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THE BULK-ASSAY CALORIMETER:

PART 1. SYSTEM DESIGN AND OPERATION

PART 2. CALIBRATION AND TESTING

by

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NOTE

This report describes the design and operating principles of the ANL Bulk-Assay Calorimeter. The calorimeter was originally constructed at ANL under ISPO Task A-9. Subsequent modifications to the data analysis routines were incorporated into the unit as part of a joint task (ISPO A-47) with Mound Facility-Monsanto. The software supplied by Mound enables the unit to predict and/or recognize the electrical end-point power applied when the system reequilibrates after sample insertion. The prediction algorithm is described in "Real-Time Prediction of Calorimeter Equilibrium," by C. L. Fellers and P. W. Seabaugh.¹ Questions concerning the details of the prediction code should be addressed to C. Fellers or J. Wetzel at Mound-Monsanto, Miamishburg, Ohio 45342.

PART 1

THE BULK-ASSAY CALORIMETER: SYSTEM DESIGN AND OPERATION

I. SYSTEM DESCRIPTION

The Bulk-Assay Calorimeter (see Fig. 1) 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 five calorimeter canisters for containing the samples are also included. 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: $T_3 = 47.56^\circ\text{C}$, $T_2 = 45.58^\circ\text{C}$, $T_1 = 43.49^\circ\text{C}$, $T_B = 40.73^\circ\text{C}$, $T_S = 39.83^\circ\text{C}$ (all temperatures approximately $\pm 0.1^\circ\text{C}$). 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)

Fig. 1. Bulk-Assay Calorimeter.

will zero on the coarse scale. A set of 32 test points which monitor voltages at points within the calorimeter and its control circuitry 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.

II. SYSTEM START-UP

A. Component Interconnection and Power-up

Operator Action

1. Connect calorimeter measurement chamber (CMC) to the control circuit power bin (CCPB),
2 cables.
2. Connect the data-acquisition system (DAS) to the CCPB,
2 cables.

Note: The cable connectors have been chosen so that only one connection sequence is possible.

3. Connect the system power cord to the building power.

Operator ActionSystem Response

Note: The system is designed
for 230/240 V, 50/60 cycle AC.

4. Turn the system on with the
switch on the front panel of
the CCPB.

Bulk-Assay CalorimeterOperation Sequence:

1. ELECT CALIB
2. INPUT PARAMETERS
3. ASSAY SAMPLE
4. BASELINE ASSAY

B. Thermal Stabilization Period

Note: If the DAS did not respond after "power-up," turn power
off momentarily and turn on again.

The following automatic start-up sequence has been programmed into
the DAS under the assumption that the system is at room temperature
(20°C) when powered up. The operator may abort this sequence at any
time by pressing RST.

After printing the messages listed above, the DAS will wait for the
operator to respond to

"DLY (M)"

with a delay period (min) during which the instrument will reach operating equilibrium. No data will be processed during this period. The default value is 0 min. Since the DAS will determine when the system actually reaches equilibrium, an "0" response will result in a calibration being completed in minimum time. However, if the user allows the calorimeter to warm up overnight, a delay may be entered to ensure that the following calibration is performed just prior to sample assay in the morning. Allow 120 min for the electrical calibration when determining the delay time.

C. Internal Electrical Calibration

After the delay period, the system will enter the automatic electrical calibration sequence. The DAS will determine the baseline power necessary to maintain thermal equilibrium in the absence of a sample. This procedure takes approximately 30 min. Next, the system will calculate the incremental power necessary to obtain a six (6) point electrical calibration. This power is applied to the electrical calibration coil, and both the applied power and the system response are measured. This procedure takes an additional 90 min. A linear least squares fit is performed on the six-point data set, and the slope and intercept are stored. At this time, the system requires operator interaction. The user may elect to perform a more detailed calibration checkout or to begin sample assay.

III. SYSTEM CHECK-OUT

A. DAS A/D Certification

Note: Alteration of the A/D calibration will affect the system Pu calibration. Do not adjust the slope control until you are sure of the test instrument calibration.

Connect the 4-1/2 digit multimeter supplied with the instrument to the test points at the bottom of the calibration module marked COM and VR. Check the A/D zero point by requesting test point 30.

SYSTEM RESPONSE

OPERATOR RESPONSE

TEST PTS

PRESS "TP"

A/D CALIBRATE?

PRESS "ENTR" (NO)

ALL?

PRESS "3,0,ENTR"

The display should now read 00000. (Note: The display will blank if the voltage is between ± 0.1 V and is not 0. If this occurs, a reading may be obtained by pressing "PRNT".) The zero adjustment is on the right side of the DAS panel above the keyboard and is screwdriver adjustable.

The slope of the A/D may be obtained by using the voltage-averaging

A/D calibrate option of the TP routine.

SYSTEM RESPONSE

OPERATOR RESPONSE

TEST PTS

PRESS "TP"

A/D CALIBRATE

PRESS "YES"

PRINT ALL

PRESS "ENTR" (NO)

DELAY - MIN

PRESS "0 ENTR"

#DP/RUN

PRESS "100 ENTR"

RUNS

PRESS "1 ENTR"

TEST POINT #

PRESS "32 ENTR"

The program will average 100 voltage readings and will print the mean and the standard deviation of the mean. The average will also appear in the liquid crystal display (LCD). This number should agree with the DVM readings. To check the linearity of the A/D, measure 5 voltages covering the range 2-10 V. The voltage is adjusted by turning the black knob on the calibration module in the control bin. Press "YES" after each voltage adjustment to obtain the reading and record the DVM reading. After this procedure is completed, a zero reading may be obtained by pressing "ENTR" and changing the test point number to 30. A least squares analysis of the data should be used to determine if the slope is significantly different from 1 before adjusting the slope. The slope control is at the top, right, of the DAS. Adjustment should be made with the voltage near 10 V. (Note: The DAS has a 12-bit A/D with

a resolution of 0.0037 volts. This should be taken into consideration when attempting to adjust the slope.)

B. Power- Measurement Circuit Check

Connect the DVM to the red and black test points on the front of the calibration module. With the switch in the E1 position, the voltage at the test points is the voltage across the calibration coil. With the switch in the E2 position, the voltage is proportional to the current through the calibration coil and standard resistor. The power

$$P_{DVM} = (E1 \times E2)/R_{STD}$$

where $R_{STD} = 0.97514$ ohms

measured by the DVM should agree with the power printed by the program. The measurement must be made while the calibration program is running, and the results should be fitted to a line using a linear least squares code. The calibration routine is started by pressing "CAL". The routine will first determine the baseline power and will calculate the incremental calibration power steps. After the "CALB" LED comes on, measure E1 and E2. Repeat for each of the data points. Do not read the DVM while the printer is operating as noise is introduced into the system. The fit of $P_{DVM}(x)$ vs $P_{CAL}(y)$ should give a slope of 1 and an intercept of 0 within the measurement precision.

IV. SAMPLE ASSAY

A. Parameter Input

Prior to sample assay, the operator may enter parameters which can be used in calculating sample Pu mass from the power determination.

SYSTEM RESPONSEOPERATOR RESPONSE

ASSAY PARAMETERS

(W)/(KG)

3.674 +/- 0.01

NORM

1.0

SIG(SYS)

0.0

PRESS "INIT"

Enter effective specific power to convert thermal power to Pu mass. If unknown, press ENTR

Enter optional user-determined normalization factor
1.0 must be entered if change in calibration is not wanted

Enter systematic uncertainty from table for sample power range used.
(See Part 2, Table II.)
Default value is zero.

