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| **Working with Cesium Ampoules** |
| Dept:  | **MSE** | Laboratory:  | **Advanced Materials Laboratory** | Rm:  | **MEC 213** |
| PI | Reviewed by: | Enter name here | **X** | Date: |
| EHS | Reviewed by: | Enter name here | **X** | Date: |

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| **Brief Overview & Scope** | This protocol describes the procedure for loading and unloading elemental cesium (99.98%) with various experimental substrates of SiC into fused quartz ampoules. It covers the safe handling of the waste products after the samples are removed from the ampoules. |
| **Potential Hazards** | Cesium (Cs) reacts violently with air or water (water vapor) liberating extremely flammable gases (hydrogen). Causes burns. Corrosive. |
| **Haz. Mitigation Device** | Keep an open container of mineral oil available to immerse loose Cs.Special powder to extinguish Cs fire: Use class D fire agent, dry salt or sand. |
| **Engr. Controls** | Glove box, Mineral oil, portable glove bag |
| **PPE** | Safety glasses, lab coat, gloves, closed toed shoes |
| **Equipment, Tools:** | Ampoule sealing occurs using the Schlenk line in Eric Brown’s chemistry lab. Portable glove bag,  |
| **Mat’ls, Supplies:** | Ampoules: Quartz EPR tubes, 5 mm OD from Wilmad Glass P/N 701-PQ7Cesium: 10 g ampoule, 99.98% pure (or greater)Waste container: HDPE container with mineral oil for collecting all waste streams (must have secondary containment in the glove box)Portable glovebag (Grainger p/n 3NNZ9)Glass jar for storing excess Csmineral oildewar of liquid nitrogen (in chemistry for the sealing step).TorchCompressed UHP nitrogen or argon for purging glovebag. |
| **Assoc. Training Requirements**  | none |
| **Special Requirements:** |
| **Handling & Facilities** | All procedures occur under an inert atmosphere (Ar or N2) in a glove box, glove bag, or Schlenk line. |
| **Decontamination/ Waste Disposal** | All contaminated wastes are stored in a mineral oil bath. |

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| Warnings: |
| * Cesium (Cs) reacts violently with air or water (water vapor) liberating extremely flammable gases (hydrogen).
* Extinguish with special powder for metal fires. Do not use water.
* All contaminated wastes are stored in a mineral oil bath.
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| NOTES: |
| * This documents the steps taken to prepare sample ampoules containing Cs.
* The work is to be performed by Dr. John Youngsman and Dr. Eric Brown. It is not intended to be a training document for students.
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| chemicals |
| **Instructions:** Review the MSDSs for all chemicals used in this procedure. *Complete the (\*) information below only for chemicals that have a hazard level 3 or greater in any category.* |

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| Chemical:  | **Cesium 99.98%** | CAS #: | 7440-46-2 |
| Mfr: | Science Lab | Mfr: p/n | SLC4678 |
| Purpose: | Cesium will be diffused into SiC samples through a thermal annealing process. |
| Ingredients | Cesium |
| **Hazard Information for NFPA-HMIS ratings 2 or greater**  |
| **Health Hazard**  | 3 | May cause eye and skin irritation. May cause respiratory and digestive tract irritation. May cause central nervous system effects. May cause cardiac disturbances. |
| **FlammabilityHazard**  | 4 | Material will react with water and may release a flammable and/or toxic gas. May ignite or explode on contact with steam or moist air. |
| **Reactivity Hazard** | 3 | Water-reactive. Reacts violently with water.  |
| **PPE** |  |  |
| **First Aid** |  |  |

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| Chemical:  | **Cesium 99.98%** | CAS #: | 7440-46-2 |
| Mfr: | Alpha Aesar | Mfr: p/n | 10146 |
| Purpose: | Cesium will be diffused into SiC samples through a thermal annealing process. |
| Ingredients | Cesium |
| **Hazard Identification**  |
| Flame | In contact with water releases flammable gases (hydrogen) which may ignite spontaneously.  |
| Corrosion | Causes severe skin burns and eye damage.Causes serious eye damage. |
| First Aid |  |

