SNF-10618
Revision 0

Spent Nuclear Fuel Project Canister Storage Building Multi-Canister Overpack Sampling System Validation (OCRWM)

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Fluor Hanford
P.O. Box 1000
Richland, Washington

Contractor for the U.S. Department of Energy
Richland Operations Office under Contract DE-AC06-96RL13200

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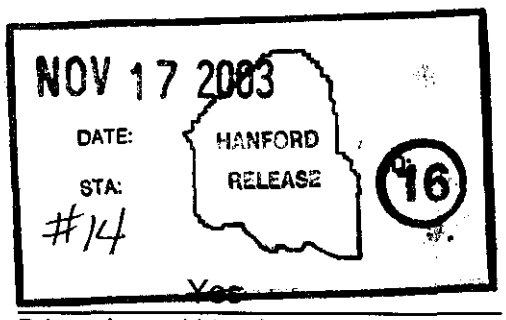

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ALARA	As Low As Reasonably Achievable
CSB	Canister Storage Building
DCS	Distributive Control System
HEPA	High Efficiency Particular Air
MCO	Multi-canister Overpack
MHM	Multi-canister Overpack Handling Machine
MP	Maintenance Procedure
N/A	Not Available
OCRWM	Office of Civilian Radioactive Waste Management
PAT	Preoperational Acceptance Test
PCV	Pressure Control Valve
PIC	Pressure Indicating Control
PIT	Pressure Indicating Transmitter
PNNL	Pacific Northwest National Laboratory
PS	Process Standard
QA	Quality Assurance
QARD	Quality Assurance Requirements Descriptions
RTU	Remote Terminal Unit
SCF	Sample Correlation Factor
TCF	Theoretical Correlation Factor
SNF	Spent Nuclear Fuel
SP	Surveillance Procedure
THC	Total Hydrocarbon
TIT	Temperature Indicating Transmitter
TPCR	Technical Procedure Change Request
TSR	Technical Safety Requirement
WP	Work Package

1.0 INTRODUCTION

Approximately 400 Multi-canister overpacks (MCO) containing spent nuclear fuel are to be interim stored at the Canister Storage Building (CSB). Several MCOs (monitored MCOs) are designated to be gas sampled periodically at the CSB sampling/weld station (Bader 2002a). The monitoring program includes pressure, temperature and gas composition measurements of monitored MCOs during their first two years of interim storage at the CSB.

The MCO sample cart (CART-001) is used at the sampling/weld station to measure the monitored MCO gas temperature and pressure, obtain gas samples for laboratory analysis and refill the monitored MCO with high purity helium as needed. The sample cart and support equipment were functionally and operationally tested and validated before sampling of the first monitored MCO (H-036). This report documents the results of validation testing using training MCO (TR-003) at the CSB. Another report (Bader 2002b) documents the sample results from gas sampling of the first monitored MCO (H-036).

1.1 Objective and Purpose

This document is required by *Spent Nuclear Project Testing Process Administrative Procedure* (FH 2000). It verifies that the MCO sample cart and support equipment perform as designed and demonstrates satisfactory performance for continued service.

The objectives of MCO gas sampling are:

- Obtain representative samples from monitored MCOs during their first two years of interim storage at the CSB
- Minimize sampling error as practical
- Meet MCO safety and quality requirements including:
  - Maintain adequate MCO gas pressure
  - Prevent introduction of contaminants into an MCO

2.0 SUMMARY

Validation testing of the MCO gas sampling system showed the equipment and procedure as originally constituted will satisfactorily sample the first monitored MCO. Subsequent system and procedural improvements will provide increased flexibility and reliability for future MCO gas sampling.

The physical operation of the sampling equipment during testing provided evidence that theoretical correlation factors for extrapolating MCO gas composition from sample results are unnecessarily conservative. Empirically derived correlation factors showed adequate conservatism and support use of the sample system for ongoing monitored MCO sampling.

3.0 SAMPLING PROCESS TEST RESULTS

The main features of the sampling system are represented in Figure 3-1. These features are the MCO in the sample/weld pit, sample system, Office of Civil Radioactive Waste Management (OCRWM) helium supply, and the sample hood exhaust system.

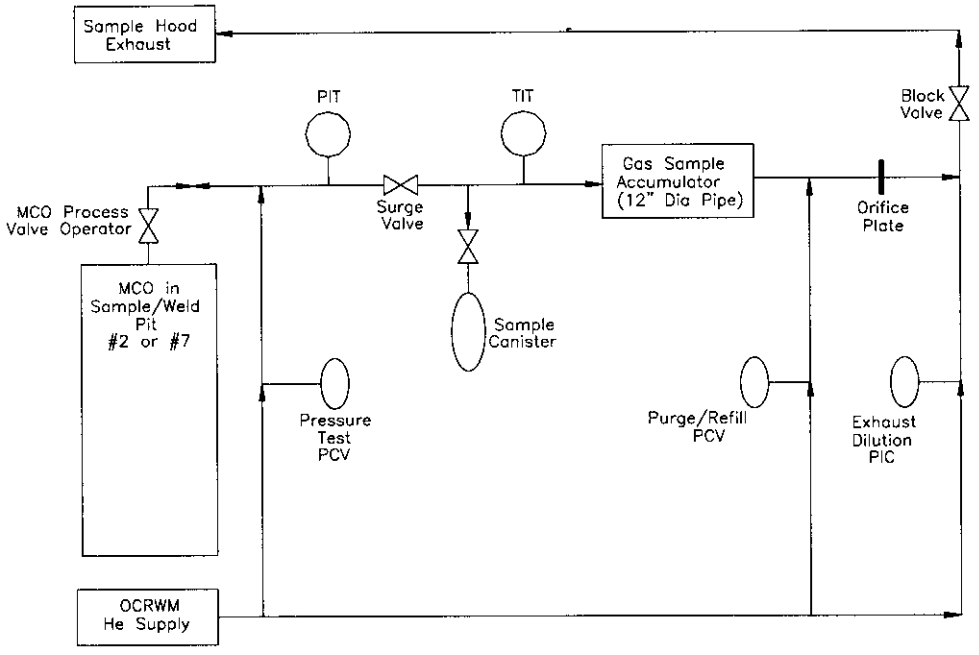


Figure 3-1: Simplified Schematic Diagram of MCO Gas Sampling System

Operating procedure OP-23-002S *Operate MCO Sample Station (OCRWM)* is used to obtain a sample of MCO gas. This sample includes residual helium from pressure testing and purging of the sampling system. The residual helium is accounted for in order to understand the actual MCO gas composition.

The sampling equipment contains residual helium at close to atmospheric pressure (about 15 psia) prior to accessing gas via the MCO process valve. MCO gas flows into the sample accumulator and increases the pressure in the cart by about 10 psi to a final pressure of about 25 psia. The resulting inventory is a combination of residual helium (> 99.5 % pure) and gas from the MCO. The MCO gas is about 10/25 or 2/5 of the cart's gas inventory; residual helium makes up the other 3/5-gas inventory. The 10 psi pressure increase and MCO gas dilution by the residual gas in the sampling system results in a theoretical correlation factor (TCF) of about 2.5. The actual TCF varies in accordance with actual MCO and cart system pressures.

An equation relating gas pressures and concentrations can be developed based on Dalton's Law. Equation 1 relates the final pressure of the sampling system to the sum of the residual pressure of the system before addition of gas from the MCO, and the pressure change resulting from addition of the MCO gas.

$$P_f = P_r + \Delta P_{mco} \quad (1)$$

$$\Delta P_{mco} = P_f - P_r \quad (2)$$

where :
P_f = final pressure of sample system at equilibrium, psia
P_r = residual pressure of sample system before addition of MCO gas, psia
ΔP_{mco} = pressure increase of sampling system by MCO gas, psi

The partial pressure of a constituent is related to the mole fraction and the gas pressure as shown in Equation 3.

$$y_f P_f = y_r P_r + y_{mco} \Delta P_{mco} = y_r P_r + y_{mco} (P_f - P_r) \quad (3)$$

where:
y_f = mole fraction of any constituent in sample system at equilibrium = [Sample Result]
y_r = mole fraction of any constituent in residual gas before addition of MCO gas
y_{mco} = mole fraction of any constituent in MCO gas = [MCO]

Equation 3 is rearranged and solved for the mole fraction or concentration of any constituent in the MCO at equilibrium.

$$y_{mco} (P_f - P_r) = y_f P_f - y_r P_r$$

$$y_{mco} = (y_f P_f - y_r P_r) / (P_f - P_r) \quad (4)$$

The pressure testing and purging of the sampling system with >99.5 % helium reduces the impurity content (y_r) in the sampling system to negligible values before addition of MCO gas. The negligible impurity values simplify Equation 4 to the below equation.

$$y_{mco} = (y_f) P_f / (P_f - P_r) \quad (5)$$

The TCF is related to the pressure changes of the sampling system as discussed above and shown by Equation 6.

$$TCF = P_f / (P_f - P_r) \quad (6)$$

Combining Equation 5 and Equation 6 gives the following equation for calculation of the MCO gas concentration after perfect mixing of the residual helium gas and added MCO gas in the sampling system.

$$y_{mco} = (y_f) (TCF) = [MCO] = [Sample Result](TCF) \quad (7)$$

Table 3-1 lists example conditions and calculated values for TCF and MCO gas concentrations at perfect mixing equilibrium based on the hydrogen concentration.

Table 3-1 Calculated Values of TCF and MCO Hydrogen Concentration at Equilibrium

Residual Pressure Sample System P_r	Equilibrium Pressure After MCO Gas Addition P_f	T C F	Hydrogen Concentration at Equilibrium Pressure [Sample Result]	Calculated Hydrogen Concentration [MCO]
psia	psia	-	ppm	ppm
15	26	2.36	10	23.6
15	25	2.50	10	25
15	24	2.67	10	26.7

Even though a TCF of about 2.5 can be seen to represent the expected pressure changes during MCO sampling and assuming perfect mixing, validation test results show that the sample drawn matched quite closely the actual MCO gas concentration. This result implies that the residual He was pushed from the sample system piping into the downstream accumulator by the inrush of MCO gas and that the sample was drawn before the gasses in the system were fully mixed. Application of a TCF is, therefore, very conservative. Complete gas mixing in a short period of time is seriously retarded by the presence of the 1/8" pipe T that houses the thermocouple (TE-723) just upstream of the gas accumulator and downstream of the sample take-off connection. This restriction provides a reasonable explanation for the close agreement between the actual MCO gas concentration and the sample from the cart.

Because the TCF is too conservative, a more useful sample-based correlation factor (SCF) relating the results from sample cart samples and direct samples of training MCO gas was developed for several major gas constituents. The average SCF was about 1.15 for argon, carbon dioxide, nitrogen and oxygen and 1.00 for helium. The SCF for oxygen was 1.14. These empirically derived SFCs reflect the close match between directly sampled MCO gas and sample cart samples.

The procedure used for sampling of the training MCO does not ensure complete mixing of the gas in the sample cart. The surge valve is left open allowing communication between the MCO and sampling system until equilibrium pressure is achieved after about thirty seconds. It is postulated that the movement of MCO gas through the sample system piping carries the purge He out of the smaller diameter piping and into the downstream gas accumulator. The sample was drawn a few minutes after closure of the surge valve allowing limited time for diffusion to equilibrate the concentration between the surge valve and block valve (see Figure 3-1). The sampling procedure must be performed so as to minimize the time between MCO process valve opening and sample bottle filling to eliminate uncertainty in results of future samples. Changes in the design of the sampling system hardware could also reduce sampling uncertainty. Additional tests using the training MCO to improve understanding of the plug flow and slow mixing behavior in the cart could also be performed. Samples could be taken after set time periods to determine the rate at which mixing occurs and procedure changes suggested by test results could improve sampling accuracy.

Several possible improvements were suggested during testing; they include both hardware and procedural changes. Suggested equipment upgrades included modifications of the helium gas supply, MCO sample cart valving, and interface manifold connections. Improvements to the sampling procedure, OP-23-002S, include changes to reflect hardware upgrades and replacement, provision for performing certain steps in parallel (reduces time to complete sampling), and increased time for helium purging of air from the MCO sample cart piping. Extensive redesign/new design of the sampling equipment to greatly reduce the volume of gas taken from an MCO has also been suggested.

4.0 DESCRIPTION OF GAS SAMPLING SYSTEM

The MCO gas sampling system and support equipment are described in the following sections. The discussion includes equipment, equipment testing, procedures, OCRWM considerations and sample analysis. Each is discussed in some detail.

4.1 Equipment

The gas sampling equipment consists of two sampling/weld stations, an MCO sample hood, the sample hood exhaust system, MCO sample cart, sample interface manifold, and helium supply system.

4.1.1 MCO Sampling/Weld Station

The MCO Sampling/Weld Station System is used to sample gas from monitored MCOs and to weld a Canister Cover Assembly to each MCO. The MCO Handling Machine (MHM) is used to transport the designated MCO from the cask-receiving pit or storage tube to a sampling/weld station pit located at the south end of the CSB operating deck and back from the sampling/weld pit to the designated storage tube. The major components of the sampling/weld station are:

- Two radiation shielded Sampling/Weld Station Pits (Pits #2 and #7) with impact absorbers and two support utilities pits (Pits #3 and #6) are provided for sampling and welding operations. Impact absorbers prevent damage to the MCOs upon placement in the Sampling/Weld Stations in the unlikely drop of the MCO from the MHM. Pits #2 and #7 have removable guardrails to protect personnel against accidental falls. Pits #3 and #6 are covered with removable steel grating.

- Two 7.5-ton capacity gantry cranes, one for each of the two stations are used to position the Sample Hood (one Sample Hood to be shared between the two stations). Each gantry crane is equipped with one 5.0 ton and two 1.0 ton hoists.

- Temporary shielding is used to protect operating personnel against radiation streaming during MCO movement into or out of the sampling/weld pits. The shielding reduces the radiation dose rate to within as low as reasonably achievable (ALARA) limits.

- Fixed shielding with internal coolant circulation and MCO cooling caps (at each station) is used to cool any high temperature MCO top section for operator safety during sampling or welding.

- A shared, permanently installed Sampling/Weld station high efficiency particular air (HEPA) filter/exhauster is provided to ventilate the sample and/or weld hoods (AH-006).

- A sample cart, connected to the sample hood process gas HEPA filter provides equipment for gas sampling and MCO refill operations (CART-001).

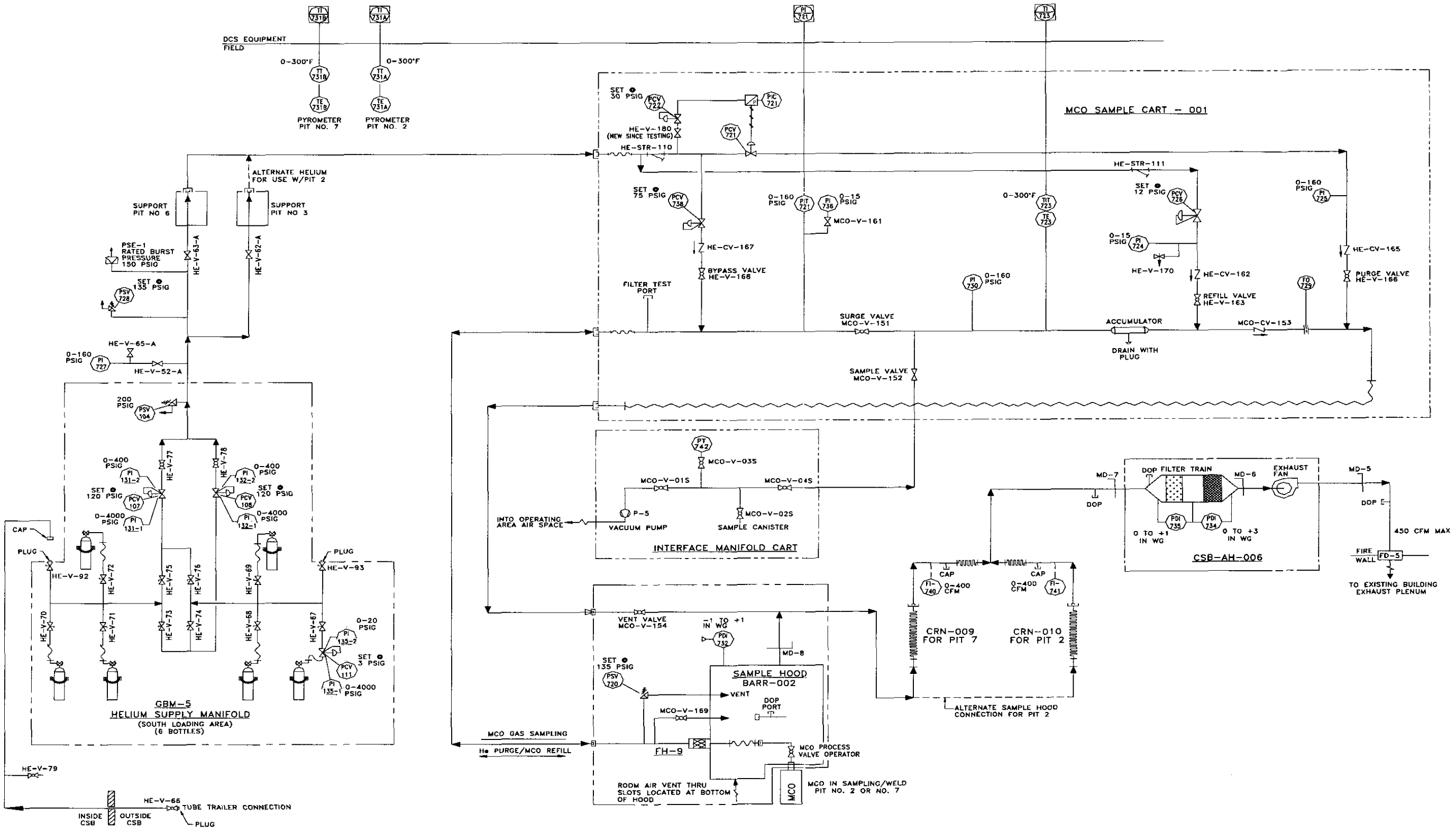
- An interface manifold is used to obtain gas samples for laboratory analysis. The interface manifold is connected to the sample cart.

Additional support equipment includes a fixed optical pyrometer at each station to measure the MCO skin temperature for a general temperature profile; a hand-held pyrometer to measure the temperature of the MCO shield plug; one process valve operator for MCO gas access, tools for opening/closing the MCO process port valve and cover plate removal/installation, and non destructive examination equipment to test integrity of the MCO Port #2 cover plate seal after gas sampling.

4.1.2 Sample Hood

Sample Hood (BARR-002) fulfills several functions: it provides confinement of any release of the MCO's internal atmosphere thereby preventing a release into the CSB operating area atmosphere during sampling operations; confines the exhaust from the Sample Cart; and provides a seismically secure mounting location for the high efficiency particulate air (HEPA) filter FH-9. The sample hood is stainless steel and has an open bottom, two glove ports, equipment pass through port, view windows, exterior lighting, ventilation control damper, and sample hose with valve operator and accessories. Figure 4-1 shows a schematic illustration of the sample hood and support equipment. See drawing H-2-120400 for additional details.

A valve operator with 1-inch diameter flexible hose connects the MCO port #2 (after port cover removal) to the HEPA filter FH-9. FH-9 removes particulate contamination that may pass through the internal MCO HEPA (not testable) from the MCO sample gas before the gas exits the sample hood and enters another 1-inch diameter flexible hose that connects to the sample cart. A 1-1/2 inch diameter flexible hose is used to route exhaust gas from the sample cart to the sample hood exhaust line. Confinement is accomplished by maintaining a negative pressure within the sample hood so that any airflow is from the operating area into the sample hood. Air is exhausted from the sample hood to the building ventilation system through a HEPA filtered unit CSB-AH-006.


DCS EQUIPMENT
FIELD
TT 731B
TT 731A
0-300°F
TE 731B
TE 731A
PYROMETER PIT NO. 7
PYROMETER PIT NO. 2
PI 721
TI 723
MCO SAMPLE CART - 001
SET 30 PSIG
PCV 722
PIC 721
HE-V-180 (NEW SINCE TESTING)
PCV 721
HE-STR-110
HE-STR-111
SET 75 PSIG
PCV 738
0-160 PSIG
PIT 721
PI 736
0-15 PSIG
MCO-V-161
TIT 723
TE 723
SET 12 PSIG
PCV 726
PI 725
PI 724
HE-V-170
HE-CV-167
BYPASS VALVE HE-V-168
FILTER TEST PORT
HE-CV-162
REFILL VALVE HE-V-163
HE-CV-165
PURGE VALVE HE-V-166
FQ 729
PI 730
SURGE VALVE MCO-V-151
ACCUMULATOR
DRAIN WITH PLUG
MCO-CV-153
SAMPLE VALVE MCO-V-152
ALTERNATE HELIUM FOR USE W/PIT 2
SUPPORT PIT NO 6
SUPPORT PIT NO 3
PSE-1 RATED BURST PRESSURE 150 PSIG
HE-V-63-A
HE-V-62-A
SET 135 PSIG
PSV 728
HE-V-65-A
HE-V-52-A
PI 727
200 PSIG
PSV 104
0-400 PSIG
PI 131-2
PI 132-2
SET 120 PSIG
PCV 107
PCV 108
PI 131-1
PI 132-1
0-4000 PSIG
HE-V-77
HE-V-78
HE-V-92
HE-V-93
HE-V-72
HE-V-75
HE-V-76
HE-V-69
HE-V-70
HE-V-71
HE-V-73
HE-V-74
HE-V-68
HE-V-67
CAP
PLUG
0-20 PSIG
PI 135-2
SET 3 PSIG
PCV 111
PI 135-1
GBM-5
HELIUM SUPPLY MANIFOLD
(SOUTH LOADING AREA)
(6 BOTTLES)
HE-V-79
HE-V-66
TUBE TRAILER CONNECTION
INSIDE CSB
OUTSIDE CSB
PT 742
MCO-V-03S
MCO-V-01S
MCO-V-04S
INTO OPERATING AREA AIR SPACE
P-5
VACUUM PUMP
MCO-V-02S
SAMPLE CANISTER
INTERFACE MANIFOLD CART
VENT VALVE MCO-V-154
-1 TO +1 IN WG
PDI 732
MD-8
SET 135 PSIG
PSV 720
SAMPLE HOOD
BARR-002
VENT
DOP PORT
MCO-V-169
MCO GAS SAMPLING
He PURGE/MCO REFILL
FH-9
MCO PROCESS VALVE OPERATOR
ROOM AIR VENT THRU SLOTS LOCATED AT BOTTOM OF HOOD
MCO
MCO IN SAMPLING/WELD PIT NO. 2 OR NO. 7
MD-7
DOP
DOP FILTER TRAIN
MD-6
EXHAUST FAN
MD-5
DOP E
450 CFM MAX
FIRE WALL
FD-5
TO EXISTING BUILDING EXHAUST PLENUM
PDI 735
PDI 734
0 TO +1 IN WG
0 TO +3 IN WG
CSB-AH-006
CAP
FI-740
0-400 CFM
FI-741
CRN-009 FOR PIT 7
CRN-010 FOR PIT 2
ALTERNATE SAMPLE HOOD CONNECTION FOR PIT 2

HEPA filter FH-9 is constructed of stainless steel and contains a sinter bonded and reinforced composite filter. HEPA Filter FH-9 is challenge testable in-place. The sample hood gloves and pass through port were upgraded to address operational enhancements before validation testing (Black and McClellan 2001).

