SECTION A. Project Title: Pyroprocessing at Energy Innovation Laboratory (EIL)

SECTION B. Project Description and Purpose:

This Tent Environmental Compliance Permit (ECP) for Pyroprocessing at Energy Innovation Laboratory (EIL) Labs B208, B209, and C213, using non-radioactive materials, incorporates the requirements, operational aspects and scope from the following ECPs to provide clarity and consistency:

- *INL-19-030, Advanced Spectroscopy Furnace (ASF) in VTI Glovebox at EIL B208*
- *INL-19-072 (OA18), Density and Level Measurements in Fluids*
- *INL-19-133 R2, Activities for Molten Salt Systems and Pyroprocessing Department*
- *INL-21-072 Pyroprocessing*
- *INL-21-178 (OA37), A Dynamic Single Bubbler for Nuclear Material Accountancy Measurements in Bulk Liquids (NHS)*

The objective of this program is to obtain fundamental data to support the development of molten salt systems, electrochemical separations, waste form production and development, and other technology development associated with missions and programs at EIL using non-radioactive materials (cold testing). The laboratories contain chemicals, equipment, and tools used in the preparation and experimentation of research activities such as sample preparation, powder preparation, molten salt experiments, electrochemical experiments, general chemistry methods, and assembly/operation/disassembly of experiments. Activities will include experiments using molten salts in furnaces as well as pyrophoric material experiments in an inert glovebox, fume hood, and or benchtop. These R&D activities are intended to explore and discover original and enhanced methods, techniques, or materials. The following discussion describes this work:

Pyrochemistry and Molten Salt Systems department performs research and development activities with the specific goals of:

1. Determining fundamental physical and chemical properties of molten salt systems

2. Developing and implementing electrochemical separations

3. Performing chemical and physical measurements of various systems, such as: molten salts, pyrophoric materials, metals, ceramics, glasses, and other engineered systems.

4. Designing and conducting experiments on above mentioned systems.

5. Fabricating small-scale test specimens for experimental research.

6. Developing materials, sensors, and instrumentations for molten salt systems.

Pyroprocessing Tent (INL-21-072)

Research toward the stated objectives is performed in the two labs located at the EIL: Lab B208 and Lab C213 using non-radioactive materials. The laboratory contains chemicals, equipment, and tools used in the preparation and experimentation of research activities such as sample preparation, powder preparation, electrochemical experiments, sample characterization, thermal property determination, and general chemistry methods. Some chemicals are sensitive to moisture and oxygen and are handled in inert (argon) atmosphere gloveboxes and all are listed on the 420.07 form for LI-764.

An off-the-shelf lab-scale glass and ceramic melter is in the walk-in-hood in EIL C-213. Operation involves the fabrication of a small-scale glass and ceramic samples for testing and characterization. The materials used in fabrication include $BSiO_4$ and $FePO_4$ glasses, and SiO_4, AlO_4, ZrO_2, TiO_2 ceramics. No hazardous constituents will be included in the formulations and off-gas from the fabrication will include water vapor and the above-mentioned constituents. Closed chiller or house water will be running to and from the furnace for cooling. Different atmosphere hookups will be utilized for furnace operations. These hookups are located with the walk-in hood. The furnace utilizes the ventilation already present within the hood. Research on molten salt chemistry and corrosion control of chloride salt salts systems will be performed in B208 in EIL, using non-radioactive salts such as NaCl-$MgCl_2$.

Quartz tubes and capillaries loaded with salt samples may be transferred to a gamma irradiator located at the EIL facility for irradiation of salt samples.

Flowing salt properties will be investigated using a natural circulation molten-salt flow loop equipped with instrumentation located within the C-213 glovebox. Specifically, the molten-salt flow loop will be observed to analyze the molten salt flow characteristics (thermal gradients and flow dynamics). The corrosion samples of various metals and alloys will be introduced in the loop through the surge tank. Salt samples will be collected before, during, and after the corrosion tests to monitor the metal alloy corrosion. Various techniques (i.e., ICP analysis, spectroscopy techniques etc.) will be used to analyze the salt samples collected during and after the measurement; and surface characterization techniques (SEM, FIB, optical microscopy etc.) will be used for analysis of metal/alloy surface after the corrosion test. In addition to the corrosion samples, a specimen of stainless steel (SS) 316 pipe will be characterized after the loop operation to evaluate the corrosion during the operation. For salt and metal alloy characterization, the samples will be sent to various facilities including INL Research Center (IRC), Center for Advanced Energy Studies (CAES), and Irradiated Materials Characterization Laboratory (IMCL) at MFC. For some analysis techniques that might not be available at INL sites, X-ray Photoelectron Spectroscopy (XPS).

