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TEST AND EVALUATION OF THE ARGONNE BPAC10 SERIES AIR CHAMBER CALORIMETER DESIGNED FOR 20 MINUTE MEASUREMENTS

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Ronald B. Perry, Sidney Fiarman, Erwin A. Jung, and Teresa Cremers

ABSTRACT

This paper is the final report on DOE-OSS Task ANLE88002 "Fast Air Chamber Calorimetry". The task objective was to design, construct, and test an isothermal air chamber calorimeter for plutonium assay of bulk samples that would meet the following requirements for sample power measurement:

Average sample measurement time less than 20 minutes.
Measurement of samples with power output up to 10 Watts.
Precision of better than 1% RSD for sample power greater than 1 watt.
Precision better than 0.010 watt SD, for sample power less than 1 watt.

This report gives a description of the calorimeter hardware and software and discusses the test results. The instrument operating procedure, included as an appendix, gives examples of typical input/output and explains the menu driven software.

Sample measurement time of less than 20 minutes was attained by pre-equilibration of the samples in low cost precision preheaters and by prediction of equilibrium measurements. Tests at the TA55 Plutonium Facility at Los Alamos National Laboratory, on typical samples, indicates that the instrument meets all the measurement requirements.

I. INTRODUCTION

Calorimetric assay combined with gamma-ray isotopic assay has been developed into a highly precise and accurate NDA (nondestructive assay) method for plutonium safeguards measurements. Real progress has been made in reducing measurement time for gamma-ray isotopic measurements without loss of precision and accuracy. Reduction of calorimetric assay measurement time has been recognized as a critical 'User Need'. Satisfying this need could broaden the application of calorimetry to samples that are currently measured by less accurate, faster methods. Calorimeters currently in use for measurement of bulk quantities of plutonium bearing materials typically require measurement times of 2 to 6 hours or longer. Increasing the number of samples measured by calorimetry by procuring multiple units is a costly approach and does not reduce the unit cost of the measurement. A cost effective approach to satisfying this 'User Need' is development of an instrument with higher throughput.

The isothermal, air chamber, calorimeter system designed for meeting this need incorporates prediction of equilibrium with very precise preheating to reduce the measurement time. Samples are pre-equilibrated simultaneously in multiple low cost preheaters and measured in a calorimeter chamber that has a very short time constant. Since the calorimeter is an isothermal type, all of the samples can be precisely preheated to the calorimeter operating temperature reguardless of sample power output. Average measurement time is less than 20 minutes for samples preheated overnight.

The long sample time constant has essentially been eliminated by pre-equilibration in the preheater. Tests with a few samples, preheated for 6 hours, demonstrated that a second batch of samples could be measured with one set of preheaters during the same shift. Up to eight preheaters have been utilized with one BPAC10 Series calorimeter.

The instrument described in this report (BPAC10C) was designed and constructed under DOE-OSS Task ANLE88002 and has been installed at LANL TA55 Plutonium Facility. Responsibility for test and evaluation of BPAC10C has been assumed by LANL's Nuclear Materials Measurement & Accountability Group. Two additional instruments (BPAC10A and BPAC10B) were purchased by EG&G Rocky Flats Plant, and have been installed in that facility. Test and evaluation of BPAC10A and B will be the subject of a separate report.

The BPAC10 system, shown in Figure 1, measures the thermal power emitted by plutonium-containing samples with sample power up to 10 watts. The instrument package consists of the calorimeter measurement chamber (CMC), instrument rack, and 5 sample preheating chambers (SPC). Five sample canisters for transfering the samples from the SPC to the CMC are also included. The sample canister is designed to accept samples contained in an 8802 Vollrath can or any sample container up to 4.976" in diameter and up to 7" tall. BPAC10C operates on 115 V, 60 Hz AC and the system, including preheaters, can be operated on a 20 amp circuit.

II. SYSTEM DESCRIPTION

A. Calorimeter Measurement Chamber

The calorimeter measurement chamber consists of five temperature controlled concentric aluminum cylinders separated by epoxy. The aluminum cylinders are essentially isothermal elements separated by a thermal resistance that allows their temperature to be independently controlled. Styrofoam insulation at the ends of the inner cylinders limit axial heat flow so heat flow from the inner cylinder is primarily radial. The steady state, empty chamber, design temperatures of the cylinders are shown in Table I.

