Standard Operating Procedures

Experiment: High Temperature Calorimetry on Non-rad, U-, Th-, and Pu-containing Samples
Location: Dodgen Facility, Room B21

Activity Description:
This SOP covers using the Setaram AlexSys-1000 high-temperature oxide-melt calorimeter and SetSys high-temperature differiential scanning calorimeter to conduct experiments on non-rad, U-, Th-, Pu-containing materials. Future revisions will be made when necessary to accomodate the use of other radionuclides for calorimetric studies. This SOP specifically addresses *i*) the issues and risks associated with handling and measuring U-, Th-, and Pu-containing samples, *ii*) corresonding controls to ensure safety and security conducting the proposed work; and *iii*) required training plans for personnel to be authorized for conducting the experiments described by this SOP.

Work Tasks/Steps	**Hazards, Concerns, and Potential Accidents/Incidents**	**Controls, Preventive Measures, and Bounding Conditions**	**Training Plan and Reference Documents**
<u>All Tasks:</u> General guidelines - applicable to all the steps	Clutter/housekeeping issues: Crowded work area Facility deficiencies: Check the building status prior to performing work, notify NSC Point of Contact (POC) if applicable prior to work.	- Good housekeeping and communication - Be aware of potentially unsafe conditions in the facility - Wear appropriate Personal Protective Equipment(PPE) which include but are not limited to safety glasses and gloves (chemically/thermally resistant when applicable) - All instrument use will comply with training documents, protocols, and instrument manuals.	- NSC Accident Prevention Plan - NSC Facility Procedures - Chemical Hygiene Plan - Lab Specific Standard Operating Procedures - Calorimetry training (Joy of Calorimetry, calorimetry training plan) - Rad worker training in the case of working with radioactive materials (RAM)
<u>Task 1:</u> Chemicals transfer plan between Fulmer synthesis lab and Dodgen calorimetry lab	• Miscommunication that affects the chemical inventory management • Chemical exposure (contact with skin or eyes; inhalation)	**Administrative Controls:** - Notify the NSC POC with detailed information of the (both non-rad and rad) chemicals that are planned to be transferred to the calorimetry lab. If RAM is transferred, additionally notify in writing and	- NSC Accident Prevention Plan C.4, C.7 - NSC Facility Procedures C.6 - Chemical hygiene Plan

	• Spread of RAM and other chemicals during transferring/transporting • Potential exposure of individuals to low levels of radiation and contamination	coordinate with Radiation Safety Office (RSO) with standardized procedures. - PI approval or consent need to be reached prior to sample transfer. - RSO will perform all transfers of RAM across WSU. - RSO will perform and document the survey on RAM packages/containers, and the PI will place the material in secure storage. - RSO will update inventory and ensure compliance with the AU application and DOH reporting regulations. **Engineering Controls:** - Use double chemical containment appropriately shielded for RAM transfer between Fulmer and Dodgen.	- Rad worker training in the case of working with RAM - RSO procedures and notifications for transfer of RAM (RAM transfer form) - EHS/ORA procedures for transferring chemicals.
<u>Task 2:</u> Radioactive chemicals storage and security plan of rad-related experiments	• Chemical missing or stolen • Unwanted interference by unauthorized personnel during experiments • Risk of loss of control of special nuclear material	**Administrative Controls:** - Closely work with NSC POC on sample storage and coordinate on sign posting before planned calorimetric experiments. - A list of Authorized Experimenters will be maintained by PI and distributed to the NSC POC. - If RAM samples are used: ▪ notify in writing the NSC POC about planned experiments; ▪ send out notification email to NSC POC for notification of all personnel working in Dodgen about coming experiments which could impact facility personnel, ▪ consult with the NSC POC about limiting the use of elevator to the basement calorimetry lab while access to the lab can be done by taking the stairs for specific dates/times;	- NSC Accident Prevention Plan C.7, C.9 - NSC Facility Procedures C.6 - Chemical hygiene Plan - Rad worker training in the case of working with RAM

