Phase 2 Methyl Iodide Deep-Bed Adsorption Tests

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INL

Idaho National Laboratory

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Fuel Cycle Research & Development

Prepared for
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by

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SUMMARY

Nuclear fission produces fission products (FPs) and activation products, including iodine-129, which could evolve into used fuel reprocessing facility off-gas systems, and could require control to limit emissions to levels within acceptable limits. Research, demonstrations, and some reprocessing plant experience have indicated that diatomic iodine can be captured with efficiencies high enough to meet regulatory requirements. Research on the capture of organic iodides has also been performed, but to a lesser extent. Several questions remain open regarding the capture of iodine bound in organic compounds.

Deep-bed methyl iodide adsorption testing has progressed according to a multi-laboratory methyl iodide adsorption test plan. This report summarizes the second phase of methyl iodide adsorption work performed according to this test plan using the deep-bed iodine adsorption test system at the Idaho National Laboratory (INL), performed during the second half of Fiscal Year (FY) 2014.

Test results continue to show that methyl iodide adsorption using AgZ can achieve total iodine decontamination factors (DFs, ratios of uncontrolled and controlled total iodine levels) above 1,000, until breakthrough occurred. However, mass transfer zone depths are deeper for methyl iodide adsorption when compared to diatomic iodine (I_2) adsorption, and exceeded 8 inches in one test.

Methyl iodide DFs for the Ag Aerogel test were less than 1,000, and the methyl iodide mass transfer zone depth exceeded 8 inches.

Additional deep-bed testing and analyses are recommended to (a) expand the data base for methyl iodide adsorption under various conditions specified in the methyl iodide test plan, and (b) provide more data for evaluating organic iodide reactions and reaction byproducts for different potential adsorption conditions.

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ACRONYMS

AgZ   silver zeolite

DF   decontamination factors

DL   detection limit

DOE   Department of Energy

ECD   electron capture detector

FCT   Fuel Cycle Technology

FID   flame ionization detector

FP   fission product

FY   fiscal year

GC   gas chromatograph

GCMS   gas chromatography with mass spectrometry

ICPMS   inductively coupled plasma mass spectrometry

INL   Idaho National Laboratory

MTZ   mass transfer zone

ORNL   Oak Ridge National Laboratory

PNNL   Pacific Northwest National Laboratory

SPME   solid-phase micro-extraction

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1.   INTRODUCTION

Nuclear fission results in the production of fission products (FPs) and activation products. If used nuclear fuel is reprocessed, some of those FPs and activation products, including iodine-129, could evolve into the process off-gas systems, and would likely require control to limit emissions to acceptable levels.

The Department of Energy (DOE) Fuel Cycle Technology (FCT) Program has supported research and development on iodine control and iodine waste forms for the past several years. The Offgas Sigma Team was formed in 2009 within the Separations and Waste Forms Campaign (now called the FCT Material Recovery & Waste Form Development Campaign) to establish a team of researchers from different DOE sites to focus on research and development for emissions control and waste forms for volatile radionuclides.

Capture efficiencies for I-129 may need to be high enough to achieve off-gas decontamination factors (DFs) of at least 3,000x (99.9% capture efficiency) to comply with applicable reprocessing facility regulatory requirements [Jubin 2012a]. Iodine control may need to be used on multiple reprocessing facility off-gas streams (such as the head end off-gas, dissolver off-gas, vessel off-gas, and waste treatment off-gas streams), because I-129 in the used fuel is likely to distribute at up to percent levels to different off-gas streams [Jubin 2013]. Iodine is known to exist in off-gas streams as diatomic I_2, and also react with organic species such as organic solvents or organic diluents used in actinide separations or degradation products of those organic compounds, to form organic iodides.

Research, demonstrations, and some reprocessing plant experience have indicated that diatomic iodine can be captured with efficiencies high enough to meet regulatory requirements [Soelberg 2013]. Research on the capture of organic iodides has also been performed, but to a lesser extent [Jubin 2012b]. Several questions remain open regarding the capture of iodine bound in organic compounds.

A test plan was designed which identifies organic iodide data gaps and outlines a multi-year test program for advancing the state of understanding and capabilities for capturing organic iodides on solid sorbents [Jubin 2012b]. This report summarizes the adsorption work performed according to this test plan using the deep-bed iodine adsorption test system at the Idaho National Laboratory (INL), performed during Fiscal Year (FY) 2013 and early FY-2014.

All work was performed in compliance with work control documentation that was updated in FY-2013 to ensure data quality, worker safety, environmental protection, and regulatory compliance during testing (INL 2013).

2.   DEEP BED IODINE SORBENT TEST SYSTEM

Figure 2-1 shows a process diagram for the iodine test system. The main components are the:

- Process gas supply and blending system, which supplies gases from gas cylinders, iodine gas species generators, and a humidifier

