^\circ\text{F} = 10.4 \times 10^{-7} \exp(-7770/311) = 1.84 \times 10^{-17} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}^{1/2}$$

$$K @ 200^\circ\text{F} = 10.4 \times 10^{-7} \exp(-7770/367) = 8.04 \times 10^{-16} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}^{1/2}$$

$$P @ 100^\circ\text{F} = 3 \text{ atm } (311 \text{ K}/339 \text{ K}) = 2.53 \text{ atm} = 278,868 \text{ Pa}$$

$$P @ 200^\circ\text{F} = 3 \text{ atm } (367 \text{ K}/339 \text{ K}) = 3.25 \text{ atm} = 329,082 \text{ Pa}$$

$$A = \pi (1.126) (19) = 67.21 \text{ in}^2 = 0.0434 \text{ m}^2$$

$$\Delta x = 0.187 \text{ in.} = 0.00475 \text{ m}$$

$$Q/t @ 100^\circ\text{F} = (1.84 \times 10^{-17} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}^{1/2}) \left(\frac{0.0434 \text{ m}^2}{0.00475 \text{ m}} \right) (278,868 \text{ Pa})^{1/2}$$

$$= 8.88 \times 10^{-14} \text{ mol/s} = 7.67 \times 10^{-9} \text{ mol/day}$$

$$= 2.3 \times 10^{-5} \%/\text{day}$$

$$Q/t @ 200^\circ\text{F} = (8.04 \times 10^{-16} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}^{1/2}) \left(\frac{0.0434 \text{ m}^2}{0.00475 \text{ m}} \right) (329,082 \text{ Pa})^{1/2}$$

$$= 4.21 \times 10^{-12} \text{ mol/s} = 3.64 \times 10^{-7} \text{ mol/day}$$

$$= 0.001 \%/\text{day}$$

Permeation Through O-Ring

The permeation through elastomers is related directly to the hydrogen partial pressure rather than the square root of the hydrogen partial pressure (Irwin 1992)

$$Q/t = K \frac{A}{\Delta x} \Delta P$$

where Q/t = mass permeation rate (mol/s)

K = permeability (mol/m·s·Pa)

A = surface area (m²)

Δx = wall thickness (m)

ΔP = differential hydrogen partial pressure (Pa)

The O-ring material is a fluorocarbon (Viton). From (Irwin 1992), the permeability for Viton as a function of temperature (T = °C) is given by

$$K \text{ (mol/m·s·Pa)} = -2.51 \times 10^{-15} + 2.96 \times 10^{-16} T - 8.21 \times 10^{-18} T^2 \\ + 1.338 \times 10^{-19} T^3 - 4.635 \times 10^{-22} T^4$$

For 100 °F (38 °C), $K = 3.26 \times 10^{-15}$ mol/m·s·Pa

For 200 °F (93 °C), $K = 2.73 \times 10^{-14}$ mol/m·s·Pa

$P @ 100 \text{ °F} = 278,868 \text{ Pa}$

$P @ 200 \text{ °F} = 329,082 \text{ Pa}$

Area based on region between housing inner wall and bottom of O-ring groove (Drawing H-2-85097, Rev 2, Sheet 4)

$$A = \pi/4 (1.126^2 - 0.885^2) = 0.381 \text{ in}^2 = 2.456 \times 10^{-4} \text{ m}^2$$

Determine the average lateral thickness in the circular cross section of an O-ring. The results are conservative, because compression of the O-ring will deform it into a geometry that increases the effective thickness to resist gas permeation.