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| Chemical:  | **Cesium 99.98%** | CAS #: | 7440-46-2 |
| Mfr: | Espi Metals | Mfr: p/n | n/a |
| Purpose: | Cesium will be diffused into SiC samples through a thermal annealing process. |
| Ingredients | Cesium |
| **Hazard Identification**  |
| Fire | DANGEROUS WHEN WET. If involved in a fire, burning metal may produce severely corrosive fumes of cesium oxide and hydroxide. Cesium reacts violently with water, liberating and igniting hydrogen, perhaps explosively. Cesium may ignite spontaneously on contact with air. Pyrophoric metal. |
| Health | Cesium is highly reactive, especially with moisture.  Extremely corrosive to tissue.  If cesium ignites, it will cause thermal burns.  The metal rapidly forms caustic and highly corrosive alkali hydroxide with evolution of heat.  Severe thermal and chemical burns will result at every point of human contact.  Cesium compounds may cause hyper-irritability, including spasms, dizziness, abdominal cramps, vomiting, diarrhea, convulsions and collapse.  Chronic ingestion of cesium has been fatal to laboratory animals, possibly due to the replacement of potassium.  Chronic exposure also can have blood and neuromuscular effects. |
| Reactivity | **Conditions to Avoid**:  Contact with water or air.**Incompatibility (Materials to Avoid)**:  Water, alcohols, oxidizers, halogens, oxygen, carbon dioxide, halogenated halocarbons, sulfur, phosphorus, and mineral acids. |
| First Aid |  |

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| Tasks |

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|  | **Potential Hazards** | **Engr Cntl, Haz. Mitigation Device, PPE** |
| 1. Ampoule loading in MEC 213
 | Reaction with water or air | Argon glove box, mineral oil receptacle for waste, safety glasses |
| 1. Load the necessary cleaned samples and ampoules into the glove box. Cycle Load Chamber 3 times.
2. Measure appropriate amount of Cs (use scale in glove box).
3. For EPR tube sealed at 5 cm, use ~500 mg Cs. This will provide a concentration of ~5 e 20 atoms/cm3
4. Place Cs and SiC (or other material) samples into the tube and cap tube, seal tube cap with tape.
5. Load sample tubes and packing material into a transport container such as a glass jar or plastic container, place the lid on tightly and tape joint after removed from glove box. Sample tubes are now under an inert atmosphere.
6. When unloading tubes from glove box, **Do Not Cycle Load Chamber** (risk of uncapping sample tubes).
7. Clean up in the glove box:
	1. Place opened supply of Cs in jar with tight fitting lid.
	2. Unload from glove box and seal lid tightly with tape.
	3. Clearly label the container indicating contents and hazards (Cesium, reactive to air and water) include that the jar contains Argon or Nitrogen.
8. Transport samples and portable glovebag to Chemistry (Eric Brown’s lab)
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|  | **Potential Hazards** | **Engr Cntl, Haz. Mitigation Device, PPE** |
| 1. Ampoule sealing in Chemistry Dept.
 | Reaction with water or air | Nitrogen glove bag, mineral oil receptacle for waste, safety glasses |
| 1. Prepare an open container of mineral oil as an emergency measure in case the sample tube becomes cracked or damaged. The container should be sufficiently wide enough and deep enough to completely cover the sample tube with oil when immersed. Remove a sample tube from transport container and place into portable glovebag with Schlenk line attachment. Prior to sealing bag, separate the clamp screw and o-ring from the Schlenk line attachment. **Note: be careful with the glovebag (plastic) as it is susceptible to damage from sharp objects or heat.**
2. Seal and purge glovebag with nitrogen or argon (UHP 99.9995% purity) approximately two bag changes.
3. Remove tape and tube cap. Slide o-ring **seal onto the end of the tube and insert EPR tube into Schlenk line attachment. Ensure valve is closed**. Slide attachment cap up the EPR tube and secure. Insure the o-ring seats into the attachment.
4. Remove assembly from the bag and attach to the Schlenk line in the fume hood.
5. Vacuum purge and refill with nitrogen or argon (UHP 99.9995% purity) 2 times using Schlenk line apparatus.
6. Open vacuum valve on Schlenk line attachment, leave open through the remainder of the procedure (tube is under continuous vacuum).
7. Immerse lower portion of EPR tube in the bottle of liquid nitrogen for a minute.
8. With a small neutral flame (small inner blue flame), heat the tube equally by moving the flame around the perimeter. When tube softens sufficiently, twist off to seal. **In the event that the lower portion of the tube begins to heat above ~30°C, immerse into the liquid nitrogen.**
9. Place sealed tube and packing material into transport container.
10. Close valve(s).
11. Repeat steps as required to complete all samples.

