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Tc-99 Decontamination From Heat Treated Gaseous Diffusion Membrane -Phase I

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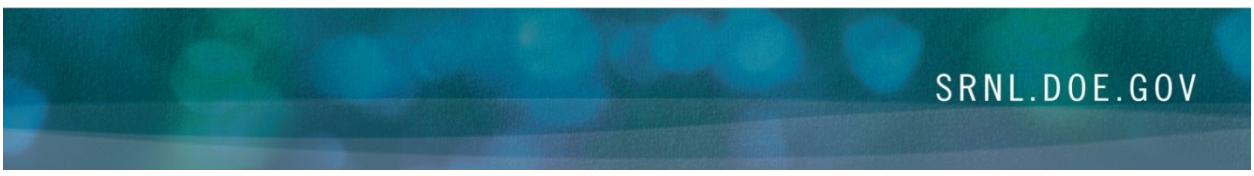
March 2017

SRNL-STI-2016-00740, Revision 0



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EXECUTIVE SUMMARY

Uranium gaseous diffusion cascades represent a significant environmental challenge to dismantle, containerize and dispose as low-level radioactive waste. Baseline technologies rely on manual manipulations involving direct access to technetium-contaminated piping and materials. There is a potential to utilize novel thermal decontamination technologies to remove the technetium and allow for on-site disposal of the very large uranium converters.

Technetium entered these gaseous diffusion cascades as a hexafluoride complex in the same fashion as uranium. Technetium, as the isotope Tc-99, is an impurity that follows uranium in the first cycle of the Plutonium and Uranium Extraction (PUREX) process. The technetium speciation or exact form in the gas diffusion cascades is not well defined. Several forms of Tc-99 compounds, mostly the fluorinated technetium compounds with varying degrees of volatility have been speculated by the scientific community to be present in these cascades. Therefore, there may be a possibility of using thermal desorption, which is independent of the technetium oxidation states, to perform an in situ removal of the technetium as a volatile species and trap the radionuclide on sorbent traps which could be disposed as low-level waste.

These initial scoping tests have shown the following:

- The results for both vacuum and flowing air treated barrier samples, at 450 °F for 72 hours, show that there is no significant Tc-99 or uranium removal from the barrier material pieces under these two test conditions. However, hot air treatment at 200-250 °F on gaseous diffusion equipment at all three sites with much higher Tc-99 concentrations successfully removed significant amounts of technetium. The system chemistry on the gaseous diffusion equipment was still in a fluorinating environment. For samples removed for this study, the volatile and semi volatile Tc-99 and uranium compounds in the cascades, now in a non-fluorinating, moisture/air environment may have been converted to their corresponding non-volatile forms like the technetium oxides (TcO_2 and Tc_2O_7) and uranium oxide (UO_2F_2). The lack of success in Tc-99 thermal decontamination with flowing air and vacuum at 450 °F may be attributed to the existence of these non-volatile oxide forms of both Tc-99 and uranium compounds in the barrier material.

- Superheated steam treatment of the barrier materials at 450 °F (232 °C), for as little as an hour, can initiate technetium and uranium isotopes removal from the barrier material. The percent technetium and uranium removal within the first one to eight hours of superheated steam treatment at 450 °F averaged 38 ± 10 % Tc-99 removal and 40 ± 4% uranium removal. As measured by both Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and Liquid Scintillation Counting (LSC), the average Tc-99 concentration remaining after steam treatment was 970 ± 29 pCi/g barrier material. When this post steam treatment Tc-99 concentration is converted to a Tc-99 concentration on a converter basis, the post steam Tc-99 concentration result is 176 ± 16 pCi/g of converter. The Paducah waste acceptance criteria (WAC) for on-site disposal cell is currently being developed. However, the Tc-99 WAC for the on-site disposal at Oak Ridge is 172 pCi/g of waste. This superheated steam treatment of the barrier material may have the potential to reduce Tc-99 concentrations to levels near the Oak Ridge's WAC. However, the use of steam treatments on fissile equipment will need to be thoroughly assessed by Nuclear Criticality Safety personnel.

- Analysis results of sub-samples of the "as-received" barrier material, taken along the length of sample 23008-Mid for Tc-99 baseline concentration, indicate that Tc-99 concentration along the length of the barrier material varies. However, the variations were within the measurement uncertainties for Tc-99.

The following recommendations are proposed:

Optimization tests on thermal decontamination of both Tc-99 and uranium isotopes from the Paducah barrier materials should be performed with emphasis on superheated steam treatment and not on flowing air or vacuum thermal treatments. The testing should also include lower temperature steam treatments, which would be easier to implement in the Paducah process.

The barrier material should be mounted in the test reactor in such a manner as to enhance contact between the interior of the barrier material and the flowing superheated steam. Such an experimental setup may improve thermal desorption/mass transfer of both Tc-99 and uranium isotopes out of the barrier material.

The chemical nature or speciation of the technetium compounds in the barrier materials need to be investigated along with determination of their distribution within the barrier material.

If optimization with superheated steam fails to attain a technetium decontamination waste acceptance level of 172 pCi/g converter or lower, it is recommended that an examination of other techniques such as fluorinating agents, to convert the technetium compounds in the barrier material into more volatile technetium hexafluorides or other volatile technetium compounds should be considered.

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LIST OF ABBREVIATIONS

AD	Analytical Development
COC	Chain-of-Custody
D_f	Decontamination factor
DL	Detection Limit
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
LSC	Liquid Scintillation Counting
MDA	Minimum Detectable Activity
PUREX	Plutonium Uranium Extraction
SRNL	Savannah River National Laboratory

1.0 INTRODUCTION

The Department of Energy's Portsmouth/Paducah Project Office (PPPO) is responsible for the large uranium gaseous diffusion plants that are now shutdown. As the plants operated to enrich uranium, some of the source of uranium included material that had been irradiated in the production reactors at Hanford and Savannah River. The spent fuels were processed using the Plutonium and Uranium Extraction Process (PUREX) to recover the uranium and remove the bulk of the fission product impurities. The first cycle PUREX product stream contained the purified uranium but also contained a small amount of impurities. The most notable one was technetium, in the form of Tc-99 pertechnetate. Following the Paducah flowsheet, the uranium oxide was reacted with anhydrous hydrogen fluoride to produce uranium tetrafluoride. Subsequently, the tetrafluoride reacted with fluorine to produce uranium hexafluoride. Golliher, et al,[i] determined that the technetium was entrained within the uranium in each of these steps. Upon entering the gaseous diffusion cascade, some of the technetium deposited within the barrier material.

It is estimated that more than 500 kilograms[ii] of Tc-99 were originally fed to the cascades, with the current Tc-99 inventory in the Paducah cascades at less than 50 kilograms[iii]. In addition to the cessation of feeding of Tc-99 contaminated UF_6 to the cascades, most of the Tc-99 had been removed during cascade and maintenance operation activities over the last 30 years of operations. The current baseline is to manually remove the barrier material and ship it off-site for disposal as low-level waste. This baseline is labor intensive and will be very costly. Therefore, developing and deploying an in-situ decontamination process, one which can reduce the manual labor, and risk of personnel radiological exposure, offers advantages in terms of schedule duration, costs and safety. An on-site disposal cell at Paducah is also being considered, and the waste acceptance criteria (WAC) is under development. The on-site disposal cell at Oak Ridge has a Tc-99 WAC[iv] of 172 pCi/g of waste and is a good reference point for this testing. To compare results from this testing in Tc-99 pCi/g of barrier to the WAC of 172 pCi/g of waste material, a correction factor was needed to convert from barrier to a converter. This factor can be obtained from the Paducah Technetium-99 Sampling Results Graph [v].

Early studies [vi] showed some removal of technetium from cascade components in the form of barrier material. Additionally, a statistical design set of experiments[vii, viii] showed removal of technetium at 30 – 60% at temperatures below 270 °F. The only direct evidence of a volatile technetium species in the cascades was determined by infrared spectroscopy to be in the pertechnetyl fluoride, TcO_3F[ix] form. However, there are other Tc-99 compounds with little volatility such as the oxides of technetium (TcO_2 and Tc_2O_7), pertechnetic acid ($HTcO_4$) and the oxyfluorides of technetium (TcO_2F_3 and $TcOF_4$).

Therefore, based on these early findings, there is the potential to utilize higher temperatures and perhaps different atmospheres to affect the decontamination, such as thermal desorption under vacuum, flowing air or flowing steam prior to the application of gases like hydrogen fluoride, fluorine or chlorine trifluoride.

2.0 SCOPE OF PROJECT

The objective of this SRNL scoping experiment was to demonstrate whether thermal Tc-99 decontamination of the barrier material, with a large decontamination factor, is possible with a technology based on flowing superheated steam, flowing air or vacuum conditions at a constant temperature of 450 °F (232 °C). The preliminary laboratory data could eventually be used for the scale up of a viable technology for Tc-99 decontamination from the barrier material.

This report documents the scoping investigations performed at SRNL to demonstrate Tc-99 decontamination ability of all three atmospheres mentioned above using Tc-99 laden Paducah barrier materials sent to SRNL.

3.0 SAMPLE RECEIPT

The Paducah barrier material samples were delivered to SRS/SRNL on April 13, 2016 and were transported and received at SRNL under a Chain-of-Custody (COC) form. After a general health physics survey and smears for external radioactive contamination, the sample container was moved into a security safe for storage. There were a total of 10 barrier material samples. One of them, a reference sample (NEW), had not been tainted with Tc-99 as confirmed through analytical data. Paducah provided 3 samples (top, mid, bottom) from each of 3 locations. The mid sample for each location was analyzed and considered representative of the other two samples. All SRNL test results presented in this report (Phase 1) were based on one of those 10 samples, which is referred to as either sample 23008-Mid (33-2.5.4-Paducah sample identification) or P_3 based on SRNL sample identification.

The Tc-99 analytical data provided by Paducah for three of the middle portion samples including the reference sample is presented in Table 1. The Tc-99 concentration in the reference sample was less than the minimum detection limits. In Table 1, the Paducah sample identification and analytical results are, respectively, in columns 1 and 2, along with their corresponding SRNL sample identifications and analysis results where applicable (columns 3 and 4). The Paducah analytical data for these four samples also included other radionuclides and select transition metals, which have not been included in the Table 1. No analytical data is provided for the other six barrier material pieces by either laboratory and sample 23008-Mid (33-2.5.4) was selected for the tests because the Paducah analytical result for Tc-99 was highest in this sample.

Table 1. Comparative Tc-99 Analytical Results by Paducah Analytical Laboratory and SRNL for Select "As-Received" Paducah Barrier Material Samples.

Paducah IDs	**Paducah Tc-99 Results, pCi/g**	**SRNL sample IDs**	**SRNL Average Tc-99, pCi/g**	**1 sigma % analytical uncertainty**	**Comments**
NEW	<4.55E+00, MDA	P_0	<9.0E+00	MDA	
C713-Mid (35-1.6.4)	1770 ± *19.7*	P_1	Not analyzed	Not applicable	
23327-Mid (33-5.5.4)	2370 ± *22.5*	P_2	Not analyzed	Not applicable	
23008-Mid (33-2.5.4)	*5670 ± 34.6***	*P_3*	1.62E+03 ± *382*	8.25 (LSC)	Average Tc-99 initial Concentration by LSC
23008-Mid (33-2.5.4)	*Not applicable*	*P_3*	1.53E+03 ± *153*	10 (ICP-MS)	Average Tc-99 initial Concentration by ICP-MS

** The same barrier material was analyzed by both laboratories but the sample preparations were different. The reason for the difference was understood and the decision was made to focus on the percent change for this study.

4.0 EXPERIMENTAL APPROACH, TEST METHODOLOGY and EQUIPMENT DESCRIPTION

In the preparation of pieces of the barrier material (subsamples) for testing, a small metal template was used as a guide to cut out small pieces of the barrier material to ensure that all the tests pieces of the barrier material used for experimentation were essentially equal in weight. Each subsample was put into an opaque (black) plastic centrifuge tube, sealed and doubled bagged for classified storage. The sealed tubes inside the double plastic bags were then individually surveyed by health physics (sample smears followed by LSC to account for total alpha/beta) to ensure that radioactive hood limits, whole body and extremity were not exceeded when running tests with subsample pieces. The sample preparations and all tests were performed inside a radioactive hood. The test methods involved the exposure of subsamples of

the barrier material (sample 23008-Mid) to a constant temperature of 232 °C (450 °F). It is worth noting that 450 °F is 200 °F higher than the typical Tc-99 hot air treatment at the Paducah plant facility. During these tests, the atmospheric conditions in the reactors were one of three conditions, superheated steam, vacuum or flowing air.

The first test environment was superheated steam. Under this scenario, the reactor containing subsamples of the barrier material was externally heated using a heating mantle to a preset temperature of 450 °F ± 5 °F while superheated steam was flowing around the sample inside the reactor for a given period of time. In the second and third test environments, vacuum and flowing air took take the place of the flowing superheated steam with new subsamples and reactors in use. Where necessary, as in the runs with vacuum and flowing air conditions, two new reactors, each containing a new subsampled piece of the barrier material, was run at the same time inside the security safe. These parallel runs reduced the time requirements to complete the Tc-99 thermal desorption experiments. Following the exposure, each sample of the barrier material was removed from the reactor and the technetium content analyzed after digestion of the material as described below. All these test activities, with the exception of the effluent gas handling section and the steam generation, took place inside a class 5 security safe, because of the classified nature of the material.

Both thermally treated (under the three conditions cited above) and untreated Paducah barrier materials, after complete digestions, were analyzed for Tc-99 by liquid scintillation counting (LSC) and inductively-coupled plasma-mass spectroscopy (ICP-MS). Uranium isotopes were analyzed by ICP-MS. It is assumed that Tc-99 is uniformly distributed throughout the length of the barrier material used for this thermal desorption study.

In the digestion and analytical processes, samples of the barrier material were digested in a combination of concentrated nitric and hydrochloric acids (aqua regia) and a few drops of 50% hydrogen peroxide. In preparation for LSC analysis of the digested material, matrix blanks were prepared and spiked with a Tc-99 standard. The samples were then spiked with Tc-99m and the technetium species were extracted from the matrix using an Aliquat-336 based solid phase extractant. Tc-99 concentrations were measured by LSC analysis and ICP-MS analysis. Tc-99m yields were measured with a NaI-well gamma spectrometer, and were used to correct the Tc-99 analyses for any technetium losses from the radiochemical separations. Portions of the digested samples were also analyzed for uranium isotopes by ICP-MS.

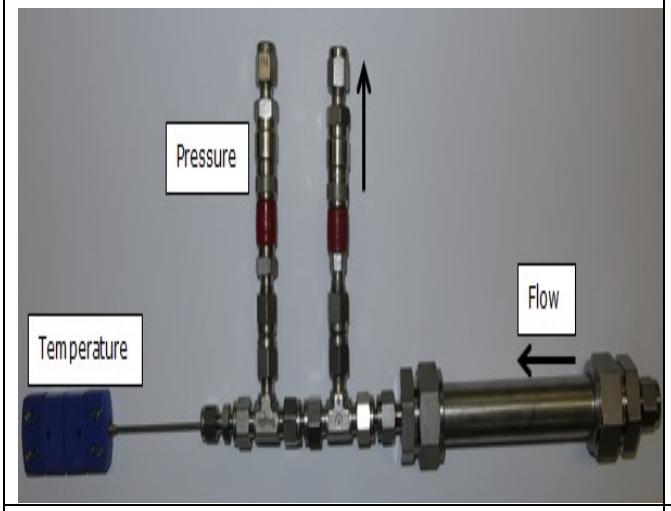
Table 1 shows a comparative analytical result summary for the same untreated Paducah barrier sample material (23008-Mid) for their Tc-99 content as reported by Paducah and SRNL. The untreated barrier materials ("as-received") apparently came from the same sources and were both analyzed by LSC methods. Please note that although the same barrier material was analyzed by both laboratories, the sample preparations were different.