Research on molten salt chemistry and corrosion control of chloride salt salts systems will be performed in B208 in EIL, using non-radioactive salts such as NaCl-MgCl2.

The labs at EIL will also support projects related to pyrochemical R&D. It is desired to design equipment (e.g., furnace systems) and develop techniques to separate salt and metal products and then to cast them into usable forms. A small furnace is installed in the MBRaun glovebox in the EIL. The furnace is small enough to fit into the transfer chamber and will support the project adequately. It will also be necessary to produce surrogate dendrites (copper) for feed into these tests using molten salt furnaces.

Experimental samples will be prepared, processed, and characterized using equipment that is standard for the industry. This includes using instruments such as an autosiever, balance, cameras, differential scanning calorimeter (DSC), gloveboxes, hand tools, high or slow speed saws, high-temperature furnace, hot plate, impact mortar and pestle, Inductively coupled plasma mass spectrometry (ICP-MS), lab-scale glass and ceramic melter, lasers, moister analyzer, laboratory glassware, laboratory oven, laboratory stirrer, micromill grinder, microscopes, microwave, molten-salt flowloop, pH/ion selective electrode meter, potentiostat, pycnometer, riffler, sonic sifter, spectroscopic analysis, thermogravimetric analyzer (TGA), three-dimensional electrochemical manufacturing and sensing (3DEMS), vacuum pump, water hydrogen torch, and other similar equipment.

Typical samples are under 5 kg for powders or solids. Liquid samples are also small and are typically one liter or less. Most characterization activities utilize smaller samples up to 200 g or 200 ml. Some samples will require heating and furnace temperatures typically between 500 - 650 deg C but up to 1200 deg C. Materials are procured, used, and stored in the laboratory. These materials include acids, powders, salts, and gases. A glovebox with HEPA filtered exhaust or fume hood with HEPA filtered exhaust is utilized as necessary. Activities may prepare mixtures with the composition desired for a given experiment or set of experiments. Typically, the process uses simple mixtures to focus on the partitioning of a specific compound. In other experiments, the process uses complex mixtures to imitate conditions expected during treatment of spent nuclear fuel. All materials used in this laboratory are non-radioactive and commercially available. Samples in secondary containers are labeled. Samples will be discarded when no longer needed.

SolGel additive manufacturing process (SGAM) fabrication process(es) for net shaped integrated fuel systems with embedded sensors to enable in-situ real time measurement of local properties (e.g. temperature) at multiple spatial locations, important to reactor safety, reactor operation and/or fuel performance.

Testing a dynamic bubbler performance in aqueous fluids to further develop in situ density and level measurements. In these experiments, tubing will be immersed in the fluid via a linear actuator and argon gas will be bubbled into the fluid. Bubble pressures will be monitored using differential pressure transducers while gas flow is controlled via mass flow controllers. The pressure transducers and flow controllers are contained in a control panel system.

For salt and metal alloy characterization, the samples will be sent to various facilities including INL Research Center (IRC), Center for Advanced Energy Studies (CAES), and Irradiated Materials Characterization Laboratory (IMCL) at Materials Fuel Complex (MFC). For some analysis techniques that might not be available at INL sites, X-ray Photoelectron Spectroscopy (XPS) for example, the samples can be shipped to university collaborators listed in this ECP.

Advanced Spectroscopy Furnace (ASF) in VTI Glovebox at EIL B208 (INL-19-030)

- The Pyrochemistry and Molten Salt Systems department proposes to install an advanced spectroscopy furnace in the VTI glovebox in Room B208 at the EIL to support anticipated future research. The ASF will be dedicated to work on spectroelectrochemistry. Future work may involve topics such as:
- Molten salt chemistry characterization using combined spectroscopy and electrochemical techniques.
- Reactor-related material performance and development
- Molten salt process chemical management
- Chemical reduction and precipitation

Molten salt technology development utilizing the ASF requires an inert atmosphere VTI glovebox because of the hygroscopic nature of the salts. The glovebox is not for radioactive materials but for materials that are sensitive to air. No building modifications are required for installation of the furnace, with a total molten salt volume of 25 mL and operation temperature of 650 deg C. The project will utilize the furnace in the glovebox coupled to a laser system via fiber optic for performing raman, UV-Vis and near IR spectroscopy on a small molten salt pool. The glovebox will be modified to perform as a Class 1 laser enclosure.

