ORNL/TM-2024/3334

# Plutonium-238 Loadout Calorimeter: Return to Service


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**March 2024**

OAK RIDGE National Laboratory

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Plutonium-238 Supply Program

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Prepared by
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managed by
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## ABBREVIATIONS

| | |
|---|---|
| FSO | Fuel Storage Outer |
| ORISC | Oak Ridge Inner Shipping Capsule |
| TC | Thermocouple |

# 1. INTRODUCTION

The calorimeter in the transfer area in Building 7920 at the US Department of Energy's Oak Ridge National Laboratory is used to measure the amount of $^{238}$Pu in the Plutonium Heat Source Oxide. The calorimetry measurement is taken after the oxide is packaged in an ORISC (Oak Ridge Inner Shipping Capsule)/FSO (Fuel Storage Outer) assembly before loading into the Containment Vessel and shipping container. The equipment is used in three specific configurations: zero measurement (nothing in the sample cup), FSO measurement (actual measurement of $^{238}$Pu), and reference calibration measurement (using the reference FSO as a known heat source). System checks were performed in July 2023 in preparation for scheduled material loadout. During these checks, personnel discovered that the reference FSO had experienced an electrical short in the heater cable and was no longer functional. The calorimeter was in the reference calibration measurement configuration at this time. Facility management was notified when the damage was discovered. The data from the Yokogawa recorder were pulled and analyzed for a span of time between September 2022 and July 2023. An unusual spike was found in the sample cup temperature data that started on October 19, 2022, and ended on October 20, 2022. The sample cup reached a temperature of 191.1°C. The maximum allowable temperature for the calorimeter thermopile to reach is 220°C based on the manufacturer statement provided in Appendix B of this report [1]. The calorimeter thermopile and the sample cup thermocouple are physically close to each other; therefore, the data suggest that the calorimeter itself never saw a temperature in excess of 191.1°C. Physical damage present on the cable, tape, and insulation suggests that the temperature reached a higher value in some locations within the sample cup than the data shows. Because of this discrepancy, testing of the calorimeter was required to confirm that the calorimeter is functioning the same as it was before the electrical short. The intent of this report is to assess the functionality of the calorimeter and determine if it is acceptable to use for $^{238}$Pu loadout material measurement. Testing showed that the calorimeter is functionally equivalent to the initial calibration that was performed in the summer of 2021, and thus, the calorimeter was cleared to be used for its intended purposes.

## 2. PHYSICAL DAMAGE

The physical damage found in July 2023 affected the heater cable, heater thermocouple (TC) tape, and the insulation on the outside of the sample cup. The calorimeter was in the configuration shown in Figure 1 when the damage took place.

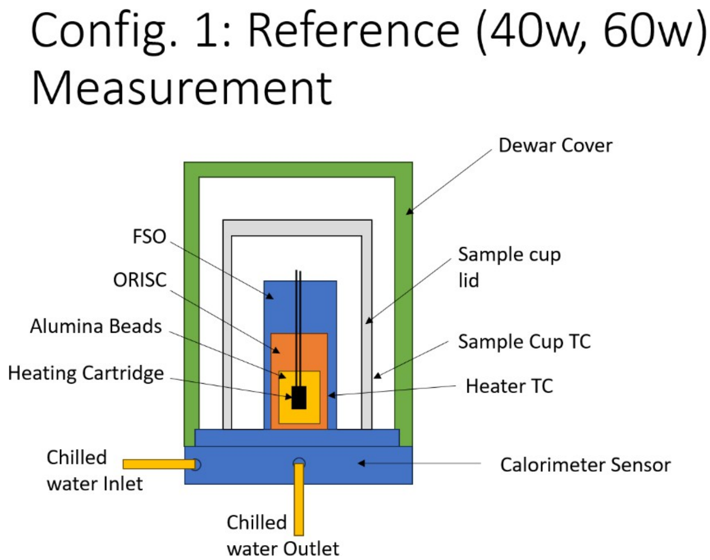


**Figure 1. Reference FSO measurement configuration.**

The reference FSO is an electric heater inside of an ORISC and FSO that was used to simulate a real $^{238}$Pu-filled FSO. It was supplied with a known wattage and was used to confirm the calibration of the calorimeter. All the TCs are type K, and the bare junctions were taped to the metal surface in each location. The heater cable was found to be shredded in July of 2023, as shown in Figure 2.


**Figure 2. Reference FSO with damaged heater wires.**

The cable was damaged by rubbing against the internal threads in the through-hole on the lid of the FSO/ORISC assembly. Over time, the threads destroyed the heat shrink, which allowed the wires to create a direct short in the circuit, melting the heat shrink casing and creating the temperature spike seen in the data analysis. This damage to the heat shrink was determined to be the root cause of the fault incident.

The heater thermocouple was located on the outside of the ORISC and secured in place with high-temperature tape. This tape was reported to be RG48 high-temperature glass cloth tape from Intertape Polymer Group. The RG48 tape spec sheet states that it can “withstand exposure to temperatures up to and in excess of 260°C” [2]. Following the discovery of damage to the calorimeter in July 2023, the tape had been charred and burned off, suggesting that the ORISC surface temperature has exceeded the tape’s rated temperature of 260°C. No photos exist of the tape as found. Although this burnt tape does not definitively conclude that the calorimeter thermopile felt temperatures of 260°C or more, it does suggest that the reference FSO likely surpassed that temperature for some amount of time.

The final evidence of physical damage was the insulation on the outside of the sample cup. Figure 3 shows the state of the insulation as found in July 2023.

**Figure 3. Discolored insulation outside the sample cup.**

The insulation shows general discoloration where it contacted the sample cup along with some charring and black residue on top of it. The discoloration could be due to excessive heat or could be where the fumes from the melted wire were filtered through the insulation. Very excessive heat would have been necessary to char the insulation in this way. More likely, this damage was due to fumes from the electrical short. If fumes were present when the wire was shorted, it would have escaped out the top of the FSO/ORISC assembly and then exited the assembly out the bottom of the sample cup lid, depositing particulate on the insulation layer.

The heater TC was disconnected during this time, thus, no data exists for the temperature attained. Facility management was notified by email on July 17, 2023 when the damage was discovered.

## 3. INITIAL DATA ANALYSIS

Once the damage was discovered, data were pulled from the Yokogawa logger and analyzed with the universal viewer software. The last known test of the calorimeter with the heater thermocouple connected was between September 28, 2022, and October 3, 2022 (this disconnection occurred to test the calorimeter calibration before measuring the actual $^{238}$Pu FSO). The heater TC was only connected during the test, so no heater TC data were recorded for the time before or after.

These data line up with the data sheet dates and times shown in Figures 4 and 5. The calorimeter was known to be functioning properly during this time.