		▪ post signs of “Radioactive material in use”, “ONLY authorized calorimetry-trained personnel access” on the fence of the calorimetry lab. ▪ Place brightly colored rope or chain control barrier across the entry to the calorimetry lab. **Engineering Controls:** - First barrier: only personnel authorized by NSC can enter the building - Second barrier: Fence structure with key-locked door on the boundary of the calorimetry lab - Third barriers: RAM samples will be either ▪ locked inside of secure storage or a safe (when not used for experiments) in the calorimetry lab; all samples will be labeled with the date produced and experiment. Dated samples related to completed experiments will be safely disposed of in order to reduce clutter in storage space. ▪ dissolved in the molten salt matrix kept at 700°C inside the AlexSYS calorimeter; ▪ (not Pu), kept in crucibles inside SetSYS DSC under temperature from 20 °C to 2400 °C.	
<u>Task 3:</u> Inspect the instrument for possible unsafe operation or malfunction conditions	• AlexSYS: Overheating (above the operation temperature; typically 700 °C, not exceeding 800 °C), abnormal odors due to burning • SetSYS: too low water level in the chiller and insufficient inert protective gas • Electrical hazards due to operation of the instruments.	- Visually inspect the HV cabling to verify it is correctly installed and the condition is acceptable. (No rips, breaks, coiling, etc.) - Inspect the external temperature readouts for the three (3) zones, and ensure the temperature is at its desired set point. - Log into the control software and verify there are no alarms, and all controls are functioning properly. - Stop work if overheating has been triggered or controller is malfunctioning.	- NSC Accident Prevention Plan C.15 - NSC Facility Operating Procedures C.7 - Calorimetry training (Joy of Calorimetry, calorimetry training plan

		- check water level of the chiller and gas tank pressure prior running DSC experiments	
<u>Task 4:</u> Prepare and lower the glassware assembly into the calorimeter • Ensure glassware is clean, and if needed wipe it with Kimwipes; visually inspect and verify that all the glass pieces are free of cracks. • Assemble glassware by placing dropping tubes and Pt crucibles inside the glass liners. • Install bubbling tubes if needed, depending on the types of experiment (transposed temperature drop or solution) and sample. • Lower the glassware assembly into the calorimeter slowly (1/3 of the way down every 10 minutes) using clamps. Attach plastic gas tubing and lower the bubbling tubes into solvent if applicable.	• Thermal hazards: Burns • Sharps/broken glasses: Whenever glassware is used, there is a risk of breakage.	- Allow 10 minutes after each step of glassware lowering to achieve thermal equilibrium. - Handle all glassware in a manner that avoids inducement of thermal shock. - If the solvent is metal gallium or other molten metals, only purge Ar gas from the beginning when the glassware setups are initially inserted.	- NSC Accident Prevention Plan C.10, C.15 - NSC Facility Operating Procedures C.3, C.7 - Calorimetry training (Joy of Calorimetry, calorimetry training plan)

<u>Task 5:</u> Performing high-T drop calorimetric experiments (AlexSYS) • Verify the baseline is stable • Drop the sample pellet or chunk into one side of the calorimeter via the dropping tube. • Allow the experiment to run to completion (typically 1-2 h). • Drop another sample into the other side of the calorimeter and continue this until a desired number of samples have been dropped and data been collected.	• Platform use: Slips/trips/falls • Thermal: Burns • Sharps/glass: Cuts/abrasions, etc.	Be aware of small step footing on the platform and ensure it is always stable during its use. Provide postings of pressures and identification of materials under pressure when systems under pressure are left unattended.	- Calorimetry training (Joy of Calorimetry, calorimetry training plan)

