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7. Abstract

The Retained Gas Sampler (RGS) system is being developed to permit characterization of the gas phase component of waste tank core samples. Several laboratory experiments have been conducted which have affirmed the proof-of-principle for separating the gas phase materials from waste tank material in a quantitative manner. However, experiments conducted thus far have dealt only with representative materials and simulated hardware mock-ups. This test plan deals with the operation and testing of actual devices in the hot cell environment. This test plan covers all aspects of the RGS system including: sampler load-in, extrusion, gas extraction, quantitative separation, sample collection, and quantitative analysis. Sample material used in this test plan will be waste tank simulants and will not be radioactive. The work environment, however, will be an operating hot cell facility and will have radioactive contaminated surfaces. Operation of the system will therefore require an official radiation work permit (RWP).

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8. RELEASE STAMP

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1.0 INTRODUCTION

Some slurries in Hanford's underground high level radioactive waste tanks generate gases (hydrogen, nitrogen, nitrous oxide) due to chemical and radiolytical mechanisms. These gases may be retained within the slurry and accumulate there. When a sufficient amount of gas has been accumulated, it may be suddenly released to the surface. This is termed a gas release event (GRE).

In order to determine whether the slurry in a given waste tank retains flammable gas mixtures, samples of the slurry must be retrieved and delivered to an analytical laboratory. The retained gas must be extracted from the slurry and analyzed. The analyses must be comprehensive enough to determine the chemical identity and concentration of the primary constituents of the retained gas and its association with the slurry as free (unbound) gas, physically bound gas, or chemically bound gas.

The purpose of the Retained Gas Sampler project is to develop the means for carrying out such an analysis (Reference 1). The functional design requirements (Reference 2) for the RGS system were established and a prototype RGS system is under development.

The design of the RGS prototype has centered on the concept of extracting gas from an entire waste tank core sample (approximately 310 ml). The core sample is extruded into an extractor device where the gas is "extracted" from the slurry through the application of several driving mechanisms (stirring, heating, dilution). The extracted gas is then pumped from the extractor to either a collection vessel or directly to an analytical instrument. The extractor device will be situated inside the hot cell whereas the pumping mechanism and collector/ analyzer instruments will be situated outside the hot cell.

A test plan was written (Reference 3) to test the proof-of-principle of the above concept. The plan primarily focuses on quantitative pumping of the gases from the extractor to the collection vessel. The results of those tests (Reference 4) verified that the conceptual design path was sufficient for quantifying the extracted gases. In addition, other aspects of this design which concern the sampler functionality, gas composition analysis accuracy, and void fraction measurement accuracy were also tested and the results of those tests (Reference 5) were also supportive of the design concept.

The next step in the RGS development is a full-scale test using simulated waste tank material. [The use of simulated waste tank material for cold testing would facilitate manned re-entry of the hot cell to modify equipment if required.] This document represents the test plan for these tests. The tests are designed to evaluate the performance of the RGS system as if it were installed and operated in an authentic application. Waste tank simulant material of which the chemical characteristics have already been determined will be extruded into the extractor from prepared samplers. The samplers too

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will be loaded into hot cell as if they actually contained genuine waste tank sample (Reference 6).

2.0 OBJECTIVE

This test plan has two objectives: 1) to effect a performance review to evaluate the operability and efficiency of the RGS system, and 2) to provide experience in operating the RGS system in order to develop a formal test procedure for operating the RGS system.

The performance evaluation will be conducted in a "dress rehearsal" type test environment. The RGS system will be installed in the 1E-2 hot cell as if genuine waste tank material was being characterized. All aspects of the gas extraction operation (start to finish) will be evaluated.

A Laboratory Technology (LT) procedure will be developed according to the evaluation of a user test procedure (Attached)--an untried (but modifiable) test procedure for operating the RGS system.

3.0 SCOPE

This test plan is the second test plan written to support the laboratory's efforts in the development of the RGS system. The first test plan (Reference 3) was written to test the proof-of-principle of the gas phase mass transfer operation and other (untried) concepts of the RGS system. That test plan was followed up with a results report document (Reference 4) which reported the results of those first test experiments. Below is a table which illustrates the order of the laboratory's major activities in the development of the RGS system. The star (*) indicates the current activity.

Phase I: GAS PHASE MASS TRANSPORT EVALUATION <ul style="list-style-type: none">• Operating Parameters Test Plan Laboratory System Setup Laboratory Testing• Test Results Report
Phase II: FULL SCALE PROTOTYPE EVALUATION <ul style="list-style-type: none">★ Waste Tank Simulant Test Plan Full Scale RGS Setup Full Scale RGS Testing• Test Results Report
Phase III: AUTHENTIC APPLICATION <ul style="list-style-type: none">• Authentic Application Test Plan Sample Acquisition Perform Separation and Analysis• Separation and Analysis Report

4.0 LABORATORY SETUP

Figure 4-1(a) represents the general layout of the collector side of the RGS system (outside the hot cell) and Figure 4-1(b) represents the extractor side components (inside the hot cell). This equipment will be installed in room 1E of the 222-S building in the Hanford 200 west area. Inside the hot cell will be placed the extruder device, sampler/extractor coupler, and the extractor section including the secondary volume. Just outside the hot cell, adjacent to the wall port, will be the control cabinet and carrier and back fill gas supply. The cabinet will contain the readout panels for all the pressure sensors and thermocouples of the system as well as the mercury positive displacement pump and a personal computer for data acquisition and post processing of acquired data.