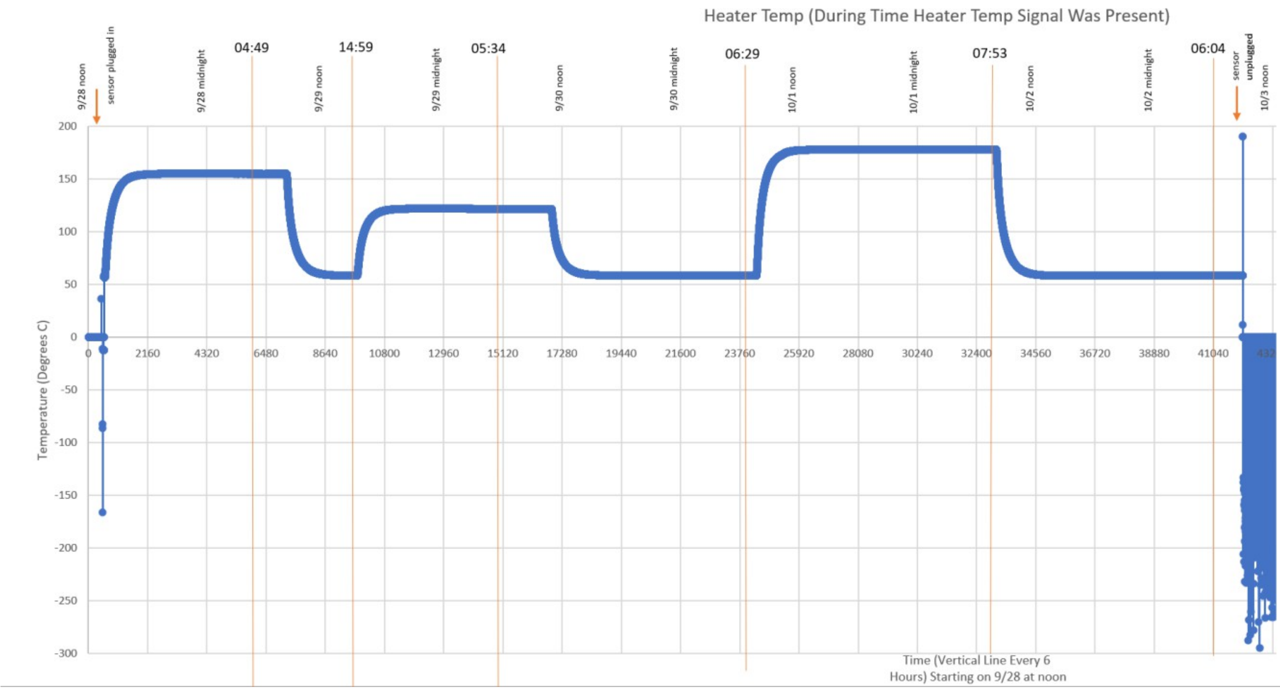


**Figure 4. Heater temperature data plotted over time.**

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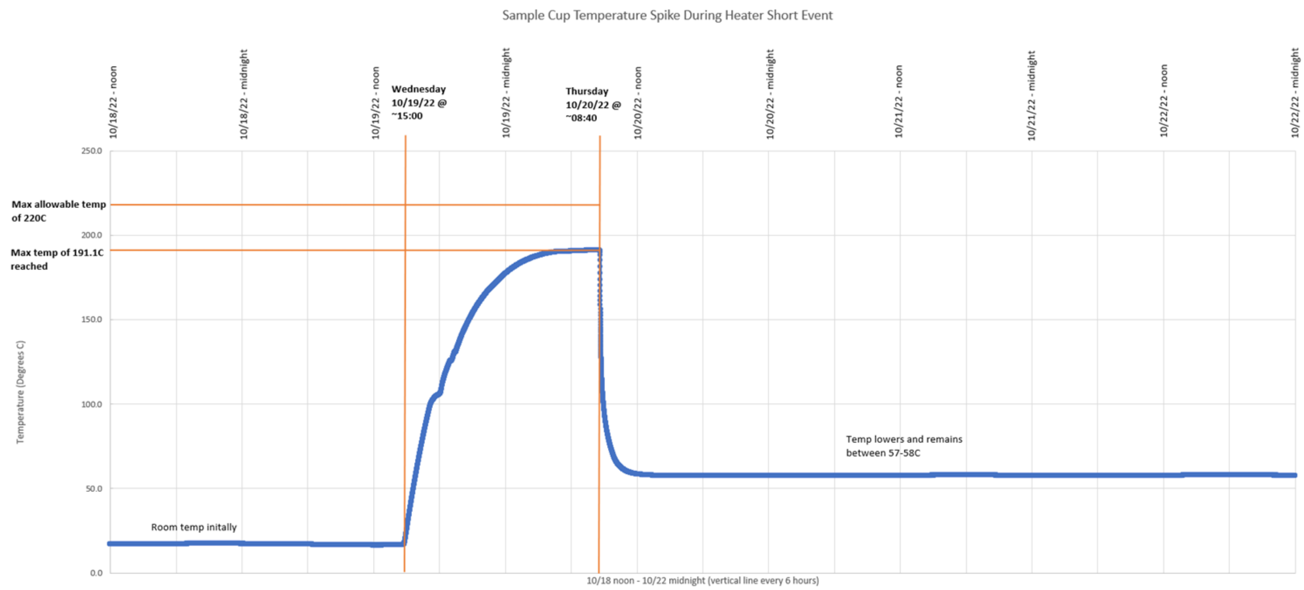
|---|---|

## APPENDIX D
### Data Sheet for Calorimetric Determination of Watts Per FSO

| Date | Container ID or "zero" | Level Trace Verified | | | 1-hr Level Trace End | | | Recorder Calorimeter Measurement | | | | | | Initials |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Calorimeter mV @ time interval, min. | | | | | Time Complete | |
| | | Time | Calorimeter mV | Water Bath Temp °C | Time | Calorimeter mV | Water Bath Temp °C | 1 | 2 | 3 | 4 | 5 | | |
| 9/28/22 | Zero ~~Heater 60~~ | 0940 | -0.0434 | 60 | 1040 | -0.0427 | 60 | -0.0427 | -0.0427 | -0.0424 | -0.0430 | -0.0423 | 1044 | CD/JR |
| 9/29/22 | Heater 60W | 0345 | 0.7445 | 60 | 0445 | 0.7439 | 60 | 0.7439 | 0.7439 | 0.7439 | 0.7437 | 0.7446 | 0449 | CD/JR |
| | 60.01 Act. Watt | 27.5 Am Temp. | ↑ | | | | | | | | | | | |
| 9/29/22 | Am. Temp 27.6 Zero | 1354 | -0.0381 | 60 | 1454 | -0.0384 | 60 | -0.0384 | -0.0380 | -0.0378 | -0.0382 | -0.0379 | 1459 | |
| 9/30/22 | Heater 40W AW38.28 / 30.0 Am Temp | 0430 | 0.4680 | 60 | 0530 | 0.4675 | 60 | 0.4675 | 0.4665 | 0.4664 | 0.4663 | 0.4674 | 0534 | CD/JR |
| 10/1/22 | Am-30.2 Zero | 0525 | -0.0391 | 60 | Am-29.5 0625 | -0.0396 | 60 | -0.0396 | -0.0397 | -0.0397 | -0.0397 | -0.0394 | 0629 | CD |
| 10/2/22 | AW 74.84 Amb 27.9 Heater 75W | 0649 | 0.9448 | 60 | 0749 | 0.9433 | 60 | 0.9433 ~~9.433~~ | 0.9433 | 0.9436 | 0.9438 | 0.9435 | 0753 | CD |
| 10/3/22 | Amb: 28.3 Zero | 0500AM | -0.0402 | 60 | 0600 | -0.0396 | 60 | -0.0396 | -0.0396 | -0.0396 | -0.0393 | -0.0393 | 0604 | |

**Figure 5. Data sheet for calorimetric determination of watts per FSO.**

Some data exist from the calorimeter thermopile and sample cup temperature that correspond to the actual $^{238}$Pu FSO measurements between October 3 and 8, 2022, but after October 8, the heater controller appears to have been turned down to 0 W, and the sample cup temperature lowers to room temperature. The heater controller appears to have never been fully turned off during this time. The unit remained at room temperature until a spike was seen on October 19, 2022. On October 19 at approximately 3:00 p.m., the sample cup temperature started a spike from room temperature up to a maximum of 191.1°C on October 20 at approximately 8:40 a.m. On October 20 at approximately 8:40 a.m., the temperature rapidly decreased from 191.1°C to the water bath set point temperature of approximately 57°C and remained at that set point temperature for several weeks. On December 2, 2022, the unit appears to have been turned off, and the sample cup temperature lowered to room temperature. Because this electrical short happened between campaigns, the damage was not found immediately. The data are shown in the graphs in Figure 6.