- Multiple sorbent bed system inside a heated oven

- Process gas bypass

- Inlet and bed segment outlet gas sampling system.
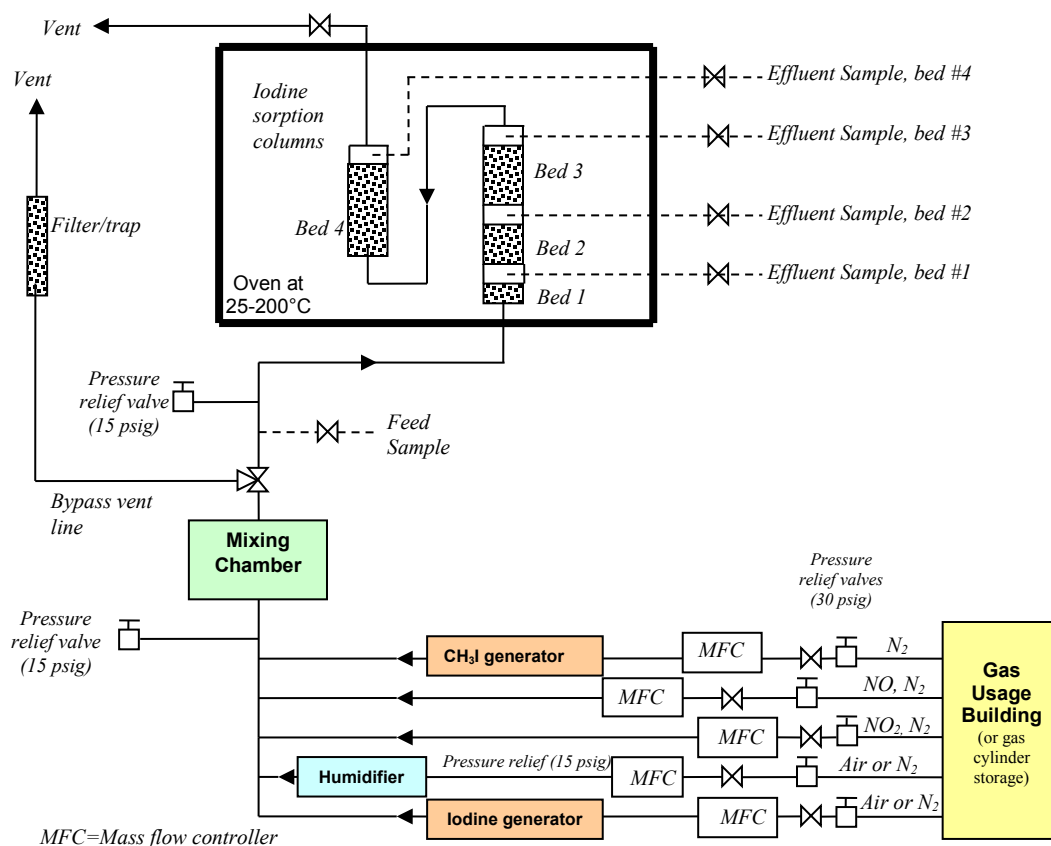
Figure 2-1. Deep-bed adsorption test system.

2.1   Process Gas Supply System

The process gas supply system consists of pressurized gas cylinders to supply the gases that are blended together creating the test gas mixture that is passed through the sorbent beds. These gases can include (depending on the test) pure air, nitrogen, NO_x, water vapor, diatomic iodine, and methyl iodide (as a surrogate for organic iodide). Air or N_2 can be supplied through mass flow controllers separately to the iodine and methyl iodide generators and the humidifier. NO and NO_2 gases, with balance N_2, are supplied from compressed gas cylinders through mass flow controllers.

Methyl iodide and iodine gases can be provided using compressed gas cylinders, permeation tubes, or a fixed bed iodine generator. The choice of methyl iodide and iodine source depends on the flowrate needed to achieve the target concentration in the test gas mixture. For lower methyl iodide concentrations under about 1 ppm, compressed gas cylinders or a permeation tube system may be used. For low iodine concentrations (under about 2 ppm) or for methyl iodide concentrations above about 1 ppm, the permeation tube system is used. For higher iodine concentrations above about 2 ppm, a fixed bed of iodine crystals interspersed in glass beads (which prevents iodine crystal agglomeration) is used.

The permeation tube system uses semi-permeable tubes that contain liquid methyl iodide (or solid iodine crystals) to emit a known flowrate of methyl iodide (or iodine) at a constant rate which is controlled by the operating temperature of the tube. The tubes (up to two), from VICI Metronics, are

placed inside a Dynacalibrator Model 190 constant temperature permeation tube system (shown in Figure 2-2) also from VICI Metronics.

The gas flowrates and the generation rates of vaporized iodine, methyl iodide, and water are set to achieve the target gas composition and blended in a mixing chamber upstream of the sorbent beds. All process lines that contain vaporized iodine, methyl iodide, or water are electrically heat traced.

I_2 impingers

Permeation tube I_2 or CH_3I generator
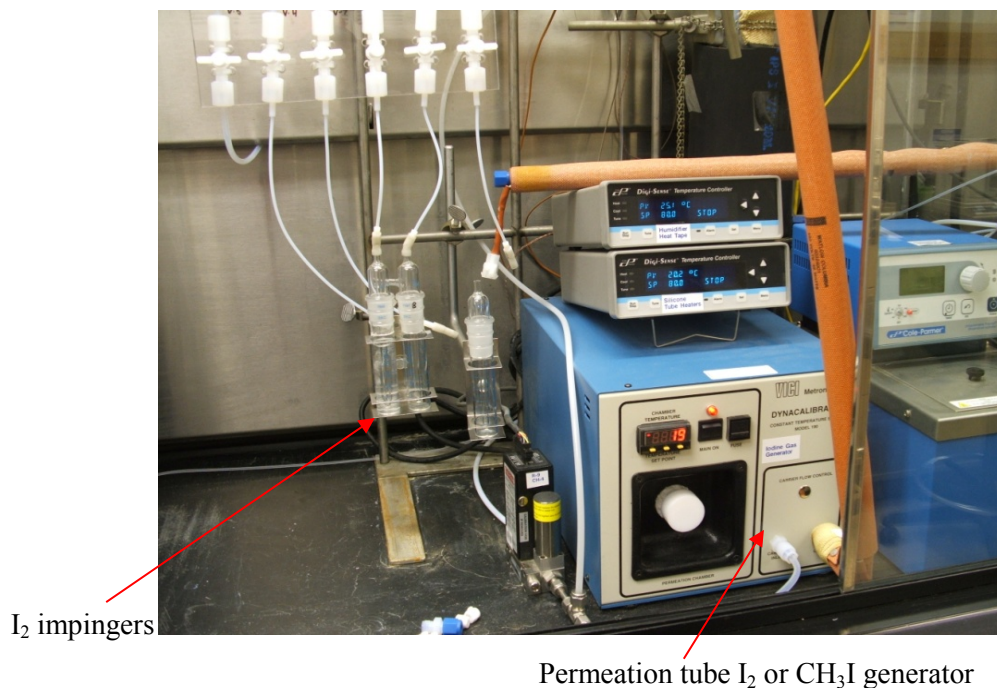
Figure 2-2. View of the iodine impingers and the permeation tube iodine or methyl iodide generator

Humidified air is produced by passing air or nitrogen through a fritted glass bubbler submerged in a constant temperature water bath. A thermocouple in the headspace of the bubbler provides the temperature of the water-saturated gas. The concentration of water in the blended gas is controlled by adjusting the gas flowrate through the humidifier and the operating temperature.

