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FIELD TEST OF THE BULK-ASSAY CALORIMETER

by

R. B. Perry and A. Keddar*

Nondestructive Assay Section
SPECIAL MATERIALS

ARGONNE NATIONAL LABORATORY
9700 S. Cass Avenue
Argonne, Illinois 60439

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*International Atomic Energy Agency

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R. B. Perry

ARGONNE NATIONAL LABORATORY

Argonne, Illinois

and

A. Keddar

INTERNATIONAL ATOMIC ENERGY AGENCY

Vienna, Austria

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INTRODUCTION

The Bulk-Assay Calorimeter described in ANL-NDA-9/ISPO-14 was field tested at the Belgonucleaire mixed-oxide fuel fabrication plant at Dessel, Belgium, May 13-19, 1982. This instrument was developed under ISPO Tasks A-9 and A-47 at Argonne National Laboratory and was supplied to the IAEA through the U.S. support program. Five containers of plutonium-oxide feed stock used in the manufacture of mixed-oxide LMFBR-type fuel were assayed during the test. Electrical measurements to verify the calibration of the calorimeter were also made.

EQUIPMENT DESCRIPTION

The Bulk-Assay Calorimeter is designed to measure the thermal power emitted by plutonium-containing samples. The sample power range of this instrument is 1.4-22.4 W. The maximum sample size is 10.8 cm in diameter and 25.0 cm in length. The instrument package consists of the calorimeter measurement chamber (CMC), the control circuit power bin (CCPB), and the data-acquisition system (DAS). Two sample preheating chambers and calorimeter canisters of two different sizes for containing the samples are also included. The inside dimensions of the canisters are 10.5-cm dia x 20.3-cm high and 10.8-cm dia x 25-cm high. This system is designed to operate on 230/240 V, 50/60 cycle AC.

The calorimeter consists of five (5) concentric cylinders, four of which are temperature-controlled. Their operating temperatures may be measured on the temperature-readout module. The outside cylinder has a measurement point on the inner and outer walls, labeled T_B and T_S, respectively. The empty chamber equilibrium values are: T_3 = 47.56°C ± 0.1°C; T_2 = 45.58°C ± 0.1°C; T_1 = 43.49°C ± 0.1°C; T_B = 40.73°C ± 0.1°C; T_S = 39.83°C ± 0.1°C. The modules in the control bin have front-panel meters which show the status of the various servo-circuits. When the system reaches operating status, all the circuits (T_3, T_2, T_1, T_S) will zero on the coarse scale. A set of 32 test points which monitor voltages at points within the calorimeter and its control circuits are accessed by the DAS. The points of interest to the operator are #1, #2 (measurement-chamber voltage and current) and #30-#32 (A/D calibration points). The use of the test points is described below.

ELECTRICAL CALIBRATION

There are three electrical calibration checks that can be performed with the Bulk-Assay Calorimeter to verify the instrument calibration.

1. Analog/Digital (A/D) Converter Test.

All power measurements are made by measuring voltages with a built-in A/D converter. The accuracy can be checked by measuring the voltage from a built-in voltage reference source (test point 32) and comparing the reading to that obtained with a calibrated digital voltmeter (DVM).

The A/D readings are displayed on a 5-digit liquid crystal display. The A/D was checked at 0, 2, 4, 6, 8, and 10 volts and was compared to the readings of a 4-1/2 digit DVM. No significant differences in the voltage readings were observed. The zero of the A/D is checked by calling test point 30, which grounds the input.

2. Linearity Test

The linearity of the power measurement circuits can be tested by applying power to a built-in calibration coil and measuring the calorimeter response. This test is part of the microprocessor operating program and is performed automatically. The test was performed five times during the exercise. The results are given in Table I.

Table I. Linearity test results.

Run #	Slope	Intercept	P_0
1	-1.0005 ± 0.0005	24.724 ± 0.007	24.716 ± 0.002
2	-1.0002 ± 0.0002	24.737 ± 0.003	24.737 ± 0.002
3	-1.0002 ± 0.0002	24.757 ± 0.002	24.757 ± 0.002
4	-0.99990 ± 0.0002	24.724 ± 0.002	24.724 ± 0.002
5	-1.0005 ± 0.0003	24.769 ± 0.002	24.768 ± 0.001

The slope should be -1.0 and the intercept should be equal to P_0. No significant differences from the expected values were observed.

3. Power Measurement Test

The final calibration check requires measurement with an external DVM of the current and voltage applied to the calibration coil during the linearity test while the test program is running. This test was performed once during run #2 of the linearity test. The results of the test are given in Table II. When the slope was compared to 1.0 and the intercept was compared to 0, no significant difference was observed.

Table II. Calibration of electric coil.

E_1 (V)	E_2 (V)	Power applied $E_1 \cdot E_2/0.97514$ (W)	Power measured
9.213	0.4544	4.2931	4.2894
12.987	0.6399	8.5183	8.5151
15.811	0.7792	12.634	12.633
18.161	0.8949	16.667	16.665
20.34	1.0023	20.907	20.919

Least squares fit: $y = a + bx$

$a = -0.00748$

$b = 1.00055$

$r^2 = 0.999999$

$S_{y \cdot x} = 0.00288$

$S_a = 0.00305$

$S_b = 0.00022$

ASSAY PROCEDURE

The calorimeter and one preheater were set up at Belgonucleaire and were allowed to warm up over the weekend. The first can of PuO_2 was preheated for 30 min before it was transferred to the calorimeter. The second can was preheated while the first one was measured. The cans were preheated and measured in O-ring sealed canisters provided with the calorimeter. The cans were placed in the canisters without any additional heat sinking, such as copper shot. The prediction-of-equilibrium program (developed by ANL and Mound Laboratory under ISPO Task A-47) was used for all measurements.