There are also two furnaces (one large furnace and one smaller transparent furnace) currently located at CAES in the Radiochemistry Laboratory that will be moved to EIL Room B208. The large furnace is a two-zone crystal growing furnace (Oxy-gon, Inc) with a footprint of approximately 3ft wide by 3 ft deep by 5 ft tall. The furnace has its own vacuum pump and inert gas system built in. The transparent furnace is a Therm-Craft tube furnace. Building modifications may include power connections for the furnaces.

Density and Level Measurements in Fluids (INL-19-072)

· Testing bubbler configurations and performance in aqueous and molten salt fluids to further develop in situ density and level measurements. In these experiments, tubing will be immersed in the fluid and argon gas will be bubbled into the fluid. Bubble pressures will be monitored using differential pressure transducers while gas flow is controlled via mass flow controllers. The pressure transducers and flow controllers are contained

in a control panel system. External to the panel are thermocouple sensors and height gauges. The majority of the system has been purchased, manufactured, and assembled; however, some minor modifications may be necessary throughout the duration of testing. In addition to the control panel, two furnaces are being relocated into EIL B208 under EC INL-19-030 (OA 27). The inert atmosphere glovebox, addressed by INL-18-114, will be used to prepare the chloride salts used in the experiments. Experiments will release small amounts of argon gas into the laboratory. The aqueous experiments will be conducted in pure water or chloride salt solutions (calcium chloride and sodium chloride). These solutions will be disposed following use. In addition, ethanol or methanol will be used as cleaning agents.

Activities for Molten Salt Systems and Pyroprocessing Department (INL-19-133 R2)

· Revision 2: This revision includes addition of two tasks to the ongoing research activities performed at the Energy Innovation Laboratory (EIL) labs B208 and C213 under LI-654 and LI-784. (The original ECP incorrectly referenced LI-764.) The overall project goals are same as listed in original ECP: 1)The procurement, installation and operation of a water hydrogen torch. The torch is a benchtop system that splits an electrolyte solution into hydrogen and oxygen atoms. The torch will be used to flame seal quartz tubes and capillaries loaded with salt samples. Operation will include igniting the flame, sealing the tube containing salts and argon, and maintenance of the torch including refilling of methyl alcohol, flux, and electrolyte solutions. The salts to be handled will include chlorides and fluorides of Li, K, Mg, Zn, Na, Cs, Ca, etc. The sealed tubes maybe transferred to gamma irradiator located at EIL facility for irradiation of salt samples. The sealed tubes/capillaries will also be shipped to the collaborating labs for characterization including Brookhaven National Lab (BNL), Oak Ridge National Lab (ORNL) and University of Notre Dame (UND). The sealed tubes once shipped outside INL will not be returned to INL, and will be disposed of at the receiving site.2)The purpose of this task is to investigate flowing salt properties. A natural circulation molten-salt flow loop equipped with instrumentation will be operated inside C213 glovebox, to analyze the molten salt flow characteristics (thermal gradients and flow dynamics). The corrosion samples of various metal/alloys will be introduced in the loop through the surge tank. Salt samples will be collected before, after and during the corrosion tests to monitor the metal alloy corrosion. Various techniques (i.e. ICP analysis, spectroscopy techniques etc.) will be used to perform analysis of salt samples collected during and after the measurement; and surface characterization techniques (SEM, FIB, optical microscopy etc.) will be used for analysis of metal/alloy surface after the corrosion test. In addition to the corrosion samples, a specimen of SS316 pipe will be characterized after the loop operation, to evaluate the corrosion during the operation. For salt and metal alloy characterization, the samples will be sent to labs outside EIL, to various facilities including INL Research Center, Center for Advanced Energy Studies, and Irradiated Materials Characterization Laboratory at the Materials and Fuels Complex. For some analysis techniques that might not be available at INL sites, X-ray photoelectron Spectroscopy (XPS) for example, the samples can be shipped to university collaborators.

