

Test Plan to Demonstrate Removal of Iodine and Tritium from Simulated Nuclear Fuel Recycle Plant Off-Gas Streams Using Adsorption Processes



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Nuclear Security & Isotope Technology

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Objective

The objective of this test plan is to describe research that will determine the effectiveness of silver mordenite and molecular sieve beds to remove iodine and water (tritium) from off-gas streams arising from used nuclear fuel recycling processes, and to demonstrate that the iodine and water can be recovered separately from one another.

Background

The need to control emissions of the gaseous radionuclides to the air during nuclear fuel recycling is widely recognized. Gaseous radionuclides can partition to off-gas streams emanating from the various individual processes used in the recycle plant, for example, from the mechanical head end (shear), fuel dissolver, vessel vents, cell ventilation, and melting operations. In traditional recycle plants the bulk of the volatile fission products (i.e., Kr, Xe, I, and H) are released during the dissolution process. The noble gases report almost quantitatively to the dissolver off-gas stream. About 95% of the iodine partitions to the dissolver off-gas stream. Tritium is retained in the aqueous dissolver solution as tritiated water and enters the dissolver off-gas stream as a small fraction of the water vapor rising from the solution. Most of the water vapor is returned to the process using a condenser, but nevertheless some water vapor escapes into the off-gas system. Dissolver off-gas is also accompanied by nitrogen oxides and nitric acid vapors. Because of the corrosive nature of these gases and vapors, it has been proposed that silver substituted mordenite may be used for iodine removal because mordenite is resistant to degradation by these oxy-acids. Tritium is allowed to accumulate in the plant water systems until the concentration is such that the water vapor escaping the plant causes release limits to be reached. The plant water may then be replaced and the contaminated water appropriately treated and disposed. Other techniques for tritium disposal, such as water feed-and-bleed, can be implemented but all involve disposing of significant amounts of water that must be immobilized for decades.

A seriously considered variation on traditional fuel recycling includes the addition of a tritium removal step between the shearing and dissolution processes. Introduction of the tritium into the plant water systems is thus avoided. The tritium removal step involves oxidizing the UO_2 fuel matrix to U_3O_8 at elevated temperatures (e.g., 500°C), which releases all the tritium and radiologically significant fractions of the noble gases and iodine. This process was formerly called voloxidation. The off-gas from the process contains tritium in higher concentrations than the off-gas from the dissolver, but it also contains iodine. Water may be efficiently removed from off-gas streams using molecular sieves, specifically of the 3A type (3AMS). The presence of iodine is a complicating factor because it can coadsorb on the molecular sieve, elevating the low-level tritium waste to a high level waste. Iodine could be removed first with silver mordenite (AgZ), but studies have shown that the mordenite coadsorbs water, which can be released in the high temperature processes used to convert the mordenite to a suitable waste form.

Based on the qualitative description of the off-gases arising from dissolution and the process formerly known as voloxidation, we assert that the most likely need for a system for removing iodine and tritiated water from an off-gas stream and separate these radionuclides for cost effective disposal is the process formerly known as voloxidation. A basis for, and estimate of, the iodine and water concentrations in such an off-gas stream is provided in Appendix A. Because of the low water concentrations that arise from hydrogen isotopes evolved from the fuel, addition of water vapor via the process feed gases (or elsewhere before sorption) is required to obtain both measureable changes in water concentration across the

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molecular sieve bed and acceptable decontamination factors for water (tritium). Such considerations are discussed in Appendix B.

The characteristics of the co-adsorption and desorption of iodine and water using the sorbents (i.e., silver mordenite and molecular sieve) make possible an operating sequence that recovers these off-gas contaminants in forms separate from one-another. It involves placing the AgZ sorbent upstream of the 3AMS. Further, when breakthrough of iodine occurs in the AgZ column, a second column is placed on-line that accepts both the process off-gas and a low flow rate purge stream from the first column which is heated to remove physisorbed iodine. The cycle repeats with respect to the AgZ column. Essentially no iodine reaches the 3AMS column, which would be regenerated for reuse while a companion 3AMS column goes on-line.

Data Gap Analysis

In testing conducted over the previous years, several important questions were raised regarding the use of reduced silver mordenite (AgZ) and type 3A molecular sieve (3AMS) beds to sequester iodine and water separately.

First, the breakthrough time and mass transfer zone length for iodine co-loaded with water onto a silver mordenite bed has yet to be completely determined. This test plan will begin by conducting a few brief tests to fill-in this knowledge gap.

Second, testing on deep beds of mordenite showed that a desorption phase following co-loading of iodine and water resulted in extended periods of iodine release from the bed. The quantity of this release appears to be impacted by the temperature of desorption. This test plan aims to select a recommended desorption temperature and quantify the total iodine released from the bed at that condition.

Third, no work has yet been conducted to determine the ability of 3AMS to tolerate multiple capture and regeneration cycles. This test plan will evaluate whether multiple capture/regeneration cycles causes any of the following to occur: a) decrease in water capacity, b) lengthened mass transfer zone, and c) residual contaminant build-up in the capture material.