4.1 Experiment Setup

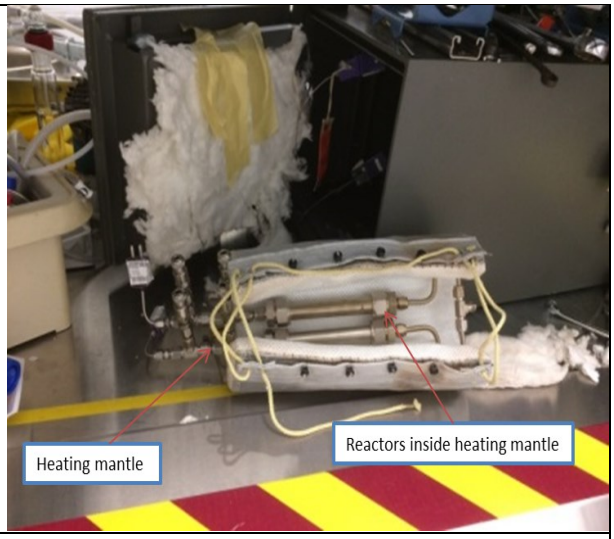
The overall experimental setup utilized one or two reactors to contain the test specimen, a heating mantle to house the reactors, superheated steam generator, a class 5 security safe needed to secure the sample specimen during unattended testing periods, and a radiological hood to protect the workers and environment from the radiological nature of the test materials. All tests were to be performed at a constant reaction temperature of 232 °C (450 °F). The piping and instrument diagrams (P&IDs) for flowing superheated steam, vacuum and flowing air setup are shown in Appendix A (Figures 19, 20, and 21). The data acquisition systems for the reactor temperatures and pressures, steam pressures and temperatures and the security safe interior temperatures are shown and described in detail in Appendix B, Figure 22 and the general the equipment/instrumentation used for the data acquisition are summarized in Appendix D.

4.2 Reactor

The device that held the barrier material was a 15-cm (6 inches) long stainless steel tube with an outside diameter of 1.9 cm (3/4 inches) and an inside diameter of 1.6 cm (5/8 inches). Figure 1, insert A shows a reactor with the flow from right to left and the two ports to measure temperature and pressure and an exit port. Figure 1, insert B shows the arrangement of two reactors inside the heating mantle which is designed to maintain a temperature of 232 °C ± 5 °C during the tests durations. There were two basic reactor set ups; one using a single reactor for steam and two reactors either air or vacuum. Figure 1 insert B and Figure 2 insert A show the setup with two reactors as they were covered with a heating mantle to maintain the reactors temperatures at 232 °C ± 5 °C during the test durations.



Insert A: Singles reactor showing thermocouple, pressure and exit connection port locations.



Insert B: Two reactors inside an open heating mantle shroud.

Figure 1. Test reactor experimental set up

4.3 Security safe

To ensure classified matter controls were in place, the test material was contained inside a classified repository as shown in Figure 2, inserts A and B. This work utilized a repository from Trusted Systems, Inc. and specifically the TSM131V IPS Security Container. The security safe is equipped with two interior exhaust fans to ensure adequate exhausting of hot air from the interior of the security safe during heating operations. Figure 2, insert A shows the reactors and heating mantle inside the repository before it was locked for testing.

The exterior plumbing on the rear of the security safe is shown in Figure 3, inserts A and B. Interior wiring, both inside the security safe and directly outside the heating mantle, consists of twisted/shielded thermocouple wires and extension wires with polyvinyl insulation (Newport Electronics EXPP-K-20-TWSH-UL-1000 UL Listed Shielded Extension Grade Thermocouple Wire) and with low noise connectors from Omega. These polyvinyl insulation grade wires and connectors have a maximum temperature rating of 90 °C. The immediate transfer lines, into and out of the security safe and reaction chambers (reactors),which carry the gases (flowing air and superheated steam) onto the barrier sample material being heated at 450 °F, were made of core convoluted 316L SS, followed by an outer

reinforcement of 321 SS braided hose. This had a maximum temperature rating of 454 °C. E-type thermocouples were used throughout the set up. The transfer lines from the exit section of the reactors, outside the security safe, to the chemical traps were made of 1/4th inch stainless steel tubing. The transfer lines for the two air driers which were connected in series were also made of 1/4th inch stainless steel tubing. The rest of the test equipment set up transfer lines inside the radioactive hood, from the exit portion of the chemical traps and scrubber solution, were made of flexible tubing.

The class-5 security safe, as described above and shown in Figure 2, insert B, was placed on a 36-inch diameter circular aluminum "Lazy Susan" (360-degrees rotating turn table) inside the radioactive hood. The 360-degrees swivel turn table made it easy to rotate the security safe inside the hood to ensure easy access into the safe and to open the safe during operations.

Insert A: Barrier materials are located in reactors that are surrounded by a heating mantle inside an open security safe. The heating mantle is required to maintain the reactors at 232 °C ± 5 °C for the test duration.

Insert B: Barrier materials, reactors, and heating mantle are inside closed security safe.

Figure 2. Interior and exterior of the class-5 security safe and the location of the reactors

The interior door of the security safe was covered with insulating glass wool material to prevent the temperature of the security safe door from reaching 30 °C (the safe electronic lock temperature requirement condition is 68 °C). The maximum temperatures recorded for the security door during the longest test durations of 72 hours were 23 °C and 21 °C for the security safe interior. It is worth noting that both ends of the heating mantle housing the reactors inside the safe were also insulated with glass wool and the interior security safe exhaust fan was allowed to operate throughout the duration of both the flowing air and vacuum tests. During the steam test, the safe fans did not operate as expected. They made a lot winding noises, which was similar to a failed bearing and so the safe fans were intentional turned off and the security safe door was cracked slightly open, about an inch, to minimize temperature build up on the security safe door and interior of the safe (heating mantle exterior).

4.4 Chemical Traps and Scrubber Solution

At the end of all the tests, under the three atmospheric conditions mentioned earlier, the goal was to perform a Tc-99 mass balance to account for most of the Tc-99 thermally desorbed from the barrier material if there was a significant thermal desorption of Tc-99 ($D_f \geq 11$). Previous studies [x] [xi] have shown

the ability to use chemical entities to remove technetium from gaseous stream. So, chemical traps consisting of inorganic sorbents and scrubber solution, which were connected in series with the reactor discharge, were to be analyzed for trapped Tc-99 at the end of the tests if the Tc-99 D_f were high.

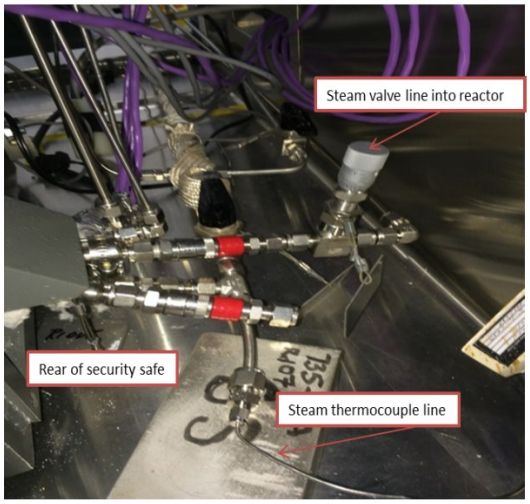
The pair of traps per reactor contained absorbent materials, magnesium fluoride (MgF_2 from Morita Chemical with a surface area of approximately 90 m^2/g), and activated aluminum oxide (Al_2O_3 from BASF).

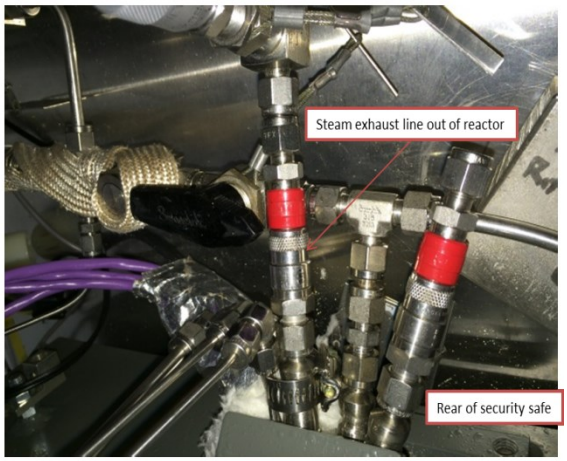
The air first entered the air driers and then passed directly into the two reactors in parallel flow. The flow exiting each reactor was then directed to two different pairs of chemical traps. The discharge of one reactor migrated through one pair of traps with MgF_2 first and Al_2O_3 second. The discharge of the second reactor migrated through the other pair of traps with Al_2O_3 first and MgF_2 second. This was done to determine if the order of trapping was important. After the air, or vacuum, left the traps it was routed through a rotameter and a pressure gauge indicator. Finally, the air stream was sent through a scrubber solution to remove any trace materials before the discharge of the scrubber was exhausted to the ventilation system inside the hood.

A typical glass sorbent trap container (volume capacity of 2 mL, length of 4 cm and internal diameter of 8 mm), was loaded with inorganic sorbent, magnesium fluoride (MgF_2) or activated alumina (Al_2O_3). The inorganic sorbents were provided by Paducah. The MgF_2 sorbent particles were cylindrical in shape, with an average length of 12.07 ± 2.32 mm and a diameter of 3.13 ± 0.07 mm. During the runs (Vacuum and Air flow runs), the MgF_2 sorbent trap columns were loaded with MgF_2 particles in which the individual particles were cut into halves to increase this sorbent loading inside the 2 mL capacity sorbent containers. The diameter of the "as received" spherical activated alumina balls averaged 3.52 ± 0.41mm.

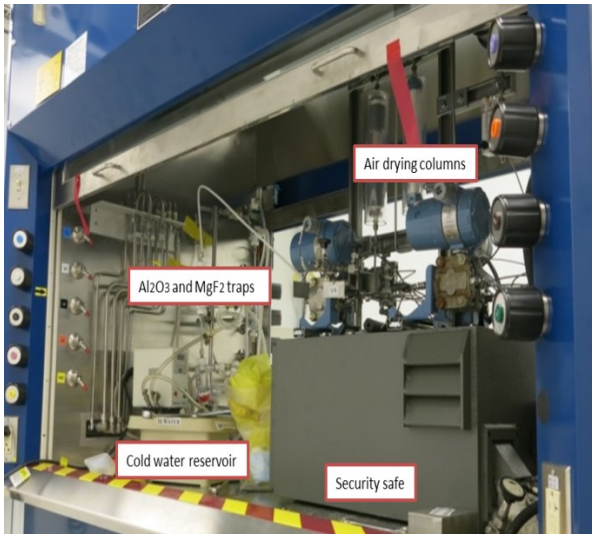
It is worth noting that the "as received" sorbent particles from Paducah were very large in size, only allowing a few to fit into the available glass trap tubes. Therefore, to be able to utilize more sorbent material and increase the exposed surface area of the sorbent particles, they were ground down to a smaller size ranging from 300 to 1000 microns. Unfortunately, the smaller sized particles caused a prohibitive pressure drop in the system during trial runs. It was decided to use the original sorbent materials from Paducah with a slight modification of the length of the MgF_2 particles only, as mentioned above, in order to fit better into the trap tubes. No further treatment, such as heat treatment to drive off moisture, was performed on the "as received" sorbents.

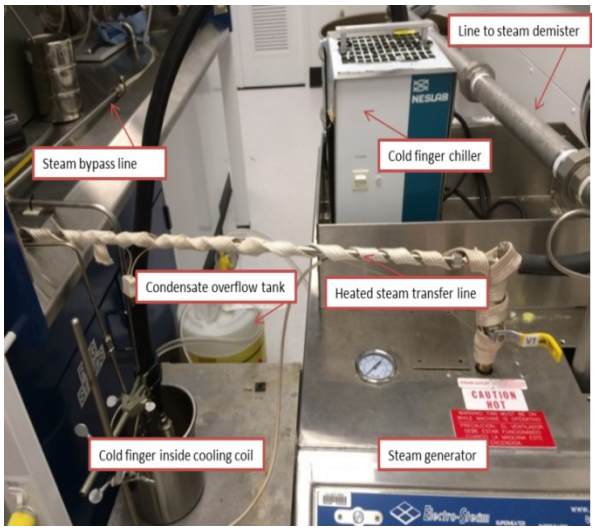
A mesh-100 stainless steel screen was attached to the bottom and top of each of the 2 mL capacity glass sorbent trap receptacles, with the 1.7-2.0 grams sorbent material being held between the screens (sorbent bed volume). After leaving the sorbent traps, but before entering the radioactive hood vent, the exhaust gases (effluents) from flowing air and vacuum treatments were passed through a solution containing 1.0 M ammonium carbonate solution (scrubber solution) inside a glass cylinder fitted with quick connects on both the entry and exit ends. For the steam test, the exhaust steam (after it left the reactor) did not travel through the sorbent traps, but was condensed in a separate container before being routed to the scrubber. That is both the air drier and sorbent columns were bypassed during steam runs because the steam would have condensed in the columns creating flow problems. However, samples of the condensate were taken to perform a Tc-99 mass balance on the steam if it were needed.





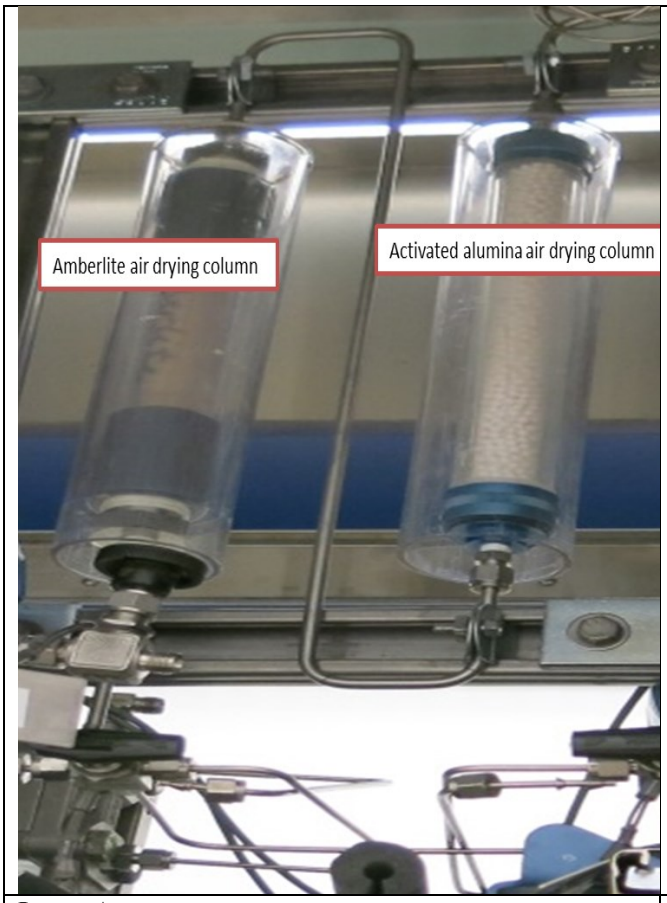
Insert A: Rear piping into and out of the security safe (steam)



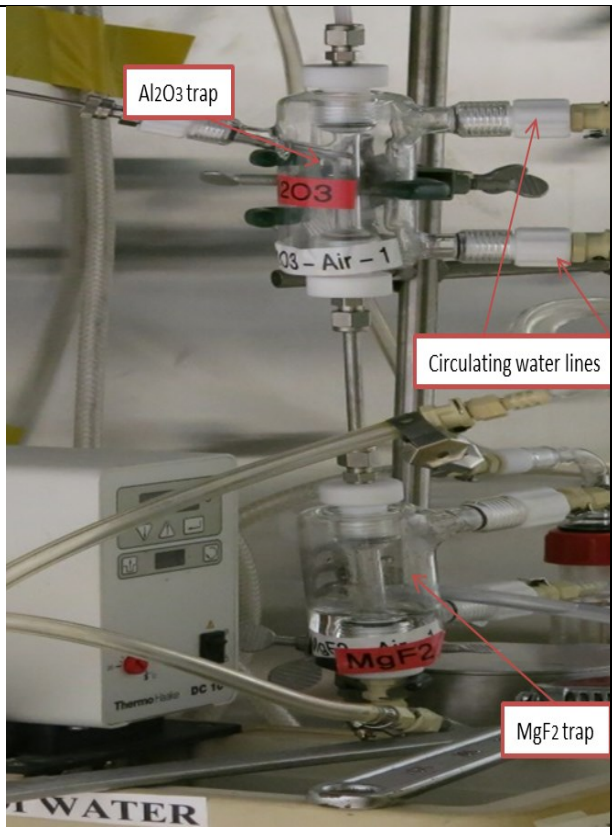


Insert B: Overall profile of test equipment in a radiological hood. Note the air driers on top of the safe and in the background are the chemical traps required to adsorb products from the reactor effluents. The heated steam transfer line in shown in the fore ground.

Figure 3. Test equipment set up in a radiological hood



Insert A: Two external air driers connected in series with the reactors inside the safe.



Insert B: One of two pairs of chemical traps. Each reactor is connected in series with a pair of inorganic traps.

Figure 4.Pre-reactor (influent) and post-reactor (effluent) gas treatment setup for air and vacuum tests.