Revision 1: This revision includes the procurement, installation and operation of an off-the-shelf lab-scale glass and ceramic melter in the walk-in-hood in EIL C-213. Operation will involve the fabrication of small-scale glass and ceramic samples for testing and characterization involved with the Materials and Disposition Capabilities Program sponsored by DOE NA-22. The materials to be fabricated include $BSiO_4$ and $FePO_4$ glasses, and SiO_4, AlO_4, ZrO_4, TiO_2 ceramics. No hazardous constituents will be included in the formulations and off-gas from the fabrication will include water vapor and the above-mentioned constituents. Lab modifications will include adding a single-phase 208-volt outlet with 70 amps, using closed chiller or house water run to and from the furnace for cooling, and hookups for different atmospheres (already set up in the · hood). Ventilation is also already set up, since this will be operated exclusively in the hood. Project goals are the same as those listed in the original EC.

· Original ECP: The objective of this program is to obtain fundamental data to support the development of molten salt systems, electrochemical separations, waste form production and development, and other technology development associated with missions and programs at the Idaho National Laboratory (INL) Energy Innovation Laboratory (EIL) using non-radioactive materials (cold testing). Pyrochemistry and Molten Salt Systems department performs research and development activities with the specific goals of:

1. Determining fundamental physical and chemical properties of molten salt systems
2. Developing and implementing electrochemical separations
3. Performing chemical and physical measurements of various systems, such as: molten salts, metals, ceramics, glasses and other engineered systems.
4. Designing and conducting experiments on above mentioned systems.
5. Fabricating small-scale test speciments for experimental research.

Research toward the stated objectives is performed in the two labs located at the EIL: Lab B208 and Lab C213 using non-radioactive materials. The laboratory contains chemicals, equipment, and tools used in the preparation and experimentation of research activities such as sample preparation, powder preparation, electrochemical experiments, sample characterization, thermal property determination, and general chemistry methods. Some chemicals are sensitive to mosture and oxygen and are handled in inert (argon) atmosphere gloveboxes but all are listed on the 420.07 for LI-764.

Experimental samples will be prepared, processed, and characterized using equipment that is standard for the industry. This includes using instruments such as an autosiever, balance, cameras, differential scanning calorimeter (DSC), gloveboxes, hand tools, high or slow speed saws, high-temperature furnace, hot plate, impact mortar and pestle, Inductively coupled plasma mass spectrometry (ICP-MS), moister analyzer, laboratory glassware, laboratory oven, laboratory stirrer, micromill grinder, microscopes, microwave, pH/ion selective electrode meter, potentiostat, pycnometer, sonic sifter, spectroscopic analysis, thermogravimetric analyzer (TGA), vacuum pump, and other similar equipment.

Typical samples are under 5 kg for powders or solids. Liquid samples are also small and are typically one liter or less. Most characterization activities utilize smaller samples up to 200 g or 200 ml. Some samples will require heating and furnace temperatures typically between 500 - 650 deg C but up to 1200 deg C. Materials are procured, used, and stored in the laboratory. These materials include acids, powders, salts, and gases. A glovebox with HEPA filtered exhaust or fume hood with HEPA filtered exhaust is utilized as necessary. Activities may prepare mixtures with the composition desired for a given experiment or set of experiments. Typically, the process uses simple mixtures to focus on the partitioning of a

specific compound. In other experiments, the process uses complex mixtures to imitate conditions expected during treatment of spent nuclear fuel. All materials used in this laboratory are non-radioactive and commercially available. Samples in secondary containers are labeled. Samples will be discarded when no longer needed.

Dynamic Single Bubbler for Nuclear Material Accountancy Measurements in Bulk Liquids (EIL)(INL-21-178)

· The purpose of this project is to test a dynamic bubbler performance in aqueous fluids to further develop in situ density and level measurements. In these experiments, tubing will be immersed in the fluid via a linear actuator and argon gas will be bubbled into the fluid. Bubble pressures will be monitored using differential pressure transducers while gas flow is controlled via mass flow controllers. The pressure transducers and flow controllers are contained in a control panel system. External to the panel are thermocouple sensors, the linear actuator, and height gauges. The single bubbler system has been purchased, manufactured, and assembled and no building modifications are needed. Experiments will release small amounts of argon gas into the laboratory. The aqueous experiments may be conducted in pure water, chloride salt solutions (calcium chloride and sodium chloride), mineral oil, nitric acid, and kerosene. These solutions will be disposed of properly following completion of the testing into the Idaho Falls sewer system. The mineral oil and kerosene will be disposed of by Waste Generator Services. In addition, ethanol, acetone, and methanol may be used as cleaning agents.