Finally, after completing testing to address the three listed knowledge gaps, a demonstration of iodine and water (tritium) capture will be performed using beds of silver mordenite and molecular sieve to collect iodine and water separately. This demonstration will be performed under prototypical conditions that might be expected in an off-gas capture system such as would be required for the oxidative pretreatment (the process formerly known as voloxidation) of used nuclear fuel. It will provide assurance that there is sufficiently low cross-contamination of the beds to meet two objectives: (1) iodine does not elevate the low-level tritium waste stream to a high level waste stream and (2) there is insufficient tritium in the iodine-containing mordenite sorbent that high temperature processing of the mordenite to a waste form cannot release enough tritium to exceed the overall tritium release limit of the plant. The tests are designed to demonstrate a system that can achieve the decontamination factors (D_F) required in future plant designs.

Test Plan

A conceptual design for the test system is illustrated in Figure 1. A potential arrangement to use an iodine back-stop in AgZ columns is illustrated in Figure 2. General requirements for the test system are given in Appendix C.

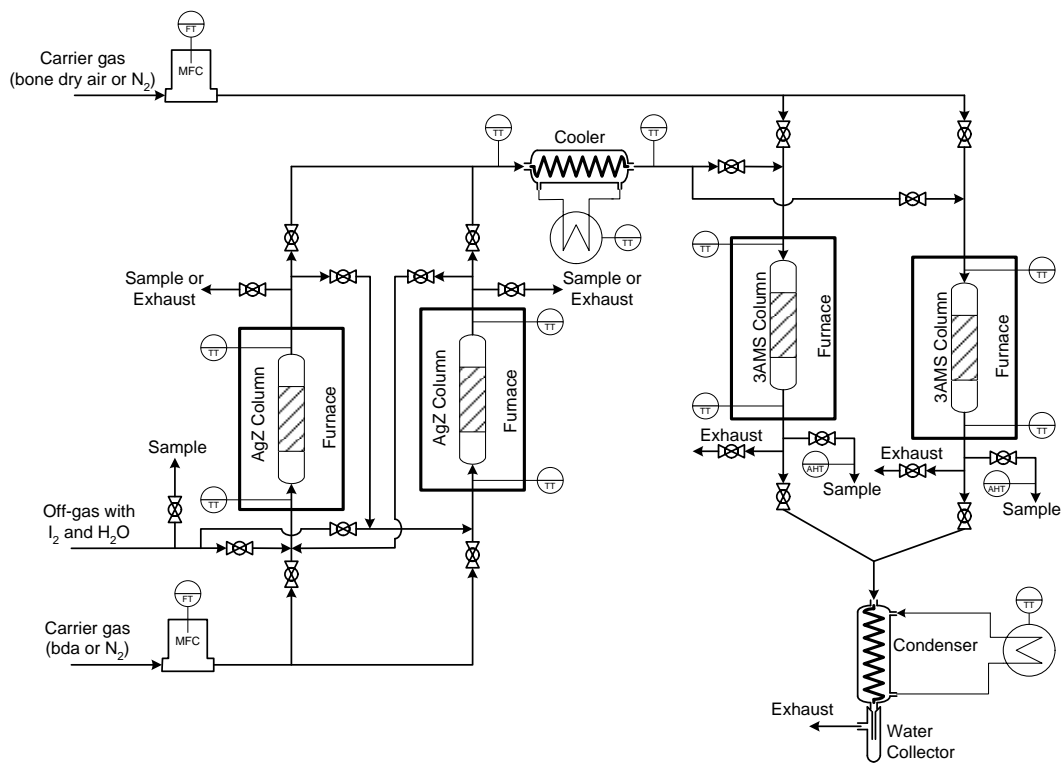


Figure 1. Concept of flow pattern for iodine and water adsorption train.

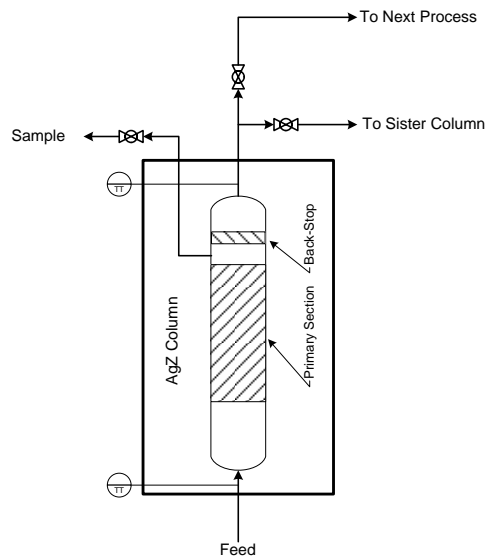


Figure 2. Concept of adsorption column with backstop section.

Phase 1: Measure iodine breakthrough time and length of mass transfer zone at operating conditions.

Load bed of AgZ at operating conditions. Obtain breakthrough curve by analysis of effluent stream I₂ concentrations. Test complete when effluent [I₂] = inlet [I₂]. [Conduct test twice for quality assurance.]

Phase 2: Determine the recommended desorption temperature for iodine bed.

Load ½-size AgZ bed with I₂ at set operating conditions. Load column until the break point is reached, as monitored by a starch test. Flow dry air at ½ loading velocity through column and increase temperature to 250°C [Run 1], 400°C [Run 2] and 550°C [Run 3]. Effluent is directed through a small second bed of AgZ for capture of any desorbed iodine. Periodically, the effluent will be tested to determine if iodine is still present. Once the iodine is fully desorbed from the bed [i.e. no iodine is detected in the effluent] the second AgZ bed will be homogenized and analyzed for iodine by neutron activation analysis (NAA), thus quantifying the amount of iodine desorbed. The first AgZ bed will also be analyzed for iodine retention by NAA.

