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AN EIGHT UNIT ISOTHERMAL
CALORIMETER FOR ROUTINE PLUTONIUM ANALYSES

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AN EIGHT UNIT ISOTHERMAL CALORIMETER FOR ROUTINE PLUTONIUM ANALYSES

Robert G. White and Franklin L. Oetting

ABSTRACT. This report describes the design and operation of a multi-unit isothermal calorimeter whose primary function is to analyze for plutonium. Although each individual unit is similar in design to the corresponding unit in the twin isothermal calorimeter, the overall design and operation of the multi-unit calorimeter is unique.

INTRODUCTION

The eight-unit isothermal calorimeter is patterned after the twin isothermal calorimeter which has been previously described.¹ Although the basic design and capabilities of the eight-unit calorimeter are the same as those of the twin-unit calorimeter, the capacity of the multi-unit instrument is seven times greater. The optimum capacity of twenty samples per week can be increased to twenty-eight when necessary. Because of its size and relative complexity, the eight-unit calorimeter is in some ways unique and requires additional explanation. It is designed for use in routine plutonium assays of two or three kilogram samples of plutonium metal or oxide and has been used for analysis of smaller amounts of miscellaneous radioactive samples such as americium-241 and plutonium-238. The purpose of this report is to introduce the eight-unit calorimeter by presenting a survey of its design and operation, as well as some of the theory of isothermal calorimetry.

THEORY

The function of a radiometric calorimeter as an analytical instrument is to measure the heat produced by radioactive decay. The amount of heat is related to the specific isotopes present and to the amounts of these isotopes.

For any given radioactive isotope, whether it decays by emission of alpha particles or by emission of beta particles, there is a characteristic energy associated with its decay. For alpha emitting nuclides this energy is equal to the product of the maximum energy of the alpha particles and the number of disintegrations per unit time plus the recoil energy. The maximum value for the alpha

energy is used because, although there are several energy levels to which the parent nucleus may decay, the excited daughter nucleus instantaneously undergoes a transition to the ground state emitting energy as gamma radiations. This gamma radiation energy is quite small compared to the alpha energy.

The majority of the energy associated with the gamma radiation, as well as all of the energy associated with the alpha radiation, is captured by the calorimeter. On the other hand, a beta-emitter decays by loss of both a beta particle and a neutrino. The neutrino is not captured by the calorimeter, and its energy is not measured. Since the disintegration energy is divided between the beta particle and the neutrino in varying proportions, it is necessary to use an average beta energy in calculating the energy output of a beta-emitter, multiplying the average beta energy by the number of disintegrations per unit time.

Therefore, the thermal power generated by the decay of one gram of radioactive material (specific power) may be calculated using the isotopic composition as determined by mass spectrographic analysis, the alpha or beta energies (Q values) of the isotopes present, and the half-lives of these isotopes. This specific power can be measured directly by means of calorimetry, and is conveniently expressed as watts per gram. Table I lists the values currently in use (January 1969) for the specific powers of selected isotopes.

TABLE I. Specific Power of Selected Isotopes

Isotope	Specific Power (watts/gram)
²⁴¹ Am	0.1145 ^a
²³⁸ Pu	0.569 ^b
²³⁹ Pu	0.001923 ^b
²⁴⁰ Pu	0.007097 ^c
²⁴¹ Pu	0.00362 ^d
²⁴² Pu	0.00012 ^b

a. F. L. Oetting and S. R. Gunn, *J. Inorg. Nucl. Chem.*, 29, 2659 (1967).

b. F. L. Oetting, *J. Inorg. Nucl. Chem.*, 27, 2151 (1965).

c. F. L. Oetting, "Thermodynamics of Nuclear Materials, 1967", Proceedings of the Symposium on Thermodynamics, IAEA, 1967, Vienna, Austria.

d. F. L. Oetting, *Phys. Rev.*, 168, 1398 (1968).