An electrical conduit containing the thermocouple leads, pressure sensor leads, and extruder and extractor motor controller leads will extend from the control cabinet to the extractor section via the hot cell wall port. Also, extending from the control cabinet to the extractor section (through the wall port) will be a back fill gas line and the sample line. [An in-line depth filter will be installed in the sample line to prevent the spread of contamination to the control cabinet.] The hot cell wall port will also allow the extruder hydraulic lines and the temperature controller heat exchanger fluid lines to connect between the extractor and the heater/chiller unit. In fact, all lines which connect the devices inside the hot cell to the controlling devices outside the hot cell will connect through the hot cell wall port.

The control cabinet will sit on the floor (perhaps on wheels) adjacent to the hot cell wall port. Behind the control cabinet and mounted in a smaller cabinet will be a collection of gas cylinders containing nitrogen, argon, and helium for use as carrier gas and system back filling purposes. In this same cabinet will also be located a heater/chiller unit and a mechanical vacuum pump. Elsewhere in the 1E laboratory will be a rolling cart carrying the extruder hydraulic controller. The hydraulic lines will be made of polymeric material approximately 15 meters in length for optimal re-positioning capability.

The moving part of the mercury displacement pump (the reservoir) will be completely housed inside the control cabinet. The reservoir will be raised and lowered by rotating a ball-bearing screw shaft. The shaft will be powered and controlled by an electric stepper motor which will allow the user the ability to vary the speed and direction (up/down) of the reservoir. The control cabinet will possess a containment well at the bottom of the cabinet to contain liquid mercury should a leak in the mercury pump occur.

Figure 4-1(a): RGS System (Collector Side)

Figure 4-1(b): RGS System (Extractor Side)

5.0 TEST PROCEDURE

The user test procedure was written to provide the most comprehensive gas extraction technique that the RGS system is capable of performing. This test plan will follow as closely as possible to the user test procedure so that all aspects of the gas extraction operation can be evaluated. Where it becomes necessary to deviate from the user test procedure, remarks will be logged into a controlled laboratory log book. Since the user test procedure was written before any practical experience was gained about operating the RGS system, it is possible that the procedure might not be wholly adequate for the complete gas extraction process: certain procedure steps might require rearranging, elimination, or alteration, and perhaps new procedure steps will need to be added. The important thing is that when the user test procedure is found to provide inadequate support for the proper operation activities, careful notes will be taken to explain the inadequacy of the user test procedure and the procedure steps which were actually performed will be accurately recorded for later evaluation.

6.0 EXPECTED RESULTS

The data to be obtained during this test shall be sufficient to assess the following operability features:

- Ease and integrity of securing the sampler to the coupler.
- Ability to actuate the system valves with the manipulators.
- Ability to fully evacuate and calibrate the system volume.
- Ability to determine the system leak rate.
- Ability to extrude the sample into the extractor.
- Ability to monitor the motor function and the pressure transducers and thermocouples.
- Ability to regulate the temperature of the extractor and secondary volume.
- Ability to extract the gas from the extractor and to determine that particular point at which essentially all the extracted gas has been pumped out of the extractor leaving only the vapor pressure of the sample material.
- The ability to isolate a portion of the extracted gas in the secondary volume.
- The ability to empty the contents of the ammonia extractant solution to the secondary volume.
- The ability to collect the ammonia extractate solution into the collector.
- The ability to re-evacuate the secondary volume.
- The ability to add a specified amount of diluent to the extractor.
- The ability to disassemble the extractor for clean-up.
- The ability to reassemble the extractor for the next gas extraction process.

This test plan is also concerned with the applicability of the user test procedure. Careful notes will be maintained during these tests about:

- Necessary or recommended deviations from the procedure.

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- Problems with the procedure.
- Problems with the RGS system.
- Procedure steps or aspects which should be done differently.

After the sampler is loaded into the hot cell, this test plan should be started by beginning with step 8.1.1 and ending with 8.7.11 of the user test procedure. Careful attention should be paid to the applicability of each procedure step and careful observation of the RGS system performance should be maintained.

7.0 SAFETY

There are two significant safety concerns inherent in this test plan. The concern and precautions taken are described in the following paragraphs.

1. Liquid mercury metal will be used as the displacement fluid for the RGS mercury displacement pump. Mercury metal is considered a hazardous material because the substance possesses a substantial vapor pressure, and as vapor, is toxic to humans. However, the mercury will be contained in a closed system between a displacement cylinder and a reservoir. The reservoir will be open to atmosphere but will be located inside a metal cabinet which should reduce air from stirring the mercury saturated air inside the reservoir. Also, a containment skirt will be built into the cabinet so that if liquid mercury should accidentally spill from the pump, it would be contained in the containment skirt and easily recovered.

Prior to use, an industrial hygienist shall perform tests of the air near the mercury pump to determine if there is a significant risk of human exposure to mercury vapor.

2. Gas will be pumped from the hot cell to a collection vessel or analytical instrument outside the hot cell. The gas could contain radioactive particles so an in-line depth filter shall be installed on the sample line connecting the extractor to the pump (situated outside the hot cell). The filter will stop the particulates before reaching the pump, collection vessel or analyzer. The filter will be changed as needed.

