UCRL-ID-129732

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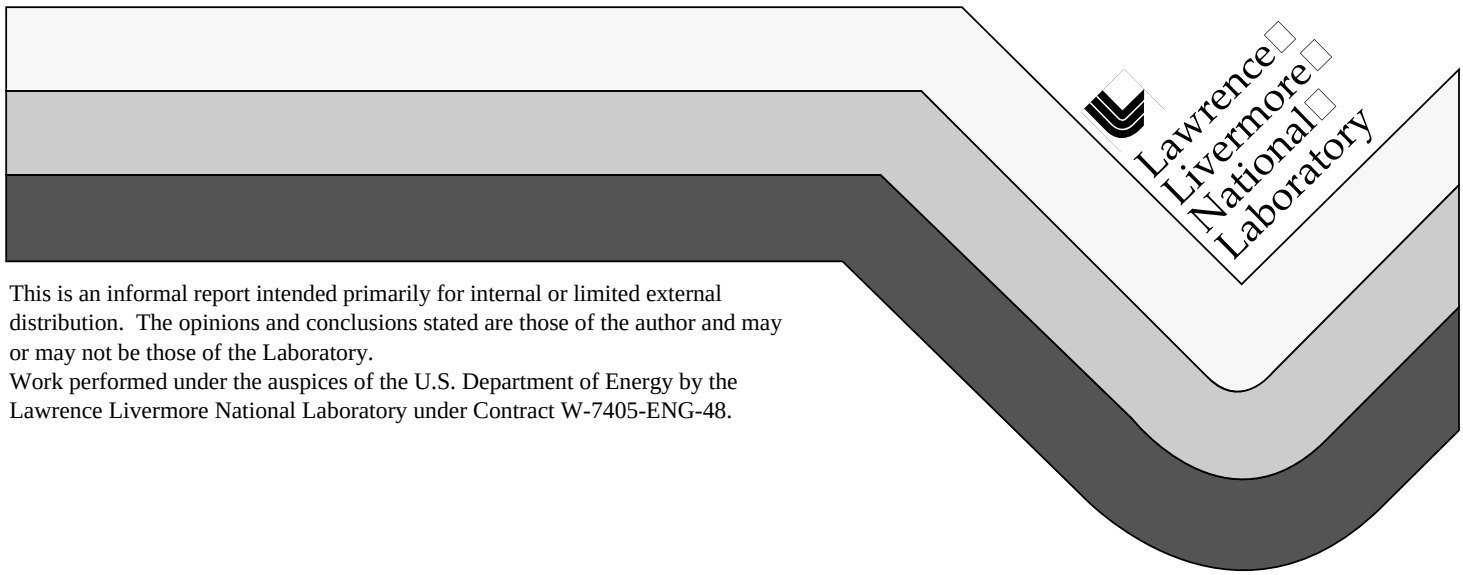
January 12, 1998



Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

Gas Sampling in the DST

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LLNL

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Introduction

Characterization of the rock-fluid interactions in the DST will play an important role in understanding the performance of waste package materials and radionuclide transport through the altered zone of a repository. Consequently, the chemistry of fluids and gases originating in the pore space of the rock and the changing compositions observed with time and temperature will be targeted for study in the chemistry boreholes of the DST.

The chemical holes have been lined with SEAMIST (Science Engineering Associate Membrane Insitu Sampling Technology) liners that allow gas and fluid from the pore spaces of the rock walls to be sampled on-site periodically. The concentrations of certain chemical species in the gases and fluids sampled at those locations will then be analyzed back in the laboratory. The baseline sampling of the rock-pore gases (prior to heater turn-on) is described.

Proposed Baseline Gas Sampling

The intent of the original gas sampling plan was that a background baseline of the rock pore gas chemistry would be established using at least three sets of sample analyses obtained during the pre-heat phase. The actual baseline sampling has been modified, however, to a single set of gas samples collected during pre-heat. The change resulted from the following factors:

i. The Seamist liner installation was completed late in the pre-heat stage such that possible sample collection of baseline gases was delayed.
ii. During the first attempt to collect baseline gases, air injection into the hydrology boreholes located immediately adjacent to the chemistry holes was simultaneously ongoing (LBNL) for permeability testing. The resulting gas samples obtained were considered corrupted and not suitable to establish baseline.
iii. Gas sampling opportunities were restricted by the TCO management in order to meet the modified heater turn-on date.

Gas Collection Procedure:

A gas sampling plan was devised to draw gas from the tubing mounted on the SEAMIST liners within the chemistry boreholes in increasingly “pore-space” -rich aliquots. Gas present in the length of the teflon-tubing at the time of sampling, is a mixture of rock-pore gas from the borehole walls and atmospheric gases diffused in from the observation drift. The first gas samples pulled from the tubing will be dominated by this mix of gases, but as the tubing is depleted, the gas originating predominantly from the rock walls will move down the tube to fill the space. Subsequent samples will therefore be rich in the desired gases.