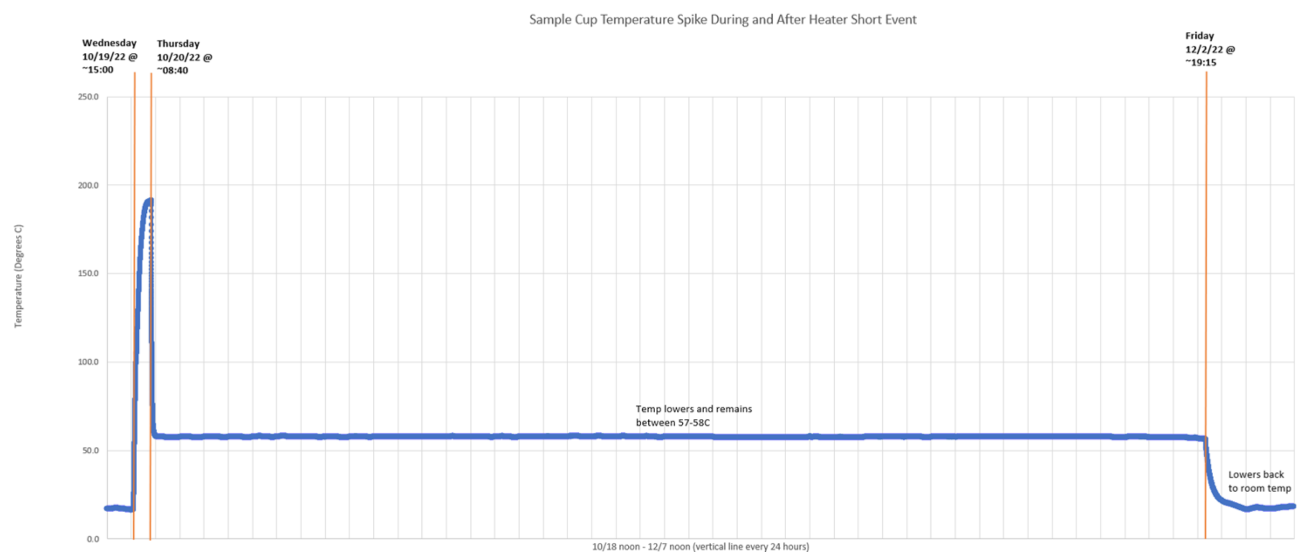




**Figure 6. Sample cup temperature data during heater spike.**

Although the data presented in Figures 4, 5, and 6 were exported to Microsoft Excel and formatted for ease of viewing, the raw data from the universal viewer software demonstrates the heater wattage and calorimeter thermopile voltage at the exact time that the temperature spike began, on October 19. The data are shown in Figure 7 (the red line is the calorimeter thermopile voltage, the green is the sample cup temperature, and the purple is the heater wattage).

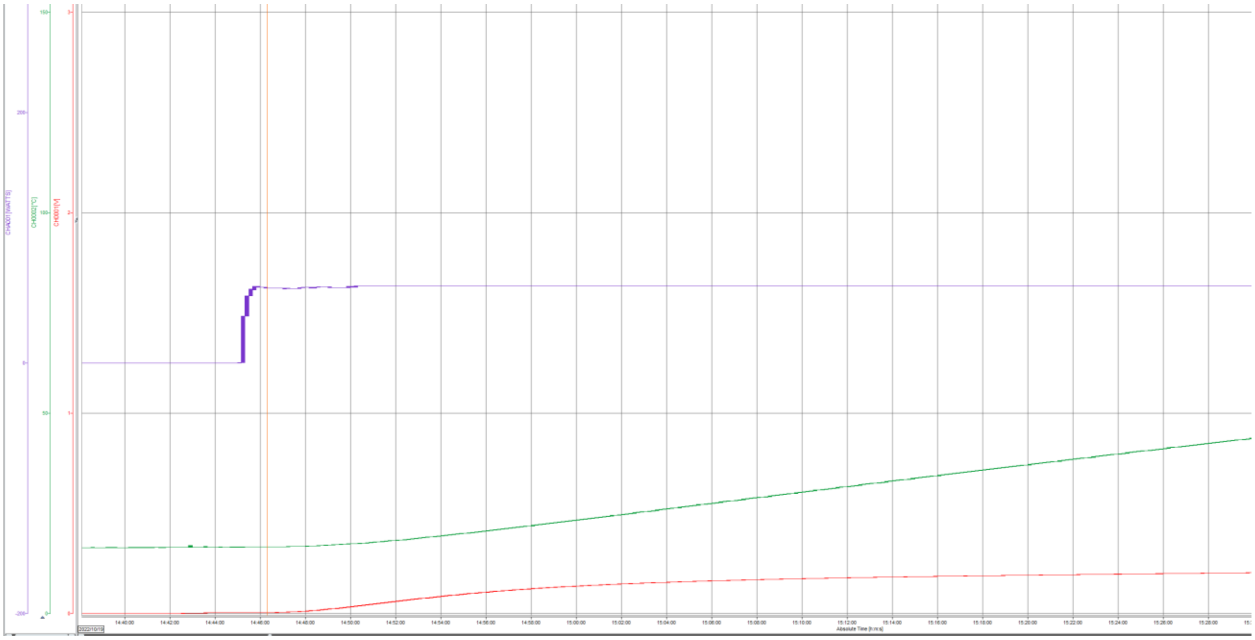


**Figure 7. Raw data plot when temperature spike began.**

The raw data also show what happened when the temperature dropped, on October 20. The data are shown in Figure 8 (the red line is the calorimeter thermopile voltage, the green is the sample cup temperature, and the purple is the heater wattage).