The method of steady-state is used with this isothermal calorimeter. The steady-state method requires a source of constant and continuous heat and an isothermal jacket

¹ F. L. Oetting, *J. Inorg. Nucl. Chem.* 27:2151 (1965).

around the calorimeter. When the heat source is placed into the calorimeter, it begins to heat the calorimeter body. The heat is conducted horizontally through the calorimeter into the isothermal environment where it is absorbed. Eventually the conditions in the system are such that the rate at which heat is generated by the source is equal to the rate at which the isothermal bath absorbs the heat.

During steady-state the heat flux may be measured. This approach is well-suited for isothermal calorimetry since a radioactive heat source provides a constant heat output which can be neither initiated nor terminated. For this particular calorimeter the time required to reach steady-state varies from ten to twenty hours, with most samples coming into steady-state at fourteen to sixteen hours.

The application of isothermal calorimetry as an analytical technique to radioactive sources is limited to measurements taken on dry solids. Solutions of radioactive materials cannot be accurately assayed because of the extraneous heat generated by radiolysis of the solvent. Oxidation of the dry metallic samples, greatly enhanced by traces of moisture, introduces significant positive error. On the other hand there are several distinct advantages to the calorimetric approach to radiometric analysis: (1) the method is nondestructive and direct, (2) the operation is radioactively cold, and (3) the accuracy of results is independent of the degree of homogeneity of the sample and of the sample geometry.

DESIGN AND OPERATION

The following topics are discussed: the calorimeter unit, the calibration heater circuit, the Maier bridge circuit, the oil bath, the control cabinet, and the operation and calculations.

The Calorimeter Unit

The eight calorimeter units are arranged in a fixed circular pattern and are numbered clockwise beginning with the "dummy" or reference unit as number one (See Figure 1).

In effect the eight-unit calorimeter is a seven-twins instrument since seven measuring units operate against one reference unit. All eight units are identical so that their heat leaks, heat capacities, thermal conduction paths, and heat transfer coefficients are similar. It is essential that each set of twins be identical so that fluctuations of the jacket temperature affect both the reference unit and the measuring unit in the same way. It should be noted that point-to-point temperature variations

are greater in the large oil bath than in the bath of the single-twin instrument although this is not to be considered serious. For this reason the twin method of minimizing the effects of these variations is not as effective with the larger instrument.

The main body of the calorimeter unit consists of two thin-walled concentric copper cylinders, open at both ends (See Figure 2). The two cylinders are held in place by Lucite supports fastened to the top and bottom of the concentric cylinders by brass metal screws. A brass sample container, designed to hold a number -3 tin can, slip-fits inside this main body and is held at the geometric center of the unit by a lip machined on the inside of the inner copper cylinder. Two insulating plugs of Styrofoam® (a Dow product) also slip-fit into the inner cylinder, one above and one below the sample holder. These two plugs minimize the amount of heat that escapes in a vertical direction. The calorimeter body, with its sample container and plugs of Styrofoam®, is housed in a nineteen-inch deep brass submarine well. The Lucite supports slip-fit into the brass well and provide both thermal and electrical insulation between the calorimeter body and the well.

This submarine well is a part of a large brass wheel consisting of eight wells suspended in a circular array from eight brass spokes. The oil of the isothermal bath comes to within two inches of the tops of the walls.

Figure 1. Brass Wheel (Top View).

Figure 2. Cutaway View of Calorimetric Measuring Unit.

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The outer surface of the inner copper cylinder has machined grooves which hold the calibration heater wire, which is bifilarly wound to prevent induced emf's. The grooves hold the wire in position so that the heat is evenly distributed along the cylinder. Electrical insulation between the heater wire and the cylinder is provided by enamel on the wire and by several coats of plastic aerosol insulation on the copper. Nylon lacing cord is wound tightly over the heater to hold it in the grooves.

