



Standard Test Method for Nondestructive Assay of Plutonium, Tritium and ²⁴¹Am by Calorimetric Assay¹

This standard is issued under the fixed designation C1458; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Section 6.2 was corrected editorially in March 2009.

1. Scope

1.1 This test method describes the nondestructive assay (NDA) of plutonium, tritium, and ²⁴¹Am using heat flow calorimetry. For plutonium the typical range of applicability corresponds to ~1 g to ~2000 g quantities while for tritium the typical range extends from ~0.001 g to ~10 g. This test method can be applied to materials in a wide range of container sizes up to 50 L. It has been used routinely to assay items whose thermal power ranges from 0.001 W to 135 W.

1.2 This test method requires knowledge of the relative abundances of the plutonium isotopes and the ²⁴¹Am/Pu mass ratio to determine the total plutonium mass.

1.3 This test method provides a direct measure of tritium content.

1.4 This test method provides a measure of ²⁴¹Am either as a single isotope or mixed with plutonium.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

C697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.10 on Non Destructive Assay.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Dioxide Powders and Pellets

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

C1592 Guide for Nondestructive Assay Measurements

C1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 ANSI Standards:³

ANSI N15.22 Plutonium—Bearing Solids—Calibration Techniques for Calorimetric Assay

ANSI N15.54 Radiometric Calorimeters—Measurement Control Program

3. Terminology

3.1 Definitions:

3.1.1 Terms shall be defined in accordance with C26.10 Terminology **C1673** except for the following:

3.1.2 *baseline, n*—the calorimeter output signal with no heat-generating item in the calorimeter item chamber.

3.1.3 *basepower, n*—a constant thermal power applied in a calorimeter through an electrical resistance heater with no heat-generating item in the item chamber.

3.1.4 *equilibrium, n*—the point at which the temperature of the calorimeter measurement cell and the item being measured stops changing.

3.1.5 *heat distribution error, n*—the bias arising from the location of the heat source within the calorimeter chamber.

3.1.6 *passive mode, n*—a mode of calorimeter operation where no external power is applied to the calorimeter except in the case of Wheatstone bridge temperature sensors where electrical current is needed to excite the bridge circuit.

3.1.7 *sensitivity, n*—the change in calorimeter response per Watt of thermal power (usually in units of micro Volts per Watt) for a heat flow calorimeter.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3.1.8 *servo control mode, n*—a mode of calorimeter operation where a constant applied thermal power is maintained in a calorimeter measurement chamber through the use of an electric resistance heater in a closed loop control system.

3.1.9 *specific power, n*—the rate of energy emission by ionizing radiation per unit mass of a radionuclide, such as ²⁴¹Am or tritium.

3.1.10 *thermal diffusivity, n*—the ratio of thermal conductivity to the heat capacity. It measures the ability of a material to conduct thermal energy relative to its ability to store thermal energy.

3.1.11 *thermal power, n*—the rate at which heat is generated in a radioactively decaying item.

3.1.12 *thermal resistance, n*—ratio of the temperature difference at two different surfaces to the heat flux through the surfaces at equilibrium.

3.1.13 *thermal time constant, n*—an exponential decay constant describing the rate at which a temperature approaches a constant value. An item container combination will have numerous thermal time constants.

3.1.14 *thermel, n*—the THERMaL ELement of the calorimeter, including the item chamber, and temperature sensor.

4. Summary of Test Method

4.1 The item is placed in the calorimeter measurement chamber and the heat flow at equilibrium, that is, the thermal power, from the item is determined by temperature sensors and associated electronic equipment.

4.2 The thermal power emitted by a test item is directly related to the quantity of radioactive material in it. The power generated by ionizing radiation absorbed in the item is measured by the calorimeter.

4.3 The mass (m) of Pu, tritium, or ²⁴¹Am is calculated from the measured thermal power of an item (W_i) using the following relationship:

$$m = \frac{W_i}{P_{eff}} \quad (1)$$

where:

P_{eff} = the effective specific power calculated from the isotopic composition of the item (see 11.3.2 for details of the calculation of P_{eff} for plutonium).

4.3.1 When tritium is the only heat source the measured thermal power can be directly converted into mass using the specific power of tritium, $P_{eff} = (0.3240 \pm 0.00045)$ (SD) W/g (1).⁴

4.3.2 For ²⁴¹Am as a single isotope the measured thermal power can be directly converted into mass using the specific power of ²⁴¹Am, $P_{eff} = (0.1142 \pm 0.00042)$ (SD) W/g (see Table 1).

4.3.3 For ²⁴¹Am mixed with plutonium, the ²⁴¹Am mass, M_{Am} , is determined by

$$M_{Am} = R_{Am} M_{Pu} \quad (2)$$

where:

R_{Am} = the mass ratio of ²⁴¹Am to Pu, and

M_{Pu} = the mass of Pu.

5. Significance and Use

5.1 This test method is the most accurate NDA technique for the assay of many physical forms of Pu. Isotopic measurements by gamma-ray spectroscopy or destructive analysis techniques are part of the test method when it is applied to the assay of Pu.

5.1.1 Calorimetry has been applied to a wide variety of Pu-bearing solids including metals, alloys, oxides, fluorides, mixed Pu-U oxides, mixed oxide fuel pins, waste, and scrap, for example, ash, ash heels, salts, crucibles, and graphite scarfings) (19,20). This test method has been routinely used at U.S. and European facilities for Pu process measurements and nuclear material accountability for the last 40 years (19-26).

5.1.2 Pu-bearing materials have been measured in calorimeter containers ranging in size from about 0.025 m to about 0.60 m in diameter and from about 0.076 m to about 0.9 m in height.

5.1.3 Gamma-ray spectroscopy typically is used to determine the Pu-relative isotopic composition and ²⁴¹Am to Pu ratio (see Test Method C1030). Isotopic information from mass spectrometry and alpha counting measurements may be used (see Test Method C697).