The remaining parameters permit the operator to calculate the effective specific power from a set of sample isotopic data. The user may also update the isotopic data to account for radioactive decay occurring since the isotopic assay. Either the elapsed time may be entered or the program will calculate the number of days between two dates. The isotopic percents are entered following the prompt statements. The Am-241 must be entered as a percent of the total Pu. If the Am-241 is given as percent of total sample weight, then the correct value to enter is obtained by dividing the Am-241 by the weight

fraction of Pu. The keys controlling the calculations are "FRAC" (decay) and "SP" (effective specific power). If the effective specific power is known, this section can be skipped by pressing "RST".

B. Assay of Fissile Samples

Before a sample is measured in the Bulk Calorimeter, the sample container should be checked for surface contamination and loaded into one of the calorimeter canisters provided with the instrument. The calorimeter canister containing the sample should then be placed into the preheater for a minimum of 30 minutes. (For additional information on preheating, see Part 2, Sec. VI.) Cans of plutonium oxide, mixed oxide or metal, with or without external plastic bagging, can be placed directly into the calorimeter canister without heat sinking the container with shot. Small, concentrated heat sources, such as Pu-238 heat standards, can be packed in copper or aluminum shot to reduce the measurement time and reduce the sample temperature. DO NOT PLACE A SAMPLE DIRECTLY INTO THE CALORIMETER MEASUREMENT CHAMBER WITHOUT USING A CALORIMETER CANISTER. A significant loss of measurement precision and accuracy will result if a calorimeter canister is not used. In addition, the placing of concentrated, high-power heat sources directly into the calorimeter measurement chamber without using a calorimeter canister could damage the styrofoam insulating materials at the bottom of the measurement chamber, because these sources can have surface temperatures above the melting point of styrofoam.

After the calorimeter canister containing the sample is preheated, the canister should be quickly transferred to the calorimeter

measurement chamber. Any delay during the transferring of the canister or in replacing the lid of the measurement chamber will increase measurement time.

A sample assay is a two-step process. The sample power is proportional to the difference between the calorimeter-applied electrical power necessary to maintain the system at equilibrium in the presence or in the absence of a Pu sample. First, the Pu sample is assayed (press "RUN"). The assay time varies from 2-5 hrs, depending on sample composition and the degree of preheating. After the DAS has determined the equilibrium electrical power with the sample present, a baseline measurement (press "P₀") must be taken. This takes approximately 45 min. The sample power and mass are calculated at the end of this run.

The assay routines may be operated by using a number of run-time options. The user may choose to: 1) print the intermediate data points; 2) have the instrument predict the final thermal equilibrium value before it is reached; 3) halt data acquisition after a prediction or continue until the final equilibrium value is recognized.

Normal operation is covered by the default options (1 = NO; 2 = YES; 3 = YES). This may be selected by responding to the microprocessor prompt "DEFAULT OPTIONS?" with "YES". The system will monitor the calorimeter power until a thermal end point is predicted. It will print the final value with no intermediate output.

A continuous I/O mode is available by responding as follows:

DEFAULT OPTIONS?	ENTR (NO)
DUMP MEAS PT?	YES
USE PREDICT?	ENTR (NO)

A program check may be performed by requesting both prediction and equilibrium recognition with intermediate I/O.

DEFAULT OPTIONS?	ENTR (NO)
DUMP MEAS PT?	YES
USE PREDICT?	YES
STOP AFTER PREDICT?	ENTR (NO)

C. Baseline Assay and Mass Computation

A system baseline measurement ends the assay sequence. It consists of determining the calorimeter applied power necessary to maintain thermal equilibrium in the absence of a sample. This P_0 check must be performed after the sample assay to use the mass calculation routines.

The sample power is calculated as the difference between the "RUN" and " P_0 " assays. This value is corrected for the slope determined in the electrical calibration and the operator input "NORM" constant

(default value = 1.0)

$$P_{\text{sample}} = [\text{NORM} * (P_0 - P_C) - A_0] / A_1$$

The uncertainty in this value is given by:

$$s(P_S) = \text{NORM} * [s(P_0)^2 + s(P_C)^2]^{1/2} / A_1 \quad (A_0 \text{ and } A_1 \text{ have been treated as constants})$$

The sample Pu mass is determined using the operator-provided effective specific power (ESP W/KG)

$$M = P_{\text{sample}} / \text{ESP}$$

The uncertainty in this value is given by

$$s(M) = M * [(s(P_S)/P_S)^2 + (s(\text{ESP})/\text{ESP})^2 + \text{SSQ}]^{1/2}$$

where SSQ is the operator-supplied systematic uncertainty (default value = 0.0).

SAMPLE DATA TAPEDAS RESPONSEOPERATOR RESPONSE

Assemble instrument and
power up

BULK ASSAY CALORIMETER

OPERATION SEQUENCE

- 1 - ELECT. CALIB.
- 2 - INPUT PARAMETERS
- 3 - ASSAY SAMPLE
- 4 - BASELINE ASSAY

DLY (M) 300

Press 300 ENTR
(5 hr delay)

CALIBRATE

EQUIB POWER (W)
24.749 +/- 0.00199

06 CAL PNTS
EQUIB DLY (M) 05
0250 DP/RUN

CALIBRATE POWER (W)
0

MEAS POWER (W)
24.749 +/- 0.00199

CALIBRATE (W)
4.2884 +/- 1.3091 E-4
MEAS POWER (W)
20.457 +/- 3.4047 E-4

CALIBRATE (W)
8.3593 +/- 2.1760 E-4
MEAS POWER (W)
16.396 +/- 4.1555 E-4

CALIBRATE (W)
12.523 +/- 2.4123 E-4
MEAS POWER (W)
12.221 +/- 3.1050 E-4

CALIBRATE (W)
16.785 +/- 3.0178 E-4
MEAS POWER (W)
7.9650 +/- 2.8623 E-4

CALIBRATE (W)
 20.854 +/- 2.0715 E-4
 MEAS POWER (W)
 3.8916 +/- 2.6435 E-4

SLOPE
 -1.0000

INTCPT
 24.748

ASSAY PARAMETERS

(W)/(KG)
 3.674 +/- 0.01

Enter effective specific power
 Press 3.674 ENTR
 Press 0.01 ENTR

NORM

Enter power normalization
 constant

1.0

Press 1.0 ENTR

SIG(SYS)
 0.0

Enter systematic error
 Press 0.0 ENTR

The systematic error is
 approximately $0.0013P + 0.0015$
 watts. A value of 0.004 could
 be entered as a conservative
 estimate.

TIME

Skip ESP calculation
 Press RST

DAS READY

Insert sample
 Press RUN

SYSTEM POWER (W)

63 DP/RUN
 DEFAULT OPTIONS?

Press YES
 (1.1-2.5 hr wait)

YES

PREDICT POWER (W)
 20.947 +/- 0.00911

Remove sample
 Press P₀

BASELINE POWER (W)

63 DP/RUN

DEFAULT OPTIONS?

Press YES

(0.4-1.0 hr wait)

YES

EQUB POWER (W)

24.749 +/- 0.00285

BASELINE POWER (W)

24.749 +/- 0.00285

ASSAY POWER (W)

20.947 +/- 0.00911

SAMPLE POWER (W)

3.8101 +/- 0.00955

Sample power = $P_0 - P + a_0$
 $a_0 = 0.008$ is added as an
 intercept correction.

(W)/(KG)

3.674 +/- 0.00999

PII (KG)

1.0371 +/- 0.00384

V. PREHEATER OPERATION

1. Plug the line cord into a 230/240 V, 50/60 cycle AC outlet.
2. Turn the power switch to on, and press the reset button.