<u>Task 6:</u> Performing high-T differential scanning calorimetry (SetSYS) • Verify the baseline is stable • Weight the sample (powder, pellet or chunk) in the microbalance • Load the weighted sample into crucible (type of crucible depends on the nature of chemicals, heating temperature and atmosphere). • Remove the contained sample from the instrument and contain in a vial for future post-characterization.	• Platform use: Slips/trips/falls • Thermal: Burns • Sharps/glass: Cuts/abrasions, etc.	Be aware of small step footing on the platform and ensure it is always stable during its use. Provide postings of pressures and identification of materials under pressure when systems under pressure are left unattended.	- Calorimetry training (DSC document, calorimetry training plan)
<u>Task 7:</u> Handle/measure U- and Th-bearing samples (solids containing depleted U and/or Th), using calorimetry described by both Task 5 and 6.	**<u>For all parts of task 7</u>** • Chemicals may be flammable, hazardous, toxic, acutely toxic, etc. • Chemical exposure (contact with skin or eyes; inhalation) • Spread of radioactive materials (RAM), including depleted uranium (primarily isotope ^{238}U) and natural thorium (^{232}Th)	**Administrative Controls:** - Perform prejob briefing and discuss all potential risks - Define and mark out (by rad tape) a benchtop area for rad samples unpacked and preparation. - Constrain the potential contaminated area. - Facility will provide instruments for self-frisk for accessing the radiologically-controlled area. - Always self-monitor when exiting a radiologically-controlled area. - Wear TLD in radiologically-controlled areas. - <u>For any spill of radioactive material, personal contamination, or any injury compromising the integrity of the skin (cuts or abrasions), contact PI, NSC PM and RSO immediately.</u>	- NSC Accident Prevention Plan C.7, C.10 - NSC Facility Procedures C.6, C.7 - Calorimetry training (Joy of the calorimetry, calorimetry training plan) - Chemical hygiene plan - Rad worker training - SPPM on Radiation Safety - WSU Radiation Protection Plan - NSC Emergency Plan

1) Conducting calorimetric measurements on U/Th-containing sample pellets	• Chemical: sample dependent • Potential to spread radioactive contaminants and expose individuals to low levels of radiation	**PPE:** - Lab coats are required for entry into a rad controlled area. - Wear gloves and safety glasses when working and handling RAM. **Administrative Controls:** - If spills occur, use moistened disposable paper towels for cleaning, and have facility personnel to inspect the surrounding area. - If the exterior of the instrument is potentially contaminated, minimize potential spread when cleaning by blotting whenever feasible rather than wiping. - Dispose the used paper towels in rad trash boxes. - Do not clean potentially rad-contaminated equipment with compressed air. - After usage and cleaning, store the transferring container at an appropriate location noting that the items used in rad controlled areas cannot be free released without the facility personnel or RSO approval. - <u>For any spill of radioactive material, personal contamination, or any injury compromising the integrity of the skin (cuts or abrasions), contact PI, NSC POC and RSO immediately.</u> - If any questions or doubts arise, consult PI, facility managers and RSO for help.	

2) Disposal of used quartz-glass crucibles	• Burns from handling hot glass crucibles • Mechanical: using hands tool during solvent removal • Cuts from glass	**Engineering Controls:** - A container will be used for transferring samples from the safe to the designed working area or microbalance table in the calorimetry lab - A small plastic scoop or weight paper will be additionally used for transferring the pellets from the container to i) the weighting balance; ii) the top opening of the dropping tube of the AlexSYS calorimeter; iii) into the crucibles used in SetSYS. - Aluminum funnels are recommended to be placed on the tops of the two dropping tubes to avoid off-dropping of the sample pellets/chucks. **PPE:** - Lab coats are required for entering a rad controlled area. - Wear gloves and safety glasses when working and handling RAM. - Carefully remove the used quartz-glass crucibles (which contain the solidified molten sodium molybdate plus dissolved U/Th sample pellets) and do swipe tests on certain glassware so those can be reused; the quartz sample containers/crucibles will be disposed as rad wastes. - <u>For any spill of radioactive material, personal contamination, or any injury compromising the integrity of the skin (cuts or abrasions), contact PI, NSC POC and RSO immediately.</u> - Hand and foot monitoring into and out of the lab is required upon entry and exit. - If any questions or doubts arise, consult PI, or NSC POC and RSOs for help.	

