INL/EXT-10-19657
Rev. 1

Summary of FY 2010 Iodine Capture Studies at the INL

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August 2010

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SUMMARY

Packed bed tests were conducted to determine the sorption characteristics of two candidate sorbents for iodine from a simulated dissolver off-gas. The test gas was composed of methyl iodine (iodine source), NO, NO_2, and water vapor in a carrier gas of air. A single, baseline set of operating parameters was chosen to operate the sorption column and included a gas flow rate of 1200 SCCM and a sorption temperature of 150°C. The candidate sorbents were both silver mordenites and included unaltered, commercial IONEX Ag-900 and a silver reduced material received from Oak Ridge. Tests were planned to operate until an iodine breakthrough occurred in a 2" (Bed 2) or 4" (Bed 3) portion of the packed bed.

Three breakthrough runs were conducted and a dynamic sorption capacity estimated based on methyl iodine (CH_3I) breakthrough from the 2" bed. However, it is now believed that data for the first 2 runs is incomplete because the contributions from elemental iodine (I_2) were not included. Although the only source of iodine was CH_3I, elemental iodine was generated within the sorbent bed, presumably from a recombination reaction likely catalyzed by silver mordenite. On-line effluent analysis with a GC was only capable of analyzing methyl iodine, not I_2. Scrub samples drawn during Run #3, which are specific for I_2, show significant levels of I_2 being emitted, ranging from about 4% of the emitted iodine early in the run and increasing to over 95% by the end. By combining CH_3I and I_2 analyses, a well defined total iodine breakthrough curve was generated for Run #3. These findings have resulted in modification of the sampling protocol to add more extensive sampling using midget bubblers (scrub samples).

At the conclusion of Run #3 (using IONEX Ag-900) the effluent level from the 2" bed (Bed 2) was approaching 70% of the feed concentration. The leading bed section (Bed 1) had an estimated average loading of 66 mg I/g sorbent, while for Bed 2 it was 52 mg I/g, and Bed 3 it was 47 mg I/g. The corresponding silver utilizations (assuming formation of AgI) were about 59%, 46%, and 42%, respectively. A similar iodine loading gradient with bed depth was also noted for the 2 earlier runs. The spent sorbents are being sent to Sandia National Laboratories for confirmatory analysis of iodine and silver content and as source material for waste form development.

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ACRONYMS

BET	Brunauer, Emmett, and Teller (adsorption model of gas on solid surface)
D@250	dynamic sorption capacity at 250 ppb
DOG	dissolver off-gas
GC	gas chromatograph
ICP-MS	inductively coupled plasma mass spectroscopy
INL	Idaho National Laboratory
MeI	methyl iodine
PNNL	Pacific Northwest National Laboratory
SCCM	standard cubic centimeters per minute

Summary of FY 2010
Iodine Capture Studies at the INL

1. BACKGROUND

Iodine is a gaseous fission product generated in nuclear fuel, but is largely retained in the solid matrix of the fuel. However, it will be released if the fuel undergoes reprocessing with most of it evolving to the dissolver off-gas (DOG), with lesser quantities going to other off-gas streams (voloxidation off-gas, vessel off-gas, cell off-gas, etc.) and/or possibly carried-out in solidified waste. After a moderately short cooling period (~3 months), about 80% of the iodine will be the radioactive isotope I-129, with the balance being stable I-127. The I-129 isotope is very long-lived ($t_{½} = 15.7\times10^6$ years) and will persist essentially forever. Unmitigated release of gaseous iodine would be very mobile in the environment, possibly resulting in an uncontrolled and/or problematic dose. For these reasons very efficient capture of iodine is anticipated to meet stringent emission limits likely for future reprocessing facilities.

Experimental adsorption tests at the INL began in fiscal year 2009. Tests used commercial silver mordenite (IONEX Ag-900) as the sorption media. Data from thin beds (½ inch depth) was used to evaluate the effects of process variables (temperature, gas flow rate, co-constituent gases) on capture efficiency. These tests were attractive since a lot of information could be obtained in a relatively short time period – a matrix on a single feed gas with varying sorption temperatures and gas flow rates could be completed in a day (Haefner 2009).

1.1 FY 2010 Objectives

The emphasis this year was to collect information on deep beds to obtain effluent breakthrough curves. Sorbents targeted included the unaltered, commercial sorbent IONEX Ag-900 (silver mordenite from Molecular Products, Inc.) and a silver mordenite supplied by Oak Ridge labeled as "AgZ natural." The base sorbent materials are very similar. A BET analysis estimated the surface area of the IONEX to be about 21 m^2/g, while that for "AgZ natural" was 18 m^2/g. Pore diameter distributions were essentially the same with about 56% of the pores in the 20-80 nm range (Garn 2009). The silver content was also very similar at about 9 wt%. The primary difference was that "AgZ natural" had undergone a hydrogen reduction step to convert silver to the zero valence state. A direct comparison of these sorbents should be possible by subjecting them to similar process conditions.

2. EXPERIMENTAL APPROACH

Test gases were prepared to mimic off-gases that would evolve from a reprocessing plant, however, only non-radioactive surrogates were used. The iodine source was methyl iodine (MeI) since this compound could easily produce the levels of iodine desired. Initially it had been hoped to use elemental iodine, however, we were not able to generate a reliable concentration of ~25 ppm I_2 for an extended period. This level was desirable to achieve breakthrough in a reasonable time frame. Co-constituent gases were water vapor, NO, and NO_2, using a carrier gas of nitrogen or air. The test gas, having a known feed concentration, was routed to the adsorption column where effluent samples were withdrawn and analyzed for iodine at known times. The test was operated until the effluent iodine rose to an arbitrary breakthrough value. A segmented bed allowed gas samples to be withdrawn from various bed heights, so in theory, several breakthrough curves could be obtained from a single run.