$$x = R \cos \theta$$

$$\bar{x} = \frac{\int_0^{\pi/2} R \cos \theta \, d\theta}{\pi/2} = \frac{R [\sin(\pi/2) - \sin(0)]}{\pi/2}$$

$$\bar{x} = \frac{R (1 - 0)}{\pi/2} = 0.637 R$$

$$\text{Average thickness} = 2 * \bar{x} = 0.637 D$$

$$\Delta x = 0.637 \left(\frac{1.126 - 0.885}{2} \right) = 0.077 \text{ in.} = 0.00195 \text{ m}$$

$$Q/t @ 100^\circ\text{F} = (3.26 \times 10^{-15} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}) \left(\frac{2.456 \times 10^{-4} \text{ m}^2}{0.00195 \text{ m}} \right) (278,868 \text{ Pa})$$

$$= 1.15 \times 10^{-10} \text{ mol/s} = 9.89 \times 10^{-6} \text{ mol/day}$$

$$= 0.030 \text{ \%/day}$$

$$Q/t @ 200^\circ\text{F} = (2.73 \times 10^{-16} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}) \left(\frac{2.456 \times 10^{-4} \text{ m}^2}{0.00195 \text{ m}} \right) (329,082 \text{ Pa})$$

$$= 1.13 \times 10^{-9} \text{ mol/s} = 9.78 \times 10^{-5} \text{ mol/day}$$

$$= 0.292 \text{ \%/day}$$

Permeation Through Viton Seal in Valve

The formulation for hydrogen permeation through the Viton seal in the rotary valve is the same as that for the O-ring given above.

$$K @ 100^{\circ}\text{F} = 3.26 \times 10^{-15} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}$$

$$K @ 200^{\circ}\text{F} = 2.73 \times 10^{-14} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}$$

$$P @ 100^{\circ}\text{F} = 278,868 \text{ Pa}$$

$$P @ 200^{\circ}\text{F} = 329,082 \text{ Pa}$$

Determine the inner exposure area of the Viton seal on the rotary valve.
From Drawing H-2-85097, Rev 2, Sheet 5:

$$(x + 0.5625)^2 + (0.465)^2 = (0.860)^2$$

$$x = 0.161 \text{ in.}$$

$$D = 1.125 + 2 \times 0.161 = 1.447 \text{ in.}$$

$$A = \pi/4 (1.697^2 - 1.447^2)$$

$$= 0.6173 \text{ in}^2 = 3.983 \times 10^{-4} \text{ m}^2$$

$$\Delta x = 0.260 \text{ in.} = 0.00660 \text{ m}$$

$$\begin{aligned}
 Q/t @ 100^\circ\text{F} &= (3.26 \times 10^{-15} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}) \left(\frac{3.983 \times 10^{-6} \text{ m}^2}{0.00660 \text{ m}} \right) (278,868 \text{ Pa}) \\
 &= 5.49 \times 10^{-11} \text{ mol/s} = 4.74 \times 10^{-6} \text{ mol/day} \\
 &= 0.014 \text{ \%/day}
 \end{aligned}$$

$$\begin{aligned}
 Q/t @ 200^\circ\text{F} &= (2.73 \times 10^{-14} \text{ mol/m}\cdot\text{s}\cdot\text{Pa}) \left(\frac{3.983 \times 10^{-6} \text{ m}^2}{0.00660 \text{ m}} \right) (329,082 \text{ Pa}) \\
 &= 5.42 \times 10^{-10} \text{ mol/s} = 4.68 \times 10^{-5} \text{ mol/day} \\
 &= 0.140 \text{ \%/day}
 \end{aligned}$$

Total Permeation Rate

The total permeation rate of hydrogen through the universal sampler containment is the sum of the above constituents.

$$Q/t @ 100^\circ\text{F} = 0.044 \text{ \%/day}$$

$$Q/t @ 200^\circ\text{F} = 0.433 \text{ \%/day}$$

References

Irwin, J. J., 1992, "A Survey of Fluid Flow Leakage," WHC-SD-TP-RPT-003, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Steward, S. A., 1983, "Review of Hydrogen Isotope Permeability Through Materials," UCRL-53441, Lawrence Livermore National Laboratory, Livermore, California.