2.2   Sorbent Bed Segments

Figures 2-3 and 2-4 show the detail of the sorbent beds and how the sorbent beds are configured in a temperature-controlled oven. The current test design includes up to four sorbent bed segments. The sorbent bed segments are made of borosilicate glass with a glass frit at the bottom of each bed segment to support the granular sorbent.
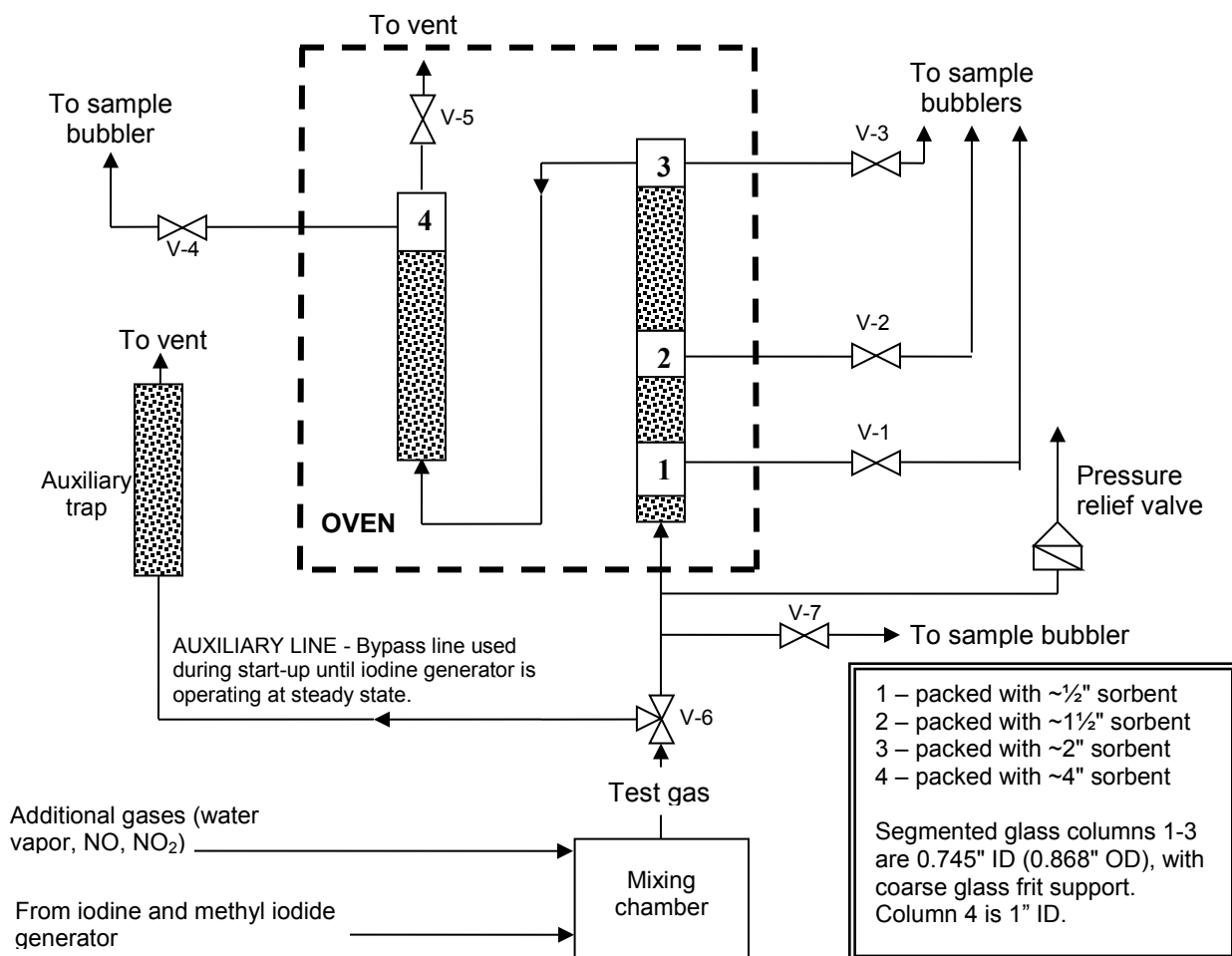
Figure 2-3. Detail of the sorbent beds. At times, only three of the four beds are used.

2.3   Sample Collection and Analysis

Iodine and methyl iodide concentrations in the test gas can be measured at up to five locations in the test system – at the inlet to the sorbent bed segments, and at the outlet of each of up to four bed segments. Since the gas flowrate is essentially the same at all five sample locations, the removal efficiencies for the sorbent in all four beds can be determined by measuring the iodine and methyl iodide concentrations at these locations. By integrating over time and using the test gas flowrate, the adsorption efficiencies and the amounts of iodine and methyl iodide adsorbed on each of the beds can be calculated. The amount of adsorbed iodine can also be determined gravimetrically by weighing the sorbent before and after each test.

Figure 2-4. Configuration of the sorbent beds inside the temperature-controlled oven.

2.3.1   Iodine Sample Collection and Analysis

Iodine measurements are made even when iodine is not included in the test gas mixture to determine if, or how much, iodine is being formed from reactions of methyl iodide. For measuring the gaseous iodine concentration, the process gas from any of the five sample locations is passed through 25-ml "midget" impingers that contain 0.1 N NaOH for scrubbing halogen gases including I_2 and HI, if present. This technique is modeled after EPA Method 26 "Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources, Non-Isokinetic Method" (40 CFR 60 Appendix A). The caustic solution captures halogens by hydrolyzing halogen gases to form a proton (H^+) and hypohalous acid.

Any HI, if present, will dissociate in the caustic solution, and is included with the I_2 in the analysis. As a result, this test method does not discriminate between I_2 and HI or other iodine species that are soluble in 0.1 N NaOH. Testing was initially tried using an impinger of 0.1 N sulfuric acid upstream of the NaOH impingers, intended to quantitatively separate HI (which is soluble in 0.1 N sulfuric acid) from the I_2. This concept is modeled after EPA Method 6, which uses the separate sulfuric acid and NaOH impingers to separate HCl, HF, and HBr from Cl_2, F_2, and Br_2. However, these initial test results showed that too much I_2 was retained in the sulfuric acid impinger to provide a valid HI-I_2 separation.