5.2 This test method is the most accurate NDA method for the measurement of tritium. For many physical forms of tritium compounds calorimetry is the only practical measurement technique available.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

TABLE 1 Nuclear Decay Parameters for Pu Calorimetric Assay^A

Isotope	Half-Life, Years	Standard Deviation, Years		Specific Power, W/g	Standard Deviation, W/g		References
²³⁸ Pu	87.74	0.04	(0.05 %)	0.56757	0.00026	(0.05 %)	(2,3)
²³⁹ Pu	24 119	16	(0.11 %)	1.9288 × 10 ⁻³	0.0003 × 10 ⁻³	(0.02 %)	(3-5)
²⁴⁰ Pu	6564	11	(0.17 %)	7.0824 × 10 ⁻³	0.0020 × 10 ⁻³	(0.03 %)	(6-11)
²⁴¹ Pu	14.348	0.022	(0.15 %)	3.412 × 10 ⁻³	0.002 × 10 ⁻³	(0.06 %)	(12-16)
²⁴² Pu	376 300	900	(0.24 %)	0.1159 × 10 ⁻³	0.00026 × 10 ⁻³	(0.22 %)	(17)
²⁴¹ Am	433.6	1.4	(0.32 %)	0.1142	0.00042	(0.37 %)	(15,18)

^A Numbers in parentheses are % relative standard deviation (RSD).

5.3 Physical standards representative of the materials being assayed are not required for the test method.

5.3.1 This test method is largely independent of the elemental distribution of the nuclear materials in the matrix.

5.3.2 The accuracy of the method can be degraded for materials with inhomogeneous isotopic composition.

5.4 The thermal power measurement is traceable to national measurement systems through electrical standards used to directly calibrate the calorimeters or to calibrate secondary ²³⁸Pu heat standards.

5.5 Heat-flow calorimetry has been used to prepare secondary standards for neutron and gamma-ray assay systems (24-29).

5.6 Calorimetry measurement times are typically longer than other NDA techniques. Four parameters of the item and the item packaging affect measurement time. These four parameters are density, mass, thermal conductivity, and change in temperature. The measurement well of passive calorimeters will also affect measurement time because it too will need to come to the new equilibrium temperature. Calorimeters operated in servo mode maintain a constant measurement well temperature and have no effect on measurement time.

5.6.1 Calorimeter measurement times range from 20 minutes (30) for smaller, temperature-conditioned, containers up to 24 h for larger containers and items with long thermal-time constants.

5.6.2 Measurement times may be reduced by using equilibrium prediction techniques, by temperature preconditioning of the item to be measured, or operating the calorimeter using the servo-control technique.

6. Interferences

6.1 Interferences for calorimetry are those processes that would add or subtract thermal power from the power of the radionuclides being assayed. Some examples include phase changes, endothermic or exothermic chemical reactions, such as oxidation, radiolysis of liquids, and bacterial action.

6.2 Heat-generating radionuclides that are not included in the P_{eff} determination will bias the measurement results high.

7. Apparatus

7.1 Calorimeters are designed to measure different sizes and quantities of nuclear material. Different types of heat-flow calorimeter systems share the common attributes listed below.

7.1.1 *Measurement Chamber*—Heat flow calorimeters have a cylindrical measurement chamber from which all of the heat flow generated by radioactive decay is directed through temperature sensors.

7.1.1.1 An electrical heater may be built into the walls or the base of the chamber to provide measured amounts of thermal power into the calorimeter well.

7.1.1.2 Insulation or active heaters (or both) are used to shield the chamber from outside temperature variations that would influence the thermal power measurement. Typically, an insulated plug is inserted above the item container inside the calorimeter. For some calorimeter types an insulating plug is installed permanently below the measurement chamber.

7.1.2 *Calorimeter Can*—The item to be measured may be placed in a special can that is designed to be inserted and removed easily from the calorimeter. It will typically have only a small air gap to provide good thermal conductivity between the outer surface of the can and the inner surface of the measurement chamber.

7.1.3 *Temperature Sensors*—Temperature sensors consist of thermistors, thermocouples, temperature sensitive resistance wire, or thermopiles.

7.1.4 *Thermal Sink*—The temperature increases due to heat flows generated by items are measured against a reference temperature of a thermal sink. The thermal sink could be a water bath, air bath, or a solid, usually metal, maintained at a constant temperature.

7.1.5 *Electrical Components*—Sensitive, stable electronic components are required for accurate calorimeter measurements.

7.1.5.1 High precision voltmeters are required to measure the voltage changes generated from the temperature sensors. The resolution of the voltmeters should be better than one part per million of the voltage range.

7.1.5.2 Stable power supplies are necessary to provide constant current to Wheatstone bridge sensors and calorimeter heaters.

7.1.5.3 Precision resistors with certified resistances traceable to a national measurement system may be used with calibrated voltmeters to accurately determine electrical power delivered to heaters in the calorimeter chamber. If radioactive heat standards are used as part of the measurement control program the calorimeter voltmeters need not be calibrated, nor are precision resistors required.

7.1.5.4 For a calorimeter operated in the servo (power replacement) mode digital-to-analog controller units are used to supply power to an internal resistance heater to maintain a constant temperature differential across thermal resistances.

7.1.6 *Heat Standards*—Thermal power standards are required to calibrate the calorimeter and may be used as measurement control standards to check the stability of calorimeter performance (31-34).

7.1.6.1 Radioactive heat standards, typically powered by ²³⁸Pu, also may be used to calibrate calorimeters over a range of thermal powers. These standards are calibrated against electrical standards traceable to a national measurement system. The certified power is typically decay corrected to the nearest day using certified decay tables.

7.1.6.2 Removable electrical heaters may be used to calibrate calorimeters. For this type of standard the power generated by the heater must be measured with electrical equipment regularly calibrated against standards or standard methods traceable to a national measurement system. The power supplied to the electrical calibration heater may be varied over the calibration range.

7.1.7 *Wheatstone Bridge*—When temperature sensitive resistance wire is used as the sensor, it is arranged in a Wheatstone bridge configuration shown in Fig. 1.