4.1.3 MCO Sample Cart

The MCO Sample Cart (CART-001) is a pushcart with piping, valves, fittings, flexible hose with quick connects, instrumentation, and gas accumulator. The cart connects to the 120-psig-helium supply, distributive control system (DCS) via a remote terminal unit (RTU-2), 120-v ac supply, interface manifold, and sample hood (MCO gas sample line and exhaust system). All piping is stainless steel. Flexible hoses (five total) are stainless steel braided over Teflon[1] or stainless steel braided over stainless steel or corrugated stainless steel. Figure 4-1 shows a schematic illustration of the MCO sample cart. See drawings H-2-120403 and H-2-128000 for additional details.

The 120-psig-helium supply system is connected to the sample cart through a 1-inch diameter flexible hose and quick connects near the sample/weld pits. Sample cart equipment divides the 120-psig-helium supply into four separate delivery lines. Nominal 12-psig helium is used to purge air from the sample cart and hoses and to refill the monitored MCO to designated gas pressure (as needed), 75-psig helium is used to leak rate test the MCO sample line and valve operator connections before sampling the MCO, and 20-psig helium is used to operate the purge gas control valve actuator for dilution of sample cart exhaust gas. The 120-psig-helium supply is used to purge and dilute MCO sample gas as it is released to the exhaust system at the sample hood.

The RTU provides electrical power for operation and readout of the OCRWM pressure (PIT-721) and temperature (TIT-723) instruments on the sample cart. These readings are also transmitted to the DCS through RTU-2.

The 120–v ac supply voltage at utility Pits #3 or #6 provides electrical power to the process controller for helium purge of the MCO sample gas from the sample cart.

[1] Teflon is a trademark of E. I. DuPont de Nemours and Company

4.1.4 Interface Manifold

The interface manifold is used to obtain the helium blank sample and MCO gas sample from the sample cart. The interface manifold contains valves, vacuum seal fittings, a flexible line, mechanical vacuum pump, pressure indicator instrument, and single port gas sample canister. Helium blank gas or MCO gas is accumulated in the sample cart at positive pressure and sampled by drawing a vacuum (<0.1 torr per OP-23-002S) on the interface manifold (including the flex hose, tubing, and sample canister) and then opening the sample valve (MCO-V-152) on the sample cart for pressurized gas flow into the evacuated interface manifold and sample canister. The gas sample is collected and the valve on the sample canister is closed. The excess gas in the sample cart and interface manifold is vented to the sample hood before disconnecting the closed sample canister.

A 120–v ac supply voltage on the south wall of the CSB operating deck provides electrical power to the vacuum pump and vacuum indicator instrument. Figure 4-1 shows a schematic illustration of the sample interface manifold and support equipment. Figure 4-2 is an expanded illustration of the sample interface manifold. See Drawing H-2-120403, Sheet 3 for additional details.

Pacific Northwest National Laboratory (PNNL) provided the pre-evacuated sample canisters (75 cm^3) and analyzed the helium blank and training MCO gas samples. The sample canisters are evacuated to <0.1 torr (as indicated in PI-743) on the interface manifold before taking of the sample to ensure no air has leaked into the canister during storage.

4.1.5 Helium Supply System

The helium supply system (GBM-5) for the sample/weld stations is an eight bottle double manifold (six bottles shown) with manual valves, pressure indicators and pressure safety relief valves. GBM-5 is located in the south vestibule of the CSB. One gas bottle is used to provide a low-pressure purge of the helium supply piping between MCO gas sampling events. Figure 4-1 is a schematic illustration showing the 4,000-psig helium supply system. See Drawing H-2-125162, Sheet 9 for additional details.

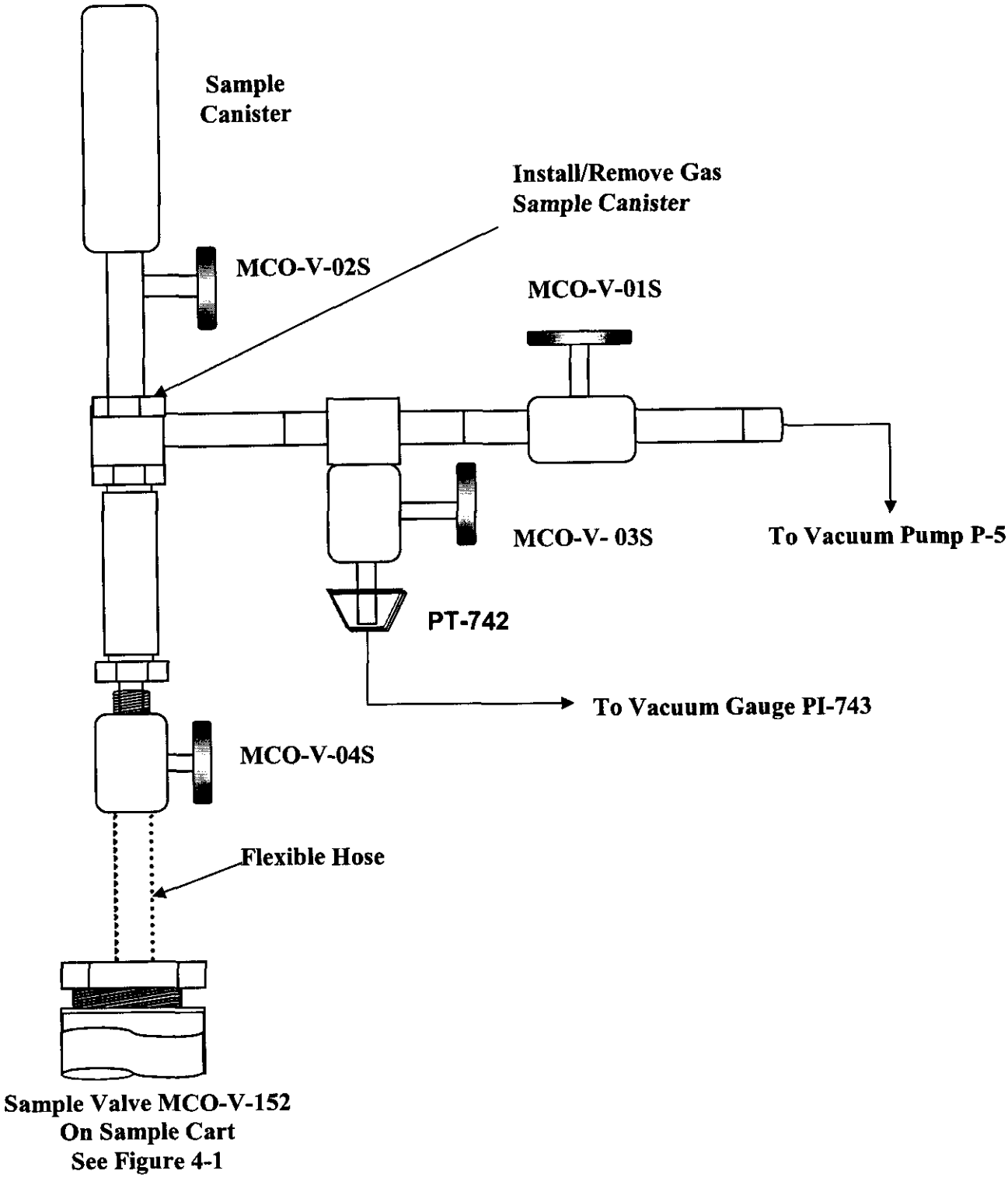
4.2 Equipment Testing

In 1999, Startup Test Engineering directed and supervised a preoperational acceptance test (PAT) of the sampling system (*Test Results Package SNF-W379-TRP-023*). This initial testing demonstrated that the sampling process (including the sample cart, cranes, sample hood, rotational drives, exhaust system and chiller) functioned as designed.

In 2001, CSB Operations performed subsequent validation tests of the equipment at normal operating conditions and in its final in-service configuration under the direction of CSB Engineering. Operations support and Quality Assurance organizations participated in the tests. Approved procedures were used to test and validate the sampling system before monitored MCO gas sampling.

The validation testing included chain-of-custody delivery of gas samples to PNNL for analysis by a mass spectrograph. Preliminary sample results from helium blank and training MCO samples were available within a few hours after sampling. Final results are included in Appendix B.

Figure 4-2: Sample Interface for CART-001



4.3 Procedures

Procedures that were used to implement validation testing included the work package, operating procedures, maintenance procedures, and analytical procedures.

4.3.1 Work Package

Work package 1S-01-00586 *Sample/Weld Station Hands On Work* was used to perform sample station hands on work, personnel training, procedure verification, and functional test of the equipment used for training MCO gas sampling.

4.3.2 Operating Procedures

Operating procedure OP-23-002S *Operate MCO Sample Station (OCRWM)* was used to operate the MCO sampling equipment. If a limit and/or control were not in compliance or if the procedure could not be performed in compliance with the limit or control, then management was notified immediately and Technical Procedure Change Requests (TPCR) were generated and implemented when appropriate.

The operating procedure was controlled in accordance with requirements identified in OCRWM/Quality Assurance Requirements and Descriptions (QARD) DeKlever (2000). OCRWM and QARD designators are included in the body of the procedure. The procedure contains Traveler Pointer markers to indicate associated steps in the traveler that require information to be documented (CP-70-007) and reference to TSR/process standard limits and controls (PS 610).

4.3.3 Maintenance Procedures

Maintenance procedures include surveillance procedures (SP), maintenance procedures (MP), and work packages (WP). Surveillance procedures and MPs were used to calibrate instruments and inspect equipment. Work packages were used to correct discrepancies found during training and validation testing. Table 4-1 is a list of the procedures used for MCO gas sample training and validation testing.

Table 4-1: Maintenance Procedures and Work Packages for MCO Gas Sample Training and Validation Testing

Procedure/ Work Package Number	Title
SP-23-001S	Calibration of Sample/Weld Hood Differential Pressure Indicators PDI-732, PDI-737, PDI-739
SP-23-002S	Calibration of Sample/Weld Station Flow Indicators FI-740, FI-741, And Differential Pressure Indicators PDI-734, PDI-735
SP-23-003S	Calibration of Sample Cart Pressure Instrument PIT-721 (OCRWM)
SP-23-004S	Calibration of Sample Cart Temperature Instrument TIT-723 (OCRWM)
SP-23-005S	Periodic Inspection of Sample/Weld Stations Cranes CRN-009 And CRN-010
SP-23-006S	Calibration of Sample Station Exhaust Differential Pressure Indicators PDI-734 and PDI-735
MP-23-001S	Inspect-Service MCO Sample Cart
MP-23-006S	Frequent Inspection of Sample/Weld Station Cranes CRN-009 And CRN-010
MP-23-014S	Inspect Sample/Weld Station Exhauster Motor AH-006
1S-00-00341/M	Modify/Repair Helium Supply System
1S-01-00586	Sample/Weld Station Hands On Work
1S-01-00591	Instrument Calibration
1S-01-00601	Change PIT-721 Local Display to Read in Psi
1S-01-00655	Trouble Shoot and Repair MCO Sample Cart Leaks
1S-01-00656	Add Strain Relief to Amphenol at Sample Station
1S-01-00669	Function Test Foxboro Controller Sample Cart
1S-01-00682	Take Flow Measurements From Sample Cart
1S-01-00710	Functional Check of TE-731B and TT-731B
1S-01-00711	Obtain Sample From Dummy MCO
1S-02-00013	Obtain Sample From Dummy MCO in Sample Pit
1S-02-00021	CSB Annual Sample Cart Temperature Instrument Calibration
1S-02-00022	TE-723 Functional Check Prior to Sample Analysis
1S-02-00447	Install TE-723 in Sample Cart

4.3.4 Sample Control and Laboratory Analysis Procedures

Procedure OP-23-002S and Administrative Procedure EN-6-016-02, *Spent Nuclear Fuel Project OCRWM Sample Control* (FH 2002d) were used to obtain and control the helium blank samples and the training MCO gas samples. A chain of custody was used for each sample. Analysis of the helium and training MCO gas samples was performed by PNNL using QARD compliant procedures approved by the CSB Project.

4.3.5 MCO Sampling Process

Two sampling processes were used to obtain gas samples from the training MCO. The conventional MCO sampling process is designed to use the MCO sample cart and interface manifold system for obtaining helium blank and MCO gas samples as described in Procedure OP-23-002S. The conventional process includes refilling of the MCO with helium that meets OCRWM requirements.

The conventional process includes the following steps:

- Install sample hood over training MCO. Start sample hood exhaust system.
- Connect sample cart to sample hood, helium supply, and DCS system.
- Remove training MCO Port #2 cover plate.
- Purge air from the sample cart piping, flexible hoses, and valve operator. Secure process valve operator to MCO Port #2 while purging.
- Perform initial 75-psig-pressure check of sampling piping connection to MCO process port valve.
- Vent gas from pressure test to sample hood to a final pressure >0 psig and <1.0 psig.
- Attach sample canister to interface manifold. Evacuate interface manifold and sample canister to less than 0.1 torr as read on PI-743 using mechanical vacuum pump P-5.
- Adjust helium pressure of sample cart to about 12 psig, open valves and obtain a blank helium sample in evacuated sample canister.
- Close valves, disconnect sample canister and deliver sample canister to PNNL for analysis. Maintain chain-of-custody for helium blank sample.
- Evaluate laboratory results and ensure blank helium sample meets OCRWM requirements.
- Perform final 75-psig-pressure check of the sampling piping connection to MCO process port valve to verify system leakage is < 40 std cm^3/sec at 75 psig (LCO 3.2.2).
- Vent gas from pressure test to sample hood to a final pressure >0 psig and <1.0 psig.
- Attach sample canister to interface manifold. Evacuate interface manifold and sample canister to less than 0.1 torr as read on PI-743 using mechanical vacuum pump P-5.
- Open MCO valve operator and take first pressure reading of MCO on PIT-721.
- Open surge valve to fill MCO cart with MCO gas. Take temperature reading on TIT-723 and second pressure reading of MCO on PIT-721.
- Open sampling valves and obtain an MCO gas sample in evacuated canister. Close sampling valves.
- Compare first pressure reading on PIT-721 and temperature reading on TIT-723 and refill MCO if needed to meet requirements of PS 610.
- Close MCO process valve operator.
- Vent residual sample cart gas to sample hood exhaust while diluting with controlled 120-psig-purge helium supply (PIC-721 and PCV-721).
- Disconnect gas sample canister and deliver to PNNL for analysis. Maintain chain-of-custody for MCO sample.
- Perform leak check of MCO valve operator using PIT-721.
- Shut down and disconnect equipment. Remove sample hood.
- Install MCO Port #2 cover plate on MCO and leak test to 1E-05 std cm^3/sec.

The direct sampling process does not require helium purging of the sample cart or taking of the helium blank sample. The interface manifold is relocated to the filter test port tee on the inlet line to the sample cart from the training MCO (See Figure 4-1). PIT-721 is disconnected and the fitting plugged. The interface manifold, sample canister and line between the MCO process valve operator and tee are evacuated before opening the MCO process valve operator and taking the training MCO gas sample.

The direct sampling process includes the following steps:

- Relocate interface manifold to the filter test port tee on the sample cart inlet line from the training MCO.
- Disconnect PIT-721 from sample cart and plug fitting.
- Install sample hood over training MCO. Start sample hood exhaust system.
- Connect sample cart to sample hood and helium supply.
- Remove MCO Port # 2 cover plate and attach valve operator to training MCO.
- Attach sample canister to interface manifold. Evacuate interface manifold, sample canister and hose to MCO process valve operator to less than 0.1 torr as read on PI-743 using mechanical vacuum pump P-5.
- Open MCO process valve, pressurize system, and obtain an MCO gas sample in the canister.
- Close process valve operator and sampling valves.
- Vent sample cart gas to sample hood exhaust while diluting with controlled 120-psig-purge helium supply. Operate PIC-721 on manual mode.
- Disconnect sample canister and deliver sample canister to PNNL for analysis. Maintain chain-of-custody for MCO gas sample.
- Shut down and disconnect equipment. Remove sample hood.
- Install MCO Port #2 cover plate on training MCO and leak test to 1E-05 std. cc/sec.
- Return sample cart equipment to standard configuration.

4.4 OCRWM Certification

MCOs containing spent nuclear fuel (SNF) stored at the CSB will eventually be transferred to a federal repository for permanent storage. To assure acceptance by the repository, the SNF Project must meet OCRWM/QARD requirements. DeKlever (2000) identified items and activities that require application of OCRWM/QARD requirements ("Q-List").

Items that require certification to meet QARD requirements for MCO gas sampling are:

- The pressure (PIT-721) and temperature (TE-723/TIT-723) instruments on the sample cart, which measure OCRWM data from the monitored MCOs.
- Helium gas purity (>99.5 %).

Activities that require documentation to meet OCWRM requirements for MCO gas sampling are:

- Records of helium received, OCWRM certification package(s) of refill helium for OCRWM/QARD application and the sampling and analysis of helium to ensure non-

approved materials are not admitted to a monitored MCO and are OCRWM lifetime Quality Assurance (QA) records. Calibration records of the pressure and temperature instruments used to provide these data are also considered OCRWM lifetime records.

- Data developed by CSB that are needed by OCRWM as identified in contractual direction from U.S. Department of Energy, Richland Operations Office (none identified at this time).

4.4.1 MCO Sample Cart Pressure Indicating Transmitter

Pressure indicating transmitter PIT-721 is a "Q-List" item because it provides gas pressure data required for OCRWM hydrogen gas calculations for the monitored MCOs. It will also measure the pressure of helium added to the sampled MCO to re-establish a baseline pressure before the MCO is returned to a storage tube.

PIT-721 was purchased as a general service, Quality Level 3-item. It meets the standard for National Fire Protection Association Class 1, Division 2 instruments. Critical characteristics for OCRWM dedication and verification are listed in Table 4-2. Initial calibration was performed under work package 1S-01-00591. Future calibrations will be performed under new work packages on a set frequency determined by CSB Engineering.

Table 4-2: Critical Characteristics and Verification Methods PIT-721

Critical Characteristics				Verification	
Characteristic	Acceptance	ID	Function	Method	Procedure
Manufacturer	Rosemount	X		Inspect	N/A
Model No.	3051CGA22A1AB4E5M5	X		Inspect	N/A
Functional Pressure Readout	Local display calibrated		X	Test	SP-23-003S
Connections Integrity	No faults detected		X	Test	SP-23-003S
Accuracy	+/- 0.5 % range of instrument (+/- 8 psig)		X	Test	SP-23-003S
Range of Instrument	0 to 160 psig		X	Test	SP-23-003S

4.4.2 MCO Sample Cart Temperature Element and Transmitter

Temperature Element TE-723 and the associated Temperature Indicating Transmitter TIT-723 are "Q-List" items because they provide temperature data required for OCRWM hydrogen gas calculation for the monitored MCOs. Temperature of the internal gas is used to calculate the amount of helium and hydrogen in the MCO.

The instrument was purchased as a general service, Quality Level 3 item. It meets the standard for National Fire Protection Association Class 1, Division 2 instruments. Critical characteristics for OCRWM dedication and verification are listed in Table 4-3. Calibration was performed under work package 1S-02-00021. Functional check of TE-723 was performed under work package 1S-02-00022. Future calibrations will be performed under new work packages on a set frequency determined by CSB Engineering.

Table 4-3: Critical Characteristics and Verification Methods for TE/TIT-723

Critical Characteristics				Verification	
Characteristic	Acceptance	ID	Function	Method	Procedure
Manufacturer	Watlow & Rosemount	X		Inspect	N/A
Model No.	ARGHFOA030-GT000 or ARGHFOF030-GT000 & 444TT1U1B2K5	X		Inspect	N/A
Connections Integrity	TE-723 to TIT-723 Connected		X	Test	SP-23-004S
Accuracy	+/- 2 % range of instrument (+/-6 °F)		X	Test	SP-23-004S
Range of Instrument	0 °F to 300 °F		X	Test	SP-23-004S

4.4.3 Helium Gas Supply

High purity helium is the only material introduced into the monitored MCOs at the CSB. The monitored MCO helium is a "Q-List" item because it could potentially be added to the MCO and assures that repository requirements are met for protection of the MCO from excessive oxidants and impurities.