On the plastic-coated outer surface of the outer cylinder, extending beyond the ends of the cylinder onto the Lucite supports, are the two bifilarly wound bridge wires. These nylon-coated nickel wires are not held in grooves, but they are wound by lathe and held in place by a winding of lacing cord.

Eight copper lugs are embedded in the lip of the upper Lucite support. They are numbered one through eight in a clockwise direction. The first four lugs are attached to leads from the manganin heater wire (two leads from each end), and the last four lugs are attached to leads from the two nickel-bridge wires.

All lead wires and all connections are made of copper to minimize lead wire resistance and to prevent the generation of small emf's at the contact interfaces.

The brass sample holder is made in two longitudinal halves — a number —3 tin can, the outside surface of which is radioactively cold, fits snugly inside the holder and contains the sample. This arrangement prevents radioactive contamination of the calorimeter. The brass container is held closed by two cap screws, one each in the top and in the bottom clamp lug. The top clamp lug is provided with a hole through which a hook is inserted to remove the holder from the unit. The upper plug of Styrofoam® must be removed to gain access to the sample holder (See Figure 3).

The Heater Circuit

The calorimeter heating circuit is used to calibrate the seven measuring units by means of electrical equivalence of heat experiments (See Figure 4). Each unit, including the reference unit, has a manganin wire heater (25B and S) located between the two copper cylinders as described. A four-pole twelve-position selector switch makes it possible to use only one heater at a time. The power supply is a NJE model RB36-2 which has a maximum output of two amperes. The current through the heater is determined by measuring the potential drop across an 0.2 ohm precision resistor (RII) which is placed in series with the heater. A voltage divider made up of a 100 ohm and a

100 kilohm precision resistors (RI and RV) in series, is in parallel with the heater. The potential drop across the 100 ohm resistor is measured to determine the voltage across the heater. Table II lists the exact values for the resistances of the precision resistors and the eight heaters.

TABLE II. Heater Circuit Resistances.

Resistor	Resistance (ohms)
Precision Resistor I	100.00923
Precision Resistor II	0.19188
Precision Resistor V	99,999.23
Heater 1	44.380
Heater 2	47.620
Heater 3	44.320
Heater 4	45.690
Heater 5	44.840
Heater 6	44.550
Heater 7	44.750
Heater 8	45.993

The end product of a calibration run is the sensitivity of the unit at the chosen power input. This is expressed as microvolts per watt. Since it is impossible to calibrate the unit at every possible power, it is necessary to make a plot of sensitivities at a variety of powers and then to interpolate the desired sensitivity from the plot. Since a calibration curve is not a straight line, best results are obtained when each unit is calibrated over a small power range and then used exclusively for samples in that range. Table III lists the units and the power ranges which they operate.

TABLE III. Power Range of Each Unit.

Unit No.	Power Range (watts)	Sensitivity ($\mu\text{V}/\text{watt}$)
2	2 to 4	765-780
3	2 to 4	757-773
4	0 to 0.5	838-848
5	1 to 2	798-808
6	0.5 to 1	788-791
7	3 to 5	806-820
8	2 to 4	766-782

With time and use the bridge wires "age" and cause a change in the sensitivity of the unit. The sensitivities have both increased and decreased, and there is no way to predict these changes. For examples, over a four-month period unit four's sensitivity changed twice. Each change was a 0.4% decrease over two months. Unit five increased 0.4% over two months, and over a four-month period unit two decreased 0.3% in the 0-to 300- μV range while it increased 0.1% in the 3000-to 4000- μV range. For this reason it is necessary to calibrate each unit as often as possible to maintain a well-defined and up-to-date sensitivity curve for each unit.

Figure 3. Exploded View of Calorimetric Measuring Unit.

Figure 4. Heater Circuit.

Figure 5. Bridge Circuit.