The bubbler solutions are analyzed by inductively coupled plasma mass spectrometry (ICPMS) per EPA Method 6020A (SW-846, "Test Methods for Evaluating Solid Wastes Physical/Chemical Methods," http://www.epa.gov/osw/hazard/testmethods/sw846/online/). The gaseous iodine detection limit (DL) is about 0.08 ppb with this method. Higher–concentration samples for gas streams with 1 ppm or higher iodine concentrations are typically diluted for analysis.

2.3.2   Organic Compound Sampling and Analysis

Methyl iodide analysis using gas chromatography (GC) has evolved over time due to the corrosive nature of the typical test gas. Early testing used a Hewlett-Packard model 5890 Series II gas chromatograph (GC) installed in-line with the sample loop, thereby allowing near real-time analysis of methyl iodide [Haefner 2010]. An Rt-Q-BOND fused silica capillary column was used in the GC. The

GC was equipped with an electron capture detector (ECD), which is very sensitive for measuring halogenated organic compounds. The minimum detection limit for this GC setup was about 5 ppb.

This early testing indicated that the use of the sample loop (which enables frequent and automatic sampling) and the highly sensitive ECD resulted in apparent corrosion of GC components resulting in erroneous measurements. The GC was frequently down for maintenance. Essentially all components that contacted the sample gas were eventually replaced, and the GC continued to frequently malfunction.

Beginning in FY-2013, the GC was replaced with another Hewlett Packard 5890 GC, equipped with a RTX-624, 30 m x 0.32 mm ID, 1.8 µm df column, and a flame ionization detector (FID). The sample loop was not used causing the sampling and analysis to be more operator time-intensive, and reduced the number of GC measurements that are practical, but reduced the amount of time that the GC components are exposed to the corrosive sample gas.

The FID is not as sensitive for methyl iodide analysis as the ECD resulting in a methyl iodide detection limit of about 1 ppm using direct injections. When lower detection limits were desired a solid-phase micro-extraction (SPME) syringe was used. A SPME adsorbs organic compounds onto a solid-phase sorbent in a needle, thereby concentrating the amount of analyte. The adsorbed analytes are then desorbed into the GC carrier gas at an elevated temperature.

The SPME syringe is a Supelco brand, containing 75 µm carboxen/polydimethylsiloxane fiber. The SPME and GC are calibrated together for specified adsorption and desorption times and temperatures. Using a SPME improves the methyl iodide detection limit by approximately 100x to about 10 ppb.

Unknown organic compounds formed by reactions of methyl iodide can appear as additional peaks on the GC-FID chromatograms. When this occurs, they can be tentatively identified using gas chromatography – mass spectroscopy (GCMS) analysis. The GCMS used for this work is a Shimadzu GC2010 with GCMS-QP2010 (with autosampler). The column is a J&W Scientific DB-1 (dimethyl polysiloxane) column, 30 m x 0.25 mm ID x 1 µm df.

3. DEEP BED METHYL IODIDE TEST RESULTS

Methyl iodide testing proceeded according to the joint methyl iodide test plan [Jubin 2012b]. Results of Phase 1 testing through the first half of FY-2014 are reported in Soelberg 2014. Results through the second half of FY-14 are reported in this Phase 2 report. These results are summarized in Table 3-1, and are further discussed in more detail in the following sections.

Table 3-1. Results of the Phase 2 methyl iodide tests performed through the second half of FY-2014.

Question from Jubin 2012b	Test conditions	Results	
Methyl iodide adsorption on AgZ, with NO_x but almost no H_2O – is I_2/HI formed?			**Yes.**
CH3I-6 AgZ test (1.b.iii.3. Test 3 in test plan)	33 ppm average CH_3I, AgZ sorbent, 4.3 m/s air velocity, 822 ppm NO_2, 822 ppm NO_2, 3 ppm H_2O	Initial CH_3I DF ~40,000, decreasing more rapidly than DFs for iodine adsorption, indicating MTZ > 8 inches.	
		Bed 1 outlet CH_3I levels increased from nondetectable (0.01 ppm) to 2 ppm (6% of input CH_3I) after breakthrough.	
		Bed 1 outlet I_2/HI levels increased from 0.0001 ppm to 12 ppm (73% of input iodine) after breakthrough.	
		Of the total Bed 4 outlet unabsorbed iodine after breakthrough, about 0.1% remained CH_3I; about 99.9% was in the form of I_2/HI.	
		With NO_x present and very low H_2O in the gas stream, most of the unabsorbed iodine was converted to I_2/HI after breakthrough. This result is consistent with the CH_3I-4 test (Soelberg 2014), in which most of the unabsorbed iodine after breakthrough was in the form of I_2/HI in the presence of NO_x and 1.8% H_2O.	
		Dimethyl ether (CH_2OCH_3) and nitromethane (CH_3NO_2) have been tentatively identified in the sorbent bed outlet gas, along with at least one other compound that could not be identified. The estimated levels of dimethyl ether are high enough to account for most of the CH_3 moiety separated from the adsorbed iodine, which is different than was observed in prior tests (Soelberg 2014), when the total amount of detected organics was not high enough to account for the CH_3 moiety.	
Methyl iodide adsorption on Ag Aerogel, with NO_x and H_2O – Is CH_3I sorbed, and is I_2/HI formed?		**CH_3I adsorption is not as high as for I_2 adsorption on Ag Aerogel or CH_3I adsorption on AgZ. I_2/HI, formed in early in the bed, was mostly adsorbed further downstream in the beds.**	
CH3I-7 AgAerogel test not included in the Jubin 2012b test plan	24 ppm average CH_3I, AgZ sorbent, 4.3 m/s air velocity, 824 ppm NO_2, 824 ppm NO_2, 1.8% H_2O	Initial CH_3I DF ~130, decreasing more rapidly than DFs for iodine adsorption, indicating MTZ > 8 inches.	
		Bed 4 Outlet CH_3I levels increased from ~0.2 ppm 15 ppm (63% of input CH_3I) after breakthrough.	
		Bed 1 outlet I_2/HI levels increased from undetectable (0.0002 ppm detection limit) to 2 ppm (17% of input iodine) after breakthrough. The maximum unadsorbed I_2/HI at the outlet of Bed 4 was lower at 0.001 ppm.	
		Of the total Bed 4 outlet unabsorbed iodine after breakthrough, over 99.9% remained CH_3I; under 0.02% was in the form of I_2/HI.	
		With NO_x and H_2O in the gas stream, most of the unabsorbed iodine remained CH_3I after breakthrough. This result is not consistent with the CH_3I adsorption tests on AgZ (Soelberg 2014), in which most of the unabsorbed iodine after breakthrough was in the form of I_2/HI in the presence of NO_x and 1.8% H_2O.	