Wootan, D. W., and Bridges, A. E., 1993, "Preliminary Design Requirements and Criteria for the Prototype Retained Gas Sampler and Extraction (RGS) System," WHC-SD-WM-CR-054, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

APPENDIX G

Two Volume Method Ammonia Vapor Pressure Approximation

The Two Volume method for determining gas quantity and soluble gas vapor pressure (P_v) relies on the approximation that P_v remains relatively constant during the expansion of the extractor volume to include the 2nd Volume. Without this assumption, there are too many unknowns to produce Equations 5 and 6. Opening the valve to combine the extractor volume with the 2nd Volume results in only a 25 percent increase in the total volume to which the sample is exposed; thus superficially, the approximation that P_v is constant seems reasonable when dealing with soluble gases.

However, Shekarritz and Norton (PNLFGP: 091595) have analyzed the approximation of constant ammonia vapor pressure using Henry's Law considerations, and point out some potential problems. Consequently, a further analysis will be performed here specific to the current RGSS geometry and Two Volume method.

G.1 Ammonia Vapor Pressure Variation on Volume Expansion

Two equations from Shekarritz and Norton are borrowed (A.2 and A.4) to start the analysis development:

$$P_{v_{NH_3}} = H \frac{n_{1_{NH_3}}}{m_L} \quad (G-1)$$

which is Henry's Law, where H is Henry's constant for ammonia. The total mass of water in the waste is m_L , and $n_{1_{NH_3}}$ is the number of moles of ammonia in the liquid. The second equation was derived by Shekarritz and Norton as

$$\frac{n_{1_{NH_3}}}{n_{NH_3}} = \frac{(RT/V_g)}{H/m_L + (RT/V_g)} \quad (G-2)$$

where n_{NH_3} is the total number of moles of ammonia both in solution and in vapor. The volume available to the ammonia gas is V_g .

Dropping the NH_3 subscripts, and combining the ratios of Equations G-1 and G-2 resulting from two different volume states, then

$$\frac{P_{v1}}{P_{v2}} = \frac{V_{g2} + RT(m_L/H)}{V_{g1} + RT(m_L/H)} \quad (G-3)$$

where at temperature T, P_{v1} is the ammonia vapor pressure in volume V_{g1} and P_{v2} is the ammonia vapor pressure in the new volume V_{g2} .

When H is very small (corresponding to high solubility), then Equation G-3 predicts that $P_{v1}/P_{v2} \approx 1$ and the approximation of constant vapor pressure is valid; when H is very large (insoluble condition) then $P_{v1}/P_{v2} \approx V_{g2}/V_{g1}$ as would be predicted by the perfect gas law for insoluble gases.

For acceptance testing, the extractor volume plus feed line volume totals were 1350 mL + 200 mL = 1550 mL. The 2nd Volume was approximately 320 mL. Thus, in the case of an essentially full sampler, $V_{g1} = 1550$ mL - 315 mL = 1235 mL and $V_{g2} = 1235$ mL + 320 mL = 1555 mL. The worst case change in ammonia vapor pressure would then be $P_{v1}/P_{v2} = 1.259$, or about 26 percent. For a nearly empty sampler, this worst case error would be reduced to about 20.6 percent.

By way of example, Shekarritz and Norton used $H/m_L = 2.2 \times 10^6$ Pa/mole as an approximation for real waste. With $RT = 2685.6$ J/mole (corresponding to 50 °C), then Equation G-3 becomes

$$\frac{P_{v1}}{P_{v2}} = \frac{V_{g2} + 1220.7 \text{ mL}}{V_{g1} + 1220.7 \text{ mL}} \quad (\text{G-4})$$

Thus for a test starting with a full sampler, $P_{v1}/P_{v2} = 1.13$ for a 13 percent reduction in ammonia vapor pressure when the evacuated 2nd Volume is exposed to the extractor. For a nearly empty sampler, this pressure ratio is 1.115 for an 11.5 percent vapor pressure change.

Figure G-1 indicates the sensitivity of the pressure ratio P_{v1}/P_{v2} (to the actual value of H/m_L) by plotting this ratio versus $X = \log_{10}(H/m_L/2.2 \times 10^6 \text{ Pa/mole})$.