2.1 Equipment Design and Setup

Figure 1 is a schematic diagram of the experimental set-up. All test gases are supplied from high pressure cylinders located in a gas usage building separate from the lab. Permanent stainless steel lines bring the gases into the lab where mass flow controllers (Sierra Instruments) with ranges of 0 to 150, 0 to

500, and 0 to 1000 cm^3/min (SCCM) are used to adjust flows. All flow controllers are calibrated prior to use.

Methyl iodide is supplied in cylinders at about 100 ppm CH_3I in nitrogen. Gas cylinders also supply NO and NO_2 at levels up 5000 ppm in nitrogen. Humidified air is produced by passing air through a fritted glass bubbler submerged in a constant temperature bath. A thermocouple in the headspace of the bubbler provides the temperature of the saturated gas. All test gases are combined in a glass mixing chamber prior to routing to the adsorption columns. Gas feed lines carrying iodine are Teflon, heat traced and maintained at 100-110°C.

The adsorption columns are fabricated from sections (spool pieces) of 0.745" ID glass tubing. Each spool piece has ground glass end fittings, a coarse glass frit support to hold bed material, and a sampling port. Spool pieces are interchangeable and when connected together, form the adsorption column. Spool pieces ranging in length from about 3 to 6 inches are available.

As depicted in Figure 1, two columns are used and both are placed in an oven maintained at the target sorption temperature of 150°C. The first column has 3 sections with bed depths of about ½, 1½, and 2 inches, while the second has a single section and can accommodate a bed up to 4 inches deep. Sample ports in the headspace of each section allow effluent gas samples to be drawn from each bed, i.e., interbed gas samples. All sample ports are connected to manual sampling valves located on a manifold outside the oven. Sampling lines can be connected to either gas bubblers (for elemental iodine capture/analysis) or to an on-line gas chromatograph (for methyl iodine analysis).

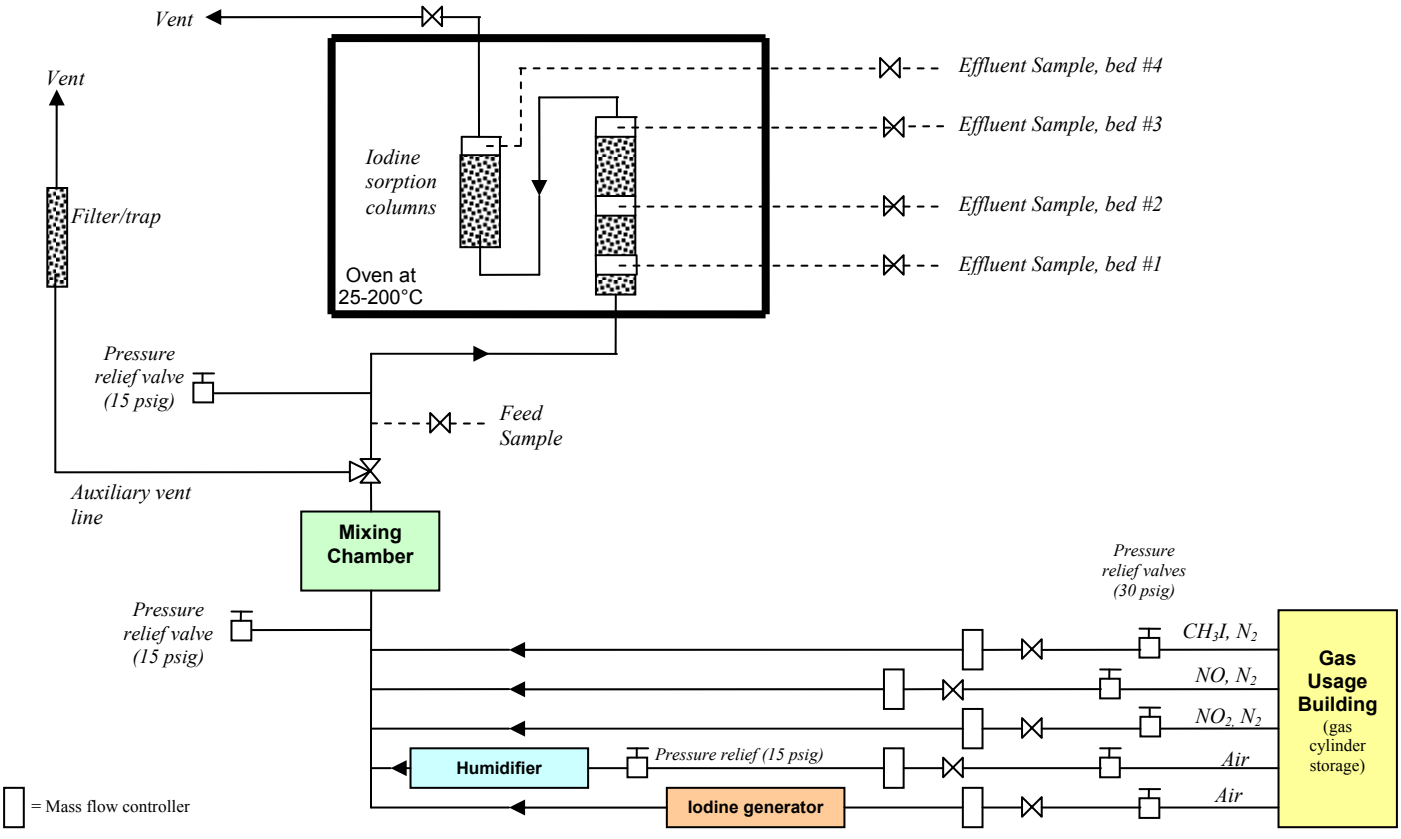

Figure 1. Schematic of Experimental Set-up.

