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**To:** Stephen Kung, Ken Marsden, and Kimberly Gray

**From:** Allison T. Greaney, Randy O. Ngelale, Katie R. Johnson

As part of the FY22 Material Recovery and Waste Form Development campaign, ORNL has prepared a new set of iodine-bearing sorbents.. Four canisters were machined and loaded with ~30 g each of iodine-bearing silver mordenite at varying degrees of sorbent saturation via hot isostatic pressing (HIP)

Reduced AgZ was used as the iodine sorbent for testing. The sorbent is supplied by IONEX as Ag-400, and the silver is further reduced in-house under a flow of 4% $H_2$/argon in an oven at 270°C for 10 days. This effectively reduces $Ag^{1+}$ to $Ag^0$, better facilitating iodine capture to form silver iodide in an off-gas column.

Iodine was loaded onto 50 g of AgZ under static conditions at 150°C. A piece of steel mesh was fitted into an ACE Glass column that sealed at both ends. Then, 50 g of AgZ were loaded onto the steel mesh, which was enough to cover the mesh in a layer one to two pellets deep. Iodine crystals ($I_2$) with varying masses (0.73 – 7.36 g) were placed at the base of each column to achieve varying degrees of sorbent saturation. The column was sealed and placed in an oven at 150°C for 10 days. Four batches of AgZ were loaded with iodine following this method to create samples: HIP-22A, HIP-22B, HIP22-C, and HIP22-D (Table 1).

**Table 1. Masses of AgZ and iodine used for each of the four samples before iodine loading, after iodine loading, and after purging with nitrogen.**

| Sample | Mass AgZ (g) | Mass $I_2$ (g) | Mass AgZ-I (g) | Mass AgZ-I (g) | Apparent mass $I_2$ loaded (g) | Mass AgZ-I loaded into canister |
|---|---|---|---|---|---|---|
| | | | (Pre-purge) | (Post-purge) | | |
| HIP22-A | 49.93 | 7.36 | 53.8913 | 52.76 | 2.83 | 32.06 |
| HIP22-B | 50.09 | 3.52 | 53.552 | 52.65 | 2.56 | 34.23 |
| HIP22-C | 49.95 | 1.738 | 52.153 | 51.57 | 1.62 | 32.64 |
| HIP22-D | 49.96 | 0.731 | 51.322 | 50.71 | 0.75 | 32.39 |

After 10 days, the iodine-loaded AgZ was weighed beforepurging with nitrogen gas to remove water and other physiosorbed components. To purge the sorbent, the AgI-Z was loaded into 1 in. diameter columns that were maintained at 150°C under 1 LPM flow of $N_2$ for 2 days. To accurately determine the iodine content of the AgZ, aliquots of each of the four samples will be analyzed for iodine concentration using neutron activation analysis (NAA) at ORNL's High Flux Isotope Reactor. These data will be sent with the HIP samples to researchers at Argonne National Laboratory for experiments on iodine-bearing wasteforms.

Canisters were designed to contain ~30 g of AgZ for HIP. The canisters are 2 in. long by 1.5 in. in diameter and made of 316 stainless steel (Figure 1). After filling with ~30 g of AgZ, the canisters are welded closed with two trepan lids and vacuum sealed using a ⅛ in. tube port welded to the top lid. The samples were pressed at American Isostatic Pressing Inc. at 900°C and 300 MPa with a 3 h hold time. During pressing, Sample D opened, possibly due to a failure in the weld.

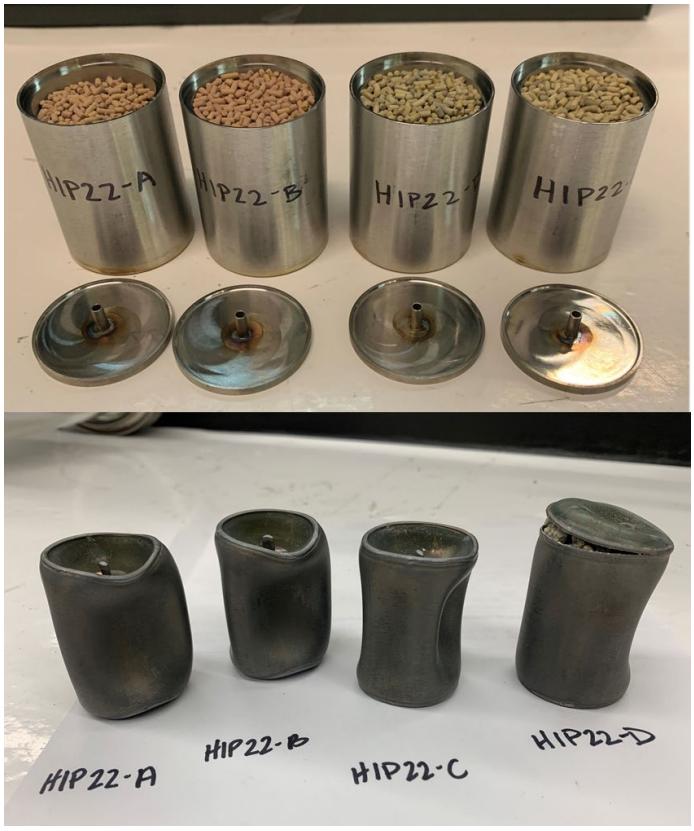


**Figure 1. HIP canisters before and after HIP.**

These HIP wasteforms are being sent to Argonne National Laboratory for wasteform stability experiments. Please do not hesitate to contact me with any questions: greaneyat@ornl.gov.

Sincerely,

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