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SECTION A. Project Title: Ceramic Sensor Experiments in the Analytical Laboratory

SECTION B. Project Description:

The purpose of the proposed action is to research the development of a sensor that has the ability to monitor real- or near real-time concentrations of actinide ions in electrorefiner salt systems. The sensor would provide a way to ensure that plutonium (Pu) is not diverted during electrorefining operations by providing the ability to monitor the Pu concentration and the ratio of the Pu to uranium (U) concentrations in the electrolyte.

Electrically conductive ceramics would be evaluated as candidate solid electrolyte materials for the sensor, including sodium beta alumina. Activities needed to reach this goal include 1) ion exchange at high temperatures, 2) chemical compatibility and sensor tests in molten salts, and 3) conductivity measurements at high temperatures.

The proposed action would use the Hot Uniaxial Press (HUP) Furnace located in the Casting Lab of the Analytical Laboratory (AL) and operate the HUP Furnace at temperatures up to 950°C. The HUP Furnace is located inside the Casting Lab glovebox--an inert atmosphere. A quartz, alumina, or stainless steel crucible would be filled with experiment materials, as described below. The crucible would then be loaded into a stainless steel liner and set in one of the HUP Furnace inserts. The insert would then be lowered into the HUP Furnace well and the furnace turned on. Experiments would remain in the furnace anywhere from several hours to several days.

The following activities would occur as part of the proposed action:

Ion Exchange

In order to function as a solid electrolyte, the sodium in the beta alumina must first be ion exchanged for the species of interest, in this case Pu. Plutonium chloride (PuCl₃) would be used as the source of Pu. For this action, the crucible (<100 ml) would be filled with $PuCl_3$ (< 50 g) and the ceramic sensor material buried in the $PuCl_3$ then lowered into the HUP Furnace. This test could also be performed using U/UCl₃. The maximum amount of UCl₃ would be 200 g.

After the ceramic sensor material has been ion-exchanged, it would be sectioned for analysis or used for chemical compatibility tests, conductivity measurements, or sensor tests.

Annealing

Due to stress on the material during ion exchange, micro-cracks propagate throughout the material compromising its mechanical integrity. Annealing of the ceramic material heals stress-related micro-cracks and other imperfections of the ceramic material. Annealing would consist of placing the ion-exchanged ceramic in a crucible or holder and "baking" in the HUP furnace.

After the annealing process the ceramic sensor would be sectioned for analysis or used for chemical compatibility tests, conductivity measurements or sensor tests. Not all ion exchanged sensor material would be annealed.

Chemical Compatibility

Material used to fabricate the sensor needs to be able to withstand submersion in the molten electrolyte. For this test, the ionexchanged sensor material would be loaded into a crucible (<100 ml) with eutectic LiCl-KCl and placed in the HUP Furnace. The eutectic salt could be used on its own or could be mixed with UCl₃ and/or PuCl₃. Depending on the test, other rare earth chlorides found in the electrorefiner could be added to the mixture.

After chemical compatibility studies, the ceramic sensor material would be sectioned for analysis or used for conductivity measurements.

Conductivity Measurements

A critical aspect for a sensor is how fast it can detect changes. Conductivity measurements on the ceramic material being considered for a sensor provide information on how the ability of the material to conduct the ions of interest in a timely fashion changes as the material is processed. These measurements can be done on the original ion-exchanged sensor disc, after it has been annealed, or after chemical stability tests.

Conductivity measurements would include platinum paint being affixed on the sides of the disc then curing the painted disc in the HUP Furnace. Once the paint has set, the disc would be setup with electrodes to measure conductivity across the disc. A ProboStat would be used to ensure the electrodes make good contact with the surface of the disc. A ProboStat measures electrical properties and uses spring force to sandwich the ceramic disc between two Pt electrodes. After the electrodes have been connected to the internal electrical feed-throughs, the end of the ProboStat holding the disc would be lowered into the furnace. A potentiostat connected to the external electrical feed-throughs would be used to make conductivity measurements at different temperatures.

Sensor Testing

Beta alumina tubes would be fashioned into sensors after ion-exchange. The largest tube would be 4 in long with an outside diameter of 10 mm and a wall thickness of 2 mm. A small amount (< 2 g) of a reference solution (i.e., LiCI-KCI-5 wt% PuCl₃) would be loaded into the tube with a wire making electrical contact with the solution. This sensor would be lowered with two other electrodes into a crucible (< 100 ml) containing a salt electrolyte (i.e., LiCI-KCI-PuCl₃) then loaded into the HUP Furnace. Different chlorides would be added

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throughout the test to change the salt composition. The sensor and the electrodes could be pulled up slightly above the salt surface to avoid being frozen into the salt when the furnace is turned off. After the test, the tube would be sectioned for analysis.

