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Retained Gas Sampler Calibration and Simulant Tests

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Richland, WA 99352

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Abstract: This test plan provides a method for calibration of the retained gas sampler (RGS) for ammonia gas analysis. Simulant solutions of ammonium hydroxide at known concentrations will be diluted with isotopically labeled 0.04 M ammonium hydroxide solution. Sea sand solids will also be mixed with ammonium hydroxide solution and diluent to determine the accuracy of the system for ammonia gas analysis.

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RETAINED GAS SAMPLER CALIBRATION AND SIMULANT TESTS

1.0 INTRODUCTION

This test plan describes a method for testing and calibrating ammonia measurements made by the system used for extraction and separation of retained gas from waste tank core samples. Under normal operation, the waste tank core segment sample is delivered to the laboratory in a special gas tight waste tank core sampler that contains the gas phase component of waste tank sample material along with the bulk material. Normally the sampler is loaded into the 1E-2 hot cell according to procedure LO-160-101, Core Segment Load-in and Load-out of 1E-2 Hot Cell. The sampler is then coupled to the extractor of the Retained Gas Sampler (RGS) system in a way that ensures vacuum integrity. The extractor is evacuated. Vapors contained inside the core sampler are extruded into the extractor with the bulk sample material then are pumped, using a liquid mercury positive displacement pump, to collection canisters located outside the hot cell. A high-efficiency in-line depth filter prevents the transfer of radioactive particles to the collection canisters. The collection canisters can then be removed from the system for analysis of the gases.

As part of this test plan the RGS system will be calibrated using a 0.04 M concentration of isotopically labeled $^{15}\text{NH}_4\text{OH}$ solution.

2.0 OBJECTIVES

This test plan describes the calibration and sampling requirements for use of the retained gas sampler (RGS) equipment for ammonia gas extraction from solid and liquid samples in the 1E-2 hot cell in the 222-S facility. Pre-analyzed solutions of ammonium hydroxide will be run through vapor extraction tests to determine the accuracy of ammonia measurements made for a water matrix in the retained gas-sampling unit. Tests will then be expanded to include analysis of NH_3 in a more concentrated ammonium hydroxide solution or one of identical concentration as the previous tests containing approximately 40 volume % solids.

3.0 QUALITY ASSURANCE

Data obtained during these tests include temperature and pressure readings. Log sheets on which data are recorded are pasted into a controlled laboratory notebook. These sheets will be sent via facsimile to PNNL for data interpretation and use.

Temperature measurements are obtained through use of K-type thermocouples. Calibration of the temperature sensing capability of the RGS system is not routinely performed; rather several readings are acquired simultaneously from different locations of the RGS system for redundant verification.

Four of the five pressure measurements are made using instruments that are calibrated annually, the fifth (non-calibrated) pressure measuring instrument is used for convenient indication only. Calibrations are performed on pressure transducers by the manufacturer at the factory. These are maintained on a preventative maintenance schedule.

4.0 SAFETY

The RGS extraction system may involve using compressed gases that are used to purge the system, periodically. Although these gases are regulated to low and easily manageable pressures, care should be exercised to ensure the system does not exceed 133 kPa (4.6 psig). This ensures sensitive pressure and temperature sensors will not be damaged. To further ensure the instruments are protected, valves should be closed when the system is not in use.

The RGS extraction system uses liquid mercury in the displacement pump. Although the mercury is safely contained within the RGS system, care should be exercised when performing plumbing operations on the system because beads of mercury may be encountered when breaching some system lines. If a spill of metallic mercury does occur, the Shift Operations Manager (SOM) must be notified after personnel in the area are warned of the spill and the spill is isolated.

In the event that the system lines and plumbing should be breached to effect repair or replacement of instruments or fittings, an HPT must be present to conduct surveys to monitor and control the spread of radioactive contamination.

5.0 WASTE GENERATION AND HANDLING

During these tests some solid waste may be generated from the use of glass silica beads with solutions being tested. These wastes will be packed and loaded out of the hot cell in accordance with LO-100-151, *Segregate and Manage Solid Laboratory Wastes*, section 3.4, 6.9 and 6.10 (see appendix A) and LO-160-101, *IE-2 Hot Cell Airlock Operations*, sections 6.1 through 6.6 as necessary (see appendix B).

Contaminated rinse water, generated during preparation and post processing wash downs, is released to the hot cell drains as per LO-100-151 housekeeping procedures (see appendix A). The building operations manager should be notified prior to wash down activities or anytime water is used in the hot cell.

6.0 PROCEDURE

6.1 Equipment Needed

Gas Sample Collection Canisters (dwg. # H-2-821611)
 Controlled Laboratory Notebook (HNF-40-2)
 Retained Gas Sampler System (dwg. #H-2-821609)
 Manifold System Control Cabinet and Contents (dwg. #H-2-85291)
 Turbo-molecular Vacuum Pump

Equipment Needed (cont.):

Rotary Vane Vacuum Pump

Temperature Controlled Water Bath (Heater/Chiller)

Assorted Tools and Mock Sampler for operation of the extruder

Sample vials, 15 mL with septum lids

Polybottles (1L)

6.2 Phase I Testing

Note – All system equilibration and system leak checks should be performed and documented prior to sample introduction according to the protocol established in LT-160-101 (appendix C) sections 10.1.2 and 10.2.2.

6.2.1 Solution Preparation and Analysis

1. Make up 500 ml 0.04 M $^{15}\text{NH}_4\text{OH}$ solution.
2. Shake solution prior to dispensing.
3. Fill a 15-ml vial with a septum lid to the top with solution.
4. Submit the 15-ml vial for ammonia analysis by ion selective electrode (ISE).

6.2.2 Introduction of Solution to Extractor and Equilibration

1. Ensure canisters are attached to ports J1 and J2 and that the canister valves are open for evacuation.
2. Ensure the extruder is isolated from the evacuated system by closing valve T.
3. Dispense approximately 100 ml diluent through valve W2 and then ensure valve W2 is closed.
4. Introduce 300 ml of the $^{15}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
5. Assure the system temperature is at 10 to 12°C.

Note – The motor speed of the extractor is 60 rpm when the controller dial on the RGS console is dialed to 999.

6. Begin mixing solution by turning on the mixing motor and adjusting the motor speed to 60 rpm.

- When system temperature and pressure stabilizes, record the date, time, event, temperature and pressure on the RGS processing worksheet and post in the controlled notebook.

Note – Temperature is maintained at 10 °C and mixing continues for 24 hours of equilibration.

- Allow the solution to equilibrate in the mixing chamber for 24 hours.

6.2.3 Pump System for Sample (24 hours only)

- Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
- Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

- Ensure system pressure is less than 0.01kPa.
- Adjust pressure indicator zero offsets as necessary.
- Close valve K1.
- Open valve T.
- When system temperature and pressure stabilizes, record date, time, event, temperature and pressure in the controlled notebook.
- Close valve F and ensure valve G is turned to the collector side.
- Perform the following steps 12 times initialing each step as performed:

Step	1	2	3	4	5	6	7	8	9	10	11	12
a. Raise the mercury reservoir above the 0-point.												
b. Open valve U.												
c. Control flow of mercury with G, sight glass and mercury reservoir level.												
d. Lower mercury reservoir to bottom of travel.												
e. Record the system temperature and pressure in notebook.												

Step	1	2	3	4	5	6	7	8	9	10	11	12
f. Turn valve G to the extractor side. and wait until mercury completely evacuates displacement bulb.												
g. Close valve U.												
h. Turn valve G to collector side.												
i. Repeat steps until completed.												

10. Raise the mercury reservoir above 0-point.
11. Open valve U and control the flow of mercury to the sight glass with valve G and height of the reservoir.
12. Lower the mercury reservoir to bottom of travel.
13. Record the temperature and pressure in the controlled notebook.
14. Close the valves on the gas sample canisters at port J1 and J2 and H1 and H2.
15. Record the vapor sample canister ID, temperature and pressure in the notebook.
16. Stop data acquisition, close file, and copy file to a floppy disk.

6.2.4 Collect Samples and Data

1. Remove the canisters at ports J1 and J2.
2. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
3. Replace canisters J1 and J2 with valves open.
4. Ensure the valves are in the configuration shown and initial each step when complete:

Valve ID	Status	Completed
B	Open	
K2	Closed	
M2	Closed	
N1	Closed	
N2	Closed	
N3	Closed	
N4	Closed	
R	Closed	
S	Closed	
U	Closed	

V	Closed	
W1	Closed	
W2	Closed	
W3	Closed	
Y	Closed	
Z	Closed	
A	Open	
C	Open	
D	Open	
E	Open	
F	Open	
G	To collector	
H1	Open	
H2	Open	
H3	Closed	
J1	Closed	
J2	Closed	
J3	Closed	
K1	Open	
M1	Open	
T	Closed	

5. Ship canisters after they have been released for shipment to PNNL for composition and isotopic ammonia ratio analysis.
6. Transmit Data Acquisition Control System (DACS) logs and copies of notebook records to the customer.

Note – Consult with the cognizant chemist and engineers if necessary to determine the method to remove solution from the extractor-mixing chamber.

7. Collect the remaining $^{15}\text{NH}_4\text{OH}$ solution from the extractor mixing chamber by best available method in 1 L polybottles.
8. Record the estimated volume, time the sample is exposed to atmosphere before containing and characteristics of the solution removed in a controlled notebook.
9. Load samples out of the hot cell under HPT coverage using LO-160-101 as applicable.
10. Submit sample for ammonia analysis by Ion Selective Electrode (ISE).
11. At conclusion of ammonia analysis transmit data to the customer (see section 7.0 for reporting requirements).
12. Repeat steps from section 6.2.1 step 1 through section 6.2.4 step 11 for a duplicate sample before proceeding to step 13.

13. Prepare the RGS for Phase II testing.

6.3 Phase II Testing

6.3.1 Solution Tests

6.3.1.1 Solution Preparation and Analysis

Note – Solutions must be made fresh the day sample is to be run.

1. Make up a solution of 0.04 M $^{15}\text{NH}_4\text{OH}$ solution.
2. Allow solution to mix gently for 5 minutes.
3. Fill a 15 mL vial with a septum lid to the top with the $^{15}\text{NH}_4\text{OH}$ solution.
4. Submit the 15 mL vial for ammonia analysis by ISE.
5. Make up a solution of 0.04 M $^{14}\text{NH}_4\text{OH}$ solution.
6. Fill a 15-ml vial with a septum lid to the top with the $^{14}\text{NH}_4\text{OH}$ solution.
7. Submit the 15-ml vial for ammonia analysis by ion selective electrode (ISE).

6.3.1.2 Introduction of Simulant Solution to Extractor and Collection of Preliminary Samples

Note – All system equilibration and system leak checks should be performed and documented prior to sample introduction according to the protocol established in LT-160-101 (appendix C) sections 10.1.2 and 10.2.2.

1. Ensure canisters are attached to ports C/D and H1/J1 and that the canister valves are open for evacuation.
2. Ensure the system temperature is at 10 to 12°C and record the date time, event, temperature and pressure in the notebook.
3. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
4. Adjust pressure indicator zero offsets as necessary.
5. Close valve T.

6. Dispense approximately 100 ml of the $^{14}\text{NH}_4\text{OH}$ solution through valve W2 and then ensure valve W2 is closed.
7. Introduce 300 ml of the $^{14}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
8. Close valve K1.
9. Open valve T.
10. Ensure the system temperature is at 10 to 12° C.

Note - The motor speed of the extractor is 60 rpm when the controller dial on the RGS console is dialed to 999.

11. Begin mixing solution by turning on the mixing motor and adjusting the motor speed to 60 rpm.
12. When system temperature and pressure stabilizes, record the date, time, event, temperature and pressure on the RGS processing worksheet and post in the controlled notebook.
13. Close valve D and record: sample/container ID, temperature, and pressure in the notebook.
14. Close valve F and ensure valve G is turned to the collector side.
15. Perform the following steps 12 times initialing each step as performed:

Step	1	2	3	4	5	6	7	8	9	10	11	12
a. Raise the mercury reservoir above the 0-point.												
b. Open valve U.												
c. Control flow of mercury with G, sight glass and level of mercury reservoir.												
d. Lower mercury reservoir to bottom of travel.												
e. Record the system temperature and pressure in notebook.												
f. Turn valve G to the extractor side and wait until mercury completely evacuates displacement bulb.												
g. Close valve U.												
h. Turn valve G to collector side.												
i. Repeat steps until completed.												

16. Raise the mercury reservoir above 0-point.

17. Open valve U and control the flow of mercury to the sight glass with valve G and height of the reservoir.
18. Lower the mercury reservoir to bottom of travel.
19. Record the temperature and pressure in the controlled notebook.
20. Close the valves on the gas sample canister at port J1 and H1.
21. Record the vapor sample canister ID, temperature and pressure in the notebook.
22. Stop data acquisition, close file, and copy file to a floppy disk.
23. Close valve T.
24. Remove the canisters at ports D and J1.
25. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
26. Place new canisters at port C/D, H1/J1 and H2/J2 and ensure valves are open.
27. Ensure auxiliary port is plugged.
28. Turn valve G to the collector side
29. Open valve K1 and ensure the rotary vane pump is running and valve M1 is open.
30. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,H3,J1,J2,J3,K1,M1

31. Turn heater/chiller unit off.

6.3.1.3 Introduction of the Diluent to the Extractor

1. Ensure the rotary vane pump is running.
2. Ensure the stirrer motor is rotating at 60 rpm.
3. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

4. Dispense approximately 100 ml diluent through valve W2 and then ensure valve W2 is closed.
5. Introduce 300 ml of the $^{15}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
6. Record date, time, event and temperature after the addition of diluent.

6.3.1.4 Sampling at 24 hour, 4 days and 16 days from Test Initiation

Note – The system is kept under ambient temperature and mixing conditions for the duration of the tests. Temperature is decreased to 10°C only during vapor pumping.

1. Ensure rotary vane pump is running.
2. Ensure valve T is closed and valves K1 and M1 are open.
3. Ensure canisters are attached at ports H1/ J1 and H2/J2 and that the valves for these canisters are open.
4. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

5. Ensure stirrer motor is rotating at 60 rpm.

Note – No DACS data is obtained during equilibration times. Data is only taken during pumping and vapor extraction.

6. For the 24-hour sample, wait until 24 hours have elapsed from the time the $^{15}\text{NH}_4\text{OH}$ solution was added before proceeding.
7. Ensure heater/chiller are on and controlling to 10° C and extractor temperature is ~10° C.
8. Set automatic data acquisition to 10-second interval data log and record date file name in notebook.
9. Ensure system pressure is less than 0.01 kPa.
10. Adjust pressure indicator zero offsets as necessary.
11. Close valve K1.

12. Open valve T and wait until system's temperature and pressure stabilizes.

13. Record system temperature and pressure in the notebook.

14. Close valve F and ensure G is turned to the collector side.

15. Perform the following sequence 18 times and initial when completed:

Step description:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
a. Raise mercury reservoir above 0-point.																		
b. Open valve U and control height of mercury in sight glass with height of reservoir.																		
c. Lower mercury reservoir to bottom of travel.																		
d. Record system pressure and temperature in notebook.																		
e. Turn valve G to extractor side and wait until mercury completely evacuates the displacement bulb.																		
f. Close valve U.																		
g. Turn valve G to collector side.																		
h. Repeat cycles until complete																		

16. Raise mercury reservoir above the 0-point.

17. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.

18. Lower mercury reservoir to bottom of travel.

19. Record system pressure and temperature in notebook.

20. Close valves J1 and J2 and H1 and H2 and record canister/sample ID, pressure and temperature in the notebook.

21. Stop data acquisition, close file and copy data file to floppy disk.

Note – Canisters should be shipped to PNNL as soon as possible after collection, contact shipper concerning shipping the day samples are ready.

22. Remove canisters at ports J1 and J2.

23. After 3 days repeat steps 1 through 22.

Note – Consult with the cognizant chemist and engineers if necessary to determine the method to remove solution from the extruder-mixing chamber.

24. After 12 days repeat steps 1 through 22 and collect the remaining NH_4OH solution from the extruder mixing chamber by best available method.

25. Record the estimated volume and characteristics of the solution removed in a controlled notebook.

26. Ship remaining canisters after they have been released for shipment to PNNL for isotopic ammonia analysis.

27. Load liquid samples out of the hot cell under HPT coverage using LO-160-101 as applicable.

28. Submit sample for ammonia analysis by ISE.

29. At conclusion of ammonia analysis transmit data to the customer (see section 7.0 for reporting requirements).

30. Repeat section 6.3.1.1 step 1 through section 6.3.1.4 step 29 for a duplicate sample before proceeding to the next test.

Note – Consult the customer for guidance in completion of either section 6.2.2 *Concentrated Solution Tests* or 6.2.3 *Solution/Solids Tests* depending on data needs. Proceed with either section under written direction of the customer.

6.3.2 Concentrated Solution Tests

6.3.2.1 Solution Preparation and Analysis

Note – Solutions must be made fresh the day sample is to be run.

1. Make up a solution of 0.2 M $^{14}\text{NH}_4\text{OH}$ solution.

2. Allow solution to mix gently for 5 minutes.

3. Fill a 15-ml vial with a septum lid to the top with the $^{15}\text{NH}_4\text{OH}$ solution.

4. Submit the 15-ml vial for ammonia analysis by ion selective electrode (ISE).
5. Make up a solution of 0.04 M $^{15}\text{NH}_4\text{OH}$ solution.
6. Fill a 15-ml vial with a septum lid to the top with the $^{15}\text{NH}_4\text{OH}$ solution.
7. Submit the 15-ml vial for ammonia analysis by ion selective electrode (ISE).