Two long-duration methyl iodide adsorption tests are reported here. Table 3-2 provides some details of these two tests.

Table 3-2. Methyl iodide adsorption tests on AgZ (Test CH$_3$I-6) and Ag Aerogel (Test CH$_3$I-7).

Run Number	CH3I 6 33 ppm CH3I w/ NOx, w/o H2O	CH3I 7 AG 33 ppm CH3I w/ NOx, H2O
Simulate what off-gas?	Dissolver	Dissolver
Test start date	19-Nov-13	11-Jun-14
Sorption conditions		
Temperature, deg. C	150	150
Total gas flowrate, sccm	730	728
Target CH3I conc, ppmv	35	50
Measured CH3I conc, ppmv	32.9	24.0
Target I2 conc, ppmv	0.0	0.0
Measured I2 conc, ppmv	0.54	0.21
Measured total I flowrate, mg/min	---	---
H2O conc, %	0.0003	1.8
NO conc., ppmv	822	824
NO2 conc., ppmv	822	824
Balance	air	air
Gas flowrate, standard l/min	0.73	0.73
Sorption gas velocity, m/min	4.3	4.3
Bed 1 out residence t, sec	0.18	0.18
Bed 2 out cumulative residence t, sec	0.70	0.71
Bed 3 out cumulative residence t, sec	1.41	1.41
Bed 4 out cumulative residence t, sec	3.91	3.92
% I conversion to I2 at bed inlet	3.3%	1.8%
Cumulative test duration, hr	462	260
Max iodine loadings from grav analysis	3.2%	Not Available
Max silver utilization, %	34%	Not Available
Max DF before breakthrough	41,767	134
Max conversion of bed outlet CH3I to I2	99.9%	0.7%
Mass transfer zone depth, inches	>8 inches	>8 inches
Organic compounds detected	dimethyl ether, C2H6O	dimethyl ether, C2H6O
	nitromethane, CH3NO2	nitromethane, CH3NO2
	At least one other unidentified compound	acetic acid, CH2H2O4
		1-Napthalene carbonitrile, C11H7N
		2,5-furandione (maleic anhydride), C4H2O3
		At least one other unidentified compound
	[iodine data 26sept14.xlsx]2013 CH3I summary	

3.1   Test CH$_3$I-6

This test was designed to be a part the test matrix for evaluating methyl iodide adsorption. The test gas included NO$_2$, but low H$_2$O. Silver zeolite (AgZ) was the sorbent, with a presumed silver content of 9.5 wt%. This sorbent was chemically reduced to convert the silver to Ag^o and provided by Oak Ridge National Laboratory (ORNL).

Figure 3-1 shows the AgZ sorbent in the sorbent beds prior to adsorption testing, and Figure 3-2 shows the AgZ sorbent following the test. The pre-test sorbent granules are colored varying shades of grey and brown. The post-test sorbent, especially in Bed 1, turned a light beige color; the sorbent in the other beds has become somewhat rose or coral-colored.

Figure 3-1. AgZ sorbent in the sorbent beds prior to the test.
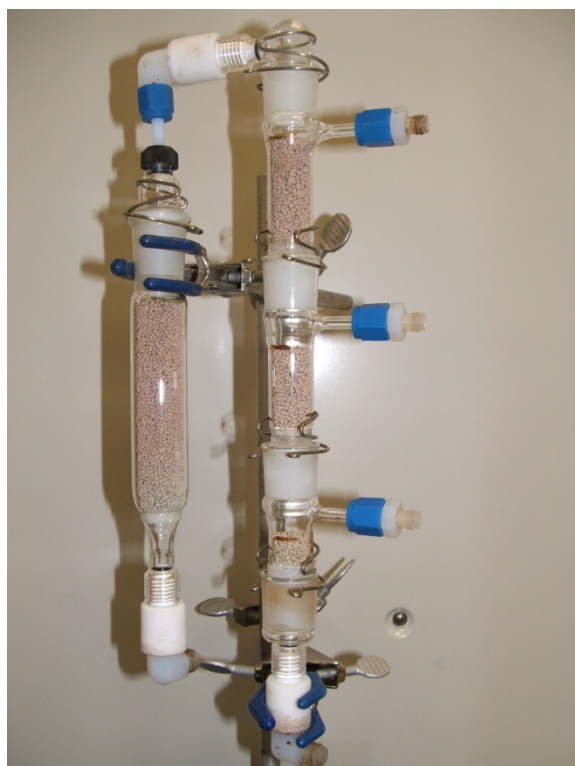

Figure 3-2. AgZ sorbent in the sorbent beds following the test.