8.0 REFERENCES

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ATTACHMENT

User Test: Retained Gas Sampler System Operation

ANALYTICAL LABORATORIES

USER TEST

1.0 SUMMARY

This user test is used to extract and separate retained gas from a waste tank core sample for qualitative and quantitative analysis. The waste tank core sample is delivered to the laboratory in a specially designed waste tank core sampler which has been fabricated to contain the gas phase component of waste tank sample material as well as the bulk material. The sampler is loaded into the 1E-2 hot cell according to Procedure LO-160-101, *Core Sample Receipt and Preparation*.

Retained Gas describes the gas phase component of waste tank core samples. The gas is associated with the bulk of the sample as 1) free and unbound, as vapor, 2) physically bound, as small gas bubbles trapped within the crystal interstices of the bulk sample, or 3) chemically bound, as dissolved gas in the interstitial liquid of the bulk sample such as ammonium hydroxide. Three different extraction operations will be used to extract each of the three types of gas from the bulk of the sample.

In order to accomplish the extraction and separation of retained gas from the sampler, a Retained Gas Sampler (RGS) system will be used. This system has two main sections; the extractor section which is located within the hot cell and the separator section located outside the hot cell. The extractor section includes three main components: an extractor vessel; a sampler/extractor vessel coupler; and an ammonia extractor. The separator section includes two main components: a liquid mercury positive displacement pump and a collector vessel. A gas analyzer may be connected to the collector vessel for immediate analysis.

An extruder device is used to extrude the sample from the sampler into the extractor via the coupler. This is the same type of extruder device used for current universal sampler core extrusions (LO-160-103) except that the sample is extruded directly into the extractor instead of onto a tray. Also, the sample is extruded with a single stroke of the hydraulic pump rather than in three or four successive strokes.

The extractor section is connected to the separator section through a port in the hot cell wall. Mass transfer of gas from the extractor to the collector involves the use of an in-line filter installed on the transfer tube to prevent the release of radioactive particles from exiting the hot cell.

Fitted on the transfer tube between the mercury displacement pump and the in-line filter is a valved port. This port is for attaching a sample vessel for internal standardization and to check the system's performance. The system may also be opened to atmosphere for venting and calibration against normal atmospheric pressure.

2.0 APPLICATIONS

This user test applies to activities involved in the use of the Retained Gas Sampler (RGS) system for the purpose of identifying and quantifying retained gas.

3.0 LIMITATIONS

An RGS System Work Plan is required to perform this procedure. Any departures from the Work Plan should be documented in controlled laboratory notebooks by the cognizant scientist. The cognizant scientist needs to be available during the operation for critical decisions should unexpected conditions arise.

The RGS system should not be used if any of the three manipulators are unable to function properly.

Gases collected in the collector vessel should be non-radioactive and should not contain any radioactive particulates.

Transfer of material from the extractor to the collector vessel should be limited to gas phase substances only.

4.0 QUALITY CONTROL PROTOCOL

This user test assumes that all pressure and temperature measurement devices used in this procedure are functioning properly and are already calibrated.

The RGS system Work Plan should be specific enough to verify strict compliance with the customer's quality assurance requirements and gives detailed instructions for achieving data quality objectives.

A controlled laboratory notebook should be used to record all operations concerning the RGS system.

5.0 SAFETY

Persons performing this procedure should review the applicable radiological work permit.

Persons performing this procedure will be trained in the use of remote manipulators and should verify that all three manipulators are in good working condition prior to operation.

Persons performing this procedure should be familiar with the function and operation of the extruder and should verify the device is in good working condition prior to operation.

Persons performing this procedure should be familiar with the function and operation of the RGS system and should verify each component is in good working condition prior to operation.

6.0 REAGENTS

Ammonia extractant solution (0.02 M H_2SO_4 acid solution)

Tank waste material diluent solution (1.0 M sodium hydroxide)

Water from Laboratory sanitary water supply

7.0 EQUIPMENT

1E-2 Hot Cell

- Three (3) EHD remote manipulators

Extruder Device

Retained Gas Sampler System

- Retained Gas Sampler
- Separator Section (Figure 11-1)
- Extractor Section (Figure 11-2)
- Data Logger

Collector vessel(s)

Gas Analyzer

8.0 PROCEDURE STEPS

8.1 Authorization and Preliminary Setup

8.1.1 Notify the Building Operations Manager about plans for disposing waste tank material down hot cell drains.

8.1.2 Inspect each manipulator and verify that each one is functioning properly and moves with normal effort.

8.1.3 Inspect the extruder device and verify that it is functioning properly and moves with normal effort.

PROCEDURE STEPS (Continued)

- 8.1.4 Inspect the RGS system and verify that all its components are functioning properly.
- 8.1.5 Remove the quadra latch and mouse trap from the sampler.
- 8.1.6 Slide the spacer and clamping collar over the threaded end of the sampler.
- 8.1.7 Install the universal press nut on the threaded side of the sampler.
- 8.1.8 Mount the sampler on the extruder.
- 8.1.9 Couple the sampler to the extractor.
- 8.1.10 Connect the ammonia extractant and extractate vessels to the secondary volume of the extractor section.
- 8.1.11 Fill the diluent reservoir with sample diluent, cover.