6.3.2.2 Introduction of Simulant Solution to Extractor and Collection of Preliminary Samples

Note – All system equilibration and system leak checks should be performed and documented prior to sample introduction according to the protocol established in LT-160-101 (appendix C) sections 10.1.2 and 10.2.2.

1. Ensure canisters are attached to ports C/D and H1/J1 and that the canister valves are open for evacuation.
2. Ensure the system temperature is at 10 to 12°C and record the date time, event, temperature and pressure in the notebook.
3. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
4. Adjust pressure indicator zero offsets as necessary.
5. Close valve T.
6. Dispense approximately 100 ml $^{14}\text{NH}_4\text{OH}$ solution through valve W2 and then ensure valve W2 is closed.
7. Introduce 300 ml of the $^{14}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
8. Close valve K1.
9. Open valve T.
10. Ensure the system temperature is at 10 to 12° C.

Note - The motor speed of the extractor is 60 rpm when the controller dial on the RGS console is dialed to 999.

11. Begin mixing solution by turning on the mixing motor and adjusting the motor speed to 60 rpm.

12. When system temperature and pressure stabilizes, record the date, time, event, temperature and pressure on the RGS processing worksheet and post in the controlled notebook.
13. Close valve D and record: sample/container ID, temperature, and pressure in the notebook.
14. Close valve F and ensure valve G is turned to the collector side.
15. Perform the following steps 12 times initialing each step as performed:

Step	1	2	3	4	5	6	7	8	9	10	11	12
a. Raise the mercury reservoir above the 0-point.												
b. Open valve U.												
c. Control flow of mercury with G, sight glass and level of mercury reservoir.												
d. Lower mercury reservoir to bottom of travel.												
e. Record the system temperature and pressure in notebook.												
f. Turn valve G to the extractor side and wait until mercury completely evacuates displacement bulb.												
g. Close valve U.												
h. Turn valve G to collector side.												
i. Repeat steps until completed.												

16. Raise the mercury reservoir above 0-point.
17. Open valve U and control the flow of mercury to the sight glass with valve G and height of the reservoir.
18. Lower the mercury reservoir to bottom of travel.
19. Record the temperature and pressure in the controlled notebook.
20. Close the valves on the gas sample canister at port J1 and H1.
21. Record the vapor sample canister ID, temperature and pressure in the notebook.
22. Stop data acquisition, close file, and copy file to a floppy disk.
23. Close valve T.
24. Remove the canisters at ports D and J1.

25. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
26. Place new canisters at port C/D, H1/J1 and H2/J2 and ensure valves are open.
27. Ensure auxiliary port is plugged.
28. Turn valve G to the collector side
29. Open valve K1 and ensure the rotary vane pump is running and valve M1 is open.
30. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,H3,J1,J2,J3,K1,M1

31. Turn heater/chiller unit off.

6.3.2.3 Introduction of the Diluent to the Extractor

1. Ensure the rotary vane pump is running.
7. Ensure the stirrer motor is rotating at 60 rpm.
8. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

9. Dispense approximately 100 ml $^{15}\text{NH}_4\text{OH}$ solution diluent through valve W2 and then ensure valve W2 is closed.
10. Introduce 300 ml of the $^{15}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
11. Record date, time, event and temperature after the addition of diluent.

6.3.2.4 Sampling at 24 hour, 4 days and 16 days from Test Initiation

Note – The system is kept under ambient temperature and mixing conditions for the duration of the tests. Temperature is decreased to 10°C only during vapor pumping.

1. Ensure rotary vane pump is running.

2. Ensure valve T is closed and valves K1 and M1 are open.
3. Ensure canisters are attached at ports H1/ J1 and H2/J2 and that the valves for these canisters are open.
4. Ensure the following valve configurations:

Closed	H3, J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

5. Ensure stirrer motor is rotating at 60 rpm.

Note – No DACS data is obtained during equilibration times. Data is only taken during pumping and vapor extraction.

6. For the 24-hour sample, wait until 24 hours have elapsed from the time of addition of the $^{15}\text{NH}_4\text{OH}$ solution diluent before proceeding.
7. Ensure heater/chiller are on and controlling to 10°C and extractor temperature is $\sim 10^\circ\text{C}$.
8. Set automatic data acquisition to 10-second interval data log and record date file name in notebook.
9. Ensure system pressure is less than 0.01 kPa.
10. Adjust pressure indicator zero offsets as necessary.
11. Close valve K1.
12. Open valve T and wait until system's temperature and pressure stabilizes.
13. Record system temperature and pressure in the notebook.
14. Close valve F and ensure G is turned to the collector side.
15. Perform the following sequence 18 times and initial each step when completed:

Step description:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
a. Raise mercury reservoir above 0-point.																		
b. Open valve U and control height of mercury in sight glass with height of reservoir.																		

Step description:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
c. Lower mercury reservoir to bottom of travel.																		
d. Record system pressure and temperature in notebook.																		
e. Turn valve G to extractor side and wait until mercury completely evacuates the displacement bulb.																		
f. Close valve U.																		
g. Turn valve G to collector side.																		
h. Repeat cycles until complete																		

16. Raise mercury reservoir above the 0-point.

17. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.

18. Lower mercury reservoir to bottom of travel.

19. Record system pressure and temperature in notebook.

20. Close valves J1 and J2 and H1 and H2 and record canister/sample ID, pressure and temperature in the notebook.

21. Stop data acquisition, close file and copy data file to floppy disk.

Note – Canisters should be shipped to PNNL as soon as possible after collection, contact shipper concerning shipping the day samples are ready.

22. Remove canisters at ports J1 and J2.

23. After 3 days repeat steps 1 through 22.

Note – Consult with the cognizant chemist and engineers if necessary to determine the method to remove solution from the extruder-mixing chamber.

24. After 12 days repeat steps 1 through 22 and collect the remaining NH_4OH solution from the extruder mixing chamber by best available method.

25. Record the estimated volume and characteristics of the solution removed in a controlled notebook.
26. Ship remaining canisters after they have been released for shipment to PNNL for isotopic ammonia analysis.
27. Load liquid samples out of the hot cell under HPT coverage using LO-160-101 as applicable.
28. Submit sample for ammonia analysis by ISE.
29. At conclusion of ammonia analysis transmit data to the customer (see section 7.0 for reporting requirements).
30. Repeat section 6.3.2.1 step 1 through section 6.3.2.4 step 29 for a duplicate sample before proceeding to the next test.

6.3.3 Solution/Solids Tests

6.3.3.1 Solution Preparation and Analysis

1. Make up a solution of 0.04 M $^{14}\text{NH}_4\text{OH}$ solution
2. Allow solutions to mix gently for 5 minutes.
3. Fill a 15 mL vial with a septum lid to the top with the $^{14}\text{NH}_4\text{OH}$ solution.
4. Submit the 15 mL vial for ammonia analysis by ion selective electrode (ISE).
5. Make up a solution of 0.04 M $^{15}\text{NH}_4\text{OH}$ solution.
6. Fill a 15 mL vial with a septum lid to the top with the $^{15}\text{NH}_4\text{OH}$ solution.
7. Submit the 15 mL vial for ammonia analysis by ion selective electrode (ISE).

6.3.3.2 Introduction of Simulant Solid and Solution to Extractor

Note – All system equilibration and system leak checks should be performed and documented prior to sample introduction according to the protocol established in LT-160-101 (appendix C) sections 10.2.1 and 10.2.2.

1. Ensure canisters are attached to ports C/D and H1/J1 and that the canister valves are open for evacuation.
2. Ensure the system temperature is at 10 to 12°C and record the date time, event, temperature and pressure in the notebook.

3. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
4. Adjust pressure indicator zero offsets as necessary.
5. Close valve T.

Note – Solid sample shall be loaded into the hot cell using LO-160-101 under HPT supervision before proceeding.

6. Weigh the equivalent of 120 mL of sea sand using the density provided by the manufacturer into a tube to be placed in the extruder.
7. Load the sea sand into the extractor by best available method.

Note - All system equilibration and leak checks must be performed after solids introduction according to the protocol established in LT-160-101 sections 10.2.1 and 10.2.1.

8. Open valve T slowly to evacuate the extractor after addition of the solids.
9. Close valve T.
10. Dispense approximately 100 ml $^{14}\text{NH}_4\text{OH}$ solution through valve W2 and then ensure valve W2 is closed.
11. Introduce 180 mL of the $^{14}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
12. Close valve K1.
13. Open valve T.
14. Ensure the system temperature is at 10 to 12° C.

Note - The motor speed of the extractor is 60 rpm when the controller dial on the RGS console is dialed to 999.

15. Begin mixing solution by turning on the mixing motor and adjusting the motor speed to 60 rpm.
16. When system temperature and pressure stabilizes, record the date, time, event, temperature and pressure on the RGS processing worksheet and post in the controlled notebook.
17. Close valve D and record: sample/container ID, temperature, and pressure in the notebook.

18. Close valve F and ensure valve G is turned to the collector side.

19. Perform the following steps 12 times initialing each step as performed:

Step	1	2	3	4	5	6	7	8	9	10	11	12
a. Raise the mercury reservoir above the 0-point.												
b. Open valve U.												
c. Control flow of mercury with G, sight glass and level of mercury reservoir.												
d. Lower mercury reservoir to bottom of travel.												
e. Record the system temperature and pressure in notebook.												
f. Turn valve G to the extractor side and wait until mercury completely evacuates displacement bulb.												
g. Close valve U.												
h. Turn valve G to collector side.												
i. Repeat steps until completed.												

20. Raise the mercury reservoir above 0-point.

21. Open valve U and control the flow of mercury to the sight glass with valve G and height of the reservoir.

22. Lower the mercury reservoir to bottom of travel.

23. Record the temperature and pressure in the controlled notebook.

24. Close the valves on the gas sample canister at port J1 and H1.

25. Record the vapor sample canister ID, temperature and pressure in the notebook.

26. Stop data acquisition, close file, and copy file to a floppy disk.

27. Close valve T.

28. Remove the canisters at ports D and J1.

29. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.

30. Place new canisters at port C/D, H1/J1 and H2/J2 and ensure valves are open.

31. Ensure auxiliary port is plugged.
32. Turn valve G to the collector side
33. Open valve K1 and ensure the rotary vane pump is running and valve M1 is open.
34. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,H3,J1,J2,K1,M1

35. Turn heater/chiller unit off.

6.3.3.3 Introduction of the Diluent to the Extractor

1. Ensure the rotary vane pump is running.
2. Ensure the stirrer motor is rotating at 60 rpm.
3. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

4. Dispense approximately 100 ml $^{15}\text{NH}_4\text{OH}$ solution diluent through valve W2 and then ensure valve W2 is closed.
5. Introduce 300 ml of the $^{15}\text{NH}_4\text{OH}$ solution to the RGS mixing chamber through valves W1 and W3 using the diluent reservoir.
6. Record date, time, event and temperature after the addition of diluent.

6.3.3.4 Sampling at 24 hour, 4 days and 16 days from Test Initiation

Note – The system is kept under ambient temperature and mixing conditions for the duration of the tests. Temperature is decreased to 10°C only during vapor pumping.

1. Ensure rotary vane pump is running.
2. Ensure valve T is closed and valves K1 and M1 are open.
3. Ensure canisters are attached at ports H1/ J1 and H2/J2 and that the valves for these canisters are open.
4. Ensure the following valve configurations:

Closed	H3,J3,K2,M2,N1,N2,N3,N4,R,S,T,U,V,W1,W2,W3,Y,Z
Open	A,B,C,D,E,F,G(to collector),H1,H2,J1,J2,K1,M1

5. Ensure stirrer motor is rotating at 60 rpm.

Note – No DACS data is obtained during equilibration times. Data is only taken during pumping and vapor extraction.

6. For the 24-hour sample, wait until 24 hours have elapsed from the addition of $^{15}\text{NH}_4\text{OH}$ solution diluent before proceeding.

7. Ensure heater/chiller are on and controlling to 10°C and extractor temperature is $\sim 10^\circ\text{C}$.

8. Set automatic data acquisition to 10-second interval data log and record date file name in notebook.

9. Ensure system pressure is less than 0.01 kPa.

10. Adjust pressure indicator zero offsets as necessary.

11. Close valve K1.

12. Open valve T and wait until system's temperature and pressure stabilizes.

13. Record system temperature and pressure in the notebook.

14. Close valve F and ensure G is turned to the collector side.

15. Perform the following sequence 18 times and initial when completed:

Step description:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
a. Raise mercury reservoir above 0-point.																		
b. Open valve U and control height of mercury in sight glass with height of reservoir.																		
c. Lower mercury reservoir to bottom of travel.																		
d. Record system pressure and temperature in notebook.																		
e. Turn valve G to																		

Step description:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
extractor side and wait until mercury completely evacuates the displacement bulb.																		
f. Close valve U.																		
g. Turn valve G to collector side.																		
h. Repeat cycles until complete																		

16. Raise mercury reservoir above the 0-point.

17. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.

18. Lower mercury reservoir to bottom of travel.

19. Record system pressure and temperature in notebook.

20. Close valves J1 and J2 and H1 and H2 and record canister/sample ID, pressure and temperature in the notebook.

21. Stop data acquisition, close file and copy data file to floppy disk.

Note – Canisters should be shipped to PNNL as soon as possible after collection, contact shipper concerning shipping the day samples are ready.

22. Remove canisters at ports J1 and J2.

23. After 3 days repeat steps 1 through 22.

Note – Consult with the cognizant chemist and engineers if necessary to determine the method to remove solution from the extruder-mixing chamber.

24. After 12 days repeat steps 1 through 22 and collect the remaining NH_4OH solution from the extruder mixing chamber by best available method.

25. Record the estimated volume and characteristics of the solution removed in a controlled notebook.

26. Ship remaining canisters after they have been released for shipment to PNNL for isotopic ammonia analysis.

27. Load liquid samples out of the hot cell under HPT coverage using LO-160-101 as applicable.

28. Submit sample for ammonia analysis by ISE.
29. At conclusion of ammonia analysis transmit data to the customer (see section 7.0 for reporting requirements).
30. Repeat section 6.3.2.1 step 1 through section 6.3.2.4 step 29 for a duplicate sample before proceeding to the next test.

7.0 REPORTING REQUIREMENTS

At the completion of each sample run, the following shall be provided to the customer: a set of DACS data obtained in accordance with LT-106-101, copies of notebook observations transmitted to the customer via facsimile, and any analytical results from sample analyses as validated data from the laboratory information management system, LabCore. No formal report or data package will be transmitted.

8.0 SCHEDULE FOR TESTS

December 15 – complete processing and approval of test plan

Phase I Solution Tests:

Jan. 13 – Run #1 set up

Jan. 14 – 24-hour sample obtained

Jan. 17 – Prepare for Run #2

Jan. 19 – 24-hour sample obtained

Jan 24-28 – First shipment of RGS samples to PNNL for ammonia analyses

Shipment to include: 4-24-hour phase I sample canisters

4-initial and 24-hour phase II run #1 test sample canisters

Submit to 222-S analytical for analysis: 2-initial aqueous samples of 0.04M

NH₄OH solution for NH₃ by ISE

2-resultant aqueous samples of 0.04M

NH₄OH solution (after extraction) for
NH₃ by ISE

Phase II Solution Tests

Jan. 24 – Run set up #1

Jan. 25 – 24-hour sample obtained

Jan. 28 – 4-day sample obtained

Feb. 9 – 16-day sample obtained

Feb. 10 – Run #2 set up

Feb. 11 – 24-hour sample obtained

Feb. 7-11 – Second shipment of RGS samples to PNNL for ammonia analyses

Shipment to include: 2-4 day phase II run #1 sample canisters

2- 16-day phase II run #1 sample canisters

Submit to 222-S analytical for analysis: 4-initial aqueous samples of 0.04M

NH₄OH solution for NH₃ by ISE

1-resultant aqueous samples of 0.04M

NH₄OH solution (after extraction) for

NH₃ by ISE

Phase II Solution Tests

Feb. 14 – 4-day sample obtained

Feb. 26 (Saturday) – 16-day sample

Feb. 26- Mar.2 - third shipment of RGS samples to PNNL for ammonia analyses

Shipment to include: 4- initial and 24-hour phase II run #2 sample canisters

2- 4-day phase II run #2 sample canisters

2- 16-day phase II run #2 sample canisters

Submit to 222-S analytical for analysis: 2-initial aqueous samples of 0.04M

NH₄OH solution for NH₃ by ISE

1-resultant aqueous samples of NH₄OH

solution (after extraction) for NH₃ by

ISE

Phase II solution/solids tests:

Feb. 28- Run #1 set up

Feb. 29 – 24-hour sample

Mar. 3 – 4-day sample

Mar. 15 – 16-day sample

Mar. 20 - Run #2 set up

Mar.20 –24 - fourth shipment of RGS samples to PNNL for ammonia analyses

Shipment to include: 4- initial and 24-hour phase II run #1 sample canisters

2- 4-day phase II run #1 sample canisters

2- 16-day phase II run #1 sample canisters

Submit to 222-S analytical for analysis: 2-initial aqueous samples of 0.04 or 0.2M
NH₄OH solution for NH₃ by ISE
1-resultant aqueous samples of NH₄OH
solution (after extraction) for NH₃ by
ISE

Mar. 21 – 24-hour sample

Mar. 24 – 4-day sample

April 5 – 16-day sample

April 10-14 - Fifth shipment of RGS samples to PNNL for ammonia analyses

Shipment to include: 4- initial and 24-hour phase II run #1 sample canisters

2- 4-day phase II run #1 sample canisters

2- 16-day phase II run #1 sample canisters

Submit to 222-S analytical for analysis: 2-initial aqueous samples of 0.04 or 0.2M
NH₄OH solution for NH₃ by ISE
1-resultant aqueous samples of NH₄OH
solution (after extraction) for NH₃ by
ISE

Appendix A
LO-100-151
Segregate and Manage Solid
Laboratory Wastes

WASTE MANAGEMENT LABORATORY
SEGREGATE AND MANAGE SOLID LABORATORY WASTES
LO-100-151

REV/CHANGE NO. E-4

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APPROVAL DESIGNATOR ES

Procedure Use Level REFERENCE

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<u>Change Level</u>	<u>Date</u>	<u>Change Document</u>	<u>Page(s)</u>	<u>Description</u>
E-0	10/07/98	LO-100-151	All	Exempt non-contaminated glass and other items from packaging and labeling requirements. Change mRad/hr to mr/hr. Format change.
E-1	10/15/98	LO-100-151	25	Editorial only. Change step 6.11.11 to read 6.11.7 through 6.11.9.
E-2	03/29/99	99-30	22 and 24	Editorial only. Change step callouts.
E-3	06/30/99	99-49	3 and 23	Delete reference LO-100-155, add step 6.10.1, change term from smearable to removable throughout procedure.
E-4	11/16/99	99-140	10	Add procedure step caveat.