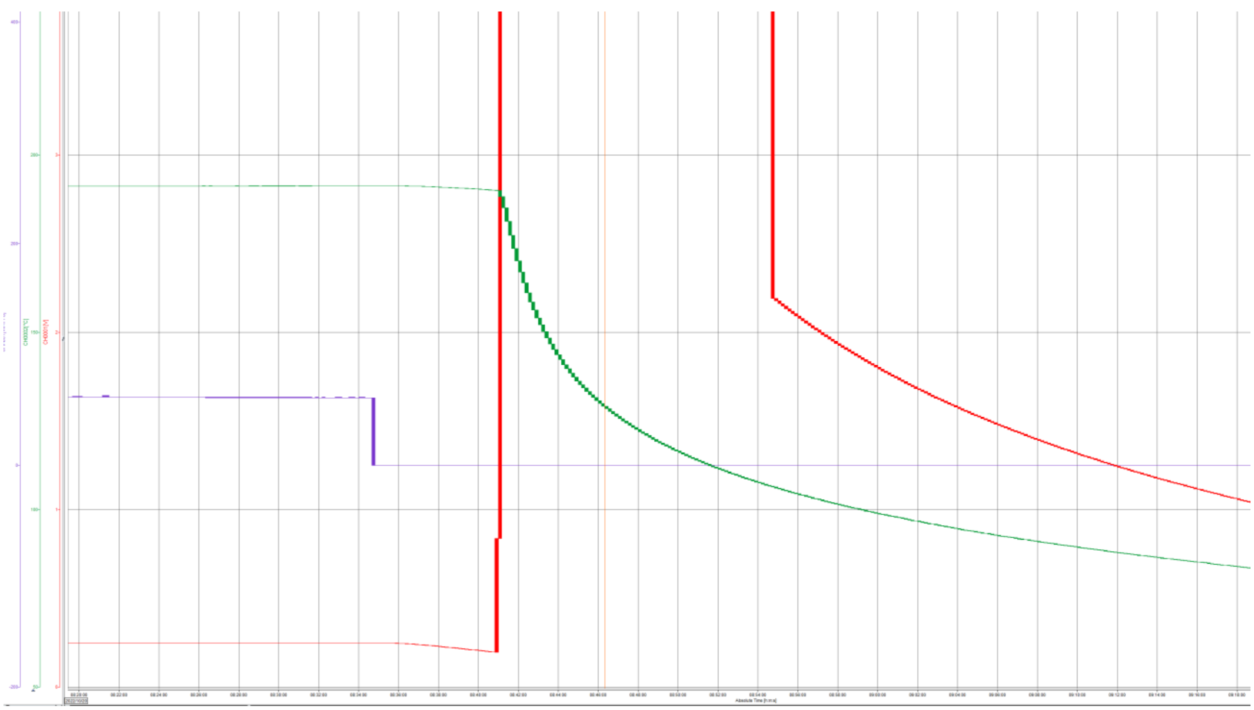


**Figure 8. Raw data plot when temperature spike ended.**

Gaps exist in data logs from December 23, 2022, to March 28, 2023, and again from April 18, 2023, to July 5, 2023, but data before December 23, 2022, and between March 28, 2023, and April 18, 2023, show that the sample cup temperature was at room temperature. No uses of the calorimeter were recorded after

December 2, 2022. This suggests that the calorimeter remained at room temperature until the damage to it was discovered in July 2023.

## 4. HARDWARE AND SOFTWARE CHANGES

After the data had been analyzed and the physical damage had been evaluated as detailed in the previous sections, several hardware and software changes were made to the calorimeter prior to testing again to ensure that this fault case would not be repeated. The following changes were made to the calorimeter components.

- The threads were machined out of the threaded hole at the top of the FSO and ORISC assembly to ensure that they would not fray the cable over time.
- The heater TC was replaced with a new one and new high-heat tape was used to hold it in place.
- The alumina beads were replaced with new, equivalent, alumina beads.
- A new heater was installed into the ORISC and the ends were crimped and heat-shrunk to replace the failed heater.
- A maximum allowable sample cup temperature cutoff of 100°C was added to the Yokogawa program to prevent future failures due to overheating (the maximum sample cup temperature during normal testing was determined to be 87.5°C).
- Ambient temperature logging was added to the Yokogawa program.
- The water level was confirmed to be at the correct level.
- The sample cup insulation was replaced.

In addition to hardware and software changes, a configuration drawing was made for the reference FSO. This drawing is presented in Appendix C.

# 5. TESTING PERFORMED

To confirm that the calorimeter was fully functional and ready for use, a full reference test was completed and compared with the results from the previous, summer 2021, calibration. The raw data tables are presented in Appendix A. The testing completed during the summer of 2021 was used as a baseline for comparison because it was completed as a functional test of the equipment upon receipt of the calorimeter from the manufacturer. The testing to check the calorimeter after the overheat event occurred from December 4 to 11, 2023.

The draft procedure NNFD-7920-OP-264 [4] was used as a guide for the testing. The recirculating water bath was turned on and set to 60°C, and a zero-measurement voltage reading was taken once the system had equilibrated. After the zero measurement was acquired, the heater was turned on and set to 40 W. Once the system had equilibrated to 40 W, the measurement was taken. This process was repeated at both 60 W and 75 W.

## 6. DATA ANALYSIS OF TESTING PERFORMED

After collecting all the voltage measurements, the data were shifted to account for the offset of the zero reading. Then, the data were plotted, and a linear regression line applied, shown in Figure 9.

| Data from 12/4/23-12/11/23 Measurements | | |
|---|---|---|
| Watts | Volts | Volts after adjusting zero |
| 0 | -0.0528 | 0.0000 |
| 40 | 0.503 | 0.5558 |
| 60 | 0.7626 | 0.8154 |
| 71.74 | 0.9285 | 0.9813 |

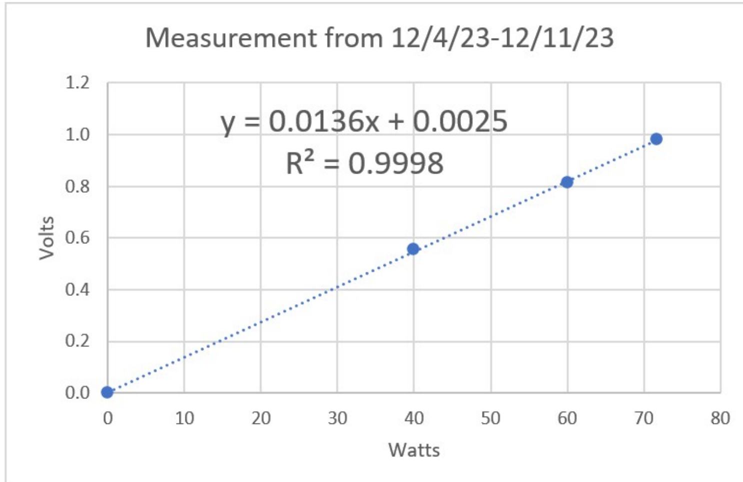



**Figure 9. Data plot from December 4–11, 2023.**

The 2021 calibration data were also zero-adjusted and plotted using the same method as that used for the data in Figure 9. This data is shown in Figure 10.

| Summer 2021 Calibration | | |
|---|---|---|
| Watts | Volts | Volts after adjusting zero |
| 0.00 | -0.0318 | 0.0000 |
| 40.01 | 0.5285 | 0.5603 |
| 74.46 | 0.9709 | 1.0027 |