2.2 Iodine Analysis

Elemental iodine was determined by bubbling a known quantity of gas through bubblers (midget impingers) containing 25 ml of 0.1 M NaOH solution. The resulting liquid was then analyzed by ICP-MS. Detection levels for the scrub liquid is typically ~0.5 ppb, which corresponds to a gas concentration of ~0.08 ppb using the following expression:

$$C_{I_2,gas} = \frac{A \times 24.45}{V_{gas} \times 253.8} \quad (1)$$

And $A = C_{liq} \times V_{liq}$ (2)

where: A = amount of iodine, µg I_2

$C_{I_2,gas}$ = iodine concentration in gas, ppmv I_2

C_{liq} = iodine concentration in liquid, µg I/mL

V_{gas} = volume of gas passed through bubbler, L

V_{liq} = volume of scrub liquid in bubbler, mL

24.45 = molar volume of air at 25°C and 760 mmHg, L/mole

253.8 = molecular weight of I_2, g/mole

Methyl iodine (MeI) was determined by a Hewlett-Packard model 5890 Series II gas chromatograph (GC) installed in-line with the sampling manifold, thereby allowing near real-time analysis of MeI. An Rt-Q-BOND fused silica capillary column was used in the GC. The GC was equipped with an electron capture detector, which is very sensitive towards halogenated organic compounds and is well suited for MeI analysis.

Initially, it was desired to quantify MeI at the lowest concentration possible since this would be used to assess the capture efficiency (decontamination factor) of fresh adsorbent. A calibration curve was prepared using standards of 5, 50, and 100 ppb methyl iodide, thereby allowing quantification of gas samples to 5 ppb (even lower when response factors are used). After some use, methyl iodine will begin passing through the sorbent without being captured in accordance with typical breakthrough behavior. Eventually, anticipated levels of MeI should approach that of the feed. It was also desired to analyze samples along the breakthrough curve using the on-line GC. To determine MeI concentrations accurately at higher concentrations, a second calibration curve was prepared for use at the higher levels using standards in the range of 3,000 to 25,000 ppb.

Two gas chromatograms are shown in the Appendix and several features of their iodine detection behavior are worth noting. The first is the retention time for methyl iodine, which was about 2.5 minutes. A single, prominent peak was typical when MeI levels were at ppm levels. However, at low MeI levels when the detector resolution was high, several "ghost peaks" were consistently present and appeared earlier than MeI. Our suspicion is that some other organic compounds are forming or being released from Teflon components used in the experimental system. We did inject a sample of methanol and it eluted at about 1.45 minutes, which is consistent with one of the early peaks we were seeing.

3. KEY RESULTS FROM FY 2010 WORK

Three extended adsorption runs lasting from 3600 to 9500 hours were performed. The objective was to use conditions representative of the dissolver off-gas and operate until the effluents reached an arbitrary defined "breakthrough" value. A breakthrough value of 250 ppb from the second bed was used as the basis for a "dynamic sorption capacity," defined as the ratio of the mass methyl iodine sorbed divided by the mass of sorbent. It is used as a convenient tool to compare performance of different sorbents. It was estimated using the following expression:

$$D_{@250} = (V \times t_{250} \times C) \div m_{sorb} \quad (3)$$

where C = concentration of CH_3I in feed (mg/cm^3)

$D_{@250}$ = estimated sorption capacity at effluent concentration of 250 ppb

m_{sorb} = mass of sorbent (g)

t_{250} = time to 250 ppb in effluent (min)

V = volumetric flow rate of gas (cm^3/min)

The run time to reach 250 ppb was determined by using a best fit of the data near the breakthrough point. A breakthrough value of 10-15% or more of the feed concentration was also desirable from a dynamic modeling perspective as this can be used to corroborate and/or adjust computer models to predict adsorption behavior for the iodine/DOG system.

3.1 Run #1

An initial breakthrough test with IONEX Ag-900 was conducted using the conditions summarized in Table 1. Effluent methyl iodine levels from the second bed were monitored throughout the run and shown in Figure 2. Initial effluent levels from the fresh bed were consistently below 5 ppb, corresponding to a DF of greater than 5000. The run was continued until the effluent methyl iodine level was consistently greater than 250 ppb.

At the conclusion of the run, the bed material was removed and the weights recorded – this information is shown in Table 2. The silver content is based on the sorbent manufacturer's specification sheet indicating 9.5% silver. For preparation of this table, the change in sorbent weight is assumed to be due to iodine adsorption – we anticipate little retention of water at the operating temperature of 150°C. The percent silver utilization was estimated assuming formation of the compound AgI. The data suggests that about half of the silver in the thin first bed is being utilized for iodine retention; as expected, the utilization drops off in subsequent beds. The total weight gain of beds 1, 2, and 3 (0.547 grams) was compared to the total iodine passed through the system, based on flow rate, time, and methyl iodine concentration. The weight gain accounted for 60.5% of the iodine passed to the system.

Table 1. Conditions and results for breakthrough Run #1.

Parameter	Value/information
Sorbent	IONEX Ag-900
Temperature	150°C
Gas flow rate (total)	1200 SCCM
Approach velocity	14.1 ft/min
Feed composition	25 ppm CH_3I 1.5 wt% water (dew pt 63.1°F) 494 ppm NO 500 ppm NO_2
$D_{@250}$	55.2 mg CH_3I/g sorbent
$t_{@250}$	4000 minutes

Table 2. Initial and final sorbent weights plus estimated silver utilization for Run #1.