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1.0 PURPOSE AND SCOPE

This procedure establishes the guidelines for collecting and packaging of solid laboratory wastes (for example, glass, metal, plastic) low-level radioactive and radioactive hazardous (mixed) wastes within the 222-S Complex.

2.0 REFERENCES

LO-100-107, *Cubicle Housekeeping and Waste Management*

LO-161-172, *Perform Complex 11A Hot Cell Operation*

LO-100-166, *Manage Satellite Accumulation Area, 90 Day Accumulation Areas and the Treatment, Storage, Disposal (TSD) Units*

3.0 PRECAUTIONS AND LIMITATIONS

Work is performed under the guidance and direction of the generator's manager or designee. The user shall apply training, working knowledge and best judgement to perform the work required. Deviation in the order which steps occur is allowed with prior approval from the Manager.

Waste is collected within laboratory hoods and laboratory rooms in approved waste container, U. S. Department of Transportation shipping containers, or appropriate containment during routine or off-normal working conditions. Waste generated during maintenance or construction activities is collected and placed in U. S. Department of Transportation shipping containers.

3.1 Solid Low Level Radioactive Waste

NOTE

Hazardous wastes, or mixed wastes, are not disposed of or mixed with low-level radioactive or compactible wastes. Disposal of this nature will be in violation of state and or federal regulations and waste acceptance criteria.

- 3.1.1 **Compactable waste** - The approved contents for waste to be compacted are paper, dry rags, gloves, clothing, plastic wraps, and other dry, low density, compactible wastes. These wastes are managed as low-level radioactive materials. Dry, less than detectable smear media are acceptable for disposal to a compactible room waste drum.

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- Wastes that are prohibited in compactible room waste drums include glass, empty plastic bottles, stir bars, metal, hard plastic, rigid cardboard, hazardous waste, and radioactively contaminated articles including contaminated smear media or unidentified and uncharacterized wastes. Free standing liquids are not allowed.
- 3.1.2 **For burial - Solid low level radioactive wastes generated at the 222-S Complex** include empty (by definition in 3.2) polybottles, scintillation vials, T-9 vials, alpha and beta discs, planchettes, glass vials, and miscellaneous wastes.
- 3.1.3 **Contaminated Smear Media -** Contaminated smear media which has not come into direct contact with a listed waste sample or a listed waste, can be disposed of as a low-level radioactive waste. For example: A contamination smear of a hood in which the smear media (to the knowledge of the person performing the smear) does not directly contact the sample material or waste in the hood, but radioactive contamination is detected on the smear media. The smear media can be disposed of as low-level radioactive waste.

3.2 Empty Containers

A container (for example, poly bottles, scintillation vials, pipet tips, and so forth) is considered empty and nonhazardous when all wastes in it have been taken out that can be removed using commonly employed practices and it contains no visible sludge or free-flowing liquids.

If the container held acutely hazardous waste ("P" waste codes), toxic extremely hazardous waste (EHW waste codes WT01) or pesticides bearing the danger or warning label, the container must be rinsed at least three times with an appropriate cleaner or solvent. The volume of the cleaner or solvent used for each rinsing must be ten percent or more of the container's capacity or of sufficient quantity to thoroughly decontaminate the container. Any rinsate or residue which results from the cleaning of containers must be properly managed as a hazardous waste.

Hood Waste-(Contaminated Glass Containers) may be taken to 2B hood 16 (North side), to be crushed. The container must be empty and dry.

3.3 Solid Mixed Waste

- 3.3.1 **Solid mixed waste derived from listed tank samples -** Any solid waste which comes into contact with, or is derived from the analysis or management of tank farms sample or waste is to be assigned the listed waste codes F001, F002, F003, F004, and F005.

Waste known through process knowledge, not to be derived from Tank Farm samples or waste, do not carry the above listed waste codes unless otherwise directed by Hazardous Materials Control (HMC) manager and Environmental Compliance Officer (ECO).

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- 3.3.2 Solid mixed waste derived from other listed waste sources - Any solid waste which comes into contact with or is derived from the analysis or management of a sample or waste which carries a listed waste code is to be assigned the listed waste code of that sample or waste (examples would be U or P coded wastes).

Example: If a listed waste sample is brought into a hood for analysis, any items which come into contact with the sample (Examples of Mixed Waste in hoods: Plastic bags, gloves, dental rolls, stir bars, tissues, paper towels, metal scoops, dried pens and dried sharpies) and are determined to be waste shall be handled as mixed waste. Containers or vials can be disposed of as nonhazardous low-level radioactive waste, if they are empty by definition.

3.4 Hot Cell and Shielded Waste

Waste is generated from analytical procedures which are performed on undiluted Single Shell Tank or Double Shell Tank samples. This waste is typically generated, (but not limited to) hoods performing the following analyses: TGA/DSC, TIC/TOC, percent water, acid digest, fusion, water digest, and includes tank waste generated within the hot cells.

Containers with a wall thickness greater than a metal lard can, are considered a shielded container and the waste in them must be managed as a shielded waste in accordance with Section 6.9.

3.5 Lead Management/Usage

NOTE

Contaminated lead solids, scrap lead, lead bricks, etc. must be disposed of separately. Contact HMC for guidance.

Lead can be used for shielding of non-hazardous and hazardous radioactive waste packages providing other means of shielding have been used to try and reduce the dose rate. When these other means fail, lead may be used provided the package is clearly marked with the word "Lead used as shielding" and the container is disposed of as shielded mixed waste. Lead waste (for example, lead tape, lead lined gloves, lead contacts on light bulbs) must be noted as lead waste on the inventory sheet. Lead is only to be disposed of as hazardous or mixed waste. Lead is strictly forbidden in low level radioactive waste drums. Improper disposal of lead can result in drum having to be sorted and repackaged. Improper disposal of lead is a violation of state and federal regulations.

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3.6 Acutely Hazardous Waste

An Acutely Hazardous Waste is any discarded pure chemical product(s) (listed in WAC-173-303-9903) that are identified with a dangerous waste number beginning with "P" (for example, copper cyanide PO29). Additionally any of the following waste codes are acutely Hazardous FO20, FO21, FO22, FO23, FO26, or FO27.

3.7 Free Liquids

NOTE

Diatomaceous Earth is not an approved absorbent but is used as a void space filler.

Free liquids are not allowed in Low-Level Radioactive waste containers. Any bottles or vials which have held free liquids are required to have an approved absorbent placed in them if they are not dry. Absorbants approved for use at the laboratories include Imbiber beads for organics, and Radsorb for aqueous. If an approved absorbent is not available contact the Hazardous Materials Control group (HMC) for guidance.

3.8 Radioactive Contaminated Rags

NOTE

Acid contaminated waste such as gloves, rags, paper towels must be rinsed with water and dried prior to disposal.

3.8.1 **Radioactive Contaminated Acid/Caustic Rags** may be rendered as nonhazardous by rinsing the rags in water and drying the rags. The rinsate must be managed as mixed waste if it exhibits the characteristic of corrosivity (for example, $2.0 < \text{pH} < 12.5$)

3.8.2 Radioactive Oily Rags

Non hazardous radioactive oily rags must be moved outside by the end of each day to the low-level radioactive non-regulated oily rag drum by HMC.

Hazardous radioactive oily rags must be moved outside by the end of each day to the mixed waste oily rag drum by HMC.

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3.9 Unknown Wastes

Unknown or abandoned wastes or containers of waste are to be immediately reported to immediate manager, facility manager, ECO, and/or HMC manager. All unknown wastes will be labeled with the date of sampling and the words "Waste Pending Analysis" and stored in a 90-Day Area until analytical data is complete enough to designate waste.

3.10 Incompatible Materials

Do not place incompatible materials into a single container. Examples of these include oxidizing acids (e. g. nitric acid and sulfuric acid) and rags or paper, acid waste, and cyanide or sulfides, oxidizers and reductants, and peroxides and paper. If a waste is believed to be incompatible with other wastes being handled, consult the MSDS for guidance on compatibility of the waste.

3.11 Reactive Materials

Do not place reactive materials into containers. These include alkali metals, explosives, pyrophorics (uranium metal), and chelating compounds. If a waste is believed to be reactive with other wastes being handled, consult the MSDS for guidance on compatibility of the waste.

3.12 Container Compatibility

To help prevent chemical reactions and container deterioration, waste collection containers must be compatible with the waste being placed into them. Containers must be made of or lined with materials that will not react with, and are otherwise compatible with, the waste to be stored, so that the ability of the container to contain waste is not impaired. For example, Hydrofluoric Acid will etch glass and should be collected in polyethylene containers. Consult WHC-SD-CP-LB-028, *Laboratory Reagents*, in determining the type of container to be used for collecting wastes.

3.13 Other Safety Precautions

Wear leather gloves over surgical gloves when handling sharp objects.

Use proper lifting techniques when lifting heavy or awkward loads.

A cart should be used for transporting containers of waste which are heavy (lead lined) or pose an ALARA concern.

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4.0 SPECIAL TOOLS AND EQUIPMENT

Approved absorbent (e. g., radsorb, imbiber beads, universal poly propylene)

Approved void space filler [e. g., Diatomaceous earth (Clean Up IV), pyrofoam]

Approved containers

Magnetic Compactable Waste Contents Sign (Appendix A)

Polyethylene liners

Rubber and Lead Shielding

Survey equipment

Tape

Waste Contents Sheet (see Appendix B)

222-S Hazardous Waste label (see Appendix C)

6 Point Survey Data Contents Sheet (See Appendix E)

5.0 PREREQUISITES

Not Applicable.

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6.0 INSTRUCTIONS

NOTES

Sections, or steps within sections, of this procedure may be performed out of sequence, as required.

Labels may be obtained from HMC or stockroom.

6.1 Prepare Compactible Room Waste Drums

- 6.1.1 OBTAIN a drum.
- 6.1.2 REMOVE drum lid and accessories.
- 6.1.3 PLACE drum into laboratory room or area for collection of waste.
- 6.1.4 MARK drum with three radioactive waste stickers 120° apart on the side of the drum.
- 6.1.5 ATTACH a magnetic Compactible Waste Contents Sheet sign to the drum (See Appendix A).
- 6.1.6 OBTAIN two (4 mil minimum) polyethylene yellow liners.
- 6.1.7 INSPECT polyethylene liners for defects (punctures and tears).
- 6.1.8 DISCARD and REPLACE any faulty liners.
- 6.1.9 INSERT polyethylene liner into drum.
- 6.1.10 FOLD top of liner over rim of drum.
- 6.1.11 REPEAT steps 6.1.9 and 6.1.10 to install second liner.

NOTE

A swing lid is not required for compactible room waste drums. On a case by case basis a swing lid may be used.

- 6.1.12 PLACE swing lid on drum if one is to be used.

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EFFECTIVE DATE 11/16/99**6.2 Prepare Low Level Radioactive Waste Containers**

- 6.2.1 **OBTAIN** approved container and appropriately sized poly liner for collection of low-level radioactive (LLR) waste.
- 6.2.2 **MARK** or **LABEL** side and top of the container "Low-Level Radioactive Waste", "Low-Level Waste", "Low-Level" or "LLR".
- a. IF waste to be collected is known to contain PCBs
PLACE a yellow and black "Known PCB" label on container noting the project location.
- This label can be obtained from HMC or the stockroom.
- b. IF waste to be collected is suspected to contain PCBs
PLACE a white and red "Suspect PCB" label on the container noting the project location.
- This label can be obtained from HMC or the stockroom.
- 6.2.3 **WRITE** room number on side of waste container.
- 6.2.4 **PLACE** poly liner inside container.
- 6.2.5 **FOLD** top of liner over lip of container, and **PRESS** liner toward sides and bottom.
- 6.2.6 **PLACE** prepared container into area for waste collection and **ENSURE** Low-Level Radioactive marking is visible.
- 6.2.7 **OBTAIN** a Waste Contents Sheet and fill out while collecting low-level radioactive waste. (Appendix B)
- a. **CHECK** appropriate square for type of waste being collected
- b. **RECORD** room number
- c. **RECORD** hood number (if applicable)
- d. **ITEMIZE** miscellaneous items placed in the waste container.

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6.3 Prepare Hazardous/Mixed Waste Containers

NOTE

When working with a sample which contains a listed waste other than F001 - F005 a separate waste container must be used.

- 6.3.1 OBTAIN approved container and appropriately sized poly liner.
- 6.3.2 PLACE poly liner inside container.
- 6.3.3 FOLD top of liner over lip of container, and PRESS liner towards sides and bottom.
- 6.3.4 OBTAIN two blank hazardous waste labels. (Appendix C)
 - a. AFFIX one to container side and one to the lid of the container.
 - b. IF a Hazardous Waste label is not available, or used
 - 1. ENSURE the words "Hazardous Waste" are placed visibly on the container.
 - 1. ENSURE that the major risk (e. g. toxic, flammable, corrosive) is placed visibly on the container.
- 6.3.5 IF waste to be collected is known to contain PCBs
PLACE a yellow and black "Known PCB" label on the container noting the project location.
 - This label can be obtained from HMC or the stockroom.
- 6.3.6 IF waste to be collected is suspected to contain PCBs
PLACE a white and red "Suspect PCB" label on the container noting the project location.
 - This label can be obtained from HMC or the stockroom.
- 6.3.7 RECORD room number and hood number on hazardous waste label or container.
 - a. On shielded waste container, write tank number or area from which waste was generated (for example, 101-SC, T-111).
 - When more than two tanks are present use the 219-S as the tank number.

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- 6.3.8 **PLACE** labeled container into another container for stability and/or shielding purposes, if necessary.
- 6.3.9 **ENSURE** either the inner container label is visible, or the outer container has an identical label and is visible.

NOTE

Waste container lid is securely in place at all times when work is not being performed.

- 6.3.10 **PLACE** prepared container into area for waste collection.
- a. **ENSURE** the hazardous waste label is visible.
- 6.3.11 **OBTAIN** a Waste Contents Sheet and fill out while collecting mixed waste.
- a. **CHECK** appropriate square for type of waste being collected.
- b. **RECORD** room number.
- c. **RECORD** hood number (if applicable)
- d. **ITEMIZE** miscellaneous items placed in the waste container.

6.4 Prepare Hot Cell Waste Container (See the Appendix D.)

NOTE

- When working with a sample which contains a listed waste other than F001 - F005 a separate waste container must be used.
- The following description is intended as a guide only. Individual users will develop their own techniques for waste container preparation.

- 6.4.1 **PREPARE** and
LABEL inner waste container.
- a. **USE** an approved inner container.

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- b. **TIE** knots in the end of any suitable material (for example, Tygon¹ tubing) to form a bail.
- c. **TAPE** the ends of the bail to opposite sides of the container leaving room for the lid.
- d. **TAPE** a 2-in. piece of Tygon to the lid to provide a handle to grasp with the manipulator.
- e. **WRITE** on or
LABEL the container with the words "Hazardous Waste."
- f. **WRITE** on or
LABEL the container with the major risk associated with the waste (e. g. Toxic, flammable, corrosive).
- g. **WRITE** tank number or area from which waste was generated (e. g. 101-SC, T-110, K-Basin).
- h. **IF** waste to be collected is known to contain PCBs
PLACE a yellow and black "Known PCB" label on the container noting the project location.
 - This label can be obtained from HMC or the stockroom.
- i. **IF** waste to be collected is suspected to contain PCBs
PLACE a white and red "Suspect PCB" label on the container noting the project location.
 - This label can be obtained from HMC or the stockroom.

6.4.2

PREPARE and
LABEL outer waste container.

- a. **USE** an approved outer container.
- b. **TIE** knots in the end of any suitable material (e.g., Tygon tubing) to form a bail.
- c. **TAPE** the ends of the bail to opposite sides of the container leaving room for the lid.

¹Tygon is a trademark of Norton Performance Plastics.

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NOTE

For extra shielding, additional rubber matting or lead shielding may be used.

- d. **LINE** the can with rubber matting and/or other suitable material as necessary.
- e. **PLACE** a plastic bag in the center of the waste container and fold bag over the outside of the container.
- f. **TAPE** a 2-in. piece of Tygon to the lid to provide a handle to grasp with the manipulator.
- g. **ATTACH** shielding material to the inside of the container lid.