NOTE: Preliminary setup complete.

8.2 System Vacuum Integrity Check.

NOTE: Figure 11-1 illustrates the separator section of the RGS system and includes an identifying letter for each valve. Figure 11-2 illustrates the extractor section of the RGS system and also includes a letter identifier for each valve (different from Figure 11-1).

- 8.2.1 Power-up the RGS system (thermocouples, pressure transducers, display panels, computers, and data acquisition devices).
- 8.2.2 Start data acquisition (pressure, temperature).
- 8.2.3 Turn all valves, on the RGS system, to their fully-closed position.
- 8.2.4 Verify the mercury reservoir is positioned at its lowest level.
- 8.2.5 Verify valve Q, on the extractor, is closed.
- 8.2.6 Turn on the mechanical vacuum pump.
- 8.2.7 Open valves M, K, H, J, F, E, B, A on the separator section.
- 8.2.8 Open valves R, U, T on the extractor section.

PROCEDURE STEPS (Continued)

- 8.2.9 Turn valve G, on the mercury displacement pump, to the collector side.
- 8.2.10 Evacuate the system to less than 1 Pascal (absolute) using the mechanical vacuum pump.
- 8.2.11 Close valve K to isolate the mechanical vacuum pump from the separator section.
- 8.2.12 Turn off the mechanical vacuum pump.
- 8.2.13 Mark this time in the log book as "Start of RGS System Vacuum Leak Check."
- 8.2.14 Monitor the RGS system for approximately one-half hour.
- 8.2.15 Mark this time in the log book as "End of RGS System Vacuum Leak Check."
- 8.2.16 Determine the RGS system leak rate in units of mmole/sec (see CALCULATIONS) and record in the log book the determined leak rate.

NOTE: If the RGS system is found to possess a leak rate greater than 0.01 μ mole per second, the procedure should stop here and the leaks tended to. If, on the same day, the RGS system leaks have been remedied, continue the procedure starting with Step 8.2.1. If the procedure must be restarted on a later day, restart the procedure beginning with Step 8.1.1.

8.3 Free Gas Extraction and Separation Operation

NOTES: The procedure assumes that the RGS system has been maintained in the power up mode since the beginning of Section 8.2.

The data acquisition routine may be stopped at this point and the acquired data saved before continuing. The important thing to make sure of is that data acquisition continues uninterrupted from start to finish in this section (8.3).

- 8.3.1 Turn all valves, on the RGS system, to their fully-closed position.
- 8.3.2 Verify the mercury reservoir is positioned at its lowest level.
- 8.3.3 Verify valve Q, on the extractor section, is closed.
- 8.3.4 Turn on the mechanical vacuum pump.
- 8.3.5 Open valves M, K, J, H, F, E, B, A on the separator section.

PROCEDURE STEPS (Continued)

- 8.3.6 Open valves R, U, T on the extractor section.
 - 8.3.7 Turn valve G, on the mercury displacement pump, to the collector side.
 - 8.3.8 Raise the mercury, in the pump displacement volume, to the sight glass by controlling both the height of the mercury reservoir and valve G.
 - 8.3.9 Verify valve G is in the fully closed position.
 - 8.3.10 Lower the mercury reservoir to the lowest position.
 - 8.3.11 Evacuate the system to less than 1 Pascal (absolute) using the mechanical vacuum pump.
 - 8.3.12 Close valve B to isolate the extractor section from the separator section.
 - 8.3.13 Mark this time in the log book as "RGS System Preparatory Evacuation for Waste Tank Sample Introduction."
- NOTE: At this point in the procedure, the whole interior of the RGS system has been evacuated and is ready for waste tank sample introduction.
- 8.3.14 Close valves R and U on the extractor section to isolate the secondary volume from the extractor main volume.
 - 8.3.15 Close valve K to isolate the mechanical vacuum pump from the separator section.
 - 8.3.16 Turn off the mechanical vacuum pump.
 - 8.3.17 Verify valve S, on the extractor section, is closed.
 - 8.3.18 Turn on the extractor mixer motor.
 - 8.3.19 Open the sampler valve (valve X).
 - 8.3.20 Extrude the sample material into the extractor.
 - 8.3.21 Mark this time in the log book as "Waste Tank Sample Extrusion Operation Complete."
 - 8.3.22 Operate the heater/chiller unit to bring the waste tank sample to the desired temperature.

PROCEDURE STEPS (Continued)

8.3.23 Monitor the RGS system until the pressure and temperature of the extractor section equilibrate.

8.3.24 Mark this time in the log book as "Waste Tank Sample Initial Temperature Conditioning Operation Complete."

8.3.25 Open valve S on the extractor section to expose the secondary volume to the extractor main volume.

8.3.26 Monitor the system until the pressure and temperature of the extractor section equilibrate.

8.3.27 Close valve S, on the extractor section, to isolate the secondary volume from the extractor main volume.

NOTE: At this point all the free gas associated with the waste tank sample has accumulated in the vapor space of the extractor section. Some of this gas has been isolated in the secondary volume of the extractor section for ammonia determination.

8.3.28 Open valve Q, on the extractor section, to empty the contents of the ammonia extractant vessel into the secondary volume.