7.1.8 *Data Acquisition System*—Calorimeter data collection is performed using computer-based data acquisition systems. The system should be able to read signal voltages or resistances

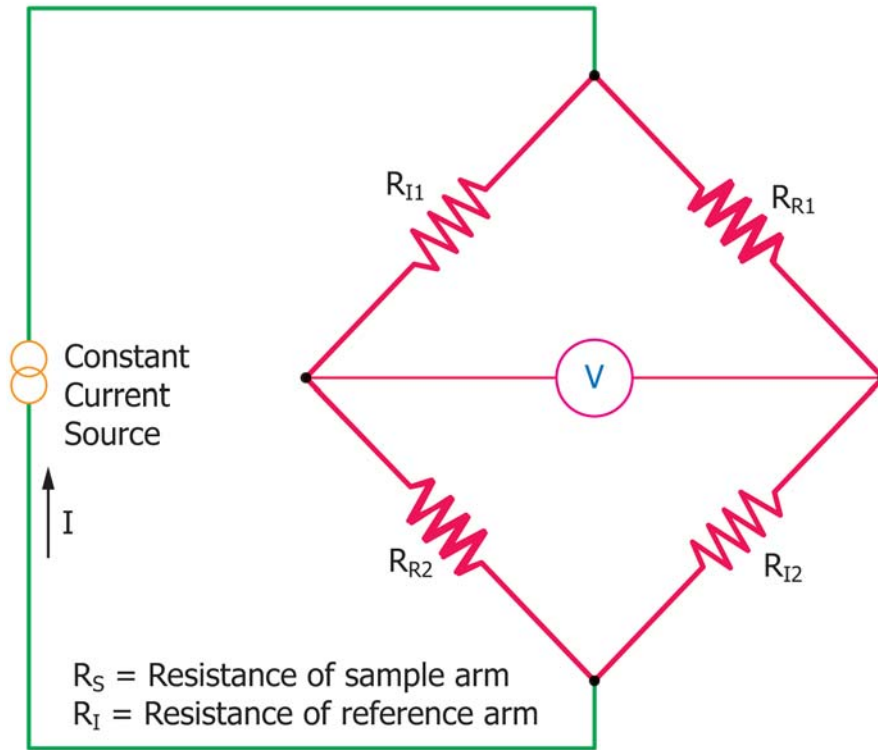


FIG. 1 Calorimeter Wheatstone Bridge Circuit

at a fixed time frequency and be able to calculate and report a power value from the item using software that detects equilibrium. Graphics and numerical data indicating system power and temperatures may be displayed to aid the operator.

7.1.9 Adapters—Low mass cylindrical metal adapters may be fabricated to accept smaller calorimeter containers in the calorimeter well, and thus, provide good thermal contact between the outer container surface and calorimeter inner wall. Heat-conducting metal foil or metal gauze fill material, typically Al or Cu, or metal shot can be used in place of machined metal adapters. Smaller items may be placed in the calorimeter container and the void space inside the container may be filled with metal fill material or shot to provide good thermal contact. Lack of fill material does not preclude measurement of the item.

7.1.10 Loading Apparatus—A hoist or assist may be used to load and unload items. Robotic loading systems may be used to handle the items.

8. Heat-Flow Calorimeter Systems

8.1 Equilibrium—A heat flow calorimeter consists of an item chamber thermally insulated from a constant temperature environment by a thermal resistance. When an item is placed in the calorimeter the temperature difference across the thermal resistance is disturbed and the difference changes with time until it converges to a constant value and equilibrium is achieved. The magnitude of the shift in the measured voltage (passive mode) or supplied power (servo mode) is used to determine the thermal power of the item in the calorimeter.

8.1.1 The curve describing the approach to equilibrium of the temperature difference is a function of several exponentials

with different time constants related to the specific heats and thermal conductivities of the item matrix material, packaging, and the calorimeter.

8.1.1.1 Equilibrium may be detected by visual inspection of the measurement data versus time or through statistical tests performed on a set of the latest data points in the time series.

8.1.1.2 Statistical prediction algorithms may be used earlier during transient temperature conditions to predict equilibrium and reduce measurement time. These typically consist of exponential functions that are used to fit the measurement data. The fitted parameters then are used to predict the final equilibrium power.

8.1.1.3 The temperature of the item to be measured may be adjusted through the use of preconditioning heaters or baths in order to decrease the time required to reach equilibrium.

8.2 Heat-flow calorimeters are operated typically in one of two modes, passive or servo controlled (power-replacement).

8.2.1 Passive Mode—In this mode of calorimeter operation a Wheatstone bridge is frequently used as the heat sensor. The only heat generated comes from the item being measured and the current required to excite the bridge inside the thermel. A graph of the calorimeter response to a heat source is shown in Fig. 2. This plot shows that after a period of time the temperature transient caused by the insertion of the item into the calorimeter disappears and the calorimeter and item are in thermal equilibrium.

8.2.1.1 The item wattage, W_i , is calculated by the following:

$$W_i = (BP_s - BP_0)/S \quad (3)$$

where:

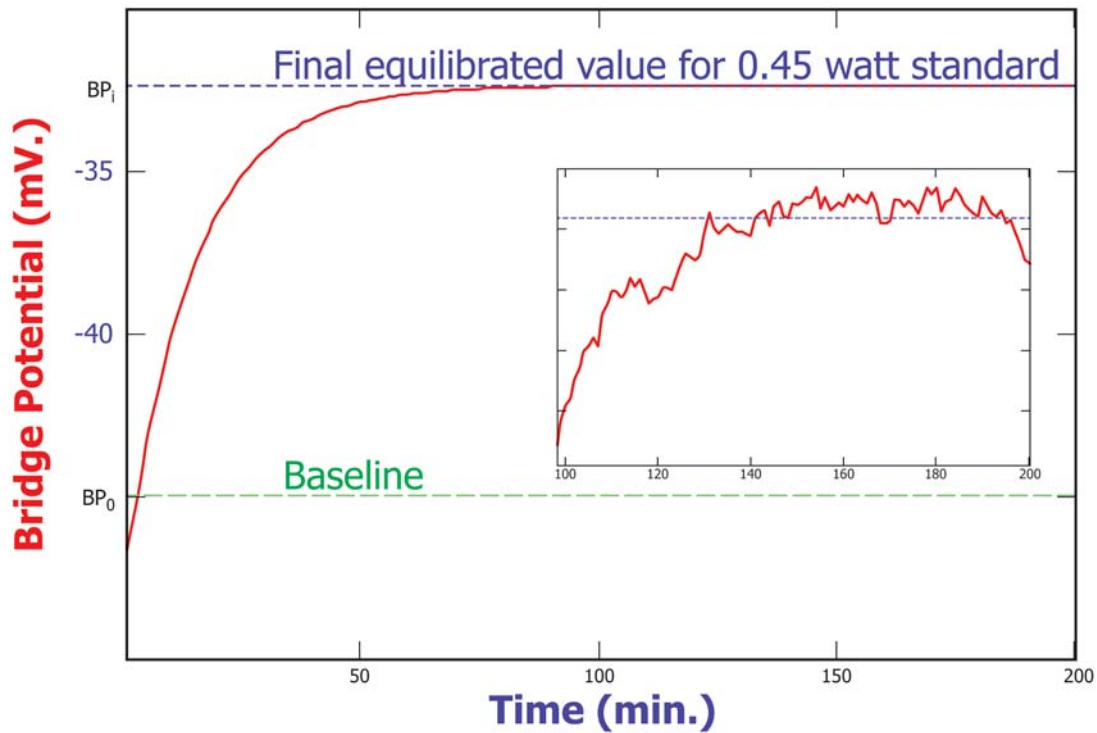


FIG. 2 Approach to Equilibrium for a Calorimeter Operated in the Passive Mode

- S = the calorimeter sensitivity (microvolts/Watt) at the power level, determined by electrical or ^{238}Pu standards,
- BP_s = the equilibrium sensor response with the item in the calorimeter, and
- BP_0 = the baseline sensor response with no item in the calorimeter.

8.2.2 Servo Mode (Power Replacement Mode)—In this mode of operation, a constant amount of thermal power is applied to the item chamber by electrical heaters. The temperature of the calorimeter item chamber is held at a constant temperature difference above the temperature of the thermal sink by means of a servo-controller. The constant power causes a constant temperature differential to be maintained across the thermal resistance separating the measurement chamber from a controlled reference temperature. The temperature differential is proportional to the signal, voltage or resistance, and is the temperature difference between a sensor (or sensors) located adjacent to the item being measured and the other(s) located at the reference temperature. A closed-loop controller monitors the output signal, and if a radioactive heat-generating item is inserted, the external power applied is decreased to precisely maintain the same signal differential. When the unknown item is placed in the calorimeter, the control power drops over time to a lower level. The power of the unknown is the difference between the two control power readings at equilibrium. A graph of the calorimeter response is shown in Fig. 3.

8.2.2.1 The item wattage, W_i , is calculated by the following:

$$W_i = W_0 - W_H \quad (4)$$

where:

- W_0 = the basepower with no item in the calorimeter, and
- W_H = the power supplied to the calorimeter with the item in the calorimeter.

8.2.2.2 The measurement time for the servo mode of operation is normally shorter than for the passive mode because the calorimeter components are at the equilibrium temperature and the servo-controlled internal heater can supply heat actively to bring the item to equilibrium.

9. Hazards

9.1 Safety Hazards:

9.1.1 It is recommended that a criticality evaluation be carried out if fissile material is to be measured.

9.1.2 Precautions should be taken to minimize electrical shock hazards.

9.1.3 Precautions should be taken to avoid contamination with radioactive materials.

9.1.4 Precautions should be taken to minimize personnel radiation exposure to ionizing radiation.

9.1.5 Pinch-point and lifting hazards may be present during the loading and unloading of heavy items with calorimeters. Mechanical aids, such as a hoist, should be used for movement of heavy items.

9.1.6 A burn hazard can exist for high-power items. Caution should be taken to avoid burns.

9.2 Technical Hazards:

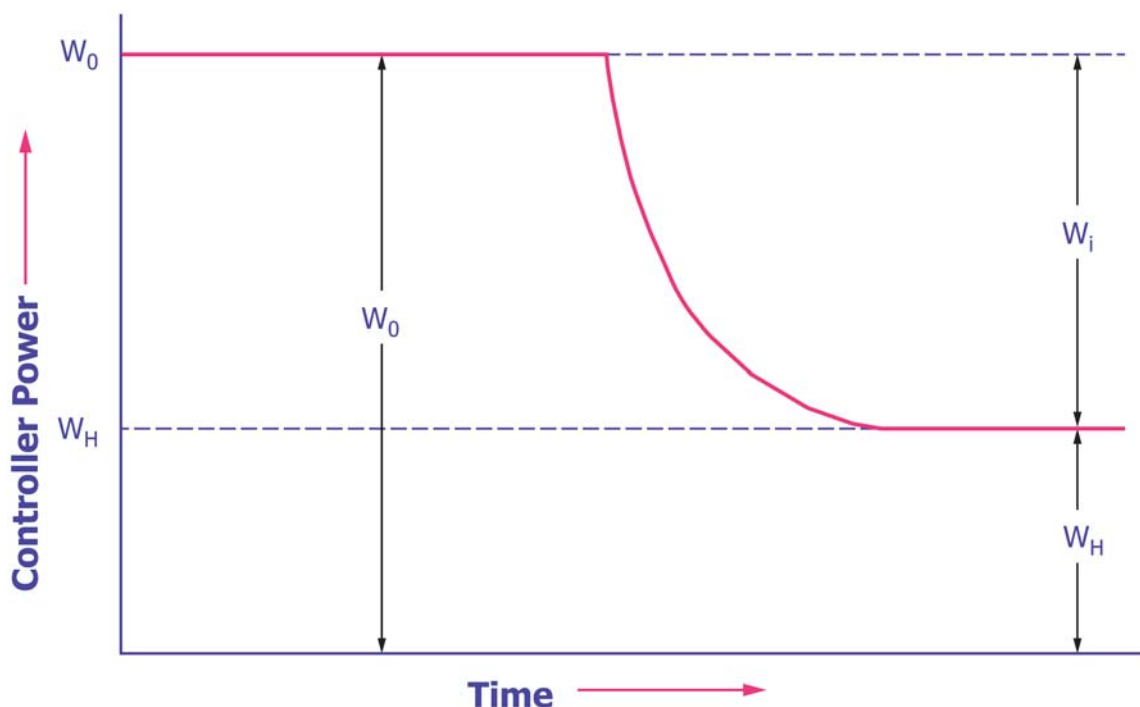


FIG. 3 Approach to Equilibrium for a Calorimeter Operated in the Servo (Power Replacement) Mode

9.2.1 Room temperature variations may affect the stability of the reference temperature and increase measurement uncertainty.

9.2.2 Using a measurement result outside of the range of the calibration is not recommended.

9.2.3 Care should be taken in the insertion or removal of the calorimeter can so that it is not jammed in the calorimeter well.

9.2.4 Noise in the electronics AC supply power generated by machinery may increase the measurement uncertainty.

9.2.5 The base power for servo-operated calorimeters must not be less than the highest power expected from items.

9.2.6 Mechanical stress on the item chamber from the weight of the item may cause a bias in the final result.

9.2.7 The calorimeter may exhibit a small heat distribution error dependent on calorimeter design and item characteristics.