Figure 3-3 shows the results of this test. At the test start, the highest measured DF (at the outlet of Bed 4, at 8 inches total bed depth) was over 40,000.   The DF at the outlet of Bed 4 was not measured until later in the test. The initial DF at the outlet of Bed 1, at 0.5 inches bed depth, was about 400, indicating that break-through at this 0.5-inch depth had already occurred prior to the first sample period. The DFs at the outlets of all four beds rapidly decreased such that the DFs had all decreased to 1,000 or less by hour 10 and to 100 or less by hour 100. The rapid decrease in the DFs for the bed depth up to 8 inches, indicate the depth of the mass transfer zone (MTZ) was at least 8 inches. This MTZ depth is deeper than prior MTZ estimates for either CH_3I or I_2 adsorption on either AgZ or Ag Aerogel.
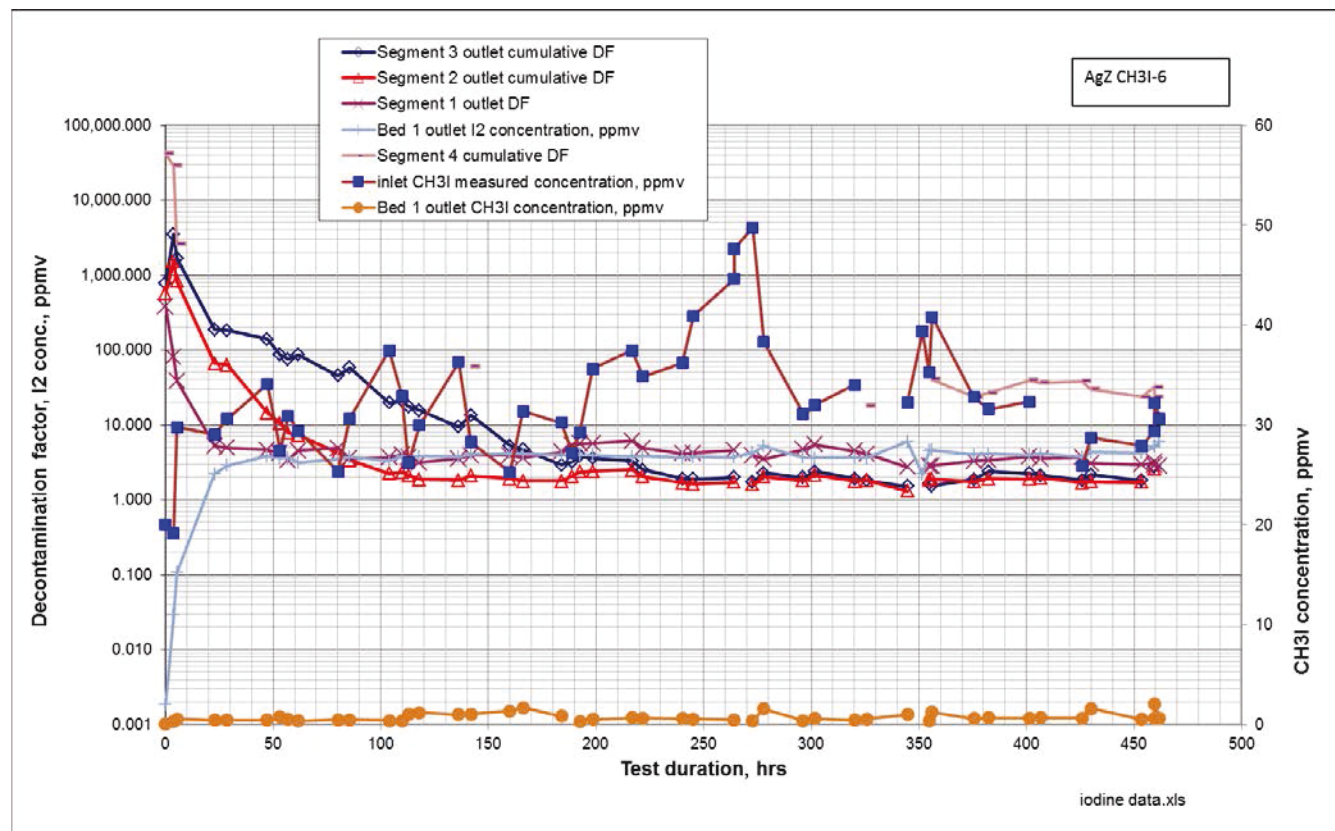

Figure 3-3. Test CH_3I-6 methyl iodide adsorption results.

The DFs for Beds 1, 2, and 3 decreased to about 2-3 during the test, indicating that the first four inches of bed depth did not reach saturation for the 460-hr duration of this test. The DF for Bed 4 (8 inches total depth) decreased to about 20-30.

As each bed became progressively laden with iodine, gaseous CH_3I levels still stayed below about 2 ppmv. Simultaneously, levels of Bed 1 outlet I_2/HI increased from near zero to about 4-6 ppm. The combined gaseous CH_3I and I_2/HI levels in the Bed 1 outlet gas was only about 40-50% of the CH_3I.

The GC chromatograms showed several peaks in addition to the CH_3I peak. Samples of gas from the bed outlets were periodically analyzed by GCMS in an attempt to tentatively identify these organic compounds.   The GCMS was not as sensitive for these organic compounds as was the GC-FID, so some peaks detected on the GC-FID chromatograms were not detected by the GCMS. The two compounds that were detected and tentatively identified were dimethyl ether and nitromethane. Additional evaluation and analysis of methyl iodide reactions and iodine adsorption on AgZ are reported in Nenoff 2014.

After the test was completed, the sorbent beds were purged with dry air to desorb any amounts of iodine that may be loosely or physisorbed. The purge results for this test are shown in Figure 3-4. This figure shows that only a small fraction of the iodine adsorbed on Bed 1 was desorbed during the 24-hr purge period. During the purge period, Bed 1 outlet I_2/HI and CH_3I levels decreased to 2 to 4 orders of magnitude lower than Bed 1 outlet I_2/HI and CH_3I levels that occurred near the end of the adsorption test. The cumulative amount of total iodine that was desorbed from Bed 1 increased from about 0.6% at the start of the purge period to about 3.3%.
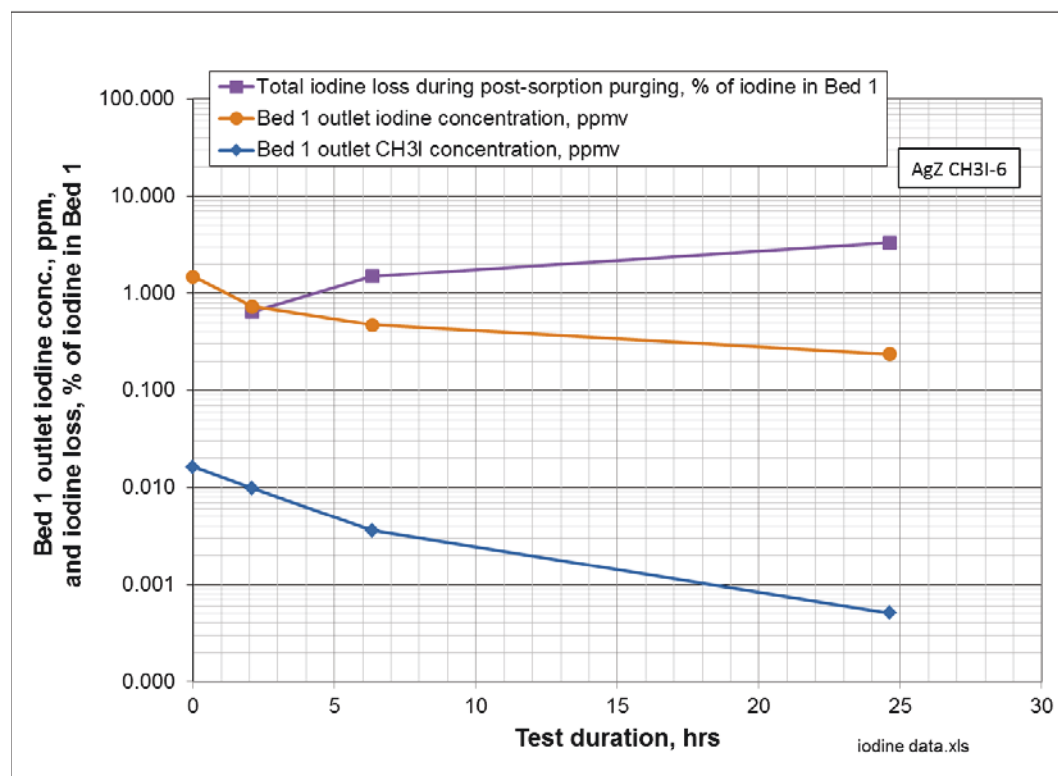