Waste Type	Location	Anticipated Volume
Metal recycling	C208/C213	5 ft^3/yr
Wipes	C208/C213	<0.5 ft^3/yr
Industrial waste	C208/C213	<25 kg/month
RCRA metals	C208/C213	<50 g/month
Corrosive solids (e.g. salts)	C208/C213	<5 kg/yr
Flammable solids	C208/C213	<50 g/month
Reactive metals	C208/C213	<50 g/month
Liquid solutions (i.e., acids or flammable liquids)	C208/C213	<20 L/yr

Additional Referenced Documents (not to supersede) ECPs:

- LI-654 Pyroprocessing General Laboratory Activities
- LI-784 NS&T Pyroprocessing Experiments
- LI-928 General Fabrication & Assembly Activities Within Pyrochemistry & Molten Salt System Laboratories
- INL-21-072, Pyroprocessing Tent

Referenced Air Permitting Applicability Determination (APADs) not to supersede:

- APAD-13-007 R2, REL/EIL Operations

SECTION C. Environmental Aspects or Potential Sources of Impact:

Air Emissions

The processes may generate very small amounts of chemical emissions. These emissions are covered by APAD INL-13-007 R2. The proposed action has the potential to generate air emissions from bubbling of argon gas that will be distributed into the open laboratory space. This will

eventually be exhausted through the lab's exhaust system.

Discharging to Surface-, Storm-, or Ground Water

NA

Disturbing Cultural or Biological Resources

Cultural: Pursuant to the 2023 Programmatic Agreement, this federal undertaking is excluded from Section 106 review as the proposed activity has no effects to historic properties.

Generating and Managing Waste

Small amounts of common office trash are expected. Industrial waste such as boxes, plastic, and paper would be generated from packaging and office work. Some discharges of water may occur, if a closed-loop chiller is not used. It is anticipated that the samples will need to be disposed. WGS will assist in the characterization and management of any waste that is generated. Dilute acidic and basic solutions will be disposed of using SAA set up in lab B208.
Additional standard industrial waste will be generated at a small scale (INL-21-165). Waste includes typical industrial waste such as PPE, etc. Additional waste is from aqueous solutions including water, salt water (NaCl and/or CaCl), mineral oil, nitric acid, and kerosene. The test solution volumes will be less than 2 liters. The material waste (kerosene and mineral oil) will be disposed of by waste management services and will generate less than 4 liters.

Releasing Contaminants

When chemicals are used during the project there is the potential for spills that could impact the environment (air, water, soil).

Using, Reusing, and Conserving Natural Resources

Project personnel would use every opportunity to recycle, reuse, and recover materials and divert waste from the landfill when possible.

Environmental Justice

NA

SECTION D. Determine Recommended Level of Environmental Review, Identify Reference(s), and State Justification: 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: B1.31 "Installation or relocation of machinery and equipment", B3.6 "Small-scale research and development, laboratory operations, and pilot projects"

Justification: B1.31 Installation or relocation of machinery and equipment. Installation or relocation and operation of machinery and equipment (including, but not limited to, laboratory equipment, electronic hardware, manufacturing machinery, maintenance equipment, and health and safety equipment), provided that uses of the installed or relocated items are consistent with the general missions of the receiving structure. Covered actions include modifications to an existing building, within or contiguous to a previously disturbed or developed area, that are necessary for equipment installation and relocation. Such modifications would not appreciably increase the footprint or height of the existing building or have the potential to cause significant changes to the type and magnitude of environmental impacts.

B3.6 Small-scale research and development, laboratory operations, and pilot projects. 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); and small-scale pilot projects (generally less than 2 years) frequently conducted to verify a concept before demonstration actions, provided that construction or modification would be within or contiguous to a previously disturbed or developed 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.

Is the project funded by the American Recovery and Reinvestment Act of 2009 (Recovery Act) ☐ Yes ☒ No

Approved by Robert Douglas Herzog, DOE-ID NEPA Compliance Officer on: 12/15/2023