4.5 Flow Systems-Vacuum and Air flow

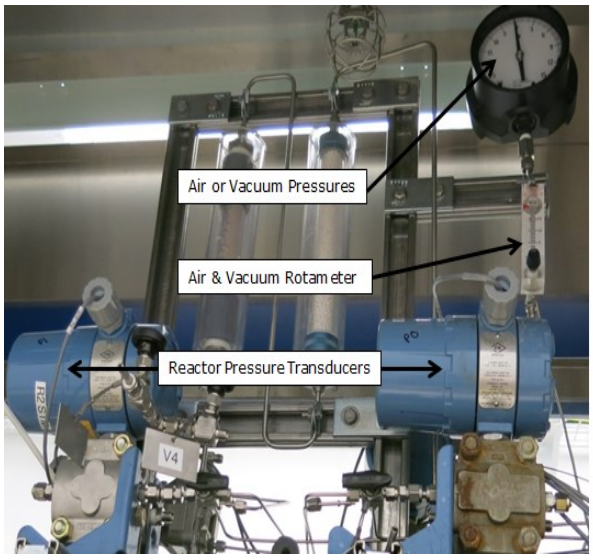
For either air or vacuum, the flow system of the test set up (Figure 4, inserts A, B; Figure 5, insert A and the P&IDs in Figures 20 and 21, Appendix A) was basically the same. The sole difference between the two systems was the motive forces and the two air driers were bypassed during the vacuum test to minimize leaks in the setup. The air was supplied by existing low pressure building air, which was connected to the intake of the two air driers (commercial grade activated alumina and amberlite) as shown in Figure 4, insert A. The vacuum condition in the setup was created with a corrosion resistant, oil-free diaphragm process pump (KNF Neuberger vacuum pump) and was connected just downstream of the traps, as show in Figure 4, insert B. Circulating water lines, as shown in Figure 4, insert B, were included in the setup and were to be used only if the temperature inside the chemical traps exceeded room temperature. These were never used because the interior chemical trap receptacles temperatures never exceeded 25 ^{o}C.

4.6 Flow Systems-Super-heated Steam

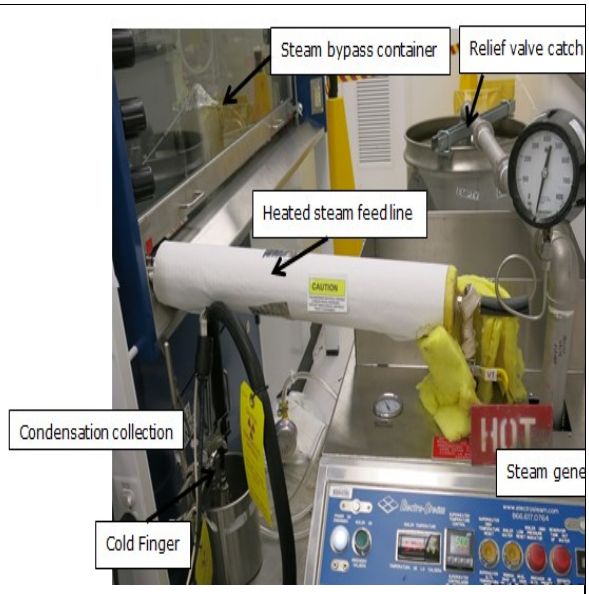
The steam equipment setup differed from the air and vacuum setup because steam had to be diverted from the air driers and chemical traps as well as only using a single reactor as explained below. The steam was simply collected and condensed as it discharged from a test reactor, but it has several handling issues. With steam, the water had to be handled in both its liquid and gas forms. The steam leaving the steam generator, Figure 5, insert B, had to be maintained above its saturated temperature at all times or it would

begin to condense (crash out). Therefore, the flow system up to and through a test reactor had to be heated. Working with superheated steam is challenging in many ways. Originally, the test setup had two reactors in parallel, as used in the air and vacuum tests. However, the upstream and downstream communication between the two reactors caused the steam conditions to be unstable and very difficult to handle. The decision to limit the test to a single reactor made the system much more stable.

Furthermore, the need to maintain the reactor and test specimen in a security safe meant that some of the tubing needed to be flexible and accessible. No modifications of the safe were permitted. There were certain sections of the tubing that could only be heated by the steam itself, which meant temperature fluctuations could occur and sometimes did and this led to saturation and water condensation. These conditions led to a temporary accumulation of condensed steam, which required saturated conditions of pressure and temperature. This saturated temperature was approximately 160 °C at the pressures provided by the stream generator, well below the 232 °C needed because of the pressure requirements. As the system was brought up to temperature, a steam bypass system with its liquid collection container as shown in Figure 5, insert B, was designed to heat the reactor upstream tubing. When the tubing was at temperature the steam was then slowly directed to the test reactor to heat the rest of the upstream tubing and to stabilize the temperature to the target of 232 °C. At this point the steam bypass was slowly closed so that all of the steam was flowing into and through the reactor and around the test specimen.



Insert A: Flow systems for air and vacuum being routed: (a) through two air driers before entering the reactors and (b) into chemical traps after leaving the reactors.



Insert B: Top of steam generator showing its control panel, pressure gauge, and the insulated and heated feed line leading to the test reactor inside the vent hood.

Figure 5. Flow systems for air and vacuum and steam generator control panel.

5.0 RESULTS and DISCUSSION

5.1 Data Quality and Presentations for Radionuclides

In the analytical results by LSC method, the one sigma percent uncertainty for Tc-99 reported in all tables is based on the pooled[1] estimate derived from the individual uncertainties for each replicate measurement for that radionuclide using an excel function, SQRT ((SUMSQ (x_i)/n)), where n is the number of replicates and x_i is the individual uncertainty associated with each radionuclide for each run. Here it is assumed that the radioanalytical processes, be it counting or other techniques, are of the same precision for each individual measurement.

In the analyses results presented in all tables in this report, values preceded by "<" (less than sign) indicate values were below minimum detection limits (MDLs), and values proceeded by "≤" (less than or equal to sign) indicate that for replicates, at least one of the analysis values was at or above the instrument detection limit or MDL and at least one of the analysis values was below the MDL. Thus, where replicate analyses were both above and below the detection limit, the average of all replicates above and below the detection limit is given and a "≤ " sign precedes the average value. The standard deviations reported in the analyte tables were calculated only for values that were all above the detection limits. The minimum detectable activity (MDA) is defined as the value above which instrument signal can be considered quantitative relative to the signal-to-noise ratio. The detection limit (DL) as used in ICP-MS analyses is equivalent to three times the standard deviation of the blank measurements.

5.2 Steam Operation Results

Following many hours of non-radioactive mock up testing, the safe and barrier material was installed in a radiological fume hood as described above. Four tests were conducted at 232 °C under a steam atmosphere. The temperature and pressure targets for these 1-hour, 4-hour and duplicate 8-hour steam exposures were 232 °C and 65 psig and are shown in Figures 6, 7, 8 and 9, respectively. As can be seen in all the tests, there were some issues in achieving these targets. Working with steam was much more challenging than air or vacuum because of the need to maintain the entire flow system above the saturation temperature after steam leaves the steam generation. This meant preheating and insulating most of the flow system. For example, in the one hour test, temperatures and pressures reached nearly 300 °C and 100 psig, respectively, during the first fifteen minutes of the testing. At that time, the automatic temperature controller was overcompensating while it was trying to reach the temperature target of 232 °C. As shown in Figure 6, the average temperature turned out to be 222 °C for the one hour test with a large temperature fluctuation with a standard deviation of ± 39 °C. The spike in the pressure was the direct result of the increased temperature. With time and experience the subsequent test runs became more stable.

[1] Pooled estimate (pooled standard deviation) is a method of estimating a single standard deviation to represent all independent deviation data. It is a weighted average of each group's standard deviation and not a simple average where all groups have equal effect.

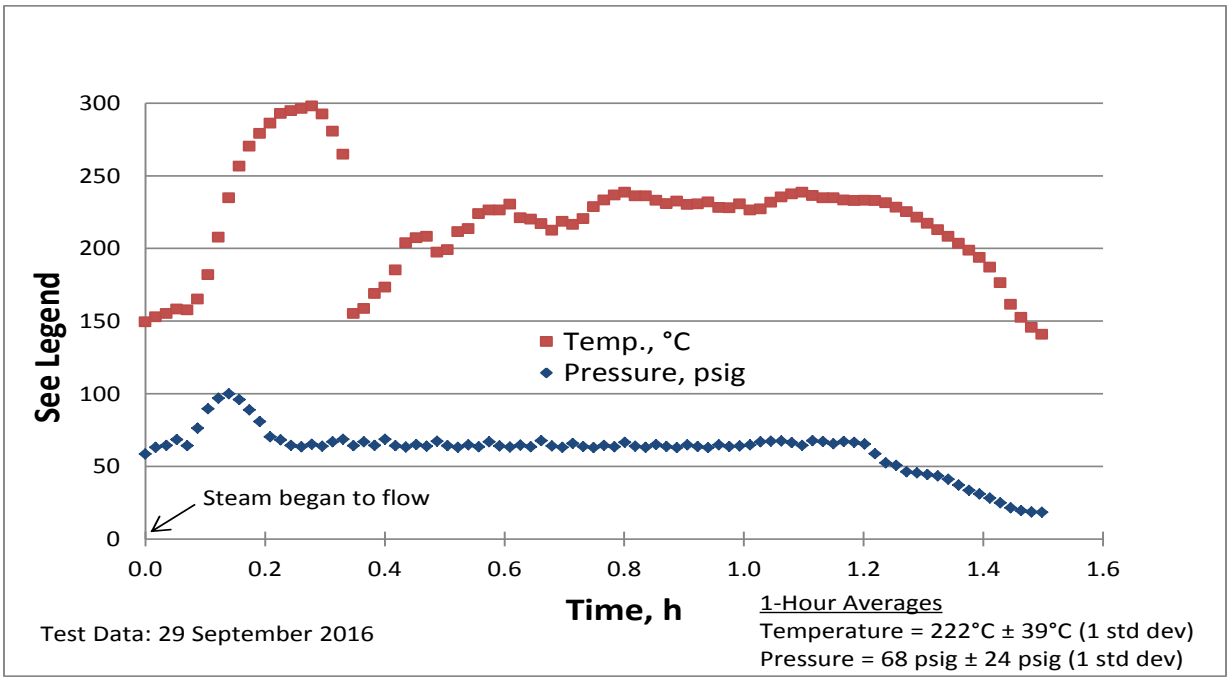


Figure 6. Pressure and temperature history of the 1-hour test with steam

As shown in Figure 7, the 4-hour test run with steam still showed significant fluctuations, but the automatic heater did a better job to stay near 232 °C, with a reduced standard deviation in the temperature of ± 24 °C, and ± 5 psig in pressure changes. The overall average temperature was 224 °C and the target pressure of 65 psig was maintained.

A lot of operational experience was gained from the 1-hour and 4-hour tests, so the two 8-hours test were much more stable. Figure 8 shows the temperature and pressure of the reactor during the entire first 8-hour test period. The average reactor temperature at 229 °C was within 3 °C of the target and the temperature fluctuations were considerably reduced with a standard deviation of ± 15 °C. The reactor pressure was very stable with an average of 65 psig and a standard deviation of ± 2 psig.

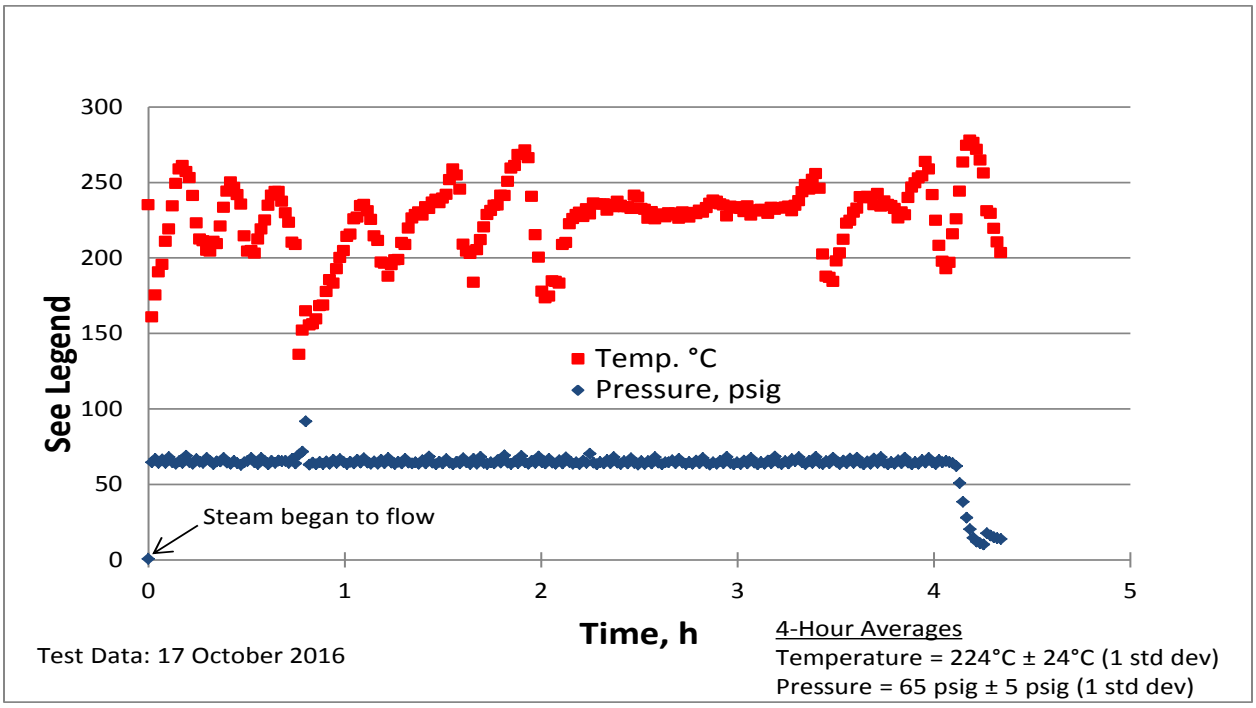


Figure 7. Pressure and temperature history of the 4-hour test with steam

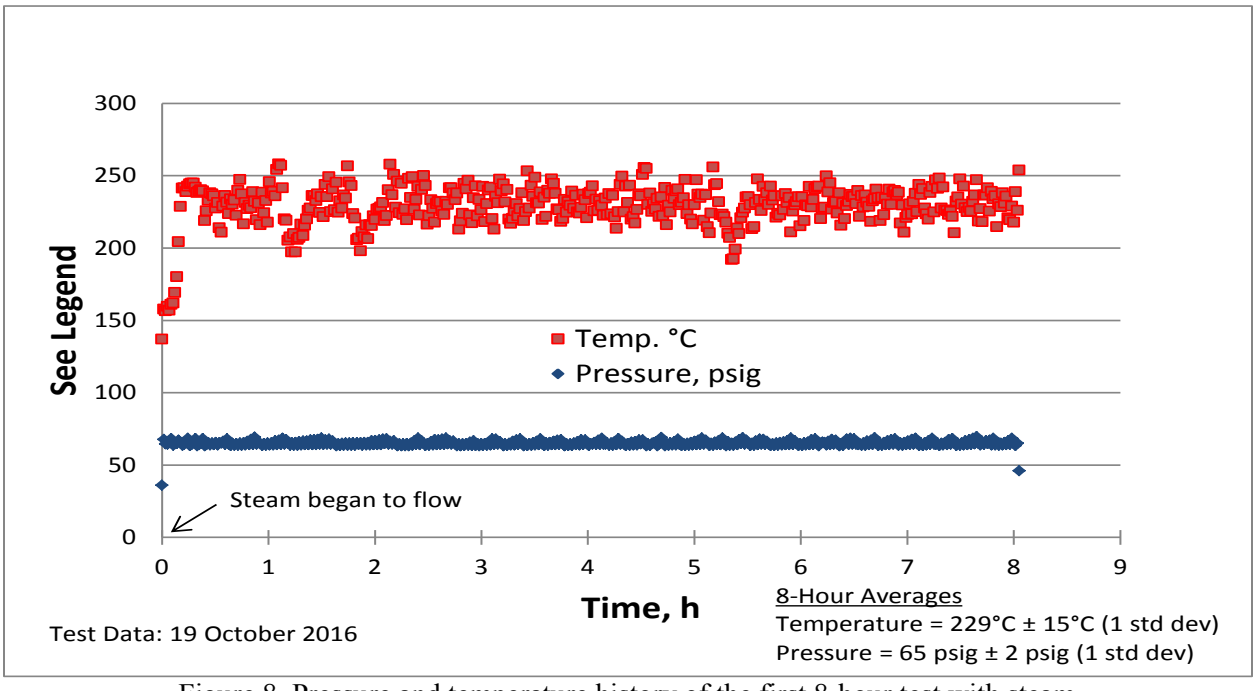


Figure 8. Pressure and temperature history of the first 8-hour test with steam

The temperature and pressure data for final steam test, i.e., the second 8-hour test, are shown in Figure 9. These results were similar to the first 8-hour test (Figure 8). So it appears the stability of the test apparatus with steam could not improve any further. In fact, it was the cycling of the steam generator and the heated conditions of the steam tubes that limited further stability. That is, the pressure and steam were

held constant within the limits of the steam generator used. The small steam generator utilized needed to continually cycle its boiler to maintain the needed temperature and this cycling traveled through the entire system. The cycling can be seen in Figure 10, which shows the fluctuation realized during the third hour of the first 8-hour test.