9.2.8 The following conditions could extend measurement time.

9.2.8.1 Large masses of material.

9.2.8.2 Items that make poor thermal contact with their containers.

9.2.8.3 Items that contain a large amount of insulating material or dead air spaces caused by several layers of containment.

9.2.9 Errors can result from incorrect algorithms used for equilibrium detection or prediction. The algorithm used for equilibrium prediction that is suitable for a ^{238}Pu heat standard or electrical standard in a conductive matrix may not be suitable for items with Pu in a matrix with poor thermal conductivity.

9.2.10 Bias in the determination of P_{eff} will lead to a bias in the assay result.

9.2.11 The item introduced into the calorimeter should not generate enough heat such that the temperature inside the calorimeter would exceed safe limits.

9.2.12 For removable electrical heat standards the heater leads to the standard will serve as a heat path for heat to exchange between the measurement chamber and the environment; this may bias the calibration.

9.2.13 Improperly closing the calorimeter (for example, not completely inserting the insulating baffle) may bias the measurement results, degrade precision, or both.

9.2.14 Cables or any other conductor providing a thermal conduction path into and out of the calorimeter. Variability in these paths is a more severe hazard.

10. Calibration Procedure

10.1 The type of calibration procedure depends on whether the calorimeter is operated in the passive or servo mode. In the passive mode calibration consists of determining the calorimeter sensitivity, S , the conversion factor between the differential voltage or resistance output of the sensor system and the thermal power of the item being measured. In the servo mode calibration is setting the sensor output setpoint voltage that corresponds to a specific base power.

10.2 Calibration—Passive Mode:

10.2.1 Select a series of ^{238}Pu heat standards or calibrated electrical standard power settings that span the expected power range of items to be measured. A minimum of three different standard powers must be used.

10.2.2 Initiate a baseline measurement of the bridge potential (voltage) with a calorimeter can filled with conductive material in the calorimeter chamber. There should be no heat

source in the calorimeter can. Record the baseline, BP_0 (1), after equilibrium is reached.

10.2.3 Remove the calorimeter can and place the ^{238}Pu or electrical standard in the can. Center the standard in the can.

10.2.3.1 Whether using radioactive or electrical heat standards, the calorimeter can must be removed from the calorimeter between each measurement, baseline, or standard. This removal is necessary even when using electrical standards to simulate as closely as possible real calorimeter operating conditions.

10.2.4 Close the calorimeter can and place it in the calorimeter well. For all measurements, the calorimeter should be properly closed before a measurement is commenced.

10.2.5 Initiate the calorimeter run. Record the bridge potential voltage output (BP_{std}) after equilibrium has been achieved.

10.2.6 Remove the calorimeter can from the calorimeter, then remove the heat standard from the can and place the can back in the calorimeter.

10.2.7 Repeat another baseline run. Record the baseline BP_0 (19) after equilibrium is reached.

10.2.8 Calculate the average baseline, $BP_{0(av)} = [BP_0 (1) + BP_0 (19)]/2$.

10.2.9 Use the known power output of the heat standard, W_{std} , the calorimeter sensor value, BP_{std} , and the calculated average baseline, $BP_{0(av)}$ to calculate the calorimeter sensitivity, S .

$$S = \frac{BP_{std} - BP_{0(av)}}{W_{std}} \quad (5)$$

10.2.9.1 The sensitivity S is not usually a constant, but varies slightly with the wattage of the standard, W_{std} , over the measurement range of the calorimeter. For one calorimeter, with an air gap thermal resistance, a standard measurement at 1 W yielded a sensitivity of 28 950 $\mu\text{V}/\text{W}$ while for another standard measurement at 11 W, it was 28 450 $\mu\text{V}/\text{W}$, a decrease in sensitivity of 1.7 %. The decreased sensitivity is qualitatively consistent with increased thermal conductivity of material forming the primary thermal resistance. For example the conductivity of dry air, a common thermal gap material, increases at the rate of 0.3 %/°C. The change in sensitivity with power is described by:

$$S = S_0 + k \times W_{std} \quad (6)$$

where:

S_0 = the mathematically determined sensitivity for zero power, and

k = the slope of the varying sensitivity.

10.2.10 Perform a minimum of three replicate measurements at each power level using steps 10.2.1 – 10.2.8. This sensitivity determination at different power levels typically need only be done once after a calorimeter is installed.

10.2.11 Combine Eq 5 and Eq 6 and rearrange for net sensor output, $BP_{std} - BP_{0(av)}$, to obtain the equation to relate sensor output to watts:

$$(BP_{std} - BP_{0(av)}) = S_0 \times W_{std} + k \times W_{std}^2 \quad (7)$$

The fit parameters S_0 and k are determined by a least-squares fit to a plot of net sensor output as a function of standard watts. Since calorimeter noise is a function of the power of

the item being measured, increasing variability with increasing power, the residuals of the least-squares fit will not be normally distributed. However the parameter estimates are not affected by this abnormal distribution.

10.3 Calibration–Servo Mode:

10.3.1 Select an approximate basepower, W , at which the servo system will operate. It should be 10–20 % higher than the highest expected item power.

10.3.2 Determine the sensitivity S of the calorimeter by performing steps 10.2.2 – 10.2.9 once for one ^{238}Pu or electrical standard power. Any power within the range of item powers is satisfactory.

10.3.3 Calculate the setpoint bridge potential (BP_{sp}) as follows:

$$BP_{sp} = BP_{0(av)} + S \times W \quad (8)$$

10.3.4 Set the controller to adjust heater power from the external power source so that BP_{sp} is maintained.

10.3.4.1 The actual supplied heater power (W_0) used to maintain BP_{sp} may be slightly different than the target value, W , because of the uncertainty in S used to calculate the setpoint.

10.3.5 Place a calorimeter can that is filled with conductive material but with no heat generating item in the calorimeter.

10.3.6 Close the calorimeter and initiate the measurement run. Once equilibrium is reached take a heater power measurement. This is the basepower W_0 corresponding to BP_{sp} .

10.3.7 Repeat the basepower measurement with no item in the item chamber at least three times. This involves removing the calorimeter can between each of the basepower measurements at least three times and reinserting it in the calorimeter. The standard deviation of an individual basepower measurement should typically be less than 0.1 %.