Phase 3: Verifying the regeneration behavior of 3AMS.

Load bed of 3AMS with moist air to break point. Regenerate at 270°C under low air flow while monitoring effluent stream with hygrometer and condensing water in cold trap. Verify time required for regeneration and quantify water released during regeneration. [Conduct test twice.]

Phase 4: Integrated testing.

Bed 1a: 5x1 AgZ

Bed 1b: 2x1 AgZ

Bed 2a: 5x1 AgZ

Bed 2b: 2x1 AgZ

Bed 3: 3AMS

Bed 4: 3AMS

Load Bed 1a with iodine under selected operating conditions until break point is observed through use of starch test. Upon observation of break point, Bed 2 will be placed in line for primary iodine capture. Bed 1a and 1b will be held at the selected desorption temperature under low air flow until desorption is complete. Effluent from 1a and 1b will be combined into Bed 2 inlet stream. This design allows for no aging of Bed 2 prior to becoming the primary iodine capture bed. The majority of iodine desorbed from Bed 1a will be captured by Bed 1b, allowing the Bed 2 inlet to have inlet iodine concentrations similar to those experienced by Bed 1.

3AMS columns will be online for the duration of this test. It is expected that they will require multiple regeneration cycles during this length of test. As the regeneration time is expected to be less than the breakthrough time, one column will always be in line while a second column is being regenerated. The effluent from regeneration will be condensed in a cold trap, allowing quantification of the water desorbed from each 3AMS bed and quantification of any residual iodine desorbed. Other contaminants, if any,

may be identified. Effluent from the inline bed will be continuously run through a scrubber and this scrubber will be periodically analyzed for the presence of iodine. The test is considered complete upon completion of desorption for Bed 1. This test will be conducted 2–3 times.

Final Data Obtained:

- 1) Iodine captured on Bed 1
- 2) Iodine desorbed from Bed 1
- 3) Iodine presence on Bed 3 and 4
- 4) Remaining water on Bed 1
- 5) Regeneration characteristics of Bed 3 and 4 [residual iodine, residual water capacity, contaminants in cold trap]
- 6) Estimated iodine breakthrough time/mass transfer zone at operating conditions
- 7) Recommended temperature and duration for desorption of AgZ bed

Appendix A. Estimated Concentrations of Iodine and Tritium in Voloxidizer Off-Gas

The process formerly known as voloxidation is based on the oxidation of used nuclear fuel. Density changes that occur, when the fuel is oxidized from UO_2 to U_3O_8 , restructures the fuel from a monolithic solid to a fine powder, and various volatile fission products are released to the gas phase. The most volatile isotopes released from the fuel are listed in Table A.1. Also shown in the table are the amounts of each isotope calculated to be present in fuel irradiated to 55 GWd/MTIHM and cooled 5 years; the calculations were performed using the ORIGEN code.

Table A.1. Quantities of selected volatile gases in typical used fuel^a

Isotope	Activation products		Fission products		Activation + fission products	
	(g/MTIHM)	(Ci/MTIHM)	(g/MTIHM)	(Ci/MTIHM)	(g/MTIHM)	(Ci/MTIHM)
¹ H	1.35E-2	0	0	0	1.35E-2	0
² H	1.32E-5	0	0	0	1.32E-5	0
³ H	2.19E-2	2.11E+2	6.84E-2	6.60E+2	9.03E-2	8.71E+2
¹² C	8.83E+1	0	0	0	8.83E+1	0
¹³ C	1.15E+1	0	0	0	1.15E+1	0
¹⁴ C	1.66E-1	7.40E-1	4.38E-5	1.95E-4	1.66E-1	7.40E-1
³⁵ Cl	3.53E+0	0	0	0	3.53E+0	0
³⁶ Cl	4.42E-1	4.46E-2	0	0	4.42E-1	4.46E-2
³⁷ Cl	1.34E+0	0	0	0	1.34E+0	0
⁷⁹ Br	0	0	6.00E-4	0	6.00E-4	0
⁸¹ Br	0	0	3.36E+1	0	3.36E+1	0
⁸⁰ Kr	0	0	1.01E-4	0	1.01E-4	0
⁸¹ Kr	0	0	2.20E-5	4.63E-7	2.20E-5	4.63E-7
⁸² Kr	0	0	2.00E+0	0	2.00E+0	0
⁸³ Kr	0	0	6.08E+1	0	6.08E+1	0
⁸⁴ Kr	0	0	1.79E+2	0	1.79E+2	0
⁸⁵ Kr	0	0	2.96E+1	1.16E4	2.96E+1	1.16E+4
⁸⁶ Kr	0	0	3.26E+2	0	3.26E+2	0
¹²⁷ I	2.58E-7	0	7.33E+1	0	7.33E+1	0
¹²⁹ I	7.24E-13	1.28E-16	2.35E+2	4.15E-2	2.35E+2	4.15E-2
¹²⁸ Xe	1.05E-8	0	6.15E+0	0	6.15E+0	0
¹²⁹ Xe	0	0	4.64E-2	0	4.64E-2	0
¹³⁰ Xe	0	0	2.16E+1	0	2.16E+1	0
¹³¹ Xe	0	0	6.01E+2	0	6.01E+2	0
¹³² Xe	0	0	1.91E+3	0	1.91E+3	0
¹³⁴ Xe	0	0	2.52E+3	0	2.52E+3	0
¹³⁶ Xe	0	0	3.79E+3	0	3.79E+3	0

^aOak Ridge National Laboratory (ORNL) Isotope Generation and Depletion Code (ORIGEN) calculation based on pressurized water reactor (PWR) fuel at a burnup of 55 GWd/MTIHM and cooling time of 5 years.