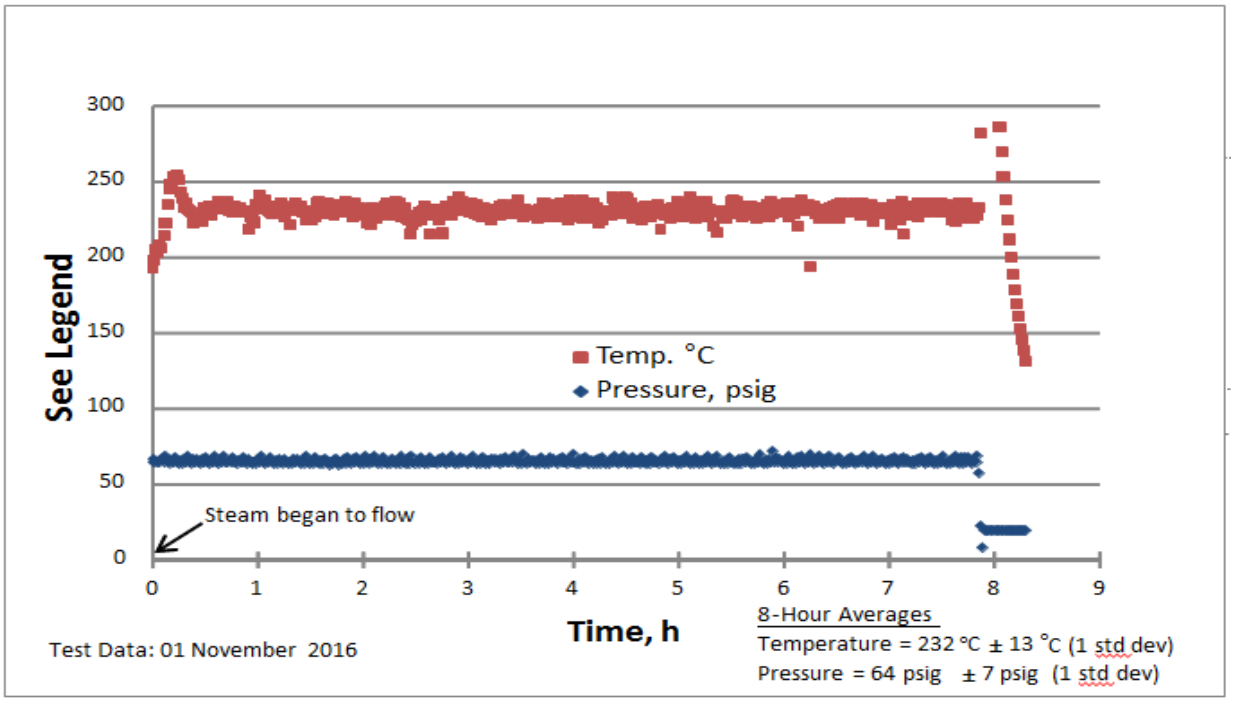


Figure 9. Pressure and temperature history of the second 8-hour test with steam

Actually, two cycle trends are seen and the steam generator is the cause of the lower frequency. That is, about every 12 minutes or 0.2 h, the increasing pressure represents the steam generator building up and then losing pressure. This pressure fluctuation was a direct result of the main boiler heating elements being switched on and off to maintain the required output temperature. The higher frequency cycle is a little more subtle to understand. As the stream travels out of the steam generator it first enters a heated and insulated stainless steel transfer tube. However, it then needs to enter through the safe wall where it cannot be heated. Once inside the safe the tube is fle^{x}ible to allow the reactor to be pulled out and is not heated for about 0.6 meters, until it is connected to the reactor. The volume of this unheated section of transfer tube is approximately 4.8 mL and the measured steam flowrate was approximately 4 mL/minute, which gives the residence time of steam in the unheated tube at approximately 1.2 minutes. Figure 10 shows 58 points in 60 minutes, which results in a periodic fluctuation of approximately 1 minute. This matches fairly closely to the steam residence time in the flexible heated tube. What seems to be happening is that the steam cools and heats through this section of tube which causes the pressure to rise and drop.

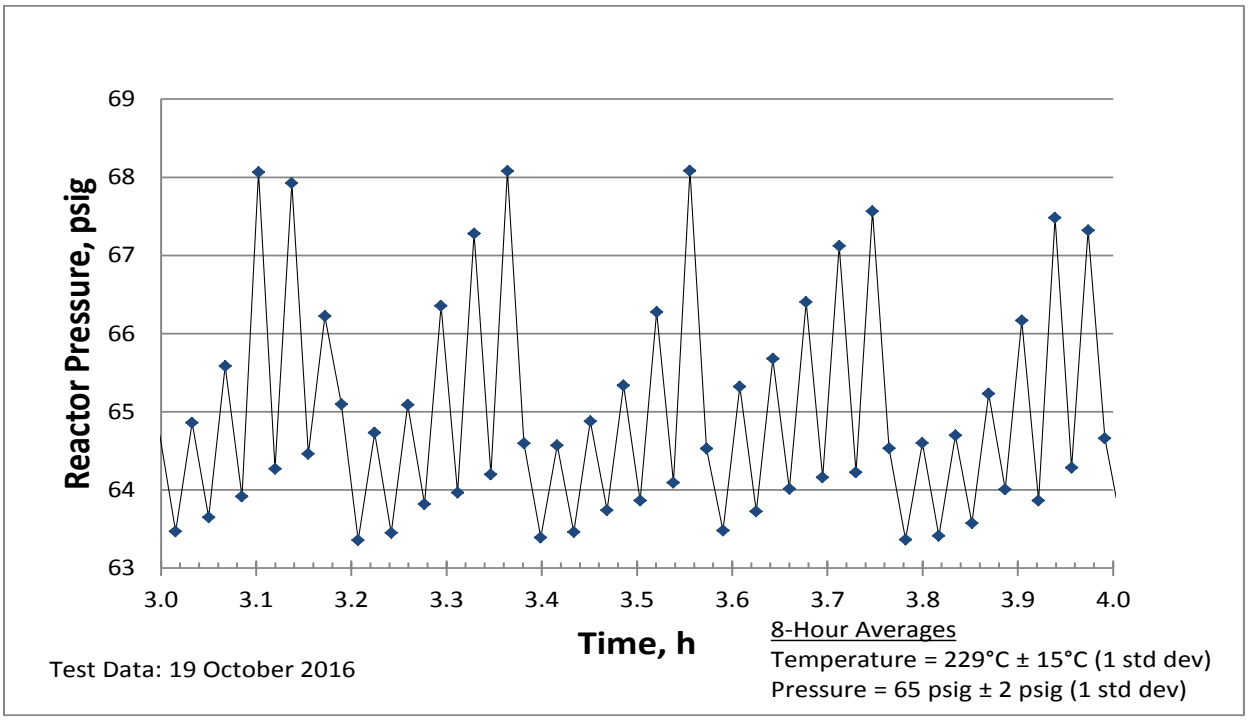


Figure 10. Pressure fluctuations in the reactor due to steam generator cycling; first 8-hour test

5.3 Decontamination Results with Steam

Table 2 shows the results for the removal of technetium from the pieces of barrier material that were placed in the reactor during steam exposure. The data for one and 4-hour runs were corrected to the concentration of the piece of barrier placed into the reactor. At end of these tests (one-hour and 4-hour tests only) water condensed in the reactor vessel during cool down and therefore the pieces were wet and weighed more than the starting weight. At the conclusion of the duplicate eight hour tests, an air purge was applied during cool down and it resulted in identical pre and post barrier material sample exposure weights. The data presented in Table 3 includes the technetium results by ICP-MS, with the ICP-MS complementary method used mainly to account for uranium isotopes in the samples as shown in Tables 4 and 5. In general, there was good agreement for Tc-99 analytical results using the two methods (Tables 2 and 3).

After a one hour steam exposure, about one third of the technetium was removed (28 % average based on LSC measurements and 27% average based on ICP-MS measurements) as presented in Tables 2 and 3. The Tc-99 decontamination factor (D_f) was 1.4 based on both LSC and ICP-MS analytical methods. The removal efficiency improved to about 50% after the 4-hour exposure (50% average based on LSC and average 52% based on ICP-MS data) with corresponding D_f values of 2.0 and 2.1. Although the 4-hour exposure showed the most percent removal of Tc-99 (average of 51%), one cannot conclude that the 4-hour run appears to be the optimum run time for the superheated steam treatment because of the presence of condensate at the end of the 4-hour run, which led to a Tc-99 data correction to account for the weight increase at the end of the run.

The technetium removal did not improve with additional exposure (8 hours). With duplicate runs, the percent of technetium removed averaged 42% based on LSC analysis and 32% based on ICP-MS, with an average D_f values of 1.7 and 1.5, respectively. Due the high test condition stability attained with the 8-

hour steam tests, the decontamination results from these tests are assumed to be more accurate at these steam conditions.

Although the results do not show decontamination to a level acceptable for onsite disposal at Paducah, the results do show a significant amount of technetium is removed. The steam results indicate several positive aspects in that it appears the removal kinetics for steam is very fast and is complete in less than four hours. One interpretation of this data is that the steam only permeated to a certain amount of the barrier material and removed that portion's technetium and the configuration was such that steam did not permeate throughout the entire piece of barrier material. Additional testing, such as directing the steam into the barrier material instead of 'bathing' it with steam and possibly cutting open the barrier material (longitudinal cutting into about two equal halves) for maximum exposure to steam to enhance mass transfer is warranted to improve the technetium removal efficiency with flowing superheated steam.

Table 2. Post-steam treatment analysis for Tc-99 by LSC in Sample 23008-Mid

Run description	**[Tc-99] pCi/g ; Post steam treatment**	**D_f**	**% Tc-99 removed**	**Comments**
Initial [Tc-99], pCi/g	1,620*	NA	NA	
ONE Hour RUN	1160	1.40	28.4	Corrected for weight changes due to the presence of condensate on sample.
Four Hour RUN	806	2.01	50.3	Corrected for weight changes due to the presence of condensate on sample.
Eight Hour Run-1	955	1.70	41.0	
Eight Hour Run-2	910	1.78	43.8	
Average	**958**	**1.72**	**40.9**	
Std. Dev.	*149*	*0.25*	*9.2*	

*Average of all four LSC analysis result for Tc-99 in the "as-received" barrier material. The one sigma percent counting uncertainty for Tc-99 by LSC was 6.74% (pooled estimate).

Table 3. Post-steam treatment analysis for Tc-99 by ICP-MS in Sample 23008-Mid

Run description	**[Tc-99] pCi/g ; Post steam treatment**	**D_f**	**% Tc-99 removed**	**Comments**
Initial [Tc-99], pCi/g	1,530*	NA	NA	
ONE Hour RUN	1120	1.36	26.7	Corrected for weight changes due to condensate
Four Hour RUN	729	2.09	52.3	Corrected for weight changes due to condensate
Eight Hour Run-1	1040	1.47	31.9	
Eight Hour Run-2	1030	1.48	32.6	
Average	**980**	**1.60**	**35.8**	
Std. Dev.	*172*	*0.33*	*11.3*	

*Initial Tc-99 concentration in the "as-received" sample 23008-Mid by ICP-MS was 1528 ± 156 pCi/g by ICP-MS

The digested barrier material pre- and post-steam exposure was analyzed for uranium by ICP-MS as well. In the "as-received" barrier material (pre-steam exposure), concentrations of the U-233 and U-236 isotopes were below the instrument detection limits. The concentrations of the other uranium isotopes (U-234, U-235 and U-238) along with Np-237 are shown in Table 4. However, after steam treatment of the

barrier material samples (one-hour, 4-hour and two 8-hours runs) the ICP-MS analytical results for the uranium isotopes did not show any measurable quantities of U-234 and Np-237, although U-235 and U-238 were measurable. As presented in Tables 4 and 5, an average loss of combined U-235 and U-238 was 40% after treatment with steam. It is worth noting that uranium, possibly in the oxy-fluoride forms, is slightly soluble [xii] in water. The decrease in uranium and neptunium concentrations, which were significantly lower in the "as-received" barrier material, may be attributed to the solubility of their oxy-fluoride forms in steam during the steam treatments.

Table 4. "As-received" analytical results for uranium isotopes in sample 23008-Mid

Analytes	Average, pCi/g	St. dev.
U-234	**1.55E+01**	*1.05E-01*
U-235	**1.27E+00**	*8.0E-02*
U-238	**7.87E+01**	*6.2E+00*
Np-237	**3.75E+00**	*1.18E+00*

Table 5. Post-steam treatment analytical results for uranium isotopes

Analytes	One-hour run, pCi/g	Four-hours run, pCi/g	Eight-hour Run-1, pCi/g	Eight-hour Run-2, pCi/g	Average, pCi/g	St. dev.
U-235	7.65E-01	7.61E-01	8.15E-01	7.07E-01	**7.62E-01**	*0.044*
U-238	4.68E+01	4.74E+01	5.11E+01	4.27E+01	**4.70E+01**	*3.44*
		D_f values				
U-235	1.66E+00	1.67E+00	1.56E+00	1.80E+00	**1.67E+00**	*9.8E-02*
U-238	1.68E+00	1.66E+00	1.54E+00	1.84E+00	**1.68E+00**	*1.20E-01*
		%Removed				
U-235	39.8	40.1	35.8	44.3	**40.0**	3.5
U-238	40.5	39.8	35.1	45.7	**40.3**	4.3

5.4 Vacuum Operation Results

Following the steam test, the equipment was set up for vacuum testing. The vacuum setup was similar to the flowing air setup with the only difference being the introduction of an oil-free diaphragm pump inside the radiological hood, downstream of the reactors and the chemical traps, to pull a vacuum through both reactors and the chemical traps. Thus, the air drier assemblies (activated alumina and amberlite®) as shown in Figure 4, insert A and Figure 5 insert A, which were not leak tight, were bypassed to improve the vacuum conditions inside the setup. The feed tube to the two reactors was also capped to minimize leakage. That is, there was no flow of air through the reactors and whatever exited the reactors during the vacuum test is what came off of the test material within the reactors. Once the reactors were capped off the vacuum testing proceeded smoothly. From the reactors, the vacuum flow stream traveled through the four inorganic traps to the vacuum pump, the scrubber solution, and then out to the hood vent as shown on the P&ID in Appendix A. Duplicate experiments were performed on pieces of the barrier material in two separate reactors. The residual vacuum air flow (leaked air) through the heated reactors and the inorganic sorbent columns (Al_2O_3 and MgF_2) was at a superficial velocity of 0.313 meters/second (1.03 ft. /sec). The average vacuum pressure inside the two reactors containing samples was 412.8 ± 2.0 Torr.

Figure 11 shows the pressure and temperature history for the first 3 hours of the vacuum test which lasted a total of 72.5 hours. Only the first three hours are shown in Figure 11 because both parameters (temperature and pressure) were very stable throughout the test. The interior temperatures and pressures

inside both reactors averaged 235 ± 3 °C and 415 ± 2 Torr[2], respectively. The fluctuations are one sigma standard deviation based on the data collected over the entire test period. There was a small, momentary drop in temperature at about 1.5 hours into the test because the temperature controller needed to be reset, which required the heater to be turned off and on. However, the impact was insignificant.

5.5 Decontamination Analytical Results under Vacuum Conditions

The average Tc-99 analysis results for the "as-received" barrier material was 1,530 ± 156 pCi/g based on ICP-MS method (Tables 6, 7 and Appendix C). The Tc-99 concentration in the post vacuum treated barrier sample pieces in the two reactors averaged 1,475 ± 148.5 pCi/g based on ICP-MS. The percent Tc-99 removed with vacuum treatment of the barrier material and the D_f values are presented in Table 7. The average percent Tc-99 removed was less than 5%; which is statistically not significant. As presented in Table 6 also, there are no measurable changes in uranium isotope concentrations in the post-vacuum treated barrier material. The post-vacuum treated barrier material uranium concentrations (U-235 and U-238) at, respectively, 1.26 ±0.01 and 76.9 ± 1.2 pCi/g are not statistically different from their initial "as-received" concentrations in the barrier material, which were 1.27 ± 0.08 and 78.7 ± 6.2 pCi/g, respectively. Therefore, it can be concluded that there was no significant decontamination of Tc-99 or uranium isotopes with vacuum treatments of the barrier material at 232 °C for 72 hours. There was a lot of scatter in the LSC data for Tc-99 post vacuum treatment and so the LSC data was not considered useful (Appendix C).