8.3.29 Close valve Q to isolate the secondary volume, of the extractor section, from the ammonia extractant vessel.

NOTE: At this point the ammonia extractant solution has been added to the secondary volume of the extractor section to absorb ammonia from the gas phase. The ammonia absorption process will continue undisturbed while the remainder of the gas in the extractor main volume is pumped to the collector vessel.

8.3.30 Open valve B to connect the extractor section to the separator section.

8.3.31 Close valve F, of the separator section, to isolate the collector side from the extractor side of the mercury displacement pump.

NOTE: The next seven steps in this procedure are the principle steps for operating the mercury positive displacement pump.

8.3.32 Turn valve G, on the mercury displacement pump, to the extractor side.

8.3.33 Monitor the system until the pressure and temperature on the extractor side equilibrate.

8.3.34 Turn valve G, on the mercury displacement pump, to the collector side.

PROCEDURE STEPS (Continued)

CAUTION

The pressure accumulating in the collector vessel must be closely observed. When the pressure reaches 100 kPa, the pumping operation should stop and the collector vessel should be emptied or exchanged.

8.3.35 Raise the mercury, in the pump displacement volume, to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.3.36 Verify valve G is in the fully-closed position.

8.3.37 Lower the mercury reservoir to the lowest position.

8.3.38 Wait until the mercury settles to the lowest level in the pump displacement volume.

8.3.39 Repeat Steps 8.3.32 through 8.3.39 until the only vapor subjecting pressure to the extractor side of the mercury displacement pump is the sample vapor pressure itself.

8.3.40 Turn valve G, on the mercury displacement pump, to the extractor side.

8.3.41 Wait until pressure and temperature on the extractor side equilibrate.

8.3.42 Turn valve G, on the mercury displacement pump, to the collector side.

8.3.43 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.3.44 Make sure valve G is in the fully-closed position.

8.3.45 Lower the mercury reservoir to the lowest position.

8.3.46 Mark this time in the log book as "Free Gas Extraction and Separation Operation Complete."

8.3.47 Close valves H and J to isolate the collector vessel.

NOTE: At this point all the free gas from the waste tank sample has been collected either in the collector vessel (isolated by valve J) or in the secondary volume of the extractor section.

8.3.48 Open valve U to expose the ammonia extractate vessel to the secondary volume of the extractor section.

PROCEDURE STEPS (Continued)

8.3.49 Close valve U to isolate the ammonia extractate vessel from the secondary volume of the extractor section.

NOTE: The free gas extraction and separation operation is complete. The collector vessel (at valve H) may be removed and replaced with another vessel if so desired. The ammonia extractate vessel may also be removed for eventual load out of the hot cell. Removal of the ammonia extractate vessel will compromise the vacuum in the secondary volume of the extractor section.

8.4 Physically Bound Gas Extraction and Separation Operation

NOTES: The procedure assumes the RGS system has been maintained in the power up mode since the beginning of Section 8.2.

The procedure assumes that the extractor mixer motor has run continuously since Step 8.3.18 and that the waste tank sample temperature has remained constant since Step 8.3.24.

The data acquisition routine may be stopped at this point and the acquired data saved before continuing. The important thing to make sure of is that data acquisition continues uninterrupted from start to finish in this Section (8.4).

- 8.4.1 Turn all valves of the RGS system to the fully-closed position.
- 8.4.2 Verify the mercury reservoir is positioned at its lowest level.
- 8.4.3 Replace the ammonia extractant and extractate vessels on the extractor section.
- 8.4.4 Make sure valve Q, on the extractor section, is closed.
- 8.4.5 Turn on the mechanical vacuum pump.
- 8.4.6 Open valves M, K, J, H, F, E, B, A on the separator section.
- 8.4.7 Open valves R, U, T on the extractor section.
- 8.4.8 Turn valve G, on the mercury displacement pump, to the collector side.
- 8.4.9 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.4.10 Verify valve G is in the fully-closed position.

PROCEDURE STEPS (Continued)

- 8.4.11 Lower the mercury reservoir to the lowest position.
- 8.4.12 Evacuate the system to 4000 Pa (absolute) using the mechanical vacuum pump.
- 8.4.13 Close valve K to isolate the mechanical vacuum pump from the separator section.
- 8.4.14 Turn off the mechanical vacuum pump.
- 8.4.15 Turn valve G, on the mercury displacement pump, to the extractor side.
- 8.4.16 Monitor the system until the pressure and temperature on the extractor side equilibrate.
- 8.4.17 Turn valve G, on the mercury displacement pump, to the collector side.

CAUTION

The pressure accumulating in the collector vessel must be closely observed. When the pressure reaches 100 kPa, the pumping operation should stop and the collector vessel should be emptied or exchanged.

- 8.4.18 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.4.19 Verify valve G is in the fully-closed position.
- 8.4.20 Lower the mercury reservoir to the lowest position.
- 8.4.21 Wait until the mercury settles to the lowest level in the pump displacement volume.
- 8.4.22 Repeat Steps 8.4.15 through 8.4.22 until the only vapor subjecting pressure to the extractor side of the mercury displacement pump is the sample vapor pressure itself.
- 8.4.23 Turn valve G, on the mercury displacement pump, to the extractor side.
- 8.4.24 Wait until pressure and temperature, on the extractor side, equilibrate.
- 8.4.25 Turn valve G, on the mercury displacement pump, to the collector side.