G.2 Ammonia Vapor Pressure Variation Effects on Two Volume Method Results

A 26 percent change (for example) in ammonia pressure during expansion to include the 2nd Volume does not translate directly into a 26 percent error in gas quantity and vapor pressure calculated using the TV method, and in fact the error can be considerably less, as will be shown.

The basic equations used to derive Equations 5 and 6 for the TV method are reproduced here from the proof test report (WHC-SD-WM-TRP-232):

$$P_{EXL} = \frac{n_g RT}{V_{EXL} - V_{SL}} + P_v \quad (G-5)$$

and

$$P_2 = \frac{n_g RT}{V_{EXL} - V_{SL} + V_2} + P_v \quad (G-6)$$

The V_{SL} determination is not affected by the ammonia vapor pressure change provided that the BT "charge" volume ($V_2 + V_L$) has been exposed to the waste sample in the extractor before it is valved out and charged with argon or air to P_g (see Equation 2). Thus, there appear to be only two unknowns (n_g and P_v) in Equations G-5 and G-6, and they can be solved simultaneously to produce Equations 5 and 6.

However, if P_v actually varies during the TV method, then an error is introduced into Equations 5 and 6. Let $P_v = P_{H_2O} + P_{vi}$ where P_{vi} is the vapor pressure of ammonia in either volume State 1 (V_x) or State 2 ($V_x + V_2$); let $P_{v1} = f * P_{v2}$, and redetermine Equations 5 and 6 as

$$n_g = [P_{EXL} - P_2 - (f - 1)P_{v2}] \left(\frac{V_x}{V_2} \right) \left[\frac{V_x + V_2}{RT} \right] \quad (G-7)$$

and

$$P_{v2} = P_2 - \frac{n_g RT}{V_{EXL} - V_{SL} + V_2} - P_{H_2O} \quad (G-8)$$

where $V_x = V_{EXL} - V_{SL}$, and P_{H_2O} is the vapor pressure of water at temperature T.

G.2.1 Insoluble Gas Quantity Error

The fractional error (FE) in the insoluble gas quantity determination is defined as

$$FE = \frac{n_{g5} - n_{gF7}}{n_{gF7}} = \frac{n_{g5}}{n_{gF7}} - 1 \quad (G-9)$$

where n_{g5} is the insoluble gas quantity determined from Equation 5, and n_{gF7} is the "actual" gas quantity based on Equation G-7. Then

$$FE = \frac{(f - 1) P_{v2}}{[P_{exL} - P_2 - (f - 1) P_{v2}]} \quad (G-10)$$

Thus, the error in TV measured gas quantity is small if $(f - 1)P_{v2}$ is small compared to $P_{exL} - P_2$. The FE can be evaluated further by noting that $P_{exL} = P_{ins} + P_{H2O} + P_{v1}$ and $P_2 = P_{ins2} + P_{H2O} + P_{v2}$ where insoluble gas pressures are given by P_{ins} and P_{ins2} , and are related by $P_{ins}/P_{ins2} = (V_x + V_2)/V_x$. Subtracting P_2 from P_{exL} then yields

$$P_{exL} - P_2 = (1 - \frac{V_x}{V_x + V_2}) P_{ins} + P_{v1} - P_{v2} \quad (G-11)$$

Assume that the ammonia vapor pressure initially in the extractor constitutes a fraction (g) of the total gas pressure (excluding water vapor), such that $P_{v1} = g * (P_{ins} + P_{v1})$. Then

$$P_{exL} - P_2 = [(1 - \frac{V_x}{V_x + V_2}) (\frac{1}{g} - 1) f + (f - 1)] P_{v2} \quad (G-12)$$

Substituting Equation G-12 into Equation G-10 gives the relationship for the fractional error as

$$FE = \frac{f - 1}{[(1 - \frac{V_x}{V_x + V_2}) (\frac{1}{g} - 1) f]} \quad (G-13)$$

As an example, for waste gas consisting of 5 percent ammonia (excluding water vapor), $g = 0.05$ and assuming the worst case value of $f = 1.26$, then $FE = 0.0527$ for a 5.3 percent error in insoluble gas quantity. Coincidentally, assuming the worst case values for f previously described for a full or empty sampler results in $FE \approx g$. However, if $H/m_L = 2.2 \times 10^6$ Pa/mole giving $f = 1.13$, then for $g = 0.05$, $FE = 0.029$, or a 2.9 percent error in n_{g5} .