The Bridge Circuit

The flux-generated heat from the heat source through the calorimeter and into the isothermal bath is detected by a Maier bridge (See Figure 5). The Maier bridge is a modified Wheatstone bridge having two opposite arms in a reference (dummy) unit and kept at a reference temperature (oil bath). The other two opposite arms are located in a measuring unit whose temperature varies with different samples. If there is no heat source in the measuring unit, then all four bridge arms are at the reference temperature. Under this condition a small amount of current flows across the bridge because of differences in the physical dimensions of the four arms (See Table IV), and the potential drop across RIV is the *bridge null*. When a heat source is in the measuring unit the two arms inside this unit are heated, and their resistances are raised. The result is that more current runs across RIV. The potential drop across RIV is measured, and the bridge null is subtracted from it to give the true potential caused by the thermal output of the sample. Since the null cannot be measured simultaneously with the sample reading, it is necessary to make a plot of bridge nulls from week-to-week and then to interpolate the desired null from the plot. For those units operating in power

TABLE IV. Bridge Circuit Resistances.

Unit No.	Arm 1 (ohms)	Arm 2 (ohms)
1	1678.2	1679.8
2	1717.8	1713.1
3	1699.4	1690.1
4	1681.3	1683.1
5	1688.4	1687.4
6	1686.6	1686.0
7	1741.6	1708.0
8	1683.3	1700.9

Resistor	Resistance (ohms)
III	100.00923
IV	100.00923

ranges below one watt (unit four and six) it is desirable to obtain null readings before and after each sample and each calibration for maximum accuracy.

The power supply for the bridge circuit is a twelve-volt, 205 amp-hour storage battery. The 2 milliamps of current drawn by the bridge are controlled by two micropotentiometers in series with the load. The current is measured by the digital voltmeter across a 100 ohm precision resistor (RIII). Another 100 ohm resistor (RIV) is connected across the bridge and is used to determine the potential drop across the bridge.

In order to prevent the reference (dummy) arms of the bridge from being heated by the current while the other arms in the measuring units remain unheated, it was necessary to use a double-pole double-throw switch to switch the current from the bridge circuit to a dummy circuit of approximately the same resistance. The dummy circuit is used to prevent the sudden burst of current into the bridge circuit which would result from completely stopping the flow of current from the battery, and then starting it again by closing the bridge circuit.

The Oil Bath

The isothermal environment for the eight units is provided by the 120 gallons of 643 hydraulic oil contained in a large water-cooled steel tub. The bath temperature is maintained at $29.3^{\circ} \pm 0.2^{\circ}\text{C}$ by two 2000-watt coils, and is monitored by a platinum resistance thermometer. The bath is cooled continuously by water circulating in the copper tubing inside the tub. When the temperature drops below the limit, a Hallikainen Thermotrol closes the solid-state relay which feeds 18 amperes of current into the heating coils. The bath temperature is set by the Thermotrol controls: the coarse adjustment is set at 5.03, and the fine adjustment at 1.25.

The five-inch, three-blade impeller which stirs the oil is suspended on its shaft from a bearing housing located at the center of the brass wheel. It is driven via a V-belt by a one-half horsepower direct-current motor. A powerstat is used to control the speed of the motor and is set at 50%.

The Control Cabinet

The control cabinet houses the digital voltmeter, the Hallikainen Thermotrol, the power supply for the calibration heater circuit, the control switches, and other parts of the electrical circuitry such as precision resistors, solid-state relay, and terminal boards. There are two panels of controls. The top panel has two micropotentiometers which are used to adjust the current in the Maier bridge circuit. One is a 5000 ohm potentiometer, and the other is a 500 ohm potentiometer; the latter is used for fine adjustments.

The lower panel holds a double-pole, double-throw (DPDT) switch and three selector switches (SS) marked "SHUNT", "HEATER", and "SENSOR". The DPDT switch is used to switch current into the bridge circuit, or out of the bridge circuit into a dummy circuit.

The SHUNT Switch is a two-pole, twelve-position switch used to connect the digital voltmeter with four precision resistors, one at a time, to measure the current or voltage of either the calibration heaters or the bridge. Table V shows the function of each switch position.