Monitored MCO helium is procured as a general service Quality Level 3 item and verified acceptable based on documentation supplied by the vendor and reviewed during receipt inspections and subsequently certified in accordance with Administrative Procedure *Certification of Q-Listed Items to OCRWM/QARD*, (AP-EN-6-038), prior to use in sampling activities. Prior to start of MCO sampling activities, the associated helium supply lines, sample cart and flexible hoses are purged with helium and a confirmatory helium (blank) sample is taken and tested by an approved vendor (PNNL) to ensure desired helium purity levels are maintained. The gas sample analysis is compliant with the vendor OCRWM/QARD requirements approved by the SNF Project.

Critical characteristics for OCRWM dedication and verification of the monitored MCO helium are listed in Table 4-4.

Table 4-4: Critical Characteristics and Verification of Monitored MCO Helium

Critical Characteristics			
Characteristic	Acceptance Criteria	Method	ID
Batch Conformance	Vendor certificate of conformance >99.999% He purity and following impurity limits are met[1]. H_2O ≤ 3 ppm THC (as CH_4) ≤ 2 ppm O_2 ≤ 2 ppm N_2 ≤ 3 ppm	Inspect	-
He Bottle ID	Verify vendor He bottles contain batch number tag	Inspect	X
Sample Analysis (Helium blank)	Representative sample and laboratory analysis. Verify >99.5 % He purity and following impurities limits are met[1]. H_2O ≤ 4580 ppm THC (as CH_4) ≤ 10 ppm O_2 + Ar ≤ 1020 ppm N_2 ≤ 3600 ppm H_2 ≤ 10 ppm CO_2 ≤ 11 ppm	Test	X

Notes:1) Source of information is Krieg (2002)

4.4.4 Gas Sample Canisters

The sample canisters (single valve, nominal 75 cm^3 void volume and fabricated of stainless steel) are supplied by PNNL and used for both the helium blank and MCO gas samples. A vacuum seal fitting is used to connect the sample canister to the MCO sample interface manifold. The sample canisters have been factory cleaned initially and evacuated to 1E-05 torr by PNNL before transport to CSB. (Note: The sample interface manifold and sample canister are re-evacuated to less than 0.1 torr using the mechanical vacuum pump before the gas sample is obtained from the sample cart).

4.5 Analysis of Gas Samples

The helium blank and MCO gas samples are released from the CSB and transported under chain-of-custody to PNNL for receipt and analysis. Trained personnel use a mass spectrograph, support equipment and QARD compliant procedures to analyze the gas samples. The helium blank gas sample and MCO sample gas are analyzed to OCRWM requirements (FH 2002a).

Pacific Northwest National Laboratory issues a letter with preliminary sample results and then a final report that contains the results of the analyses. These results include a summary data table and narrative describing any anomalies with the data, chain-of-custody forms, evidence of adherence to the OCRWM QA program, results of Quality Control and calibrations required by the analysis procedure, traceability of any laboratory sub-samples to original field samples, and evacuation record of the sample canisters for use at CSB (See Appendix B for details). Table 4-5 lists the constituents and precision requirements (FH 2002e, FH 2002f).

Table 4-5: Gas Sample Analysis and Precision Requirements

Sample	Constituent	Analysis Precision
Helium Blank	He	▪ +/- 2 % for constituents with concentrations > 500 ppm ▪ Best effort for constituents with concentrations ≤ 500 ppm
	N_2	
	H_2	
	O_2	
	Ar	
	Ne	
	CO	
	CO_2	
	NO_x	
	Total hydrocarbons as CH_4	
	H_2O	
MCO	He	▪ +/- 2 % for constituents with concentrations > 500 ppm ▪ Best effort for constituents with concentrations ≤ 500 ppm
	H_2	
	O_2	
	N_2	
	Ar	
	Kr	
	Xe	
	CO	
	CO_2	
	NO_x	
	Total hydrocarbons as CH_4	
	H_2O	

5.0 EQUIPMENT TESTS

The 1999 PAT demonstrated that the sampling process (including the sample cart, cranes, sample hood, rotational drives, exhaust system and chiller) works as designed (Test Results Package SNF-W-379-TRP-023). Design changes to the sampling system and helium supply since completion of the PAT necessitated additional testing of the equipment. After modifications to the sampling system were made, validation tests were performed at normal operating conditions and in-service configuration to validate the sampling equipment system for MCO gas sampling. Maintenance procedures complemented the validation testing. This section describes the final equipment tests for the sample cart, gas sampling, and helium refill of the training MCO.

5.1 MCO Sample Cart

Equipment modification to the sample cart and interface manifold were completed on December 11, 2001. Validation testing started on December 12, 2001 using WP 1S-01-00586 and OP-23-002S. CSB Operations performed the tests at normal operating conditions and final in-service configuration. Training MCO TR-003 was used for the validation testing.

CSB Operations performed the tests under the direction of CSB Engineering. Operations support and QA organizations were also involved. Maintenance performed leak tests and instrument calibrations under work package control. All testing, maintenance and qualifications were completed on January 16, 2002.

Some procedure changes were required to improve operation and/or clarify instructions. If a limit and/or control were not in compliance or if the procedure could not be performed in compliance with the limit or control, then management was notified immediately and a TPCR was written and incorporated as necessary.

5.1.1 Validation Tests

Helium blank samples and training MCO gas samples were obtained during validation testing. Training MCO gas samples were obtained using two sampling methods in order to develop a correlation factor between the helium diluted sample cart samples and undiluted MCO samples (See Section 6.2 for results). The training MCO was refilled with helium to 11.5 psig after the first gas sample because the initial MCO gas pressure (as found) was too low to confidently identify its contents. There was no additional refill of the training MCO during the remaining validation tests.

Helium blank samples were obtained using the sample cart system and instructions in OP-23-002S, which is the MCO standard sampling method (see Figure 4-1). The direct sampling method reconfigured the equipment and used instructions as directed in work packages 1S-01-00711 and 1S-02-0013. Four sets of combined helium blank samples and training MCO gas samples were obtained using OP-23-002S. Two direct MCO gas samples were obtained using reconfigured equipment and instructions in work packages 1S-01-00711 and 1S-02-0013.

Direct MCO gas sampling used the sample hood, valve operator, front-end portion of the sample cart, and interface manifold. The interface manifold connection was moved from the sample valve (MCO-V-152) to the FH-9 HEPA filter test port tee on the MCO gas supply line of the sample cart. The interface manifold vacuum pump evacuated the supply line and flexible hose back to the MCO process valve operator, interface manifold and sample canister to <0.1 torr as read on PI-743. This evacuation activity took about 3 ½ hours. The valve operator was opened, pressurizing the sampling system with MCO gas before taking the sample. See Figure 4-1 and Figure 4-2 for information.

5.1.2 Leak Tests

The MCO sample cart, sample cart exhaust hose to the sample hood and interface manifold were leak tested before and after validation testing. The systems were pressurized to 30 psig with helium and all fittings and valves were visually checked for leaks (~ 1xE-03 std cm^3/sec) using WP 1S-01-00655 and leak detection liquid.

The interface manifold (vacuum seal fittings, valves and pressure indicator) and sample valve MCO-V-152 were gross leak tested by COGEMA Engineering Corporation before validation testing. No leakage was detected after tightening the bolts on the body of sample valve MCO-V-152.

The MCO process valve operator, sample supply hoses, HEPA filter FH-9, fittings and associated valves are pressure leak tested as a unit at 75 psig before opening the MCO process valve operator. Leakage was less than a 1-psi loss after a 10 minute hold period, which meets criteria of less than 40 cm^3/s (see Appendix A, Section A.4 for details).

5.1.3 Exhaust Gas Dilution

The MCO gas in the sample cart is diluted using the 120-psig-supply of helium before it enters the flexible hose to the sample hood exhaust system. The gas pressure of the cart is adjusted for MCO refill (normally about 12 psig) before it is released to the exhaust system at the sample hood. Proportional controller PIC–721 automatically controls the purge helium. As the gas pressure inside the sample cart (measured by PIT-721) decreases to near atmospheric pressure, the purge helium flow is throttled using valve PCV-721.

A dilution ratio of ten-to-one is used to dilute the sample cart gas. The ten to one dilution assures safe dilution of up to 8 percent hydrogen to less than 25 percent of the lower flammability limit in air based on theoretical amount of hydrogen from sampling a "best estimate" for a single scrap basket MCO after two years of interim storage. This "best estimate" single scrap basket MCO is the baseline MCO in the CSB process flow diagram by Klem and Pajunen (2000). Duncan (2001) shows that expected hydrogen concentrations for this case are 6.0 percent at zero year and 8.0 percent at two year storage time intervals.

Work packages 1S-01-00669 and 1S-01-00682 were used to establish a correlation between sample cart gas pressures and dilution ratio. A dilution ratio of ten-to-one was achieved by: carefully measuring the bleed flow rate of the cart gas with respect to cart pressure; carefully

measuring helium purge gas flow rate with respect to throttle valve position; and finally calibration of the proportional controller to provide ten times the helium purge gas with respect to the current cart gas pressure using throttle Valve PCV-721.

5.2 Helium Supply System

The helium supply system (GBM-5) for the sample/weld stations is an eight bottle double manifold (six bottles used for MCO gas sampling) with manual valves, pressure indicators and pressure safety relief valves. Work Package 1S-00-00341/M included removal of impurities from the inside of the piping and pressure testing of the system.

6.0 EQUIPMENT TEST RESULTS

This section discusses the test criteria, results of the tests and environmental considerations during the testing.

6.1 Test Criteria and Findings

The validation test criteria and findings are discussed in the following sections, which include helium purity, integrity of equipment, and operability/reliability of the equipment system.

6.1.1 Helium Purity

The helium purity criteria include meeting OCRWM requirements for helium that is used to refill MCOs. Table 4-4 lists the acceptance criteria and control method for ensuring helium purity requirements are met

Inspection and documentation methods are used to control batch conformance and helium bottle identification of purchased helium. During validation testing, no problems were identified with qualification of purchased helium; however the OCRWM document (FH 2002a) must be revised to show batch conformance and bottle identification before new batches of purchased helium maybe used for MCO gas sampling.

Laboratory analysis of sample cart helium is used to qualify MCO refill helium. Table 6-1 compares sample results to the criteria. A few of the blank helium samples did not meet criteria. Loose valves on two sample canisters and/or incomplete helium purging of the sample cart equipment resulted in excess air in helium blank samples 212H-121201-1348 and 212H-010402-1048. There was excess carbon dioxide in sample 212H-010702-2209.

Instruction was provided in OP-23-002S to improve removal of carbon dioxide and air from the sample equipment by the following actions: a) doubling the purge times to minimum 60 seconds, b) lengthening the time for vacuum pump down of the interface manifold and sample canister to minimum 4 minutes, c) firmly closing the sample canister valve after filling, and d) securing the sample valve closed using tamper-indicating tape. Purity of residual helium in the sample cart is expected to be better than shown in Table 6-1. The second 75 psig pressure test and system venting before opening the MCO process valve operator are performed after taking the helium

blank sample. This pressurization and venting purges additional contaminants from the sampling system resulting in improved helium purity.

Table 6-1: Acceptance Criteria and Laboratory Results of Helium Blank Samples[1, 2]

Constituent	Acceptance Criteria	Sample[3] 212H-121201-1348	Sample[4] 212H-121201-2156	Sample 212H-122001-1336	Sample[3] 212H-010402-1048	Sample 212H-010702-2209	Sample 212H-010902-2144
He	>99.5 %	99.5 %	99.99 %	99.97 %	99.28 %	99.98 %	99.98 %
H_2O	≤ 4580 ppm	<1000 ppm	<1000 ppm	<100 ppm	<100 ppm	Not detected	Not detected
THC (CH_4)	≤ 10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm
O_2+Ar	≤1020 ppm	970 ppm	<30 ppm	<60 ppm	1390 ppm	<30 ppm	<30 ppm
N_2	≤ 3600 ppm	4130 ppm	90 ppm	230 ppm	5800 ppm	180 ppm	170 ppm
H_2	≤ 10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm	<10 ppm
CO_2	≤ 11 ppm	<10 ppm	<10 ppm	<10 ppm	10 ppm	20 ppm	<10 ppm

Notes:
1) Key to sample number system: 212H is CSB; 121201 is date (month day year) and 1348 is time.
2) Results shown do not include estimate of precision value. See Appendix B for PNNL data transmittal letters.
3) Sample canister valve was reported loose by PNNL. Sample results exceed acceptance criteria.
4) Helium used to refill training MCO to 11.5 psig.

6.1.2 Sampling System Integrity

The sampling system integrity criterion includes the MCO process valve operator, sample hood, hoses, sample cart, interface manifold, and helium supply system.

The MCO sample cart, interface manifold, and sample cart exhaust hose to the sample hood were leak tested before and after validation testing. The systems were pressurized to 30 psig with helium and all fittings and valves were visually checked for leaks (~ 1xE-03 std cm^3/sec) using Work Package 1S-01-00655 and leak detection liquid. All visible leaks were repaired. The operator for valve PCV-721 utilities continual helium pressure bleed down. The manufacturer reports that the PCV-721 operator has a maximum consumption rate of 0.2 scfm at 20 psig based on air.

The interface manifold (vacuum seal fittings, valves and pressure indicator) and sample valve MCO-V-152 were gross leak tested by COGEMA Engineering Corporation before validation testing. Leakage was detected initially at the body of sample valve MCO-V-152. After tightening the nuts and bolts on the sample valve body, no helium leakage was detected on the interface manifold.

The MCO process valve operator, sample supply hoses, HEPA Filter FH-9, fittings, and associated valves are pressure leak tested at 75 psig before opening the MCO process valve operator as instructed in OP-23-002S. Typical pressure loss over a 10-minute period was < 1 psi (a pressure drop ≤5 psi is ≤ 4 std cm^3/sec - see Appendix A, Section A.4) and satisfies the TSR maximum leak rate of less than 40 std cm^3/sec inside the sample hood.

The modified helium supply system for MCO gas sampling was internally cleaned of impurities and the entire system (GBM-5 to support utility pits 3 and 6) pressure leak tested in accordance with Work Package 1S-00-00341/M. Two leak tests were performed. The first test criterion was less than a 2-psi-pressure loss in 15 minutes at 165-psig-test pressure. The second test criterion was a helium leakage rate of less than 1xE-03 std. cm^3/sec at 150-psig using commercial leak detection liquid. Both criteria were met.

6.2 Training MCO Gas Sample Test Results

This section discusses the MCO gas sample test results obtained using the conventional and direct sampling methods.

6.2.1 MCO Sample Cart Results

Four gas samples were obtained from the training MCO using OP-23-002S. The results are summarized in Table 6-2.

Sample 212H-121201-2243 was the first training MCO gas sample. The MCO gas pressure was 0.43 psig as measured on the sample supply line to the sample cart (surge valve MCO-V-151 closed) and 0.44 psig when the MCO gas was added to fill the main part of the sample cart (surge valve MCO-V-151 open). Both pressures were measured using PIT-721. The first training gas sample was mostly sample cart helium (sample cart helium pressure reduced to >0 psig and <1.0 psig before addition of training MCO gas to the main part of the sample cart). The first MCO sample (212H-121201-2243) could not be used to develop the correction factor for the MCO sample cart because of low gas pressure in the "as found" training MCO. The history of training MCO operations at K Basins did not provide adequate information about training MCO gas contents. The training MCO was refilled to 11.5 psig with helium represented by helium blank sample 212H-121201-2156 (see Table 6-1).

PNNL Radio Chemistry Laboratory personnel found a loose valve on the second MCO gas sample canister. The loose valve and/or air contamination of residual helium blank in the sample cart resulted in additional contamination of MCO sample 212H-010402-1143. Since these sample results are very close to the results of third and fourth sample events, air contamination is judged as minor for process calculations. Therefore MCO gas samples 212H-010402-1143, 212H-010702-2235 and 212H-010902-2232 were used to develop the gas correction factors for the MCO sample cart. There were no helium refills of the training MCO after the second sampling event.

Table 6-2: Training MCO Gas Sample Results Based on Conventional Usage of Sample Cart[1, 2]

Constituent	Sample[3] 212H-121201-2243	Sample[4] 212H-010402-1143	Sample 212H-010702-2235	Sample 212H-010902-2232
Ar	0.006 %	0.024 %	0.024 %	0.023 %
CO_2	<0.001 %	0.002 %	0.003 %	0.002 %
CO	<0.01 %	<0.01 %	<0.01 %	<0.01 %
He	99.4 %	97.5 %	97.5 %	97.5 %
H_2	<0.001 %	0.001 %	<0.001 %	<0.001 %
Ne	<0.001 %	<0.001 %	<0.001 %	<0.001 %
N_2	0.5 %	2 %	1.99 %	1.95 %
O_2	0.126 %	0.51 %	0.49 %	0.48 %
N_2O	<0.001	<0.005 %	<0.005 %	<0.005 %
NO_x	<0.001 %	<0.005 %	<0.005 %	<0.005 %
THC	<0.001 %	<0.001 %	<0.001 %	<0.001 %
H_2O	<1000 ppm	<1000 ppm	Not Detected	Not Detected
MCO Pressure in Supply Line[5]	0.43 psig	10.1 psig	9.9 psig	9.2 psig
Pressure of MCO Sample[6]	0.44 psig	9.8 psig	9.6 psig	8.9 psig
He Blank Residual Pressure of Sample Cart[7]	Not Available	Not Available	0.97 psig	0.77 psig

Notes:

1) Key to sample number system: 212H is CSB; 121201 is date (month day year) and 2243 is time.
2) Results do not include estimate of precision value. See Appendix B for PNNL data transmittal letters.
3) Low pressure training MCO. Sample obtained before helium refill to 11.5 psig using helium represented by blank sample 212H-121201-2156.
4) Sample canister valve was reported loose by PNNL.
5) MCO gas pressure measured on PIT-721 after opening port valve #2 and filling sample supply line. Ambient temperature was about 62 °F. Actual MCO would be refilled to this pressure.
6) MCO gas pressure measured on PIT-721 after opening MCO-V-152 surge valve to fill sample cart. Ambient temperature was about 62 °F.
7) Pressure of residual He in sample cart before addition of MCO gas. Ambient temperature was about 62 °F.

6.2.2 MCO Direct Sample Results

Two direct MCO gas samples were obtained using instructions in WP 1S-01-00711/W and 1S-02-00013/W. The results are nearly identical and are summarized in Table 6-3. The direct sampling equipment configuration moved the interface manifold from the sample cart valve (MCO-V-152) to the HEPA filter (FH-9) test port tee, disconnected tubing to PIT-721 on the sample cart and plugged the pressure tap pipe connection. Measurement of the MCO gas pressure could not be obtained during direct sampling because of equipment configuration. MCO gas pressures are estimated at about 10.1 psig for sample 212H-121901-2332 and about 9.2 psig for sample 212H-011002-1517 based on the ideal gas law. The training MCO was not refilled with helium after the direct sampling events.

Table 6-3: Training MCO Gas Sample Results Based on Direct Samples[1, 2]

Constituent	Sample 212H-121901-2332	Sample 212H-011002-1517	Average
Ar	0.028 %	0.027 %	0.0275 %
CO_2	0.003 %	0.002 %	0.0025 %
CO	<0.01 %	<0.01 %	<0.01 %
He	97.1 %	97.1 %	97.1 %
H_2	<0.001 %	<0.001 %	<0.001 %
Ne	<0.001 %	<0.001 %	<0.001 %
N_2	2.27%	2.28%	2.275 %
O_2	0.55 %	0.57 %	0.56 %
N_2O	<0.005	<0.005 %	<0.005 %
NO_x	<0.005	<0.005 %	<0.005 %
THC	<0.001	<0.001 %	<0.001 %
H_2O	<100 ppm	Not Detected	<100 ppm
Estimated Pressure of MCO Sample[3]	~10.1 psig	~9.2 psig	-

Notes: 1) Sample number system: 212H is CSB; 121201 is date (month day year) and 1348 is time.
2) Results do not include estimate of precision value. see Appendix B for PNNL data transmittal letters.
3) Actual MCO gas pressure could not be obtained because of equipment configuration. Estimated gas pressure based on ideal gas law.

6.2.3 Estimated MCO Gas Correlation Factors

Two correlation factors were developed from test data. The SCF is based on a ratio of constituent concentrations from sample results of the direct MCO gas sample and the MCO sample cart gas sample. It does not apply corrections for the MCO and sample cart temperature, pressure or void space volume. Equations 8 and 9 summarize this method.

SCF= [Direct MCO sample constituent concentration]/[MCO sample cart sample constituent concentration] (8)

[MCO] = [Sample Result] x SCF (9)

The average SCFs are about 1.15 for argon, carbon dioxide, nitrogen and oxygen, and 1.00 for helium. See Table 6-4 and Appendix A, Section A.1. The SCFs provide "best estimate" values of the constituent concentrations inside the MCO. Use of correlation factor of 1.14 is recommended to determine the oxygen concentration of the actual MCO.

The TCF is developed from residual helium pressure and final gas pressure conditions in the sample system and represents equilibrium conditions for perfect mixing of all gas in the sample system. Equation 6 and 7 summarize this method.

$$TCF = P_f / (P_f - P_r) \quad (6)$$

$$[MCO] = [Sample\ Result] \times TCF \quad (7)$$

The average TCF is 2.86. It is significantly larger than measured during sampling and represents the span of results assuming perfect mixing of all gas in the sample system. See Table 6-4 and Appendix A.1

Table 6-4: Correlation Factors for MCO Gas Sampling

He Blank Sample	212H-010402-1048[2]	212H-010702-2209	212H-010902-2144	Average Correlation Factor
MCO Sample Cart Sample	212H-010402-1143[2]	212H-010702-2235	212H-0100902-2232	
Major Constituent	Sample Based Correlation Factor[3]			
Ar	1.15	1.15	1.20	1.17
CO_2	1.25	0.88	1.25	1.13[1]
He	0.996	0.996	0.996	1.00
N_2	1.14	1.14	1.17	1.15
O_2	1.10	1.14	1.17	1.14
Overall	1.13	1.06	1.16	1.12
	Theoretical (Pressure Based) Correlation Factor[4]			
Overall	N/A[5]	2.82	2.90	2.86

Note:
1) Use value with caution because of difficulty in CO_2 removal from equipment.
2) Potential air contamination of this sample because of reported loose sample canister valve by PNNL and/or incomplete purging.
3) Based on Equation 8 (see previous page)
4) Based on Equation 6
5) Not available because residual helium pressure of sample cart was not recorded.