The calorimeter is an isothermal type and is maintained at constant temperature by an electrical, servo-controlled heater. The baseline power, P_0, is the power required to maintain the calorimeter at constant temperature when the sample chamber is empty. The power, P, required while a sample is being measured is lower than the baseline power. The sample power is

$$P_S = P_0 - P + a$$

where a is the intercept. The value of a was determined to be

$$a = 0.008 \text{ watts}$$

and is added to the power measurement by the microprocessor program.

The baseline power, P_0, was measured immediately after each can was removed from the sample chamber. Each baseline power was an equilibrium value, because the calorimeter reached equilibrium before sufficient data could be collected by the prediction routine to give a predicted value. The measurement data is shown in Table III.

The measurement time for can #1, after 30 min in the preheater, was 4 hrs 45 min, including the baseline measurement. Can #2 was preheated for 4 hrs 45 min while can #1 was measured. This reduced the measurement time for can #2 to 2 hrs, including the baseline measurement. A third can was measured overnight. Can #1 was repeated using the option of allowing the measurement to continue to equilibrium after the predicted value was determined. The predicted and equilibrium values agreed within 0.006 watts, which was less than one standard deviation of the predicted value. The two measurements of can #1 agreed within 0.003 watts, which is equivalent to less than 1 g in 1.634 kg.

Belgonucleaire gave the data for the five cans, corrected for decay to the date of measurement, shown in Table IV. All measurements agree with the declared values within the measurement precision. The largest part of the error given for the measurements in Table III is due to the uncertainty in the specific power. The average uncertainty in the measured power was 0.1%.

Table III. ANL-IAEA Bulk-Assay Calorimeter measurements, Belgonucleaire, 17-19 May, 1982

Can #	P_0	Sample Power (W)	Specific Power (W/kg)	Pu Meas (kg)	Pu Declared	Diff (kg)	% Diff.
1	24.773 ± 0.002	8.0366 ± 0.0098	4.9110 ± 0.017	1.6363 ± 0.0060	1.6343	0.0020	0.12
2	24.772 ± 0.0009	8.7080 ± 0.0150	4.9766 ± 0.017	1.7498 ± 0.0067	1.7516	-0.0018	-0.10
3	24.771 ± 0.001	9.3126 ± 0.0019	4.9766 ± 0.017	1.8713 ± 0.0064	1.8669	0.0044	0.24
4	24.775 ± 0.002	8.7871 ± 0.0083	5.0794 ± 0.018	1.7299 ± 0.0063	1.7270	0.0029	0.17
5	24.770 ± 0.002	8.1784 ± 0.0091	5.0584 ± 0.018	1.6167 ± 0.0060	1.6081	0.0086	0.53
1	24.768 ± 0.001	8.0400 ± 0.0110	4.9110 ± 0.017	1.6371 ± 0.0061	1.6343	0.0028	0.17

Table IV. Isotopic data supplied by Belgonucleaire, corrected for decay to 17-18 May, 1982

Lot	Can No.	g Pu 17/5/82	238	239	240	241	242	Am * 241
1	S00839	1634.3	0.133 ± 0.0013	75.846 ± 0.025	20.712 ± 0.021	2.657 ± 0.0087	0.652 ± 0.0022	0.9920 ± 0.0133
2	S00847	1751.6	0.137 ± 0.0013	75.454 ± 0.025	21.042 ± 0.021	2.696 ± 0.0087	0.671 ± 0.0022	1.0140 ± 0.0135
3	S00850	1866.9	0.137 ± 0.0013	75.454 ± 0.025	21.042 ± 0.021	2.696 ± 0.0087	0.671 ± 0.0022	1.0140 ± 0.0135
4	S00907	1727.0	0.142 ± 0.0014	75.381 ± 0.025	21.094 ± 0.021	2.687 ± 0.0087	0.696 ± 0.0022	1.0771 ± 0.0143
5	S00719	1608.1	0.127 ± 0.0013	74.990 ± 0.025	21.466 ± 0.021	2.709 ± 0.0087	0.708 ± 0.0022	1.1162 ± 0.0148

* Am is expressed as a percent of the total plutonium.

INSTRUMENT PROBLEMS

The printer used in the DAS has proven to be unreliable for this application. The printer shipped with the Bulk-Assay Calorimeter failed after less than 4 hours of use. The calorimeter is inoperable without the printer because it is the primary output device for the microprocessor. It is recommended that the printer be replaced with the type of printer that the ANL Electronics Division has used and found to be reliable in portable equipment.

The microprocessor program was interrupted several times a day during preliminary testing at CEN. This was attributed to power-line noise by CEN personnel, because they have experienced problems with other equipment in the building. This problem did not recur at Belgonucleaire. A simple program change to avoid this problem has been tested with the Small-Sample Calorimeter at ANL. It is recommended that this program fix be incorporated into the Bulk-Assay Calorimeter program.

Most of the cans used at Belgonucleaire are larger than 10.8 cm in diameter and could not be measured in the Bulk-Assay Calorimeter. A survey of container sizes used at other safeguarded facilities is needed to determine if a larger measurement chamber is necessary for optimum use of the Bulk-Assay Calorimeter. It is estimated that six to nine months would be required for fabrication and calibration of a larger chamber.