TABLE V. Shunt Position Functions.

SS Position	Resistor	Function
1	RI	heater voltage
2	RII	heater current
3	RIII	bridge current
4	RIV	bridge voltage

The HEATER Switch is a four-pole, twelve-position selector switch used to place the calibration heaters into the circuit, one at a time. The position numbers correspond to the heater numbers. Position one is never used since it is connected to the reference unit heater.

The two-pole, twelve position SENSOR Switch completes the bridge circuit, one bridge (or unit) at a time. Since unit one (the reference) is connected to position "0", unit two is connected to position "1", unit three to position "2", and so on to unit eight which is connected to position "7".

All potentiometric measurements are taken with a digital voltmeter (DVM), the DANA model 5700.

Operation and Calculations

Operation of the calorimeter is divided into four parts: determining bridge nulls, calibrating the measuring units, standardizing the measuring units, and assaying samples. Regardless of which part of the operation one is concerned with, the original settings of the control switches are as follows:

1. DPDT on "DUMMY"
2. SHUNT on "3"
3. HEATER — see explanation no. 1 below
4. SENSOR on "1"
5. DVM reading 200.00 μV — see explanation no. 2 below.

Explanation No. 1

During the course of a given day's reading the HEATER Switch is not disturbed. At the end of the readings it is reset to the number of the calorimeter unit which is to be calibrated the next day. If no unit is to be calibrated, the HEATER Switch is set to "0".

Explanation No. 2

If the DVM does not read 200.00 μV , check to see that the controls are on their original settings. If they are, then use the FINE bridge X current adjust knob, turning it counter-clockwise to increase the reading, or clockwise to decrease the reading.

HOW TO DETERMINE BRIDGE NULLS

Those units whose nulls are to be measured should contain an empty, number three can which has been in the unit overnight to allow the system to reach steady-state. When steady-state has been reached, the following procedure should be repeated every hour for three or four hours.

1. Controls on original settings before starting
2. DPDT on "BRIDGE"
3. SHUNT on "4"
4. HEATER — see explanation no. 1 above
5. SENSOR on "X" — see explanation no. 3 below
6. Record DVM reading — this is the null for unit "X"
7. Return to original settings

Explanation No. 3

"X" may be any position on SENSOR from "2" through "7". Since unit one is connected to position "0", unit two is on position "1", unit three on "2", and so on to unit eight which is on position "7".

When the null readings are taken, the average null is plotted against the date.

HOW TO CALIBRATE A MEASURING UNIT

For this calorimeter only one unit can be calibrated per day. On the day before the calibration is to be done, the HEATER is set to the unit's number (never set it on "1" — this is the reference unit). The next day, after the system has come to steady-state, calibration readings are taken each hour for three or four hours.

1. Controls on original settings before starting.
2. DPDT on "BRIDGE"
3. SHUNT on "4"
4. HEATER — do not disturb -- see explanation no. 1 above
5. SENSOR on the unit's number — see explanation no. 3 above

6. Record DVM reading — this is bridge reading for the unit
7. SHUNT on "2"
8. Record DVM reading — this is heater current
9. SHUNT on "1"
10. Record DVM reading — this is heater voltage
11. Return to original setting

To calculate the unit's sensitivity at the measured power:

$$S = R / N/K \times V \times I$$

where: S — Sensitivity ($\mu\text{V}/\text{watt}$)
 R — Bridge reading (μV)
 N — Bridge null from graph (μV)
 V — Heater voltage (μV)
 I — Heater current (μV)
 K — Calibration factor = $5.2139 \times 10^{-9} \text{ volts}^2 \mu\text{V}^{-2} \text{ ohm}^{-1}$

A graph of S versus R-N is made; from this graph the sensitivity at any reading may be interpolated.