(The preheater is protected from overheating by a high-temperature limit switch that will de-energize the control relay and turn off the power. Press the reset button to resume operation. If the preheater does not cool with the power off, the sample may be overheating the preheater. If the relay continues to trip when the preheater is empty, there is a malfunction in the control circuit. An open control thermistor may be the cause.)

3. Connect the temperature output to the calorimeter temperature-readout module.
4. After the calorimeter and the preheater have reached a stable operating temperature, the temperature of T_3 and the preheater can be compared by rotating the switch between T_3 and PH1 or PH2. To raise the temperature of the preheater, rotate the temperature adjustment control clockwise. The control calibration is approximately 1.5°C/turn.

The temperature of the preheater will read higher when there is a sample in the preheater. The temperature difference will depend on the sample power and the location of the sample in the calorimeter canister. Readjustment of the preheater temperature with a calorimeter canister in place is not required.

A sample in its calorimeter canister should usually be preheated a minimum of 30 minutes. (See Part 2, Section III.) Normally, while one sample is being measured, another sample would be preheated, and several hours would therefore be available for preheating, except for the first sample. If the first sample is preheated for more than 30 minutes, the time spent in the preheater would probably not be made up by time saved in the calorimeter.

The calorimeter power is plotted in Fig. 2 as a function of time, for preheating times from 30 min to 320 min, to show the effect of preheating on the measurement time. (This data is summarized in Table V, Part 2.) Samples with power outputs greater than 12 watts require less preheating. If the sample power is greater than 20 watts, the calorimeter canister may become hotter than the calorimeter operating temperature when the canister is allowed to warm up to a steady-state temperature in room-temperature air. When the calorimeter canister is placed into the calorimeter, both will cool slowly, and the measurement time will be longer than normal. Therefore, a high-power sample should be placed in a cold calorimeter canister and preheated until the preheater temperature reaches the calorimeter operating temperature. This will only take 10-15 min. The calorimeter canister should then be placed immediately into the calorimeter measurement chamber to prevent overheating.

Fig. 2. Effect of preheating on measurement time.

VI. DATA ACQUISITION SYSTEM

A. Physical Description

The data-acquisition system (DAS) is housed in a 47-cm x 35-cm x 16-cm attache case and has a total weight of 5 kg. It is a totally dedicated microprocessor-controlled device, designed around the INTEL 8085A. The system memory consists of 16K bytes of erasable, programmable, read-only memory (EPROM) and 1K bytes of random-access memory (RAM). The program resides in the permanent memory, EPROM, and does not need to be reentered after the device is powered down. The EPROM is organized on four 4K-byte INTEL-2732 chips, the last 4K bytes being exchangeable. The RAM is organized on four 256-byte INTEL-8156 chips and serves as scratch-pad storage for the program. The microprocessor receives input either from the calorimeter through the 12-bit ADC or from the 25-key multifunctional keyboard. System output is through either the BOMAR TP3120-2 printer or the liquid crystal display (LCD). A set of 6 LEDs are available to reflect system status. Detailed schematics are included in Appendix B.

B. Keyboard Action-Response

1. RST = Restart

Function: To place the system in an inactive mode, where it will monitor the keyboard for a user command. All data processing is stopped. The results of any uncompleted calculation will not be available to the user. The system will also return to the inactive mode after completing any noncyclical routine.

Use: This key should be used when aborting a particular routine.

Status Light: READY

Printer: DAS READY

2. ENTR = Enter

Function: To signal the end of a numeric input.

Use: This key should be used when responding to a microprocessor prompt statement with a numerical value. It also acts as a "NO" in a situation requiring a YES/NO response.

Printer: Carriage return - Line feed

3. BS = Backspace

Function: To permit the user to erase an undesired response to a microprocessor prompt statement prior to pressing ENTR.

Use: This key repositions the input ASCII array by one character each time it is pressed.

Printer: Backspace

4. EXT = Execute external program

Function: To enable the user to expand the computational abilities of the DAS. The DAS is designed so that the last 4K of the 16K PROM memory is interchangeable by replacing the chip in the zero force socket with a different INTEL 2732 containing a new program. (CAUTION: Insertion of the chip upside down may cause damage to the system.)

Use: Reserved for future expansion.

5. TP = Diagnostic test points

Function: To enable the user to examine the voltages of any

of 32 selected locations within the calorimeter (see Appendix A for TP locations within the calorimeter).

Use: This routine may be used to

- a) Continuously monitor a particular point on the liquid crystal display;
- b) Obtain a printout of all 32 points;
- c) Obtain an average and standard deviation of the voltage of a particular test point.

The use of this routine is described in Part I, Section 3.A, entitled "DAS A/D Certification."

Status Light: A/D

Liquid Crystal Display: TP voltage

6. PRINT = Print

Function: To allow the user to obtain a printed record of the value on the liquid crystal display.

7. INIT = Initial parameter input

Function: To permit the user to input parameters which will be used in data analysis by the microprocessor.

Use: The use of this routine is described in Part I, Section 4.A, entitled "Parameter Input."

8. FRAC = Mass fraction decay correction and effective specific power calculation

Function: To calculate the change in the mass percentages in a previously isotopically analyzed sample resulting from radioactive decay. A current set of mass percent will be generated which will then be used to calculate effective specific power.

Use: The program requires that the initial mass percent and the elapsed time be entered through INIT prior to execution.

Printer: The decay-corrected isotopic mass percent and the effective specific power are printed.

9. SP = Effective specific power readout

Function: To check the value used for thermal power emitted per gram of the material to be assayed as well as the associated uncertainty.

Printer: The effective specific power and its uncertainty.

10. P_0 = Empty chamber baseline analysis

Function: To determine the power supplied by the calorimeter to maintain a constant temperature when no heat-producing source is present; to compare this with the power when the sample is in place and derive the sample power and mass.

11. RUN = Sample assay

Function: To determine the calorimeter-supplied power in the presence of a Pu-containing material.

12. CAL = Electrical calibration

Function: To supply a series of known electrical powers to the sample chamber, and to determine the resulting measured powers, thus allowing the user to derive a relation between the measured and actual sample power.

NOTE: A simulated output tape showing the results of typical operations is given in Section IV.

VII. DATA ANALYSIS SOFTWARE

A. Effective Specific Power

In order to obtain maximum utility from the calorimeter, the measured sample power must be combined with additional isotopic information to determine the plutonium content of the sample. If the unknown contains only one isotope of plutonium, the generated heat is related to the sample mass by the equation

$$W_U = 2.1193 \times M \times Q/T_{1/2} \times A$$

where

W = power (nW)

M = mass of Pu (g)

Q = total disintegration energy (MeV)

$T_{1/2}$ = half-life of the isotope of Pu (yrs)

A = gram atomic weight (g)

Under normal assay conditions, nuclear fuel will contain a mixture of radionuclides, the principal power producers being $^{238-242}\text{Pu}$ and ^{241}Am . Consequently, the sample power may be obtained from

$$W_U = M \cdot \sum R_i \cdot P_i = M \cdot P_{\text{eff}}$$

where

- R_i = mass fraction of the radionuclide
 P_i = specific power of a Pu isotope mW/g
 P_{eff} = effective specific power of the sample

Two techniques are suggested for determining the effective specific power.² Representative samples may be subsequently analyzed chemically to determine the Pu content (mW/g). This method usually requires multiple measurements distributed in time to account for isotopic decay. The second technique requires that the isotopic composition be determined by gamma-ray or mass spectrometry.³ Determination of radionuclide abundances by gamma-ray spectrometry has the advantage of being nondestructive and may be conducted concurrently with a calorimetric analysis. While the method chosen may differ from assay to assay, the programs supplied will work with either technique.