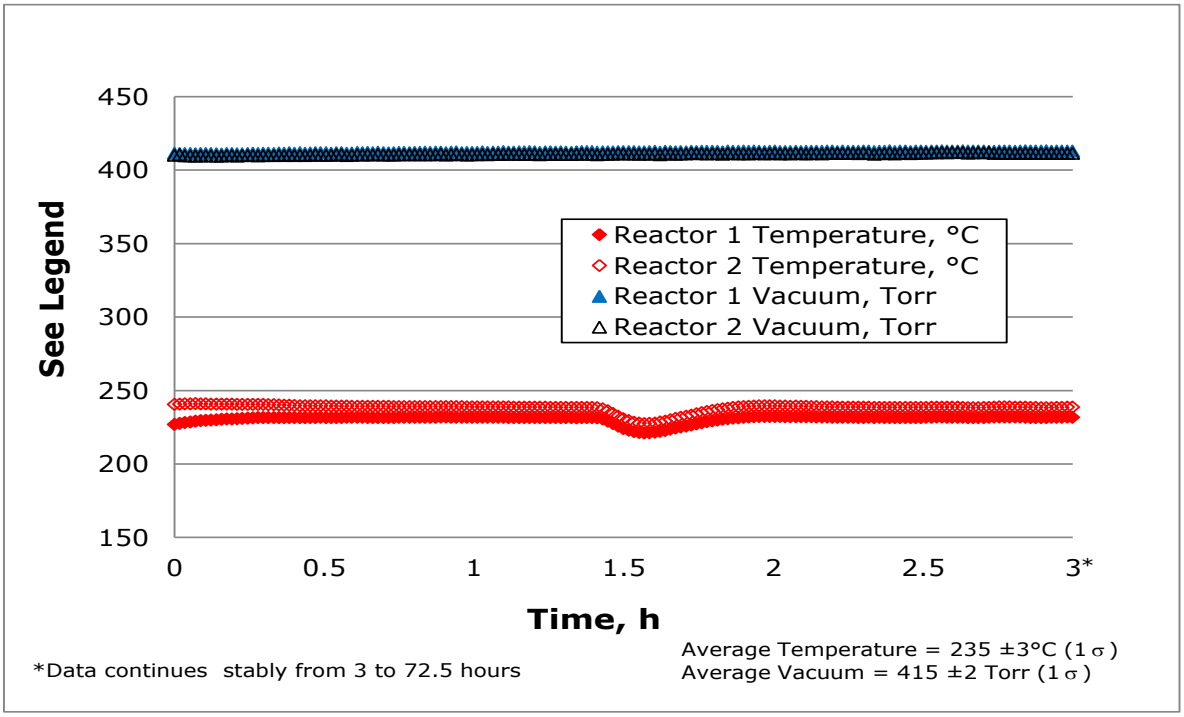


Figure 11. Pressure and temperature profiles for the 72-hour test under vacuum conditions

[2] Over the entire test period the atmospheric pressure was 752 ±3 Torr. The difference of the pressure inside to the outside of the radiological hood was insignificant, 0.05 Torr.

Table 6. Post-vacuum treatment analytical results for uranium isotopes and Tc-99, by ICP-MS

Analytes	Initial concentrations, pCi/g	72-Hour Run-1, pCi/g	72-Hour Run-2, pCi/g	**Average,** pCi/g	**Std. Dev**
U-234	1.55E+01	1.42E+01	1.44E+01	**1.43E+01**	*1.17E-01*
U-235	1.27E+00	1.25E+00	1.26E+00	**1.26E+00**	*1.01E-02*
Np-237	3.75E+00	2.95E+00	4.61E+00	**3.78E+00**	*1.18E+00*
U-238	7.87E+01	7.77E+01	7.60E+01	**7.69E+01**	*1.20E+00*
Tc-99	1.53E+03	1.58E+03	1.37E+03	**1.48E+03**	*1.50E+02*

Table 7. Post-vacuum treatment analysis for Tc-99 by ICP-MS in Sample 23008-Mid

Run description	**[Tc-99] pCi/g ; Post vacuum treatment**	**D_f**	**% Tc-99 removed**	**Comments**
Initial [Tc-99], pCi/g	1.53E+03	NA	NA	This was the average of three ICP-MS analysis results. Standard deviation = 150 pCi/g
72-Hour React-1	1.58E+03	0.97	~0	
72-Hour Reactor-2	1.37E+03	1.12	10	D_f and percent removal not statistically significant based on analytical uncertainty.
Average	**1.48E+03**	***1.04***	**~5**	
Std. Dev.	*1.50E+02*	*0.1*		

The one sigma percent counting uncertainty for Tc-99 by LSC was 8.25% (pooled estimate).

5.6 Flowing Air Operation Results

The final test was with the flow of air through the reactors at a target temperature of 232 °C. Duplicate experiments were performed on pieces of the barrier material with two reactors. Air was supplied just upstream of the air driers and then traveled through the two reactors in parallel, to the traps, through the rotameter, and then to the scrubber solution and exited out through the hood vent. The air flow rate was set at 2 scf/hr. (5.66E-02 m^3/hr.). This air flow rate translates to a superficial velocity of 1.03 ft/second or 3.13E-01 m/second through the inorganic sorbent columns of Al_2O_3 and MgF_2. However, there were some initial problems. Figure 12 shows the entire pressure and temperature history of the two reactors for the 75.5 hours of the test. The test was extended past the designated 72 hours of the testing to 75.5 hours because of the initial problems which took about 3 hours to correct. During the first 33 hours there were a few challenges to keep the test running smoothly. During that initial period the scrubber contained 1.0 M ammonium carbonate (a good reagent to capture excess technetium not trapped by the inorganic reagents- Al_2O_3 and MgF_2), however, it may have been the source of the instabilities. The air flow rate was set at approximately 2 scf/hr. with the reactor pressure at about 6.5 psig. However, periodically the pressure would increase up to 10 psig while the airflow rate would drop. During one of the experiments, the downstream flow system was dismantled to see what was causing the problem. The problem was attributed to the swelling of the internal rubber 'o' rings of the quick connect on the scrubber outlet sections in contact with ammonium carbonate solution or its vapors. This closing prevented the free flow of air and the resulting pressure build up. After 33 hours of testing, and several attempts to clean the system, the ammonium carbonate solution was replaced with just plain distilled water and the problem went away because plain distilled water was quite compatible with the rubber 'O' ring used. From the 33rd hour to the end of the test the pressures and temperatures were stable. The flow rate was slightly adjusted from 1.9 to 2.0 scf/hr. at the 45th hour, but in general the test was very stable. Even with all the upsets, the average temperature was 232 ± 4 °C (1 sigma), which was right on target, and the pressures inside the two reactors averaged 6.4 ± 1.1psig (1 sigma). The average absolute air pressure inside the two reactors containing samples was 20.85 ± 0.52 psi (1078.01 ± 12.95 Torr).

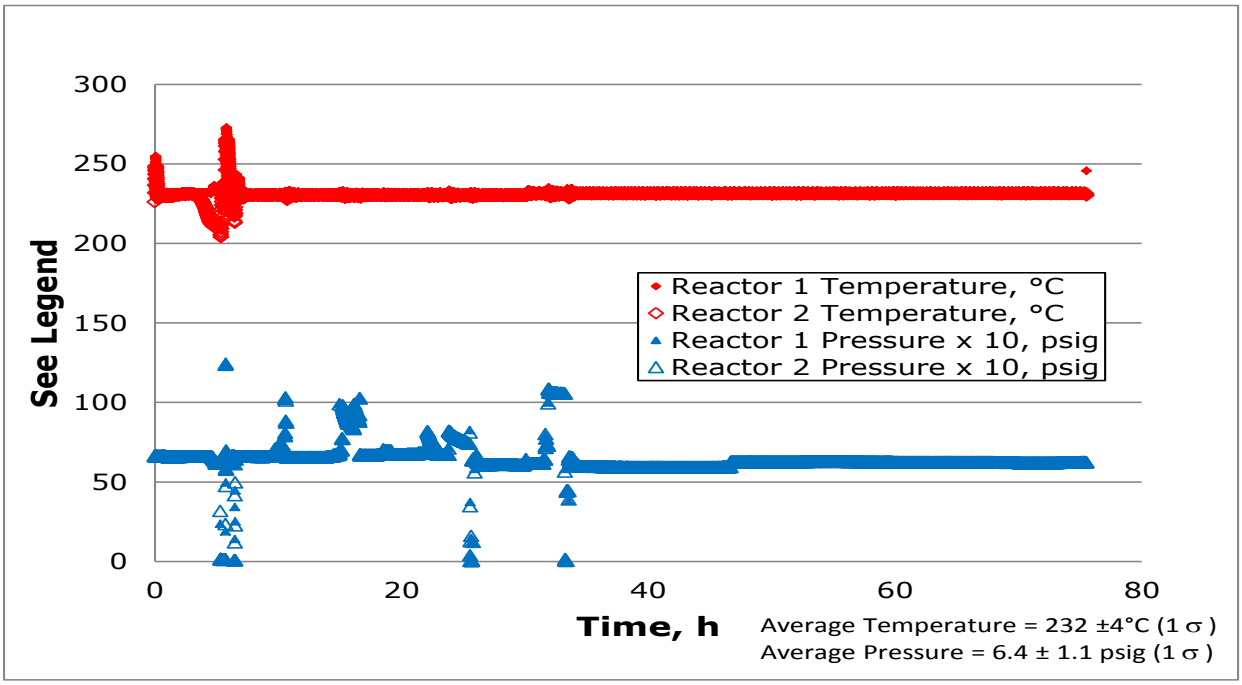


Figure 12. Pressure and temperature profiles for the 72-hour test under flowing air condition

5.7 Decontamination Results under Flowing Air Condition

There was statistically insignificant decontamination of Tc-99 or uranium isotopes with flowing air treatment of the barrier material at 232 °C for 72 hours. The data presented in Tables 8, 9 and Appendix C shows that there were no significant changes in the concentration of Tc-99 or uranium isotopes in the post-flowing air treatment of the barrier material at 232 ^{0}C for 72 hours when compared with the concentration of these species in the "as-received" barrier material. The average Tc-99 analysis results for the "as-received" barrier material was 1,530 ± 156 pCi/g based on the ICP-MS method (Tables 8, 9 and Appendix C). The Tc-99 concentration in the post vacuum treated barrier sample pieces in the two reactors averaged 1,400 ± 64 pCi/g based on ICP-MS. The percent Tc-99 removed with vacuum treatment of the barrier material and the D_f values are presented in Table 9. The average percent Tc-99 removed was less than 10%; which is not statistically significant. As presented in Table 8 also, there are no significant changes in uranium isotope concentrations in the post-vacuum treated barrier material. The post-vacuum treated barrier material uranium concentrations (U-235 and U-238) at, respectively, 1.23 ± 0.01 and 75.8 ± 0.7 pCi/g are not statistically different from their initial "as-received" concentrations in the barrier material, which were 1.27 ± 0.08 and 78.7 ± 6.2 pCi/g, respectively. Therefore, it can be concluded that there was no significant decontamination of both Tc-99 and uranium isotopes with flowing air treatments of the barrier material at 232 °C for 72 hours. There was a lot of scatter in the LSC data for Tc-99 post-flowing air treatments and so the LSC data was not considered useful (Appendix C).

Again, one runs into the same problem as in the vacuum test where the Tc-99 concentrations in the flowing air treated samples were not statistically different from the Tc-99 concentration in the "as-received" barrier material sample pieces. It is also worth noting that the question of Tc-99 cross

contamination during runs under the three test conditions is not an issue because new reactor vessels were used for every new test condition.

Table 8. Post-flowing air treatment analytical results for uranium isotopes and Tc-99 by ICP-MS

	*Initial [U], pCi/g	72-Hour Run-1, pCi/g	72-Hour Run-2, pCi/g	**Average,** pCi/g	**Std. Dev**
U-234	1.55E+01	1.40E+01	1.42E+01	**1.41E+01**	*1.23E-01*
U-235	1.27E+00	1.23E+00	1.24E+00	**1.23E+00**	*7.1E-03*
U-238	7.87E+01	7.53E+01	7.63E+01	**7.58E+01**	*7.13E-01*
Np-237	3.75E+00	< 1.60E+00	4.05E+00	**≤ 2.83E+00**	-
Tc-99	1.53E+03	1.35E+03	1.44E+03	**1.44E+03**	*6.36E+01*

*Single ICP-MS analysis of the "as-received" barrier for uranium isotopes.

Table 9. Post flowing air treatment analysis for Tc-99 by ICP-MS in Sample 23008-Mid (P3)

Run description	**[Tc-99] pCi/g; Post flowing air treatment**	**D_f**	**% Tc-99 removed**	**Comments**
Initial [Tc-99], pCi/g	1.53E+03	NA	NA	This was the average of three ICP-MS analysis results. Stand. deviation = 156 pCi/g
72-Hour Reactor-1	1.35E+03	1.13	11.7	D_f and percent removal not statistically significant based on analytical uncertainty.
72-Hour Reactor-2	1.44E+03	1.06	5.9	D_f and percent removal not statistically significant based on analytical uncertainty.
Average	**1.44E+03**	**1.10**	***8.8***	
Std. Dev.	*6.36E+01*	*0.05*	*4.2*	

5.8 Analytical Result Summary for All Test Conditions

In the course of this barrier material Tc-99 decontamination scoping test, the "as-received" barrier material (sample 23008-Mid) was analyzed before and after the tests for Tc-99 and uranium isotopes. Before the tests, two specimens of this material were digested and analyzed to obtain an average "as-received" baseline Tc-99 concentration. At the end of the tests, a final remaining piece of the "as-received" barrier material was digested and analyzed for Tc-99 (single digestion and analysis for Tc-99 by LSC and ICP-MS; uranium isotopes were also obtained from IC-MS analysis). In the pre-test sample, the LSC analyses measured an average of 1810 ± 185 pCi/g (average of the first two Tc-99 data points under LSC analysis for Tc-99 in the "as-received" sample as shown in Appendix C). Overall, the average LSC analysis data of the "as-received" barrier material was 1620 ± 382 pCi/g. This Tc-99 concentration average is based on the "as received" Tc-99 analytical results of 1,676, 1,937, 1,070 and 1797 pCi/g as summarized in Appendix C. This Tc-99 average concentration value of 1,620 ± 382 pCi/g in the "as-received" barrier material is the basis of the ± 2 sigma overlay plot in Figure 13 for Tc-99 analysis results by LSC method as well as the D_f and percent Tc-99 removal calculations.

In the case of Tc-99 based on ICP-MS, the initial analytical results for Tc-99 in the "as-received" material averaged 1620 ± 8 pCi/g (average of the first two Tc-99 data points under ICP-MS analysis for Tc-99 in the "as-received" sample as shown in Appendix C). The average Tc-99 result of 1,528 ± 156 pCi/g was based on ICP-MS analysis results of 1624, 1612 and 1348 pCi/g for the "as-received" samples pieces (Appendix C). This average Tc-99 concentration value of 1,528 ± 156 pCi/g is the basis of the ± 2 sigma overlay plot (Figure 14), D_f and percent Tc-99 removal for all analysis results by ICP-MS. There was a small difference in Tc-99 concentrations between the initial analysis result of the "as received material" at the beginning of the tests and the re-measurement at the end of the tests (see Tables 1, 3 and 6 and Appendix C).

It is worth noting that the storage and handling of the barrier materials were the same throughout the scoping tests. So, these differences cannot be attributed to handling or storage of the barrier materials. The differences in uranium concentrations, by ICP-MS, in the "as-received" sample analyses cited above are not significant as shown in Tables 4, 6 and 8. The differences in uranium concentrations between the initial analysis of the "as- received" sample and the measurement of the same sample at the end of all tests are within the 20%, two sigma, measurement uncertainty for uranium based on ICP-MS.

Figures 13, 14, 15 and 16 are overlay plots of average "as-received" Tc-99 by LSC, average "as-received" Tc-99 by ICP-MS, average uranium isotopes (U-235 and U-238) and their corresponding ± 2 sigma values for all test conditions (superheated steam, vacuum and flowing air), respectively. These overlay plots are summaries of the post-thermal treatment changes in concentrations for Tc-99 (LSC analytical data), Tc-99 (ICP-MS analytical data), U-235 and U-238 in comparison with their average individual initial concentrations in the original "as-received" barrier material sample and the 95% confidence intervals.