PROCEDURE STEPS (Continued)

8.4.26 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.4.27 Verify valve G is in the fully-closed position.

8.4.28 Lower the mercury reservoir to the lowest position.

8.4.29 Close valve B to isolate the extractor section from the separator section.

8.4.30 Mark this time in the log book as "RGS System Preparatory Evacuation for Physically Bound Gas Extraction and Separation."

NOTE: At this point in the procedure, the whole interior of the RGS system has been evacuated (to the vapor pressure of the waste tank sample) and is ready for waste tank sample heating to liberate the physically bound gas.

8.4.31 Close valves R and U on the extractor section to isolate the secondary volume from the extractor main volume.

8.4.32 Verify valve S, on the extractor section, is closed.

8.4.33 Adjust the heater/chiller unit to bring the waste tank sample to the desired temperature.

8.4.34 Monitor RGS system until pressure and temperature in the extractor section equilibrate.

8.4.35 Mark this time in the log book as "Waste Tank Sample Heating Operation Complete."

8.4.36 Open valve S, on the extractor section, to expose the secondary volume to the extractor main volume.

8.4.37 Monitor the system until the pressure and temperature of the extractor section equilibrate.

8.4.38 Close valve S on the extractor section to isolate the secondary volume from the extractor main volume.

NOTE: At this point the physically bound gas associated with the waste tank sample has been liberated and accumulated in the vapor space of the extractor section. Some of this gas has been isolated in the secondary volume of the extractor section for ammonia determination.

PROCEDURE STEPS (Continued)

8.4.39 Open valve Q on the extractor section to empty the contents of the ammonia extractant vessel into the secondary volume of the extractor section.

8.4.40 Close valve Q to isolate the secondary volume of the extractor section from the ammonia extractant vessel.

NOTE: At this point the ammonia extractant solution has been added to the secondary volume of the extractor section to absorb ammonia from the gas phase. The ammonia absorption process will continue undisturbed while the remainder of the gas in the extractor main volume is pumped to the collector vessel.

8.4.41 Open valve B to connect the extractor section to the separator section.

8.4.42 Close valve F, of the separator section, to isolate the collector side from the extractor side of the mercury displacement pump.

8.4.43 Turn valve G, on the mercury displacement pump, to the extractor side.

8.4.44 Monitor the system until the pressure and temperature on the extractor side equilibrate.

8.4.45 Turn valve G on the mercury displacement pump to the collector side.

CAUTION

The pressure accumulating in the collector vessel must be closely observed. When the pressure reaches 100 kPa, the pumping operation should stop and the collector vessel should be emptied or exchanged.

8.4.46 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.4.47 Verify valve G is in the fully-closed position.

8.4.48 Lower the mercury reservoir to the lowest position.

8.4.49 Wait until the mercury settles to the lowest level in the pump displacement volume.

8.4.50 Repeat steps 8.4.43 through 8.4.50 until the only vapor subjecting pressure to the extractor side of the mercury displacement pump is the sample vapor pressure itself.

PROCEDURE STEPS (Continued)

- 8.4.51 Turn valve G, on the mercury displacement pump, to the extractor side.
- 8.4.52 Wait until pressure and temperature on the extractor side equilibrate.
- 8.4.53 Turn valve G, on the mercury displacement pump, to the collector side.
- 8.4.54 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.4.55 Make sure valve G is in the fully closed position.
- 8.4.56 Lower the mercury reservoir to the lowest position.
- 8.4.57 Mark this time in the log book as "Physically Bound Gas Extraction and Separation Operation Complete."
- 8.4.58 Close valves H and J to isolate the collector vessel.

NOTE: At this point all the liberated gas from the waste tank sample has been collected either in the collector vessel (isolated by valve J) or in the secondary volume of the extractor section.

8.4.59 Open valve U to expose the ammonia extractate vessel to the secondary volume of the extractor section.

8.4.60 Close valve U to isolate the ammonia extractate vessel from the secondary volume of the extractor section.

NOTE: The physically bound gas extraction and separation operation is complete. The collector vessel (at valve H) may be removed and replaced with another vessel if so desired. The ammonia extractate vessel may also be removed for eventual load out of the hot cell. Removal of the ammonia extractate vessel will compromise the vacuum in the secondary volume of the extractor section.

8.5 Bulk Sample Volume Determination

NOTES: The procedure assumes that the RGS system has been maintained in the power-up mode since the beginning of Section 8.2.

The procedure assumes that the extractor mixer motor has run continuously since step 8.3.18 and that the waste tank sample temperature has remained constant since Step 8.3.24.

The data acquisition routine may be stopped at this point and the acquired data saved before continuing. The important thing to

PROCEDURE STEPS (Continued)

make sure of is that data acquisition continues uninterrupted from start to finish in this Section (8.4).