An engineering analysis of the standard voloxidation process has been documented, (Spencer and DelCul, 2010), and may serve as a starting point for estimation of iodine and tritium concentrations in voloxidizer off-gas. The most typically cited implementation of the voloxidation process involves the use of a rotary calciner. Rotary seals on calciners are expected to leak under pressure or vacuum, so the voloxidizer would be operated at pressures slightly lower than its surroundings (i.e. cell atmosphere) to ensure that leakages enter the voloxidizer instead of flowing out into the processing cell. Air or oxygen for the standard process must be supplied to sustain the oxidation reaction. The amount that must be supplied depends on fractional oxygen usage, the composition of the feed gas (e.g., air or oxygen), and the composition of the cell atmosphere leaking into the system. Inleakage rates have been estimated and gas balances for three potential operational conditions using a calciner designed to process 500 MTIHM/yr have been completed (Spencer and DelCul, 2010). The basis for these calculations assumes 200 operational days per year and 24 hr/day operation. The case definitions and results are shown in Table A.2.

Table A.2. Gas balance for base case voloxidizer unit capable of processing 500 MTIHM/yr.^a

Stream	Flow rate, scfm		
	Case 1 ^b	Case 2 ^c	Case 3 ^d
Feed Gas			
Oxygen	3.44	3.09	3.93
Nitrogen	12.96	0	0
Inleakage			
Oxygen	0.42	0.42	0
Nitrogen	1.58	1.58	2.00
Effluent			
Oxygen	1.93	1.58	2.00
Nitrogen	14.54	1.58	2.00

^aVoloxidizer processing 118.2 kg/h of UO₂ requires 1.93 scfm (54.7 slpm) of O₂ for reaction.

^bVoloxidizer in cell with air atmosphere and air feed, oxygen usage 50%.

^cVoloxidizer in cell with air atmosphere and oxygen feed, effluent oxygen concentration 50%.

^dVoloxidizer in cell with nitrogen atmosphere and oxygen feed, effluent oxygen concentration 50%.

The total flow rate of effluent gases from the voloxidizer are the residual feed and inleaking gases (shown in Table A.2) and the gases released from the fuel. The amount released from the fuel is negligible in terms of total volume, but the specific activity can be quite large. To estimate the concentrations of tritium and iodine in the effluent stream, it is necessary to have some data on the fraction of these elements that are released from the fuel. The fractional release of iodine covers a range of values and there is considerable scatter in the data available in the literature. Some examples are:

- Voloxidation at a temperature of 480°C and a 4 h processing time removes approximately 1% of iodine from the fuel (Goode et al., 1973; Goode and Stacy, 1978).
- Oxidation of the fuel at about 490°C under a minimum of 20% v/v oxygen for 4 h released 0.3% to 7.9% of the iodine depending on fuel type and burnup (Stone and Johnson, 1978).
- Oxidation of CANDU fuel in air for 100 min at 1100°C released 90% of the iodine (Iglesias et al., 1986).

Based on these data, it is reasonable to expect that voloxidation between 500°C and 600°C would release between 1% and 5% of the iodine. A value of 1% will be used as a basis of calculation. In addition, the same fractional removal will be assumed for chlorine and bromine found in the fuel (although that is a poor assumption for the behavior of chlorine).

Under the range of conditions described above for iodine release, tritium release ranged from 90% to 100%. As a basis for calculation, 100% removal of tritium will be used.

Using the information in Tables A.1 and A.2, the concentration of selected elements in voloxidizer off-gas were calculated. The results are given in Table A.3

Table A.3. Estimated concentrations of water and halides in voloxidizer off-gas when used fuel is only source of indicated element.^a

Species	Concentration (ppmv)		
	Case 1	Case 2	Case 3
H as H ₂ O	1.82	9.45	7.47
Cl as Cl ₂	0.06	0.32	0.26
Br as Br ₂	0.17	0.90	0.71
I as I ₂	1.00	5.21	4.12

^a Removal of halides is assumed to be 1% in this table; were removal as high as 5%, multiply entry for halides in this table by 5.