SECTION C. Environmental Aspects or Potential Sources of Impact:

<u>Air Emissions</u> - This work has the potential to generate emissions of radionuclides and minor amounts of toxics. Because of the amount of Pu being processed an Air Permitting Applicability Determination will be completed.

<u>Generating and Managing Waste</u> - This work is expected to generate small amounts of contact-handled transuranic (TRU) waste and low-level radioactive waste. Most TRU materials would be reused. The maximum amounts of plutonium to be used, and that could be considered waste, is 38 g. The maximum amount of uranium is approximately 52 g. All radioactive waste has a path for disposal. Very small amounts of mixed waste may also be generated. Waste will be managed by Waste Generator Services (WGS).

SECTION D. Determine the Recommended Level of Environmental Review (or Documentation) and Reference(s): Identify the applicable categorical exclusion from 10 Code of Federal Regulation (CFR) 1021, Appendix B, give the appropriate justification, and the approval date.

For Categorical Exclusions (CXs), the proposed action must not: (1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, or similar requirements of Department of Energy (DOE) or Executive Orders; (2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment or facilities; (3) disturb hazardous substances, pollutants, contaminants, or Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)-excluded petroleum and natural gas products that pre-exist in the environment such that there would be uncontrolled or unpermitted releases; (4) have the potential to cause significant impacts on environmentally sensitive resources (see 10 CFR 1021). In addition, no extraordinary circumstances related to the proposal exist that would affect the significance of the action. In addition, the action is not "connected" to other action actions (40 CFR 1508.25(a)(1) and is not related to other actions with individually insignificant but cumulatively significant impacts (40 CFR 1608.27(b)(7)).

References: 10 CFR 1021, Appendix B to Subpart D item B3.6 "Small-scale research and development, laboratory operations, and pilot projects"

Final Environmental Impact Statement for the Waste Isolation Pilot Plant (WIPP) (DOE/Environmental Impact Statement [DOE/EIS]-0026, October 1980) and Final Supplement Environmental Impact Statement for the Waste Isolation Pilot Plant (SEIS-I) (DOE/EIS-0026-FS, January 1990)

Final Waste Management Programmatic Environmental Impact Statement [WM PEIS] (DOE/EIS-0200-F, May 1997) and Waste Isolation Plant Disposal Phase Supplemental EIS (SEIS-II) (DOE/EIS-0026-S-2, Sept. 1997)

Justification: The proposed research and development (R&D) activities are consistent with CX B3.6 "Siting, construction, modification, operation, and decommissioning of facilities for small-scale research and development projects; conventional laboratory operations (such as preparation of chemical standards and sample analysis); small-scale pilot projects (generally less than 2 years) frequently conducted to verify a concept before decomonstration actions, provided that construction or modification would be within or contiguous to a previously disturbed area (where active utilities and currently used roads are readily accessible). Not included in this category are demonstration actions, meaning actions that are undertaken at a scale to show whether a technology would be viable on a larger scale and suitable for commercial deployment."

The impacts of transporting and disposing of waste resulting from defense activities that was placed in retrievable storage pursuant to a 1970 Atomic Energy Commission policy (see Section 1.2) and TRU waste that was reasonably expected to be generated by ongoing activities and programs was analyzed in DOE/EIS-0026 (October 1980) and the Final Supplement Environmental Impact Statement for the Waste Isolation Pilot Plant (SEIS-I) (DOE/EIS-0026-FS, January 1990).

NEPA coverage for the transportation and disposal of waste to WIPP are found in DOE/EIS-0200-F (May 1997) and Waste Isolation Plant Disposal Phase Supplemental EIS (SEIS-II) (DOE/EIS-0026-S-2, Sept. 1997), respectively. The 1990 Record of Decision (ROD) also stated that a more detailed analysis of the impacts of processing and handling TRU waste at the generator-storage facilities would be conducted. DOE has analyzed TRU waste management activities in DOE /EIS-200-F (May 1997). The WM PEIS analyzes environmental impacts at the potential locations of treatment and storage sites for TRU waste; SEIS-II addresses impacts associated with alternative treatment methods, the disposal of TRU waste at WIPP and alternatives to that disposal, and the transportation to WIPP. (SEIS-II also includes potential transportation between generator sites.)

Is the project funded by the American Recovery and Reinvestment Act of 2009 (Recovery Act)

Approved by Jack Depperschmidt, DOE-ID NEPA Compliance Officer on: 2/17/2015