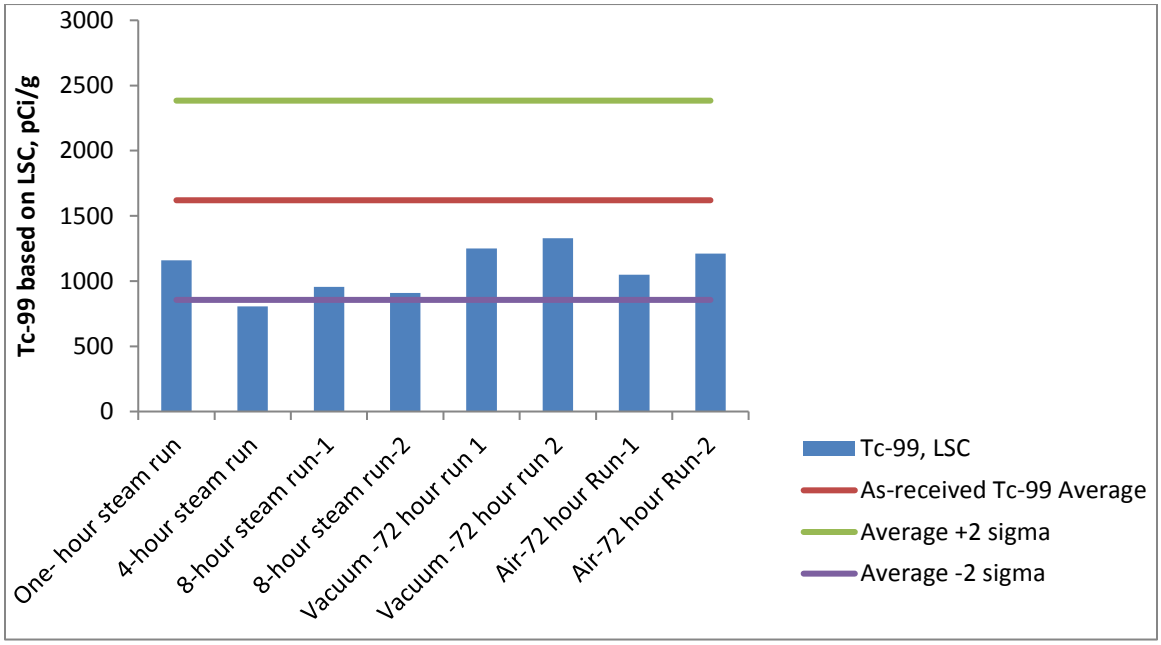


Figure 13. Overlay plots of average "as-received" Tc-99 concentration in the barrier material, ± 2 sigma values and post-thermal treatment Tc-99 concentration for all test conditions (LSC analysis).

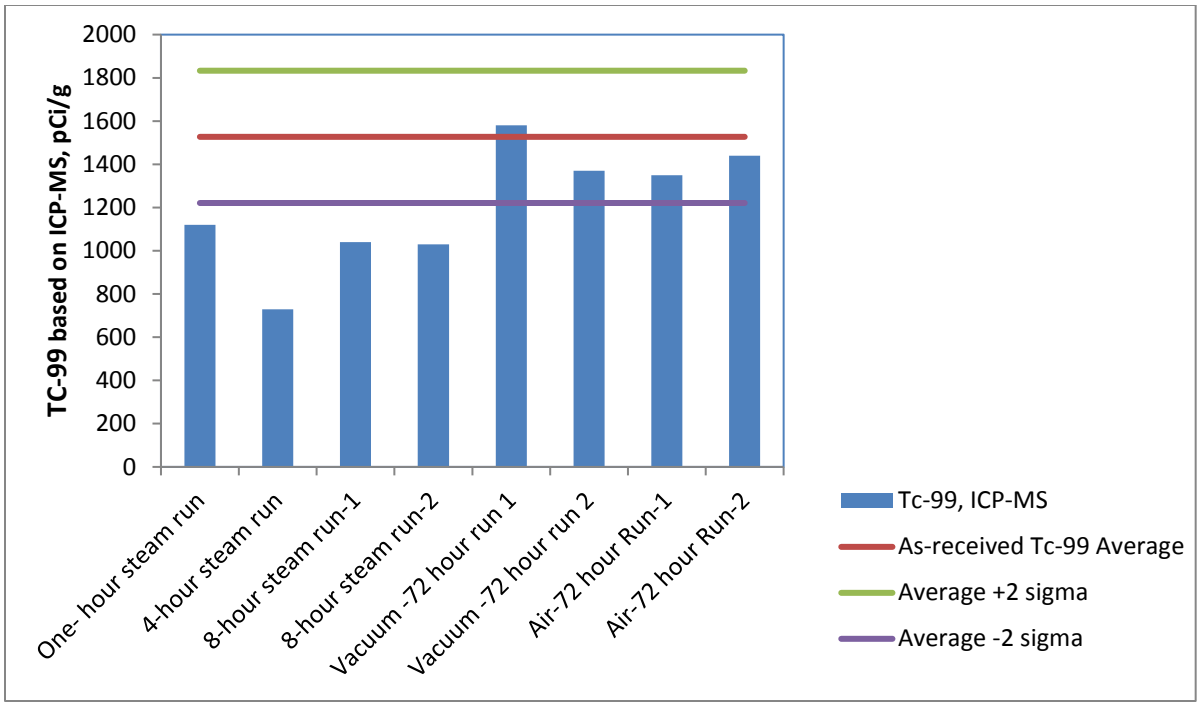


Figure 14. Overlay plots of average "as-received" Tc-99 concentration in the barrier material, ± 2 sigma values and post-thermal treatment Tc-99 concentration for all test conditions (ICP-MS)

The Tc-99 concentrations in the post-steam treatment of the barrier material, as presented in Figures 13, (data from Table 2 and Appendix C; analyses for Tc-99 by LSC), for the one-hour, 4-hour and duplicated 8-hour superheated steam treatments in comparison to the average Tc-99 concentration in the barrier material (1620 ± 764 pCi/g [± 2 sigma]) does not clearly show a 95 % confidence that Tc-99 was thermally desorbed from the barrier material based on steam treatment. The one hour post steam treatment and the duplicated 8-hour Tc-99 post steam treatment concentrations fall within the ± 2 sigma uncertainty region of the overlay plot in Figure 13. Only the 4-hour Tc-99 post steam treatment concentration is below the ± 2 sigma error bars; however, the duplicated 8 hour Tc-99 post steam treatments are close to the -2 sigma line. This was interpreted to indicate that there was Tc-99 removed from the barrier material during the 4-hour and possibly the duplicated 8-hour superheated steam treatments. Results from other test conditions (Vacuum and flowing air) as shown in Figure 13 do not show Tc-99 removal after vacuum and flowing air treatments at 232 °C for 72 hours. The Tc-99 concentrations in the vacuum and flowing air treated sample are statistically not different from the average Tc-99 concentration in the" as-received" sample.

However, the analytical results for the same set of post steam, vacuum and air flow thermally treated samples presented in Figure 13 from LSC analysis are presented again in overlay plots in Figure 14 for ICP-MS analysis for Tc-99 for the same set of samples (data from Tables 3, 7 and 9). In Figure 14 overlay plots, the one-hour, 4-hour and duplicated 8-hour superheated steam treatments in comparison to the average Tc-99 concentration in the barrier material (1528 ± 312 pCi/g [± 2 sigma]) does show a 95 % confidence that Tc-99 was thermally desorbed from the barrier material based on steam treatment. All the steam treated samples show a residual technetium concentration less than 1220 pCi/g (± 2 sigma) in the post-steam treated samples, which indicates that some Tc-99 was removed for the barrier material as a

result of superheated steam treatment relative to the Tc-99 concentration in the "as received" barrier material. Again, based on Figure 14 overlay plots, there is no appreciable Tc-99 removal after vacuum and flowing air thermal treatments at 232 °C for 72 hours.

Overall, the average Tc-99 remaining on the barrier material sample 23008-Mid after steam treatment comes to 960 ± 150 pCi/g based on LSC analysis and 980 ± 170 pCi/g based on Tc-99 analysis by ICP-MS (Tables 2 and 3). This gives a combined Tc-99 average concentration remaining on the barrier material with steam treatment of 970 ± 16 pCi/g of barrier material. To compare this barrier material Tc-99 decontamination scoping test results with the expected disposal WAC of 172 pCi/g of converter (172 pCi/g of waste material), a conversion factor, as provided by Paducah, was used [v]. When this conversion is made (Tc-99 concentration on a 000 size converter[3] basis), the result is 176 ± 29 pCi/g of converter (1 sigma). This WAC value of 176 pCi/g of converter is essentially the same as the Oak Ridge on-site disposal WAC of 172 pCi/g of waste[iv] when the analytical uncertainties are taken into consideration. Hence, it is concluded that superheated steam treatment of the barrier material at 232 °C has the potential to reduce the Tc-99 concentrations in the Paducah barrier material to levels comparable to both the Oak Ridge WAC and the yet to be developed Paducah Tc-99 WAC for on-site disposal.

The overlay plots in Figures 15 and 16 for post thermal treatments concentration changes in both U-235 and 238 concentrations show a remarkable similarity. All data used in Figures 15 and 16 overlay plots were presented earlier in Tables 4, 5, 6, 8 and summarized in Appendix C. For both isotopes, the steam treated samples show U-235 and U-238 concentrations well below their average concentrations in the "as-received" barrier material and are not within the ± 2 sigma uncertainty range in Figures 15 and 16. The average U-235 concentration in the "as-received" barrier material was 1.27 ± 0.16 (± 2 sigma uncertainty). The average, post steam treatment U-235 concentration was 0.762 ± 0.044 pCi/g which is not within the range of the average "as-received" U-235 concentration in the barrier material. Similarly, the average U-238 concentration in the post steam treated barrier materials was 47.0 ± 3.4 pCi/g, which is also smaller than the average U-238 concentration in the "as-received barrier material (78.7 ± 12.4 [± 2 sigma]. Therefore, the conclusion is made that steam treatment of the barrier material did remove both uranium isotopes. The concentrations of the uranium isotopes (U-235 and U-238) in the post vacuum and flowing air treated barrier material were about the same order of magnitude as their concentrations in the "as-received" barrier material. Therefore, in these thermal treatment options, vacuum and flowing air at 232 °C for 72 hours, these uranium isotopes were not removed from the barrier material in any measurable quantities. It is also worth noting that although other uranium isotopes like U-234 and U-236, including traces of Np-237, were identified in the ICP-MS analysis results for the "as-received" samples, these analytes were below the instrument detection limits in the thermally treated sample, especially the post steam treated samples.

[3] Paducah notations

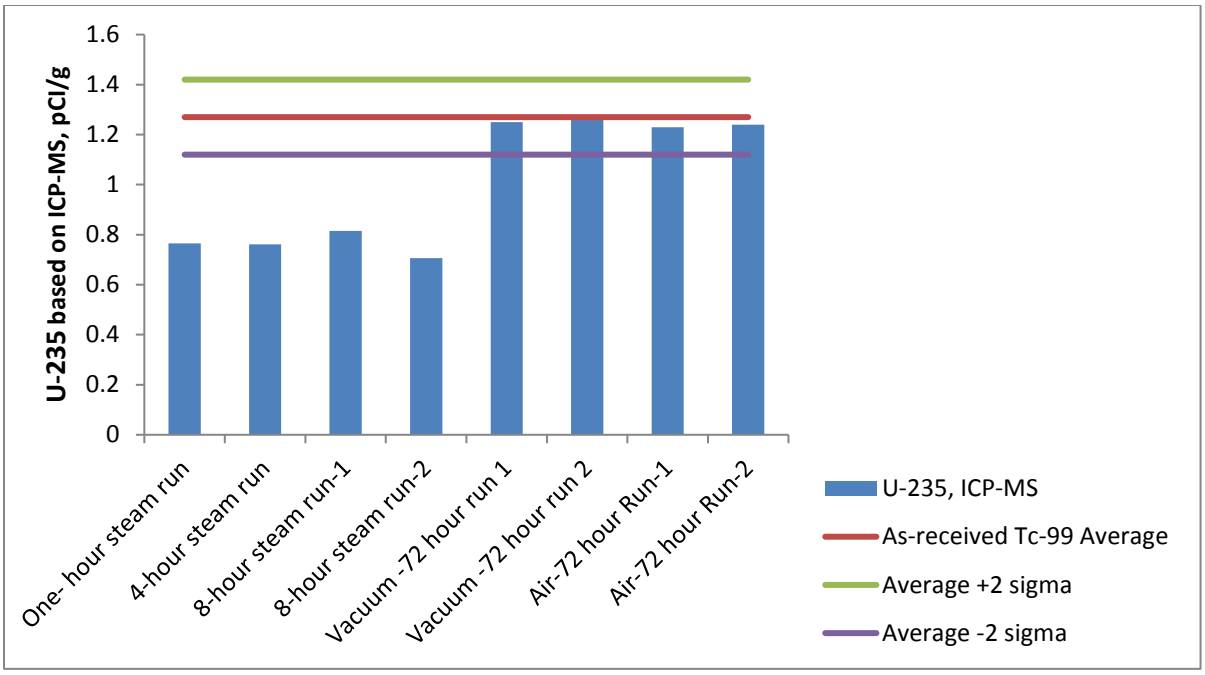


Figure 15. Overlay plots of average "as-received" U-235 concentration in the barrier material, ± 2 sigma values and post-thermal treatment U-235 concentration for all test conditions (ICP-MS analysis).

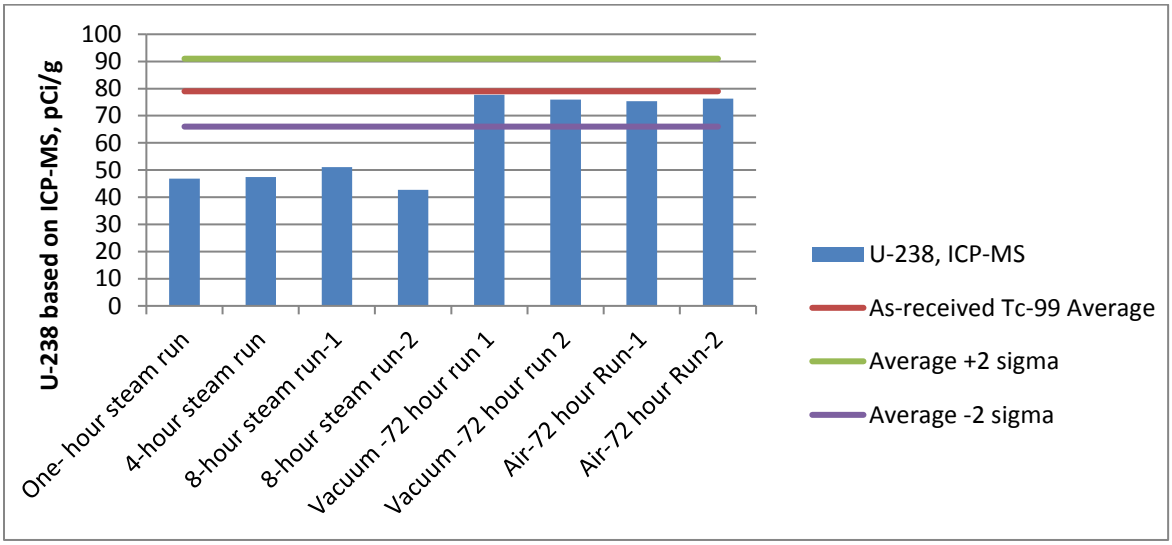


Figure 16. Overlay plot of average "as-received" U-238 concentration in the barrier material, ± 2sigma values and post-thermal treatment U-238 concentration for all test conditions (ICP-MS analysis).

The plots in Figures 17 and 18 shows, respectively, the percent Tc-99 and uranium isotopes (U-235 and U-238) removed from the superheated steam treatment of the barrier material as previously presented in Tables 2 to 5 and Appendix C. The analysis results for Tc-99 by both methods (LSC and ICP-MS) are comparable as shown in Figure 17, although the ICP-MS values were slightly lower in most cases. With

superheated steam treatment of the barrier material, an average of 41 ± 9% of the Tc-99 was removed based on LSC measurements and an average of 36 ± 11% Tc-99 was removal based on ICP-MS analysis. Based on both analytical technique results and average of 38 ± 10% Tc-99 was removed with steam treatment. An average of 40 ± 4 % of both U-235 and U-238 were removed. In summary, the conclusion is therefore made that steam treatment of the barrier material is more promising in the thermal Tc-99 and uranium isotope decontamination of the barrier material at 450 °F (232 °C).