	Initial wt sorbent (g)	Final wt sorbent (g)	Wt change (g)	Wt% change	Silver wt† (g)	Silver utilization (%)
Bed 1	3.39	3.58	0.19	5.5	0.32	50.6
Bed 2	10.38	10.67	0.29	2.7	0.99	24.7
Bed 3	12.26	12.33	0.067	0.5	1.16	4.9

† Based on IONEX specification sheet value of 9.5%

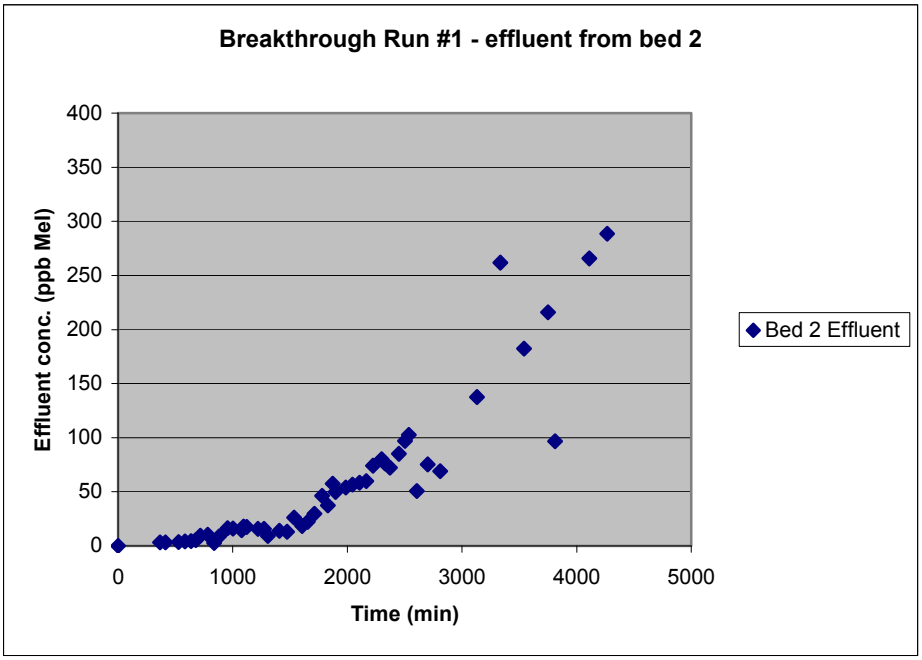

Figure 2. Effluent methyl iodine data for Run #1.

3.2 Run #2

A second test was conducted using silver mordenite that had undergone silver reduction by passing a hot stream of hydrogen/nitrogen through the sorbent. This material was received from Oak Ridge and labeled as "Natural AgZ." The test conditions for the run are given in Table 3, and the resulting data are plotted in Figures 3 and 4. Significantly more data was acquired for Run #2, including more extensive monitoring – most notably the effluents from beds 1 and 3. The data shown in Figure 3 indicates that bed 1 is picking up only about half of the MeI being fed into it – the mass transfer zone is spread well beyond that bed depth. In fact the data in Figure 4 shows that measurable levels of MeI are even leaving the fresh second bed. It is anticipated that the effluent level should continually rise until reaching the same value as the feed. The system was operated for over 3600 minutes – it was terminated prematurely when the gas cylinders of MeI and NO_2 were running low. Several mass flow controllers were also approaching their calibration expiration dates and were removed from service for recalibration.

Table 4 shows the increase in sorbent weight for the various beds. The trends are similar to those in the first test: the silver utilization dropping as the bed depth increases. The weight gain shown by Beds 1, 2, and 3 accounted for an estimated 94.2% of the total iodine passed to the system.

Table 3. Conditions and results for breakthrough Run #2.

Parameter	Value/information
Sorbent	Natural AgZ (reduced)
Temperature	150°C
Gas flow rate (total)	1200 SCCM
Approach velocity	14.1 ft/min
Feed composition	33.7 ppm CH_3I 1.95 wt% water (dew pt 71.2°F) 833 ppm NO 833 ppm NO_2
$D_{@250}$	59.5 mg MeI/g sorbent
$t_{@250}$	3030 min

Table 4. Initial and final sorbent weights plus estimated silver utilization for Run #2.

	Initial wt (g)	Final wt (g)	Wt change (g)	Wt% change	Silver wt† (g)	Silver utilization (%)
Bed 1	2.99	3.15	0.16	5.2	0.28	46.5
Bed 2	10.06	10.47	0.41	4.0	0.96	36.2
Bed 3	13.07	13.30	0.23	1.7	1.24	15.5

† Based on 9.5% silver content

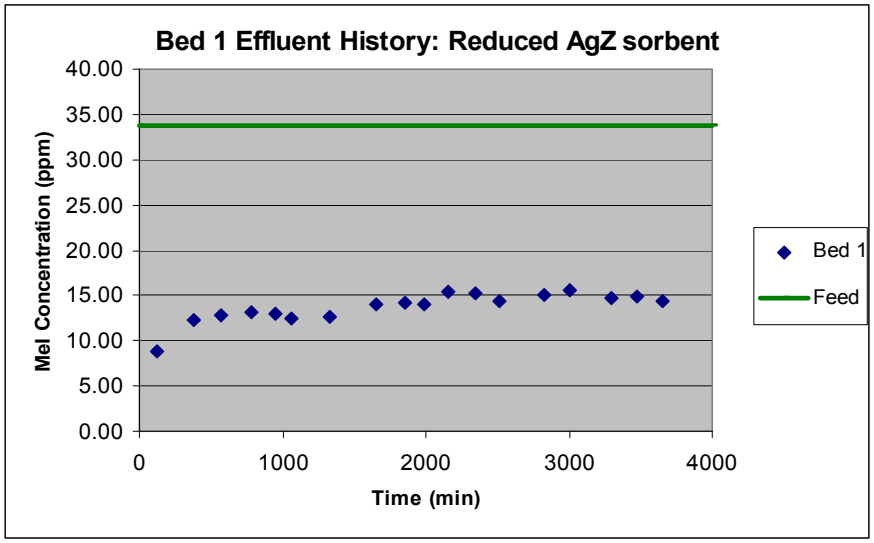

Figure 3. Effluent methyl iodine levels from Bed 1 (Run #2).