Figure 3-4. CH_3I-6 post-test sorbent purge results.

3.2   Test CH₃I-7

This test was an initial evaluation of the performance of Ag Aerogel for adsorbing methyl iodide. This test included both NO_x and H_2O in the gas mixture. The Ag Aerogel was provided by Pacific Northwest National Laboratory (PNNL).

Figure 3-5 shows the Ag Aerogel prior to this test. The Ag Aerogel particles are a range of colors between red-orange to black. Figure 3-6 shows the Ag Aerogel at the end of the test. The sorbent in Beds 1-3, and at the front of Bed 4, turned to a beige-white color. The rest of Bed 4 changed from the dark red-black overall color to a mingling of lighter-colored particles. Clearly there has been a change or changes in the sorbent particles that has caused this significant color change.

Figure 3-5. Ag Aerogel before the adsorption test.

Figure 3-6. Ag Aerogel after the adsorption test.

Figure 3-7 shows results of this test. At the test start, the highest measured DF (at the outlet of Bed 4, at 8 inches total bed depth) was about 130.

The DFs at the outlets of all four beds rapidly decreased such that the DFs had all decreased to 10 or less by hour 5. While there was some variation the measured DFs from hour 10 until the end of the 260-hr test, the DFs for all 4 beds ranged between about 3-20. None of the beds reached saturation levels, but the depth of the MTZ appears to be at least 8 inches, the total depth of all four beds.
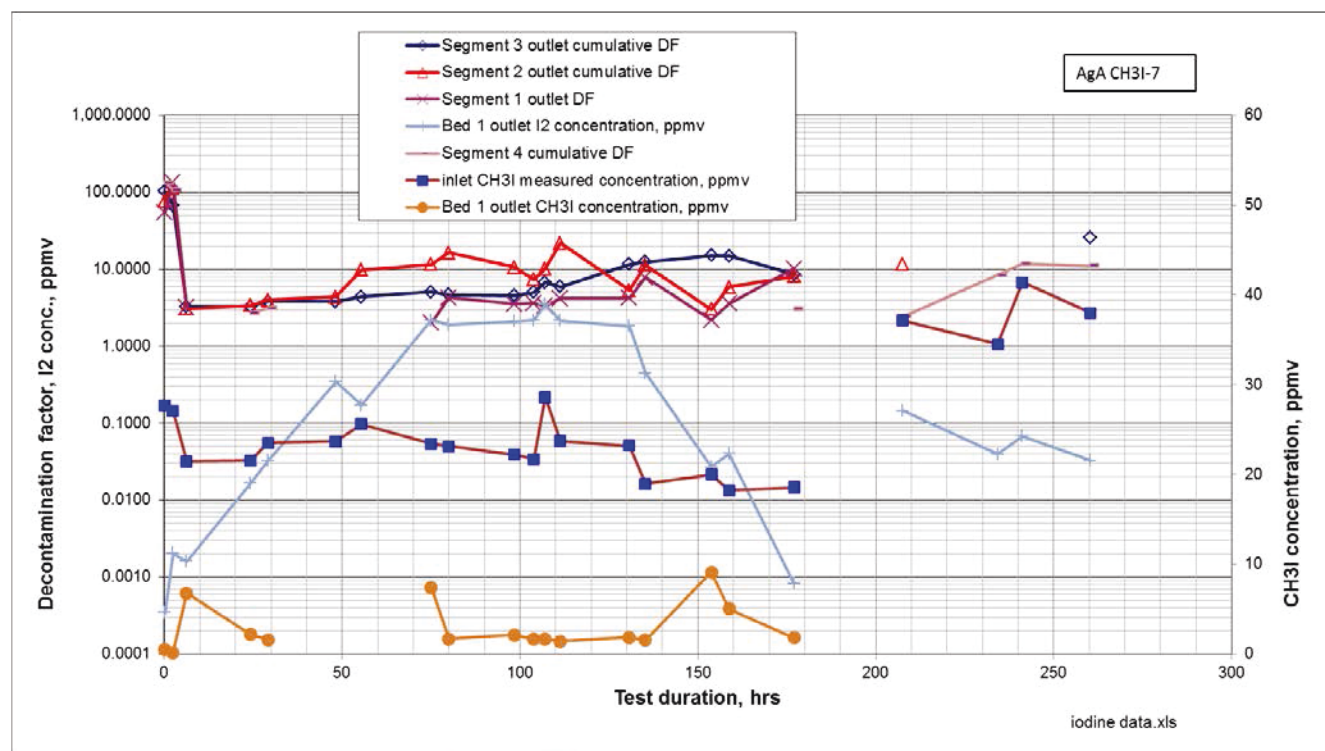

Figure 3-7. Test CH_3I-7 methyl iodide adsorption results.