1. Radioactive Decay Correction

The isotopic mass ratios are continually changing owing to radioactive decay within the sample. In particular, the beta decay of ^{241}Pu to ^{241}Am , which is a short-lived alpha emitter, results in an increase in the effective specific power with time. If a detailed isotopic analysis of the sample material has been made at an earlier date, an on-site determination of the mass fractions may be unnecessary. The present composition may be calculated by means of the following decay equations:

$$F_i^0 = F_i^0 \exp(-\lambda_i t) / \sum F_i^0 \exp(-\lambda_i t)$$

$$F_{AM} = [1. \sum F_i^0 \exp(-\lambda_i t)] \cdot [F_{AM}^0 \exp(-\lambda_{AM} t)$$

$$+ [\lambda_{241} \cdot F_{241}^0 / (\lambda_{241} - \lambda_{AM})] \cdot [\exp(-\lambda_{AM} t) - \exp(-\lambda_{241} t)] \}$$

where

F_i^0 = mass % of Pu isotope at last analysis

F_{AM}^0 = mass % of ^{241}Am (relative to Pu) at last analysis

λ_i = decay constant of Pu isotope (day^{-1})

λ_{AM} = decay constant of ^{241}Am (day^{-1})

t = time elapsed since last analysis (days)

<u>Isotope</u>	<u>Decay Constant (day^{-1})</u>
^{238}Pu	$2.1617 \cdot 10^{-5}$
^{239}Pu	$7.880 \cdot 10^{-8}$
^{240}Pu	$2.903 \cdot 10^{-7}$
^{241}Pu	$1.322 \cdot 10^{-4}$
^{242}Pu	$5.08 \cdot 10^{-9}$
^{241}Am	$4.372 \cdot 10^{-6}$

See reference 2.

2. Effective Specific Power Calculation

The effective specific power is defined as the weighted sum of the isotopic specific powers. The uncertainty in this value is dependent upon the measurement errors in both the isotopic mass fractions and the specific power.

$$P_{\text{eff}} = \sum_i F_i \cdot P_i / \sum_i F_i$$

$$\sigma^2(P_{\text{eff}}) = \sum_i \left(\frac{F_i P_i}{\sum F_i} \right)^2 \left(\frac{\sigma^2(F_i)}{F_i^2} + \frac{\sigma^2(P_i)}{P_i^2} \right)$$

where

- F_i = mass % of the radioisotope
 $\sigma(F_i)$ = uncertainty in F_i
 P_i = isotopic specific power (mW/g)
 $\sigma(P_i)$ = uncertainty in P_i (mW/g)
 P_{eff} = effective specific power (mW/g)
 $\sigma(P_{\text{eff}})$ = uncertainty in P_{eff} (mW/g)

<u>Isotope</u>	<u>Specific Power (mW/g)</u>
^{238}Pu	567.16 $\pm$ 0.57
^{239}Pu	1.9293 $\pm$ 0.0053
^{240}Pu	7.098 $\pm$ 0.015
^{241}Pu	3.390 $\pm$ 0.002
^{242}Pu	0.1146 $\pm$ 0.0003
^{241}Am	114.23 $\pm$ 0.16

See reference 2.

In general, there is a negligible increase in the uncertainty reported in the isotopic mass % as a result of radioactive decay. This is not always the case for ^{241}Am . If ^{241}Am was absent at the time of the original analysis, or only present in minute amounts, then changes in (^{241}Am) must be considered.

$$\sigma^2(^{241}\text{Am}) \approx [1.034t(\lambda_{241} - \lambda_{\text{AM}}) \cdot \sigma(^{241}\text{Pu})]^2 + \sigma^2(^{241}\text{Am})$$

B. Analysis of Sample Power

1. Data Acquisition

The calorimeter feedback circuitry adjusts the power supplied to the inner cylinder coils so that a constant temperature environment for the sample chamber is maintained. The inner coil (T_3) voltage and current are alternately monitored by a 12-bit analog-to-digital converter (ADC), which transmits data to the microprocessor at a rate of ten readings/sec. Software multiplication of each voltage-current pair produces an "instantaneous" T_3 power. Ten such measurements are averaged to produce a two-second reading which is displayed on the liquid crystal display. The system will then calculate the average power $[E(P_i)]$ supplied by the calorimeter and its standard deviation. Since the microprocessor performs floating-point arithmetic with five-digit software (NOVONICS FP 708), it is necessary to use the following biased-average approach to maintain sufficient accuracy.

Given a set of power measurements

$$P_i \quad i = 1, N$$

Define a new set

$$\hat{P}_i = (P_i - P_1) \quad i = 1, N$$

then

$$E(\hat{P}_i) = \Sigma \hat{P}_i / N = E(P_i) - P_1$$

$$\therefore E(P_i) = E(\hat{P}_i) + P_1$$

Also, since

$$P_i - E(P_i) = \hat{P}_i - E(\hat{P}_i),$$

$$s^2\{E(P_i)\} = \Sigma[\hat{P}_i - E(\hat{P}_i)]^2 / (N - 1)(N)$$

2. Data Analysis

The sample chamber (T_3) is held at a constant temperature of 47.5°C. When no heat-producing source is present, the calorimeter supplies a constant power, P_0 , of approximately 24 W to maintain this temperature. If a sample containing heat-producing plutonium is placed in the calorimeter, the power which the circuitry must supply to maintain 47.5°C drops to a lower value P_c . The radioactive decay produced power is the difference between the electrically supplied power in the presence (P_c) and absence (P_0) of a sample when the calorimeter is at thermal equilibrium. The time elapsed between insertion of the sample and the reestablishment of thermal equilibrium is dependent on sample temperature, sample composition, and thermal coupling between the sample and measurement chamber. This time may vary from 2-5 hours, depending upon the extent of sample preheating (see Section V).

In order to increase sample throughput, a subroutine which predicts final equilibrium power has been incorporated into the data analysis code. The user may reduce sample measurement time 30-50% with this routine (see Part 2, Section III). This code was developed by J. Wetzel and C. Fellers of Monsanto Mound Laboratory using a previously tested algorithm.¹ The algorithm is based on the assumption that, although the

approach to equilibrium is exponential with multiple time constants, the final portion of this approach may be approximated by a single time constant. In this region, if the electrically supplied power is monitored at constant time intervals, the equilibrium value may be predicted by solving a single exponential growth/decay equation

$$P_t = P_E(1 + Be^{-\lambda t})$$

where

- P_t = power at time t
- P_E = equilibrium power
- λ = system time constant
- t = time
- B = scaling constant

For a set of three data points equally spaced in time, P_F may be solved as follows:

$$P_F = \frac{P_2 - P_1 e^{-\lambda \Delta t}}{1 - e^{-\lambda \Delta t}}$$

The time constant is related to the measured power by

$$e^{-\lambda \Delta t} = \frac{P_3 - P_2}{P_2 - P_1}$$

If the assumption of a single time constant is valid, successive predictions will agree within random noise. The operation of this routine is shown schematically in Fig. 3.

Fig. 3. Flowchart for operation of prediction routine.

PART 2

CALIBRATION AND TESTING OF THE
BULK-ASSAY CALORIMETER

The Bulk-Assay Calorimeter was calibrated at 23 points with heat sources from 1.4 to 22.4 watts. The 23 measurement points defined a linear response function with sufficient precision to reduce the systematic error to less than the random error. The combined measurement error varied with sample power from 1.4% to 0.1% over the range of calibration measurements.