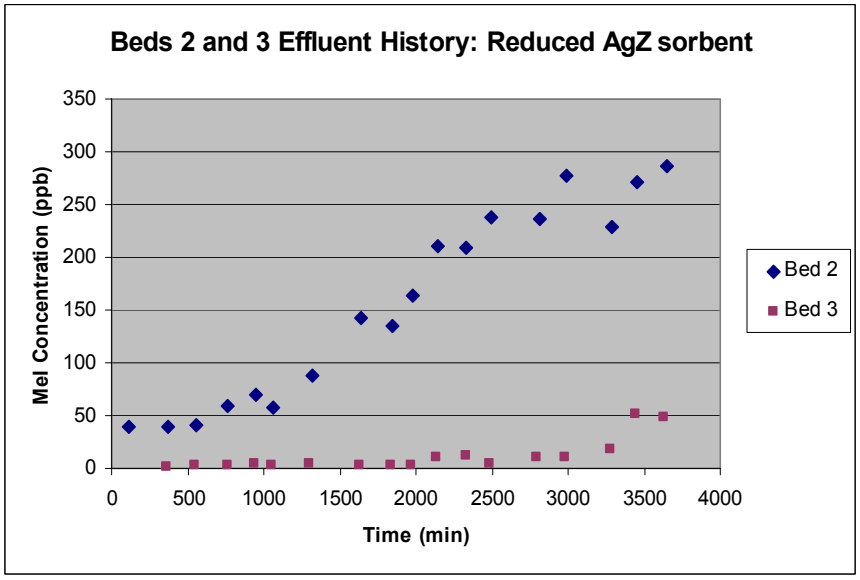

Figure 4. Effluent methyl iodine levels from Beds 2 and 3 (Run #2).

3.3 Run #3

A third breakthrough run was conducted with essentially identical conditions as Run #2 except that a different sorbent was used – this time with unaltered IONEX Ag-900. The operating conditions are summarized in Table 5. In addition to on-line MeI monitoring, scrub samples were drawn from the second bed at a frequency of one sample per day, the analysis of which indicates the level of I_2. The MeI effluent data are plotted in Figures 5 and 6. Similar to previous runs, bed 1 is removing about half of the MeI. On-line MeI data was gathered for nearly 9500 minutes (158 hrs), however, the second bed effluents seemed to nearly level off after modestly rising for a period of time. The anticipated steep rise of a MeI breakthrough did not occur, which was troubling, however, the scrub data revealed the fate of the iodine.

In Figure 7 the scrub data over time is plotted for the Bed 2 effluents. The caustic scrub retains elemental iodine and very little MeI. This data indicates that as the second bed becomes spent, iodine is being **emitted as I_2** even though the iodine source is CH_3I. This suggests that (moderately warm, 150°C) silver mordenite may be catalyzing a reaction where CH_3I is converted to I_2. The organic portion of the methyl iodine would also generate some compound(s) and this may explain the appearance of additional peaks seen on the chromatograms. Figure 7 shows that a significantly higher concentration of iodine is being emitted than MeI analysis would indicate – at the end of the test, the Bed 2 effluent was about 70% of the feed concentration.

Table 6 was prepared to show the comparative amounts of MeI and I_2 being emitted from Bed 2 as a function of time. Initially almost all the iodine is MeI (~96%), but as the sorbent is used, a progressively higher percent is elemental. In fact, at the end of the test, only about 3% of the emitted iodine occurs as MeI. Figure 8 is a plot of the total iodine emitted (from Bed 2) as a function of time and shows a more typical breakthrough curve.

At the conclusion of the test, the column was disassembled and the weights of all 4 beds determined. The results are summarized in Table 7. As expected the silver utilization was highest in the first bed, and then dropped off in successive beds. The total weight gained by the sorbent accounted for about 89% of the iodine passed to the system.

Table 5. Conditions and results for breakthrough Run #3.

Parameter	Value/information
Sorbent	IONEX Ag-900
Temperature	150°C
Gas flow rate (total)	1200 SCCM
Approach velocity	14.1 ft/min
Feed composition	33.3 ppm CH_3I 1.95 wt% water (dew pt 71.2°F) 833 ppm NO 833 ppm NO_2
$D_{@250}$	71.4 mg MeI/g sorbent
$t_{@250}$	3920 min

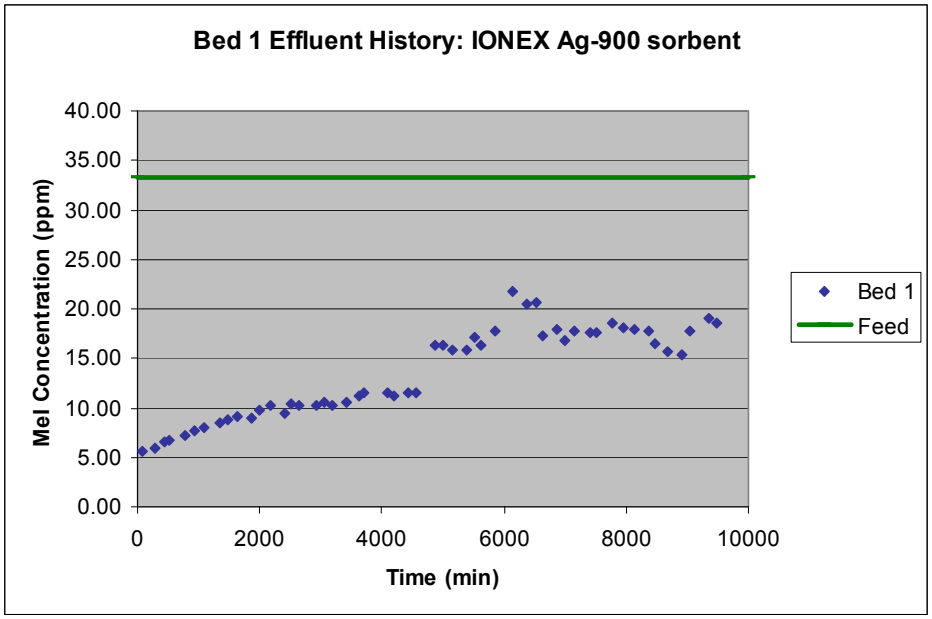

Figure 5. Effluent methyl iodine levels from Bed 1 (Run #3).