The volume of gas approximately equivalent to the volume of the line was determined based on estimates of the teflon tubing length and diameter. The gas in the line is then removed in small increments: several samples are collected representing a fraction of the volume in each draw. Then after the full line volume is removed, a couple of samples are

consecutively pulled. The procedure we have developed for the gas collections has been tested and employed during two sampling trips. It utilizes gas tight syringes for the immediate on-site collection and injection of the collected gases into previously evacuated vessels for their containment and transport to the analytical labs. The resulting gas analyses of all the samples are then examined to observe the compositional differences, if any, of the gases originating in the lines and in the pore space.

We have obtained stainless steel sampling vessels which can be evacuated and leak tested before use. The vessels are prepared and evacuated according to standard practices employed in the LLNL Gas Mass Spectrometry Lab, B222, rm 1418. Several vessels are simultaneously attached to a manifold and pulled under house vacuum to < 0.1 mTorr. The evacuated vessels are then able to be carried or shipped to the ESF without loss of vacuum.

The collection of gas samples from a borehole proceeds by attaching a gas-tight syringe with a Luer-tip Mininert valve that fits into the compression fitting of the teflon tubing located on the Seamist liner. With the Mininert valve opened, an approximate volume of gas may be removed (as judged by the position of the plunger and the graduated markings of the glass syringe after the plunger has been drawn back). The Mininert valve is then closed and the syringe and valve tip are removed from the tubing. Immediately, the syringe and valve are connected to an on/off flow valve that has been fitted via a Luer-lock connection to the stainless steel gas sampling vessel. Once the syringe and vessel are attached, the flow valve and the Mininert valve may be opened. (Although the fittings and valves have a very small dead volume associated with their path openings, air is pulled from their pathways using the gas-tight syringe in a separate step. Consequently, the contamination from the dead volume is minimized thereby keeping the associated volume to a fraction of a percent of the overall volume of the gas sample collected.) The sampling vessel may then be fully opened to allow the vacuum to pull the sampled gases from the syringe and fittings; although to completely empty the syringe it may be necessary to inject the gases using the plunger. The stainless steel vessel may then be closed again and the sample labeled for identification. Although the sample vessels will be reused over and over for new sample collections and the gases will not be retained after completion of analysis, we will be generating YMP sample collection reports and correspondingly using the SMF bar code tags that we can attach to tags on the sample collection vessels.

Gas Analysis

Currently, upon the immediate return of samples to LLNL, the samples are sent for analyses to the Gas Mass Spectrometry lab here on-site. There the gas is analyzed for the volume percent of N_2, O_2, Ar, CO_2, CO, CH_4, He, and H_2 using a VG MM30-01 Gas Mass Spectrometer. The presence of other molecules up to molecular weight 160 are scanned but not necessarily quantified.

The attached table has the analytical results of gas samples from our background baseline collecting trip. All sample designations utilize an abbreviated borehole number and reference to one of six possible sample collecting ports located on each liner. The ports are located in pairs with ports 1 and 2 the farthest into the borehole, 3 and 4 are midway along the length, and 5 and 6 are closest to the collar or opening of the observation drift. The samples increase alphabetically wherever multiple and sequential samples from a single hole and port have been drawn. Samples correspond to the SMF bar-coded designations that are collected in controlled scientific notebook, #00342. Representative analyses of both the observation drift (OD) air and the heater drift (HD) air are also included.

Gas Analysis Summary

12/2/97 Samples

Sample Name	Nitrogen	Oxygen	Argon	Carbon Dioxide	Carbon Monoxide	Methane	Helium	Hydrogen	Total %
#54-1A	77.48%	21.55%	0.95%	0.02%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-1B	76.94%	22.07%	0.94%	0.04%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-1C	76.98%	22.02%	0.94%	0.06%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-1D	77.38%	21.65%	0.92%	0.05%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-1E	77.19%	21.86%	0.93%	0.02%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-1F	77.15%	21.86%	0.92%	0.07%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3A	77.49%	21.55%	0.93%	0.04%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3B	76.92%	22.11%	0.92%	0.05%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3C	76.79%	22.15%	0.93%	0.13%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3D	77.26%	21.76%	0.91%	0.07%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3E	77.14%	21.73%	0.91%	0.22%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-3F	77.52%	21.50%	0.91%	0.07%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-5A	77.00%	22.03%	0.93%	0.05%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-5B	76.93%	22.09%	0.93%	0.05%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-5C	77.46%	21.57%	0.92%	0.06%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-5D	77.21%	21.82%	0.92%	0.05%	<0.50	<0.01	<0.01	<0.01	100.0%
#54-5E	77.14%	21.87%	0.92%	0.07%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-1A	77.85%	21.22%	0.90%	0.02%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-1B	77.50%	21.54%	0.91%	0.06%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-1C	77.64%	21.39%	0.91%	0.06%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-1D	75.02%	23.89%	1.01%	0.08%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-1E	77.63%	21.42%	0.92%	0.03%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-3A	78.12%	20.94%	0.92%	0.02%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-3B	77.43%	21.59%	0.91%	0.07%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-3C	77.66%	21.39%	0.92%	0.03%	<0.50	<0.01	<0.01	<0.01	100.0%
#56-3D	77.56%	21.48%	0.92%	0.04%	co.50	<0.01	co.01	co.01	100.0%
#56-3E	77.48%	21.57%	0.92%	0.03%	<0.50	<0.01	<0.01	<0.01	100.0%
Average	77.25%	21.76%	0.92%	0.06%					

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