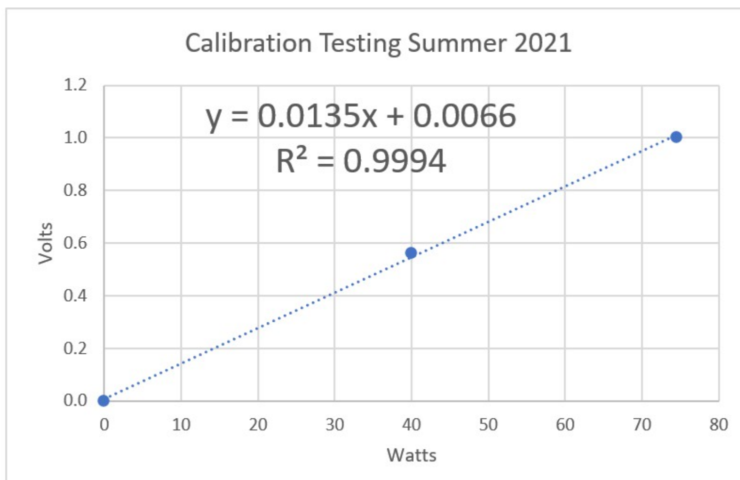



**Figure 10. Data plot from summer 2021 calibration.**

The percent error between the new data and the 2021 calibration data was determined by implementing the new data wattages in the calibration equation ($y = 0.0135x + 0.0066$) to calculate the expected voltages for each point. Then, percent error was calculated using the equation;

$$Percent\ error = \left(\frac{Measured\ value - Expected\ value}{Expected\ value}\right) \times 100.$$

The average percent error between the new data and 2021 calibration data was calculated as 0.72%, as shown in Figure 11.

| New Data (12/5/23-12/8/23) | | Expected voltage based on summer 2021 calibration (y = 0.0135x +0.0066) | Difference | Percent Error |
|---|---|---|---|---|
| Watts | Volts | | | |
| 0 | 0 | 0.0066 | -0.0066 | n/a |
| 40 | 0.5558 | 0.5466 | 0.0092 | 1.683 |
| 60.01 | 0.8154 | 0.816735 | -0.001335 | -0.163 |
| 71.74 | 0.9813 | 0.97509 | 0.00621 | 0.637 |

| Average Percent Error |
|---|
| 0.72 % |

**Figure 11. Average percent error table between new data and 2021 calibration.**

While investigating data from previous testing, the test data from September 28, 2022, to October 3, 2022, was also compared with the initial 2021 calibration data. These data were used to determine if the calorimeter was functioning properly prior to measuring the first shipment of $^{238}$Pu. The average percent error between the pre-$^{238}$Pu shipment testing and the 2021 calibration data was calculated to be −3.46%, as shown in Figure 12.

| Pre-Pu Shipment Testing (9/28/22-10/3/22) | | Expected voltage based on summer 2021 calibration (y = 0.0135x +0.0066) | Difference | Percent Error |
|---|---|---|---|---|
| Watts | Volts | | | |
| 0 | 0 | 0.0066 | -0.0066 | n/a |
| 38.28 | 0.507 | 0.52338 | -0.01638 | -3.130 |
| 60.01 | 0.784 | 0.816735 | -0.032735 | -4.008 |
| 74.84 | 0.984 | 1.01694 | -0.03294 | -3.239 |

| Average Percent Error |
|---|
| -3.46 % |

**Figure 12. Average percent error table between pre-$^{238}$Pu shipment testing and 2021 calibration.**

This figure shows that the data collected after the overheat event were actually closer to the initial calibration than the data collected to confirm that the calorimeter was functioning properly prior to measuring the initial shipment of $^{238}$Pu. Both percent errors fall within the 5% calibration accuracy claimed by the manufacturer.

## 7. CONCLUSION

After assessing the physical damage and analyzing the data from the Yokogawa data logger, an overheat event clearly occurred between October 19 and 20, 2022. After this event, typical damage associated with an overheat event was observed with the FSO/ORISC assembly, as well as with the surrounding insulation. The damaged parts of the unit were replaced, and actions were taken to prevent future reoccurrences.

Though there is no way to confirm the exact temperature the calorimeter thermopile experienced, the data show that the sample cup TC registered 191.1°C. The sample cup TC location and the calorimeter thermopile location are close enough to each other that the temperatures can be expected to be nearly equal. The two locations are roughly the same distance away from the heater, with the calorimeter thermopile located closer to the temperature-controlled water bath, which implies that the use of the sample cup TC reading is a conservative estimate for the calorimeter temperature. The data and location of the TCs suggests that the calorimeter TC did not surpass 191.1°C.

The physical evidence of the overheat event suggests that some parts of the FSO and ORISC assembly did reach a higher temperature than 191.1°C; however, the calorimeter thermopile itself was insulated against the temperature spike equivalently (if not more insulated) to the sample cup TC. Because the data showed that the sample cup temperature did not reach 220°C (the maximum allowable temperature for the calorimeter thermopile to experience), testing was completed on the unit, clearing it for use.

Testing demonstrated that the calorimeter functioned equivalently to initial calibration data, with a 0.72% error. The test data, in addition to the data analysis from the logger during the temperature spike, show that the calorimeter is not malfunctioning and is cleared to be used for intended purposes. Additional safeguards have been put in place to prevent future reoccurrence of this fault scenario.

# 8. REFERENCES

[1] International Thermal Instrument Company. *ORNL Pu Water Cooled, Single Cup Calorimeter* (Del Mar, California: International Thermal Instrument Company, 2020).

[2] Intertape Polymer Group. *Technical Data Sheet: RG48 High Temperature Glass Cloth Tape* (Sarasota, Florida: Intertape Polymer Group, 2013).

[3] Nonreactor Nuclear Facilities Division. *Calorimetry for $^{238}$Pu*. NNFD-7920-OP-264 (Oak Ridge, Tennessee: Oak Ridge National Laboratory).