11. Assay Procedure

11.1 *Item Measurement*—Determine the baseline bridge potential BP_0 or basepower W_0 for the calorimeter. This should be done using step 10.2.2 for the passive mode or steps 10.3.5 and 10.3.6 for the servo mode prior to the item measurement. If BP_0 or W_0 is stable the baseline or basepower measurement need not be done prior to every item measurement. Time between zero power measurements are set based on calorimeter stability, required accuracy, and administrative requirements.

11.1.1 Load the item to be assayed into the calorimeter can, and close the can.

11.1.2 Load the can into the calorimeter measurement chamber.

11.1.3 Close the calorimeter. Be sure the baffle top is completely down.

11.1.4 Initiate the calorimeter run.

11.1.5 When thermal equilibrium has been established or predicted the run is terminated.

11.1.6 An additional baseline (passive) or basepower (servo) run may optionally be taken after the item measurement-using step 10.2.2 or steps 10.3.5 and 10.3.6. The average of the pre- and post-item measurement baselines or basepowers may be used for BP_0 or average base power W_0 .

11.2 Item Thermal Power Calculation—The calculation of the item thermal power is performed differently for passive compared to servo operation.

11.2.1 Power Calculation—Passive Mode:

11.2.1.1 Calculate the item wattage by solving the quadratic equation for W_i as follows:

$$W_i = (BP_i - BP_0)/(S_0 + k W_i) \quad (9)$$

using the bridge potential voltage BP_i for the item, the baseline BP_0 , and the fitted parameters S_0 and k , from step 10.2.11.

11.2.1.2 The solution to the quadratic equation when $S_0 > 0$, and $k < 0$ is as follows (35) :

$$W_i = \frac{-S_0}{2 \times k} - \left[\left(\frac{-S_0}{2 \times k} \right)^2 - \left(\frac{BP_0 - BP_i}{k} \right) \right]^{1/2} \quad (10)$$

and when $S_0 > 0$ and $k > 0$ then W_i is:

$$W_i = \frac{-S_0}{2 \times k} + \left[\left(\frac{-S_0}{2 \times k} \right)^2 - \left(\frac{BP_0 - BP_i}{k} \right) \right]^{1/2} \quad (11)$$

11.2.1.3 Eq 10 and Eq 11 are specialized rearrangements of the more familiar form of the quadratic equation:

$$x = \frac{-b \pm \sqrt{b^2 - 4 \times a \times c}}{2 \times a} \quad (12)$$

This solution is necessary due to the rounding errors that occur using Eq 12 when the product of a and c is much smaller than b squared.

11.2.2 Power Calculation—Servo Mode:

11.2.2.1 Calculate the item wattage, W_i as follows:

$$W_i = W_O - W_H \quad (13)$$

where:

W_O = control power with no item in the calorimeter, and

W_H = control power with an item in the calorimeter.

11.2.3 If necessary, a bias correction may be made to a calorimeter measurement based on standard measurements made under a measurement control program. It is unusual that a calorimeter require a bias correction. Proper operation of the calorimeter hardware and software should be definitively verified before resorting to bias corrections.

11.2.4 Once the thermal power is determined the quantity of radioactive material in the container is calculated using the equations in 4.3. For Pu, a separate isotopic analysis is required to determine P_{eff} .

11.3 Calorimetric Assay—Plutonium:

11.3.1 The mass of plutonium in an item can be calculated from the thermal power measured by calorimetry in units of watts and from the item effective specific power in units of watts per gram as shown in Eq 14:

$$m = \frac{W_i}{P_{eff}} \quad (14)$$

11.3.2 The effective specific power of the material in the item is calculated in Eq 15:

$$P_{eff} = \sum_i R_i P_i \quad (15)$$

where:

R_i = abundance of the i -th isotope ($i = {}^{238}\text{Pu}, {}^{239}\text{Pu}, {}^{240}\text{Pu}, {}^{241}\text{Pu}, {}^{242}\text{Pu}$, and ${}^{241}\text{Am}$) of the Pu in the item expressed as a weight fraction, g isotope/g-Pu, and

P_i = a physical constant, the specific power of the i -th isotope in the item in W/g.

11.3.2.1 The isotopic fractions, R_i , can be determined destructively by mass spectrometry and alpha spectrometry, or nondestructively by gamma-ray spectrometry. Liquid scintillation counting may be used as well as alpha spectrometry for determination of the ${}^{241}\text{Am}$ abundance by means of destructive analysis. The terms $R_i P_i$ are the contribution of each isotope to the total item power. The isotopic fractions R_i are not constant, but change as the constituent isotopes in the item decay or grow in.

11.3.2.2 The isotopic specific powers, P_i , for some Pu isotopes have been directly measured or can be derived from Eq 16. The specific powers and half-lives for Pu isotopes and ${}^{241}\text{Am}$ as listed in ANSI N15.22–1987 are listed in Table 1. The half-lives were established after a data review of collaborative and individual experiments by the USDOE Half-Life Evaluation Committee. The half-lives and specific powers of ${}^{239}\text{Pu}$ and ${}^{240}\text{Pu}$ were determined from collaborative experiments on nearly pure isotopic samples. The specific powers of ${}^{241}\text{Pu}$ and ${}^{242}\text{Pu}$ were measured by individual experimenters and the specific power of ${}^{238}\text{Pu}$ and ${}^{241}\text{Am}$ were determined using Eq 16:

$$P_i = \frac{2119.3}{T_{1/2(i)} A_i} Q_i \quad (16)$$

where:

Q_i = total disintegration energy (MeV) for alpha-particle emitters, or the average energy (MeV) of beta particle emitters,

$T_{1/2(i)}$ = half-life (years) of isotope i , and

A_i = gram atomic weight of isotope i .

12. Measurement Control Procedure

12.1 Measurement control procedures are similar for all calorimeter measurements.

12.1.1 Replicate measurements of ${}^{238}\text{Pu}$ or electrical heat standards are performed to demonstrate that the calorimeter system, hardware and software, is operating correctly. Other well-characterized nuclear materials may be used as heat standards.

12.1.2 Corrections to the thermal power of radioactive heat standards due to radioactive decay should be made.

12.1.3 Control charts formed from replicate measurements of heat standards can be beneficial in providing quantitative means for determining that the calorimeter system is operating satisfactory prior to a single assay measurement or group of assay measurements. In addition these charts can be used to demonstrate that the calorimeter was in control during the assay runs.