Task 8: Handle/measure Pu- and radionuclides (with high activities) bearing solid samples	**Applied to all task 8, In addition to all requirements from task 7** • Chemicals may be flammable, hazardous, toxic, acutely toxic, etc. • Chemical exposure (contact with skin or eyes; inhalation) • Spread of radioactive materials (RAM), plutonium (primarily isotope ^{239}Pu and ^{242}Pu).	**Administrative Controls (in addition to requirements from task 7):** - The PI shall be present anytime the Pu is out of the safe, and is responsible for cataloging all use. - ALWAYS perform prejob briefing to discuss risks - ALWAYS enforce TWO-person rule that one person is conducting the experiment or handling the RAM, while another one is standing by and providing help as needed - For any spill of radioactive material, personal contamination, any injury compromising the integrity of the skin (cuts or abrasions), or in the event of CAM alarm triggering: immediately leave the containment area and evacuate to the designated area; and notify the PI, NSC POC, and RSO immediately. Do not leave the designated area for any reason other than preservation of life until you have been certified clean of radioactive material. **Engineering Controls (in addition to requirements from task 7):** - CAM shall be setup in the calorimetry lab and operating during the experimental period. - Unused Pu samples will be locked in the safe; only one Pu sample is introduced to the calorimeter at one time with an exposure time less than 10 min. - Safe shall be locked immediately (performed by the second person) after the sample is stored inside or taken from it for calorimetric drops. **PPE (in addition to requirements from task 7):** - Designed lab coats are required for entering a rad controlled area.	- NSC Accident Prevention Plan C.7, C.10 - NSC Facility Procedures C.6 - Calorimetry training (Joy of the calorimetry, calorimetry training plan) - Chemical hygiene plan - Rad worker training - SPPM on Radiation Safety - WSU Radiation Protection Plan - NSC Emergency Plan

1) Conducting calorimetric measurements	• Chemical: sample dependent • Potential to spread radioactive contaminants and expose individuals to low levels of radiation	- Wear gloves and safety glasses when working and handling RAM. - Wear respirator while handling Pu or other radionuclides with high activities during calorimetric experiments, despite the air exposure time is relatively short (< 10 min) **Administrative Controls:** - Plan the experiment ahead and minimize the activities (distance and time) during the calorimetric experiment. - Be extremely cautious while removing samples from the container and dropping them into the AlexSys reaction chamber. - Dispose the used paper towels and gloves in a designed rad trash bag. - Compressed air is prohibited. - After experiments, use the same transferring container for left-over solids, which cannot be released from the lab without the RSO approval. - Survey performed by NSC POC shall be done on the area advised by rad workers who conduct the calorimetric experiments. All potential areas and pathways above which RAM may be moved across are subject to swipe tests. - If any questions or doubts arise, consult PI, or facility managers and RSOs for help. - Hand and foot monitoring into and out of the lab is required at all times of entry and exit. **Engineering Controls:**	

2) Transferring of used quartz-glass crucibles back to Fulmer synthesis lab	• Mechanical: using hands tool during solvent removal • Cuts from glass	- Use a container for moving samples between different area/instruments inside the calorimetry lab. - Use of aluminum funnels are REQUIRED that they are placed on the tops of the two dropping tubes to avoid off-dropping of the sample pellets/chucks. **PPE:** - Designed lab coats are required for entering a rad controlled area. - Wear gloves and safety glasses. - Wear respirator while handling Pu or other radionuclides with high activities during calorimetric experiments, despite the air exposure time is relatively short (< 10 min) - Hand and foot monitoring into and out of the lab is required at all times of entry and exit. **Engineering Controls:** - Seal the used quartz-glass crucibles (which contain the solidified molten sodium molybdate plus dissolved rad sample pellets) in plastic bags and transfer them using calorimetric container back to Fulmer synthesis lab for disassembling. - If any questions or doubts arise, consult PI, or facility managers and RSOs for help.	
<u>Task 9:</u> Remove glassware assembly from AlexSYS • Remove glassware assembly from the calorimeter and place it on cooling rack. • Cover protection tubes with metal plugs.	• Thermal: Burns Sharps/glass: Cuts/abrasions, etc.	**Administrative Controls:** - If after Pu calorimetry, the PI shall be present when the setup is removing. - If after Pu calorimetry, ALWAYS enforce TWO-person rule that one person is pulling out the calorimetric	- Calorimetry training (Joy of Calorimetry, calorimetry training plan)