# APPENDIX A. TEST DATA

# APPENDIX A. TEST DATA

| Time | Thermopile V | Sample Cup Temp C | Heater Temp C | Watts | Bath Set Temp C | Bath Temp C | Comments/Notes |
|---|---|---|---|---|---|---|---|
| 1528 | -0.0146 | 39.2 | 24.7 | 0 | 40 | 40 | Assumed Zero |
| 1529 | -0.0146 | 39.2 | 24.7 | 0 | x | x | |
| 1530 | -0.0149 | 39.2 | 24.7 | 0 | x | x | |
| 1531 | -0.0148 | 39.2 | 24.7 | 0 | x | x | |
| 1532 | -0.0145 | 39.2 | 24.7 | 0 | x | x | |
| 1533 | -0.0148 | 39.2 | 24.7 | 0 | x | x | |
| 1704 | -0.0145 | 39.2 | 24.6 | 0 | 40 | 40 | |
| 1511 | 0.5379 | 51.5 | 110.6 | 40.01 | x | x | Assumed Equilibrium |
| 1512 | 0.5379 | 51.5 | 110.6 | 40.01 | x | x | |
| 1513 | 0.5375 | 51.5 | 110.6 | 40.01 | x | x | |
| 1514 | 0.5374 | 51.5 | 110.6 | 40.01 | x | x | |
| 1515 | 0.538 | 51.5 | 110.6 | 40.01 | x | x | |
| 1315 | 0.5374 | 51.5 | 111.9 | 40.01 | x | x | Assumed Equilibrium |
| 1410 | 0.5385 | 51.5 | 112 | 40.01 | x | x | |
| 1411 | 0.5377 | 51.5 | 112 | 40.01 | x | x | |
| 1412 | 0.5382 | 51.5 | 112 | 40.01 | x | x | |
| 1413 | 0.538 | 51.5 | 112 | 40.01 | x | x | |
| 1414 | 0.5384 | 51.4 | 112 | 40.01 | 40 | 40 | |
| 1417 | 0.5379 | 51.5 | 112 | 40.01 | 40 | 40 | |
| 920 | -0.0165 | 39.2 | 39.4 | 0 | 40 | 40 | 7/2/21 Final (3rd) Zero. Heater off but in chamber. |
| 1016 | -0.0163 | 39.2 | 39.4 | 0 | x | x | |
| 1017 | -0.0165 | 39.2 | 39.4 | 0 | x | x | |
| 1018 | -0.0164 | 39.2 | 39.4 | 0 | x | x | |
| 1019 | -0.0164 | 39.2 | 39.4 | 0 | x | x | |
| 1020 | -0.0167 | 39.2 | 39.4 | 0 | 40 | 40 | |
| 1635 | -0.0401 | 57.7 | 58.6 | 0 | 60 | 60 | First Zero - Confirmed |
| 1636 | -0.0399 | 57.6 | 58.6 | 0 | 60 | 60 | |
| 1637 | -0.04 | 57.6 | 58.6 | 0 | 60 | 60 | |
| 1638 | -0.0397 | 57.6 | 58.6 | 0 | 60 | 60 | |
| 1639 | -0.0399 | 57.6 | 58.6 | 0 | 60 | 60 | |
| 1640 | -0.04 | 57.6 | 58.6 | 0 | 60 | 60 | |
| 530 | -0.0391 | 57.7 | 58.6 | 0 | x | x | 7/14/21 - Reconfirm Zero |
| 630 | -0.0392 | 57.7 | 58.6 | 0 | x | x | |
| 631 | -0.0391 | 57.7 | 58.6 | 0 | x | x | |
| 632 | -0.0393 | 57.7 | 58.6 | 0 | x | x | |
| 633 | -0.0393 | 57.7 | 58.6 | 0 | x | x | |
| 634 | -0.0393 | 57.7 | 58.6 | 0 | x | x | |
| 1518 | 0.9699 | 79.3 | 185.4 | 74.22 | x | x | Assumed Equilibrium |
| 1618 | 0.9692 | 79.3 | 185.5 | 74.22 | 60 | 61 | |
| 1619 | 0.9694 | 79.3 | 185.5 | 74.22 | 60 | 61.1 | |
| 1620 | 0.9695 | 79.3 | 185.5 | 74.22 | 60 | 61.1 | |
| 1621 | 0.9691 | 79.3 | 185.5 | 74.22 | 60 | 61.1 | |
| 1622 | 0.9689 | 79.3 | 185.5 | 74.22 | 60 | 61.1 | |
| 1603 | 0.9652 | 79.3 | 185.2 | 74.22 | 60 | 61.1 | Assumed Equilibrium |
| 1604 | 0.9652 | 79.2 | 185.2 | 74.22 | 60 | 61.1 | |
| 1605 | 0.9654 | 79.2 | 185.2 | 74.22 | 60 | 61.1 | |
| 1606 | 0.9655 | 79.3 | 185.2 | 74.22 | 60 | 61.1 | |
| 1607 | 0.9653 | 79.3 | 185.2 | 74.22 | 60 | 61.1 | |
| 856 | -0.0397 | 57.7 | 58.6 | 0 | 60 | 60 | Confirmed |
| 857 | -0.039 | 57.7 | 58.6 | 0 | 60 | 60 | |
| 858 | -0.0397 | 57.7 | 58.6 | 0 | 60 | 60 | |
| 859 | -0.0391 | 57.7 | 58.6 | 0 | 60 | 60 | |
| 900 | -0.0397 | 57.7 | 58.6 | 0 | 60 | 60 | |
| 1718 | -0.0403 | 57.6 | 58.5 | 0 | 60 | 60 | Assumed Zero/Equilibrium |
| 1719 | -0.0398 | 57.6 | 58.5 | 0 | 60 | 60 | |
| 1720 | -0.0399 | 57.6 | 58.5 | 0 | 60 | 60 | |
| 1721 | -0.0398 | 57.6 | 58.5 | 0 | 60 | 60 | |
| 1722 | -0.0396 | 57.6 | 58.5 | 0 | 60 | 60 | |
| 1727 | -0.0396 | 57.6 | 58.5 | 0 | 60 | 60 | |
| 1800 | -0.0397 | 57.6 | 58.5 | 0 | x | x | |
| 600 | -0.0396 | 57.6 | 58.5 | 0 | x | x | 7/7/2021 - First Zero Continue |
| 700 | -0.0399 | 57.7 | 58.5 | 0 | x | x | |
| 747 | -0.0397 | 57.7 | 58.6 | 0 | 60 | 60 | |
| 1326 | 0.5058 | 69 | 131.8 | 40 | x | x | Assumed Equilibrium - 40W Setting |
| 1327 | 0.5059 | 69.1 | 131.9 | 40 | x | x | |
| 1328 | 0.5058 | 69 | 131.8 | 40 | x | x | |
| 1329 | 0.5057 | 69 | 131.9 | 40 | x | x | |
| 1330 | 0.5057 | 69 | 131.8 | 40 | 60 | 60 | |
| 1917 | 0.9781 | 79.6 | 186.8 | 75 | 60 | 61.1 | Assumed Equilibrium - 75W Setting |
| 1918 | 0.9784 | 79.6 | 186.8 | 75 | 60 | 61.1 | |
| 1919 | 0.9782 | 79.6 | 186.8 | 75 | 60 | 61.1 | |
| 1920 | 0.9786 | 79.6 | 186.8 | 75 | 60 | 61.1 | |
| 1921 | 0.9785 | 79.6 | 186.8 | 75 | 60 | 61.1 | |

**Figure A-1. Raw data of initial calibration at Oak Ridge National Laboratory.**

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testing to determine if still functional

APPENDIX D
Data Sheet for Calorimetric Determination of Watts Per FSO

| Date of measurement | Container ID or "zero" | Level Trace Verified | | | 1-hr Level Trace End | | | Recorder Calorimeter Measurement | | | | | | Initials | ambient temp |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Time | Calorimeter V | Water Bath Temp °C | Time | Calorimeter V | Water Bath Temp °C | Calorimeter V @ time interval, min. 1 | 2 | 3 | 4 | 5 | Time Complete | | |
| 12/5/23 | zero | 2:15 am | -0.0516 | 60°C | 3:15 am | -0.0528 | 60°C | -0.0528 | -0.0523 | -0.0525 | -0.0526 | -0.0527 | 3:19 am | HG/CD | 17°C |
| 12/6/23 | 40 Watts | 4:15 am | 0.5027 | 60°C | 5:15 am | 0.5029 | 60°C | 0.5029 | 0.5025 | 0.5029 | 0.5025 | 0.5028 | 5:19 am | HG/CD | 18.4°C |
| 12/7/23 | 60 watts | 4:00 am | 0.7633 | 60°C | 5:00 am | 0.7628 | 60°C | 0.7628 | 0.7627 | 0.7624 | 0.7628 | 0.7627 | 5:04 am | HG/CD | 16.3°C |
| 12/8/23 | ~~75~~ 71.74 watts | 4:30 am | 0.9300 | 60°C | 5:30 am | 0.9289 | 60°C | 0.9289 | 0.9281 | 0.9282 | 0.9287 | 0.9286 | 5:34 am | HG/CD | 17.5°C |
| 12/11/23 | zero | 6:00 am | -0.0525 | 60°C | 7:00 am | -0.0528 | 60°C | -0.0528 | -0.0529 | -0.0529 | -0.0531 | -0.0529 | 7:04 am | HG/CD | 17.4°C |