The instrument performance can be summarized as follows:

Wattage range	1.4 to 22.4 watts
Systematic error	0.06% to 0.02%
Random error	0.7% to 0.05%
Uncertainty of a single measurement (95% confidence level)	1.4% to 0.1%

Assay time, including baseline measurement:

Equilibrium	3.0 hours
Prediction	2.2 hours

No bias between predicted and equilibrium values.

The heat distribution error was not significant, compared to the random measurement error.

I. CALIBRATION

A. Procedure

Two standards, consisting of encapsulated Pu-238, were obtained from Mound Laboratory. The power of standard A was quoted as 1.9343 watts, and that of standard B as 3.8566 watts, as of June 28, 1981. The decay constant used by Mound for this material was $\lambda = 0.0000213817$ days⁻¹. A third standard of 5.7909 watts was obtained by measuring A and B together. In order to calibrate over a range of 22 watts, a three-step procedure was used. In the following,

P_0 is the baseline power with no sample in the chamber,

P is the power with a sample in the chamber,

$P_m = P_0 - P$ is the measured sample power,

P_c is the known power of the standards or the corrected power of measured samples.

The power of standard A was measured twice, and that of standards B and A + B three times each. A least-squares fit of these eight points to the equation

$$P_m = \alpha + \beta P_c \quad (1)$$

gives

$$\alpha = -0.0108$$

$$S(\alpha) = 0.0069$$

$$B = 1.0091$$

$$S(B) = 0.0016$$

or

$$P_c = 0.9910P_m + 0.0107 \quad (2)$$

Power measurements were made for eight secondary standards which consisted of various numbers of plutonium-containing G-rods. The core weight of each of these rods is accurately known; they were all manufactured from the same lot of plutonium, and, therefore, all have the same specific power. Estimates of the true power, P_c , were obtained using the measured power and Eq. 2. A specific power was computed for each sample by dividing each P_c by the corresponding core weight. The average, 3.7777 ± 0.00216 mW/g, is the best estimate of the specific power of all samples. This average specific power, multiplied by the sample core weight, is the value used for the true total power, P_c , of the sample. A ninth secondary standard was made by measuring together the largest secondary standard (74G) and the two primary standards (A and B). The true value of this ninth standard was taken to be equal to the sum of the true values of its parts. A linear least-squares treatment of the P_m and P_c data for the nine secondary standards, together with the data from the eight measurements of the primary standards, gives a calibration line to about 12.4 watts. The equation of this line is

$$P_C = 0.99106P_m + 0.01064 \quad (3)$$

In order to extend the calibrated range to about 22 watts, three tertiary standards were made by measuring three samples of Pu and obtaining estimates of their true powers with the calibration Eq. 3. These samples are:

- C - a quantity of high Pu-240 plutonium
- D - 11 Pu-238 pacemakers
- E - 22 Pu-238 pacemakers

Three additional points were obtained by measuring together the standards A+B+C, A+B+C+D, and A+B+C+E. The true value of the power for the compound samples were taken as equal to the sums of the true powers of their parts. A linear least-squares treatment of all 23 data points produced the calibration equation

$$P_m = (1.00831 \pm 0.00019)P_C - (0.00812 \pm 0.00167) \quad (4)$$

or

$$P_C = 0.99170P_m + 0.00805 \quad (5)$$

All calibration data are presented in Table I. The true values for samples containing the primary standards A and/or B have been corrected for decay to the date of measurement.

TABLE I. Calibration data.

<u>Sample</u>	<u>Date</u>	<u>P₀</u>	<u>P</u>	<u>P_m</u>	<u>P_c</u>	<u>Core Wt</u>	<u>Spec Pwr</u>	<u>P_c calc</u>
A	8/14/81	24.984	23.042	1.942	1.9323			
A	8/25/81	24.989	23.052	1.937	1.9319			
B	8/14/81	24.983	21.100	3.883	3.8527			
B	8/25/81	24.990	21.117	3.873	3.8518			
B	8/26/81	24.991	21.121	3.870	3.8518			
A+B	8/13/81	24.990	19.152	5.838	5.7853			
A+B	8/26/81	24.990	19.167	5.823	5.7837			
A+B	8/26/81	24.993	19.175	5.818	5.7837			
16G	8/12/81	24.982	23.555	1.427	1.4249	377.18	3.77777	1.4249
24G	8/11/81	24.982	22.837	2.145	2.1365	565.65	3.77707	2.1369
32G	8/7/81	24.982	22.117	2.865	2.8500	753.48	3.78245	2.8464
40G	8/17/81	24.990	21.409	3.581	3.5595	941.96	3.77832	3.5584
48G	8/7/81	24.984	20.687	4.297	4.2691	1130.44	3.77649	4.2705
56G	8/10/81	24.984	19.970	5.014	4.9796	1380.87	3.77566	4.9823
64G	8/11/81	24.982	19.250	5.732	5.6912	1507.17	3.77608	5.6936
74G	8/14/81	24.990	18.359	6.631	6.5821	1742.59	3.77719	6.5830
74G+A+B	8/17/81	24.990	12.520	12.470				12.3677
C	8/27/81	24.991	13.120	11.871				11.7757
D	8/31/81	24.997	22.652	2.345				2.3347
E	8/31/81	24.998	20.297	4.701				4.6696
A+B+C	8/27/81	24.994	7.302	17.692				17.5591
A+B+C+D	8/28/81	24.994	4.952	20.042				19.8936
A+B+C+E	8/28/81	24.994	2.580	22.414				22.2285
						Av	3.77769 +0.00216	

B. Errors

In the calibration process, the standard values, P_c , were treated as the independent variable, and a regression of P_m (the measured values) on P_c was made to obtain an equation of the form of Eq. 1.

Both α and β have associated standard deviations, $S(\alpha)$ and $S(\beta)$. In practice, the measured power, $P_m = P_0 - P$, is substituted into

$$P_c = (P_m - \alpha)/\beta \quad (6)$$

to obtain P_c , the sample power corrected for bias. The measured power also has a standard deviation, $S(P_m) = [S^2(P_0) + S^2(P)]^{1/2}$.

Applying the usual rules for the propagation of errors, the variance of the corrected sample power is

$$S^2(P_c) = \frac{S^2(P_m)}{\beta^2} + \frac{S^2(\alpha)}{\beta^2} + \frac{(P_m - \alpha)^2 S^2(\beta)}{\beta^4} \quad (7)$$

The first term on the right side of this equation is the random error due to the uncertainties in the measurement being made. The last two terms are due to the uncertainties in the calibration equation and are, therefore, a systematic component of the error in all measurements in which this equation is used. Substituting the numerical values of Eq. 4 into Eq. 7, we obtain

$$\begin{aligned}
 S^2(P_c) = & 0.9836S^2(P_m) + 3.49 \times 10^{-8}P_m^2 \\
 & + 5.67 \times 10^{-10}P_m + 2.74 \times 10^{-6}
 \end{aligned}
 \tag{8}$$

The last three terms make up the systematic portion of the variance, $S^2(P_c)$. This varies from about $(0.0017)^2$ watts² for a 2-watt sample to $(0.0048)^2$ watts² for a 24-watt sample. Table II lists the systematic standard deviations for sample powers of 1 to 24 watts.

The constants of the calibration line have been incorporated into the program for the calorimeter so that the sample power that is printed is the corrected value, P_c . The standard deviation that is printed is the random measurement error and does not include the systematic error due to the calibration. This latter portion can be obtained, by interpolation, from Table II. If we let S_r be the random measurement standard deviation and S_s be the systematic calibration standard deviation, then the total standard deviation is

$$S(P_c) = [0.9836S_r^2 + S_s^2]^{1/2}$$

For example, if the sample power measured is 6.631 ± 0.00176 , then

$$S(P_c) = [0.9836(0.00176)^2 + (0.00206)^2]^{1/2} = 0.00270$$

TABLE II. Systematic standard deviations.