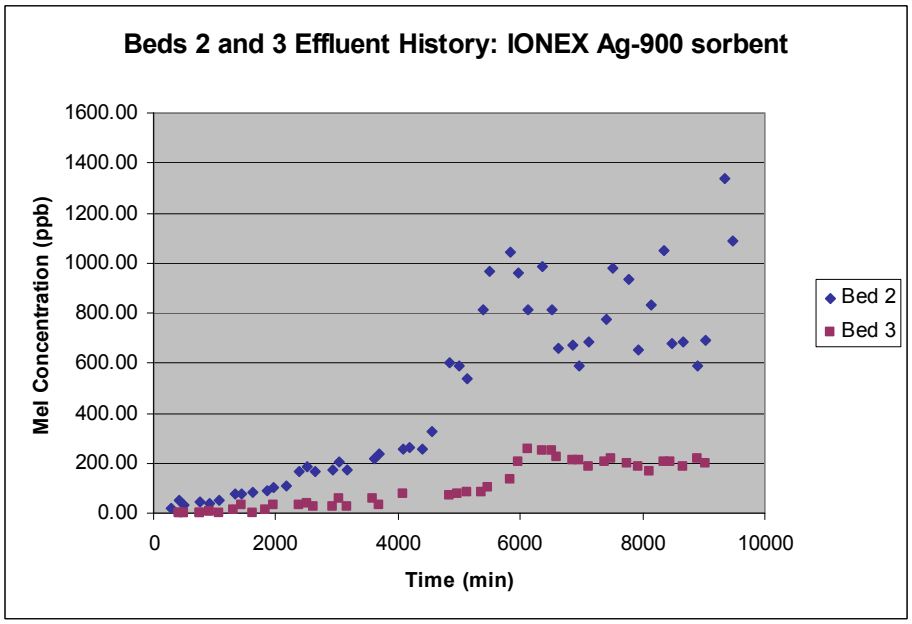

Figure 6. Effluent methyl iodine levels from Beds 2 and 3 (Run #3).

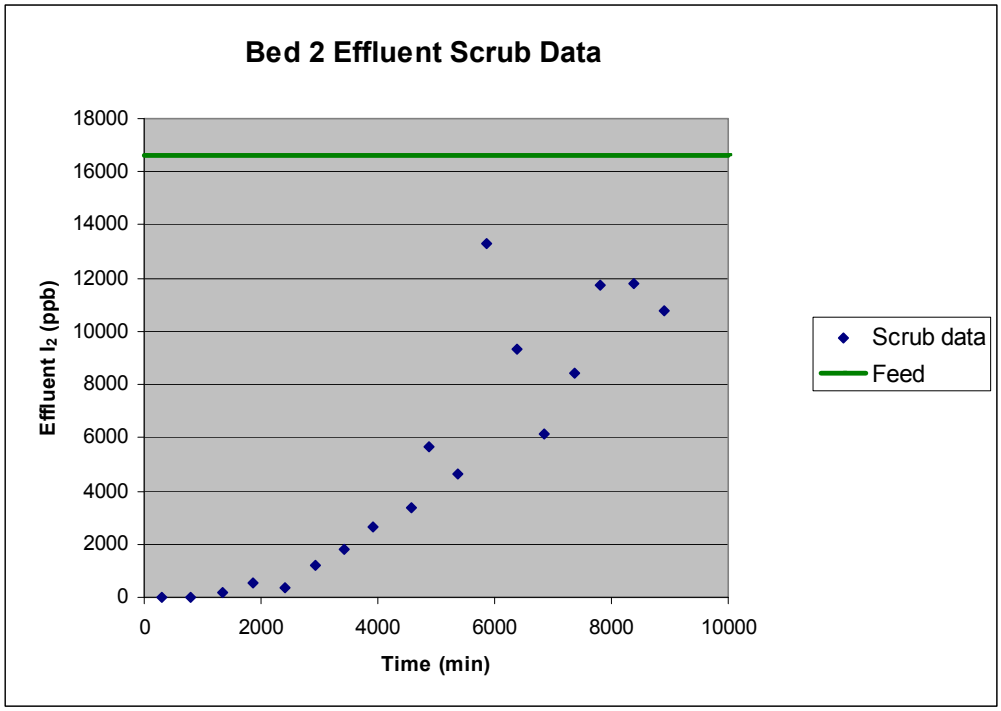

Figure 7. Effluent I_2 levels from Bed 2 (Run #3) as determined by scrub samples. (Feed line on the plot assumes all iodine in MeI is converted to I_2.)

Table 6. Comparative amounts of methyl iodine and I_2 emitted from Bed 2.

Time from start: Day No. - (min)	Effluent MeI – daily ave. (ppb)	Effluent I_2 (ppb)	Total iodide in effluent (ppb)	% iodine from MeI
1 - (303)	36.4	0.80	38.0	95.8
2 - (789)	39.4	9.61	58.6	67.2
3 - (1348)	67.6	173	414	16.3
4 - (1871)	92.4	523	1140	8.1
5 - (2404)	152	365	882	17.3
6 - (2941)	181	1,190	2,553	7.1
9 - (4572)	282	3,360	7,000	4.03
12 - (5876)	719	13,300	27,340	2.63
15 - (7369)	812	8,410	17,600	4.60
18 - (8916)	654	10,800	22,300	2.94

