# ELECTRICAL AND HEAT STANDARD CALIBRATION OF CALORIMETERS FOR PLUTONIUM AND TRITIUM MEASUREMENT

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Abstract

Recent improvements in measurement precision and reductions in measurement times have made calorimetry attractive as an alternative to some Destructive Assay (DA) for plutonium measurement. An important aspect of measurement validation is the use of an external electric calibration sample in place of Pu238 heat standards. The sample power may be supplied either from an external calibrated power source or the calorimeter internal electrical calibration power supply and measurement circuitry may be employed. The difficulties in certifying and transporting Pu238 heat standards provide an incentive to use electrical calibration methods. This paper illustrates the use of electrical calibration and presents the results of calibration measurements using both Pu238 heat standards and a variable power external electrical calibration sample. The errors associated with each method are considered and the advantages of traceable electrical calibrations are discussed.

## 1. Introduction

The advantages of calorimetry as a non-destructive technique for the assay of plutonium and tritium bearing materials are well established. The insensitivity of calorimetry to sample inhomogeneity, neutron multiplication effects and the presence of moisture makes the technique more reliable than passive neutron coincidence counting over the range of possible sample materials. In the case of tritium measurements, the insensitivity of the technique to the presence of impurities and the non-intrusive nature of the measurement are a considerable advantage.

The present paper describes and illustrates the use of the different calibration methods. The results of calibration measurements using both Pu238 heat standards and a variable power external electrical calibration sample are presented. The errors associated with each method are considered and the advantages of traceable electrical calibrations are discussed.

## 2. Calorimeter Operation

A calorimeter measures the thermal energy or heat evolved from the radioactive decay by alpha particle emission for plutonium isotopes and americium and by beta decay for tritium. In order to obtain plutonium mass from the heat measurement it is necessary to obtain measured plutonium and americium isotopic ratio data and combine these data with well established plutonium and americium specific power data. With the reduction in calorimeter measurement times to between two and four hours and recent improvements in the measurement of isotopic ratio data by high resolution gamma-ray spectrometry, the potential applicability of calorimetry for both nuclear safeguards and materials assay of plutonium has increased significantly.

Even shorter measurement times are now possible for calorimeter measurements of tritium. In general, tritium samples tend to have a more concentrated distribution of thermal energy generation and no isotopic data is required.

## 3. Electrical and Heat Standard Calibration

The sample thermal power is determined by measuring the change in the electrical power applied to the measurement chamber once a sample has been inserted. Precise measurements of the difference in the applied electrical power are obtained through measurements of electrical current and voltage and these are based on electrical calibration of the measuring circuitry. As the sample power is determined as a difference measurement the overall calorimeter thermal power calibration is achieved using either calibration Pu238 heat standards or an electrical calibration sample supplied with electrical power.

The power to the electric calibration sample is supplied by a precision power supply. The applied voltage and current are measured by electronic components for which the calibration is traceable to national standards of voltage and current. This traceability of electrical measurements is equivalent to the certification of the Pu238 standards.

Precise electrical calibration over a range of sample powers provides a means by which the performance of the calorimeter may be regularly verified as well as the calibration established. Electrical measurements of voltage and current are normally based on a NAMAS (UK National Measurement Accreditation Service) certified calibration which is traceable to standards at the National Physical Laboratory in the United Kingdom and at NIST in the United States.

The calibration external electric sample may be connected to a calibrated external electrical power supply or to the calorimeter internal electrical calibration power supply and measurement circuitry. With corrections for heating in the connecting wires this external sample provides a very accurate method of calorimeter calibration and eliminates the need for the use of Pu238 heat standards. If independence of calibration is required, for example in international safeguards, an inspector may use the electric sample with an external calibrated power supply.

## 4. Measurement Results

The results of the measurements are tabulated in Table 1 and Table 2. In Table 1 the electrical calibration data is presented. Measurements at a range of applied electrical calibration powers are presented and the results for both prediction and final equilibrium values are tabulated. In the final column the error in milliwatts between the applied calibration power and the final equilibrium power is displayed.