1. Goode, J. H., R. D. Baybarz, S. D. Clinton, L. G. Farrar, C. L. Fitzgerald, R. G. Haire, V. J. Tennery, V. C. A. Vaughen, and C. D. Watson. 1973. *Voloxidation—Removal of Volatile Fission Products from Spent LMFBR Fuels*. ORNL-TM-3723, Union Carbide Corporation, Oak Ridge National Laboratory, Oak Ridge, Tenn., January (Applied Technology).
2. Goode, J. H. and R. G. Stacy. 1978. *Head-end Reprocessing Studies with H. B. Robinson 2 Fuel*. ORNL/TM-6037, Oak Ridge National Laboratory, Oak Ridge, Tenn., June (Applied Technology).
3. Spencer, B. B. and G. D. DeCul. 2010. *Engineering Analysis of Voloxidation Process*. ORNL/TM-2009/208, UT—Battelle LLC, Oak Ridge National Laboratory, Oak Ridge, Tenn., July (Applied Technology).
4. Stone, J. A. and D. R. Johnson. 1978. *Measurement of Radioactive Gaseous Effluents from Voloxidation and Dissolution of Spent Nuclear Fuel*. DP-1S-78-7. Savannah River Laboratory, Aiken, SC, August.
5. Iglesias, F. C., et al. 1986. “UO₂ Oxidation and Fission Product Release,” *Proceedings International Conference on CANDU Fuel*, October 6–8.

Appendix B. Achieving Required Decontamination Factor For Tritium Capture Process

Sequester of tritium from voloxidizer off-gas streams is required to meet regulatory discharge limits. The tritium will be in the form of water and will be diluted with water containing other isotopes of hydrogen. The fractional removal of tritium to meet regulations is the same fraction of all water that must be removed from the off-gas stream. If the decontamination factor, D_F , is defined as the ratio of the amount of water entering the removal process to the amount of water leaving the removal process, then,

$$D_F = \frac{x\dot{m}_{in}}{y\dot{m}_{out}} \quad (1)$$

where x = concentration of water in inlet, mol/mol,

\dot{m}_{in} = inlet flow rate, mol/time,

y = concentration of water in outlet, mol/mol, and

\dot{m}_{out} = outlet flow rate, mol/time.

A water balance may be written

$$\dot{w} = x\dot{m}_{in} - y\dot{m}_{out} \quad , \quad (2)$$

where \dot{w} = water removal rate, mol/time.

And an overall material balance is

$$\dot{m}_{in} = \dot{m}_{out} + \dot{w} \quad . \quad (3)$$

The outlet flow rate and concentration are calculated from

$$\dot{m}_{out} = \dot{m}_{in} \left[1 - x \left(1 - \frac{1}{D_F} \right) \right] \quad \text{and} \quad (4)$$

$$y = \frac{x}{[D_F(1-x) + x]} \quad . \quad (5)$$

Referring to the three cases of water concentration in voloxidizer off-gas shown in Appendix A, the concentrations of water in the off-gas of a downstream water removal system are calculated for a range of D_F and are shown in Table B.1

Table B.1. Water concentration in untreated voloxidizer off-gas and reduced concentrations at specific decontamination factors.

Case	H ₂ O Concentration (ppmv)			
	$D_F=1$ (Untreated)	$D_F=10$	$D_F=100$	$D_F=1000$
1	1.82	0.182	0.0182	0.00182
2	9.45	0.945	0.0945	0.00945
3	7.47	0.747	0.0747	0.00747

The vapor pressure of water at very low dew points is shown in Table B.2. The dew point of the voloxidizer off-gas stream will have to be reduced to very low dew points, by removal of water, to achieve the low concentrations shown in Table B.1. For example, a D_F of 100 for Case 2 requires the effluent stream to be reduced to a dew point of less than -90°C . Instead of running a cryogenic system, which would be subject to plugging with ice, water can be removed to low concentrations with type 3A molecular sieve (3AMS).

Table B.2 Calculated vapor pressure of water and equilibrium concentration in gas phase at low temperatures.

Dew Point ($^{\circ}\text{C}$)	Vapor Pressure (mmHg)	Concentration (ppmv) ^a
0	4.584473	6195.23
-5	3.013476	4072.26
-10	1.94944	2634.38
-20	0.774515	1046.64
-30	0.285116	385.29
-40	0.096318	130.16
-50	0.029524	39.90
-60	0.008103	10.95
-70	0.00196	2.65
-80	0.00041	0.55
-90	7.24E-05	0.10
-100	1.05E-05	0.01

^a Volume fraction assumes total pressure of 740 mmHg.

Adsorption isotherms for 3AMS are shown in Fig. B.1. Referring again to Case 2, the partial pressure of water in the treated stream must be reduced to 0.00007 mmHg to achieve a D_F of 100. This is below the range of available data shown in Fig. B.1. If the partial pressure of water in the gas phase was 0.001 mmHg, which is equivalent to a dew point of about -70°C , the 3AMS can adsorb up to 2.5% water by weight when the sorbent is at a temperature of 25°C . Examination of the chart indicates that water can be sorbed at very low partial pressures of water, because sorption capacity exists before equilibrium is achieved. It is possible to remove water from a very lean gas stream with well dried sorbent, which necessarily restricts water accumulation to small fractions of the sorbent mass. This might be a preferred operation were it desirable to recover the tritium in as concentrated a form as possible. Such an operation

would also require a reliable means to measure water concentration in the gas phase to very low values, equivalent to dew points of around -100°C . Alternatively, water vapor could be added to the stream to be processed which would allow removal of the necessary fraction of water/tritium while raising the concentration of water to more easily measured values. In such an approach, the sorbent can be loaded to higher water concentrations, but the loading rate would increase. When loaded to higher concentrations, the regeneration process can be done at lower temperatures because bone dry solid would not be required for the next cycle and the risk of damaging the crystal structure (and thus drying capability) would be reduced. The point is that there exists a trade-off among factors including, concentration of tritium in the product, the quantity of product, the operating time for a regenerated column, the temperature of regeneration, and the water content of the gas used as a carrier during regeneration.