It may be right to assume that superheated steam treated barrier material pieces showed promising results in the decontamination of Tc-99 and uranium isotopes from the barrier material because the moisture media provided by the presence of steam may have aided in the dissolution and desorption of a fraction of the Tc-99 and other uranium compounds in the barrier material. Of course, only those Tc-99 and uranium compounds in the barrier material which are easily dissolved or water soluble under this superheated steam conditions are removed from the barrier material with this thermal treatment.

Thermally treated barrier material in flowing air conditions did not show any measurable decontamination of Tc-99 or uranium isotopes because there may be no volatile Tc-99 or uranium compounds in the barrier material. On the other hand, Tc-99 or uranium compounds in the barrier materials which may be semi-volatile at that treatment temperature may react with oxygen in the flowing air and further be converted into fixed and non-volatile species which flowing air cannot transport out of the barrier material surface. Exposure of the "as-received" barrier materials during sample preparations and storage at both Paducah and SRNL to moisture laden ambient conditions may also affect the chemical behavior of both Tc-99 and uranium compounds in the barrier material.

The expected behavior of volatile and semi-volatile Tc-99 and uranium compounds under thermal conditions with vacuum was their desorption with ease from the barrier material because of the increase in their vapor pressure due to the vacuum condition. However, there was no evidence of the thermal desorption of Tc-99 or uranium isotopes in the "as-received" barrier material, which may support the notion that there may not be volatile Tc-99 or uranium species inside the "as-received" barrier material to begin with. On the other hand, volatile Tc-99 species, if at all they were present in the "as-received" barrier materials, under both vacuum and flowing air treatment at 232 °C may have been converted to oxides of Tc-99, especially the heptoxide (Tc_2O_7) whose boiling temperature, at 310.6 °C, is greater than the scoping test operating temperature of 232 °C. Therefore, the complete removal of Tc-99 and its related compounds, if so desired, would require the running of a scoping test at temperatures greater than 310.6 °C (591 °F).

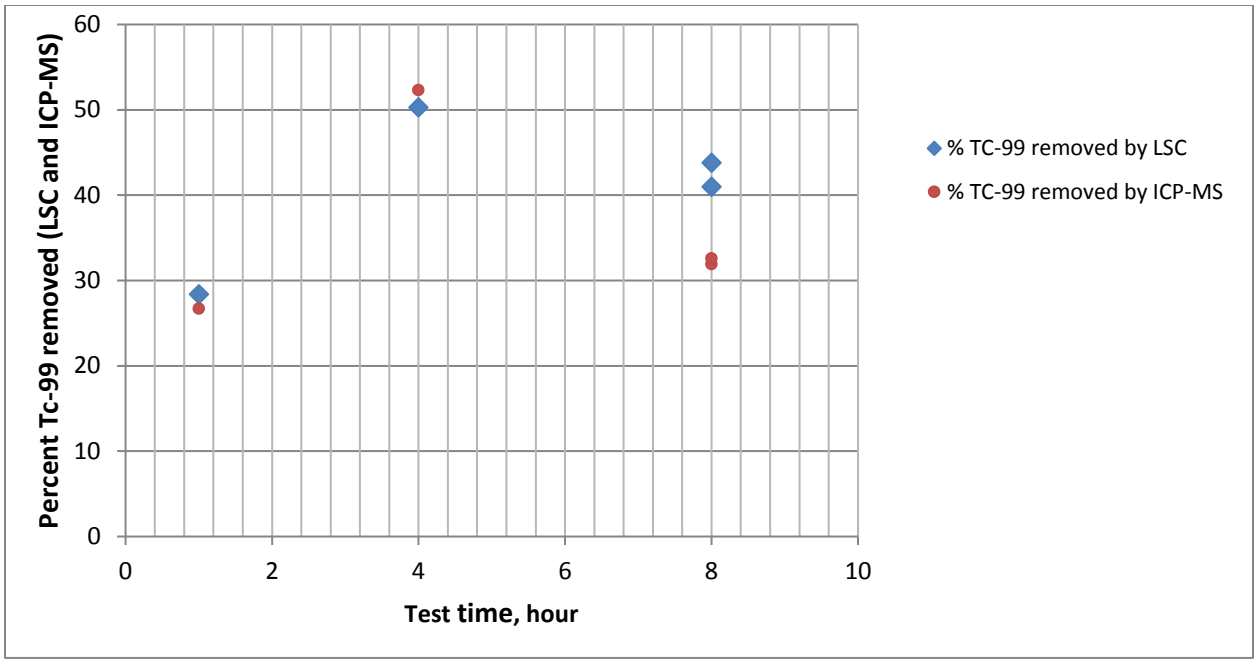


Figure 17. Percent technetium removed from steam testing

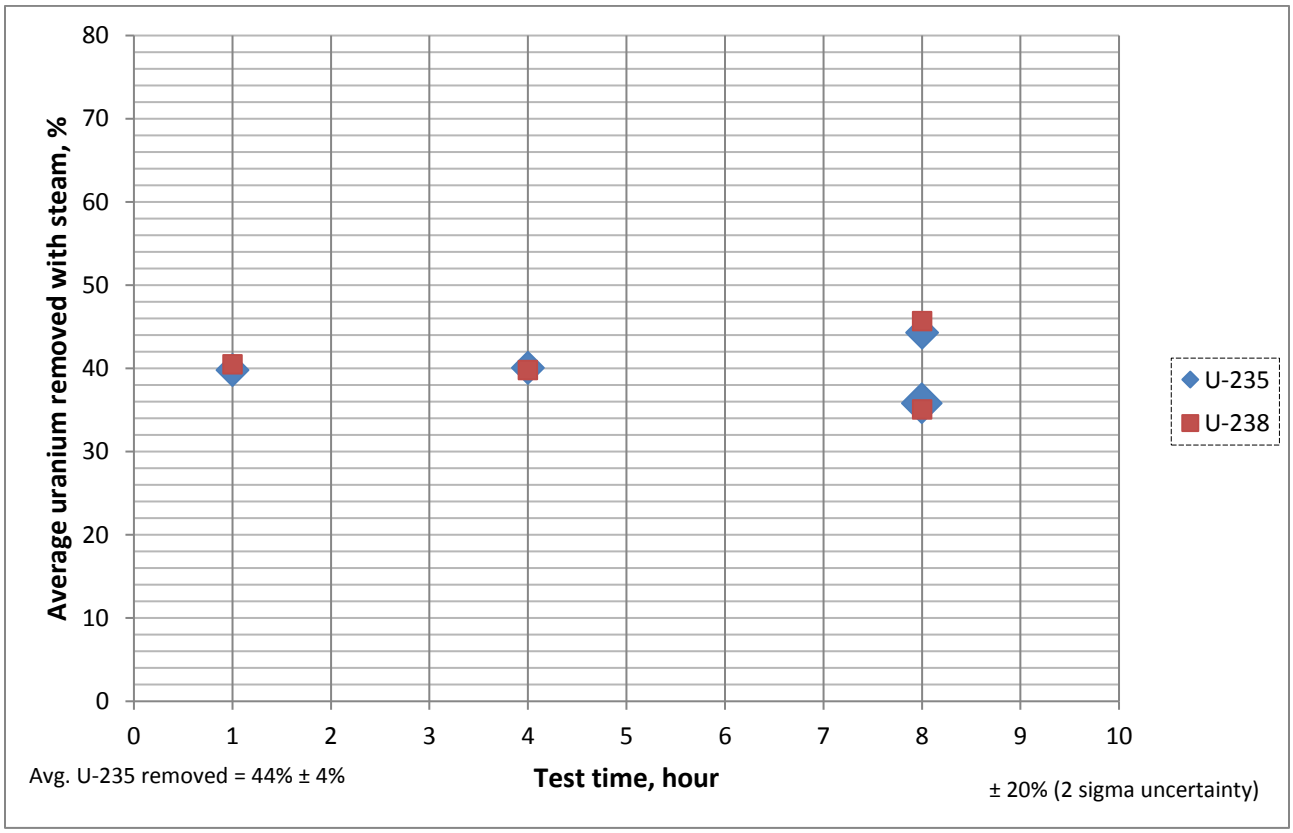


Figure 18. Percent uranium removed from steam testing

6.0 CONCLUSIONS and RECOMMENDATIONS

At best, it is perceived that the "as received" barrier materials sent to SRNL for the scoping tests contained traces of remaining volatile Tc-99 compounds and mostly Tc-99 compound in the oxidized Tc2O7 and reduced TcO2 forms, which are both non-water soluble and with their boiling temperatures greater than the scoping experimental thermal desorption temperature of 232 °C. This will seem to indicate that it will require greater efforts than thermal treatment at 232 °C to dislodge all the technetium compounds from the barrier materials to meet or exceed the waste acceptance criteria for disposal at Paducah.

The reason for no significant reduction in Tc-99 concentration in the barrier material with the vacuum and flowing air scoping tests may be due to a change in the Tc-99 chemistry. While in operation, the gaseous diffusion cascade was in a fluorinating chemistry environment. At shutdown, the equipment was purged to a UF6 negative, and pressured close to atmospheric pressure to minimize moist-air intrusion. It is also worth mentioning that prior to the sampling of barrier material for delivery to SRNL, the cell was probably still in a fluorinating environment and that even though the barrier material samples were promptly bagged at Paducah, the exposure to wet air had already started during the bagging process, and probably continued at SRNL where the samples were further exposed to wet air during sample preparations and storage. With passage of time and exposure to air and moisture as described above, the more volatile Tc-99 compounds such as pertechnetyl fluoride, TcO_3F and other fluorinated technetium compounds may have been converted to oxides (TcO_2, and TcO_7) of Tc-99. These Tc-99 oxides, especially the heptaoxide with boiling temperature [viii, ix] of 310.6 °C, will not under normal conditions be completely thermally dislodged at 232 °C; the maximum operating temperature for this scoping test. The lack of removal of Tc-99 compounds may indicate the necessity of establishing the fluorinating environment prior to heat treatments for effective removal. Additional studies with the fluorinated gases on barrier material before heat treatment could be pursued during Phase II activities.

In Phase I of this project, the "as-received" Paducah barrier material was exposed to three different atmospheric conditions, i.e., vacuum, flowing air and flowing superheated steam at varying periods of thermal decontaminations at 232 °C. The results from the scoping tests demonstrated that some of the Tc-99 was removed from the material (superheated steam treatment) while thermal treatments under both vacuum and flowing air showed no significant reduction.

Superheated steam treatment data of the barrier material show that both Tc-99 and uranium isotopes were removed from the barrier material within the first one to eight hour superheated steam/thermal treatment at 232 °C, with an averaged percent Tc-99 removal of 38 ± 10% and 40 ± 4% for uranium removal. The superheated steam/moisture may be interacting chemically with Tc-99/uranium compounds in the barrier material interior surface in a way that both vacuum and flowing air are not capable of doing, even after 72 hours of thermal treatment.

The target Tc-99 concentration after thermal desorption treatments under any of the test conditions was 172 pCi/g of converter; the proposed waste acceptance criteria (WAC) for on-site barrier material disposal at Paducah. While treatment with superheated steam showed the most promise, none of the scoping test results quite met or exceeded the WAC limit of 172 pCi/g of converter as expected, including the superheated steam test results. The average Tc-99 concentration remaining after steam treatment (970 ± 16 pCi/g barrier material) when converted to a Tc-99 concentration on converter basis is 176 ± 29 pCi/g of converter. This Tc-99 concentration per converter basis is about equal in magnitude to the proposed target on-site Paducah disposal WAC of 172 pCi/g waste. Hence, it is concluded that superheated steam treatment of the barrier material at 232 °C has the potential to reduce the Tc-99 concentrations in the Paducah barrier material to levels comparable to both the Oak Ridge WAC and the yet to be developed Paducah Tc-99 WAC for on-site disposal.

The original plan for phase 1 of this scoping test called for the analysis of the inorganic absorbents, leaching solutions and steam condensate to do a Tc-99 mass balance at the end each test condition if a significant Tc-99 D_f was obtained. It was not necessary to analyze the inorganic trap materials (MgF_2 and Al_2O_3), the leaching solutions or steam condensates to account for total desorbed Tc-99 because of the low decontamination factor obtained under these test conditions.

Therefore, the following recommendations are proposed:

- Optimization tests on thermal decontamination of both Tc-99 and uranium isotopes from the Paducah barrier materials should be performed with emphasis on superheated steam treatment.

- The barrier material should be mounted in the test reactor in such a manner as to enhance adequate contact between the interior of the barrier material and the flowing superheated steam. Such experimental setup will enhance thermal desorption/mass transfer of both Tc-99 and uranium isotopes out of the barrier material. For example, instead of putting the barrier material piece (s) under steam investigation anywhere inside the reactor chamber (steam bathing the sample), the sample piece (s) should be directly coupled or mounted at a known location inside the reactor chamber so that the superheated steam directly goes through and around it to enhance steam/surface interactions. Alternatively, cutting open the barrier material and exposing the interior surface area to the superheated steam may enhance the release of more volatile products including Tc-99 in its various forms.

- The chemical nature or speciation of the Tc-99 /technetium compounds in the barrier materials needs to be investigated along with determination of their distribution within the barrier material.

- The Tc-99 thermal decontamination of the Paducah barrier materials should also be performed at lower temperatures to meet Paducah plant operational capabilities.

- If optimization with superheated steam fails to attain technetium decontamination waste acceptance level of 172 pCi/g or lower, it is recommended that an examination of other techniques such as fluorinating agents, to convert the technetium compounds in the barrier material into more volatile technetium hexafluorides or other volatile technetium compounds should be considered.

7.0 QUALITY ASSURANCE

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

The Task Technical and Quality Assurance Plan (TTQAP) details the planned activities and associated quality assurance implementing procedures for the Tc-99 Decontamination of a Gaseous Diffusion Membrane - Phase I (TTQAP, SRNL-RP-2016-00180, Rev. 0, April 30, 2016) task. The documents referenced in the TTQAP include the following: Laboratory Notebook SRNL-NB-2016-00005. Various AD notebooks contain the analytical data.

8.0 REFERENCES

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APPENDIX A: Piping and instrument diagram: Superheated steam, Vacuum and Flowing Air setup.

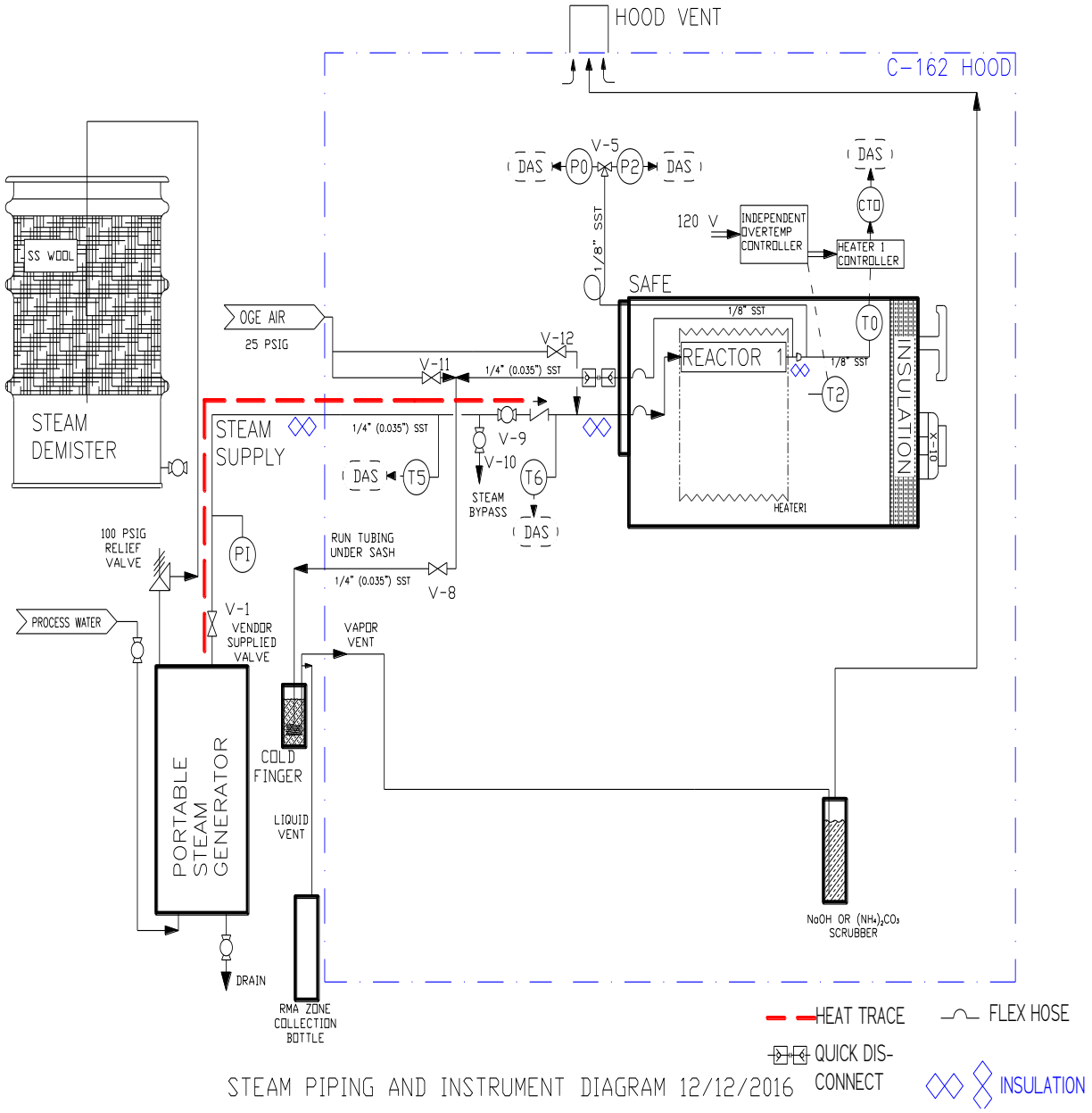

Figure 19. Steam test condition piping and instrument diagram for barrier material testing