• Once the glassware is cool, disassemble it.		setup, while another one is standing by and providing help as needed. - For removal of the glassware, be cautious and remove the glassware smoothly from the instrument, handling with thermal insulated gloves. - For removal of glass tubes, be aware of possible sharps if glass is broken during the process and use puncture proof gloves, tape, etc. to minimize sharps hazard - <u>For any spill of radioactive material, personal contamination, any injury compromising the integrity of the skin (cuts or abrasions), or in the event of CAM alarm triggering: immediately leave the containment area and evacuate to the designated area; and notify the PI, NSC POC, and RSO immediately. Do not leave the designated area for any reason other than preservation of life until you have been certified clean of radioactive material.</u> **Engineering Controls:** - If after Pu calorimetry, CAM shall be remained active in the calorimetry lab during the removing period. **PPE:** - Designed lab coats are required for entering a rad controlled area. - Wear gloves and safety glasses when working and handling RAM. - If after Pu calorimetry, wear respirator.	
<u>Task 10:</u> Leakage of molten solvent during high-T drop calorimetric experiments:	• Thermal: Burns • Sharps/glass: Cuts/abrasions, etc.	**Engineering Controls:** - Use thermal gloves and/or oven mitt gloves when inserting and removing the setup into and from the calorimeter.	- NSC Accident Prevention Plan C.8, C.9 - NSC Facility Procedures C.10

If a leakage is suspected (which could be indicated by an unstable baseline—not due to temperature fluctuation): 1. Follow Task 9 to remove both setups 2. Inspect the setups on the cooling rack: i) A good sign is that there are no cracks → reinsertion of the setup should be conducted in a much slower manner to avoid sudden expansion of the air/gas inside the setup ii) A bad sign is that there is a crack(s) in the quartz-glass crucible containing the sample which may occur during the cooling process; or there are cracks in both the large and small quartz-glass crucibles so the whole setup needs to be disposed iii) An ugly sign is that the solvent has been leaked though the liner and generated cracks → immediately notify people working nearby, and notify PI, lab manager, and RSOs for help and survey the	• Chemical exposure (contact with skin or eyes; inhalation). • Potential to spread radioactive contaminants and expose individuals to low levels of radiation.	**Administrative Controls:** - The PI will be present when the setup is being removed from the calorimeter chamber, If after Pu or other rad calorimetry. - If the glassware setup is accidentally dropped and cracked, immediately notify people working in the lab, and notify PI, NSC POC and RSOs for help. Considering the rad-containing material is solidified in the solid chunk protected by three layers of quartz glasses, the spread of RAM is unlikely. - <u>For any spill of radioactive material, personal contamination, any injury compromising the integrity of the skin (cuts or abrasions), or in the event of CAM alarm triggering: immediately leave the containment area and evacuate to the designated area; and notify the PI, NSC POC, and RSO immediately. Do not leave the designated area for any reason other than preservation of life until you have been certified clean of radioactive material.</u> **Engineering Controls:** - If after Pu calorimetry, CAM shall be remained active during the inspection and removing period. **PPEs:** - Always be alert when handling calorimetric components (*e.g.*, dropper, adapter, glass), and exercise caution when doing so. - Lab coats are required for entry into the rad controlled area. - Wear gloves and safety glasses. - If this occurs after Pu calorimetry, wear respirator	- Calorimetry training (Joy of the calorimetry, calorimetry training plan) - Chemical hygiene plan - Rad worker training - SPPM on Radiation Safety - WSU Radiation Protection Plan - NSC Emergency Plan