HOW TO STANDARDIZE A MEASURING UNIT

A standard containing a certified amount of plutonium or other radioactive material is used to obtain a power correction factor. It is essential that the following information about the standard be available:

1. Sample weight to nearest 0.01 g
2. Exact assay (g/g)
3. Complete isotopic analysis
4. Radiochemical ^{241}Am and ^{238}Pu
5. Date of the ^{241}Am analysis

A computer program is used to calculate the standard's power output every year by days.

When the standard has been run several times in a given unit, a power correction factor for this unit may be calculated:

$$F = R - N/S \times p$$

where: F — Power correction factor (no units)
 R — Bridge reading (μV)
 N — Bridge null from graph (μV)
 S — Unit's sensitivity from graph ($\mu\text{V}/\text{watt}$)
 p — Theoretical power output of the standard (watts)

The dates of the theoretical power output, the bridge null, and the bridge reading must be the same, and the sensitivity must be current with this date. The power correction factor for the seven operating units did not differ from one by more than $\pm 0.2\%$.

ASSAY OF SAMPLES

Samples must be packaged in number —3 tin cans. Metals should be protected from the air inside the can by a plastic bag or some other seal in addition to the can. For convenience in handling and to guard against contamination from a leak in the can seal, oxides should also be sealed in an inner container.

For samples of a single isotope only the isotope's identity and sample weight are needed. For plutonium samples one must know:

1. Sample weight to nearest 0.1 g
2. Approximate assay
3. Complete isotopic analysis
4. Radiochemical ^{241}Am and ^{238}Pu
5. Date of the ^{241}Am analysis

The sample is placed in the correct unit (depends on the estimated power output of the sample) and allowed about twenty hours to reach steady-state. The following procedure is repeated every hour for three or four hours after steady-state has been reached.

1. Controls on original settings before starting
2. DPDT on "BRIDGE"
3. SHUNT on "4"
4. HEATER — do not disturb — see explanation no. 1
5. SENSOR - see explanation no. 3
6. Record DVM reading — this is the bridge reading
7. Return to original settings

The power output of the sample is calculated by:

$$P = F(R - N)/S$$

where: P — Power output of the sample (watts)
 F — Power correction factor (no units)
 R — Bridge reading (μV)
 N — Bridge null from graph (μV)
 S — Unit's sensitivity from graph ($\mu\text{V}/\text{watt}$)

The assay for single isotope samples:

$$G = P/P_s \text{ (weight of sample)}$$

where: G — Assay of the isotope (g/g)
 P — Power output of the sample (watts)
 P_s — Specific power of the isotope (watts/g)

The calculation is somewhat more involved for materials containing a mixture of plutonium isotopes. However, if one knows the theoretical weight of the plutonium in the sample, the expected power can be evaluated. Plutonium samples usually contain a mixture of the following isotopes: ^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Pu , ^{242}Pu , and ^{241}Am . The theoretical power resulting from plutonium is given as ΣP_i where P_i is defined as follows:

$$P_i = (\text{isotopic composition}) (P_s) (\text{theoretical weight of Pu}).$$

The individual power percentage is defined as: $\% P_i = \frac{P_i}{\Sigma P_i}$ which when multiplied by the measured plutonium power, $P_M - P_{Am}^{241}$, gives the amount of power resulting from the i th isotope in the sample, p_i . Thus $p_i = \% P_i (P_M - P_{Am}^{241})$. The amount in grams of the i th isotope is then G_i where $G_i = \frac{p_i}{P_s}$

It follows that ΣG_i is equivalent to the total plutonium weight in grams in the given sample. Division by the total weight of the sample gives the desired assay in gram per gram.

SUMMARY AND CONCLUSION

The multi-unit calorimeter serving as an analytical instrument has been found to be as accurate as the previously described twin isothermal calorimeter, i.e., $\pm 0.2\%$. The most important feature of the multi-unit instrument is its capability. Whereas the twin calorimeter can measure the power output of one given source in a certain period of time, the multi-unit calorimeter is capable of measuring the power output of seven sources in this same period of time.