Figure B.2 shows a water breakthrough curve for a small experimental system using 3AMS to remove water from a gas stream. The breakthrough curve is shown in two units, water concentration in ppmv and dew point in $^{\circ}\text{C}$. The instrument used to monitor water dew point in the column effluent gas had a measurement range of -70°C to $+30^{\circ}\text{C}$. Dew point of the feed stream was somewhat over -15°C . In this particular system, breakthrough in concentration units begins at nearly 30 hours, but the measured dew point is rising rapidly to -50°C and is a sensitive indicator of breakthrough.

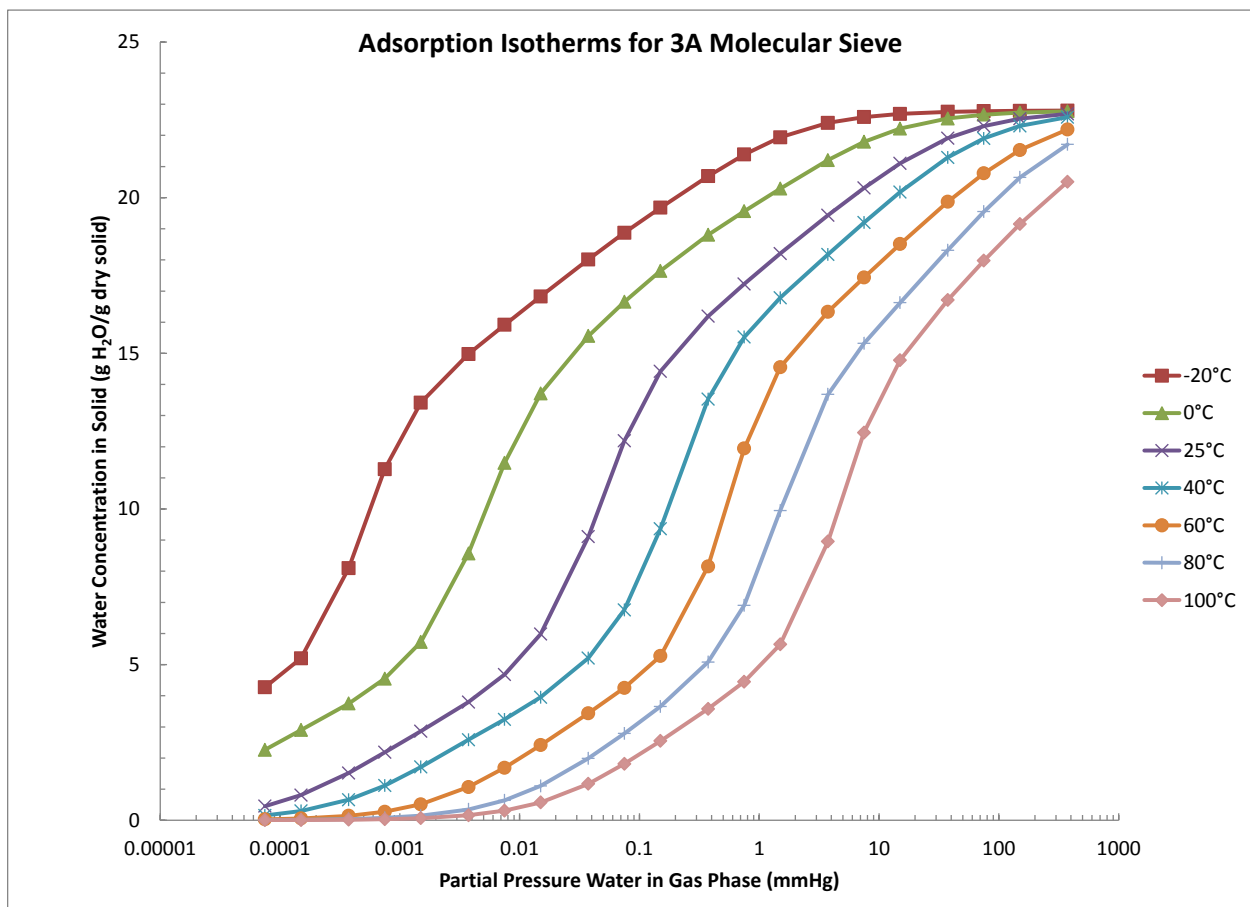


Figure B.1. Adsorption isotherms for 3AMS.

Example estimation: Make the assertion that the D_F at any point in time cannot be below 100 and that breakthrough occurs at a dew point of -50°C . In Case 2, the water concentration in the voloxidizer off-gas is 9.45 ppmv, while at -50°C the water concentration would be 39.90 ppmv. Increasing the water in the feed stream to 3990 ppmv (dew point about -5.3°C) insures that the required D_F will be achieved at each point in time until breakthrough (actually D_F will be higher at all times until breakthrough). The amount of water added depends on the gas phase throughput. For the full scale unit described in Appendix A, the water addition rate would be 0.29 g/min. Most of this will be captured in the 3AMS along with about 99% of the tritium.