6.3 System Deficiencies

This section discusses deficiencies of the MCO sample system that were observed during the validation tests. These deficiencies are specifically related to support equipment, MCO Sample Cart, interface manifold (cart), and operating procedure.

6.3.1 Support Equipment

Pressure regulator PCV-108 of GBM-5 failed to control the helium supply set point pressure at 120 psig. PCV-107 and PCV-108 are parallel pressure regulator valves that are used to control the helium supply pressure to the set point of 120 psig for the sample weld stations. PCV-108 failed to operate properly during validation testing and this required use of PCV-107 to regulate the helium supply pressure. CSB Engineering recommends replacement of PCV-108 with new regulator valve.

Manual valve HE-V-77 of GBM-5 made a loud popping sound when opened. HE-V-77 is an isolation valve on one side of the double manifold 120-psig-helium supply to the sample weld stations. Telephone conversations with the supplier indicate that the valve is questionable and should be replaced; CSB Engineering concurs with the supplier's recommendation.

The low-pressure helium regulator of GBM-5 did not function as designed. The low-pressure system was designed to maintain (via PCV-111) a 3-psig pressure on the piping from GBM-5 to the sample pit isolation valves (HE-V-62-A at Pit 2 and HE-V-63-A at Pit 7) when the system is not in use. CSB Engineering recommends an evaluation and repair of the low-pressure maintenance helium system.

6.3.2 MCO Sample Cart

Validation testing has shown that the pressure regulator PCV-726 does not control the sample cart helium at the set point pressure nor does it have the capacity to supply 30-psig-helium for refill of a maximum pressure MCO. PCV-726 provides nominal 12-psig-helium supply for purging of the sample cart, flexible hoses, and refill of the monitored MCOs. Bleed valve HE-V-170 is used to maintain the set point pressure of the helium purge. CSB Engineering recommends replacement of PCV-726 with a new regulator with pressure control capacity greater than 30 psig. The new regulator should be easily adjustable by the operator and control the pressure to within plus or minus 1 psig of the set point. Bleed valve (HE-V-170) is used to reduce the downstream pressure if the pressure goes above the set point.

Thermocouple (TE-723) was found defective during validation testing and a replacement was ordered. Due to the tight sampling schedule, TE-723 was repaired and re-qualified before the new thermocouple was available. CSB Engineering recommends installation of the replacement thermocouple when convenient. Subsequent calibration information has shown that the repaired thermocouple data collected was valid.

The continuous helium bleed at the operator for valve PCV-721 is a significant source of helium usage. CSB Engineering recommends the installation of a manual valve in the helium supply line to isolate PCV-721 and significantly reduce helium usage.

Temperature indicator (TIT-723) does not have appropriate resolution to accurately observe the sample cart gas temperature. The 20 °F increments make it difficult to interpolate with good precision the actual MCO gas temperature. CBS Engineering recommends that the current analog temperature indicator be replaced with digital temperature indicator. The digital indicator

must be OCRWM certified and have appropriate resolution of the temperature over the same range (0 °F – 300 °F).

6.3.3 Interface Manifold

The interface manifold vacuum gauge (PI-743) did not give reliable and repeatable pressure measurement under ambient pressure (non vacuum) conditions. The ambient pressure readings of PI-743 were 1000 torr instead of about 750 torr. CSB Engineering recommends an evaluation of pressure transducer (PT-742) and pressure indicator (PI-743) instruments. Spares for both PT-742 and PI-743 are available for use.

6.3.4 Operating Procedure

Improvements to Operating Procedure OP-23-002S will be required because of recommended equipment changes. Further procedure changes could result from proposed follow-on sampling of another training MCO or an MCO with known gas composition. OP-23-002S was developed from drawings of the MCO sampling equipment, vendor equipment information, validation tests results, and initial training of MCO gas sampling at normal operating conditions.

CSB Engineering recommends follow-on evaluation of the sampling operation after completion of proposed equipment changes. The follow-on evaluation is desired in order to better evaluate purging efficiency, potential ingress of air, check correlation factors, improve understanding of mixing behavior of helium and MCO gas, and provide a basis for additional procedure changes.

7.0 DISPOSITION OF GAS SAMPLES

The helium blank and training MCO gas samples are discharged to the PNNL exhaust system and the sample canisters are evacuated to 1E-05 torr after completion of the analysis and approval of the sample report. Empty and evacuated sample canisters (75 cm^3) are stored at PNNL or CSB until needed for the next gas-sampling event.

The potential in leakage of air into the PNNL evacuated sample canisters during storage necessitates that the canister be evacuated to <0.1 torr using the mechanical vacuum pump on the interface manifold before taking of the sample. The vacuum pump has the capability to achieve a vacuum of 0.01 torr.

8.0 CONCLUSIONS AND RECOMMENDATIONS

The validation testing of the MCO sampling system showed that although some minor equipment deficiencies were identified the equipment could be used to satisfactorily sample the first monitored MCO. There is some uncertainty in the results from the training MCO. System improvements are needed to provide flexibility and reduce uncertainty for future MCO gas sampling.

The following recommendations are provided to improve the gas sampling system.

Support Equipment Improvements:

- Replace failed Pressure Regulator PCV-108 and Manual Valve HE-V-77 of GBM-5.
- Perform evaluation and repair of the low-pressure maintenance system of GBM-5.

MCO Sample Cart Improvements:

- Replace rebuilt Thermocouple TE-723 with the replacement thermocouple.
- Perform calibration checks of repaired and replacement TE-723 thermocouples to meet OCRWM requirements. Calibration verification of the rebuilt thermocouple validates the temperature data obtained during sampling of the actual MCO.
- Replace current analog Temperature Indicator TIT-723 with digital temperature indicator that is OCRWM certified and has more useful readout resolution of the temperature over the same range (0 °F – 300 °F).
- Replace PCV-726 with a new regulator having higher-pressure control range.
- Install new manual isolation valve on helium supply line to PCV-721 to reduce helium usage.

Interface Manifold Improvements:

- Perform an evaluation of pressure transducer (PT-742) and pressure indicator instruments to determine source of irregular measurements.

Operating Procedure Improvements:

- Revise OP-23-002S to reflect and validate corrected equipment deficiencies.
- Revise OP-23-002S to include the option to perform certain steps in parallel and reduce the time to complete the sampling event.
- Revise OP-23-002S to lengthen the purge time and reduce potential air contamination during future MCO gas sampling.
- Revise OP-23-002S to reduce gas-mixing uncertainty after completion of additional sampling tests.

Additional MCO Sampling Tests:

- Perform additional sampling tests to improve understanding of gas mixing behavior after closing the sample cart surge valve.

Correlation factors were developed to relate results of sample cart samples to results of direct samples of gas inside the training MCO. The average SCF is about 1.15 for argon, carbon dioxide, nitrogen, and oxygen and 1.00 for helium. The oxygen correction factor is 1.14. These SCFs are "best estimate", their basis is understood, and they can be used without reservation until further notice. TCFs were developed from gas pressure data and represent the span of results for perfect mixing of all gas in the sample system. The average TCF is 2.86.

Changes to equipment and to the operating procedure should be implemented to guard against potential air contamination of the sample cart during future MCO gas sampling. Follow-on MCO gas sampling tests are needed to validate the equipment improvements and reduce mixing uncertainty.

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APPENDIX A

SUMMARY OF SAMPLING PROCESS CALCULATIONS

A.1 Calculate Gas Sample Correlation Factors for Training MCO

Two correlation factors were developed from test data of training MCO TR-003. The sample based correlation factor (SCF) is the ratio of major constituent concentrations from the direct MCO gas sample and the conventional MCO sample cart gas sample. The MCO gas sample was taken a few minutes after the MCO gas was admitted to the sample cart and mixed with some residual helium in the sample cart system. Concentration ratios do not require corrections for temperature and pressure. The gas temperature in the training MCO and sample cart was assumed to be the same as the CSB operating deck temperature (62 ° F or 16.7 ° C) because TIT-723 was not operational. Equations A-1and A-2 summarize this method.

SCF = Direct MCO sample constituent concentration / MCO sample cart constituent concentration (A-1)

[MCO] = [Sample Result] x SCF (A-2)

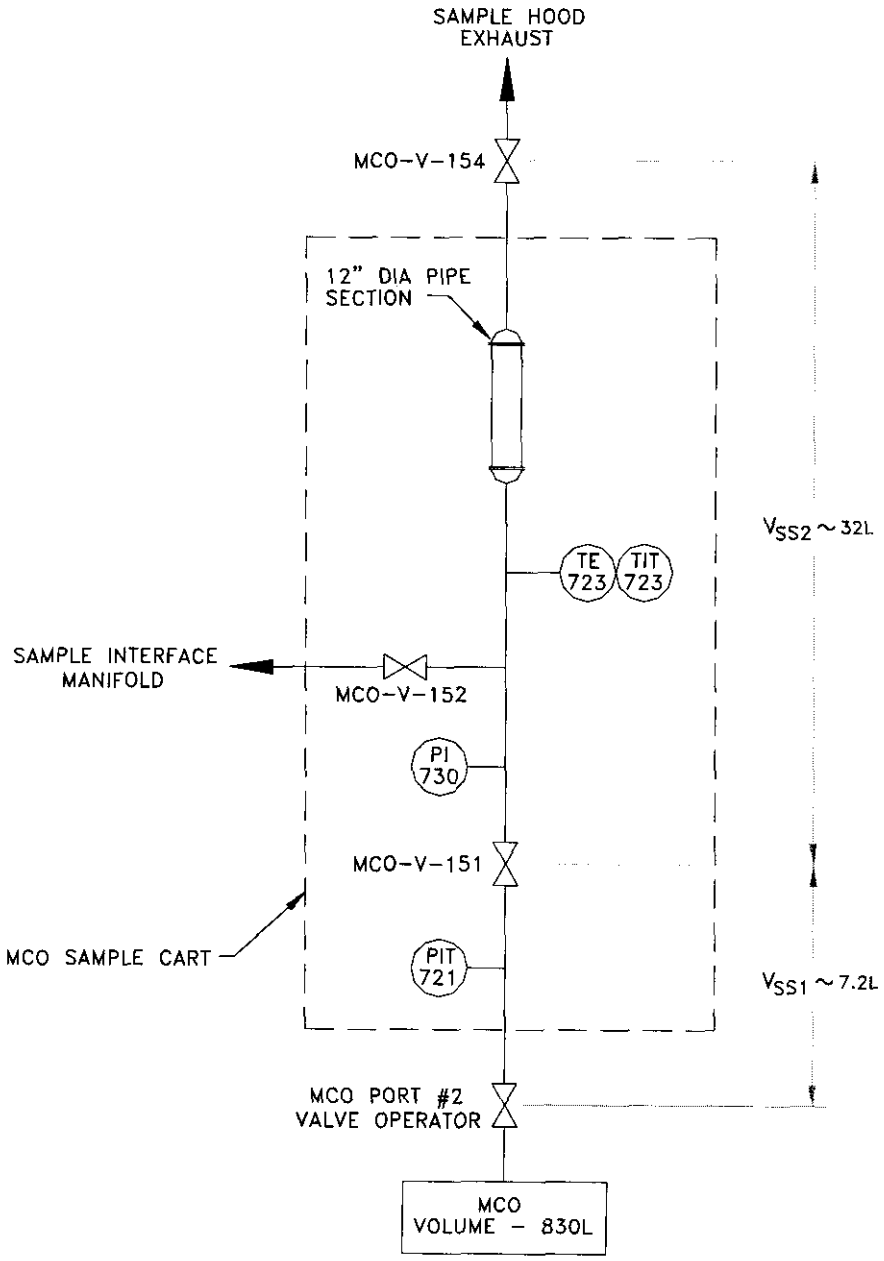
The theoretical pressure based correlation factor (TCF) is the ratio of the final gas pressure in the sample system after addition of MCO gas and the final gas pressure corrected for residual helium pressure of the sample cart system before addition of MCO gas. The TCF represents the equilibrium conditions for perfect mixing of all gas in the sample system and upper bound of the correlation factor. There was no correction of pressure due to temperature because the gas in the sample system was at the same temperature. Pressure corrections would be dependent on any temperature change after completion of gas mixing. Equations A-2 and A-3 summarize this method.

$$TCF = P_f / (P_f - P_r) \quad \text{(A-3)}$$

Where: P_f = pressure at time of sampling, psia
P_r = pressure of residual helium in system before sampling, psia

[MCO] = [Sample Result] x TCF (A-4)

Figure A-1 is a schematic showing MCO and MCO sample cart system void volumes. The MCO is attached to the sample cart using a process valve operator and flexible hoses. The process valve operator isolates the MCO from the sampling system. Surge valve MCO-V-151 isolates the MCO gas from the main portion of the sample cart and remains closed to allow for the best measurement of the MCO gas pressure on PIT-721. Opening surge valve MCO-V-151 fills the sample cart (including gas accumulator or reservoir) with MCO gas, permits measurement of the MCO gas temperature on TIT-723 and gas sampling at valve MCO-V-152 using the interface manifold. Valve MCO-V-154 is used to exhaust the sample cart gas to the sample hood exhaust system via a flexible hose. The sample hood and sample cart contain additional equipment that will not be discussed in this section.



MCO SAMPLING SYSTEM VOID VOLUMES
FIGURE A-1

Table A-1 summarizes the helium blank sample results from the conventional sample system. Purity of residual helium is expected to be better than shown below because second 75-psig-pressure test and venting operation is performed before opening the MCO valve operator and after taking the helium blank sample. Table A-2 summarizes the valve positions and void volume of residual helium blank in the sample system

Table A-1: Sample Results Helium Blank Major Constituents Based on Conventional Sampling Method

He Blank Cart Sample	Ar Mole %	CO_2 Mole %	He Mole %	N_2 Mole %	O_2 Mole %	Residual Pressure Psig
212H-010402-1048	7E-03	1E-03	9.928E01	5.8E-01	1.32E-01	N/A
212H-010702-2209	<1E-03	2E-03	9.998E01	1.8E-02	2E-03	0.97
212H-010902-2144	<1E-03	1E-03	9.998E01	1.7E-02	2E-03	0.77

Table A-2: Residual Helium Blank Only Valve Positions

Component	Valve Position	Est. System Void Volume[1], L
MCO TR-003	N/A	830
TR-003 to Port 2 valve operator	Closed	Small
Port 2 valve operator to MCO-V-151	Open	7.2
MCO-V-151 to MCO-V-154	Closed	32

Note: 1) Estimated volume based on physical dimensions of components.

Table A-3 summarizes initial valve positions of the sample system before gas sampling of the training MCO. Table A-4 summarizes the combined residual blank helium and training MCO gas pressures at the head end of the sample cart with valve MCO-V-151 closed. Table A-5 and Table A-6 summarize final valve positions and conditions of the residual blank helium and training MCO gas in the sample system with valve MCO-V-151 open.

Table A-7 lists the sample results of training MCO gas major constituents based on conventional sampling. Table A-8 lists the composition of the training MCO gas major constituents based on direct sampling. Table A-9 summarizes correlation factors as calculated by Equations A-1 and A-3.

Table A-3: Initial Valve Positions for Training MCO Gas

Component	Valve Position	Est. System Void Volume[1], L
MCO TR-003	N/A	830
TR-003 to Port 2valve operator	Open	Small
Port 2 valve operator to MCO-V-151	Closed	7.2
MCO-V-151 to MCO-V-154	Closed	32

Note: 1) Estimated volume based on physical dimensions of components.

Table A-4: Training MCO Gas In Head End of Sample Cart with Valve MCO-V-151 Closed

MCO Cart Sample	PIT-721 psig	TIT-723 °F
212H-010402-1143	10.15	N/A
212H-010702-2235	9.94	N/A
212H-010902-2232	9.2	N/A

Table A-5: Final Valve Positions for Training MCO Gas

Component	Valve Position
MCO TR-003	N/A
TR-003 to Port 2 valve operator	Open
Port 2 valve operator to MCO-V-151	Open
MCO-V-151 to MCO-V-154	Closed

Table A-6: Training MCO Gas In Sample Cart with MCO-V-151 Open

MCO Cart Sample	PIT-721 psig	TIT-723 °F
212H-010402-1143	9.81	N/A
212H-010702-2235	9.60	N/A
212H-010902-2232	8.93	N/A

Table A-7: Training MCO Gas Major Constituents Based on Conventional Sampling Method

Training MCO Conventional Sample	Ar Mole %	CO_2 Mole %	He Mole %	N_2 Mole %	O_2 Mole %
212H-010402-1143	2.4E-02	2E-03	9.75E01	2.0E00	5.1E-01
212H-010702-2235	2.4E-02	3E-03	9.75E01	1.99E00	4.9E-01
212H-010902-2232	2.3E-02	2E-03	9.75E01	1.95E00	4.8E-01
Average	2.37E-02	2.3E-03	9.75E01	1.98E00	4.93E-01

Table A-8: Training MCO Gas Composition Based on Direct Sampling Method

Training MCO Direct Sample	Ar Mole %	CO_2 Mole %	He Mole %	N_2 Mole %	O_2 Mole %
212H-121901-2332	2.8E-02	3E-03	9.71E01	2.27E00	5.5E-01
212H-011002-1517	2.7E-02	2E-03	9.71E01	2.28E00	5.7E-01
Average	2.75E-02	2.5E-03	9.71E01	2.275E00	5.6E-01

Table A-9: Major Constituent Correlation Factors

MCO Cart Sample	Ar		CO_2		He		N_2		O_2	
	SCF	TCF	SCF	TCF	SCF	TCF	CSF	TCF	SCF	TCF
212H-010402-1143	1.15	N/A	1.25	N/A	0.996	N/A	1.14	N/A	1.10	N/A
212H-010702-2235	1.15	2.82	0.83	2.82	0.996	2.82	1.14	2.82	1.14	2.82
212H-010902-2232	1.20	2.90	1.25	2.90	0.996	2.90	1.17	2.90	1.17	2.90
Average	1.17	2.86	1.13	2.86	1.00	2.86	1.15	2.86	1.14	2.86

Example calculations of the SCF and TCF are shown below for MCO cart sample 212-0107-02-2235.

MCO direct sample average result for Ar = 2.75E-02 mole % (See Table A-8)
MCO sample cart result for Ar = 2.4E-02 mole % (See Table A-7)
Residual blank He pressure before addition of MCO gas = P_r = 0.97 psig (See Table A-1)
Residual blank He pressure and training gas pressure MCO = P_f = 9.60 psig (See Table A-6)

SCF Ar = [Direct MCO sample]/[Cart MCO sample] = 2.75E-02 %/2.4E-02 % = 1.15 (A-5)

TCF overall = $P_f/(P_f - P_r)$ = (9.60 +14.7 psia)/((9.60-0.97) psi) = 2.82 (A-6)

A.2 Calculate Training MCO Void Volume

The Ideal Gas Law is used to check the estimated void volume of the training MCO. Equation A-7 relates the observed initial pressures and volumes of the combined training MCO and sample system lines (between process valve operator, surge valve MCO-V-151 and block valve MCO-V-154) to the observed final pressure and volumes at the same temperature. The temperature of the equipment was assumed to be the same as the CSB deck temperature (62 °F or 16.7 °C). See Figure A-1.

$$P_{MCO1} \times (V_{MCO} + V_{SS1}) + P_{SS} \times V_{SS2} = P_{MCO2} \times (V_{MCO} + V_{SS2} + V_{SS1}) \quad (A\text{-}7)$$

Substituting and solving Equation A-7 for V_{MCO} gives equation A-8.

$$V_{MCO} = [39.2\ L \times P_{MCO2}\ psia - 32\ L \times P_{SS}\ psia - 7.2\ L \times P_{MCO1}\ psia]/[(P_{MCO1} - P_{MCO2})\ psi] \quad (A\text{-}8)$$

Where:

P_{MCO1} is gas pressure of training MCO as measured on PIT-721 with process valve operator open and MCO-V-151 closed psia.
P_{MCO2} is gas pressure of training MCO as measured on PIT-721 with process valve operator open, MCO-V-151 open and MCO-V-154 closed psia.
V_{MCO} is the training MCO void volume, L.
V_{SS1} is void volume between Port 2 valve operator and MCO-V-151 $\cong$ 7.2 L.
V_{SS2} is void volume between MCO-V-151 and MCO-V-154 $\cong$ 32 L.
P_{SS} is gas pressure of residual helium blank in sample system before addition of training MCO gas as measured on PIT-721, psia.

Equation A-9 shows an example calculation based on Equation A-8. Table A-10 lists the gas pressures and calculated void volume of the training MCO. The validation test derived results are within 20 percent of the 830 L void volume that was estimated from physical dimensions and knowledge of the empty baskets inside the training MCO based on information in Bader (2002b).

V_{MCO} = [39.2 L x (9.60 + 14.7 psia) – 32 L x (0.97 + 14.7 psia) – 7.2 L x (9.94 + 14.7 psia)]/[9.94 – 9.60 psi] = 805 L (A-9)

Table A-10: Void Volume of Training MCO Based on Ideal Gas Law

MCO Cart Sample	MCO Pressure, psig			Sample System Residual Helium Pressure, psig	MCO Void Vol., L	
	P_{MCO1}	P_{MCO2}	ΔP_{MCO}	P_{SS}	Gas Law	Physical Dimensions
212H-010402-1143	10.15	9.81	0.34	N/A	N/A	830
212H-010702-2235	9.94	9.60	0.34	0.97	805	
212H-010901-2232	9.20	8.93	0.27	0.77	960	

Calculation of the training MCO void volume based on physical dimensions is presented below.