6.4.3 **OBTAIN** two blank hazardous waste labels. (Appendix C)

- a. **AFFIX** one to container side and one to the lid of the container.
- b. **IF** a Hazardous Waste label is not available, or used
ENSURE the words "Hazardous Waste" are placed visibly on the container.
 - 1. **ENSURE** the major risk (e. g. toxic, flammable, corrosive) is placed visibly on the container.

6.4.4 **WRITE** the hot cell location where the waste was generated on the container.

- a. **IF** waste to be collected is from Tank Farm samples
ATTACH an "F-Listed" waste label to the container.
 - hot cell waste containers must be labeled with the tank number or area from which the waste was generated (for example, 101-SC, T-111 K-Basin).

6.4.5 **IF** waste to be collected is waste known to contain PCBs
PLACE a yellow and black "Known PCB" label on the container noting the project location.

- This label can be obtained from HMC or the stockroom.

6.4.6 **IF** waste to be collected is waste suspected to contain PCBs
PLACE a white and red "Suspect PCB" label on the container noting the project location.

- This label can be obtained from HMC or the stockroom.

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- 6.4.7 **PLACE** prepared container into area for waste collection ensuring that the hazardous waste label is visible.
- 6.4.8 **OBTAIN** a Waste Contents Sheet and fill out while collecting hot cell radioactive waste.
- a. **CHECK** appropriate square for type of waste being collected
 - b. **RECORD** room number
 - c. **RECORD** hood number (if applicable)
 - d. **ITEMIZE** miscellaneous items placed in the waste container.

6.5 Collect and Manage Compactible Room Waste

NOTE

- Any questions regarding collection and packaging of compactible waste should be directed to the Hazardous Material Control Manager.
- Hazardous wastes or mixed wastes, are not disposed of or mixed with Low-Level Radioactive compactible wastes. Disposal of this nature will be in violation of state and or federal regulations and waste acceptance criteria.

- 6.5.1 **SEGREGATE** low-level radioactive compactible waste from other waste types, as it is generated, in predesignated drums located in the laboratory rooms.
- 6.5.2 **CONTINUE** filling drum until drum is visibly full.
- 6.5.3 **CHANGE** out bagged drum waste following Sections 6.11 or 6.12.

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EFFECTIVE DATE 11/16/99**6.6 Collect and Manage Low-Level Radioactive Waste**

NOTE

When disposing of equipment, the property number, serial numbers and any other ID numbers (when present) needs to be recorded on property disposal paperwork. HMC can provide assistance with property disposal paperwork issues.

WARNING

Sharp objects and sharp edges must be padded and taped to prevent injury.

- 6.6.1 **CONTACT** HMC for guidance prior to packing waste items too large to fit into a standard collection container.
- 6.6.2 IF the container has a Standards Laboratory bar code, **CONTACT** the Standards Laboratory and report the bar code number (located directly below the bar code) as an empty container.

NOTE

Vial and bottle caps must be removed prior to disposal in waste container.

- 6.6.3 **PLACE** only empty, and nonhazardous items into the waste container.
- 6.6.4 **ADD** approved absorbent to containers which may have residual liquids present as follows:
- a. 10 ml minimum of imbiber beads for residual organic liquids.
 - b. 2 ml minimum of radsorb for residual aqueous liquids.
- 6.6.5 **FILL** out Waste Contents Sheet when collecting waste.
- a. **ITEMIZE** miscellaneous items placed in the waste container.

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6.6.6 IF container is full or ready for removal from hood,
HORSETAIL and
TAPE liner.

- a. **TAPE** or
SECURE lid onto container.

NOTE

Lead shielded containers must be managed as mixed waste.

6.6.7 **NOTIFY** HPT to take contact readings of waste.

- a. IF readings are greater than 100 mr/hr at contact,
SHIELD container to less than 100 mr/hr on contact.

- IF lead shielding is used container must be managed as mixed waste. (Go to Section 6.9)

6.6.8 **DOUBLE BAG** container,
HORSETAIL, and
TAPE bags.

- Outer bag must be yellow.

6.6.9 **ENSURE** outer bag has no removable contamination.

NOTE

Percentages of waste includes the container, items inside the container, void space filler, absorbent, liner, tape, and bags. The percentages are based on volume.

6.6.10 **COMPLETE** Waste Contents Sheet as follows:

- a. **RECORD** size of container
- b. **RECORD** percentages of waste
- c. **RECORD** room number.
- d. **RECORD** hood number.

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- e. **RECORD** the date
- f. **PRINT** your name.
- 6.6.11 **ATTACH** Waste Contents Sheet to outside bag.
- 6.6.12 **ENSURE** container is labeled and dose rate established by HPT is written on container prior to transporting.
- 6.6.13 **TRANSPORT** the container to Room 4E Low-Level Radioactive storage area.
 - a. IF waste is suspect or known PCB waste,
HAVE HMC personnel present to receive waste.

6.7 Low Level Radioactive Waste to be Crushed or Shredded

NOTE

- Free standing liquid is not allowed in shredder or hot crusher.
- PCB waste is not allowed in shredder or hot crusher.
- When collecting vials/bottles to be crushed or shredded void space filler is not required. However, the container **MUST** be empty, non-hazardous, and dry.
- The following are examples of acceptable materials:

Shredder-empty nonhazardous, nonremovable, Plastic and glass bottles, cardboard, small pieces of wood. Materials generated inside a hood that have not contained radioactive material and have no removable contamination.

Glass Crusher-contaminated empty nonhazardous glass and plastic vials, bottles, reading <100 mr/hr at contact.

- 6.7.1 **CONTACT** HMC for guidance on what materials are acceptable for shredding or crushing.
- 6.7.2 **IDENTIFY** items to be shredded or crushed.
- 6.7.3 IF items(s) are non-contaminated such as empty reagent bottles, or other items collected from rooms or benchtops,
LABEL as "Non-Rad waste for shredder" and
SKIP to step 6.7.9.

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- 6.7.4 NOTIFY HPT to take contact readings of waste to be shredded or crushed.
- Containers must be reading <100 mr/hr at contact.
- 6.7.5 BAG container, HORSETAIL, and TAPE bag for any items being taken to the shredder or crusher.
- Outer bag must be yellow.
- 6.7.6 ENSURE outer bag has no removable contamination before removing from collection area.
- 6.7.7 LABEL outer bag or container as "shredder" or "Glass Crusher" waste.
- This can be done with a marking pen, a piece of tape with the appropriate labeling, etc.
- 6.7.8 ENSURE container is labeled as "shredder" or "glass crusher" and dose rate established by HPT and written on container, prior to storing in staging area.
- 6.7.9 TRANSPORT container to designated staging area for either the hot crusher or the shredder.

6.8 Collect and Manage Mixed Waste**WARNING**

All corners and sharp edges must be padded and taped to prevent injury.

- 6.8.1 CONTACT HMC for guidance prior to packaging when working with waste items too large to fit into a standard collection container,
- 6.8.2 IF waste to be collected is shielded waste, PROCEED to Section 6.9.
- 6.8.3 IF waste to be collected is Hot Cell Waste, PROCEED to Section 6.10.

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6.8.4 **CONTACT** HMC for assistance in disposal of the following waste types.

- Waste containers shielded with lead or rubber.
- All waste generated in hot cells.
- Items too large to fit in a standard container (ice cream cartons or lard cans).
- PCB waste.

NOTE

Waste container lid is in place at all times when work is not being performed.

6.8.5 **PLACE** only mixed waste into container.

- a. **IF** packaging containers which may have residual liquids present,
ADD approved absorbent to container as follows:
1. 10 ml minimum of imbibor beads for residual organic liquids.
 2. 2 ml minimum of radsorb for residual aqueous liquids.

6.8.6 **FILL OUT** Waste Contents Sheet when collecting waste.

- a. **ITEMIZE** miscellaneous items placed in the waste container.

6.8.7 **PERFORM** the following when container has been filled to within 25 mm (1 in.) of the top

- a. **HORSETAIL** and tape liner
- b. **TAPE** lid onto container
- c. **NOTIFY** HPT to take contact reading of waste.
 1. **IF** contact reading is greater than 100 mr/hr GO to Section 6.9.

6.8.8 **DOUBLE BAG** container,
HORSETAIL, and
TAPE bags.

- Outer bag must be yellow.

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- 6.8.9 ENSURE outer bag has no removable contamination before removing from collection area.

NOTE

Percentages of waste include the container, items inside container liner, tape, and bags. The percentages are based on volume.

- 6.8.10 COMPLETE Waste Contents Sheet as follows:

- a. RECORD size of container
- b. RECORD percentages of waste
- c. RECORD room number
- d. RECORD hood number
- e. RECORD the date
- f. PRINT your name.

- 6.8.11 ATTACH Waste Contents Sheet to outside of bag.

- 6.8.12 ENSURE the container is labeled and dose rate established by HPT is written on container(s) or label(s) prior to transferring to a 90-day accumulation area.

NOTE

State regulations require dangerous waste/hazardous waste be under control of the generator at all times. DO NOT leave waste container unattended, outside of a Satellite Accumulation Area or a <90-Day Accumulation Area.

- 6.8.13 TRANSFER waste to room 4E 90-Day Accumulation Area.

- 6.8.14 LOG waste into room 4E 90 Day Accumulation Area per LO-100-166.

- IF waste is suspect or known PCB waste, HMC personnel must be present to receive waste.

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EFFECTIVE DATE 11/16/99**6.9 Manage Shielded Waste**

NOTE

Obtain HPT coverage while performing the following steps.

- 6.9.1 **SHIELD** container to less than 100 mr/hr using rubber and/or lead.
- IF lead shielding is used
MARK container with the words "Lead used as shielding."
- 6.9.2 **DOUBLE BAG** container, **HORSETAIL**, and **TAPE** bags.
- Outer bag must be yellow.
- 6.9.3 **ENSURE** outer bag has no removable contamination prior to moving from collection area.
- 6.9.4 **FILL OUT** the appropriate sections of the 6-Point Survey Data Sheet (Appendix E) (See Section 6.1.16 for complete instructions on filling out the 6-Point Survey Data Sheet.)

NOTE

State regulations require dangerous waste/hazardous waste be under control of the generator at all times. **DO NOT** leave waste container unattended outside of a Satellite Accumulation Area or a <90-Day Accumulation Area.

- 6.9.5 **REQUEST** an HPT to perform a 6-Point Survey and fill out the appropriate sections of the 6-Point Survey Data Sheet.

NOTE

HMC personnel are responsible for logging all mixed waste containers into the 90 day accumulation area.

- 6.9.6 **TRANSPORT** container to the 90-Day Accumulation Area as coordinated with the HMC.

- a. **PROVIDE** HMC with the completed 6-Point Survey Data Sheet for the container.

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EFFECTIVE DATE 11/16/99**6.10 Collect and Manage Hot Cell Waste**

6.10.1 **CONTACT** Shift Operations Manager (SOM) and
REQUEST authorization to discharge to the 219-S tank system.

6.10.2 Sample and Equipment Disposal

- a. **REQUEST** assistance from the Hazardous Material Control group (HMC) for disposal of liquid organic or other listed waste (for example pure chemical products).

CAUTION

Excess water from cubicle drains may cause overfilling of 222-S waste tanks.

- b. **POUR** samples or dilutions into the cubicle drains while water is running for disposal.
1. **TURN** water OFF at completion of task.
- c. **RINSE** vials and jars a minimum of three times with water.
1. **AIR DRY** vials
- d. **SET** vials aside for disposal.
- e. **RINSE** paper and fabric items well.
1. **SQUEEZE DRY** three times
2. **ALLOW** to dry completely.
- f. **RINSE** other plastic items, glassware, and equipment well.
1. **ALLOW** to dry
2. **CONSOLIDATE** as required.
- g. **COLLECT** all glass items.
- h. **BREAK** the glass using a heavy object.

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NOTE

Waste container lid is securely in place at all times when work is not being performed.

- i. **FILL OUT** a Waste Contents Sheet as items are placed in the container. Include shielding materials.
 - 1. **ITEMIZE** miscellaneous items placed in the waste container.
- j. **CONSOLIDATE** miscellaneous glass, plastic, paper, fabric, and metal materials as much as possible.
- k. **PLACE** disposable items into the inner can.
 - No free liquids are allowed in the container.
 - 1. **USE** two pound (quart) lard cans for highly radioactive waste. Use five pound lard cans for lower level waste.
- l. **CONSOLIDATE** plastic in a can for future melting.
- m. WHEN the container is full,
PLACE the lid evenly on the can and remove from hot cell per Section 6.15.

6.11 Routine Changeout of Compactible Waste Drum Liners

- 6.11.1 **CHECK** each compactible waste drum daily for fullness.
- 6.11.2 **OBTAIN** a HEPA filtered shop vacuum.
 - a. IF HEPA filtered shop vacuum is not available
CHANGEOUT drum in accordance with Section 6.12.
- 6.11.3 **CHECK** the following on the vacuum.
 - a. **ENSURE** the seal between the HEPA filter and the vacuum is not broken.
 - a. IF the seal is broken
CONTACT building operations and do not use vacuum.
 - b. **VERIFY** DOP filter test date has not expired.

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- 6.11.4 REMOVE lid from drum if one is used.
- 6.11.5 TURN vacuum ON and
REMOVE tape from end of hose.
- 6.11.6 PULL excess inner liner up and
WRAP around the end of the vacuum hose.
- 6.11.7 CONTINUE until excess air is removed from the liner.
- 6.11.8 REMOVE hose from liner.
- 6.11.9 HORSETAIL and
TAPE liner.
- 6.11.10 PULL excess outer liner up and
WRAP around the end of the vacuum hose.
- 6.11.11 REPEAT steps 6.11.7 through 6.11.9 for outer liner.
- 6.11.12 TAPE end of hose.
- 6.11.13 TURN vacuum OFF.
- 6.11.14 REMOVE double-contained waste.
- 6.11.15 CHECK outside of bag for tears.
 - a. IF liners are torn,
PLACE into another liner,
HORSETAIL, and
TAPE.
- 6.11.16 INSPECT contents of bag.
 - a. ESTIMATE the total volume (container size) in liters (gallons), and volume percent
of paper, rubber, plastic, and cloth.
- 6.11.17 RECORD room number, date, name, and volume percent of materials on Waste Contents
Sheet.
- 6.11.18 WEIGH bag.

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- 6.11.19 **RECORD** total weight in kg on Waste Contents Sheet.
- 6.11.20 **ATTACH** Waste Contents Sheet to bag.
- 6.11.21 **HAVE** HPT dose rate, survey and label bag prior to transferring to designated staging area.
- 6.11.22 **IF** contamination is found:
 - a. **DECONTAMINATE** as necessary.
 - b. **NOTIFY** immediate manager.
 - c. **SEGREGATE** contaminated waste per section 6.13.

NOTE

Use the following steps if a vacuum is not available for use.

6.12 Alternate Changeout of Compactible Waste Drum

- 6.12.1 **REMOVE** lid from drum, if one is being used.
- 6.12.2 **HORSETAIL** and **TAPE** inner liner and then outer liner.
- 6.12.3 **REMOVE** double-contained waste.
 - a. **CHECK** outside of bag for tears.
 - b. **IF** liners are torn, **PLACE** into another liner, **HORSETAIL**, and **TAPE**.
- 6.12.4 **INSPECT** contents of bag.
 - a. **ESTIMATE** the total volume (container size) in liters (gallons), and volume percent of paper, rubber, plastic, and cloth.
- 6.12.5 **RECORD** room number, date, name, and volume percent of the materials on Waste Contents Sheet.

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- 6.12.6 **WEIGH** bag.
- 6.12.7 **RECORD** total weight in kg on Waste Contents Sheet.
- 6.12.8 **ATTACH** Waste Contents Sheet to bag.
- 6.12.9 **HAVE** HPT dose rate , survey, and label bag prior to transferring to designated staging area.
- 6.12.10 IF contamination if found:
 - a. **DECONTAMINATE** as necessary.
 - b. **NOTIFY** immediate manager.
 - c. **SEGREGATE** contaminated waste per section 6.13.

6.13 Manage Radioactively contaminated Non-Routine Waste

- 6.13.1 **SEGREGATE** any radiologically contaminated waste from the waste container.
- 6.13.2 **PLACE** contaminated waste into a separate bag or container.
- 6.13.3 **DOUBLE BAG** and **TAPE** bag or waste container.
- 6.13.4 IF waste is known to have been derived from or contacted a listed waste source:
 - a. **LABEL** container with a completed Hazardous Waste Label
 - b. **LABEL** container with the major risk.
 - 1. IF major risk is not known, **CONTACT** HMC for guidance).
 - c. **MOVE** container to a 90-Day accumulation area per LO-100-166.
- 6.13.5 IF waste is not known to have been derived from or contacted a listed waste source:
 - a. **LABEL** container as Low-Level Radioactive waste and manage per section 6.6 of this procedure.
 - b. **DOCUMENT** all corrective actions per the Radiological Problem Report.

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EFFECTIVE DATE 11/16/99**6.14 Collect and Manage Solid Chemical Waste**

NOTE

Whether managing solid chemical wastes as Low-Level Radioactive or Solid Mixed waste the miscellaneous section on the Waste Contents Sheet must have the constituents, procedure number, or test plan listed.

6.14.1 **MANAGE** solid chemical wastes as instructed by the solid waste stream fact sheet.

- a. **DISPOSE** of nonhazardous solid chemical wastes as Low-Level Radioactive waste per Section 6.6 of this procedure.
- b. **DISPOSE** of hazardous/mixed solid chemical waste either as solid mixed waste per Section 6.8, or as solid chemical mixed waste per this section.