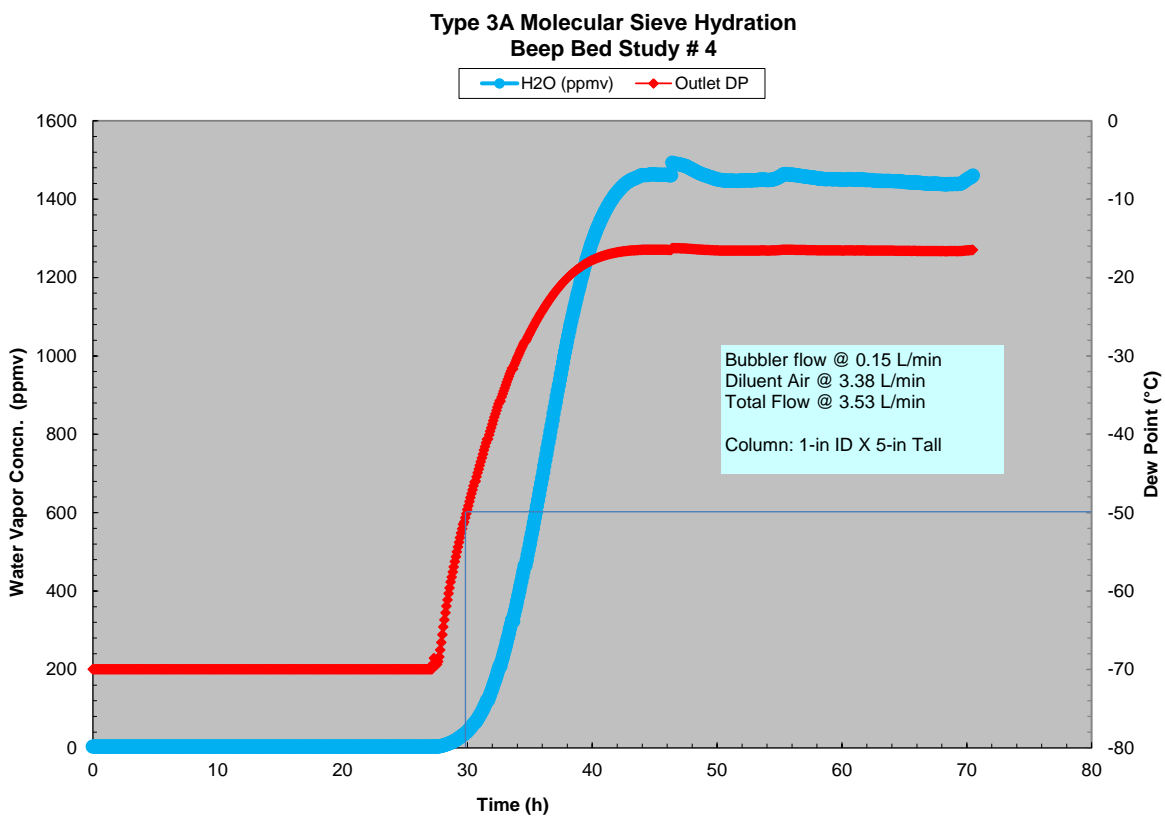


Figure B.2. Measured water breakthrough curve for small experimental 3AMS column.

Appendix C. Design Requirements for AgZ/3AMS Co-adsorption Test System

1. Overall test system
 - a. A co-adsorption test system will be designed to remove iodine and water from a flowing gas stream (which may be called the synthetic off-gas stream). Iodine concentrations in the gas stream will range from 0–100 ppmv, and the water dew point will range from -70°C – 25°C .
 - b. Silver mordenite (AgZ) will be used to remove iodine from the synthetic off-gas stream. Efficient removal requires that the AgZ be heated to at least 150°C to promote the reaction between silver and iodine. (Reduced silver mordenite [Ag^0Z] is usually preferred, but that detail is superficial to the mechanical design requirements.) When the column is saturated, heating the media above 150°C will remove iodine and water that is only physisorbed (i.e. unreacted with the silver). Temperatures up to 550°C may be tested.
 - c. Type 3A molecular sieve (3AMS) will be used to remove moisture (water) from the synthetic off-gas stream. Efficient removal and high sorption capacity of water occurs at temperatures of 25°C or lower. The 3AMS is regenerated at temperatures up to 275°C .
 - d. AgZ Columns
 - i. The system will utilize a minimum of two AgZ sorption columns such that one column is in service to treat the iodine-water laden gas stream (synthetic off-gas stream) while the other column is in a desorption or media replacement mode.
 - ii. During desorption to remove physisorbed iodine, the carrier gas stream from the desorbing column will be fed to the off-gas processing column along with the synthetic off-gas stream.
 - iii. After desorption, the column is cooled to near room temperature and the AgZ media may be replaced.
 - iv. Columns may be designed such that a short section of media above the primary section will serve as a back-stop for iodine as break through from the primary section begins.
 - e. 3AMS Columns
 - i. The system will utilize a minimum of two 3AMS sorption columns such that one column is in service to treat the water-laden synthetic off-gas stream while the other column is in a regeneration or media replacement mode.
 - ii. During regeneration, a carrier gas stream will be fed to the column and the effluent will be routed to a condenser to recover the water.
 - iii. After regeneration, the column is cooled to near room temperature and maintained at the operating temperature (e.g., 25°C).
2. Columns
 - a. Column dimensions will be determined by:
 - i. Minimum column inside diameter shall be at least 15 times the characteristic length of the sorbent media pellets.
 - ii. AgZ characteristic length (i.e. diameter of cylindrical pellet) is 1.6 mm [1/16 in.].
 - iii. 3AMS characteristic length (i.e. the diameter of the spherical pellet) is 1.6–2.6 mm [\sim 1/16–1/10 in.].
 - iv. Column height is limited by $L/D < 30$ where L=length of column, D=inside diameter of column; typical values of $L/D=5$ will be sufficient and preferred for these sorbents.
 - v. Column height for the primary sorbent section shall be at least $L/D > 3$.
 - b. Operating characteristics