12.1.3.1 Control charts may also be used to monitor baseline or basepower measurements. This can provide auxiliary information in case out-of-control operating conditions are detected.

12.1.4 Calorimeter bath temperatures can be monitored continuously to flag changes that will affect calorimeter performance. The temperatures may be evaluated using control charts or administrative limits.

12.1.5 The frequency of standards and baseline measurements are dependent on how well the calorimeter meets performance requirements and environmental conditions. Other factors to consider are throughput/day, portable or permanent instruments, and data collection systems.

12.1.5.1 More details on calorimeter measurement control may be found in ANSI N15.54–1988. More general aspects of measurement control relevant to calorimetry may be found in Guide C1009.

12.1.6 Data collected from a measurement control program can be used to calculate the precision and bias of the power measurement. A summary of the precision and bias of the power measurement obtained from replicate measurements of ²³⁸Pu heat standards in production facilities over a 0.5–1.0-year period is shown in Table 2.

13. Assay Precision and Bias

13.1 Precision:

13.1.1 Calorimetric assay of plutonium requires both calorimetry and isotopic analysis, thus, the precision and bias of the assay will have components due to uncertainties in both techniques.

13.1.2 The precision of a calorimeter measurement is dependent on the sensitivity of the calorimeter, baseline stability, and item power.

13.1.3 A list of major factors that can affect the precision of the gamma-ray isotopic assay and mass spectrometric modes of isotopic analysis are described in Test Methods C1030 and C697, respectively.

13.1.4 *Plutonium in PuO₂-UO₂ Mixed Oxide*—Generally, the greater the thermal power in a calorimeter, the better the relative precision. Absolute calorimeter random noise increases with increasing power. To illustrate this the relative precision observed from repetitive calorimeter measurements of six items containing 26 to 258 g of Pu (17 % ²⁴⁰Pu) in PuO₂-UO₂ (26 % Pu) was calculated and the results are shown in Table 3. These measurements were made over a 56-day period with a water bath twin-bridge over-under calorimeter. The items were loaded robotically allowing for continuous operation. A common P_{eff} factor for all six items was determined using the plutonium isotopic composition and ²⁴¹Am content that was determined by mass spectrometry and alpha counting. The calorimeter can size was 0.06-m diameter × 0.16-m high. The calorimeter measurement time was fixed at 1 h. The calorim-

TABLE 3 Calorimetry/Mass Spectrometry Measurements of Plutonium in PuO₂-UO₂ Mixed Oxide^{A,B}

ID	Mass ^{B,C} , g by Chem	Mass, g ^C by Cal _D /MS/α ^E	Precision, g 1 SD	Precision %RSD ^F	Bias, g	Bias, %
4	257.70	257.54	0.14	0.06	−0.16	−0.06
5	206.09	206.06	0.13	0.06	−0.03	−0.02
6	206.18	206.12	0.14	0.07	−0.06	−0.03
7	128.81	128.94	0.12	0.09	0.13	0.10
8	77.28	77.35	0.12	0.15	0.07	0.09
9	25.79	25.99	0.11	0.42	0.20	0.78

^A Unpublished results.

^B Mass of plutonium determined by coulometry using reference material NBS 949E. Pu percentage of mixed oxide, 0.25759, based on triplicate measurements of six samples.

^C Pu masses reported here decayed to a common date.

^D Final results based on 117 replicate calorimeter measurements/item.

^E Isotopic composition determined by 12 replicate measurements by mass spectrometry (²³⁹Pu, ²⁴⁰Pu, ²⁴¹Pu, ²⁴²Pu) and six replicate alpha counting (²³⁸Pu, ²⁴¹Am) measurements. Average Pu isotopic and ²⁴¹Am results were used to calculate P_{eff} used for all calorimeter measurements.

^F Precision due to calorimetry power replicate measurements.

eter was run in the servo mode and the items were preconditioned to reduce measurement time.

13.1.5 *Plutonium in PuO₂*—The results of multiple calorimetric and gamma-ray isotopic measurements by three facilities on identical standards each containing 400 g of well-characterized Pu (6 % ²⁴⁰Pu) in PuO₂ are shown in Table 4. The Pu content and isotopic composition used as reference values of the mother lot of PuO₂ material used for these standards were measured by coulometry and mass spectrometry/alpha counting by four analytical laboratories. The calorimetry and gamma-ray measurements used to determine Pu mass variabilities and biases reported in Table 4 were taken over a one-year period. The within-facility variability and the bias of the calorimetric/gamma-ray assay were calculated from results reported by each facility decayed to a common date. Each facility used different gamma-ray analysis codes for the isotopic measurements. Some of the reported values are the averages of measurements of the standard item with different calorimeters.

13.2 Bias:

13.2.1 The bias of calorimetric assay can be determined by the measurement of certified reference materials or well-characterized items with known elemental and isotopic compositions.

13.2.2 Plutonium in PuO₂-UO₂ and PuO₂ (36). Biases for calorimetric assay of Pu in PuO₂-UO₂ mixed-oxide powder and Pu in PuO₂ powder are shown in Tables 3 and 4, respectively. This data shows that the biases for this measurement application are typically less than 0.1 %.

TABLE 2 Calorimeter Power Measurement Precision and Bias

Heat Standard Power, W	Calorimeter Diameter, m	Calorimeter Type, Operation Mode	Number of Measurements	Precision, % RSD	Bias, %
98	0.06	rod, servo	29	0.065	0.02
3.5	0.15	rod, servo	55	0.09	0.00
4.0	0.25	twin, passive ^A	22	0.05	0.03
4.9	0.30	twin, passive ^A	34	0.06	0.05

^A Pooled results from two calorimeters.

TABLE 4 Calorimetry/Gamma-Ray Assay Measurement of 400 g of Pu in PuO₂^A (32)

Facility	Within-Facility Variability, g	Within-Facility Variability, %RSD	Bias, g	Bias, %
A ^B	1.5	0.38	0.03	0.01
B ^B	1.5	0.38	−0.40	−0.10
C ^C	1.4	0.36	0.04	0.01

^A All masses are in grams of plutonium decayed to a common date.

^B Used multiple water bath twin-bridge calorimeters.

^C Used “air-bath” calorimeter.