**In the event of a crack forming in any tube, immediately immerse the tube into the container of mineral oil, insuring that the sample is completely below the liquid surface. Add mineral oil to top off container and screw on lid to seal at end of this step.** |
|  | **Potential Hazards** | **Engr Cntl, Haz. Mitigation Device, PPE** |
| 1. Unloading Cs samples from furnace
 | Reaction with water or air | Argon glove box, mineral oil receptacle for waste, safety glasses, extraction rod |
| 1. Increase gas flow (UHP argon or nitrogen) through furnace tube to > 400 sccm.
2. Disconnect ¼” Swagelok connections from exhaust side cap on furnace tube.
3. Place rod in the deflated glovebag.
4. Enclose the end of the furnace tube with the glovebag. Allow glove bag to inflate and purge for several minutes (approximately two full bag exchanges).
5. Remove exhaust cap from furnace tube. Using rod, extract sample boat from center of furnace.
6. Remove extraction rod from glovebag through exhaust outlet.
7. Seal glovebag and remove from furnace tube.
8. Turn off gas to furnace.
9. Deflate glovebag through exhaust outlet until the volume is reduced enough to fit into glove box exchange chamber. Be careful to not allow a backflow of air during deflation.
10. Load into exchange chamber, open exhaust outlet of glove bag, quickly secure chamber door and initiate vacuum purge. Cycle the exchange chamber three times.
11. In the glove box, remove sample ampoules and furnace endcap from the glove bag.
12. Remove the glove bag and furnace endcap from the glove box through the exchange chamber. Cycling is not necessary. Proceed to next step to extract samples from ampoules.
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|  | **Potential Hazards** | **Engr Cntl, Haz. Mitigation Device, PPE** |
| 1. Unloading Cs samples from tubes
 | Reaction with water or air | Mineral oil receptacle for waste, safety glasses, sample collection container |
| 1. Transfer waste container (in secondary containment) and sample collection container into glove box through transfer chamber. Cycle 3 times.
2. Transport the heat treated samples from the furnace to the glove box using the glovebag. Crack seal on glovebag and place into the transfer chamber.
3. Cycle the transfer chamber 3 times then move glovebag into the glove box.
4. Remove sample tubes from glovebag.
5. Using pliers (or ampoule cutter) carefully snap off the end of the tube. Pour the samples into a weigh boat or petri dish. Discard tube (and all other non-sample remnants) into the waste container.
6. Remove samples and waste container from glove box.
7. **As a precaution: remove samples from glove box in a sealed container under an inert atmosphere (argon/nitrogen). Move to the fume hood and carefully open container in the hood with the sash as low as practical in case remnant Cs on the surface or the samples reacts unfavorably.**
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|  | **Potential Hazards** | **Engr Cntl, Haz. Mitigation Device, PPE** |
| 1. Cleanup and Waste
 | Reaction with water or air | Glove box, safety glasses, lab coat |
| 1. Discard tubes (and all other non-sample remnants) into the waste container.
2. Place mineral oil waste container into a secondary container and seal so that an argon atmosphere remains.
3. Remove samples and waste container from glove box.
4. Label waste container: **Contains cesium and broken glass stored under an inert atmosphere. WARNING: Do not open in air due to risk of fire or explosion.**
5. Arrange with EHS to pick up waste immediately.
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|  | **Potential Hazards** | **EC, Haz. Mitigation Device, PPE** |
| 1. Chemical Storage
 | Reaction with water or air | Glove box, or sealed jar in secondary sealed container under inert atmosphere.  |
| 1. Place unused Cesium in a well-labeled, sealed, container in the glove box or
2. Place the sealed container in a secondary container under inert atmosphere with the following label:

***Contains cesium stored under an inert atmosphere. WARNING: Do not open in air due to risk of fire or explosion.***1. Store in the flammables cabinet.
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|  | **Potential Hazards** | **EC, Haz. Mitigation Device, PPE** |
| 1. Spill and Incident
 | Reaction with water or air | Mineral oil receptacle,  |
| * + In the event of a spill place unreacted waste into mineral oil receptacle.
	+ In the event of a fire, extinguish with special powder for metal fires; class D fire agent, dry salt or sand. **WARNING**: Do not use water due to risk of fire or explosion.
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