Void volume empty MCO = 1000 L

Volume of empty fuel basket = 28.3 L

The volume of the empty fuel basket was derived from density of basket material and weight of basket. Six empty fuel baskets are inside the training MCO.

Volume of six empty fuel baskets = 6 x 28.3 L = 170 L (A-10)

Void volume training MCO = Volume empty MCO - Volume empty fuel baskets (A-11)

Void volume training MCO = 1000 L – 170 L = 830 L (A-12)

A.3 Calculate MCO Gas Pressure Based on MCO Void Volume

The MCO gas pressure is calculated from the void volume and pressure information in Section A.2 at constant temperature. Equation A-13 relates the observed initial pressures and volumes of the combined training MCO and sample system lines (between process valve operator and surge valve MCO-V-151 to the observed final pressure and volumes at the same temperature. (see Figure A-1)

$V_{MCO} \times P_{MCO} + V_{SS1} \times P_{SS} = V_{MCO} \times P_{MCO1} + V_{SS1}\ L \times P_{MCO1}$ (A-13)

Substituting solving Equation A-13 for P_{MCO} gives equation A-14.

$P_{MCO} = [P_{MCO1} \times (V_{MCO} + 7.2L) - 7.2LP_{SS}]/(V_{MCO})$ (A-14)

Where:

P_{MCO} is actual gas pressure of MCO, psia
P_{MCO1} is gas pressure of training MCO as measured on PIT-721 with valve operator open and MCO-V-151 closed, psia.

P_{MCO2} is gas pressure of training MCO as measured on PIT-721 with valve operator open, MCO-V-151 open, and MCO-V-154 closed, psia (Not used in Equation A-13 but value shown in Table A-11 for continuity).
V_{MCO} is training MCO void volume, L.
V_{SS1} is void volume between Port 2 valve operator and MCO-V-151 ≅ 7.2L
P_{SS} is gas pressure of residual helium blank in sample system before addition of training MCO gas as measured on PIT-721, psia.

Equations A-15 and A-16 show an example calculation based on MCO Cart Sample 212H-010702-2235 (see Table A-10). Table A-11 lists the estimated actual gas pressures of the training MCO at constant temperature. The results show there is less than 0.1-psi pressure differences between the empirical gas pressure of the MCO (P_{MCO}) and the gas pressure as measured in the sample cart on PIT-721 with MCO-V-151 closed (P_{MCO1}).

$$P_{MCO} = [(9.94 + 14.7 \text{ psia}) \times (805 \text{ L} + 7.2 \text{ L}) - 7.2 \text{ L} \times (0.97 + 14.7 \text{ psia})]/ 805 \text{ L} = 24.7 \text{ psia} \quad \text{(A-15)}$$

$$P_{MCO} = 24.7 \text{ psia} - 14.7 \text{ psia} = 10.0 \text{ psig} \quad \text{(A-16)}$$

Table A-11: Pressure of Training MCO Based on Ideal Gas Law

MCO Cart Sample	MCO Pressure, psig			Sample System Residual Helium Pressure, psig	MCO Void Vol., L (Ideal Gas Law)
	P_{MCO}	P_{MCO1}	P_{MCO2}	P_{SS}	V_{MCO}
212H-010402-1143	N/A	10.15	9.81	N/A	N/A
212H-010702-2235	10.0	9.94	9.60	0.97	805
212H-010901-2232	9.26	9.20	8.93	0.77	960

A.4 Calculate Gas Leakage Rate for Pressure Decay Test

The TSR requires 75-psig pressure decay test of the MCO valve operator and MCO gas supply line inside sample hood BARR-002 within one hour prior to opening the valve operator (Krahn 2000). The maximum leakage rate is 40 std cm^3/sec. The pressure decay test is performed on the line between the process valve operator and surge valve MCO-V-151 and uses PIT-721 for pressure measurement. The following calculation is the basis of the maximum 5-psi pressure loss (ΔP) criteria over a 10 minute hold period on the line between the process valve operator and surge valve MCO-V-151 (V_{SS1} ≅ 7.2 L, see Figure A-1). Gas temperature is based on operating deck temperature of 62 °F (16.7 °C).

$$\Delta n \text{ g mole/sec} = \Delta P V_{SS1}/(RT600 \text{ sec}) \quad \text{(A-17)}$$

$$\Delta n = (5 \text{ psi} \times 7.2 \text{ L}) /(0.082 \text{ L atm/g mole } ^\circ\text{K} \times 289.8 \ ^\circ\text{K} \times 14.7 \text{ psia/atm} \times 600 \text{ sec}) \quad \text{(A-18)}$$

$$\Delta n = 1.72\text{E-}04 \text{ g mole/sec} \quad \text{(A-19)}$$

Leakage rate based on ideal gas g mole volume of 22,400 cm^3 /g mole at standard temperature and pressure is shown in Equation A-20.

Line leakage rate = (22,400 cm^3/g mole) x 1.72E-04 g mole/sec = 3.8 std cm^3/sec (A-20)

Therefore, line leakage rate based on the 5-psi pressure drop is well below maximum allowable rate of 40 std cm^3/sec.

APPENDIX B

PACIFIC NORTHWEST NATIONAL LABORATORY
GAS SPECIES ANALYSIS SAMPLE RESULT
TRANSMITTAL LETTERS

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

43469-L01

December 18, 2001

Frank W. Moore
Fluor Hanford, Inc.
PO Box 1000
Richland, WA 99352

Dear Mr. Moore:

TRANSMITTAL OF REPORT 43469-RPT01, "GAS SPECIES ANALYSIS OF SAMPLES FROM THE CANISTER STORAGE BUILDING RECEIVED BY PNNL ON DECEMBER 13, 2001"

Reference: Statement of Work "Analysis of Gas Samples from the Spent Nuclear Fuel (SNF) Canister Storage (CSB) Facility" transmitted to PNNL on October 26, 2001, accompanying contract 11979-36 (PNNL Project 43469).

Attached please find a copy of report 43469-RPT01, "Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001." The samples described in this report were received by PNNL on December 13, 2001, and preliminary results were transmitted on December 13, 2001. The attached report is the final deliverable associated with these samples.

The sampling media used to obtain these samples were provided by CSB staff. Therefore, PNNL did not perform a cylinder cleaning check prior to obtaining samples. No cylinder cleaning documentation is included in the attached report.

All analyses performed and results reported in the attached document were conducted in compliance with OCRWM standards. This data has been reviewed and determined to be OCRWM-compliant.

Radiochemical Processing Laboratory (RPL) sample identification number used in this report was assigned as follows:

RPL Sample ID	Client Sample ID	Client Sample Description
02-00899	212H-121201-1348	He Gas Sample
02-00900	212H-121201-2156	He Gas Sample
02-00901	212H-121201-2243	MCO Gas Sample

If you have any question, please give me a call on 372-4828.

Sincerely,

Kurt L. Silvers,
PNNL Project Manager

902 Battelle Boulevard • P.O. Box 999 • Richland, WA 99352

Telephone (509) 372-4828 ■ Email kurt.silvers@pnl.gov ■ Fax (509) 372-6515

Frank W. Moore
December 18, 2001
Page 2

Attachment

E Biebesheimer, FH (summary only)
MS Busch, FH (summary only)
DR Duncan, FH (full data package)
SL Moore, FH (summary only)
DW Smith, FH (full data package)

GD Bazinet, NHC (summary only)
JP Sloughter, NHC (summary only)

SJ Bos, PNNL (full data package)
AM Lewis, PNNL (summary only)
BM Thornton, PNNL (summary only)
43469 project file

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

December 18, 2001

Pacific Northwest National Laboratory

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Sample Analysis Letter

December 18, 2001

Pacific Northwest National Laboratory

Battelle
. . . Putting Technology To Work

43469
Project No. ~~42047~~ BMS 12/17/01

Internal Distribution
File/LB

Date December 14, 2001

To Kurt Silvers

From Stan Bos Stan Bos

Subject Canister Storage Building gas sample analysis

Gas species analyses of samples, taken at the Canister Storage Building, on December 12, 2001, have been completed. A report detailing the gas species detected is attached. Analysis was performed using the Finnigan MAT-271 (WC38625) high sensitivity quantitative mass spectrometer. The sensitivity of the instrument is checked daily prior to use with high purity N2 gas and two air standards are run weekly to insure the instrument is operating correctly. The samples were assigned RPL sequence numbers 02-00899 through 02-00901.

This analysis was performed, and the report prepared following PNNL quality assurance plan Nuclear Quality Assurance Requirements and Description (NQARD). NQARD has been evaluated and found to be in conformance with the Office of Civilian Radioactive Waste Management (OCRWM) QA Program. The data in this report is OCRWM QARD Qualified Data.

PNNL project 43469 has setup for document and financial control of the sample analyses. Sample reports and data sheets are stored in the project RIDS located in ETB room 2619. Sample analyses are charged to work package F29087.

If you have any questions please contact Stan Bos at 376-5384.

Concurrence R. K. Bunz 12/14/01

ES4-1900-001 (8/98)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Gas Analysis Summary Results

December 18, 2001

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: December 14, 2001
Subject: Gas Species Analysis

To: Kurt silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 136
Measurement and test equipment WC38625

Sample Id.	212H-121201-1348		
Analysis Date:	December 13, 2001	Mole Percent	Estimate of Precision	PPM
Log-In No.	02-00899		
Argon		0.005	± 0.001	50
Carbon dioxide		<0.001	± 0	<10
Carbon monoxide		<0.01	± 0	<100
Helium		99.5	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.413	± 0.008	4130
Oxygen		0.092	± 0.002	920
Nitrous oxide		<0.001	± 0	<10
Other nitrogen oxides		<0.001	± 0	<10
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water <1000 ppm			

Sample Id.	212H-121201-2156		
Analysis Date:	December 13, 2001	Mole Percent	Estimate of Precision	PPM
Log-In No.	02-00900		
Argon		<0.001	± 0	0
Carbon dioxide		<0.001	± 0	<10
Carbon monoxide		<0.01	± 0	<100
Helium		99.99	± 0.01	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.009	± 0.001	90
Oxygen		0.002	± 0.001	20
Nitrous oxide		<0.001	± 0	<10
Other nitrogen oxides		<0.001	± 0	<10
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water <1000 ppm			

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: December 14, 2001
Subject: Gas Species Analysis

To: Kurt silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 136
Measurement and test equipment WC38625

Sample Id.	212H-121201-2243			
Analysis Date:	December 13, 2001	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-00901			
Argon		0.006	± 0.001	60
Carbon dioxide		<0.001	± 0	<10
Carbon monoxide		<0.01	± 0	<100
Helium		99.4	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.5	± 0.01	5000
Oxygen		0.126	± 0.003	1260
Nitrous oxide		<0.001	± 0	<10
Other nitrogen oxides		<0.001	± 0	<10
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water <1000 ppm			

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Chain-of-Custody Form

December 18, 2001

Pacific Northwest National Laboratory

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-121301-1 Date 12-13-01 Field Logbook No. NA

Point of Contact Doug Black Phone No. 372-2543 MSIN 58-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300 I

Sampled By STEVEN B CARTER (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
2124-121201-1348	212H/MCO-TR003	12-12-01	1348 hrs	NA	Helium Gas Sample
2124-121201-2156	212H/MCO-TR003	12-12-01	2156 hrs	NA	Helium Gas Sample
2124-121201-2243	212H/MCO-TR003	12-12-01	2243 hrs	NA	MCO Gas Sample

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions:

Sample Analysis to be performed per CSB/212H Agreement, Ref Contract #8726 "CSB Statement of Work". COO 11979-00036 SBL 12-13-01

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
STEVEN B CARTER	12-13-01	1105	RICHARD ADAMS	12-13-01	1105
RICHARD ADAMS	12-13-01	1159	S.J. BOS	12-13-01	1159

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ _____ _____ _____

(Print) (Sign) (Date) (Time)

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Analytical Services Request

December 18, 2001

Pacific Northwest National Laboratory

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor: Signature 12/13/01
Print Name Kurt Silvers
Phone 372-4828 MSIN K9-08

PNNL Project #:
Charge Code: F29087
Date Required: 12-31-01

Matrix Type Information

- ♦ Liquids: ___ Aqueous __ Organic ___ Multi-phase
- ♦ Solids: ___ Soil ___ Sludge ___ Sediment
  ___ Glass ___ Filter __ Metal
  ___ Smear ___ Organic ___ Other
- ♦ Other: ___ Solid/Liquid Mixture, Slurry
  X Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ QA Plan:
  SBMS
  HASQARD (CAWSRP)
- ♦ Additional QA Requirements? No X
  or Reference Doc # PRS 12/14/01
- ♦ Field COC? No X Yes X
- ♦ Lab COC Required? No X Yes
- ♦ Hold Time: None X
  or RCRA CERCLA
  Other, Specify
  & Date Sampled
  Time Sampled
- ♦ Special Storage Requirements:
  None X Refrigerate (4°C)
  or Other, specify
- ♦ Data Quality Review Required? No X Yes

Disposal Information

- ♦ Disposition of Virgin Samples:
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc #
- ♦ Disposition of Treated Samples:
  Dispose Return

Waste Designation Information

- ♦ Sample Information Check List Attached? Yes
  or Reference Doc #
  or Previous ASR # 5749
  or Previous RPL ID #

Does the Waste Designation Documentation Indicate Presence of PCBs?
No X Yes

Additional or Special Instructions

Send Report To Kurt Silver Phone 372-4228
Phone

Preliminary results requested, as available? No ___ Yes ✓ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12-13-01
Delivered By (optional)
Time Delivered (optional)
Group ID (optional)
CMC Waste Sample? No X Yes
Cost Estimate, if requested: $

Received By: S. J. Bos

ASR Number: 6304
RPL Numbers: (02-00899)-(02-00901)

RPG/CMC Work Accepted By: SJ Bos Signature/Date: 12/13/01

ASR FY2000 - RPG.doc

Page 1 of 2

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
02-00899	212H-121201-134	He gas	Gas Species	H Gas Anal	H2O Inorg
-00900	212H-121201-2156				
-00901	212H-121201-2243				

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___ ; On Attachment ___]

ASR # 6304

Page 2 of 9

ASR FY2000 - RPG.doc

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Quality Control Check

December 18, 2001

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-3358 / mail slot P7-22
Date: December 10, 2001
Subject: Air standards from Finnigan MAT - 271 Mass Spectrometer

Analytical procedure: PNNL - 98523 - 284 Rev. 0
Laboratory Record Book 56998: Page 135
Measurement and test equipment WC38625

Accepted values for the composition of air :

	Mole percent
Argon	0.934
Nitrogen	78.08
Oxygen	20.95

Analyzed Values:

Analysis Date: December 10,2001

	Mole percent
Argon	0.938
Nitrogen	78.02
Oxygen	21.00

Analyzed Values:

Analysis Date: December 10,2001

	Mole percent
Argon	0.937
Nitrogen	77.89
Oxygen	21.13

Instrument Background:

Background analyses are run daily prior to sample analyses. Trace amounts of hydrogen and/or water in the 0.1 to 0.2 millivolt range were the only species detected. The background spectra is subtracted from each sample spectra.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 13, 2001

43469-RPT01, Rev. 0

Raw Instrument Data from Gas Analysis

December 18, 2001

Pacific Northwest National Laboratory

Page 1 of 6

MATRIX - EVALUATION LIST of 212H-121201-1348 13 Dec 2001 13:49

SAMPLE ID:	212H-121201-1348	ALO NO.:	02-00899
ANALYST:	STAN BOS S G Bos	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	13 Dec 2001 (13:30)	PAGE:	136
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HELIUM	99.49020	96.43703	.133487
N2	.41268	2.80008	.000554
O2	.09210	.71418	.000124
ARGON	.00502	.04870	.000007
TOTAL	100.00000	100.00000	.134171
TOTAL C1-C4		0.00000	

CLOSURE [%] : 1.419 MEASURED PRESSURE: .1323

Sample cylinder valve could have been closed tighter. May have contributed to air inleakage.

Reviewed By: P.K. Berry Date: 12/14/01

Page 2 of 6

◂‡S P E C T R U M L I S T :SAMPLE SPECTRUM 13 Dec 2001 13:46

SAMPLE ID: 212H-121201-1348
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 13 Dec 2001 (13:30)

ALO NO.: 02-00899
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 136
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 132.29 mTorr
ANALYSIS TIME : 16:32.555
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 96.22 mTorr
INTEGR.INTENSITY: 2.1800E+0 V
BASE PEAK INT. : 2.1223E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	7.5010E-5	.0035	.0034	7	30.3
2	4.002604	@	4.0	2.1223E+0	100.0000	97.3537	3	62.4
3	14.003074	@	14.0	2.7437E-3	.1293	.1259	3	172.2
4	16.010216	N	16.0	6.1746E-4	.0291	.0283	7	202.8
5	18.035573	N	18.0	1.0355E-4	.0049	.0048	6	233.3
6	20.022152	N	20.0	1.1442E-4	.0054	.0052	7	263.6
7	28.006148	@	28.0	4.4701E-2	2.1062	2.0505	3	354.5
8	29.007593	N	29.0	2.9896E-4	.0141	.0137	7	370.3
9	32.007117	N	32.0	8.2791E-3	.3901	.3798	3	416.0
10	39.962384	@	40.0	7.5580E-4	.0356	.0347	7	524.0

Reviewed By: P.K. Barney Date: 12/14/01

Page 3 of 6

MATRIX - EVALUATION LIST of 212H-121201-2156 13 Dec 2001 14:12

SAMPLE ID:	212H-121201-2156	ALO NO.:	02-00900
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	13 Dec 2001 (13:54)	PAGE:	136
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HELIUM	99.98900	99.92078	.152064
N2	.00869	.06078	.000013
O2	.00231	.01844	.000004
TOTAL	100.00000	100.00000	.152081
TOTAL C1-C4		0.00000	

CLOSURE [%] : 1.182 MEASURED PRESSURE: .1503

Reviewed By: P.K. Barry Date: 12/14/01

Page 4 of 6

◄:S P E C T R U M L I S T :SAMPLE SPECTRUM 13 Dec 2001 14:10

SAMPLE ID: 212H-121201-2156
ANALYST: STAN BOS SB Bos
CUSTOMER: KURT SILVERS
CALCULATE DATE: 13 Dec 2001 (13:54)

ALO NO.: 02-00900
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 136
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 150.3 mTorr
ANALYSIS TIME : 16:19.758
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 109.72 mTorr
INTEGR.INTENSITY: 2.4190E+0 V
BASE PEAK INT. : 2.4174E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	8.3787E-5	.0035	.0035	7	30.3
2	4.002604	@	4.0	2.4174E+0	100.0000	99.9351	3	62.8
3	16.021664	N	16.0	9.3081E-5	.0039	.0038	6	203.8
4	18.034153	N	18.0	9.0557E-5	.0037	.0037	7	234.3
5	28.006148	@	28.0	1.0668E-3	.0441	.0441	3	354.7
6	32.008157	N	32.0	2.3505E-4	.0097	.0097	7	415.3

Reviewed By: P.K Barry Date: 12/14/01

Page 5 of 6

MATRIX - EVALUATION LIST of 212H-121201-2243 13 Dec 2001 15:12

SAMPLE ID:	212H-121201-2243	ALO NO.:	02-00901
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	13 Dec 2001 (14:34)	PAGE:	136
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00038	.00018	.000001
HELIUM	99.36907	95.61370	.155831
N2	.49833	3.35650	.000781
O2	.12603	.97015	.000198
ARGON	.00618	.05948	.000010
TOTAL	100.00000	100.00000	.156820
TOTAL C1-C4		0.00000	

CLOSURE [%] : 1.223 MEASURED PRESSURE: .1549

Reviewed By: R.K. Bems Date: 12/14/01

Page 6 of 6

S P E C T R U M L I S T :SAMPLE SPECTRUM 13 Dec 2001 14:51

SAMPLE ID: 212H-121201-2243
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 13 Dec 2001 (14:34)

ALO NO.: 02-00901
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 136
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 154.93 mTorr
ANALYSIS TIME : 16:21.79
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 112.92 mTorr
INTEGR.INTENSITY: 2.5605E+0 V
BASE PEAK INT. : 2.4775E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.2006E-4	.0048	.0047	7	30.3
2	4.002604	@	4.0	2.4775E+0	100.0000	96.7576	3	62.5
3	14.003074	@	14.0	3.8649E-3	.1560	.1509	3	172.9
4	16.008502	N	16.0	9.3759E-4	.0378	.0366	7	203.4
5	18.026135	N	18.0	8.4323E-5	.0034	.0033	7	234.0
6	20.020506	N	20.0	1.5194E-4	.0061	.0059	7	264.2
7	28.006148	@	28.0	6.3086E-2	2.5464	2.4638	3	355.4
8	29.011082	N	29.0	4.4768E-4	.0181	.0175	7	371.1
9	32.007245	N	32.0	1.3241E-2	.5345	.5171	3	416.2
10	39.962384	@	40.0	1.0889E-3	.0440	.0425	3	521.5

Reviewed By: P.K Barry Date: 12/14/01

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

43469-L02

January 7, 2002

Frank W. Moore
Fluor Hanford, Inc.
PO Box 1000
Richland, WA 99352

Dear Mr. Moore:

TRANSMITTAL OF REPORT 43469-RPT02, "GAS SPECIES ANALYSIS OF SAMPLES FROM THE CANISTER STORAGE BUILDING RECEIVED BY PNNL ON DECEMBER 20, 2001"

Reference: Statement of Work "Analysis of Gas Samples from the Spent Nuclear Fuel (SNF) Canister Storage (CSB) Facility" transmitted to PNNL on October 26, 2001, accompanying contract 11979-36 (PNNL Project 43469).