**Figure A-2. Data sheet from December 4–11, 2023, testing.**

| Container | | | 1-h Level Trace Start | | | 1-h Level Trace End | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Campaign or Run Number | ID | Start Date | Time | Calorimeter (V) | RCW Temp (C) | Time | Calorimeter (V) | RCW Temp (C) | Transfer Area Temp (C) | 1 | 2 | 3 | 4 | 5 | Time End | Avg. V | Before & After zero | Container V | Cantainer Watts |
| Zero | Zero | 09/28/22 | 9:40 | -0.0434 | 60.0 | 10:40 | -0.0427 | 60.0 | | -0.0427 | -0.0427 | -0.0424 | -0.0430 | -0.0423 | 10:44 | -0.043 | | | |
| **~60 watt Standard** | **60.01** | **09/29/22** | **3:45** | **0.7445** | **60.0** | **4:45** | **0.7439** | **60.0** | **27.5** | **0.7439** | **0.7439** | **0.7439** | **0.7437** | **0.7446** | **4:49** | **0.744** | **-0.040** | **0.784** | **60.01** |
| Zero | Zero | 09/29/22 | 13:54 | -0.0381 | 60.0 | 14:54 | -0.0384 | 60.0 | 27.6 | -0.0384 | -0.0380 | -0.0378 | -0.0382 | -0.0379 | 14:59 | -0.038 | | | |
| Zero | Zero | 09/29/22 | 13:54 | -0.0381 | 60.0 | 14:54 | -0.0384 | 60.0 | 27.6 | -0.0384 | -0.0380 | -0.0378 | -0.0382 | -0.0379 | 14:59 | -0.038 | | | |
| **~ 40 watt Standard** | **38.28** | **09/30/22** | **4:30** | **0.4680** | **60.0** | **5:30** | **0.4675** | **60.0** | **30.0** | **0.4675** | **0.4665** | **0.4664** | **0.4663** | **0.4674** | **5:34** | **0.467** | **-0.039** | **0.51** | **38.28** |
| Zero | Zero | 10/01/22 | 5:25 | -0.0391 | 60.0 | 6:25 | -0.0396 | 60.0 | 29.5 | -0.0396 | -0.0397 | -0.0397 | -0.0397 | -0.0394 | 6:29 | -0.040 | | | |
| Zero | Zero | 10/01/22 | 5:25 | -0.0391 | 60.0 | 6:25 | -0.0396 | 60.0 | 29.5 | -0.0396 | -0.0397 | -0.0397 | -0.0397 | -0.0394 | 6:29 | -0.040 | | | |
| **~75 watt Standard** | **74.84** | **10/02/22** | **6:49** | **0.9448** | **60.0** | **7:49** | **0.9433** | **60.0** | **27.9** | **0.9433** | **0.9433** | **0.9436** | **0.9438** | **0.9435** | **7:53** | **0.944** | **-0.040** | **0.98** | **74.84** |
| Zero | Zero | 10/03/22 | 5:00 | -0.0402 | 60.0 | 6:00 | -0.0396 | 60.0 | 28.3 | -0.0396 | -0.0396 | -0.0396 | -0.0393 | -0.0393 | 6:04 | -0.039 | | | |
| Zero | Zero | 10/03/22 | 5:00 | -0.0402 | 60.0 | 6:00 | -0.0396 | 60.0 | 28.3 | -0.0396 | -0.0396 | -0.0396 | -0.0393 | -0.0393 | 6:04 | -0.039 | | | |

**Figure A-3. Data sheet from pre-$^{238}$Pu shipment testing on September 28–October 3, 2022.**

# APPENDIX B. INTERNATIONAL THERMAL INSTRUMENT COMPANY DOCUMENTATION

# APPENDIX B. INTERNATIONAL THERMAL INSTRUMENT COMPANY DOCUMENTATION

INTERNATIONAL THERMAL INSTRUMENT COMPANY
P.O. Box 309, Del Mar, California, 92014
Phone (858) 755-4436 Fax (858) 755-6878 www.iticompany.com

ORNL Pu Water Cooled, Single Cup Calorimeter

This custom water cooled, single cup calorimeter was calibrated at three different Watt Inputs. By employing a ½" dimeter heater, imbedded in an aluminum reaction vessel model, then fully heat sunk with thermal compound and alumina nodules to the calorimeter sample cup. Two power inputs of 40.7 and 106.4 Watts were liberated within the enclosed calorimeter. The corresponding Calibration Constants are as follows:

**ORNL Pu Single Cup Calorimeter (Al Construction)**
*ITI Model No:* CA-100-ORNL-3.125"Dia. (Custom - #030)

$K_{40.7\ Watts}$ = **15.47 mVolts DC / Thermal Watt Input**

$K_{106.4\ Watts}$ = **15.30 mVolts DC / Thermal Watt Input**

*Calibration Accuracy* = 5 % ________ 03/27/2020

The stability of this unit was satisfactory throughout the requested power input regime. For most of the calibration process, a cooling water flow rate of **2 liters/minute at 60°C** seemed to be quite sufficient at keeping the temperature of heat sink at a constant temperature. The recorded temperatures of the upper Heat Release Chamber were 39.8°C @ 40.7Watts and 61.7°C @ 106.4Watts. **(NOTE: At no time should the temperature of this units Heat Release Chamber be allowed to exceed 220°C.)**

The overall response time of this unit was longer than the previous version. From a cold start, at 106.4 Watts input, equilibrium was achieved in 2:30 hours. Additionally, response time to variable power inputs, at temperature, seemed to be on the order of one hour. It is ITI's opinion, that the size of this single sensor Calorimeter has reached the limit for accurate and timely data acquisition.

Any further questions, please feel free to contact ITI Inc.