- i. Columns shall remain leak tight over the operating range (see Furnaces below).
 - ii. Columns shall be designed to permit replacement of the media, but on-line media replacement will not be required (i.e., media may be replaced when the column is out of service and cooled to room temperature).
 - iii. Time to breakthrough is recommended to be an integer multiple of a 24 hr period. This will aid in scheduling replacement or regeneration of the media on the day shift.
 - c. Fluid distributors
 - i. Intra-column fluid re-distributors will not be required if column height is limited by $L/D \leq 5$, otherwise place re-distributors $5D$ apart in the column.
 - ii. The first re-distributor in the bottom of the column shall be capable of supporting the media bed.
 - iii. Columns operated in down-flow will use a re-distributor above the sorption media. Distance above the media surface should be between 1 in. and 3 in.
 - d. Material of construction
 - i. Columns and internals shall be fabricated of monel or hastelloy, if available, or stainless steel otherwise.
 - ii. Tubing used in applications requiring heating or where the temperature may exceed 150°C shall be fabricated of monel or hastelloy, if available, or stainless steel otherwise.
 - iii. Tubing that does not require heating or that is used in low temperature applications ($<150^{\circ}\text{C}$) may be fabricated of Teflon, polyethylene, or polypropylene.
 - iv. Valves shall be fabricated of monel or hastelloy, if available, or stainless steel otherwise. Exceptions may be made for low temperature applications ($<100^{\circ}\text{C}$) where valves may be fabricated of Teflon, nylon or other polymers (as approved by the lead researcher).
3. Furnaces
 - a. Furnaces will be sized to enclose the column sufficiently that the media bed will be controlled at a uniform temperature at steady state.
 - b. Furnace design shall permit ready access to and removal-replacement of media columns.
 - c. AgZ furnace
 - i. Shall have the capability to heat the column up to 600°C and control the temperature at specific values within the range (normally adsorption takes place at 150°C , and desorption tests up to 550°C are desirable).
 - ii. Temperature ramp rate shall be at least $5^{\circ}\text{C}/\text{min}$.
 - d. 3AMS furnace
 - i. Shall have the capability to heat and control the column temperature at values ranging from 25°C to 300°C .
 - ii. Temperature ramp rate shall be at least $5^{\circ}\text{C}/\text{min}$.
4. Gas Flows
 - a. Gas flows feeding the columns will be metered using mass flow controllers.
 - b. Flow rates of gases to be treated during the sorption cycle shall be adjustable from zero to rates that will achieve a maximum superficial velocity of $10\text{ m}/\text{min}$ in the columns.
 - c. Flow rates of carrier gases used during desorption or regeneration cycles shall be adjustable from zero to rates that are $\frac{1}{4}$ of the maximum rate defined in 4b.
 - d. Both column feed and discharge pipelines shall be heat-traced to prevent condensation of any component of the gas stream. One exception is the discharge from the 3AMS column(s); during

regeneration water may condense in the metal discharge line outside the furnace, but configured properly it will drain into the downstream condenser-water collector.

- e. A means to cool the gases discharged from the AgZ columns before feeding these gases to the 3AMS column shall be provided. Cooling to 25°C–30°C is required and cooling to 25°C is preferred
5. Condenser
 - a. The condenser shall be capable of removing >99% of the water from the inlet gas stream.
 - b. Means to collect the water in either solid or liquid form for subsequent weighing and/or analysis shall be provided.
 - c. Materials of construction shall be resistant to corrosion by iodine and iodine compounds that form from iodine in aqueous media. (Glass condensers are suitable and are available in the lab.)
 6. Sampling
 - a. Pipelines to serve as sampling ports will be, at a minimum, provided at (i) the feed stream to the AgZ columns, (ii) the effluent of each AgZ column, and (iii) the effluent of each 3AMS column.
 - b. Each sampling port shall be equipped with a valve so that it may be easily placed into or removed from service.
 - c. Sampling ports may be connected to pipelines leading to electronic hygrometers or to iodine traps.
 7. Instrumentation
 - a. Online instrumentation includes electronic hygrometers, thermocouples, and mass flow controllers.
 - b. Instrument readings shall be electronically displayed and recorded using a computer based data acquisition system.
 - c. Recording frequency for all instruments shall be 1 min⁻¹ or other value as specified by the lead researcher.