Attached please find a copy of report 43469-RPT02, "Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001." The samples described in this report were received by PNNL on December 20, 2001, and preliminary results were transmitted on December 20, 2001. The attached report is the final deliverable associated with these samples.

All analyses performed and results reported in the attached document were conducted in compliance with OCRWM standards. This data has been reviewed and determined to be OCRWM-compliant.

Radiochemical Processing Laboratory (RPL) sample identification numbers used in this report were assigned as follows:

RPL Sample ID	Client Sample ID	Client Sample Description
02-00973	212H-121901-2332	MCO Gas Sample
02-00974	212H-122001-1336	He Gas Sample

If you have any question, please give me a call on 372-4828.

Sincerely,

Kurt L. Silvers,
PNNL Project Manager

Attachment

902 Battelle Boulevard • P.O. Box 999 • Richland, WA 99352

Telephone (509) 372-4828 ■ Email kurt.silvers@pnl.gov ■ Fax (509) 372-6515

Frank W. Moore
January 7, 2002
Page 2

CC: E Biebesheimer, FH (summary only)
MS Busch, FH (summary only)
DR Duncan, FH (full data package)
SL Moore, FH (summary only)
DW Smith, FH (full data package)

GD Bazinet, NHC (summary only)
JP Sloughter, NHC (summary only)

SJ Bos, PNNL (full data package)
AM Lewis, PNNL (summary only)
BM Thornton, PNNL (summary only)
43469 project file

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

January 7, 2002

Pacific Northwest National Laboratory

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Sample Analysis Letter

January 7, 2002

Pacific Northwest National Laboratory

43469 BMS 1/3/02

Battelle
. . . Putting Technology To Work

Project No. ~~42047~~

Internal Distribution
File/LB

Date January 2, 2002

To Kurt Silvers

From Stan Bos Stan Bos

Subject Canister Storage Building gas sample analysis

Gas species analyses of samples taken at the Canister Storage Building (CSB) on December 19 and 20, 2001, have been completed. A report detailing the gas species detected is attached. Analysis was performed using the Finnigan MAT-271 (WC38625) high sensitivity quantitative mass spectrometer. The sensitivity of the instrument is checked daily prior to use with high purity N2 gas, and two air standards are run weekly to insure the instrument is operating correctly. The samples were assigned RPL sequence numbers 02-00973 through 02-00974.

Analysis of the evacuated sampler prior to taking the sample was not performed. CSB evacuates the sample cylinders before inletting the sample gas. Since this is a procedural step, a sampler cleaning analysis becomes unnecessary.

This analysis was performed and the report prepared following PNNL quality assurance plan Nuclear Quality Assurance Requirements and Description (NQARD). NQARD has been evaluated and found to be in conformance with the Office of Civilian Radioactive Waste Management (OCRWM) QA Program. The data in this report is OCRWM QARD Qualified Data.

PNNL project 43469 has setup for document and financial control of the sample analyses. Sample reports and data sheets are stored in the project RIDS located in ETB room 2619. Sample analyses are charged to work package F29087.

If you have any questions please contact Stan Bos at 376-5384.

Concurrence P.K. Berg

E34-1900-001 (8/98)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Gas Analysis Summary Results

January 7, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: December 20, 2001
Subject: Gas Species Analysis

To: Kurt silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 137
Measurement and test equipment WC38625

Sample Id.	212H-121901-2332		
Analysis Date:	December 20, 2001	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-00973		
Argon		0.028	± 0.001	280
Carbon dioxide		0.003	± 0.001	30
Carbon monoxide		<0.01	± 0	<100
Helium		97.1	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		2.27	± 0.05	22700
Oxygen		0.55	± 0.01	5500
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water <100 ppm			

Sample Id.	212H-122001-1336		
Analysis Date:	December 20, 2001	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-00974		
Argon		<0.001	± 0	<10
Carbon dioxide		<0.001	± 0	<10
Carbon monoxide		<0.01	± 0	<100
Helium		99.97	± 0.01	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.023	± 0.001	230
Oxygen		0.005	± 0.001	50
Nitrous oxide		<0.001	± 0	<10
Other nitrogen oxides		<0.001	± 0	<10
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water <100 ppm			

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Chain-of-Custody Forms

January 7, 2002

Pacific Northwest National Laboratory

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-121901-1 Date 12-19-01 Field Logbook No. NA

Point of Contact Doug Black Phone No. 372-2543 MSIN S8-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300A

Sampled By Steven B Carter (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
2124-121901-2332	212H/MCO M003	12-19-01	2332 hrs	NA	MCO GAS Sample

*Matrix
S Soil
SE Sediment
SO Solid
SL Sludge/Slurry
W Water
O Oil
A Air
AF Air Filter
DS Drum Solids
DL Drum Liquids
T Tissue
WI Wipe
L Liquid
V Vegetation
X Other

Special Instructions:
Sample Analysis to be performed per CSB Agreement, Ref. 'Contract 00011979 -00036 "CSB Statement of Work"

THIS IS AN MCO GAS Sample - FAX RESULTS TO 376-3252 (373-1456) OR EMAIL RESULTS TO Doug Black, RK Pool and Matt Busch.

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No.

CHAIN OF POSSESSION

Relinquished By	Date	Time	Received By	Date	Time
Steven B Carter	12-19-01	2400	Catherine M Clements	12/20/01	0815
Catherine M Clements	12/20/01	0900	S J Bos	12-20-01	0900

FINAL SAMPLE DISPOSITION

Disposal Method Sample cylinder pumped out

Disposed By: S J Bos (Print) S J Bos (Sign) 1/2/02 (Date) 4:10pm (Time)

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-122001-01 Date 12-20-01 Field Logbook No. NA

Point of Contact Doug BLACK Phone No. 372-2543 MSIN 58-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300A

Sampled By S BLARTER (Print) SBlarter (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
212H-122001-1336	212H/MCO TR-003	12-20-01	1336	NA	HELIUM GAS SAMPLE

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions:
SAMPLE ANALYSIS TO BE PERFORM PER CSB AGREEMENT.
REF: CONTRACT 0001197A-00036 "CSB STATEMENT OF WORK"
SAL 12-20-01
THIS IS AN ~~MCO~~ HELIUM PURITY GAS SAMPLES – FAX RESULTS TO 376-3252 (373-1456) & EMAIL RESULTS TO DOUG BLACK, RK PEOL AND MATT BISCHOFF

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. ____

CHAIN OF POSSESSION

Relinquished By			Received By		
STEVEN B CARTER	12-20-01 SAL		JANIE L. HENSLEY	12-20-01	
Sign: SBCarter	Time: 1438		Sign: Janie L Hensley	Time: 1445	
JANIE L. HENSLEY	12-20-01		P.K. BERRY	12-20-01	
Sign: Janie L Hensley	Time: 1520		Sign: PK Berry	Time: 15:23	
Print	Date		Print	Date	
Sign	Time		Sign	Time	
Print	Date		Print	Date	
Sign	Time		Sign	Time	

FINAL SAMPLE DISPOSITION

Disposal Method Sample cylinder purged out

Disposed By:

Print	Sign	Date	Time
S. S. Bos	S S Bos	1/2/02	4:10 pm

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Analytical Services Request

January 7, 2002

Pacific Northwest National Laboratory

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature [signature] 12/20/01
Print Name Kurt Silvers
Phone 372-4828 MSIN K9-08

PNNL Project #: 43409
Charge Code: F29087
Date Required: 1-15-01

Matrix Type Information

- ♦ Liquids: ___ Aqueous ___ Organic ___ Multi-phase
- ♦ Solids: ___ Soil ___ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- ♦ Other: ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ QA Plan:
  SBMS _____
  HASQARD (CAWSRP) _____
- ♦ Additional QA Requirements? No _____
  or Reference Doc # _____
- ♦ Field COC? No ___ Yes X
- ♦ Lab COC Required? No X Yes ___
- ♦ Hold Time: None X
  or RCRA _____ CERCLA _____
  Other, Specify _____
  & Date Sampled _____
  Time Sampled _____
- ♦ Special Storage Requirements:
  None X Refrigerate (4°C) _____
  or Other, specify _____
- ♦ Data Quality Review Required? No X Yes ___

Disposal Information

- ♦ Disposition of Virgin Samples:
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # _____
- ♦ Disposition of Treated Samples:
  Dispose _____ Return _____

Waste Designation Information

- ♦ Sample Information Check List Attached? Yes _____
  or Reference Doc # _____
  or Previous ASR # _____
  or Previous RPL ID # _____

Does the Waste Designation Documentation Indicate Presence of PCBs?
No X Yes _____

Additional or Special Instructions _____

Send Report To Kurt Silvers Phone 372-4828
_____ Phone _____

Preliminary results requested, as available? No ___ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12-20-01
Delivered By (optional) _____
Time Delivered (optional) _____
Group ID (optional) _____
CMC Waste Sample? No _____ Yes _____
Cost Estimate, if requested: $ _____

Received By: S. L. Bos

ASR Number: _____
RPL Numbers: _____

RPG/CMC Work Accepted By: S. L Bos Signature/Date: [signature] 12-20-01

ASR FY2000 - RPG.doc

page 1 of 2

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1) Gas Species	Lab Staff Use Only Test	Lab Staff Use Only Library
02-00973	212H-121901-2332	mco gas sample		Gas Anal	Adv. Dwg
02-00974	212H-122001-1330	Helium Gas Sample SB 12/20/01			

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___ ; On Attachment ___]

ASR # 6313

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Quality Control Check

January 7, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-3358 / mail slot P7-22
Date: December 17, 2001
Subject: Air standards from Finnigan MAT - 271 Mass Spectrometer

Analytical procedure: PNNL - 98523 - 284 Rev. 0
Laboratory Record Book 56998: Page 136
Measurement and test equipment WC38625

Accepted values for the composition of air :

	Mole percent
Argon	0.934
Nitrogen	78.08
Oxygen	20.95

Analyzed Values:

Analysis Date: December 17,2001

	Mole percent
Argon	0.938
Nitrogen	78.10
Oxygen	20.92

Analyzed Values:

Analysis Date: December 17,2001

	Mole percent
Argon	0.937
Nitrogen	77.92
Oxygen	21.10

Instrument Background:

Background analyses are run daily prior to sample analyses. Trace amounts of hydrogen and/or water in the 0.1 to 0.2 millivolt range were the only species detected. The background spectra is subtracted from each sample spectra.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on December 20, 2001

43469-RPT02, Rev. 0

Raw Instrument Data from Gas Analysis

January 7, 2002

Pacific Northwest National Laboratory

Page 1 of 4

MATRIX - EVALUATION LIST of 212H-121901-2332 20 Dec 2001 10:18

SAMPLE ID:	212H-121901-2332	ALO NO.:	02-00973
ANALYST:	STAN BOS SJ Bos	LAB SAMPLE NO.:	0
CUSTOMER:	CSB	LRB NO.:	56998
CALCULATE DATE:	20 Dec 2001 (09:39)	PAGE:	137
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00044	.00019	.000001
HELIUM	97.14806	82.49898	.132800
N2	2.26745	13.47878	.003100
O2	.55357	3.76080	.000757
ARGON	.02750	.23350	.000038
CO2	.00297	.02775	.000004
TOTAL	100.00000	100.00000	.136698
TOTAL C1-C4		0.00000	

CLOSURE [%] : 1.225 MEASURED PRESSURE: .1350

Reviewed By: P.K. Barry Date: 1-2-02

Page 2 of 4

S P E C T R U M L I S T :SAMPLE SPECTRUM 20 Dec 2001 10:16

SAMPLE ID: 212H-121901-2332
ANALYST: STAN BOS
CUSTOMER: CSB
CALCULATE DATE: 20 Dec 2001 (09:39)

ALO NO.: 02-00973
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 137
M&TE NO.: WC38625

EXPERIMENT : 48 (UNUSUAL SAMPLES+Kr&Xe)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1
MASS RANGE: 2.02 - 152.06

START PRESSURE: 135.04 mTorr
ANALYSIS TIME : 36:47.475
BASE PEAK NO. : 2 (m/e= 4.002604)
END PRESSURE : 67.65 mTorr
INTEGR.INTENSITY: 2.3735E+0 V
BASE PEAK INT. : 2.0569E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.2814E-4	.0062	.0054	7	28.6
2	4.002604	@	4.0	2.0569E+0	100.0000	86.6643	3	58.1
3	14.003074	@	14.0	1.4976E-2	.7281	.6310	3	213.4
4	16.009739	N	16.0	3.4059E-3	.1656	.1435	3	244.0
5	18.028387	N	18.0	9.6444E-5	.0047	.0041	6	274.1
6	20.015790	N	20.0	6.2117E-4	.0302	.0262	7	304.1
7	28.006148	@	28.0	2.4182E-1	11.7563	10.1885	3	410.0
8	29.006535	N	29.0	1.7774E-3	.0864	.0749	3	425.3
9	32.003505	N	32.0	4.9015E-2	2.3829	2.0651	3	470.5
10	33.931466	N	34.0	2.2232E-4	.0108	.0094	6	500.6
11	39.962384	@	40.0	4.0719E-3	.1980	.1716	3	590.6
12	44.013643	N	44.0	3.8290E-4	.0186	.0161	6	651.0

Reviewed By: P.K. Barry Date: 01-02-02

Page 3 of 4

MATRIX - EVALUATION LIST of 212H-122001-1336 20 Dec 2001 15:52

SAMPLE ID:	212H-122001-1336	ALO NO.:	02-00974
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	CSB	LRB NO.:	56998
CALCULATE DATE:	20 Dec 2001 (15:40)	PAGE:	137
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00013	.00006	0.000000
HELIUM	99.96993	99.78054	.175378
N2	.02322	.16220	.000041
O2	.00540	.04313	.000009
ARGON	.00051	.00508	.000001
CO2	.00082	.00898	.000001
TOTAL	100.00000	100.00000	.175431
TOTAL C1-C4		0.00000	

CLOSURE [%] : .466 MEASURED PRESSURE: .1746

Reviewed By: [signature] Date: 1-2-02

Page 4 of 4

◄‡S P E C T R U M L I S T :SAMPLE SPECTRUM 20 Dec 2001 15:51

SAMPLE ID: 212H-122001-1336
ANALYST: STAN BOS
CUSTOMER: CSB
CALCULATE DATE: 20 Dec 2001 (15:40)

ALO NO.: 02-00974
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 137
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 174.62 mTorr
ANALYSIS TIME : 10:26.168
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 142.45 mTorr
INTEGR.INTENSITY: 2.7166E+0 V
BASE PEAK INT. : 2.7121E+0 V

Attributes: ABORTED ACQUISITION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.1263E-4	.0042	.0041	7	30.3
2	4.002604	@	4.0	2.7121E+0	100.0000	99.8326	3	63.3
3	14.003074	@	14.0	2.2988E-4	.0085	.0085	6	174.6
4	16.021179	N	16.0	1.5938E-4	.0059	.0059	6	205.0
5	28.006148	@	28.0	3.1966E-3	.1179	.1177	3	355.6
6	32.002588	N	32.0	6.1775E-4	.0228	.0227	7	415.9
7	39.962384	@	40.0	9.7407E-5	.0036	.0036	6	521.1
8	44.011099	N	44.0	1.3383E-4	.0049	.0049	7	581.2

Reviewed By: R.K. Bem Date: 1-2-02

Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

43469-L03

January 11, 2002

Frank W. Moore
Fluor Hanford, Inc.
PO Box 1000
Richland, WA 99352

Dear Mr. Moore:

TRANSMITTAL OF REPORT 43469-RPT03, "GAS SPECIES ANALYSIS OF SAMPLES FROM THE CANISTER STORAGE BUILDING RECEIVED BY PNNL ON JANUARY 4, 2002"

Reference: Statement of Work "Analysis of Gas Samples from the Spent Nuclear Fuel (SNF) Canister Storage (CSB) Facility" transmitted to PNNL on October 26, 2001, accompanying contract 11979-36 (PNNL Project 43469).

Attached please find a copy of report 43469-RPT03, "Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002." The samples described in this report were received by PNNL on January 4, 2002, and preliminary results were transmitted on January 4, 2002. The attached report is the final deliverable associated with these samples.

All analyses performed and results reported in the attached document were conducted in compliance with OCRWM standards. This data has been reviewed and determined to be OCRWM-compliant.

Radiochemical Processing Laboratory (RPL) sample identification numbers used in this report were assigned as follows:

RPL Sample ID	Client Sample ID	Client Sample Description
02-01039	212H-010402-1048	He Gas Sample
02-01040	212H-010402-1143	MCO Gas Sample

If you have any question, please give me a call on 372-4828.

Sincerely,

Kurt L. Silvers,
PNNL Project Manager

Attachment

902 Battelle Boulevard • P.O. Box 999 • Richland, WA 99352

Telephone (509) 372-4828 ■ Email kurt.silvers@pnl.gov ■ Fax (509) 372-6515

CC: E Biebesheimer, FH (summary only)
MS Busch, FH (summary only)
DR Duncan, FH (full data package)
DW Smith, FH (full data package)

GD Bazinet, NHC (summary only)
JP Sloughter, NHC (summary only)

SJ Bos, PNNL (full data package)
AM Lewis, PNNL (summary only)
BM Thornton, PNNL (summary only)
43469 project file

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

January 11, 2002

Pacific Northwest National Laboratory

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Sample Analysis Letter

January 11, 2002

Pacific Northwest National Laboratory

Battelle
. . . Putting Technology To Work

Project No. 43469

Internal Distribution
File/LB

Date January 7, 2002

To Kurt Silvers

From Stan Bos Stan Bos

Subject Canister Storage Building gas sample analysis

Gas species analyses of samples taken at the Canister Storage Building (CSB) on January 4, 2002 have been completed. A report detailing the gas species detected is attached. Analysis was performed using the Finnigan MAT-271 (WC38625) high sensitivity quantitative mass spectrometer. The sensitivity of the instrument is checked daily prior to use with high purity N2 gas, and two air standards are run weekly to insure the instrument is operating correctly. The samples were assigned RPL sequence numbers 02-01039 and 02-01040.

Analysis of the evacuated sampler prior to taking the sample was not performed. CSB evacuates the sample cylinders before inletting the sample gas. Since this is a procedural step, a sampler cleaning analysis becomes unnecessary.

This analysis was performed and the report prepared following PNNL quality assurance plan Nuclear Quality Assurance Requirements and Description (NQARD). NQARD has been evaluated and found to be in conformance with the Office of Civilian Radioactive Waste Management (OCRWM) QA Program. The data in this report is OCRWM QARD Qualified Data.

PNNL project 43469 was setup for document and financial control of the sample analyses. Sample reports and data sheets are stored in the project RIDS located in ETB room 2619. Sample analyses are charged to work package F29087.

If you have any questions please contact Stan Bos at 376-5384.

Concurrence P.K. Barney 1-9-02

ES4-1900-001 (8/98)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Gas Analysis Summary Results

January 11, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: January 04, 2002
Subject: Gas Species Analysis

To: Kurt Silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 138
Measurement and test equipment WC38625

Sample Id.	212H-010402-1048		
Analysis Date:	January 04, 2002	Mole Percent	Estimate of Precision	PPM
Log-In No.	02-01039		
Argon		0.007	± 0.001	70
Carbon dioxide		0.001	± 0.0005	10
Carbon monoxide		<0.01	± 0	<100
Helium		99.28	± 0.01	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.58	± 0.01	5800
Oxygen		0.132	± 0.003	1320
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10

Comments: Water <100 ppm, Sample cylinder valve was loose.