**Figure B-1. Manufacturer information.**

# APPENDIX C. REFERENCE FUEL STORAGE OUTER CONFIGURATION DRAWING

## APPENDIX C. REFERENCE FUEL STORAGE OUTER CONFIGURATION DRAWING

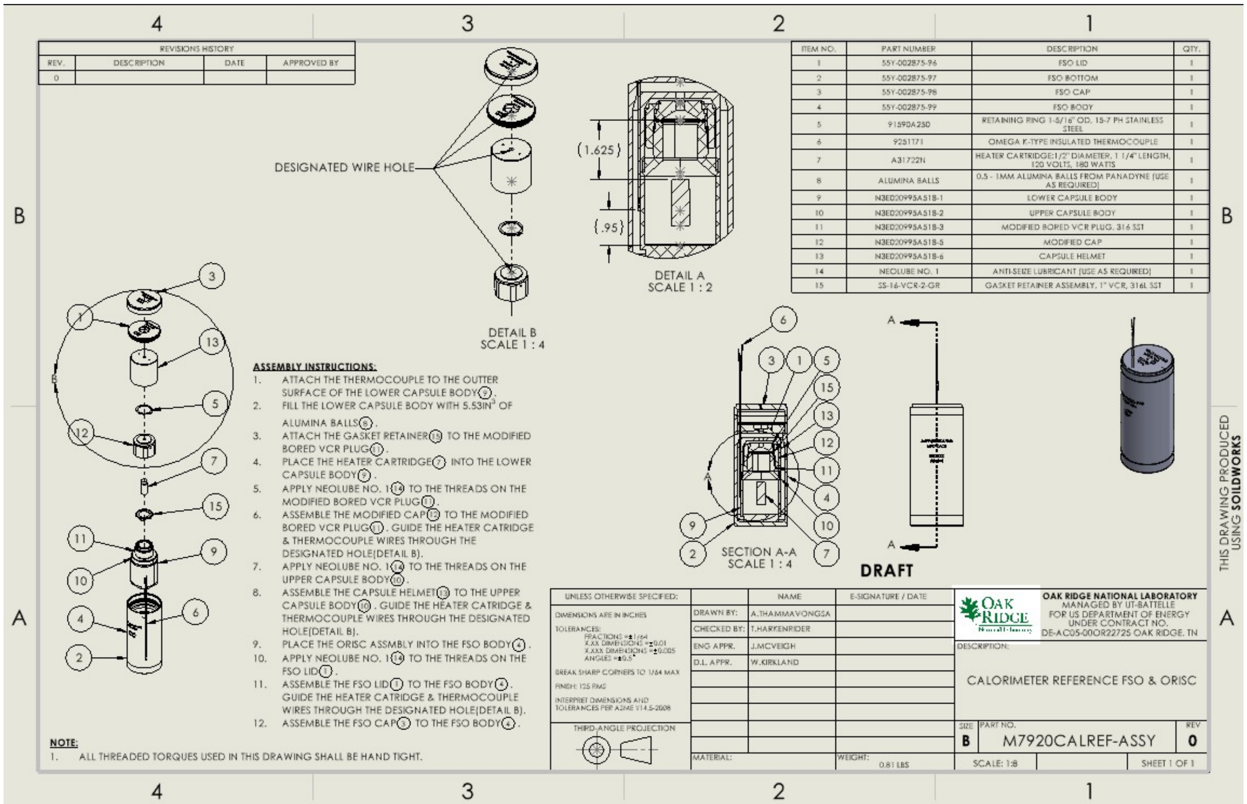


**Figure C-1. Fuel Storage Outer configuration drawing.**

# APPENDIX D. WORK PACKAGE PROCEDURE

# APPENDIX D. WORK PACKAGE PROCEDURE



5.2 **Thermal Standard Check in Passive Mode**

**NOTE 1:** *In the passive mode, calibration consists of determining the calorimeter sensitivity (S), which is the conversion factor between the differential voltage or resistance output of the sensor system and the thermal power of the item being measured.*

**NOTE 2:** *Each measurement shall be preceded and succeeded by a "zero check" of the calorimeter. Only one "zero check" is necessary to be performed in between two successive batch measurements if the measurement sequences are performed without delay.*

**NOTE 3:** *When using electrical heat standard, the calorimeter can shall be removed from the calorimeter between each measurement, baseline, or standard. If possible, this removal is necessary to simulate (as closely as possible) real calorimeter operating conditions.*

5.2.1 Select a calibrated electrical standard with power settings that span the expected power range of items to be measured.
(A minimum of three different standard powers must be used.)

5.2.2 Ensure calorimeter heat release chamber is empty,
**AND** ensure lid and Dewar Cover are in place over top of chamber.

**NOTE:** *There should be NO heat source in the calorimeter can.*

5.2.3 Ensure chart recorder is turned ON,
**AND** the recorder is recording data.

5.2.4 Turn ON recirculating water bath system,
**AND** place controller temperature to desired setpoint.

5.2.5 **WHEN** the calorimeter has equilibrated as indicated by level chart trace (typically >1 hour from Step **5.2.4** setup),
**THEN** record the data on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO* as the "zero check."

5.2.6 Continue to run the calorimeter for at least 1-hour after Level Trace Verified (minimum of 2 hours after covers are in place per Step **5.2.4**),
**THEN** record data on the Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO* at the end of the 1-hour level trace period.

5.2.7 **AFTER** ensuring that the calorimeter had equilibrated,
**THEN** take calorimeter readings every minute for five minutes,
**AND** record on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO* to complete the "zero check."



**Figure D-1. Page 9 of NNFD-7920-OP-264 [3] that includes the draft procedure for checking the calibration of the calorimeter.**

5.2 **Thermal Standard Check in Passive Mode** (cont.)

**NOTE 1:** *The equation $W = f(\Delta V)$ relates sensor output to thermal power in Watts, where $\Delta V$ is the measured voltage difference and $f$ is an algorithm function chosen to the calorimeter design and historically has the form of a quadratic function.*

**NOTE 2:** *The relative standard deviation of an individual base power measurement should typically be less than 0.1%.*

5.2.8 Remove Dewar Cover and the lid from the heat release chamber, **AND** place the appropriate standard to be measured into the heat release chamber of the calorimeter.

5.2.9 Place the lid over the heat release chamber, **AND** place the Dewar Cover to its proper closed position.

5.2.10 **WHEN** the calorimeter has equilibrated as indicated by level chart trace (typically >1 hour from Step **5.2.9** setup), **THEN** record all relevant data on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO*.

5.2.11 Continue to run the calorimeter for at least 1-hour after Level Trace Verified per Step **5.2.10**, **THEN** record the data on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO* at the end of the 1-hour level trace period.

5.2.12 **AFTER** ensuring that the calorimeter has equilibrated, **THEN** take calorimeter readings on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO* every minute for five minutes (6 readings total).

5.2.13 Record the time the standard readings were completed on Appendix D, *Data Sheet for Calorimetric Determination of Watts Per FSO*.

5.2.14 Remove Dewar cover and lid from the heat release chamber, **THEN** remove the standard that was measured, **AND** ensure the calorimeter is empty.

5.2.15 Place the lid over the heat release chamber, **AND** place Dewar Cover to its proper closed position.

5.2.16 Repeat Steps **5.2.2** through **5.2.7** to perform “zero check.”

5.2.17 Perform a minimum of three replicate measurements at each power level using Steps **5.2.8** through **5.2.13**.

5.2.18 **IF** NO further calorimetry is to be performed at this time, **THEN** turn OFF the recirculating water bath system



**Figure D-2. Page 10 of NNFD-7920-OP-264 [3] that includes the draft procedure for checking the calibration of the calorimeter.**

**NONREACTOR NUCLEAR FACILITIES DIVISION**
Calorimetry for Pu238

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**APPENDIX D**
**Data Sheet for Calorimetric Determination of Watts Per FSO**

| Date | Container ID or "zero" | Level Trace Verified | | | 1-hr Level Trace End | | | Recorder Calorimeter Measurement | | | | | | Initials |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Time | Calorimeter mV | Water Bath Temp °C | Time | Calorimeter mV | Water Bath Temp °C | Calorimeter mV @ time interval, min. | | | | | Time Complete | |
| | | | | | | | | 1 | 2 | 3 | 4 | 5 | | |
| | | | | | | | | | | | | | | |
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**Figure D-3. Page 17 of NNFD-7920-OP-264 [3] that includes the data sheet for recording the wattages and voltages.**