<u>Sample Power</u>	<u>S(Cal)</u>
1.0	0.00166
2.0	.00169
3.0	.00174
4.0	.00181
5.0	.00189
6.0	.00199
7.0	.00210
8.0	.00222
9.0	.00234
10.0	.00248
11.0	.00262
12.0	.00276
13.0	.00292
14.0	.00307
15.0	.00373
16.0	.00339
17.0	.00355
18.0	.00371
19.0	.00387
20.0	.00405
21.0	.00422
22.0	.00439
23.0	.00456
24.0	.00473

C. Bias Correction

The final values for the slope and intercept given in Eq. 5 were incorporated in the microprocessor program to remove the measurement bias. The calibration was verified by repeating measurements on the standard heat sources. These measurements are summarized in Table III.

The average difference between the measured and the expected value was - 0.0014 watts, and the t-test showed that the difference is not statistically significant. The variance $S_D^2 = S_r^2 + S_0^2$, where S_0^2 is the variance in the standards and S_r^2 is the random error variance. Since the variance in the standards is much smaller than the measurement variance, S_D^2 is a good estimate of the random error variance. When the earlier estimates, based on 9 measurements of the same source and nine heat distribution error measurements, are pooled with the above, an average value for S_r of 0.0094 is obtained. This was rounded to 0.01 watts, which agrees very well with the average value printed out by the prediction program for the error in the measured power.

II. HEAT DISTRIBUTION ERROR

Heat-distribution error measurements were made with a 2-watt heat source at the top, center, and bottom of the sample canister on the centerline and on the canister wall. The analysis-of-variance F-test for means was used to evaluate the data shown in Table IV.

TABLE III. Standard heat source measurements.

Measured Power	Expected Power	
P_S	A	$P_S - A$
5.7726 ± 0.0044	5.7804	-0.0078
$3.8354 \pm .0060$	3.8496	-0.0142
$1.9297 \pm .0038$	1.9308	-0.0011
$5.7795 \pm .0082$	5.7803	-0.0008
$1.9331 \pm .0071$	1.9308	0.0023
$3.8573 \pm .0329$	3.8495	0.0078
$5.7770 \pm .0029$	5.7803	-0.0033
$3.8452 \pm .0033$	3.8495	-0.0043
$1.9394 \pm .0075$	1.9307	0.0087

$$\bar{D} = -0.0014$$

$$S_D = 0.0072$$

$$S_{\bar{D}} = 0.0024$$

$$t = 0.583 \text{ (critical value} = 2.306)$$

TABLE IV. Heat distribution measurements.

	<u>Top</u>	<u>Center</u>	<u>Bottom</u>
Centerline	2.3435	2.3460	2.3230
Centerline	2.3484	2.3580	2.3415
Side	2.3381	2.3513	2.3386
$\bar{x}$	2.3433 ± 0.0057	2.3518 ± 0.0060	2.3344 ± 0.0099

The calculated F-ratio was 4.21 compared to the critical F-ratio of 9.55 at the 95% confidence level. The combined standard deviation of the nine measurements was 0.010 watts, compared to 0.011 for nine measurements of a source located in the center of the canister. It is concluded that the heat distribution error is not significant compared to the random error.

III. PREHEATING TESTS

A series of tests were made on identical 1.2-watt samples to determine the effect of preheating on measurement time. The test results are summarized in Table V. These results show that the time saved by preheating is approximately 50% for both prediction of equilibrium and equilibrium power measurements. The time required for prediction approaches a minimum value faster than the time for equilibrium. Therefore, when prediction is used, the first sample of the day need only be preheated for 30 min before insertion into the calorimeter. Longer preheat time will not be compensated for by the time saved in the calorimeter. Higher powered samples could be preheated faster due to the self-heating of the plutonium.

The calorimeter power is shown as a function of time in the composite plot shown in Fig. 4 for the preheat times given in Table IV. The prediction does not occur until the power has fallen to within 99.8% of its final value. Therefore, the faster the power approaches equilibrium, the shorter the measurement time when prediction is used.

Fig. 4. Effect of preheat time on prediction.

TABLE V. Effect of preheating on measurement time.

Preheat		Prediction		Equilibrium	
<u>Time</u>	<u>Minutes</u>	<u>Time</u>	<u>% Reduction</u>	<u>Time</u>	<u>% Reduction</u>
0		143		210	
30		101	29%	204	3%
60		86	40	182	13
105		83	42	128	40
168		80	44	114	46
320		74	48	106	49

IV. MEASUREMENT TIME

The measurement time was recorded for 28 sample measurements with varying sample power and sample packaging. The time for prediction of equilibrium ranged from 1 to 2.5 hours. The time to reach equilibrium ranged from 1.4 to 5.4 hours. The time for the baseline measurement varied from 0.4 to 1.3 hours. The equilibrium value for the baseline power was obtained before a prediction could be made in 23 of the 28 cases. This data is summarized in Table VI.

If prediction of equilibrium is used, the average time for a sample measurement, including the baseline measurement, is 130 min. or 2.17 hours. Measurement of three samples a day, including a 30-min preheat for the first sample, would take 7 hours. It has been demonstrated that four samples can be measured in one day, starting with room-temperature samples. The first sample was preheated for 30 min, and the other three samples were preheated in the two preheaters while the measurements were made. The measurement time, including baseline, was:

Sample 1	150.0 min (including 30-min preheat)
2	121.5 min
3	118.5 "
4	108.0 "
Total	498 min = 8.3 hours

The average time saved by the prediction of equilibrium program is 48 min/sample, or 27%.

TABLE VI. Summary of measurement-time data.

	Sample		P_0 no prediction	P_0	
	<u>Prediction</u>	<u>Equilibrium</u>	<u>Equilibrium</u>	<u>Prediction</u>	<u>Equilibrium</u>
N	28	28	23	5	5
Min.	1.1 hours	1.4 hours	0.4 hours	0.95 hours	1.4 hours
Ave.	1.5 "	2.3 "	0.65 "	0.95 "	1.2 "
Max.	2.5 "	5.4 "	1.0 "	0.95 "	1.3 "
S	0.3 "	0.8 "	0.16 "	0 "	0.05 "

A paired-difference test of the prediction and equilibrium values of measurements covering the range of the calorimeter showed no statistically significant difference. This data is summarized in Table VII.

V. ELECTRICAL CALIBRATION

There is a three-step procedure for verifying the calibration of the electrical measurement functions of the calorimeter. The first step is calibration of the analog-to-digital converter (ADC) in the data-acquisition system (DAS). The slope and zero were checked frequently during the calibration and testing of the instrument. No drift in the slope or zero was observed, and no recalibration was required. An example of the ADC test data is shown in Table VIII.

The sample chamber is provided with a calibration winding so that the response of the power measurement circuits can be verified. The microprocessor program has a calibration subroutine that measures the response at six points, covering the range of the instrument. A linear least-squares program calculates the slope and intercept. An example of the calibration test program data is given in Table IX. This program checks the linearity of the sample chamber power measurement circuits.

While this program is running, the voltage and current to the calibration coil can be measured with an external digital multimeter (DMM) at test points on the front panel of the calibration module. Data taken with the 4-1/2 digit DMM that is being supplied with the

TABLE VII. Paired-difference test.