Sample Id.	212H-010402-1143		
Analysis Date:	January 04, 2002	Mole Percent	Estimate of Precision	PPM
Log-In No.	02-01040		
Argon		0.024	± 0.001	240
Carbon dioxide		0.002	± 0.001	20
Carbon monoxide		<0.01	± 0	<100
Helium		97.5	± 0.1	
Hydrogen		0.001	± 0.0005	10
Neon		<0.001	± 0	<10
Nitrogen		2	± 0.04	20000
Oxygen		0.51	± 0.01	5100
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10

Comments: Water <1000 ppm, Sample cylinder valve was loose.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Chain-of-Custody Form

January 11, 2002

Pacific Northwest National Laboratory

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-010402-01 Date 01-04-02 Field Logbook No. NA

Point of Contact DOUG BLOCK / FRANK MOORE Phone No. 372-2543 MSIN 58-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300A

Sampled By SB CARTER
Print Sign

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
212H-010402-1048	212H/MCO-TR003	1-4-02	10:48 AM	NA	HELIUM GAS Sample
212H-010402-1143	212H/MCO-TR003	1-4-02	11:43 AM	NA	MCO GAS Sample

*Matrix
S Soil
SE Sediment
SO Solid
SL Sludge/Slurry
W Water
O Oil
A Air
AF Air Filter
DS Drum Solids
DL Drum Liquids
T Tissue
WI Wipe
L Liquid
V Vegetation
X Other

Special Instructions: SAMPLE ANALYSIS TO BE PERFORMED PER CSB AGREEMENT, REF CONTRACT 00011979-00036 "CSB STATEMENT OF WORK"
HELIUM PURITY & MCO GAS SAMPLES PER IS-01-00586/OP-23-0025
EMAIL RESULTS TO DOUG BLOCK, FRANK MOORE, RANDY PAUL PAUL GIAMBALVO, MATT BLOCH & STEVE CARTER

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By		Received By	
STEVEN B CARTER (Print)	1-4-02 (Date)	Catherine M Clements (Print)	1/4/01 (Date)
(Sign)	1225 (Time)	(Sign)	1233 (Time)
Catherine M Clements (Print)	1/4/01 (Date)	P.K. BERRY (Print)	1/4/01 (Date)
(Sign)	1300 (Time)	(Sign)	13:00 (Time)
Print	Date	Print	Date
Sign	Time	Sign	Time
Print	Date	Print	Date
Sign	Time	Sign	Time

FINAL SAMPLE DISPOSITION

Disposal Method Sample cylinders pumped out 1-7-02

Disposed By:

Print	Sign	Date	Time
S J BOS		1-7-02	7:50

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Analytical Services Request

January 11, 2002

Pacific Northwest National Laboratory

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor: Signature [signature] Print Name Kurt Silvers
Phone 372-4828 MSIN K9-08

PNNL Project #: 43469
Charge Code: F29087
Date Required: 1-15-02

Matrix Type Information

- ♦ Liquids: ___ Aqueous ___ Organic ___ Multi-phase
- ♦ Solids: ___ Soil ___ Sludge ___ Sediment ___ Glass ___ Filter ___ Metal ___ Smear ___ Organic ___ Other
- ♦ Other: ___ Solid/Liquid Mixture, Slurry X Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ QA Plan: SBMS _____ HASQARD (CAWSRP) NQARD
- ♦ Additional QA Requirements? No _____ or Reference Doc # _____
- ♦ Field COC? No ___ Yes X
- ♦ Lab COC Required? No X Yes _____
- ♦ Hold Time: None X
  or RCRA _____ CERCLA _____ Other, Specify _____
  & Date Sampled _____ Time Sampled _____
- ♦ Special Storage Requirements: None X Refrigerate (4°C) _____ or Other, specify _____
- ♦ Data Quality Review Required? No X Yes ___

Disposal Information

- ♦ Disposition of Virgin Samples: Virgin samples are returned to requestor unless archiving provisions are made with receiving group! If archiving, provide: Archiving Reference Doc # _____
- ♦ Disposition of Treated Samples: Dispose _____ Return _____

Waste Designation Information

- ♦ Sample Information Check List Attached? Yes _____
  or Reference Doc # _____
  or Previous ASR # 5949
  or Previous RPL ID # _____

Does the Waste Designation Documentation Indicate Presence of PCBs? No X Yes _____

Additional or Special Instructions _____

Send Report To Kurt Silvers Phone 372-4828

_____ Phone _____

Preliminary results requested, as available? No ___ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 01-04-02
Delivered By (optional) _____
Time Delivered (optional) 1300
Group ID (optional) _____
CMC Waste Sample? No X Yes _____
Cost Estimate, if requested: $ NA

Received By: P.K. Berry

ASR Number: 6321
RPL Numbers: 102-01039 - 102-01040 (39)
P.K. 1-4-02

RPG/CMC Work Accepted By: P.K. Berry Signature/Date: P.K. Berry 1/4/02

ASR FY2000 - RPG.doc

Page 1 of 2

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
02-01039	212H-010402-1048	212H / MCO-TR003 (1048) Hegas sample	Gas Species		
02-01040	212H-010402-1143	" " (1143) MCO gas sample			

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___; On Attachment ___]

ASR # 6521

Page 2 of 2

ASR FY2000 - RPG.doc

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Quality Control Check

January 11, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-3358 / mail slot P7-22
Date: January 02, 2002
Subject: Air standards from Finnigan MAT - 271 Mass Spectrometer

Analytical procedure: PNNL - 98523 - 284 Rev. 0
Laboratory Record Book 56998: Page 137
Measurement and test equipment WC38625

Accepted values for the composition of air :

	Mole percent
Argon	0.934
Nitrogen	78.08
Oxygen	20.95

Analyzed Values:

Analysis Date: January 02,2002

	Mole percent
Argon	0.94
Nitrogen	78.16
Oxygen	20.86

Analyzed Values:

Analysis Date: January 02,2002

	Mole percent
Argon	0.937
Nitrogen	77.94
Oxygen	21.08

Instrument Background:

Background analyses are run daily prior to sample analyses. Trace amounts of hydrogen and/or water in the 0.1 to 0.2 millivolt range were the only species detected. The background spectra is subtracted from each sample spectra.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 4, 2002

43469-RPT03, Rev. 0

Raw Instrument Data from Gas Analysis

January 11, 2002

Pacific Northwest National Laboratory

Page 1 of 4

S P E C T R U M L I S T :SAMPLE SPECTRUM 4 Jan 2002 13:38

SAMPLE ID: 212H-010402-1048
ANALYST: PK BERRY P.K. Berry
CUSTOMER: KURT SILVERS
CALCULATE DATE: 4 Jan 2002 (13:26)

ALO NO.: 02-01039
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 138
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1
MASS RANGE: 2.02 - 135.91

START PRESSURE: 141.52 mTorr
ANALYSIS TIME : 10:51.704
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 114.32 mTorr
INTEGR.INTENSITY: 2.3369E+0 V
BASE PEAK INT. : 2.2504E+0 V

Attributes: ABORTED ACQUISITION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	9.8508E-5	.0044	.0042	7	30.3
2	4.002604	@	4.0	2.2504E+0	100.0000	96.2968	3	62.8
3	14.003074	@	14.0	4.0988E-3	.1821	.1754	3	173.8
4	16.010461	N	16.0	9.2722E-4	.0412	.0397	7	204.1
5	20.006679	N	20.0	1.7381E-4	.0077	.0074	6	264.4
6	28.006148	@	28.0	6.6910E-2	2.9733	2.8632	3	353.8
7	29.007349	N	29.0	5.0269E-4	.0223	.0215	7	368.9
8	32.007119	N	32.0	1.2566E-2	.5584	.5377	3	413.7
9	39.962384	@	40.0	1.1219E-3	.0499	.0480	3	517.9
10	44.009067	N	44.0	1.4234E-4	.0063	.0061	6	577.5

Reviewed By: S.G. Date: 1/4/02

Page 2 of 4

MATRIX - EVALUATION LIST of 212H-010402-1048 4 Jan 2002 13:39

SAMPLE ID:	212H-010402-1048	ALO NO.:	02-01039
ANALYST:	PK BERRY P.K. Berry	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	4 Jan 2002 (13:26)	PAGE:	138
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HELIUM	99.27926	95.02128	.140473
N2	.58119	3.89383	.000822
O2	.13150	1.00689	.000186
ARGON	.00702	.06717	.000010
CO2	.00103	.01083	.000001
TOTAL	100.00000	100.00000	.141493
TOTAL C1-C4		0.00000	

CLOSURE [%] : -.017 MEASURED PRESSURE: .1415

cylinder valve was not Tighten enough

Reviewed By: S J Bess Date: 1/4/02

MATRIX - EVALUATION LIST of 212H-010402-1143 4 Jan 2002 14:02

SAMPLE ID:	212H-010402-1143	ALO NO.:	02-01040
ANALYST:	PK BERRY P.K. Berry	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	4 Jan 2002 (13:50)	PAGE:	138
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00103	.00044	.000001
HELIUM	97.46818	84.18930	.122161
N2	1.99533	12.06446	.002501
O2	.50982	3.52289	.000639
ARGON	.02394	.20674	.000030
CO2	.00170	.01616	.000002
TOTAL	100.00000	100.00000	.125335

TOTAL C1-C4 0.00000

CLOSURE [%] : -2.834 MEASURED PRESSURE: .1290

Sample cylinder valve was loose

Reviewed By: S G Box Date: 1/11/02

◄:S P E C T R U M L I S T :SAMPLE SPECTRUM 4 Jan 2002 14:01

SAMPLE ID: 212H-010402-1143
ANALYST: PK BERRY P.K. Berry
CUSTOMER: KURT SILVERS
CALCULATE DATE: 4 Jan 2002 (13:50)

ALO NO.: 02-01040
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 138
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 128.99 mTorr
ANALYSIS TIME : 10:51.707
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 104.44 mTorr
INTEGR.INTENSITY: 2.2253E+0 V
BASE PEAK INT. : 1.9571E+0 V

Attributes: ABORTED ACQUISITION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.5954E-4	.0082	.0072	7	30.3
2	4.002604	@	4.0	1.9571E+0	100.0000	87.9504	3	62.7
3	14.003074	@	14.0	1.2458E-2	.6366	.5599	3	173.3
4	16.009809	N	16.0	2.9571E-3	.1511	.1329	3	203.4
5	18.037371	N	18.0	1.0715E-4	.0055	.0048	6	233.8
6	20.017836	N	20.0	5.3147E-4	.0272	.0239	7	263.9
7	28.006148	@	28.0	2.0349E-1	10.3972	9.1444	3	353.6
8	29.007072	N	29.0	1.4927E-3	.0763	.0671	3	368.8
9	32.002730	N	32.0	4.3153E-2	2.2049	1.9392	3	413.7
10	33.935796	N	34.0	1.9005E-4	.0097	.0085	6	443.7
11	39.962384	@	40.0	3.3884E-3	.1731	.1523	3	518.4
12	44.014263	N	44.0	2.1204E-4	.0108	.0095	7	578.0

Reviewed By: S J Bess Date: 1/4/02

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

43469-L04

January 11, 2002

Frank W. Moore
Fluor Hanford, Inc.
PO Box 1000
Richland, WA 99352

Dear Mr. Moore:

TRANSMITTAL OF REPORT 43469-RPT04, "GAS SPECIES ANALYSIS OF SAMPLES FROM THE CANISTER STORAGE BUILDING RECEIVED BY PNNL ON JANUARY 8, 2002"

Reference: Statement of Work "Analysis of Gas Samples from the Spent Nuclear Fuel (SNF) Canister Storage (CSB) Facility" transmitted to PNNL on October 26, 2001, accompanying contract 11979-36 (PNNL Project 43469).

Attached please find a copy of report 43469-RPT04, "Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002." The samples described in this report were received by PNNL on January 8, 2002. The preliminary results were transmitted on January 8, 2002; however, due to a typographical error related to the precision of the helium measurement, the preliminary results were reissued on January 9, 2002. The attached report is the final deliverable associated with these samples.

All analyses performed and results reported in the attached document were conducted in compliance with OCRWM standards. This data has been reviewed and determined to be OCRWM-compliant.

Radiochemical Processing Laboratory (RPL) sample identification numbers used in this report were assigned as follows:

RPL Sample ID	Client Sample ID	Client Sample Description
02-01045	212H-010702-2209	He Gas Sample
02-01046	212H-010702-2235	MCO Gas Sample

If you have any question, please give me a call on 372-4828.

Sincerely,

Kurt L. Silvers,
PNNL Project Manager

Attachment

902 Battelle Boulevard • P.O. Box 999 • Richland, WA 99352

Telephone (509) 372-4828 ■ Email kurt.silvers@pnl.gov ■ Fax (509) 372-6515

Frank W. Moore
January 11, 2002
Page 2

CC: E Biebesheimer, FH (summary only)
MS Busch, FH (summary only)
DR Duncan, FH (full data package)
DW Smith, FH (full data package)

GD Bazinet, NHC (summary only)
JP Sloughter, NHC (summary only)

SJ Bos, PNNL (full data package)
AM Lewis, PNNL (summary only)
BM Thornton, PNNL (summary only)
43469 project file

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

January 11, 2002

Pacific Northwest National Laboratory

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Sample Analysis Letter

January 11, 2002

Pacific Northwest National Laboratory

Battelle
. . . Putting Technology To Work

Project No. 43469

Internal Distribution
File/LB

Date January 9, 2002

To Kurt Silvers

From Stan Bos Stan Bos

Subject Canister Storage Building gas sample analysis

Gas species analyses of samples taken at the Canister Storage Building (CSB) on January 7, 2002 have been completed. A report detailing the gas species detected is attached. Analysis was performed using the Finnigan MAT-271 (WC38625) high sensitivity quantitative mass spectrometer. The sensitivity of the instrument is checked daily prior to use with high purity N2 gas, and two air standards are run weekly to insure the instrument is operating correctly. The samples were assigned RPL sequence numbers 02-~~01046~~ and 02-01046.

BMT 1/11/02 .01045 .

Analysis of the evacuated sampler prior to taking the sample was not performed. CSB evacuates the sample cylinders before inletting the sample gas. Since this is a procedural step, a sampler cleaning analysis becomes unnecessary.

This analysis was performed and the report prepared following PNNL quality assurance plan Nuclear Quality Assurance Requirements and Description (NQARD). NQARD has been evaluated and found to be in conformance with the Office of Civilian Radioactive Waste Management (OCRWM) QA Program. The data in this report is OCRWM QARD Qualified Data.

PNNL project 43469 was setup for document and financial control of the sample analyses. Sample reports and data sheets are stored in the project RIDS located in ETB room 2619. Sample analyses are charged to work package F29087.

If you have any questions please contact Stan Bos at 376-5384.

Concurrence P.K. Barney 1-9-02

ES4-1900-001 (8/98)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Gas Analysis Summary Results

January 11, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: January 08, 2002
Subject: Gas Species Analysis

To: Kurt Sivers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 138
Measurement and test equipment WC38625

Sample Id.	212H-010702-2209		
Analysis Date:	January 08, 2002	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-01045		
Argon		<0.001	± 0	<10
Carbon dioxide		0.002	± 0.001	20
Carbon monoxide		<0.01	± 0	<100
Helium		99.98	± 0.01	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.018	± 0.001	180
Oxygen		0.002	± 0.001	20
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water was not detected			

Sample Id.	212H-010702-2235		
Analysis Date:	January 08, 2002	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-01046		
Argon		0.024	± 0.001	240
Carbon dioxide		0.003	± 0.001	30
Carbon monoxide		<0.01	± 0	<100
Helium		97.5	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		1.99	± 0.04	19900
Oxygen		0.49	± 0.01	4900
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water was not detected			

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Chain-of-Custody Form

January 11, 2002

Pacific Northwest National Laboratory

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-010702-01 Date 01/07/02 Field Logbook No. N/A

Point of Contact DOUG BLACK/FRANK MOORE Phone No. 373-2543 MSIN S8-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300A

Sampled By D. L. McKINNIS (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
212H-1-07-02-2209	212H/MCO-TR003	1-07-02	2209	N/A	HELIUM GAS SAMPLE
212H-1-07-02-2235	212H/MCO-TR003	1-07-02	2235	N/A	MCO GAS SAMPLE

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions: SAMPLE ANALYSIS TO BE PERFORMED PER CSB AGREEMENT, REF CONTRACT 0C011979-00036 "CSB STATEMENT OF WORK"
HELIUM PURITY & MCO GAS SAMPLES PER 15-01-00586/OP-23-0025 EMAIL RESULTS TO DOUG BLACK, FRANK MOORE, RANDY PIPOOL, PAUL GARELLO, MATT BUSCH & STEVE CARTER
(CONTACT @ 325 LAB STAN BOS 376-5384)

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. ____

CHAIN OF POSSESSION

Relinquished By		Received By	
Print: D. L. McKinnis	Date: 1-8-02	Print: JANIE L. HENSLEY	Date: 1-8-02
Sign: D. L. McKinnis	Time: 0725	Sign: Janie L. Hensley	Time: 0725
Print: JANIE L. HENSLEY	Date: 1-8-02	Print: S S BOS	Date: 1-8-02
Sign: Janie L. Hensley	Time: 0805	Sign: S S Bos	Time: 08-06
Print:	Date:	Print:	Date:
Sign:	Time:	Sign:	Time:
Print:	Date:	Print:	Date:
Sign:	Time:	Sign:	Time:

FINAL SAMPLE DISPOSITION

Disposal Method: Sample cylinder pumped out.

Disposed By: S S Bos (Print) (Sign) Date: 1/9/02 Time: 9:12

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Analytical Services Request

January 11, 2002

Pacific Northwest National Laboratory

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor: Signature: Kurt L. Silvers
Print Name: Kurt Silvers
Phone: 372-4828 MSIN: K9-08
PNNL Project #:
Charge Code: F29087
Date Required: 1-15-02

Matrix Type Information

- ♦ Liquids: ___ Aqueous __ Organic ___ Multi-phase
- ♦ Solids: ___ Soil ___ Sludge ___ Sediment ___ Glass ___ Filter __ Metal ___ Smear ___ Organic ___ Other
- ♦ Other: ___ Solid/Liquid Mixture, Slurry ✓ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ QA Plan: SBMS ____ HASQARD (CAWSRP) NQRD
- ♦ Additional QA Requirements? No ____ or Reference Doc # 1/8/02
- ♦ Field COC? No WTSS Yes ✓
- ♦ Lab COC Required? No ✓ Yes ____
- ♦ Hold Time: None X ____ or RCRA ____ CERCLA ____ Other, Specify ____ & Date Sampled ____ Time Sampled ____
- ♦ Special Storage Requirements: None X ____ Refrigerate (4°C) ____ or Other, specify ____
- ♦ Data Quality Review Required? No X Yes ____

Disposal Information

- ♦ Disposition of Virgin Samples: Virgin samples are returned to requestor unless archiving provisions are made with receiving group! If archiving, provide: Archiving Reference Doc # ____
- ♦ Disposition of Treated Samples: Dispose ✓ Return ____

Waste Designation Information

- ♦ Sample Information Check List Attached? Yes ____ or Reference Doc # ____ or Previous ASR # 5949 or Previous RPL ID # ____

Does the Waste Designation Documentation Indicate Presence of PCBs? No X Yes ____

Additional or Special Instructions ____

Send Report To: Kurt Silvers Phone: 372-4828
Phone:

Preliminary results requested, as available? No ✓ Yes ____ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 1/8/02
Delivered By (optional) ____
Time Delivered (optional) ____
Group ID (optional) ____
CMC Waste Sample? No ____ Yes ____
Cost Estimate, if requested: $ ____

Received By: S. J. Bos

ASR Number: 6324
RPL Numbers: 02-01045 02-01046

RPG/CMC Work Accepted By: S S Bos Signature/Date: S S Bos 1-8-02

ASR FY2000 - RPG.doc

Page 1 of 2

Analytical Service Request (ASR)

(REQUEST PAGE ---- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
02-01045	212H-010702-2209		Gas Species	GasAnal	[illegible]
02-01046	212H-010702-22[illegible]5 SO 1-7-02				

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___ ; On Attachment ___]

ASR # 6324

Page 2 of 2

ASR FY2000 - RPG.doc

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Quality Control Check

January 11, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-3358 / mail slot P7-22
Date: January 07, 2002
Subject: Air standards from Finnigan MAT - 271 Mass Spectrometer

Analytical procedure: PNNL - 98523 - 284 Rev. 0
Laboratory Record Book 56998: Page 138
Measurement and test equipment WC38625

Accepted values for the composition of air :

	Mole percent
Argon	0.934
Nitrogen	78.08
Oxygen	20.95

Analyzed Values:

Analysis Date: January 07,2002

	Mole percent
Argon	0.938
Nitrogen	78.05
Oxygen	20.97

Analyzed Values:

Analysis Date: January 07,2002

	Mole percent
Argon	0.937
Nitrogen	77.92
Oxygen	21.11

Instrument Background:

Background analyses are run daily prior to sample analyses. Trace amounts of hydrogen and/or water in the 0.1 to 0.2 millivolt range were the only species detected. The background spectra is subtracted from each sample spectra.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 8, 2002

43469-RPT04, Rev. 0

Raw Instrument Data from Gas Analysis

January 11, 2002

Pacific Northwest National Laboratory

Page 1 of 4

◄‡MATRIX - EVALUATION LIST of 212H-010702-2209 8 Jan 2002 09:07

SAMPLE ID:	212H-010702-2209	ALO NO.:	02-01045
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	8 Jan 2002 (08:47)	PAGE:	138
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES. [Torr]
HYDROGEN	.00065	.00033	.000001
HELIUM	99.97707	99.83301	.174437
N2	.01758	.12288	.000031
O2	.00243	.01941	.000004
ARGON	.00048	.00479	.000001
CO2	.00178	.01958	.000003
TOTAL	100.00000	100.00000	.174477
TOTAL C1-C4		0.00000	

CLOSURE [%] : .120 MEASURED PRESSURE: .1743

Reviewed By: MC Date: 1-8-02

Page 2 of 4

◂‡S P E C T R U M L I S T :SAMPLE SPECTRUM 8 Jan 2002 09:04

SAMPLE ID: 212H-010702-2209
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 8 Jan 2002 (08:47)

ALO NO.: 02-01045
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 138
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1

MASS RANGE: 2.02 - 135.91

START PRESSURE: 174.27 mTorr
ANALYSIS TIME : 16:47.547
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 125.84 mTorr
INTEGR.INTENSITY: 2.6021E+0 V
BASE PEAK INT. : 2.5987E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.3565E-4	.0052	.0052	6	30.3
2	4.002604	@	4.0	2.5987E+0	100.0000	99.8687	3	62.9
3	14.003074	@	14.0	1.7538E-4	.0067	.0067	7	174.1
4	16.019973	N	16.0	1.5468E-4	.0060	.0059	7	204.9
5	28.006148	@	28.0	2.3172E-3	.0892	.0891	3	359.4
6	32.001972	N	32.0	2.6608E-4	.0102	.0102	6	420.3
7	39.962384	@	40.0	8.7822E-5	.0034	.0034	6	529.7
8	44.014588	N	44.0	2.7895E-4	.0107	.0107	7	593.3

Reviewed By: R.K. Barry Date: 1-9-02

Page 3 of 4

MATRIX - EVALUATION LIST of 212H-010702-2235 8 Jan 2002 10:30

SAMPLE ID:	212H-010702-2235	ALO NO.:	02-01046
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	8 Jan 2002 (09:22)	PAGE:	138
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00082	.00035	.000001
HELIUM	97.49274	84.32744	.157773
N2	1.99010	12.04951	.003221
O2	.48926	3.38550	.000792
ARGON	.02367	.20473	.000038
CO2	.00341	.03245	.000006
TOTAL	100.00000	100.00000	.161831

TOTAL C1-C4 0.00000

CLOSURE [%] : .786 MEASURED PRESSURE: .1606

Reviewed By: MC Date: 1-8-02

Page 4 of 4

◂‡S P E C T R U M L I S T :SAMPLE SPECTRUM 8 Jan 2002 09:38

SAMPLE ID: 212H-010702-2235
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 8 Jan 2002 (09:22)

ALO NO.: 02-01046
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 138
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 160.57 mTorr
ANALYSIS TIME : 16:37.772
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 116.96 mTorr
INTEGR.INTENSITY: 2.6702E+0 V
BASE PEAK INT. : 2.3512E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e	Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650 @	2.0	1.4298E-4	.0061	.0054	7	30.3
2	4.002604 @	4.0	2.3512E+0	100.0000	88.0533	3	61.8
3	14.003074 @	14.0	1.5005E-2	.6382	.5619	3	173.2
4	16.008143 N	16.0	3.4523E-3	.1468	.1293	3	203.8
5	20.016431 N	20.0	5.8446E-4	.0249	.0219	7	264.8
6	28.006148 @	28.0	2.4362E-1	10.3618	9.1239	3	355.7
7	29.007390 N	29.0	1.7509E-3	.0745	.0656	3	372.2
8	32.004411 N	32.0	4.9698E-2	2.1138	1.8612	3	418.4
9	33.933219 N	34.0	2.1002E-4	.0089	.0079	7	449.7
10	39.962384 @	40.0	4.0272E-3	.1713	.1508	3	526.3
11	44.018545 N	44.0	5.0234E-4	.0214	.0188	7	588.5

Reviewed By: R.K. Barry Date: 1-9-02

Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

43469-L05

January 17, 2002

Frank W. Moore
Fluor Hanford, Inc.
PO Box 1000
Richland, WA 99352

Dear Mr. Moore:

TRANSMITTAL OF REPORT 43469-RPT05, "GAS SPECIES ANALYSIS OF SAMPLES FROM THE CANISTER STORAGE BUILDING RECEIVED BY PNNL ON JANUARY 10, 2002"

Reference: Statement of Work "Analysis of Gas Samples from the Spent Nuclear Fuel (SNF) Canister Storage (CSB) Facility" transmitted to PNNL on October 26, 2001, accompanying contract 11979-36 (PNNL Project 43469).