6.14.2 **OBTAIN** an approved container if required.

NOTE

A combination label can be used as long as all the information required below is included on the label.

6.14.3 **LABEL** waste or waste container with the following:

- a. A completed Waste Stream Label obtained from the Solid Waste Stream Fact Sheet (SWSFS) per WMH-310, Section 6.1.
- b. A Hazardous Waste Label.
- c. Major risk (i.e., flammable, corrosive, etc.).
- d. Room number and hood number (if applicable).

6.14.4 **PLACE** solid chemical waste into container (if one is used).

6.14.5 **COMPLETE** Waste Contents Sheet as follows:

- a. Record size of container
- b. Record percentages of waste
- c. Record room number

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- d. Record hood number
 - e. Record the date
 - f. Print your name.
- 6.14.6 **DOUBLE BAG** container or waste, **HORSETAIL** and **TAPE** bags.
- Outer bag must be yellow.
- 6.14.7 **ENSURE** outer bag has no removable contamination.
- 6.14.8 **ENSURE** the container is labeled and dose rate established by HPT is written on the container prior to transferring to Room 4E.
- 6.14.9 **CONTACT** HMC or shift manager for access to Room 4E 90-Day accumulation area.
- a. **SHIELD** container if reading 100 mr/hr or greater.
- 6.14.10 **TRANSFER** waste to Room 4E 90-day accumulation area.
- 6.14.11 **PLACE** package into 90-day accumulation area.
- 6.14.12 **LOG** container into 90-day accumulation area per procedure LO-100-166.

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EFFECTIVE DATE 11/16/99**6.15 Routine Cubicle Waste Load-Out****NOTE**

- All waste load-outs must be coordinated with the HMC.
- Waste packages reading >100 mr/hr in the airlock, must be returned to the cubicle for rework or shielded to <100 mr/hr.
- HPT coverage is required for all waste load-outs.

6.15.1 **LOAD IN/OUT** of sample/equipment according to cubicle operating procedures LO-100-110 and LO-161-172.

NOTE

For contamination control, a second bag may be needed.

6.15.2 **PLACE** container in a plastic bag,
HORSETAIL, and
TAPE.

- Outer bag must be yellow.

NOTE

State regulations require dangerous waste/hazardous waste be under control of the generator at all times. **DO NOT** leave waste container unattended outside of a Satellite Accumulation Area or a <90-Day Accumulation Area.

WARNING

Do not leave waste container unattended. It must be stored in an approved 90-day accumulation area or satellite accumulation area.

6.15.3 **PLACE** bagged waste container on a cart and then:

- a. **HAVE** HPT perform a 6-point survey on the container.
 1. **FILL** out the 6-point survey data sheet.

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NOTE

HMC personnel are responsible for logging all mixed waste containers into the 11A 90-Day Accumulation Area.

- b. **TRANSPORT** container to the 11A 90-Day Accumulation Area as coordinated and directed by the HMC.
- c. **PROVIDE** HMC with completed 6-Point Survey data Sheet for the container.

6.16 Filling out the 6-Point Survey Data Sheet

NOTE

- 6-Point Survey Data Sheets (Appendix E) may be obtained from HMC office.
- HMC will assign the carton and CIN in space provided at top of sheet.

- 6.16.1 **CIRCLE** the size of the inner container used or **PRINT** the size of the container in the "other" space provided.
- 6.16.2 **INDICATE** the number of layers of lead and/or rubber shielding used.
 - a. IF none is used
THEN CIRCLE "0".
- 6.16.3 **CIRCLE** the size of the outer container used.
 - a. IF no outer container is used
THEN CIRCLE "NO CAN".
- 6.16.4 **INDICATE** the number of layers of lead and/or rubber shielding used.
 - a. IF none is used
THEN CIRCLE "0".
- 6.16.5 **FILL IN** the waste percentages box at the bottom of the page.
 - a. **RECORD** the gross weight of the inner container.
 - HMC will figure the weight of the waste in the container.

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NOTE

Percentages DO NOT include shielding, inner or outer containers.

- b. **ESTIMATE** the percentage of each waste type in container by volume.
- c. **WRITE** in room number or area in which the waste was generated.
- d. **WRITE** in the tank number of the sample from which the waste was generated.
 - When more than two tanks are present use the 219-S as the tank number.
- e. **WRITE** in the date in which the form was filled out.
- f. **PRINT** name and sign the sheet.

6.16.6 **ASSIGN** the carton number and CIN in space provided at top of sheet.

6.16.7 **ATTACH** the 6-Point Survey Data Sheet to container and give to HMC.

7.0 RECORDS

Waste Contents Sheet and 6-Point Survey Data Sheet are records maintained by HMC.

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Appendix A. Example of Magnetic Compactible Waste Contents Sheet Sign

COMPACTIBLE WASTE CONTENTS SHEET

Process Knowledge Documents
100% Compactable Waste Including;

PAPER
PLASTIC
SURGEONS GLOVES
CLOTH

HNF-5443, rev. 0
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Appendix C. Example of 222-S Hazardous Waste Labels

(Pink background, black lettering)

<p>HAZARDOUS WASTE</p> <p>F-LISTED MIXED WASTE</p> <p>ROOM NO.____ HOOD NO.____</p> <p>WASTE CODES: F001, F002, F003, F004, F005</p> <p>TOXIC</p>
--

(Periwinkle background, black lettering)

<p>HAZARDOUS WASTE</p> <p>MIXED WASTE</p> <p>ROOM NO.____ HOOD NO.____</p> <p>WASTE CODES:_____</p>

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Appendix D. Waste Container Preparation

Inner Container

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Appendix E. Example of 6 Point Survey Data Sheet

6 POINT SURVEY DATA SHEET

CARTON NO. _____		CIN: _____
SURVEY POINT	READING AT SURFACE (mRad/hr)	
# 1	_____	
# 2	_____	
# 3	_____	
# 4	_____	
# 5	_____	
# 6	_____	

HPT SURVEY:

NAME (print) _____

SIGNATURE _____

DATE _____

HMC PERSONNEL LOGGING WASTE INTO SATELLITE DRUM

NAME (print) _____ (signature) _____

(circle one)
Inner Container: # 5 # 10 # 1 gal ice cream other _____
Lard Can # of layers 1/8" (.32 cm) thick

LEAD SHIELDING (SIDE, TOP & BOTTOM) 0 1 2 3 other _____

RUBBER SHIELDING (SIDE, TOP & BOTTOM) 0 1 2 3 other _____

(circle one)
Outer Container: # 5 # 10 # NO CAN
Lard Can # of layers 1/8" (.32 cm) thick

LEAD SHIELDING (SIDE, TOP & BOTTOM) 0 1 2 3 other _____

RUBBER SHIELDING (SIDE, TOP & BOTTOM) 0 1 2 3 other _____

Percentages DO NOT include shielding, inner or outer containers										
PERCENTAGES ARE BASED ON VOLUME										
Size Inner Can	Gross Wt. Kg	Waste Wt. Kg	Paper	Rubber	Plastic	Cloth	Glass	Metal	K.L.	Misc.
Room _____			Tank No. _____			Date _____				
Print Name _____			Signature _____							

Appendix B
LO-160-101
1E-2 Hot Cell Airlock
Operations

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LO-160-101

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APPROVAL DESIGNATOR ES

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Revision Status

<u>Change Level</u>	<u>Date</u>	<u>Change Document</u>	<u>Page(s)</u>	<u>Description</u>
F-1	11/10/98	98-67	All	Incorporate Field Change 222-98-105 to incorporate enhanced radiological work planning for sample portions. Reformat.
G-0	11/12/98	98-26	All	Changes to maintain compliance with current operating procedures.
H-0	03/23/99	99-07	All	Consolidate objectives, activities of two procedures into one and limits the application of this procedure to only one hotcell and its airlock.
H-1	06/09/99	99-40	1	Change from reference to continuous use.
H-2	11/19/99	99-140	6	Add procedure step caveat

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1.0 PURPOSE AND SCOPE

This procedure describes steps necessary to perform operations involving use or change in configuration of 1E-2 airlock. The scope of this procedure includes all aspects for airlock utilization including: load-in and load-out operations via airlock transfer tray, load-in and load-out operations via transfer-tube, and airlock configuration to support either of these modes.

2.0 REFERENCES

Not applicable.

3.0 PRECAUTIONS AND LIMITATIONS

This procedure shall be used only in situations where the field dose rate is less than 50 mrem/hr at 30 cm; or as specified in the RWP; or whichever is lower.

Application of this procedure requires authorization by one or more Radiation Work Permits (RWP).

This procedure should be performed under direction of a cognizant lead worker who shall be present at work site during entire work function.

The lead worker should conduct a thorough pre-job briefing with all task personnel present including Health Physics Technician (HPT) immediately before performing job task.

4.0 SPECIAL TOOLS AND EQUIPMENT

4.1 Core Sampler Load in:

1E Bridge Crane (trained operator use only)

Airlock configured with transfer tube (PVC, 30" x 4-1/2" OD)

Box wrench to fit cask bolt(s)

Cask table

Gloves: leather or canvas

Hex wrench to fit cask plug

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Plastic bags: yellow, assorted capacities

Plastic sheeting or Kraft paper

Plastic sleeving: clear, 6-foot sections

Ramrod w/plastic sheath, steel, 1/4" diameter, 5 feet long

Safety shoes

Tape: masking and green duct

4.2 Spent Sampler Load-Out:

Plastic sheeting/Kraft paper

Plastic bags: yellow, assorted capacities

Plastic sleeving: yellow, reinforced

Plastic sleeving: clear, six foot sections

Tape: masking and green duct

Airlock configured with transfer tube (PVC, 30" x 4-1/2" OD)

4.3 Transfer Tray Operations

Plastic sheeting/Kraft paper

Plastic bags: yellow, assorted capacities

Tape: masking and green duct

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5.0 PREREQUISITES

Sections 6.1 through 6.5 of this procedure each contain steps necessary to complete the particular task. Therefore, tasks involving any of these sections may be performed alone or in any sequence necessary provided that all the steps in each section are performed precisely as ordered.

Work requiring overhead bridge crane use shall commence with a pre-use crane and rigging inspection per LO-161-110. Only one qualified person should be designated as hoist operator and shall be the only person operating the hoist during the job task. Furthermore, the hoist operator should know the hoist emergency circuit breaker location.

Personnel involved in this job task should wear safety shoes and canvas or leather gloves where appropriate when moving and hoisting casks.

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6.0 INSTRUCTIONS

NOTE:

Sections, or steps within sections, of this procedure may be performed out of sequence, as required.

6.1 Replacement of Airlock Plug with PVC Transfer-Tube

- 6.1.1 REQUEST HPT to survey airlock and vicinity for dose rate and contamination.
 - a. CONSULT HPT for radiological conditions and instructions.
- 6.1.2 REMOVE plug from outer airlock door.
 - a. DECONTAMINATE or DOUBLE-BAG plug as necessary.
 - b. STORE plug as appropriate.
 - c. DECONTAMINATE airlock as necessary.
- 6.1.3 INSERT PVC transfer tube (without cap) into 1E-2 airlock through port in outer airlock door.
- 6.1.4 TAPE one end of a 6-ft section of 12-in. diameter polyethylene sleeve to airlock door or barrel of PVC transfer tube.
- 6.1.5 HORSETAIL other end of polyethylene sleeve.
- 6.1.6 PLACE cap over end of transfer-tub and polyethylene sleeve.
- 6.1.7 REQUEST HPT to survey area for contamination.
 - a. DECONTAMINATE are as necessary.
 - b. PERFORM housekeeping as appropriate.
 - c. DISPOSE of accumulated waste per LO-100-151.

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6.2 Replacement of PVC Transfer-Tube with Airlock Plug

- 6.2.1 REQUEST HPT to survey the airlock and vicinity for dose rate and contamination.
 - a. CONSULT HPT for radiological conditions and instructions.
 - b. PULL PVC transfer-tube into plastic sleeving attached to outer airlock door until transfer-tube is free of the penetration.
- 6.2.2 DETACH plastic sleeving from outer airlock door.
- 6.2.3 REQUEST HPT to survey bagged transfer tube for dose rate and contamination.
 - a. DECONTAMINATE and store transfer-tube as appropriate.
- 6.2.4 REQUEST HPT to survey airlock for contamination.
 - a. DECONTAMINATE area as necessary.
- 6.2.5 INSERT plug into outer airlock door.
- 6.2.6 REQUEST HPT to survey area for contamination.
- 6.2.7 DECONTAMINATE area as necessary.
 - a. PERFORM housekeeping as appropriate.
 - b. DISPOSE of accumulated waste per LO-100-151.

6.3 Core Sampler Load-in

- 6.3.1 PLACE appropriately sized polyethylene bag into waste collection can attached to cask table to collect any particulates, liquids, or loose materials which may fall out of the cask.
- 6.3.2 REQUEST HPT to survey cask and airlock vicinity for dose rate and contamination.
 - a. CONSULT HPT for radiological conditions and instructions.
- 6.3.3 REMOVE bail bolts.

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- 6_3_4 **REQUEST** HPT to survey bail bolts for contamination.
 - a_ **DECONTAMINATE** bail bolts as necessary.
- 6_3_5 **ATTACH** hoist to cask bail.
 - a_ **APPLY** duct tape to lifting hook to "mouse" the hook closed.
 - b_ **LIFT** cask out of dolly.
 - c_ **REMOVE** dolly from under cask.
- 6_3_6 **RELEASE** spring locks on cask bail.
- 6_3_7 **LOWER** cask horizontally onto cask table.
- 6_3_8 **PLACE** end of 12-inch diameter sleeve over end of closed cask and tape to base of cask flange.
- 6_3_9 **PULL** remaining sleeve over cask until sleeve is bunched, leaving cask lid clear for removal.
- 6_3_10 **REMOVE** PVC cap.
- 6_3_11 **REQUEST** HPT to survey PVC cap for contamination.
 - a_ **DECONTAMINATE** PVC cap as necessary.
- 6_3_12 **PLACE** any items suitable for load-in into transfer-tube.
- 6_3_13 **UNTAPE** end of old sleeving from face of outer airlock door.
- 6_3_14 **REMOVE** lid bolts.
- 6_3_15 **REQUEST** HPT to survey lid bolts for contamination.
 - a_ **DECONTAMINATE** lid bolts as necessary.
- 6_3_16 **REMOVE** cask lid.

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- 6_3_17 **REQUEST** HPT to survey cask lid for contamination.
- a_ **DECONTAMINATE** as necessary.
- 6_3_18 **ALIGN** cask with transfer-tube opening.
- 6_3_19 **PULL** sleeving over cask/transfer-tube interface and tape to face of airlock door.
- 6_3_20 **HOLD** absorbent smear material under cask plug to catch loose material.
- a_ **REMOVE** plug from bottom of cask, using appropriate wrench.
- 6_3_21 **REQUEST** HPT to survey plug for contamination.
- a_ **DECONTAMINATE** plug and cask as necessary.
- 6_3_22 **POSITION** plastic-covered ramrod through bottom of cask.
- 6_3_23 **USE** ramrod to push liner/sampler assembly into transfer-tube.
- 6_3_24 **OPEN** inner airlock door.
- 6_3_25 **CONTINUE** pushing liner/sampler assemble into transfer-tube.
- 6_3_26 **PULL** liner/sampler assemble completely into hot cell.
- 6_3_27 **CLOSE** inner airlock door.
- 6_3_28 **RETRACT** ramrod into its sleeving.
- 6_3_29 **DECONTAMINATE** ramrod and sleeving as appropriate.
- 6_3_30 **SCREW** plug back into cask.
- 6_3_31 **DECONTAMINATE** bottom of cask as necessary.
- 6_3_32 **UNTAPE** polyethylene sleeve from cask.
- a_ **HORSETAIL** end of polyethylene sleeve.
- 6_3_33 **REPLACE** cap over transfer-tube.

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- 6_3_34 **REPLACE** lid on cask.
 - a_ **REPLACE** lid bolts (finger tight).
- 6_3_35 **LIFT** cask from cask table.
 - a_ **RETURN** cask to vertical orientation.
- 6_3_36 **REQUEST** HPT to survey cask for external removable contamination.
 - a_ **DECONTAMINATE** exterior of cask as appropriate for release from contamination area.
- 6_3_37 **LOWER** cask vertically back into dolly.
 - a_ **DISCONNECT** hoist from cask bail.
 - b_ **REPLACE** bolts in bail (finger tight).
- 6_3_38 **STORE** cask and dolly as appropriate.
 - a_ **APPLY** identification labels and RADCON survey labels (as directed by HPT) as appropriate for release from contamination area.
- 6_3_39 **REQUEST** HPT to survey airlock vicinity and work area for contamination.
- 6_3_40 **DECONTAMINATE** area as necessary.
- 6_3_41 **PERFORM** housekeeping as appropriate.
- 6_3_42 **DISPOSE** of accumulated waste per LO-100-151.
- 6_4 **Unloading of Sampler and Liner from 1E-2 Hot Cell**
 - 6_4_1 **REQUEST** HPT survey airlock vicinity for dose rate and contamination.
 - a_ **CONSULT** HPT for radiological conditions and instructions.
 - 6_4_2 **REMOVE** cap from transfer-tube.
 - 6_4_3 **PUSH** spent sampler/liner into transfer-tube.