<u>Equilibrium</u>	<u>Prediction</u>	<u>Difference</u>
0.3593	0.3593	0
1.4267	1.4297	+0.0030
2.1443	2.1443	0
2.8647	2.8687	+0.0040
3.5804	3.5804	0
4.2954	4.3044	+0.0090
5.0123	5.0313	+0.0190
5.7311	5.7311	0
6.6289	6.6269	-0.0020
12.465	12.488	+0.0230
5.8364	5.8364	0
3.882	3.878	-0.0040
1.9412	1.9372	-0.0040
17.686	17.684	-0.002
20.035	20.034	-0.001
22.407	22.405	-0.002
Std dev. of mean diff (S_D)		
	0.0020	
Std dev. of single diff. (S_D)		
	0.0079	
Mean diff. ($\bar{D}$)		
	0.0027	
Test statistics $t =$		
	1.3686	(critical value 2.131 95% C.L.)
Deg. freedom		
	15	
N		
	16	

TABLE VIII. ADC test.

<u>DVM</u>	<u>ADC</u>	<u>Least-Squares-Fit</u>
0.0000	0.0000	$a_0 = -0.00036$
1.9647	1.9643	$a_1 = 1.000070$
4.0002	4.0001	$r^2 = 0.99999999$
6.0013	6.0016	$S_{yx} = 0.000316$
8.0011	8.0016	$S_0 = 0.00033$
9.9895	9.9895	$S_1 = 0.000050$

TABLE IX. Calibration test program.

<u>Slope</u>	<u>Intercept</u>	<u>Baseline</u>	<u>Comment</u>
-1.0004	24.951	24.953	6/17/81
-1.0003	24.975	24.975	7/8/81
-1.0000	24.977	24.980	7/22/81
-1.0002	24.982	24.982	8/7/81
-1.0000	24.748	24.749	9/30/81 after bias correction
-1.0004	24.785	24.785	10/8/81

calorimeter is given in Table X. This data provides a slope that can be used as a basis for determining if the instrument calibration has changed.

The applied power is equal to

$$P = E_V \times \frac{E_I}{R}$$

The slope determined by the least-squares-fit to the data in Table X gives the value of R. A t-test indicates that the intercept is not statistically significant, so that it is effectively equal to zero.

The "R" determined by this data includes the normalizing constant used to remove the bias determined by the heat standard calibration. Any change in the instrument calibration will be detected by repeating the electrical measurements, using "R" to calculate the power and testing to see if the data gives a slope of "1" and an intercept of zero within the measurement precision.

TABLE X. IAEA Bulk Calorimeter calibration. E_V and E_I measurements made with Keithley-135 DMM.

$\underline{E_V}$	x	$\underline{E_I}$	=	$\underline{E_V \times E_I}$	$\underline{P_{DAS}}$
9.214		0.4544		4.187	4.2884
12.852		.6335		8.142	8.3483
15.739		.7757		12.209	12.518
18.225		.8981		16.368	16.782
20.43		1.0065		20.563	21.083

Least-Squares-Fit

$$a_0 = 0.0032$$

$$a_1 = 0.97514$$

$$r^2 = 0.99999994$$

$$S_{yx} = 0.00180$$

$$S_0 = 0.00188$$

$$S_1 = 0.000135$$

REFERENCES

1. C. L. Fellers and P. W. Seabaugh, "Real-Time Prediction of Calorimeter Equilibrium," Nuclear Instruments and Methods 163, 499-505, North Holland Publishing Co., (1979).
2. "American National Standard Calibration Technique for the Calorimetric Assay of Pu-Bearing Solids Applied to Nuclear Materials Control," American National Standard AINSI N15.22-1975, American National Standards Institute, New York (1975).
3. R. Gunnink, "Determination of Plutonium Isotopic Abundances by Gamma-Ray Spectroscopy," UCRL-52879, March 1980.

PARTS LIST AND SUPPLIERS

Only parts that may be difficult to obtain are listed.

PARTSSUPPLIER

Printer, Bowmar Model TP3120
Printhead, Thermal 220722-1
Printer paper, 2-1/4 wide x 2" diameter,
#710-849-1

Bowmar Instrument Co.
4640 - 126th Ave. North
Clearwater, FL 33520

Analog-to-Digital Converter AD7550BD
Digital-to-Analog Converter AD7522KN
Multiplexer AD7506JD

Analog Devices
Route One Industrial Park
P.O. Box 280
Norwood, MA 02062

Display, LCD Hamlin 3918-312

Hamlin, Inc.
Lake & Grove Streets
Lake Mills, WI 53551

APPENDIX A

Circuit Diagrams for Bulk-Assay Calorimeter

Sheets 1 through 12

Notes :

- ① The two innermost cylinders have had threads cut in them (see detail below). The copper heater coils and nickel sensing coils were then wound in the grooves. Finally some .081" dia aluminum wire was wound over the grooves, sealing the coils inside, to keep epoxy from flowing onto them during assembly. The center cylinder has two coils wound with 30ga copper wire and two coils wound with 36ga nickel wire. The resistances of the copper coils are 18.7 Ω and 18.7 Ω . The nickel coils are 1.59K Ω and 1.58K Ω . The second cylinder has one coil wound with 30ga copper wire and two coils wound with 36ga nickel wire. The copper coil is 20.8 Ω and the nickel coils are 1401 Ω and 1404 Ω . Each cylinder has five Fenwall UUA33J1 thermistors imbedded in the cylinder wall and wired in series. Each cylinder also has a YSI temperature probe imbedded in the wall for temperature measurement.
- ② The third and fourth cylinders from the center are wound with teflon coated 32 ga hookup wire. The resistance of the third coil is 41.4 Ω , and the fourth coil measures 63.7 Ω . The third coil has nine Fenwall UUA33J1 thermistors epoxied to the inside wall. These are wired in series with a tap after every third one. The fourth coil has twelve Fenwall UUA33J1 thermistors epoxied to the inside wall. These are wired in series with a tap after every fourth one. Both coils have a YSI temperature probe imbedded in the wall for temperature measurement.
- ③ The fifth (outermost) cylinder has no coil wound on it. There are twelve Fenwall thermistors wired in series and epoxied to the inside wall. There is a tap after every fourth thermistor giving three legs of four thermistors each. Each leg consists of three GB32P2 (1000 Ω) thermistors and one GB31P2 (1000 Ω) thermistor.

Notes (cont.):

- ④ The outer cylinder has 37 fins made from 1'-1" right angle aluminum stock. These fins are to increase the heat dissipation from this cylinder.
- ⑤ A styrofoam plug is to slip fit into the plexiglass tube to insulate the sample chamber during operation.