- 8.5.1 Connect the ammonia extractant and extractate vessels to the secondary volume of the extractor section.
- 8.5.2 Turn all valves to their fully closed position.
- 8.5.3 Verify the mercury reservoir is positioned at its lowest level.
- 8.5.4 Verify valve Q, on the extractor section, is closed.
- 8.5.5 Turn on the mechanical vacuum pump.
- 8.5.6 Open valves M, K, H, J, F, E, B, A on the separator section.
- 8.5.7 Open valves R, S on the extractor section.
- 8.5.8 Turn valve G on the mercury displacement pump to the collector side.
- 8.5.9 Evacuate the system to 4000 Pa using the mechanical vacuum pump.
- 8.5.10 Mark this time in the log book as "Extractor Pressure for Waste Tank Sample Volume Determination."
- 8.5.11 Close valve S, on the extractor section.
- 8.5.12 Turn off mixer motor.
- 8.5.13 Regulate the output pressure of the inert carrier gas to 100 kPa absolute (0 kPa gauge).
- 8.5.14 Open valves Z and R to fill the secondary volume of the extractor section with 100 kPa (abs.) of inert carrier gas.
- 8.5.15 Close valves Z and R on the extractor section.
- 8.5.16 Mark this time in the log book as "Secondary Volume Pressure for Waste Tank Sample Volume Determination."
- 8.5.17 Open valve S to connect the secondary volume to the extractor main volume.
- 8.5.18 Monitor the RGS system until pressure and temperature of the extractor side equilibrate.
- 8.5.19 Mark this time in the log book as "Resultant Pressure from Combined Extractor Main and Secondary Volumes."

PROCEDURE STEPS (Continued)

NOTES: Waste tank sample volume determination operation complete.
This section (8.5) may be repeated to gain replicate results.

8.6 Chemically Bound Gas Extraction and Separation

NOTES: The procedure assumes that the RGS system has been maintained in the power-up mode since the beginning of Section 8.2.

The procedure assumes that the extractor mixer motor has been turned off since Step 8.5.12 and that the waste tank sample temperature has remained constant since Step 8.3.24.

The data acquisition routine may be stopped at this point and the acquired data saved before continuing. The important thing to make sure of is that data acquisition continues uninterrupted from start to finish in this Section (8.6).

- 8.6.1 Turn all valves to the fully-closed position.
- 8.6.2 Verify the mercury reservoir is positioned at its lowest level.
- 8.6.3 Verify the ammonia extractant and extractate vessels, on the extractor section, are in place for this operation.
- 8.6.4 Verify valve Q, on the extractor section, is closed.
- 8.6.5 Turn on extractor mixer motor.
- 8.6.6 Turn on the mechanical vacuum pump.
- 8.6.7 Open valves M, K, J, H, F, E, B, A on the separator section.
- 8.6.8 Open valves R, U, T on the extractor section.
- 8.6.9 Turn valve G, on the mercury displacement pump, to the collector side.
- 8.6.10 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.6.11 Verify valve G is in the fully-closed position.
- 8.6.12 Lower the mercury reservoir to the lowest position.
- 8.6.13 Evacuate the system to 4000 Pa (absolute) using the mechanical vacuum pump.

PROCEDURE STEPS (Continued)

- 8.6.14 Close valve K to isolate the mechanical vacuum pump from the separator section.
- 8.6.15 Turn off the mechanical vacuum pump.
- 8.6.16 Turn valve G, on the mercury displacement pump, to the extractor side.
- 8.6.17 Monitor the system until the pressure and temperature on the extractor side equilibrate.
- 8.6.18 Turn valve G, on the mercury displacement pump, to the collector side.

CAUTION

The pressure accumulating in the collector vessel must be closely observed. When the pressure reaches 100 kPa, the pumping operation should stop and the collector vessel should be emptied or exchanged.

- 8.6.19 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.6.20 Verify valve G is in the fully-closed position.
- 8.6.21 Lower the mercury reservoir to the lowest position.
- 8.6.22 Wait until the mercury settles to the lowest level in the pump displacement volume.
- 8.6.23 Repeat Steps 8.6.16 through 8.6.23 until the only vapor subjecting pressure to the extractor side of the mercury displacement pump is the waste tank sample vapor pressure itself.
- 8.6.24 Turn valve G, on the mercury displacement pump, to the extractor side.
- 8.6.25 Wait until pressure and temperature on the extractor side equilibrate.
- 8.6.26 Turn valve G, on the mercury displacement pump, to the collector side.
- 8.6.27 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.
- 8.6.28 Make sure valve G is in the fully closed position.
- 8.6.29 Lower the mercury reservoir to the lowest position.

PROCEDURE STEPS (Continued)

8.6.30 Close valve B to isolate the extractor section from the separator section.

8.6.31 Mark this time in the log book as "RGS System Preparatory Evacuation for Chemically Bound Gas Extraction and Separation."

NOTE: At this point in the procedure, the whole interior of the RGS system has been evacuated (to the vapor pressure of the waste tank material) and is ready for the addition of diluent to liberate chemically bound gas.

8.6.32 Close valves R and U on the extractor section to isolate the secondary volume from the extractor section.

8.6.33 Verify valve S, on the extractor section, is closed.

8.6.34 Open (carefully) valve W to let the desired amount of diluent into the extractor.

8.6.35 Monitor RGS system until pressure and temperature in the extractor section equilibrate.

8.6.36 Mark this time in the log book as "Diluent Addition to Waste Tank Sample Complete."

8.6.37 Open valve S, on the extractor section, to expose the secondary volume to the extractor main volume.

8.6.38 Monitor the system until the pressure and temperature of the extractor section equilibrate.

8.6.39 Close valve S on the extractor section to isolate the secondary volume from the extractor main volume.

NOTE: At this point the chemically bound gas associated with the waste tank sample has been liberated and accumulated in the vapor space of the extractor section. Some of this gas has been isolated in the secondary volume of the extractor section for ammonia determination.