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- 6.4.4 **CLOSE** inner airlock door.
- 6.4.5 **PULL** sampler/liner out of transfer-tube and into polyethylene sleeve.
- 6.4.6 **TWIST** sleeving closed approximately 4-6 inches beyond open end of sampler/liner.
- 6.4.7 **APPLY** duct tape across twist and cut across tape.
 - a. **COVER** each end of horsetailed sleeve with piece of tape.
- 6.4.8 **DOUBLE-BAG** sampler/liner with another polyethylene sleeve using yellow mesh reinforced plastic material.
 - a. **HORSETAIL** both ends of outer sleeve (bag).
 - b. **APPLY** extra duct tape to each end of the double-bagged sampler/liner to prevent chaffing of the plastic sleeving during handling.
- 6.4.9 **REQUEST** HPT to survey double-bagged sampler/liner for exterior removable contamination and dose rate.
 - a. **DECONTAMINATE** double-bagged sampler/liner as appropriate for release from the contamination area.
 - b. **APPLY** identification labels and RADCON survey labels (as directed by HPT) as appropriate for release from contamination area.
 - c. **STORE** double-bagged liner and sampler as appropriate.
- 6.4.10 **REPLACE** PVC cap on transfer-tube.
- 6.4.11 **REQUEST** HPT to survey airlock vicinity and work area for contamination.
 - a. **DECONTAMINATE** area as necessary.
 - b. **PERFORM** housekeeping as appropriate.
 - c. **DISPOSE** of accumulated waste per LO-100-151.

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6.5 Airlock Transfer Tray Load-In Operations

- 6.5.1 REQUEST HPT to survey airlock vicinity for dose rate and contamination.
 - a. CONSULT HPT for radiological conditions and instructions.
- 6.5.2 ENSURE airlock inner door is completely closed.
- 6.5.3 POSITION transfer tray near airlock outer door.
- 6.5.4 OPEN airlock outer door.
- 6.5.5 REQUEST HPT to survey airlock vicinity for dose rate and contamination.
- 6.5.6 CONSULT HPT for radiological conditions and instructions.
- 6.5.7 PLACE toweling on tray.
- 6.5.8 PLACE materials to be loaded in onto tray so it can be easily grasped with manipulator.
- 6.5.9 CLOSE airlock outer door.
- 6.5.10 OPEN airlock inner door.
- 6.5.11 EXTEND transfer tray into hot cell.
- 6.5.12 REMOVE material from transfer tray.
- 6.5.13 RETRACT transfer tray back into airlock.
- 6.5.14 CLOSE airlock inner door.
- 6.5.15 OPEN airlock outer door.
- 6.5.16 REQUEST HPT to survey airlock vicinity for dose rate and contamination.
 - a. CONSULT HPT for radiological conditions and instructions.
- 6.5.17 REMOVE toweling from airlock.
- 6.5.18 DECONTAMINATE airlock and tray as necessary.

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- 6_5_19 **CLOSE** airlock outer door.
- 6_5_20 **REQUEST** HPT to survey airlock vicinity and work area for contamination.
 - a_ **DECONTAMINATE** area as necessary.
 - b_ **PERFORM** housekeeping as appropriate.
 - c_ **DISPOSE** of accumulated waste per LO-100-151.

6_6 Airlock Transfer Tray Load-Out Operations

- 6_6_1 **REQUEST** HPT to survey airlock vicinity for dose rate and contamination.
 - a_ **CONSULT** HPT for radiological conditions and instructions.
- 6_6_2 **ENSURE** airlock inner door is completely closed.
- 6_6_3 **OPEN** airlock outer door.
- 6_6_4 **REQUEST** HPT to survey airlock vicinity for dose rate and contamination.
 - a_ **CONSULT** HPT for radiological conditions and instructions.
- 6_6_5 **PLACE** toweling on tray to prevent spread of contamination.
- 6_6_6 **CLOSE** airlock outer door.
- 6_6_7 **OPEN** airlock inner door.
- 6_6_8 **EXTEND** tray into hot cell.
- 6_6_9 **PLACE** material onto tray.
- 6_6_10 **RETRACT** transfer tray into airlock.
- 6_6_11 **CLOSE** inner airlock door.
- 6_6_12 **OPEN** outer airlock door.

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- 6_6_13 **REQUEST HPT to survey airlock vicinity for dose rate and contamination.**
 - a_ **CONSULT HPT for radiological conditions and instructions.**
- 6_6_14 **REMOVE items according to good RADCON practice.**
 - a_ **DECONTAMINATE airlock as necessary.**
- 6_6_15 **CLOSE outer airlock door.**
- 6_6_16 **REQUEST HPT to survey airlock vicinity and work area for contamination..**
 - a_ **DECONTAMINATE area as necessary.**
 - b_ **PERFORM housekeeping as appropriate.**
 - c_ **DISPOSE of accumulated waste per LO-100-151.**

7.0 RECORDS

Not Applicable.

Appendix C
LT-160-101
Retained Gas Sampler
Operation

WASTE MANAGEMENT LABORATORY

Laboratory Technology Procedure

Issued By: **L. L. Lockrem**
Manager

Title: **RETAINED GAS SAMPLER EXTRACTION
SYSTEM OPERATION**

B. E. Hey
Author

Approval Designator S

1.0 SUMMARY

This procedure is used to extract and separate retained gas from a waste tank core sample for quantitative analysis. The waste tank core segment sample is delivered to the laboratory in a special gas tight waste tank core sampler that contains the gas phase component of waste tank sample material along with the bulk material. Normally the sampler is loaded into the 1E-2 hot cell according to procedure LO-160-101, Core Segment Load-in and Load-out of 1E-2 Hot Cell.

The Sampler is coupled to the extractor of the Retained Gas Sampler (RGS) system in a way that ensures vacuum integrity. Then, the extractor is evacuated. Vapors contained inside the core sampler are extruded into the extractor with the bulk sample material then are pumped, using a liquid mercury positive displacement pump, to collection canisters located outside the hot cell. A high-efficiency in-line depth filter prevents the transfer of radioactive particles to the collection canisters. The collection canisters can be removed from the system for analysis of the gases.

2.0 APPLICATIONS

This procedure applies to activities involved in processing RGS samples and using the RGS Extraction System to identify and quantify vapors associated with core segments.

3.0 LIMITATIONS

The complete RGS system consists of more than 20 valves and includes filters, vacuum pumps, liquid mercury, pressure transducers, and thermocouples among its components. Because the system can be damaged if it is operated incorrectly, only persons trained to operate the system shall perform this work.

Approved for Use

Release Date

11/15/99

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4.0 QUALITY CONTROL PROTOCOL

Data obtained during this procedure include temperature and pressure readings. Log sheets on which data are recorded are pasted into a controlled laboratory notebook. (Usually telefacsimiles of the data sheets are transmitted out of the laboratory to avoid time and expense of radiological survey.)

Temperature measurements are provided through use of K-type thermocouples. Calibration of the temperature sensing capability of the RGS system is not routinely performed; rather several readings are acquired simultaneously from different locations of the RGS system for redundant verification.

Four of the five pressure measurements are made using instruments which are calibrated annually; the fifth (non-calibrated) pressure measuring instrument is used for convenient indication only. Usually the calibrations are performed by the manufacturer at the factory.

5.0 SAFETY (Continued)

5.0 WASTE GENERATION

No solid chemical waste is generated when performing this procedure since spent sample material is saved for other unrelated studies. Contaminated rinse water which is generated during preparation and post processing washdowns is expelled to the hot cell drain. The Building Operations Manager should be notified prior to washdown activities or anytime water is used in the hot cell.

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4.0 QUALITY CONTROL PROTOCOL (Continued)**6.0 SAFETY**

The RGS extraction system may involve using compressed gases which are used to purge the system periodically. Although these gases are regulated to low and easily manageable pressures, care should be exercised to ensure the system does not exceed 133 kPa (4.6 psig) so that sensitive instruments will not be damaged. When not in use the cylinders' valves should be closed.

The RGS extraction system uses liquid mercury in the displacement pump. Although the mercury is safely contained within the system, care should be exercised when performing plumbing operations on the system because beads of mercury may be encountered when breaching some system lines. If a spill of metallic mercury does occur, the Shift Operations Manager (SOM) must be notified and a spill clean-up plan initiated according HNF-IP-0263-222S, Building Emergency Plan for 222-S Complex.

In the event that the system lines and plumbing should be breached to effect repair or replacement of instruments or fittings, an HPT must be present to conduct surveys to monitor and control the spread of radioactive contamination.

7.0 REAGENTS

Ammonium hydroxide solutions ~0.04M, solutions containing specific non-radioactive isotopes.

8.0 EQUIPMENT

Gas Sample Collection Canisters (H-2-821611)

Laboratory Notebook, controlled

Retained Gas Sampler System (H-2-821609)

Manifold System Control Cabinet (H-2-85291) and Contents:

Data Acquisition Control Unit, DACU

Mercury Positive Displacement Pump

Pressure Transducers and Display Panels

Motor Controllers Computer Power Supplies

Turbo-Molecular Vacuum Pump

Rotary Vane Vacuum Pump

Extruder Hydraulic Pump

Temperature Controlled Water Bath (Heat/Chiller)

Small or Consumable Items Required inside the hot cell:

Grommet Pick

Piston Positioning Sleeve

Poly Bottle (1L)

Ring Positioner (H-2-821610)

Seal (H-2-821610)

Spring Assembly (H-2-85449-7)

Universal Press Nut (H-2-85441-8)

Wire/cable cutters

Wrench to remove quadralatch cap (H-2-690140-23)

Q or W canister, if desired

Sample Vial (20 mL), if desired

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9.0 PROCESS OVERVIEW

The RGS Extraction System is designed to extract vapors from core samples of tank waste material. The extractor is temperature controlled and contains a mixing rotor for continued stirring of the sample during the entire processing session. The RGS sampler (which contains the sample material) is loaded into hot cell 1E-2, prepared, and mounted onto the RGS system in a vacuum tight configuration. The RGS system is then evacuated down to less than 10 Pa². After system checks have been performed, the sampler valve is opened and the sample material is pushed (using the extruder system) into the extractor section of the RGS system.

A tube leads from the extractor, out of the hot cell, through a filter, and eventually to a liquid mercury positive displacement pump so that vapors can be pumped from the extractor to various (removable) vapor collection canisters for analysis elsewhere².

9.1 Physical Configuration

Below are four schematic drawings of the RGS system. Figures 1 and 2 are line drawings that illustrate the general physical arrangement of the RGS system: Figure 1 shows the arrangement of sections outside the hot cell and Figure 2 shows the arrangement of sections inside the hot cell.

Outside the hot cell is situated: the control cabinet, (which houses the instrumentation, collector side tube and fittings, auxiliary manifold, diluent reservoir, mercury positive displacement pump, data acquisition unit DACU, computer), heater/chiller unit, vacuum pumps (turbo-molecular and rotary vane), and the extruder hydraulic pump.

Inside the hot cell is situated the extruder device, the extractor and stirrer mechanism, and the secondary volume.

Figure 3 is a schematic diagram of the system that shows, in closer detail, the configuration of the plumbing, instrumentation, and all valves located outside the hot cell.

Figure 4 is a schematic diagram (in similar detail) of the system components which are situated inside the hot cell including: the extractor, secondary volume, and associated manifold.

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9.0 PROCESS OVERVIEW (Continued)

Figure 1: Overall RGS System Arrangement Outside Hot Cell

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9.0 PROCESS OVERVIEW (Continued)

Figure 2: Overall RGS System Arrangement Inside Hot Cell

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9.0 PROCESS OVERVIEW (Continued)

Figure 3: Detailed Schematic of RGS System Outside Hot Cell

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9.0 PROCESS OVERVIEW (Continued)

Figure 4. Detailed Schematic of RGS System Inside Hot Cell

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9.0 PROCESS OVERVIEW (Continued)

9.2 Section, Volume, and Valve Arrangement

In order to draw quantitative conclusions from the pressure and temperature data acquired during the entire processing session, it is important to know the volumetric dimensions of the vapor containing sections of the RGS system. Therefore, volumetric capacities of the important sections of the RGS system are provided in Tables 1 and 2 below.

Table 1 lists the measured volumes for various fixed-volume sections of the RGS system. These volumes were measured by a combination of gravimetry (weighing water) and ideal gas law calculations.

Table 2 shows how the various sections are concatenated during various processing phases, e.g. bulk vapor extraction, exsolution, diluent addition, etc.

Also, a list of each valve is provided in Table 3 which describes how each valve is associated with the important sections of the RGS system and their function.

TABLE 1. SYSTEM SECTION VOLUME ASSIGNMENT

Section	Description	Volume (cc)
i	Extractor to Valve T	1290
ii	Valve T to Valves B and Z	262
iii	Valve B to Valves C, F, G and Port L	15
iv	Displacement Chamber and Sight Glass	464
v	Effective Hg Pump Displacement	438
vi	Auxiliary Sample Manifold (between Valves C,D,K2,Y)	10
vii	Collector Manifold (Valves F/G to H's, K1)	34
viii	Canister Connector (e.g. Valve H to Canister Valve)	2
ix	Secondary Volume	321
diluent	Volume of Added Diluent (mL)	300 ³
sample	Bulk Sample Volume Determination	BSVD ⁴
D	Auxiliary Manifold Vapor Canister (Canister D)	D ⁵
J1	Collection Canister J1	J1
J2	Collection Canister J2	J2
J3	Collection Canister J3	J3

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9.0 PROCESS OVERVIEW (Continued)

TABLE 2. CONCATENATION OF TABLE 1 SECTIONS

PROCESS PHASE	CONCATENATION
BULK VAPOR EXTRACTION	
Extractor side (to Valve B)	i+ii-BSVD
Unpumped Vapor Sample Capture	i+ii+iii+iv+vii+(3•viii)+D+J1+J2+J3-BSVD
Extractor Side (to Valves F/G)	i+ii+iii+vi-BSVD
Collector Side (filled Sight Glass)	vii+(3•viii)+J1+J2+J3
EXSOLUTION	
Extractor	i+ii+iii+vi-BSVD
Collector Side (to Valves H1,H2,H3)	vii
Collector Side (plus Canister Volumes)	vii+(3•viii)+J1+J2+J3
EQUILIBRATED VAPOR EXTRACTION	
Extractor Side	i+ii+iii+vi-BSVD
Collector Side	vii+(3•viii)+J1+J2+J3

TABLE 3. VALVE AND PORT DESIGNATION AND FUNCTION

VALVE or PORT	SEQUENCE	FUNCTION
Valve A	R,T,Z/A/B	main sample line isolation valve
Valve B	A/B/C,E,L	wet transducer isolation valve
Valve C	B,E,L/C/D,K2,Y	auxiliary manifold isolation valve
Port D	C,K2,Y/D	sample port where D canister fits
Valve D	Valve D/Port D	valve on the canister itself (D canister)
Valve E	B,C,L/E/F,G	mercury pump isolation valve
Valve F	E,G/F/G,H1,H2,H3	mercury pump bypass valve
Valve G	E,F/G/U/G/F,H1,H2,H3	mercury pump 3-way valve
Valve H1	F,G,H2,H3/H1/Port J1	sample port J1 isolation valve
Valve H2	F,G,H1,H3/H2/Port J2	sample port J2 isolation valve
Valve H3	F,G,H1,H2/H3/Port J3	sample port J3 isolation valve
I		no valve or port designated with this letter
Port J1	H1/Port J1	sample port where vapor canister fits
Port J2	H2/Port J2	sample port where vapor canister fits
Port J3	H3/Port J3	sample port where vapor canister fits
Valve J1	Valve J1/Port J1	valve on the canister itself (J canister)
Valve J2	Valve J2/Port J2	valve on the canister itself (J canister)
Valve J3	Valve J3/Port J3	valve on the canister itself (J canister)
Valve K1	F,G,H1,H2,H3/K1/M1,M2	system main vacuum isolation valve
Valve K2	C,D,Y/K2/M1,M2	auxiliary manifold vacuum isolation valve
Port L	B,C,E/L	sample port (non-purgeable)
Valve M1	K1,K2,M2/M1/pump	rotary vane vacuum pump isolation valve
Valve M2	K1,K2,M1/M2/pump	turbo-mol. vacuum pump isolation valve
Valve N1	regulator/N1/N2,N3,N4	helium supply isolation valve
Valve N2	regulator/N2/N1,N3,N4	argon supply isolation valve
Valve N3	Port N/N3/N1,N2,N4	isolation valve (used for air inlet)
Valve N4	Y,Z/N4/N1,N2,N3	purge manifold isolation valve
O		no valve or port with this letter
P		obsolete PQ canister valve
Valve Q	Q/2nd-volume	2nd volume port (no valve present)
Valve R	2nd-volume,S/R/A,T,Z	2nd volume isolation valve
Valve S	extr.,T/S/2-vol,R	2nd volume/extractor bypass valve
Valve T	extr.,S/T/A,R,Z	extractor isolation valve
Valve U	G/U/Hg Reservoir 2-	liquid mercury cut-off valve
Valve V	Vol/V/outlet port	2nd volume port to (beaker) valve
Port W	extractor port	extractor liquid addition port
Valve W1	extr/W1/W2,W3	extractor liquid addition valve
Valve W2	W1/W2/W3	liquid addition purge valve
Valve W3	Diluent Reservoir/W3	Diluent supply shut-off valve
Valve X	sampler:x/extractor	sampler valve itself
Valve Y	N4,Z/Y/C,D,K2	aux. manifold purge-gas isolation valve
Valve Z	R,T/Z/N1,N2,N3	main purge line isolation valve

9.3 Description of Process

9.3.1 Phase I, Extrusion and Bulk Vapor Extraction

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9.0 PROCESS OVERVIEW (Continued)

After the evacuation, preliminary leak rate and instrument offset determinations have been performed, preparations need to be made to begin sample processing. The first phase in the processing scheme involves extrusion of the sample and extraction of the unbound vapors (called bulk vapors). These preparations include:

- re-acquisition of preliminary vacuum, 10 Pa or less
- extractor cooled to 10 °C
- automatic data acquisition initiated and saving to file
- stirrer turning at 3 rpm (mixer speed)
- secondary volume and vacuum pumps isolated

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9.0 PROCESS OVERVIEW (Continued)

After these preparations have been completed and the desired situations exist, the sampler valve is opened and the sample material is extruded into the extractor⁶.