TABLE 1 Electrical Calibration Results

| Date | Run No. | Base Power (W) | Declared Power (W) | Prediction Time (min) | Predicted Power (W) | Final Equilibrium Time (min) | Final Equlibrium Power (W) | Error (mW) |
|---|---|---|---|---|---|---|---|---|
| 6 Mar 95 | 95006 | 12.765 | 1.5 | 101 | 1.5012 | 897 | 1.5037 | +3.7 |
| 7 Mar 95 | 95008 | 12.756 | 1.5 | 101 | 1.4922 | 1087 | 1.4971 | -2.9 |
| 24 Mar 95 | 95033 | 12.762 | 0.13655 | 93 | 0.1353 | 481 | 0.1371 | +0.5 |
| 4 Apr 95 | 95046 | 12.758 | 0.13655 | 93 | 0.1357 | 1628 | 0.1352 | -1.3 |
| 30 Apr 95 | 95052 | 12.775 | 0.13655 | 47 | 0.1306 | 2691 | 0.1383 | +1.8 |
| 17 Mar 95 | 95023 | 12.762 | 0.0 | 85 | 0.001 | 642 | 0.0018 | +1.8 |
| 28 Mar 95 | 95038 | 12.762 | 0.0 | 91 | -0.002 | 332 | -0.0005 | -0.5 |
| 29 Mar 95 | 95040 | 12.758 | 0.0 | - | - | 2606 | -0.0035 | -3.5 |
| 4 May 95 | 95056 | 12.775 | 0.40964 | 48 | 0.3987 | 271 | 0.4089 | -0.7 |
| 5 May 95 | 95058 | 12.775 | 0.274 | 47 | 0.2654 | 322 | 0.2736 | -0.4 |

**Table 2 Heat Standard Calibration Results**

| Date | Run No. | Base Power (W) | PERLA Sample No. | Declared Power (W) | Prediction Time (mm) | Predicted Power (W) | Final Equlibrium Time (min) | Final Equlibrium Power (W) | Error (mW) |
|---|---|---|---|---|---|---|---|---|---|
| 9 Mar 95 | 95010 | 12.752 | 10 | 0.1365 | - | - | 379 | 0.1365 | -0.05 |
| 10 Mar 95 | 95012 | 12.756 | 10 | 0.1365 | 74 | 0.131 | 179 | 0.1362 | -0.3 |
| 13 Mar 95 | 95014 | 12.764 | 10 | 0.1365 | 90 | 0.1343 | 191 | 0.1369 | +0.4 |
| 14 Mar 95 | 95017 | 12.765 | 9/10 | 0.2739 | 95 | 0.2753 | 329 | 0.2758 | +1.9 |
| 15 Mar 95 | 95019 | 12.763 | 9/10 | 0.2739 | 89 | 0.2748 | 433 | 0.2763 | +2.4 |
| 16 Mar 95 | 95038 | 12.762 | 9/10 | 0.2739 | 80 | 0.2741 | 433 | 0.2755 | +1.6 |
| 22 Mar 95 | 95029 | 12.759 | 9/10 | 0.2739 | 94 | 0.2737 | 424 | 0.2752 | +1.3 |
| 23 Mar 95 | 95031 | 12.759 | 8/9/10 | 0.4100 | 75 | 0.4092 | 376 | 0.4095 | -0.5 |
| 27 Mar 95 | 95036 | 12.756 | 8/9/10 | 0.4100 | 92 | 0.4083 | 400 | 0.4094 | -0.6 |
| 3 May 95 | 95054 | 12.775 | 8/9/10 | 0.4100 | 88 | 0.4079 | 393 | 0.4117 | +1.7 |

In Table 2 the results for the heat standard calibration measurements are presented. Heat standards employed in these measurements are PERLA plutonium 238 heat standards. The declared values are based on the decay correction of chemical analysis results. Again both prediction results and the final equilibrium results are displayed. The errors in the final column are the difference between the declared value and the final equilibrium value of the measured power.

## 5. Conclusions

On the whole the results of the electrical and heat standard measurements compare favourably. There appears to be no overall difference in the performance of the instrument calibrated by either method. In general, the final equilibrium times for the electrical measurements are rather longer than the final equilibrium times for the heat standard measurements. This may have resulted in a slightly larger bias as a result of drift in the calorimeter base power.

Although it is not statistically significant there does appear to be a consistent heat bias in some of the heat standard results, in particular the results involving samples 9 and 10. This bias may be a measure of a small systematic error in the declared value of the heat standard. Overall there is no statistically significant difference between the results of the electrical and heat standard calibrations.

Electrical calibrations, when performed correctly to minimise systematic errors, can be seen as a useful and convenient alternative to the use of plutonium 238 standards.

## 6. References

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