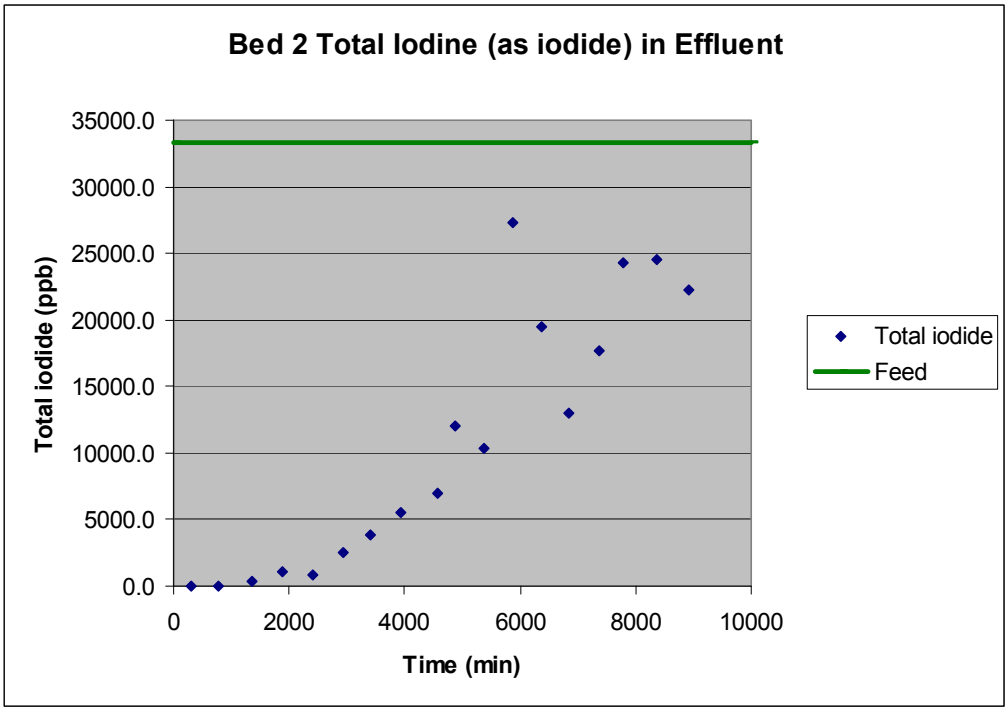

Figure 8. Total iodine (MeI plus I_2 from scrub samples) in effluent from Bed 2 (Run #3).

Table 7. Initial and final sorbent weights and the estimated silver utilization for Run #3.

	Initial wt (g)	Final wt (g)	Wt change (g)	Wt% change	Silver wt† (g)	Silver utilization (%)
Bed 1	3.31	3.53	0.22	6.4	0.31	59.5
Bed 2	10.61	11.16	0.55	5.0	1.01	46.4
Bed 3	13.77	14.42	0.65	4.6	1.31	42.2
Bed 4	40.27	40.76	0.49	1.2	3.83	10.9

† Based on IONEX specification sheet value of 9.5%

4. INTERIM CONCLUSIONS

Three breakthrough runs using silver mordenite sorbents were conducted and a dynamic sorption capacity estimated based on MeI analysis from a 2" bed. However, it is now believed the data for the first 2 runs is incomplete because the contributions from elemental iodine were not included. Although the only source of iodine was MeI, elemental iodine was generated within the sorbent bed, presumably from a recombination reaction likely catalyzed by silver mordenite. On-line effluent analysis with a GC was only capable of analyzing MeI, not I_2. Scrub samples drawn during Run #3, which are specific for I_2, show significant levels of I_2 being emitted from a partially spent Ag-mordenite bed. By combining MeI and I_2 analyses, a well defined total iodine breakthrough curve can be generated for Run #3.

At the conclusion of Run #3 (IONEX Ag-900 was the sorbent) the effluent level from Bed 2 was approaching 70% of the feed concentration. The leading bed (Bed 1) had an estimated average loading of 66 mg I/g sorbent, Bed 2's was 52 mg I/g. The corresponding silver utilizations (assuming formation of AgI) were about 59% and 46%, respectively. The spent sorbents are being sent to Sandia National Laboratories for confirmatory analysis of iodine and silver utilization as well as source material for waste form development.

Based on the FY10 studies, the experimental procedure will be modified and include:

- more extensive sampling with bubblers (scrub samples) if methyl iodine is the source of iodine to capture nascent I_2
- revisiting the generation and use of I_2 in the feed at ~20 ppm levels. In our earlier attempts, we used a column packed with iodine crystals heated to 50-70°C, but the resulting I_2 levels were inconsistent, likely due to channeling in the bed when the iodine flakes formed large chunks. Building on the experience at Oak Ridge, lower iodine column temperatures (20°C) will be used in an attempt to mitigate the clumping problems
- amending our work control documentation to allow continuous operation 24 hours a day.

5. PROPOSED PATH FORWARD

It is anticipated that several other adsorbents will be evaluated in the INL test configuration. In addition to the silver mordenite tested to date (IONEX Ag-900 and "AgZ natural"), Oak Ridge has supplied an "AgZ light phase" silver mordenite which has undergone silver reduction processing. This material has a higher silver content (12-15% vs. 9%), smaller average pore diameter, and higher BET surface area (62 vs. 20 m^2/g) than the IONEX or "AgZ natural." Sandia has developed an adsorbent material (called NC-77) and has offered to supply enough material for testing. In addition, PNNL is developing sorbents and these may be candidates as well. Finally, all of our deep bed tests have used gas rates of 1200 SCCM and a sorption temperature of 150°C. Therefore, we will investigate variations (higher flow rate and lower sorption temperature) from this baseline to determine sorption characteristics at other operating conditions.

6. References

Garn, T. G., 2009, e-mail to D. R. Haefner, Subject: Final BET analyses, Dec. 22, 2009.

Haefner , D. R., and N. Soelberg, 2009, *Experimental Sorption Testing of Elemental Iodine on Silver Mordenite*, Idaho National Laboratory report: INL/EXT-09-16837.

Appendix A

Representative Gas Chromatograms

On the following 2 pages are chromatograms from Run #3. The first page is a chromatogram from Bed #1 and shows a single prominent peak for methyl iodine eluted at about 2.5 minutes. The chromatogram on the second page is from the second bed and shows the methyl iodine peak at 2.5 minutes, but also shows two peaks for compounds eluted prior to methyl iodine. Methanol was injected into the GC and was eluted at ~1.5 minutes – about the same time as one of the earlier peaks. More elaborate means to positively identify the two phantom peaks was not undertaken. It is believed the phantom peaks are organic reaction products formed as the methyl iodine passes onto the silver mordenite surface.