13.2.3 Plutonium in Salt Residues (37,38)—In two separate studies calorimetry and gamma-ray spectroscopy measurements were used to assay items containing a mixture of KCl, MgCl₂, and NaCl residues containing PuCl₃ and AmCl₃, as well as, shards of MgO crucibles and plutonium metal shot. The material in each item was sampled and analyzed for Pu. The average relative biases between the calorimetry assay and alternative assay is shown in Table 5. A twin bridge water bath

TABLE 5 Bias—Calorimetry/Isotopic Measurements of Pu and Am in Molten Salt Residues

Mass Range, g Pu	No. of Items	Mass Range, g Am	Bias, % Pu	Bias, % Am	References
35–416	10	0.4–18	1.5 ± 0.5 ^A	0.2 ± 0.4 ^A	16
214–414	9	5–26	1.6 ± 0.6 ^B	0.2 ± 0.6 ^B	17

^A*Chemistry*—Pu and Am elemental analysis by Isotopic Dilution Mass Spectrometry. Aliquots taken of blended salt matrix for analysis. Pieces of Pu metal removed, oxidized, and returned to matrix before blending.

Calorimetry—Isotopic analysis for P_{eff} determination by gamma-ray spectroscopy.

^B*Chemistry*—Entire matrix of each item dissolved and liquid samples measured by x-ray fluorescence for Pu and gamma counting for Am.

Calorimetry—Isotopic analysis for P_{eff} determination by gamma-ray spectroscopy and facility stream averages of certain Pu isotopic ratios.

calorimeter was used for the thermal power measurements in both studies.

13.2.4 Americium in Salt Residues (37,38)—The same measurement techniques described in section 13.2.3 were used to assay the ²⁴¹Am content of the items containing molten salt residues, as well as, the Pu content. Gamma-ray spectroscopy was used to determine the ²⁴¹Am/Pu ratio simultaneously with plutonium isotopic ratios. The biases are shown in Table 5.

13.2.5 Tritium (39)—Calorimetry was used to measure the quantity of tritium gas in containers. Since tritium was the only radioactive isotope, no isotopic measurements were required for the assays. After the calorimeter measurement the gas was quantitatively transferred to tanks with calibrated volumes, and the quantity of tritium was determined using calibrated pressure and temperature transducers and mass spectrometric analyses. A comparison of measurement results between calorimetry and pressure volume temperature (PVT) combined with mass spectrometry (MS) was made for 50 containers. The tritium content of the containers ranged from 15 to 16 g. The relative mean bias for the calorimeter assay compared to PVT combined with MS was $-0.12 \pm 0.05\%$ average relative standard deviation (RSD_{mean}). A twin bridge water bath calorimeter was used for the calorimeter measurements.

APPENDIXES

(Nonmandatory Information)

X1. PROPAGATION OF ERROR FOR PLUTONIUM AND ²⁴¹AM MASS

X1.1 In Eq 14, the measurement of the thermal power, W , is independent of the measurement of the effective specific power, P_{eff} , and thus, the relative uncertainty for the plutonium mass, M , can be written as follows:

$$\frac{\sigma_M}{M} = \left[\frac{\sigma_W^2}{W^2} + \frac{\sigma_{P_{eff}}^2}{P_{eff}^2} \right]^{1/2} \quad (X1.1)$$

X1.1.1 The uncertainty in the power measurement, σ_W , can be obtained from replicate power measurements of heat standards or from historical data. It should include both precision and bias components. The uncertainty in P_{eff} , $\sigma_{P_{eff}}$, comes from the uncertainty in the isotopic fractions, R_i , and isotopic specific powers, P_i . The uncertainties in the isotopic fractions are determined from uncertainties in the various techniques that might be used for the isotopic analysis, such as mass spectroscopy, alpha counting, or gamma-ray spectroscopy. There are sufficient gamma-rays in plutonium to provide independent measured isotopic ratios of the major contributors to the item thermal power: ²³⁸Pu, ²⁴⁰Pu, ²⁴¹Pu, ²⁴²Pu, and ²⁴¹Am with respect to ²³⁹Pu that allow R_i to be calculated. Ref (40) discusses this in more detail. The uncertainties in the isotopic specific powers, P_i , as determined by different experiments, are given in Table 1. The test method for determining isotopic composition by gamma-ray spectroscopy

is described in Test Method C1030. Several gamma-ray analysis codes are available commercially that not only provide the isotopic composition, but also the uncertainties of the isotopic fractions and the specific power of the item being measured. Error propagation of the isotopic fractions is discussed in Ref (40).

X1.1.2 The uncertainty of the ²⁴¹Am mass mixed with Pu is as follows:

$$\frac{\sigma_M}{M} = \left\{ \left[\left(\frac{\sigma_W}{W} \right)^2 + \left(\frac{K}{P_{eff}} \right)^2 \left[\left(\frac{\sigma_K}{K} \right)^2 + \left(\frac{\sigma_{R_{Am}}}{R_{Am}} \right)^2 \right] + \left(\frac{R_{Am} \sigma_{P_{Am}}}{P_{eff}} \right)^2 \right] \right\}^{1/2} \quad (X1.2)$$

where:

$$K = P_{eff} - P_{Am} R_{Am} \quad (X1.3)$$

where:

- P_{Am} = the specific power of ²⁴¹Am,
- R_{Am} = the mass ratio of ²⁴¹Am to plutonium,
- W = the thermal power,
- σ_W = the thermal power uncertainty,
- $\sigma_{R_{Am}}$ = the uncertainty in the ²⁴¹Am mass ratio, and
- $\sigma_{P_{Am}}$ = the uncertainty of the ²⁴¹Am specific power.

X2. PROPAGATION OF ERROR FOR TRITIUM MASS

X2.1 The uncertainty of a calorimetric assay of tritium can be calculated using Eq X1.1. The uncertainty of the effective specific power, P_{eff} , of tritium is the same as the isotopic specific power, 0.00045 W obtained from 4.3.1. Dividing by the specific power of tritium, 0.3240 W/g, results in the following:

$$\frac{\sigma_{P_{\text{eff}}}}{P_{\text{eff}}} = 0.0014 \quad (\text{X2.1})$$

So for tritium the relative uncertainty of the tritium mass is as follows:

$$\frac{\sigma_{\text{tritium}}}{M_{\text{tritium}}} = \left[\left(\frac{\sigma_w}{W} \right)^2 + (0.0014)^2 \right]^{1/2} \quad (\text{X2.2})$$

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