As each bed became laden with iodine, gaseous CH_3I levels ranged between about 0.2 to 9 ppm at the outlet of Bed 1, when the inlet CH_3I levels averaged 24 ppm. Simultaneously, levels of I_2/HI increased from under 0.001 ppm to about 2 ppm, with some variation during the test. The combined gaseous CH_3I and I_2/HI levels in the Bed 1 outlet ranged up to about 38% of the input CH_3I levels.

As the process gas flowed through the four bed segments, the methyl iodide continued to be adsorbed or reacted. At the 8-inchbed depth (Bed 4 outlet), the I_2/HI level ranged under 0.003 ppm, and the CH_3I concentration ranged under 8 ppm. The gaseous I_2/HI represented a maximum of about 0.7% of the uncaptured iodine, and the gaseous CH_3I represented up to 99.9% of the uncaptured iodine. This ratio is very different from the results of the methyl iodide adsorption tests using AgZ, with the same gas mixture.

The GC chromatograms showed several peaks in addition to the CH_3I peak. Samples of gas from the bed outlets were periodically analyzed by GCMS to tentatively identify these organic compounds. The GCMS was not as sensitive for these organic compounds as was the GC-FID, so some peaks detected on the GC-FID chromatograms were not detected by the GCMS. Compounds that were detected and tentatively identified were dimethyl ether, nitromethane, acetic acid, 1-napthalene carbonitrile, and 2,5-furandione (maleic anhydride).

After the test was completed, the sorbent beds were purged with dry air to desorb any amounts of iodine that may be loosely or physisorbed. The purge results for this test are shown in Figure 3-8. This figure shows that only a small fraction of the iodine adsorbed on Bed 1 was desorbed during the 9-hr purge period. During the purge period, Bed 1 outlet CH_3I levels were about two orders of magnitude lower than the Bed 1 CH_3I levels were at the test end, and were near the measurement method detection limit. The Bed 1 outlet I_2/HI levels were also about 2 orders of magnitude lower than Bed 1 outlet I_2/HI levels were at the test end, and continued to decrease another order of magnitude. The cumulative amount of total iodine that was desorbed from Bed 1 increased from about 0.0004% at the start of the purge period to about 0.014% at the end of the purge period.
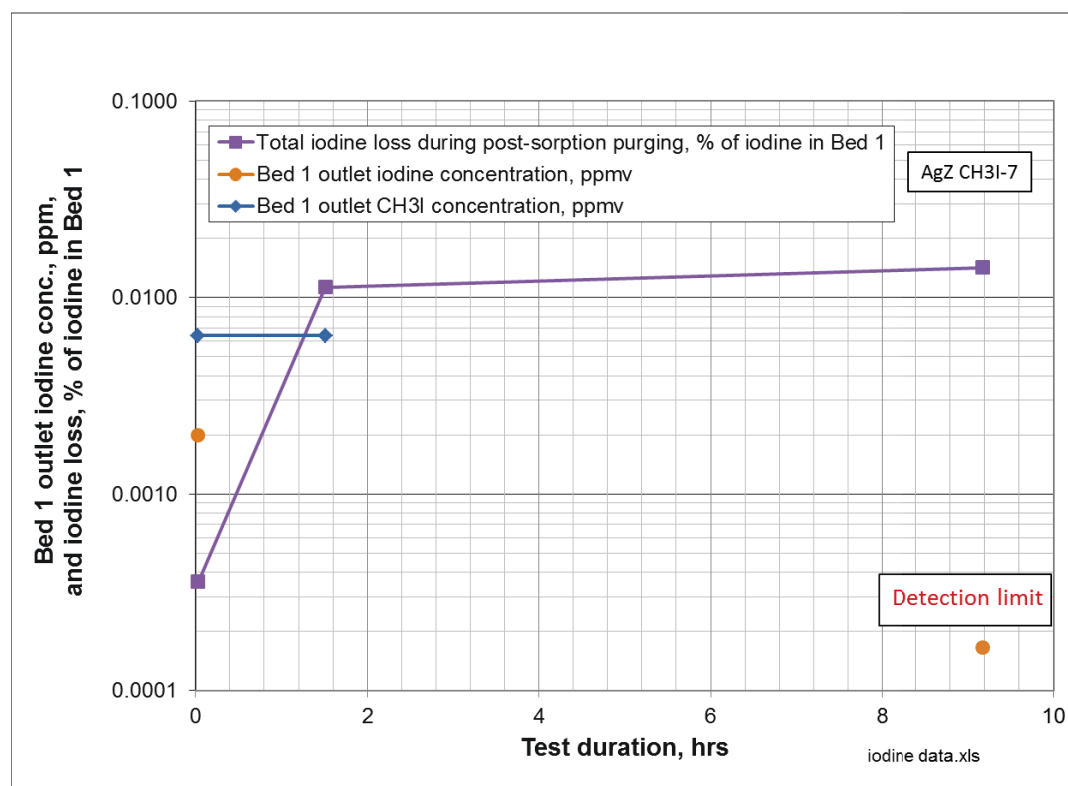

Figure 3-8. Test CH_3I-7 post-test sorbent purge results.

4.   CONCLUSIONS AND RECOMMENDATIONS

Deep-bed methyl iodide adsorption testing has progressed according to the multi-laboratory methyl iodide adsorption test plan [Jubin 2012b] and includes an initial methyl iodide adsorption test using Ag Aerogel sorbent. The reaction(s) enable separation of the iodine from the organic moiety, so that the iodine can chemisorb onto the sorbent. The organic moiety can form other compounds, some of which are organic compounds that are detected and can be tentatively identified using GC-FID and GCMS.

Methyl iodide adsorption efficiencies using AgZ continue to be high enough so that initial DFs exceed 1,000. The methyl iodide mass transfer zone depth from the last AgZ test, however, was estimated at >8 inches, deeper than mass transfer zone depths estimated for I_2 adsorption on AgZ.

The results of the Ag Aerogel test show that the initial DFs were under the nominal target value of 1,000. The MTZ for the Ag Aerogel test was >8 inches. Additional deep-bed testing and analyses are recommended to (a) expand the data base for methyl iodide adsorption under the various conditions

specified in the methyl iodide test plan, and (b) provide more data for evaluating organic iodide reactions and reaction byproducts for different potential adsorption conditions.

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