8.6.40 Open valve Q, on the extractor section, to empty the contents of the ammonia extractant vessel into the secondary volume of the extractor section.

8.6.41 Close valve Q to isolate the secondary volume, of the extractor section, from the ammonia extractant vessel.

PROCEDURE STEPS (Continued)

NOTE: At this point the ammonia extractant solution has been added to the secondary volume of the extractor section to absorb ammonia from the gas phase. The ammonia absorption process will continue undisturbed while the remainder of the gas in the extractor main volume is pumped to the collector vessel.

8.6.42 Open valve B to connect the extractor section to the separator section.

8.6.43 Close valve F, of the separator section, to isolate the collector side from the extractor side of the mercury displacement pump.

8.6.44 Turn valve G, on the mercury displacement pump, to the extractor side.

8.6.45 Monitor the system until the pressure and temperature on the extractor side equilibrate.

8.6.46 Turn valve G, on the mercury displacement pump, to the collector side.

CAUTION

The pressure accumulating in the collector vessel must be closely observed. When the pressure reaches 100 kPa, the pumping operation should stop and the collector vessel should be emptied or exchanged.

8.6.47 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.6.48 Verify valve G is in the fully-closed position.

8.6.49 Lower the mercury reservoir to the lowest position.

8.6.50 Wait until the mercury settles to the lowest level in the pump displacement volume.

8.6.51 Repeat Steps 8.6.44 through 8.6.51 until the only vapor subjecting pressure to the extractor side of the mercury displacement pump is the waste tank sample vapor pressure itself.

8.6.52 Turn valve G, on the mercury displacement pump, to the extractor side.

8.6.53 Wait until pressure and temperature on the extractor side equilibrate.

8.6.54 Turn valve G, on the mercury displacement pump, to the collector side.

PROCEDURE STEPS (Continued)

8.6.55 Raise the mercury in the pump displacement volume to the sight glass by controlling both the height of the mercury reservoir and valve G.

8.6.56 Verify valve G is in the fully-closed position.

8.6.57 Lower the mercury reservoir to the lowest position.

8.6.58 Mark this time in the log book as "Chemically Bound Gas Extraction and Separation Operation Complete."

8.6.59 Close valves H and J to isolate the collector vessel.

NOTE: At this point all the liberated gas from the waste tank sample has been collected either in the collector vessel (isolated by valve J) or in the secondary volume of the extractor section.

8.6.60 Open valve U to expose the ammonia extractate vessel to the secondary volume of the extractor section.

8.6.61 Close valve U to isolate the ammonia extractate vessel from the secondary volume of the extractor section.

NOTE: The chemically bound gas extraction and separation operation is complete. The collector vessel (at valve H) may be removed and replaced with another vessel if so desired. The ammonia extractate vessel may also be removed for eventual load out of the hot cell. Removal of the ammonia extractate vessel will compromise the vacuum in the secondary volume of the extractor section.

8.7 Extractor Section Clean-up

8.7.1 Close valve A.

8.7.2 Remove the ammonia extractant and extractate vessels from the secondary volume of the extractor section.

8.7.3 Position one manipulator under the bottom flange and lock in place.

8.7.4 Pull out the two clamp handles on either side of the extractor main body.

NOTE: The mixer blade comes out of the extractor body when the bottom flange is removed.

8.7.5 Gently lower the bottom flange from the extractor main body.

PROCEDURE STEPS (Continued)

NOTE: Only laboratory water should be used to clean out the extractor main volume. Care should be taken to prevent spraying (excessively) the exterior of the extractor section.

8.7.6 Spray water up into the extractor main volume to rinse out as much waste tank material as possible.

8.7.7 Spray water on the mixer blade and bottom flange to rinse away waste tank material from these parts.

NOTE: Care should be taken to prevent the spread of the waste tank material throughout the interior of the hot cell. It is important to wash the waste tank material down the hot cell floor drain as quickly and effortlessly as possible to avoid excessively contaminating the interior of the hot cell.

8.7.8 Rinse the waste tank material down the hot cell drain.

NOTE: While cleaning the bottom flange it is important that the gasket seal be thoroughly cleaned and not damaged in any way. If damage to the gasket is suspected, it should be replaced.

8.7.9 Replace the mixer blade in position on the bottom flange.

8.7.10 Replace the bottom flange on the extractor main body--using the aligning pins to guide the flange into the proper position.

8.7.11 Push in the clamp handles to secure the bottom flange to the extractor main body.

NOTE: Extractor section clean-up operation complete.

9.0 CALCULATIONS

Leak rate determination.

$$L = S \times \frac{V}{RT} \quad (\text{Eq. 1})$$

Where: L = Leak rate (mmoles/sec).

S = Linear least squares slope of the pressure/time curve expressed in units of Pascal/second (Pa/sec).

V = System volume expressed in milliliters (mL).

R = Gas constant, 8.314 (J/K·mol).

T = System temperature (K).

10.0 CALIBRATIONS

Instruments used on the RGS system should already be officially calibrated prior to use and shall be clearly labeled with information regarding the usable range of the instrument and the date with which it is due for recalibration.

11.0 FIGURES

Figure 11-1: RGSS Separator Section

Figure 11-2: RGSS Extractor Section