Attached please find a copy of report 43469-RPT05, "Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002." The samples described in this report were received by PNNL on January 10, 2002 in two batches; the preliminary results for samples 02-01249 and 02-01250 were transmitted on January 10, 2002 and the preliminary results for sample 02-01253 were transmitted on January 11, 2002. The attached report is the final deliverable associated with these samples.

All analyses performed and results reported in the attached document were conducted in compliance with OCRWM standards. This data has been reviewed and determined to be OCRWM-compliant.

Radiochemical Processing Laboratory (RPL) sample identification numbers used in this report were assigned as follows:

RPL Sample ID	Client Sample ID	Client Sample Description
02-01249	212H-010902-2144	He Gas Sample
02-01250	212H-010902-2232	MCO Gas Sample
02-01253	212H-011002-1517	MCO Gas Sample

If you have any question, please give me a call on 372-4828.

Sincerely,

Kurt L. Silvers,
PNNL Project Manager

Attachment

902 Battelle Boulevard • P.O. Box 999 • Richland, WA 99352

Telephone (509) 372-4828 ■ Email kurt.silvers@pnl.gov ■ Fax (509) 372-6515

CC: E Biebesheimer, FH (summary only)
MS Busch, FH (summary only)
DR Duncan, FH (full data package)
DW Smith, FH (full data package)

GD Bazinet, NHC (summary only)
JP Sloughter, NHC (summary only)

SJ Bos, PNNL (full data package)
AM Lewis, PNNL (summary only)
BM Thornton, PNNL (summary only)
43469 project file

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

January 17, 2002

Pacific Northwest National Laboratory

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Sample Analysis Letter

January 17, 2002

Pacific Northwest National Laboratory

Battelle
. . . Putting Technology To Work

Project No. 43469

Internal Distribution
File/LB

Date January 14, 2002

To Kurt Silvers

From Stan Bos Stan Bos

Subject Canister Storage Building gas sample analysis

Gas species analyses of samples taken at the Canister Storage Building (CSB) on January 10 and 11, 2002 have been completed. A report detailing the gas species detected is attached. Analysis was performed using the Finnigan MAT-271 (WC38625) high sensitivity quantitative mass spectrometer. The sensitivity of the instrument is checked daily prior to use with high purity N2 gas, and two air standards are run weekly to insure the instrument is operating correctly. The samples were assigned RPL sequence numbers 02-01249, 02-01250 and 02-01253.

Analysis of the evacuated sampler prior to taking the sample was not performed. CSB evacuates the sample cylinders before inletting the sample gas. Since this is a procedural step, a sampler cleaning analysis becomes unnecessary.

This analysis was performed and the report prepared following PNNL quality assurance plan Nuclear Quality Assurance Requirements and Description (NQARD). NQARD has been evaluated and found to be in conformance with the Office of Civilian Radioactive Waste Management (OCRWM) QA Program. The data in this report is OCRWM QARD Qualified Data.

PNNL project 43469 was setup for document and financial control of the sample analyses. Sample reports and data sheets are stored in the project RIDS located in ETB room 2619. Sample analyses are charged to work package F29087.

If you have any questions please contact Stan Bos at 376-5384.

Concurrence P.K. Barry 01-15-02

ES4-1900-001 (8/98)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Gas Analysis Summary Results

January 17, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: January 10, 2002
Subject: Gas Species Analysis

To: Kurt Silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 140
Measurement and test equipment WC38625

Sample Id.	212H-010902-2144		
Analysis Date:	January 10, 2002	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-01249		
Argon		<0.001	± 0	<10
Carbon dioxide		<0.001	± 0	<10
Carbon monoxide		<0.01	± 0	<100
Helium		99.98	± 0.01	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		0.017	± 0.001	170
Oxygen		0.002	± 0.001	20
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water was not detected			

Sample Id.	212H-010902-2232		
Analysis Date:	January 10, 2002	Mole Percent	Estimate of Precision	PPM
Log-in No.	02-01250		
Argon		0.023	± 0.001	230
Carbon dioxide		0.002	± 0.001	20
Carbon monoxide		<0.01	± 0	<100
Helium		97.5	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		1.95	± 0.04	19500
Oxygen		0.48	± 0.01	4800
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water was not detected			

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-5384 / mail slot P7-22
Date: January 11, 2002
Subject: Gas Species Analysis

To: Kurt Silvers

Analytical procedure: PNNL-98523-284 rev 0
Laboratory Record Book 56998 Page 140
Measurement and test equipment WC38625

Sample Id.	212H-011002-1517			
Analysis Date:	January 11, 2002	Mole Percent	Estimate of Precision	PPM
Log-In No.	02-01253			
Argon		0.027	± 0.001	270
Carbon dioxide		0.002	± 0.001	20
Carbon monoxide		<0.01	± 0	<100
Helium		97.1	± 0.1	
Hydrogen		<0.001	± 0	<10
Neon		<0.001	± 0	<10
Nitrogen		2.28	± 0.05	22800
Oxygen		0.57	± 0.01	5700
Nitrous oxide		<0.005	± 0	<50
Other nitrogen oxides		<0.005	± 0	<50
Total Hydrocarbon		<0.001	± 0	<10
Comments	Water was not detected			

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Chain-of-Custody Forms

January 17, 2002

Pacific Northwest National Laboratory

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-010902-01 Date 01/09/02 Field Logbook No. N/A

Point of Contact DOUG BLACK Phone No. 372-2543 MSIN S8-05

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300

Sampled By D.L. McKINNIS (Print) D.L. McK (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
212H-010902-2144	212H/MCO-TR003	01/09/02	2144	N/A	HELIUM GAS SAMPLE
212H-010902-2232	212H/MCO-TR003	01/09/02	2232	N/A	MCO CASE SEMOTE

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions: SAMPLE ANALYSIS TO BE PERFORMED PER CSB AGREEMENT, REF CONTRACT 00011979-000336 "CSB STATEMENT OF WORK"

EMAIL RESULTS TO CSB SHIFT MANAGER

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By		Received By	
STEVEN B CARTER (Print)	1-10-02 (Date)	Charles Goolsby (Print)	1-10-02 (Date)
S Blake Carter (Sign)	0712 (Time)	Charles Goolsby (Sign)	0713 (Time)
Charles Goolsby (Print)	1-10-02 (Date)	S J Bos (Print)	1-10-02 (Date)
Charles Goolsby (Sign)	0754 (Time)	S J Bos (Sign)	752 (Time)
(Print)	(Date)	(Print)	(Date)
(Sign)	(Time)	(Sign)	(Time)
(Print)	(Date)	(Print)	(Date)
(Sign)	(Time)	(Sign)	(Time)

FINAL SAMPLE DISPOSITION

Disposal Method Sample cylinders pumped out

Disposed By:

Print	Sign	Date	Time
S J Bos	S J Bos	1/14/02	0820

NOTE: This form is to accompany the sample(s) at all times.

BC-6000-560 (12/99)

SNF PROJECT CHAIN OF CUSTODY

Chain of Custody No. CSB-011002-01 Date 1-10-02 Field Logbook No. NA

Point of Contact STEVEN CARTER/Doug Black Phone No. 2-2260/ MSIN 58-03

Delivered to: ☐ OS Engineer ☐ 1706 KE Counting Facility ☐ 222-S Lab ☒ Other 325/300

Sampled By Kim Abrutis (Print) (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
2124-011002-1517	2124/MCO TR003	1-10-02	1517	NA	MCO SAMPLE (DIRECT)

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions:
SAMPLE ANALYSIS TO BE PERFORMED PER CSB AGREEMENT REF CONTRACT 00011979-000 36 "CSB STATEMENT OF WORK"

SENT REPORT TO CSB SHIFT MANAGER, DOUG BLACK, MIKE KLEM and FRANK MOORE AT A MINIMUM

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By		Received By	
Print: RK PiPoo	Date: 1-10-02	Print: Jack Ham	Date: 1-10-02
Sign: RKPoo	Time: 1529	Sign: Jack Ham	Time: 1535
Print: Jack Ham	Date: 1-10-02	Print: S J Bos	Date: 1-10-02
Sign: Jack Ham	Time: 1615	Sign: S J Bos	Time: 1615
Print:	Date:	Print:	Date:
Sign:	Time:	Sign:	Time:
Print:	Date:	Print:	Date:
Sign:	Time:	Sign:	Time:

FINAL SAMPLE DISPOSITION

Disposal Method Sample cylinder pumped out

Disposed By:

Print	Sign	Date	Time
S J Bos	S J Bos	1/14/02	0800

NOTE: This form is to accompany the sample(s) at all times. BC-6000-560 (12/99)

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Analytical Services Request

January 17, 2002

Pacific Northwest National Laboratory

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor: Signature Kurt L. Silvers
Print Name Kurt Silvers
Phone 372-4828 MSIN K9-08

PNNL Project #: 43469
Charge Code: F29087
Date Required: 1-22-02

Matrix Type Information

- ♦ Liquids: ___ Aqueous __ Organic Multi-phase
- ♦ Solids: Soil Sludge Sediment; Glass Filter __ Metal; Smear ___ Organic Other
- ♦ Other: Solid/Liquid Mixture, Slurry; X Gas Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ QA Plan: SBMS ____ HASQARD (CAWSRP) NQARD
- ♦ Additional QA Requirements? No ____ or Reference Doc # ____
- ♦ Field COC? No ___ Yes ✓
- + Lab COC Required? No ✓ Yes ____
- ♦ Hold Time: None X
  or RCRA ____ CERCLA ____ Other, Specify ____
  & Date Sampled ____ Time Sampled ____
- ♦ Special Storage Requirements: None X Refrigerate (4°C) ____ or Other, specify ____
- ♦ Data Quality Review Required? No X Yes ____

Disposal Information

- ♦ Disposition of Virgin Samples: Virgin samples are returned to requestor unless archiving provisions are made with receiving group! If archiving, provide: Archiving Reference Doc # ____
- ♦ Disposition of Treated Samples: Dispose ____ X Return ____

Waste Designation Information

- ♦ Sample Information Check List Attached? Yes ____
  or Reference Doc # ____
  or Previous ASR # 5949
  or Previous RPL ID # ____

Does the Waste Designation Documentation Indicate Presence of PCBs? No X Yes ____

Additional or Special Instructions ____

Send Report To Kurt Silvers Phone 372-4828
Phone ____

Preliminary results requested, as available? No Yes ✓ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 1-10-02
Delivered By (optional) ____
Time Delivered (optional) ____
Group ID (optional) ____
CMC Waste Sample? No ____ Yes ____
Cost Estimate, if requested: $ ____

Received By: S J Bos

ASR Number: 6332

RPL Numbers: 02-01249 - 02-01250 & 02-01253

RPG/CMC Work Accepted By: S S Bos Signature/Date: S S Bos 1-10-02

ASR FY2000 - RPG.doc

page 1 of 2

Analytical Service Request (ASR)

(REQUEST PAGE —— Information Specific to Individual Samples)

Lab Staff Use Only: RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only: Test #	Lab Staff Use Only: Library #
[illegible]	212H-010902-2144		Gas Species	[illegible]	[illegible]
[illegible]	212H-010902-2232			[illegible]	[illegible]
[illegible]	212H-010 011002 58 01-1401 -1517			[illegible]	[illegible]

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above __ ; On Attachment __]

ASR # 6332

Page 2 of 2

ASR FY2000 - RPG.doc

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Quality Control Check

January 17, 2002

Pacific Northwest National Laboratory

Pacific Northwest National Laboratory

From: 325 Gas & Isotopic Mass Spectrometry
Phone: (509) 376-3358 / mail slot P7-22
Date: January 07, 2002
Subject: Air standards from Finnigan MAT - 271 Mass Spectrometer

Analytical procedure: PNNL - 98523 - 284 Rev. 0
Laboratory Record Book 56998: Page 138
Measurement and test equipment WC38625

Accepted values for the composition of air :

	Mole percent
Argon	0.934
Nitrogen	78.08
Oxygen	20.95

Analyzed Values:

Analysis Date: January 07,2002

	Mole percent
Argon	0.938
Nitrogen	78.05
Oxygen	20.97

Analyzed Values:

Analysis Date: January 07,2002

	Mole percent
Argon	0.937
Nitrogen	77.92
Oxygen	21.11

Instrument Background:

Background analyses are run daily prior to sample analyses. Trace amounts of hydrogen and/or water in the 0.1 to 0.2 millivolt range were the only species detected. The background spectra is subtracted from each sample spectra.

Gas Species Analysis of Samples from the Canister Storage Building Received by PNNL on January 10, 2002

43469-RPT05, Rev. 0

Raw Instrument Data from Gas Analysis

January 17, 2002

Pacific Northwest National Laboratory

Page 1 of 6

MATRIX - EVALUATION LIST of 212H-010902-2144 10 Jan 2002 08:32

SAMPLE ID:	212H-010902-2144	ALO NO.:	02-01249
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	10 Jan 2002 (08:20)	PAGE:	140
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES. [Torr]
HYDROGEN	.00037	.00018	.000001
HELIUM	99.98074	99.86610	.178166
N2	.01728	.12080	.000031
O2	.00162	.01292	.000003
TOTAL	100.00000	100.00000	.178200
TOTAL C1-C4		0.00000	

CLOSURE [%] : -.007 MEASURED PRESSURE: .1782

Reviewed By: P.K.Berry Date: 1-10-02

Page 2 of 6

◄‡S P E C T R U M L I S T :SAMPLE SPECTRUM 10 Jan 2002 08:31

SAMPLE ID: 212H-010902-2144
ANALYST: STAN BOS SJ Bos
CUSTOMER: KURT SILVERS
CALCULATE DATE: 10 Jan 2002 (08:20)

ALO NO.: 02-01249
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 140
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 178.21 mTorr
ANALYSIS TIME : 10:52.205
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 143.69 mTorr
INTEGR.INTENSITY: 2.6244E+0 V
BASE PEAK INT. : 2.6215E+0 V

Attributes: ABORTED ACQUISITION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e	Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650 @	2.0	1.2673E-4	.0048	.0048	7	30.3
2	4.002604 @	4.0	2.6215E+0	100.0000	99.8911	3	62.7
3	14.003074 @	14.0	1.5483E-4	.0059	.0059	6	172.9
4	16.027623 N	16.0	9.8208E-5	.0037	.0037	6	203.4
5	28.006148 @	28.0	2.3000E-3	.0877	.0876	3	353.0
6	32.024423 N	32.0	1.7869E-4	.0068	.0068	7	413.4

Reviewed By: K.K. Barry Date: 01-10-02

Page 3 of 6

MATRIX - EVALUATION LIST of 212H-010902-2232 10 Jan 2002 09:01

SAMPLE ID:	212H-010902-2232	ALO NO.:	02-01250
ANALYST:	STAN BOS SBos	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	10 Jan 2002 (08:43)	PAGE:	140
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00078	.00034	.000001
HELIUM	97.54144	84.59407	.155627
N2	1.95116	11.84518	.003113
O2	.48135	3.33967	.000768
ARGON	.02336	.20263	.000037
CO2	.00190	.01811	.000003
TOTAL	100.00000	100.00000	.159549
TOTAL C1-C4		0.00000	
CLOSURE [%] :	.768	MEASURED PRESSURE:	.1583

Reviewed By: P.K. Berry Date: 1-10-02

Page 4 of 6

S P E C T R U M L I S T :SAMPLE SPECTRUM 10 Jan 2002 09:00

SAMPLE ID: 212H-010902-2232
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 10 Jan 2002 (08:43)

ALO NO.: 02-01250
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 140
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 158.33 mTorr
ANALYSIS TIME : 16:09.157
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 116.52 mTorr
INTEGR.INTENSITY: 2.5945E+0 V
BASE PEAK INT. : 2.2898E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.5005E-4	.0066	.0058	7	30.3
2	4.002604	@	4.0	2.2898E+0	100.0000	88.2547	3	62.8
3	14.003074	@	14.0	1.4432E-2	.6303	.5562	3	173.8
4	16.009958	N	16.0	3.2868E-3	.1435	.1267	3	204.2
5	20.015983	N	20.0	5.8815E-4	.0257	.0227	7	264.6
6	28.006148	@	28.0	2.3258E-1	10.1574	8.9644	3	355.1
7	29.007118	N	29.0	1.7166E-3	.0750	.0662	3	370.4
8	32.003129	N	32.0	4.7624E-2	2.0798	1.8355	3	415.8
9	33.931078	N	34.0	2.1147E-4	.0092	.0082	7	445.6
10	39.962384	@	40.0	3.8688E-3	.1690	.1491	3	520.6
11	44.013116	N	44.0	2.7542E-4	.0120	.0106	7	581.1

Reviewed By: P.K. Berry Date: 01-10-02

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MATRIX - EVALUATION LIST of 212H-011002-1517 11 Jan 2002 07:52

SAMPLE ID:	212H-011002-1517	ALO NO.:	02-01253
ANALYST:	STAN BOS	LAB SAMPLE NO.:	0
CUSTOMER:	KURT SILVERS	LRB NO.:	56998
CALCULATE DATE:	11 Jan 2002 (07:34)	PAGE:	140
CALIB. LIBRARY:	RDC01	M&TE NO.:	WC38625

COMPONENT	MOL%	WGT%	PART.PRES.[Torr]
HYDROGEN	.00051	.00022	.000001
HELIUM	97.12296	82.36334	.154467
N2	2.27562	13.50858	.003619
O2	.57171	3.87865	.000909
ARGON	.02728	.23132	.000043
CO2	.00192	.01789	.000003
TOTAL	100.00000	100.00000	.159043
TOTAL C1-C4		0.00000	

CLOSURE [%] : .844 MEASURED PRESSURE: .1577

Reviewed By: [signature] Date: 01-15-02

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◂‡S P E C T R U M L I S T :SAMPLE SPECTRUM 11 Jan 2002 07:51

SAMPLE ID: 212H-011002-1517
ANALYST: STAN BOS
CUSTOMER: KURT SILVERS
CALCULATE DATE: 11 Jan 2002 (07:34)

ALO NO.: 02-01253
LAB SAMPLE NO.: 0
LRB NO.: 56998
PAGE: 140
M&TE NO.: WC38625

EXPERIMENT : 47 (NORMAL RUN)
CHANNEL (RES.): FARII (220) Int.Time[s]: .1 MASS RANGE: 2.02 - 135.91

START PRESSURE: 157.71 mTorr
ANALYSIS TIME : 16:03.166
BASE PEAK NO. : 2 (m/e= 4.002604)

END PRESSURE : 116.35 mTorr
INTEGR.INTENSITY: 2.7514E+0 V
BASE PEAK INT. : 2.3797E+0 V

Attributes: NORMAL TERMINATION
ACQUIRED -SAMPLE SPECTRUM
SAVED IN FILE " "

No.	Precise m/e		Nominal m/e	Norm A [V]	Norm B [%]	Norm II [%]	Samples	Time [s]
1	2.015650	@	2.0	1.3026E-4	.0055	.0047	7	30.3
2	4.002604	@	4.0	2.3797E+0	100.0000	86.4894	3	62.7
3	14.003074	@	14.0	1.7378E-2	.7303	.6316	3	173.3
4	16.008298	N	16.0	4.0190E-3	.1689	.1461	3	203.6
5	20.017558	N	20.0	7.0789E-4	.0297	.0257	7	263.9
6	28.006148	@	28.0	2.8318E-1	11.8999	10.2922	3	353.5
7	29.010861	N	29.0	2.0365E-3	.0856	.0740	3	368.6
8	32.007062	N	32.0	5.9053E-2	2.4815	2.1463	3	413.2
9	33.930764	N	34.0	2.2051E-4	.0093	.0080	7	443.3
10	39.962384	@	40.0	4.7169E-3	.1982	.1714	3	517.8
11	44.018029	N	44.0	2.9167E-4	.0123	.0106	7	577.6

Reviewed By: P.K. Bang Date: 01-15-02