Once the sample material is fully transferred from the sampler to the extractor (via actuation of the hydraulic extruder), a short pause in processing activity is made to allow the newly expanded vapors to equilibrate with the surroundings and achieve thermal equilibrium. It is assumed that the sudden expansion of the sample material into the evacuated volume of the extractor (and associated tubing), the shear stress of the sample during the extrusion, and the continued stirring of the sample in the extractor provides sufficient agitation to free the majority of the retained gas from the bulk of the sample. Thus, temperature and pressure measurements should be continually monitored through automatic data logging and manual recording in the process log.

The initial pressure of the vapor is the first number that should be recorded under "Bulk Vapor Extraction" in the process log (Appendix A contains a sample of a process log data sheet). This is the pressure of the vapor (after achieving thermal equilibrium) in the concatenated sections of the system from the extractor to valve B (closed). Table 1 lists the volumes of the concatenated sections of the system for each phase of the process.

The first vapor sample to be acquired is captured without pumping the retained gas and is collected in "Canister D" at the auxiliary (q.v.) manifold. These vapors are acquired when valve B is opened and the vapor is allowed to fill the system up to valves F and G. Collection of the sample material in this first vapor sample capture routine occurs only as a result of allowing the vapors of the sample to fill the evacuated sections of the RGS system and the collection canister as certain valves are opened. This vapor sample contains the minimum concentration of condensable vapor components. This is to say, that the mole fractions of individual gas species are least biased in the concentrations of ammonia, water vapor, and possibly even nitrous oxide. Thus, this first sample probably represents the truest proportions of gas species that are actually present as gases in the sample.

After a portion of this "unpumped" vapor is isolated for analysis (by closing the valve of the D canister which is located at the auxiliary manifold), pumping of the extractor should commence right away in a series of thirteen pump cycles to fill the collection canisters, fitted onto the collector side manifold. The mechanism by which these extraction pump cycles are performed is simple positive displacement using liquid mercury as the volume displacement medium and a three-way valve to direct the flow. In this way, vapor is pumped from a low pressure (1-5 kPa) extractor side to a high pressure (> 5 kPa) collector side by allowing a column of liquid mercury to repeatedly rise and fall within the confines of a cylindrical displacement chamber, approximately 440 mL in capacity. Vapor is allowed into and out of the displacement chamber by cycling a three-way valve back and forth between the collector and extractor sides of the displacement pump in concert with the rise and fall of the column of mercury (about 500 mL). The collected vapor is subsequently analyzed quantitatively for gas species composition. Usually, on the collector side of the pump, there are three individual collection canisters⁸ connected to the collector manifold.

It was decided that thirteen extraction cycles was sufficient for the complete transfer and collection of the retained gas from the extractor to the collector because this would, theoretically, transfer at least 96% of the total amount of vapor in the extractor to the collector, regardless of the initial extractor pressure. There was the concern that the "representativity" of the collected vapors would diminish, however, as a result of too much pumping since the collected vapors would become more and more biased with ammonia and water vapor which exsolute from the sample with every pump. Development exercises has shown⁹ that thirteen (13) pump cycles was an appropriate balance between too little and too much pumping.

Throughout the thirteen extraction cycles, the pressure and temperature of both the extractor and collector sides of the RGS system are recorded. In doing this, a profile of the temperature and pressure is obtained which may also provide a qualitative profile of this sample material's retained gas characteristics in so far as its "free releasable" (i.e. non-dissolved) retained gas properties.

The system's valve and physical configuration for this "Bulk Vapor Extraction" session is as follows:

- a. Secondary volume (q.v.) is isolated from the extractor manifold.
- b. Extractor manifold consists of the extractor main volume and all lines running from valve Z (closed) to valve F (closed) and valve G (turned to the collector side). Valve C to the auxiliary vapor sample manifold is open but valve D (vapor sample canister) is closed before the 13 pump extraction routine begins.
- c. During the extraction pumping, valves H1, H2, and H3 are open on the collector manifold as are valves J1, J2, and J3 on the vapor sample canisters.
- d. The extractor is maintained at about 10 °C. The rest of the RGS system is subject to ambient room temperature (usually about 25 °C).

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9.0 PROCESS OVERVIEW (Continued)

- e. The stirrer turns at 3 rpm (mixer speed).

9.3.2 Phase II, Exsolution

After the thirteen-cycle extraction process is completed, an exsolution profile is generated by monitoring the system's pressure during an additional nineteen (19) extraction cycles. The purpose of generating an exsolution profile is to determine whether the amount of vapors which exsolute from the sample material during each pump cycle can be quantitatively characterized. This quantitative character could then be applied to the results of the bulk vapor extraction (first 13 cycles) as a correction for acquiring the actual quantity of retained gas from the apparent quantity which was actually determined¹⁰.

To generate the exsolution profile, vapor is pumped from the extractor to the collector while the pressure and temperature of the extractor and collector sides is recorded. The vapor in the collector side is then discarded (in some cases a portion is saved first) and then the mercury pump (through the collector manifold) is purged of any condensed vapor by exhaustively evacuating the section down to at least 10 Pa, before executing another cycle.

The reason for this exhaustive evacuation is to prevent any accumulated condensed vapors from re-evaporating inside the pump chamber, flowing back into the extractor and thus reducing the effectiveness of the pump. Without doing this the "evacuated" pump chamber would be comparable in pressure to the extractor side pressure due to re-vaporization and so very little mass transfer would occur between the extractor and the pump.

During the exsolution profile, portions of the vapor is saved for analysis. A total of nine (9) vapor samples are acquired: three from the first, tenth, and nineteenth extraction cycle. Each of these vapor samples is contained in an individual canister so the proportions of gas species could be examined as a function of the extraction sequence, in triplicate.

The system's valve and physical configuration for this part of the test is as follows:

- a. Secondary volume is isolated from the extractor manifold as before.
- b. Extractor manifold consists of the extractor main volume and all lines running from valve Z (closed) to valve F (closed) and valve G (turned to the collector side). Also, port D of the auxiliary manifold is plugged.
- c. After each vapor collection, to minimize the back flow of residual vapors from the previous extraction cycle, the collector-side manifold and the mercury displacement chamber is evacuated to less than 10 Pa prior to performing the next cycle.
- d. During each extraction cycle, the extracted vapors are pumped with the mercury pump to the collector side manifold while the H valves are all closed. At the end of the 1st, 10th, and 19 cycles, all the H valves are opened and the pressure of the collector manifold is further reduced as the three collection canisters are filled. This subsequent pressure transition is also recorded.
- e. The extractor is maintained at about 10 °C. The rest of the RGS system is subject to ambient room temperature (about 25 °C).
- f. The stirrer turns at 3 rpm (mixer speed).

9.3.3 Phase III, Standard Diluent Addition, Equilibration, and Vapor Extraction

Ammonia vapor is commonly found as a significant component in retained gas. However, due to its high solubility in aqueous media, and because of its affinity for metallic surfaces, it is difficult to obtain quantitative determinations of ammonia as a vapor in this particular mass transport system. As an attempt to compensate for these complications, an addition of 300 mL diluent consisting of approximately 0.04M ¹⁵NH₄OH is added directly to the sample in the extractor (after the bulk vapor extractions and exsolution profile extractions are completed) to function as an internal standard for total ammonia concentration determination in the sample material.

After diluent addition, the diluted sample is allowed to come to equilibrium while being stirred at 3 rpm (mixer speed) for at least 96 hours, after which a set of three vapor samples is acquired (simultaneously) for isotopic mass ratio analysis. The specific analysis to be conducted is the isotope ratio determination (IRD) of the ratio of the concentrations of ¹⁵NH₃ to ¹⁴NH₃ by way of quantitative mass analysis using ultra-sensitive gas phase mass spectrometry. With

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9.0 PROCESS OVERVIEW (Continued)

this determination it is thought that the original concentration of ammonia in the sample can be determined by using the equation:

Equation 1

$$\frac{[^{15}\text{NH}_3]}{[\text{NH}_3]} = \frac{\text{IRD}}{1 + \text{IRD}}$$

Where:

$[^{15}\text{NH}_3]$ = concentration of labeled ammonia
 $[\text{NH}_3]$ = total ammonia concentration
 IRD = $[^{15}\text{NH}_3]/[^{14}\text{NH}_3]$ isotopic ratio determination

Thus, the original amount of NH_3 in the sample can be determined by plugging in the values for $[^{15}\text{NH}_3]$ (the concentration of labeled ammonia in the diluted sample) and using the IRD results and solving for $[\text{NH}_3]$. Inaccuracy with this technique might be that the true concentration of $^{15}\text{NH}_3$ (diluent plus sample) is not accurately known since the diluted sample is still a heterogeneous mixture (non-solution).

The temperature of the extractor is maintained at about 10 °C during the addition of the diluent and the mixture is stirred at 3 rpm (mixer speed) for the duration of the equilibrium interim and during the vapor extraction exercise. The vapor sample canisters are filled simultaneously in order to replicate vapor samples for analysis in triplicate.

Usually, no attempt is made to de-gas the diluent, however, so it is expected that air components (nitrogen, oxygen, argon) should be detected in these subsequent vapor sample acquisitions if analyzed for such species. No effort is made to degas the diluent prior to its use though since the primary objective of this effort is to determine the resultant ratio of the concentration of $^{15}\text{NH}_3$ to $^{14}\text{NH}_3$ and this determination should be unaffected by the presence of other "spectator" gas species.

The system's valve configuration for the addition of the diluent and the equilibration interim:

- The extractor is isolated from the rest of the system by closing valve T.
- The heater/chiller unit is turned off.
- Diluent is added to the extractor through valve W1.
- The extractor contents are stirred at 3 rpm (mixer speed) for at least 96 hours.
- The rest of the RGS system is open to the mechanical vacuum pumps for exhaustive evacuation.

The addition of the diluent to the extractor is performed as follows:

- The diluent supply line is flushed with approximately 100 mL of the diluent solution to purge the lines of "old" diluent and fill with "new" diluent.
- 300 mL of diluent is added directly to the extractor through valve W1. Since valve T is closed, the pressure transducers are blind to this operation. Residual vacuum in the extractor is sufficient to draw the entire 300 mL aliquot of diluent into the extractor through valve W1.
- Extractor temperature is not controlled but the stirrer continues to rotate at 3 rpm (mixer speed).

After 96 hours of equilibration interim, the extractor vapors are pumped to the collector side manifold in the same fashion as the bulk vapor was pumped.

The system's valve configuration for the vapor extraction exercise of the equilibrated vapors is as follows:

- Secondary volume is isolated from the extractor manifold.
- Extractor manifold consists of the extractor main volume and all lines running from valve Z (closed) to valve F (closed) and valve G (turned to the collector side). Also, port D of the auxiliary manifold is plugged.
- The extractor is being stirred at 3 rpm (mixer speed).

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9.0 PROCESS OVERVIEW (Continued)

9.3.4 Phase IV, Bulk Sample Volume Determination

After the sample material is thoroughly exhausted of freely releasable vapors through the bulk vapor extraction, exsolution profile, and equilibrated vapor extractions, it is then possible to conduct the "Bulk Sample Volume Determination" which necessarily involves the addition of air to the extractor. Air (at ambient pressure) is used because contamination of the sample material with air components is no longer a concern and there is no danger of possibly over pressurizing the RGS system such as could happen if bottled gases such as argon or helium were used. The bulk sample volume determination procedure is followed three times for a triplicate determination.

Three main steps must be performed in this experiment:

- An initial low pressure (~4 kPa) measurement of the extractor is recorded and then the extractor is isolated from the rest of the system. The volume of the empty extractor is already known.
- Another section of the RGS system is exposed to the atmosphere and allowed to completely fill to ambient atmospheric pressure (~100 kPa). The volume of this section is also already known.
- Finally, the extractor section is valved in to this second "pressurized" section (after it is isolated from the atmosphere) and the two volumes allowed to equilibrate to a final resultant pressure (somewhere in mid range) and this third pressure measurement is recorded.

The three pressure readings (and associated volumes) can then be applied to Equation 2a (which is derived from the equation for an ideal gas) in order to 1) determine the volume of the diluted sample for use in Equation 1 for the determination of the concentration of ammonia in the sample, and 2) for use in Equation 2b to determine the volume of the sample material itself.

Equation 2a

$$D = E - A \cdot \frac{(P_3 - P_2)}{(P_1 - P_3)}$$

Equation 2b

$$V = D - S$$

Where:

A = volume of the Air let into the filled section
 D = volume of the Diluted sample material
 E = volume of the Extractor vessel (when empty)
 P₁ = initial pressure of the extractor
 P₂ = pressure of the aliquot of air
 P₃ = resultant pressure of the equilibrated system
 S = volume of the Standard diluent added
 V = volume of the Bulk sample (unknown)

10.0 PROCEDURE STEPS (Continued) NOTE: This is the end of the Procedure Steps.

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9.0 PROCESS OVERVIEW (Continued)

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10.0 PROCEDURE STEPS

NOTE: Sections, or steps within sections, of this procedure may be performed out of sequence, as required.

10.1 Sampler Preparation and Pre-Start Configuration Verification**10.1.1 Sampler Preparation**

1. Cut the valve closing cables.
2. Remove the quadralatch cap and mousetrap assembly from the sampler¹¹.

NOTE: The mouse trap assembly includes all the following parts:

Pull Block	(H-2-85097-11)
Spring	(H-2-85097-46)
Stop	(H-2-85097-25)
Plunger	(H-2-85097-27)
Release Disk	(H-2-821608-24)
Retainer Ring	(H-2-821608-41)

3. Slide the ring positioner over the threaded end of the sampler.
4. Insert the piston positioning sleeve into the sampler.
5. Install the universal press nut on the threaded side of the sampler..
6. Remove the tape and spring stops (two) from the sampler.
7. Install the spring assembly through the back of the universal press nut.
8. Place the seal in the extractor sample port or on the valve end of the sampler.
9. Notify the duty shift operations manager before using water which will wash down the hot cell drains.
10. Wash (with water) and dry the valve end of the sampler.
11. Mount the sampler on the extruder and couple it to the extractor.
12. Use the grommet pick to remove the grommet covering the valve opening screw.

10.1.2 Instrument Conditions

1. Ensure rotary vane pump is running and valve M1 is open.
2. Ensure valve M2 (to turbo-molecular vacuum pump) is closed.
3. Ensure all pressure indicator panels are operating.
4. Ensure mercury reservoir is at bottom of travel.

10.1.3 Port and Valve Configuration

1. Ensure sampler is mounted onto the extractor and its valve is closed.
2. Ensure vapor canisters are installed at the "H" ports and at the auxiliary port¹².
3. Ensure the following valve configurations: CLOSED: {M2, N(1,2,3), S, U, W(1,2,3)}; OPEN: {A, B, C, D, E, F, G (to collector), H(1,2,3), J(1,2,3), K(1,2), M1, N4, R, T, Y, Z}¹³.
4. Ensure port Q is plugged.
5. Ensure system is evacuated to less than 0.01 kPa.

10.1.4 Start-Up Sequence

1. Ensure RGS software is running and DACU is operating.
2. Ensure stepper motor controllers are on.
3. Raise mercury reservoir to the 0-point.
4. Raise mercury column to 3/4-point (by opening valve U).
5. Lower mercury reservoir to bottom and Ensure mercury column is falling.

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6. Ensure pressure readings are indicating reasonable values.

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10.2 Processing Sequences

10.2.1 Leak Rate Determination

1. Record core sample identification in notebook or process log sheet.
2. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
3. Close valves: K(1,2), N4, Y, Z.
4. Continue to acquire and log data to file for about 30 minutes.
5. Stop data acquisition, close file, and copy data file to floppy disk.
6. Compute system's leak rate as millimoles per second.

NOTE: Leak rate may be computed as:

$$L = (S \cdot V) / (R \cdot T)$$

Where:

L = leak rate (M mol/sec)
 S = slope of pressure/time curve (kPa/sec)
 V = system volume (2700 cc, conservative)
 R = gas constant (8.314 J/K·mol)
 T = temperature (K)

7. If leak rate is less than 3×10^{-6} mmol/sec then continue; otherwise correct leak rate problem.
8. Record leak rate determination in notebook.

10.2.2 Pressure Transducer Full-scale Zero Offset Check

1. Record the calibration date for all pressure transducers and the DACU instrument in the notebook.
2. Ensure the following valve configurations: CLOSED: {K(1,2), M2, N(1,2,3,4), S, U, V, W(1,2,3), X, Y, Z}; OPEN: {A, B, C, D, E, F, G (collector), H(1,2,3), J(1,2,3), M1, R, T}.
3. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
4. Open valves N3, N4, and Z, wait about 5 seconds then close valve N3.
5. Wait for system's temperature and pressure to stabilize and then record pressure indications in notebook as "High pressure indication."
6. Reduce pressure to ~10 kPa by controlling vacuum with valve K1.
7. Wait for system's temperature and pressure to stabilize and then record pressure indications in notebook as "Medium pressure indication."
8. Reduce pressure to ~1 kPa (controlling with valve K1).
9. Wait for system's temperature and pressure to stabilize and then record pressure indications in notebook as "Low pressure indication."
10. Stop data acquisition, close file, and copy data file to floppy disk.
11. Open valve K1 and allow system's pressure to continue to reduce to less than 0.01 kPa.