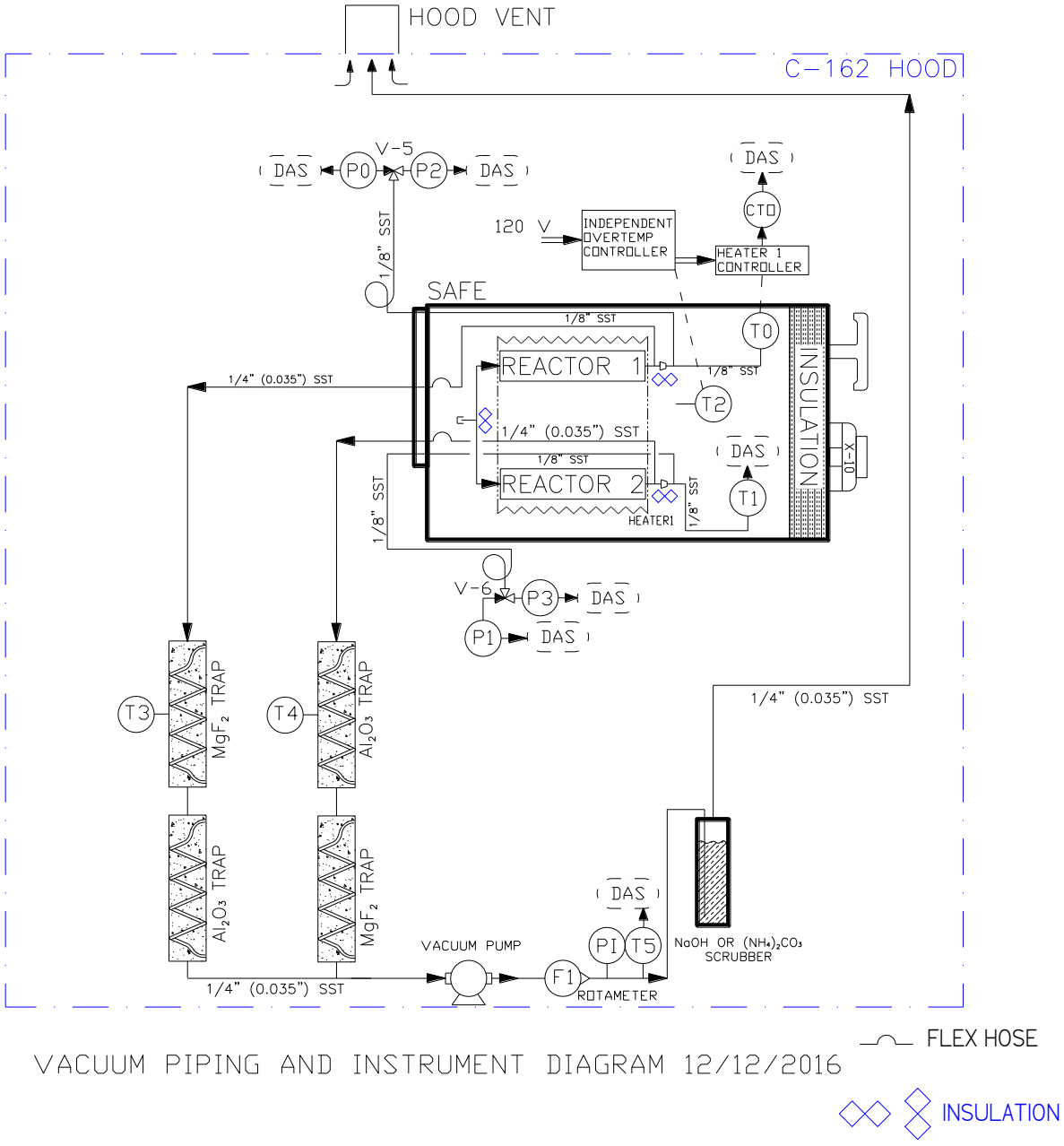

Figure 20. Vacuum test condition piping and instrument diagram for barrier material testing

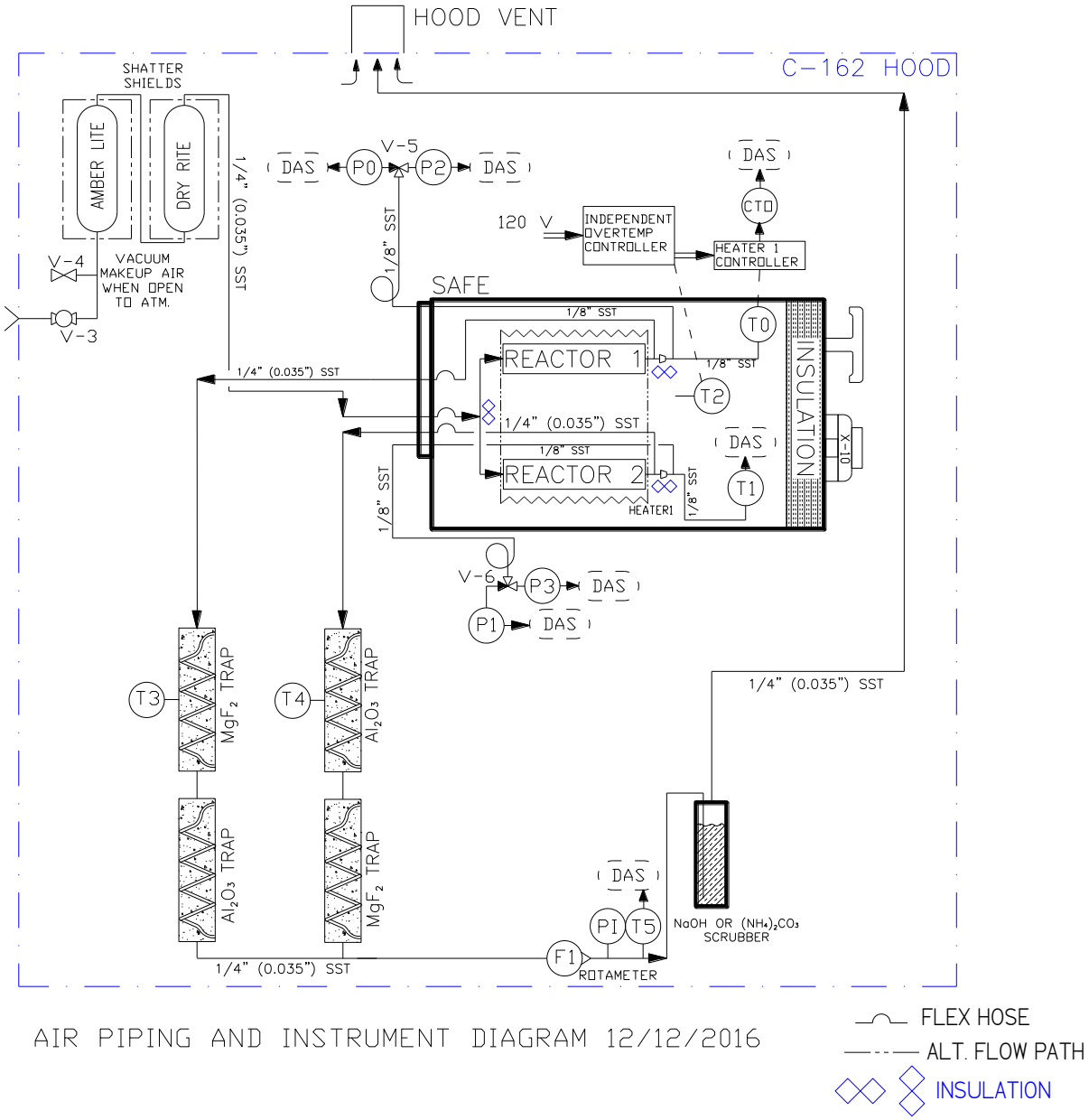

Figure 21. Flowing air piping and instrument diagram for barrier material testing

APPENDIX B: Data Acquisition System Set Up (Outside the Radiological Hood)

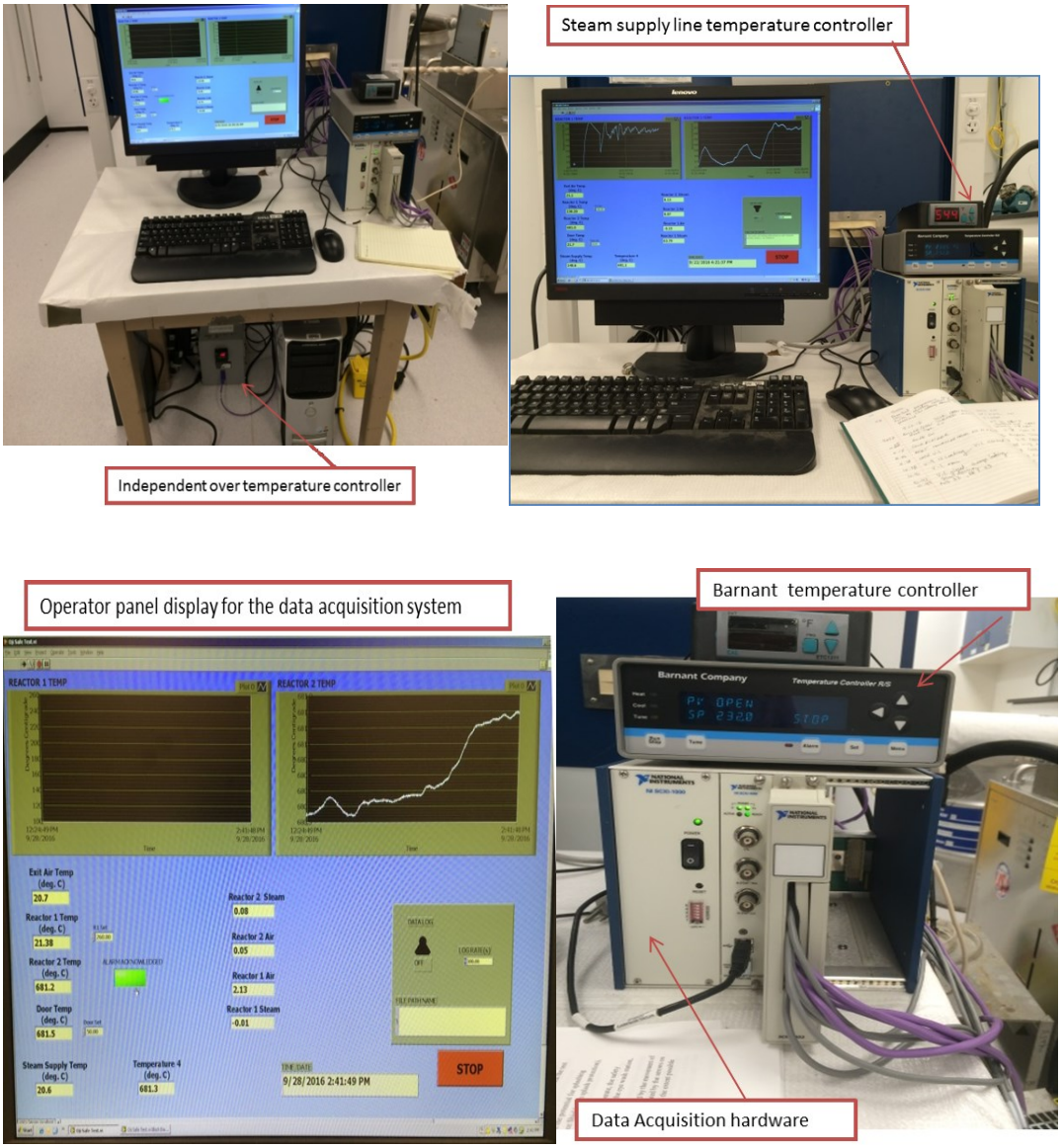

Figure 22. Various Photos of the Test Rig, including the Data Acquisition System

APPENDIX C: Summary of Analytical Results: Sample 23008-Mid

Sample ID/Test Conditions	Tc-99 by LSC, pCi/g	Tc-99 by ICP-MS, pCi/g	U-238 by ICP-MS pCi/g	U-235 by ICP-MS pCi/g	Comments
T0-1 (as-received)	1676	1624	81.4	1.31	One of the duplicate analysis results at the beginning of tests.
T0-2 (as-received)	1937	1612	83.0	1.31	One of the duplicate analysis results at the beginning of tests.
T0-3 (as-received)	1070	1348	71.6	1.18	Single analysis result at the end of the tests.
T0-4 (as-received)	1797	Not analyzed	Not analyzed	Not analyzed	Single re-analysis results at the end of the tests.
Average	**1620**	**1528**	**78.7**	**1.27**	
STDEV.	± *382*	± *156*	± *6.2*	± *0.08*	
2-Sigma Plus	2384	1840	91.1	1.42	Average +2σ
2-Sigma Minus	856	1216	66.3	1.12	Average -2σ
Test Conditions					
One- hour steam run	1160	1120	46.8	0.765	
4-hour steam run	806	729	47.4	0.761	
8-hour steam run-1	955	1040	51.1	0.815	
8-hour steam run-2	910	1030	42.7	0.707	
Vacuum -72 hour run 1	1250	1580	77.7	1.25	
Vacuum -72 hour run 2	1330	1370	76.0	1.26	
Air-72 hour Run-1	1050	1350	75.3	1.23	
Air-72 hour Run-2	1210	1440	76.3	1.24	

APPENDIX D: General Equipment list

The following is a list of the equipment/instrumentation used for the data acquisition:

Channel Name	Description	M&TE Number	Range
T0	Reactor 1 Temperature	TR-40080	0-300 °C
T1	Reactor 2 Temperature	TR-01518	0-300 °C
T2	Security Safe Internal Temperature	N/A	0-300 °C
T3	Reactor 1 Trap Temperature	N/A	0-300 °C
T4	Reactor 2 Trap Temperature	N/A	0-300 °C
T5	Rotameter Temperature	TR-01516	0-100 °C
T6	Steam Inlet Temperature	TR-40011	0-300 °C
T7	Security Safe Door Lock Temperature	TR-02827	0-300 °C
P0	Reactor 1 Steam Pressure	TR-40019	0-150 PSIG
P1	Reactor 2 Steam Pressure	TR-40018	0-150 PSIG
P2	Reactor 1 Air/Vacuum Pressure	TR-03498	0-30 PSID (Low/High Pressure Taps)
P3	Reactor 2 Air/Vacuum Pressure	TR-03745	0-30 PSID (Low/High Pressure Taps)
PI	Rotameter Pressure	TR-03788	0-15 PSIG/0-30 in Hg
PI-SG	Steam Generator Pressure	TR-03801	0-200 PSIG
FI	Flow Rotameter	TR-40284	0-2 SCFH
	Over-Temperature Controller, Custom	786-A-MLR-OTC-1	0-100 °C
	Heater Controller, Barnant Temp Controller R/S	N/A	0-300 °C
	Steam Generator, Electro Steam LG-10	N/A	0-500 °C, 0-85 PSIG
	Steam Inlet Heater, Briskheat ETC1311	N/A	0-800 °C
	Neslab CC-65 cold Finger (Cryotro)	N/A	

Data Acquisition hardware

Dell 9200 Tower running Windows™ XP

National Instruments™ Hardware

SCXI-1000 4-Slot Chassis

SCXI-1600 USB Data Acquisition and Control Module

SCXI-1102 with 1303 Terminal Block, Signal Conditioner for thermocouples and 4-20 mA Instruments

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