Data File C:\HPCHEM\1\DATA\MGJ\05041006.D Sample Name: CH3I test

Test #3 for comparisen to ORNL reduced sorbent. Gases 5
000 ppmm NO,NO2 and 100 ppm methyl iodine.

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Acq. Instrument : Instrument 1                    Inj Volume : Manually
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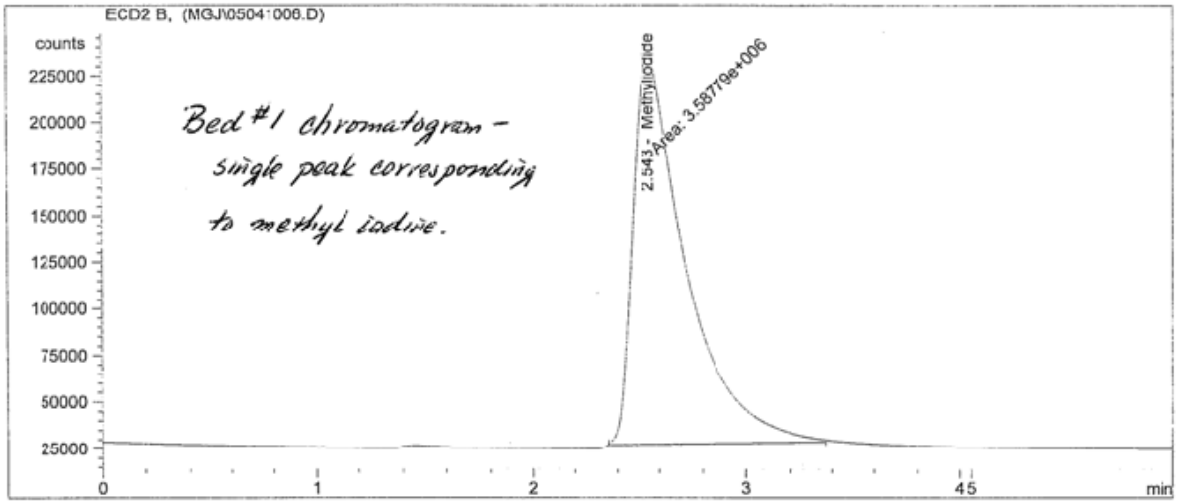


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Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD2 B,

RetTime Sig Type     Area      Amt/Area    Amount   Grp   Name
 [min]             counts*s                [ppb]
-------|--|------|----------|----------|----------|--|---------------
  2.543  1 MM     3.58779e5  2.95614e-3 1.06419e4     Methyliodide

Totals :                                 1.06419e4

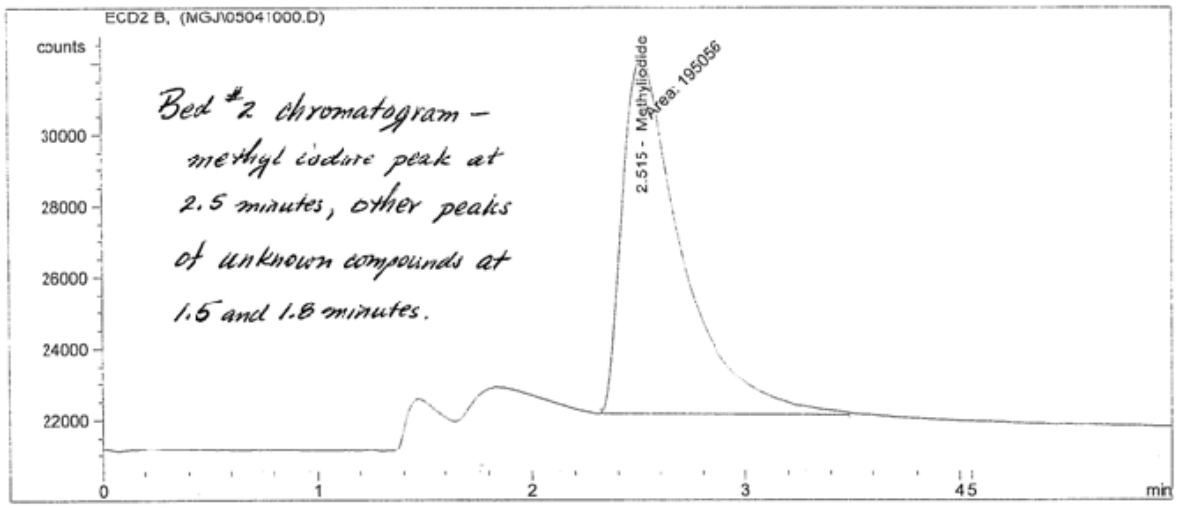
 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```


Figure A-1. Chromatogram for Bed 1 effluent.

Data File C:\HPCHEM\1\DATA\MGJ\05041000.D Sample Name: CH3I test

Test #3 for comparison to ORNL reduced sorbent. Gases 5
000 ppmm NO,NO2 and 100 ppm methyl iodine.

```
Injection Date  : 5/4/2010 9:54:24 AM
Sample Name     : CH3I test                        Location :   -
Acq. Operator   : tlw
Acq. Instrument : Instrument 1                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\1\METHODS\NPD_ECD.M\IODIDE1.M
Last changed    : 4/21/2010 8:14:41 AM by tlw
Analysis Method : C:\HPCHEM\1\METHODS\NPD_ECD.M\CH3IFULL.M
Last changed    : 5/27/2010 3:06:10 PM by tlw
```



External Standard Report

```
Sorted By             :      Retention Time
Calib. Data Modified  :      5/27/2010 3:05:59 PM
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD2 B,

RetTime Sig Type     Area      Amt/Area    Amount   Grp   Name
 [min]            counts*s                 [ppb]
-------|--|------|----------|----------|----------|--|--------------
  2.515  1 MM     1.95056e5  1.79452e-3  350.03174     Methyliodide

Totals :                                 350.03174

 Results obtained with enhanced integrator!
```

*** End of Report ***

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Figure A-2. Chromatogram for Bed 2 effluent.