Key

- Aluminum
- Epoxy
- Styrofoam
- Plexiglass

TOLERANCES: UNLESS OTHERWISE NOTED		APPROVED FOR DESIGN		ARGONNE NATIONAL LABORATORY		DATE		REV	
FRACTIONAL VOL. ± 1/10	1/10	DATE		This drawing is the property of		Bulk Calorimeter			
DECIMAL VOL. ± 0.005	0.005	APPROVED FOR MANUFACTURE		The Argonne National Laboratory		Calorimeter Sample Chamber			
ANGULAR VOL. ± 1/10	1/10	DATE		SCALE		1:2			
APPROVED BY AN ARGONNE LABORATORY		DATE		APPROVED BY AN ARGONNE LABORATORY		DATE		REV

IAEA Bulk Calorimeter Block Diagram

REVISIONS

DATE

BY

FOR

REVISIONS

ARS/ENGINE NATIONAL LABORATORY ENGINEERING NOTE		DIVISION	PROJECT	FILE NO.	PAGE
SUBJECT		NAME			
Bulk Calorimeter T ₁ Control Module		DATE

J1 CONNECTOR - BULK CALORIMETER

<u>Pin</u>	<u>Color</u>	<u>From</u>	<u>Description</u>	<u>Test Point</u>
1	N/C	--		(8)
2	Brown	T2 - 22	Sum (T2)	7
3	Gray	T2 - 24	T2 E (Heater voltage)	6
4	N/C	--		(5)
5	White	Cal - 25	Cal I (Heater current)	4
6	Gray	Cal - 24	Cal E (Heater voltage)	3
7	White	T3 - 25	T3 I (Heater current)	2
8	Gray	T3 - 24	T3 E (Heater voltage)	1
9	N/C	--		(9)
10	Brown	T1 - 22	Sum (T1)	10
11	N/C	--		(11)
12	Green(18)	Aux - 24	Aux +15 V for DAS (Com)	--
13	Black		Power supply ground	--
14	Red	+15 V	Power supply for DAS	--
15	Blue	-15 V	Power supply for DAS	--
16	Green	5 V Mod - 10	+5 V to DAS	--
17	Violet	5 V Mod - 14	+5 V sense wire	--
18	Gray	5 V Mod - 13	5 V Com. sense wire	--
19	White	5 V Mod - 3	5 V Com. to DAS	--
20	Red (18)	Aux - 22	Aux +15 V for DAS (Pos.)	--
21	N/C	--		(12)
22	Red	+15/2	Pos. from T3 & T2 preamp	13
23	Blue	-15/2	Neg. located in coil assy	14
24	Brown	T3 - 22	Sum	15
25	N/C	--		(16)

J2 CONNECTOR - BULK CALORIMETER

<u>Pin</u>	<u>Color</u>	<u>From</u>	<u>Description</u>	<u>Test Point</u>
1	Black	T3 - 35	Mea. gnd (also to Cal - 35)	--
2	Orange	Board	T2 Power amp input	25
3	N/C	--		(17)
4	N/C	--		(18)
5	N/C	--		(19)
6	N/C	--		(20)
7	Orange	Ts - 20	Ts Fine	21
8	Yellow	Ts - 21	Ts Coarse	22
9	Yellow	Board	T3 Power amp input	23
10	Yellow	Board	T3 Power amp output	24
11	Gray (2)	5 V Mod - 13	5 V Com. sense wire	--
12	Violet (2)	" - 14	+5 V Sense wire	--
13	Green (2)	" - 10	+5 V to DAS	--
14	White	Cal 2	A/D calibrator V adj.	32
15	Red	Cal 1	A/D calibrator +10 V ref	31
16	N/C	--		(30)
17	N/C	--		(29)
18	Red	Board	T1 Pwr amp output	28
19	Red	Board	T1 Pwr amp input	27
20	Orange	Board	T2 Pwr amp output	26
21	Violet	Cal 22	Drive fr DAS to calibrator	--
22	Blue (2)	-15 V	Power supply for DAS	--
23	Red (2)	+15 V	Power supply for DAS	--
24	White (2)	5 V Mod - 3	5 V Com to DAS	--
25	N/C	--		--

J3 CONNECTOR - BULK CALORIMETER

<u>Pin</u>	<u>Color</u>	<u>To</u>	<u>Description</u>
A	Orange	Ts - 12	Ts Sense thermistors
B	Blue (18)	--	
C		--	
D	Yellow	Ts - 13	
E	N/C	--	To TB heater
F	Black (18)	-15 V	
H	Orange	TB - 12	TB Sense thermistors
J	N/C	--	
K	N/C	--	
L	Yellow	TB - 13	
M	Red (18)	T1	Power amp output T1 Heater
N	Black (18)	-15 V	
P	Orange	T1 - 12	T1 Sense thermistors
R	N/C	--	
S	N/C	--	
T	Yellow	T1 - 13	
U	Orange (18)	T2	Power amp output T2 Heater
V	Black (18)	T2 - 26	
W	Blue	T2 - 18	T2 Heater voltage sense
X	Blue	T2 - 19	
Y	Violet	Ts - 26	Drive to Ts crydom [relay (S.S.)]
Z	White	Ts - 11	
a	Yellow (18)	T3 - 26	Power amp output T3 Heater
b	Yellow (18)	T3	
c	Green	T3 - 18	T3 Heater voltage sense
d	Green	T3 - 19	
e	Green (18)	Cal - 13	(Output fr cal power amp Cal heater goes to Cal-3 to connect to relay)
f	Green (18)	Cal - 26	
h	White	Cal - 18	Cal heater voltage sense
j	White	Cal - 19	
k	N/C		
l	N/C		
m	Yellow	T2 - 21	
n	Orange	T2 - 20	
p	Black (18)	Loose	
r	Yellow	T3 - 21	
s	Orange	T3 - 20	

<u>Pin</u>	<u>Color</u>	<u>To</u>	<u>Description</u>
t	Black (18)	T3 - 42	Preamp ground reference
u	Red	+15	from preamp
v	Blue	-15	from preamp
w	Red	Ts - YSI	
x	Black		
y	Green (18)	YSI Common	
z	Violet	TB - YSI	
AA	Black		
BB	White	T1 - YSI	
CC	Black		
DD	Orange	T2 - YSI	
EE	Black		
FF	Red	T3 - YSI	
HH	Black		
JJ	Black (18)	Loose	
KK	N/C		
LL	N/C		
MM	N/C		
NN	N/C		

APPENDIX B

Schematics for Data Acquisition System

Sheets 1 through 7

DRAWING NO.		ISSUE
EL -		
SHEET	OF	

Y1 - 1, 9, 17, 25
Y2 - 2, 10, 18, 33
Y3 - 3, 4, 26, 34
Y4 - 11, 12, 19, 20
Y5 - 27, 28, 35, 36

X1 - 8, 14, 30, 31, 48
X2 - 7, 13, 16, 32, 47
X3 - 6, 15, 22, 24, 46
X4 - 5, 21, 23, 32, 37

A 38 RST
B 41 EXT
C 42 BS
D 45 DP
E 49 ENTR
F PAPER ADV
GND 39, 40, 43, 44, 50, PAPER ADV.

E/K	J/M	←/O	7/8	%&
INIT	RUN	P.	SP	FRAC
3/J	✓/L	↑/N	+/#	7/%
YES		PRNT	CAL	TP
U/E	V/F	W/G	X/H	Y/I
5	6	7	8	9
P/=	9/A	R/B	S/C	T/D
L	1	2	3	4
		NUMB.	SHIFT	SPACE
RST	EXT	BS	.	ENTR

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ELECTRONICS DIVISION ARGONNE NATIONAL LABORATORY THIS DRAWING IS THE PROPERTY OF THE ARGONNE NATIONAL LABORATORY	TITLE			DRAWING NO.	
	KEYBOARD LAYOUT AND CONNECTIONS			EL -	
	DESIGN BY			DRN. BY	DATE
	BUILT FOR			CHK. BY	APPD. BY
			SHEET 1 OF 7		MODEL NO.
					SYSTEM NO.

DRAWING NO. EL -		ISSUE
SHEET	OF

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ELECTRONICS DIVISION	TITLE KEYBOARD DECODE		DRAWING NO. EL -	
	ARGONNE NATIONAL LABORATORY THIS DRAWING IS THE PROPERTY OF THE ARGONNE NATIONAL LABORATORY		SHEET 2 OF 7	
DESIGN BY	DRN. BY	DATE		MODEL NO.
BUILT FOR	DIV.	CHK. BY	APPD. BY	SYSTEM NO.