Thus, the gas quantity error resulting from the approximation of constant P_v during the TV expansion in volume (from the extractor to include the 2nd Volume) is manageable, particularly for samples with lower percentages of ammonia gas in the waste gas total.

G.2.2 Ammonia Vapor Pressure Error

The ammonia vapor pressure fractional error (PFE) due to the variation of ammonia pressure between the two measurement states when using Equation G-8 is defined as

$$PFE = \frac{P_{v25} - P_{v2}}{P_{v2}} = \frac{P_{v25}}{P_{v2}} - 1 \quad (G-14)$$

where P_{v25} is the ammonia vapor pressure in the second volume state ($V_x + V_2$) calculated from Equation G-8 using n_{g5} , and P_{v2} is the actual ammonia vapor pressure. Then

$$PFE = [P_2 - (P_{exL} - P_2) \frac{V_x}{V_2} - P_{H2O}] \frac{1}{P_{v2}} - 1 \quad (G-15)$$

resulting in

$$PFE = [(1 + \frac{V_x}{V_2}) (P_{ins2} + P_{v2}) - (P_{ins} + P_{v1}) \frac{V_x}{V_2}] \frac{1}{P_{v2}} - 1 \quad (G-16)$$

and finally

$$PFE = (1 - f) \frac{V_x}{V_2} \quad (G-17)$$

Thus, in the worst case condition of $f = 1.26$ and a nearly full sampler ($V_x = 1235 \text{ mL}$), $PFE = -1.00$, for a 100 percent error in the determination of the ammonia vapor pressure.

Clearly, the potentially large error in measured ammonia vapor pressure using the Two Volume method suggests that greater reliance will need to be placed on the Hg Pump method for this particular parameter.

It should be noted that f can be determined from a rearrangement of Equation G-17 if the Hg Pump method provides a reliable value for the ammonia vapor pressure (P_{v2}) and there are no other soluble gases present in significant quantity. A determination of the value of f would be key to characterizing the ammonia properties in a given sample. Also, if the ASA method for determining ammonia concentration can be made to work properly, then a comparison between the ASA and the Hg Pump values for ammonia vapor pressure would provide increased confidence in the value determined for f .

Figure G-1. Variation of P_{v1}/P_{v2} versus H/mL .

DISTRIBUTION SHEET

To Distribution	From Equipment Development	Page 1 of 1			
		Date June 26, 1996			
Project Title/Work Order Retained Gas Sampler System Acceptance Test Report, Revision 1		EDT No. ECN No. 624777			
Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
J. M. Bates	K7-15	X			
R. E. Bauer	S7-14	X			
C. J. Benar	R2-12	X			
R. F. Eggers	R2-12	X			
B. J. Hug	L6-35	X			
N. S. Cannon (5)	L6-38	X			
B. E. Hey	T6-09	X			
J. R. Jewett	T6-09	X			
G. D. Johnson	S7-14	X			
R. C. Knight (3)	L6-38	X			
L. S. Krogsrud	T4-07	X			
J. W. Lentsch	S7-14	X			
C. G. Linschooten	S7-12	X			
L. R. Pederson	K2-44	X			
A. L. Pitner	H0-40	X			
D. R. Rector	K7-15	X			
G. W. Rosenwald	S7-54	X			
J. S. Schofield	S7-12	X			
A. Shekariz	K7-15	X			
C. W. Stewart	K7-15	X			
J. R. Thielges	L6-38	X			
Central Files (1) + Orig.	A3-88	X			