liner. The whole setup will be then disposed 3. If the outside of the liner is contaminated, as shown by the facility survey, refer to Task 11 for removing the Inconel tube from the calorimeter chamber			
<u>Task 11:</u> Removing the Inconel tube from the calorimeter chamber due to solvent leakage: This could happen under very rare situations, since the sodium molybdate, Na_2MoO_4, solvent cannot dissolve quartz glass, and the reaction chamber has triply layered quartz-glass walls where the outer liner wall is thick and could most likely be broken/cracked by a mechanical force/shock. The steps of removing Inconel can be performed according to the procedures described in the "Operational manual – Joy of the Calorimetry" The removed Inconel tube and the calorimeter are surveyed by facility personnel.	• Thermal: Burns • Chemical exposure (contact with skin or eyes; inhalation). • Potential to spread radioactive contaminants and expose individuals to low levels of radiation.	**Engineering Controls:** - Use thermal gloves and/or oven mitt gloves when removing the Inconel tube out of the calorimeter chamber. **Administrative Controls:** - The PI and facility personnel will be present when the Inconel tube is being removed from the calorimeter chamber. - If after Pu calorimetry, CAM shall be remained active during the removing period. **PPEs:** - Always be alert when handling the Inconel tube. - Lab coats are required for entry into the rad controlled area. - Wear gloves and safety glasses. - If Pu samples are inside, wear respirator while handling Pu or other radionuclides with high activities during calorimetric experiments, despite the air exposure time is relatively short (< 1 min)	- NSC Accident Prevention Plan C.4, C.8, C.9, C.10 - NSC Facility Procedures C.10 - Calorimetry training (Joy of the calorimetry, calorimetry training plan) - Rad worker training - SPPM on Radiation Safety - WSU Radiation Protection Plan - NSC Emergency Plan

Task 12: After Use Survey	• Spread of RAM outside designated areas • Tracking of RAM outside of lab space	**Administrative Controls:** - Following all experiments, personnel will conduct a sweep for RAM of the lab space and instrumentation. These sweeps will be completed and documented by the personnel using the instrumentation. They will then be reviewed by the graduate student lab manager, then the PI. -All equipment needed for the sweeps will be maintained by the research group and will not be obtained from the NSC. -Weekly sweeps will be conducted by the NSC, and are not the responsibility of lab personnel. The lab manager and PI will coordinate with the NSC to ensure the sweeps are completed. - For any spill of radioactive material, personal contamination, any injury compromising the integrity of the skin (cuts or abrasions), or in the event of CAM alarm triggering: immediately leave the containment area and evacuate to the designated area; and notify the PI, NSC POC, and RSO immediately. Do not leave the designated area for any reason other than preservation of life until you have been certified clean of radioactive material.	- Calorimetry training (Joy of the calorimetry, calorimetry training plan)
Task 13: Plans for evacuation and contingency events	• RAM is left unattended • Thermal: Burns • Sharps/glass: Cuts/abrasions, etc. • Chemical exposure (contact with skin or eyes; inhalation). • Potential to spread radioactive contaminants and expose individuals to low levels of radiation	**Engineering Controls:** - Set up emergency light in the calorimetry lab. **Administrative Controls:** - During Pu experiment with two persons present: ▪ CAM alarms: don't panic but stop the experiment immediately, secure the Pu samples in the most closed container and leave the lab immediately. Walk up to the higher level to the designed area, and notify PI, facility manager and RSO	- NSC Accident Prevention Plan C.8, C.9, C.16 - NSC Facility Procedures C.3 - Calorimetry training (Joy of the calorimetry, calorimetry training plan) - Rad worker training - SPPM on Radiation Safety - WSU Radiation Protection Plan - NSC Emergency Plan

		immediately. Only re-enter the calorimeter lab after survey by the NSC POC. ▪ Facility emergency shutdown or under other emergency alarms: stop the experiments immediately, secure the Pu samples in the most closed container and leave the lab followed by the instruction to evacuate to the designated area outside the building. ▪ Unscheduled loss of power: don't panic as this event will not likely to result in any instrument-related catastrophic consequence as AlexSYS will be naturally cooled down. If one person has Pu sample in hand during calorimetric dropping process, he or she should stop moving, while the other person looks for backup light. Then properly put back the Pu sample into the container and lock it in the safe. ▪ Entry of unauthorized personnel: pause the experiment, secure the Pu samples in the most closed container, and warn this person of the experiment and advice of leaving immediately. Then notify this event to PI and facility manager. - During U and Th, or other non-rad experiment: ▪ Facility emergency shutdown or under other emergency alarms: stop work immediately, followed the instruction to evacuate to the designated area outside the building ▪ Loss of power: stay calm and look for emergency light. Leave the lab safely. ▪ Entry of unauthorized personnel: pause the experiment, and advise to leave immediately.	