10.2.3 Sample Extrusion and Bulk Vapor Extraction

1. Ensure heater/chiller unit is controlling to 10 °C and extractor temperature ~10 °C.
2. Turn on stirrer motor to 60 rpm.
3. Ensure system pressure less than 0.01 kPa.
4. Close valves: N4, K(1,2), Y, and Z.
5. Ensure the following valve configurations: CLOSED: {K(1,2), M2, N(1,2,3,4), U, W(1,2,3), X, Y, Z}; OPEN: {A, B, C, D, E, F, G (to collector), H(1,2,3), J(1,2,3), M1, R, T}.

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6. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
7. Adjust pressure indicator zero offsets as necessary.
8. Close valves R and B.
9. Remove rubber grommet from sampler valve.
10. Open sampler valve (valve X).
11. Extrude sample material into extractor.
12. Wait until system temperature and pressure stabilizes and then record: date, time, event, temperature, pressure in notebook.
13. Open valve B and wait for system's temperature and pressure to stabilize.
14. Record: date, time, event, temperature, pressure in notebook.
15. Close valve D and record: sample/container ID, temperature, and pressure in notebook.
16. Close valve F.
17. Perform the following steps twelve (12) times.
 - a. Raise the mercury reservoir above the 0-point.
 - b. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
 - c. Lower mercury reservoir to bottom of travel.
 - d. Record the system's temperature and pressure in notebook.
 - e. Turn valve G to extractor side.
 - f. Wait until mercury completely evacuates displacement bulb.
 - g. Close valve U.
 - h. Turn valve G to collector side.
 - i. Check off completed cycle number:

1	2	3	4	5	6
7	8	9	10	11	12

18. Raise mercury reservoir above 0-point.
19. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
20. Lower mercury reservoir to bottom of travel.
21. Record temperature and pressure in notebook.
22. Close valves J(1,2,3) and H(1,2,3) and record vapor sample/canister ID, temperature, and pressure in notebook.
23. Stop data acquisition, close file, and copy data file to floppy disk.
24. Close valve B.
25. Remove the vapor canisters from the RGS system.
26. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
27. Place three new canisters (with J valves open) at H ports and plug auxiliary port.
28. Open valves H(1,2,3).
29. Turn valve G to collector side.
30. Ensure the following valve configurations: CLOSED: (B, K2, M2, N(1,2,3,4), R, S, U, V, W(1,2,3), Y, Z); OPEN: (A, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1, T).

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10.3.2 Preparations for Interim Stand-by and Housekeeping.

1. Fit blind sampler (with gasket) onto extractor.
2. Fit bottom flange onto extractor.
3. Open valve T and allow entire system to dry and off-gas in preparation for the next processing session.
4. Ensure new vapor canisters are installed at H ports and at auxiliary port.
5. Ensure following valve configurations: CLOSED: (M(2 or 1), N(1,2,3), Q port plugged, S, U, V, W(1,2,3), Y); OPEN: (A, B, C, D, E, F, G (to collector), H(1,2,3), J(1,2,3), K(1,2), M(1 or 2), N4, R, T, Z).
6. Configure vacuum pumps as appropriate.
7. Turn off power to stepper motors.
8. Ensure heater/chiller unit is off.
9. Prepare hot cell area for next processing session and perform housekeeping as appropriate.
10. Ensure hot cell services (water and air) are turned off.

APPENDIX A. Glossary

Auxiliary Manifold A manifold situated in the control cabinet but on the extractor side of the mercury displacement pump where vapor canisters may be staged for vapor capture directly from the extractor. The auxiliary manifold is equipped with its own vacuum and purge gas capabilities.

Bottom Flange The bottom flange of the extractor where the sample material is released from the extractor after being processed. The bottom flange contains a fitted gasket (H-2-821610-47) and a bearing for the mixing rotor (H-2-821610-4).

Bulk Sample Volume Determination A procedure which employs the principle of the ideal gas law to determine the volume of the sample material that is present in the extractor. When calculating this determination, it is important that correct valve configurations are observed for accurate determinations and that diluent volume must be accounted for.

Bulk Vapor Extraction The first major extraction exercise performed on the extruded sample material. This procedure normally requires thirteen cycles of the mercury pump. Vapor samples collected during this phase may be collected simultaneously, in triplicate.

Collector Side Manifold Generally, this involves the tubing and valves of the RGS system which exist on the "discharge" side of the mercury displacement pump. Precisely, the collector side manifold includes the volume of the one-half filled sight glass, and 1) all the volume occupied through the H valves and the collection canisters during the Bulk Vapor Extraction phase and the Equilibrated Vapor Extraction phase, or 2) only the volume of the manifold to the H valves (closed) during the Exsolution (q.v.) phase.

Coupler The interface between the sample and the extractor which consists of a compression clamp and a rubber seal (H-2-821860-20).

D Collection Canister A vapor canister connected to the Auxiliary Manifold.

Exsolution The process by which gas retained in a sample is separated from the bulk of the sample through exhaustive extractions. This action describes the physical separation of gas from the sample by lowering the ambient pressure about the sample below its inherent vapor pressure. The exsolution process describes the extraction of gas as it exists already as a gas or as a condensed or solvated vapor. In contrast, "evolution" of gas from the sample implies that gas is being generated from the bulk sample through chemical activity such as the evolution of hydrogen gas from the chemical reaction of caustic solution on aluminum metal.

Extractor Side The section of the RGS system including the extractor, extractor manifold, secondary volume (q.v.), pressure transducer manifold, and all tubing, valves, and filters that exists between the extractor manifold and valves F/G and valve Z.

Extractor Manifold The section of tubing including valves R, S, and T that exists between the extractor and the secondary volume.

Extractor Volume The accepted volume of the empty, dry extractor but including the mixer rotor and equal to 1290 cc.

Extruder The actuator and ram that hydraulically ejects the sample from the sampler into the extractor. The control unit of the hydraulic ram is located outside the hot cell and consists of a reservoir of hydraulic fluid, a pump, and a system of valves and regulators to control the force and speed of the ram.

J Collection Canister The vapor canisters that are stage at the collector manifold at the H valves.

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10.2.4

Exsolution Sequence

1. Ensure rotary vane pump is running.
2. Ensure the following valve configurations: CLOSED: {B, K2, M2, N(1,2,3,4), R, S, U, V, W(1,2,3), Y, Z}; OPEN: {A, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1, T}.
3. Ensure stirrer motor is rotating at 60 rpm.
4. Ensure heater/chiller is controlling to 10 °C and extractor temperature ~10 °C.
5. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
6. Adjust pressure indicator zero offsets as necessary.
7. Record system's temperature and pressure in notebook.
8. Ensure pump pressure is less than 78 millitorr.
9. Close valve K1 and H(1,2,3).
10. Open valve B.
11. Perform the following sequence eighteen (18) times:
 - a. Raise mercury reservoir above 0-point.
 - b. Close valve F.
 - c. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
 - d. Lower mercury reservoir to bottom of travel.
 - e. Record system's pressure and temperature in notebook.
 - f. If first or tenth cycle:
 - (1) Open valves H(1,2,3).
 - (2) Wait for pressure stabilization.
 - (3) Close valves J(1,2,3), H(1,2,3) and record canister/sample ID, pressure and temperature in notebook.
 - (4) Replace vapor canisters at H ports (with J valves already open).
 - g. Turn valve G to collector side.
 - h. Open valves H(1,2,3).
 - i. Open valve K1 and wait till pump pressure falls below 78 millitorr.
 - j. Close Valves H(1,2,3), K1, and U.
 - k. Open valve F.
 - l. Check off completed cycle number:

1	2	3	4	5	6	7	8	9
10	11	12	13	14	15	16	17	18

12. Raise mercury reservoir above 0-point.
13. Close valve F.
14. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
15. Lower mercury reservoir to bottom of travel.
16. Record system's pressure and temperature in notebook.
17. Open valves H(1,2,3).

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18. Wait for pressure stabilization.
19. Close valves J(1,2,3), H(1,2,3) and record canister/sample ID, pressure and temperature in notebook.
20. Remove the vapor canisters from the RGS system.
21. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
22. Replace vapor canisters at H ports (with J valves already open).
23. Turn valve G to collector side.
24. Open valves H(1,2,3).
25. Open valve K1.
26. Close valve T.
27. Ensure rotary vane pump is running and valve M1 is open.
28. Ensure valve K1 is open.
29. Stop data acquisition, close file, and copy data file to floppy disk.
30. Turn off heater/chiller unit.
31. Ensure the following valve configurations: CLOSED: (K2, M2, N(1,2,3,4), R, S, T, U, V, W(1,2,3), Y, Z); OPEN: (A, B, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1).

10.2.5 Addition of Diluent and Equilibration Interim

1. Ensure rotary vane pump is running.
2. Ensure stirrer motor is rotation at 60 rpm.
3. Ensure the following valve configurations: CLOSED: (K2, M2, N(1,2,3,4), R, S, T, U, V, W(1,2,3), Y, Z); OPEN: (A, B, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1).
4. Dispense approximately 100 mL diluent through valve W2 and then ensure valve W2 is closed.
5. Dispense 300 mL diluent through valve W1 and the ensure valve W1 is closed.
6. Record date, time, event, and temperature.
7. Acquire a sample of the diluent and standard solution for chemical analysis (20 mL each).
8. Allow the contents of the extractor to mix, stir, and equilibrate for the required interim.

10.2.6 Equilibrated Vapor Extraction

1. Ensure rotary vane pump is running.
2. Ensure valve T is closed and valves K1 and M1 are open.
3. Ensure the following valve configurations: CLOSED: (K2, M2, N(1,2,3,4), R, S, T, U, V, W(1,2,3), Y, Z); OPEN: (A, B, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1).
4. Ensure stirrer motor is rotating at 60 rpm.
5. Ensure heater/chiller is on and controlling to 10 °C and extractor temperature ~10 °C.
6. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
7. Ensure system pressure is less than 0.01 kPa.
8. Adjust pressure indicator zero offsets as necessary.
9. Close valve K1.
10. Open valve T and wait until system's temperature and pressure stabilizes.
11. Record date, time, event, temperature, and pressure in notebook.

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12. Close valve F and ensure valve G is turned to the collector side.
13. Perform the following steps nineteen (19) times:
 - a. Raise mercury reservoir above 0-point.
 - b. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
 - c. Lower mercury reservoir to bottom of travel.
 - d. Record temperature and pressure in notebook.
 - e. Turn valve G to extractor side and wait until mercury completely evacuates displacement bulb.
 - f. Close valve U.
 - g. Turn valve G to collector side.
 - h. Check off completed cycle number:

1	2	3	4	5	6	7	8	9	10
11	12	13	14	15	16	17	18	19	

14. Raise mercury reservoir above 0-point.
15. Open valve U and control the flow of mercury to sight glass with valve G and height of reservoir.
16. Lower mercury reservoir to bottom of travel.
17. Record temperature and pressure in notebook.
18. Close valves J(1,2,3) and H(1,2,3).
19. Record sample, canister ID, temperature, and pressure in notebook.
20. Stop data acquisition, close file, and copy data file to floppy disk.
21. Remove the vapor canisters from the RGS system.
22. Survey each vapor canister by swabbing in and around the connector tube and then surveying the swab for removable radioactive contamination.
23. Place three new canisters (with J valves open) at H ports and Ensure plug at auxiliary port.
24. Open valves H(1,2,3) and turn valve G to collector side.
25. Open valve K1 and Ensure rotary vane pump is running valve M1 is open.
26. Ensure the following valve configurations: CLOSED: {K2, M2, N(1,2,3,4), R, S, T, U, V, W(1,2,3), Y, Z}; OPEN: {A, B, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1}.
27. Turn heater/chiller unit off.

10.2.7 Bulk Sample Volume Determination (BSVD)

1. Ensure rotary vane pump is running.
2. Ensure the following valve configurations: CLOSED: {K2, M2, N(1,2,3,4), R, S, T, U, V, W(1,2,3), Y, Z}; OPEN: {A, B, C, E, F, G (to collector), H(1,2,3), J(1,2,3), K1, M1}.
3. Ensure stirrer motor is rotating at 60 rpm.
4. Ensure extractor temperature is roughly equal to collector side temperature.
5. Set automatic data acquisition to 10-second interval data log and record data file name in notebook.
6. Close valve F.
7. Open valves N3, N4, R, T, and Z.
8. Allow a short pause for pressure to equilibrate and then close valve Z.

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9. Perform the following steps three (3) times:
 - a. Evacuate the system to about 4 kPa using valve F.
 - b. Wait for the system to equilibrate and then record extractor pressure (as variable P1) from 1000-torr pressure transducer display.
 - c. Close valve T.
 - d. Open valve Z.
 - e. Wait for the system to equilibrate and then record extractor pressure (as variable P2) from 1000-torr pressure transducer display.
 - f. Close valve Z and open valve T.
 - g. Wait for the system to equilibrate and then record extractor pressure (as variable P3) from 1000-torr pressure transducer display.
10. Determine "Bulk Sample Volume Determination" per equations 2a and 2b and record in notebook.
11. Ensure heater/chiller unit is off.
12. Ensure rotary vane pump is running, valves K1 and M1 are open, and valve F is closed.
13. Open valves N3, N4, and Z.
14. Pause briefly to allow pressure to equilibrate and then close valve T.
15. Close valve N3.
16. Open valve F.
17. Close valve C.
18. Exchange plug at Port D with opened canister.
19. Open valves C and K2.

10.3 Sampler Disassembly and Clean-Up

10.3.1 Disassembly and Sample Removal

1. Ensure valve T is closed.
2. Place large pan under extractor.
3. Remove sampler from extractor and prepare it for load-out.
4. Drop bottom flange from extractor.
5. Collect sample and stow as appropriate.
6. Wash entire extractor interior and parts with water and dry with air.

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APPENDIX A. Glossary (Continued)

Mercury Positive Displacement Pump A simple positive displacement pump that uses mercury metal to force the suction and flow of gas from the extractor side to the collector side of the RGS system. The direction of flow and suction is provided by a three-way valve, Valve G. The flow of mercury is motivated by the position of a reservoir which can be lifted or lowered about 100 centimeters from the 0-point (q.v.). The reservoir is vented to atmosphere to provide the necessary pressure to allow the mercury to be drawn up into the displacement chamber and sight glass.

Retained Gas Gas that is generated as a result of chemical or radiolytic decomposition of waste tank material. The gas is called "retained" because it contributes to the bulk volume of tank waste material as bubbles, foam, or even dissolved within the slurry or interstitial liquid.

Sight Glass The very top of the mercury displacement pump where the mercury can be seen when it reaches that level.

Secondary Volume An empty container whose volume has been carefully measured to be 321.46 cc and is used to verify the volume of other sections of the RGS system using the principle of the ideal gas law.

Stirrer Motor The motor affixed to the extractor which turns the mixer blade inside the extractor. Between the motor and the mixer blade is a reduction gear box that slows the mixer speed to 1/20th of the motor speed.

0-Point The point of elevation of the index of the mercury reservoir that would cause the level of mercury in the displacement pump to rise exactly to the middle of the sight glass in which 0 kPa pressure (abs.) exists in the collector side manifold.

3/4-Point The level of the column of mercury that would allow the displacement chamber to be 75% full.

Appendix C. Endnotes (continued)

- ¹ All pressure indications in this procedure are expressed in terms of ABSOLUTE pressure.
- ² An in-line depth filter has been installed in the transfer tube, just outside the hot cell wall to prevent the transfer of radioactive contamination out of the hot cell environment.
- ³ Usually 300 mL diluent is added.
- ⁴ This value is obtained from the Bulk Sample Volume Determination q.v.
- ⁵ Vapor canister volumes range from 50 to 150 cc, depending upon the canister used.
- ⁶ The extractor is cooled to about 10 °C to prevent excessive amounts of water vapor from evaporating from the sample material and fouling the in-line depth filter or condensing onto the tubing, displacement chamber, or collector manifold.
- ⁷ q.v. means "see also"
- ⁸ There are three canisters so that duplicate (or triplicate) samples can be acquired simultaneously. They are transported and analyzed as separate vapor samples for independent analyses.
- ⁹ Laboratory Controlled Notebook: WHC-N-164, pp. 64-78.
- ¹⁰ The apparent quantity of vapor contains more water vapor and ammonia than what would be observed if particular care is afforded in preventing the accumulation of these vapors.
- ¹¹ Release disk and retainer ring components do not have to be removed from the sampler.
- ¹² Vapor canisters installed at the H ports are designated as having "J" valves. Vapor canisters installed at the auxiliary port are designated as having "D" valves. Vapor canisters installed at the Q port are designated as having "P" valves.
- ¹³ The turbo-molecular vacuum pump should never be used to draw a vacuum on systems whose pressure is greater than 1 kPa. Rather, the rotary vane pump should be employed during the initial "roughing" of the vacuum. The turbo-molecular pump should only be used after the rotary vane pump has already evacuated the system to less than 1 kPa. Also, both pumps should not be allowed to pump simultaneously from a common manifold so that oily vapors are not continually vented from the rotary vane pump to the turbo-molecular pump.
- ¹⁴ It is possible for mercury to overflow the sight glass and collect in the collector side manifold. This event would have a deleterious affect on the performance of the RGS system (for a number of reasons); therefore, this activity must be performed delicately and carefully.

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Procedure No.

LT-160-101

Procedure use Type: CONTINUOUS

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