Table I
Design Temperatures

T3 = 45.4°C +/- 0.1
T2 = 41.2°C +/- 0.1
T1 = 38.5°C +/- 0.2
T0 = 34.4°C +/- 0.2
TB = 31.5°C +/- 0.4

Heat flow is radially outward with the room acting as an infinite heat sink. Heat is supplied to the T0, T1, T2,and T3 cylinders through DC temperature controllers. The outer cylinder, TB, is controlled by an AC temperature controller located in the base of the CMC. Heat is exhausted to the room by drawing air in through a series of holes around the top of the CMC cover with 4 fans located in the base. Most of the power needed to raise the temperature of the CMC to operating temperature is supplied by the AC heater. A temperature limit switch in series with the AC heater protects the outer cylinder, TB, from overheating if the AC controller malfunctions. Components contained in the CMC include:

1. Four 48 volt DC cooling fans.
2. Bridge circuits and preamplifiers for T2 and T3.
3. Dowty Model 71A AC temperature controller for TB.
4. Power indicator lamps and power relays.
5. AC voltmeter to monitor voltage to the TB heater.

B. Instrument Rack

A layout drawing of the components in the instrument rack is shown in Figure 2. The NIM (Nuclear Instrumention Module) bin containing the temperature control modules has custom wired connectors that DO NOT conform to NIM Standard TID-20893. Every connector in the NIM bin is wired for a specific module. A special keying arrangement prevents interchanging modules or insertion of standard NIM modules. The temperature control modules have front-panel meters that show the status of the various servo-circuits. When the system reaches operating status, all temperature error meters (T0, T1, T2, and T3) will read zero. Volt meters on the T0, T1, T2, T3, and calibration modules indicate actual voltage applied to the heaters.

The following components are contained in the NIM bin:

1. T0, T1, T2, and T3 temperature controllers.
2. Electrical Calibration module.
3. YSI (Yellow Springs Instrument) linear thermistor temperature readout module.

C. Temperature Controllers

Temperature control of the 4 inner cylinders is accomplished with a digital control system that is operated by the system computer. The PID (proportional, integral, differential) control algorithm receives temperature error data from an on board ADC (analog to digital converter). This multi-port ADC, operates in a fast 16 bit mode or a slower, averaging, 18 bit mode. The mode is software selectable and both modes are used by the program. Each controller is contained in a separate NIM type module.

Temperature sense elements for T2 and T3 are continuous nickel wire windings wound the full length of the cylinders. The sense coil is configured as 1 arm of a bridge circuit. The bridge reference resistors and bridge voltage source are maintained at constant temperature by locating these elements and the associated preamplifiers inside the calorimeter. The preamps provide an amplified bridge error voltage to the controller and error meter circuits in the NIM modules.

A series string of 21 thermistors is used as the temperature sense element for the T0 and T1 cylinder. All the control circuits for T0 and T1 temperature control are located in their control modules. The large temperature coefficient of thermistors (5 to 10 times larger than nickel wire) makes temperature control of these bridge circuits unnecessary.

Digital information from the control algorithm is applied to the heater power amplifiers, located in each control module, through 18 bit DACs (digital to analog converter).

D. Data Acquisition System

The BPAC10C calorimeter has a computer-based data acquisition and temperature control system. The system computer is a rack-mounted IBM-AT compatible consisting of:

1. Micronics 386 mother board with an 80386, 33 MHz microprocessor and 32K fast cache RAM.
2. 80387 math coprocessor.
3. Seagate ST277N 60 MB hard disk drive with self parking heads as C drive.
4. 1.44 MB 3-1/2" floppy drive as B drive.
5. 1.2 MB 5-1/4" floppy drive as A drive.
6. VGA monitor driven by a Paradise VGA Plus 16 card.
7. Strawberry Tree ACPC 16/16 ADC and digital I/O (Input/Output) card.
8. MS-DOS 4.01 operating system.

The Strawberry Tree card is used for all voltage measurements associated with sample power measurements, temperature control, temperature measurement, and electrical calibration. The ADC provides precision and accuracy equivalent to a 5-1/2 digit DVM (digital volt meter). In addition, the Strawberry card also provides 16 bits of digital I/O used to communicate to each of the controller modules and the YSI module. Menu-driven software written in Borland's Turbo Pascal version 5.0 language provides an operator interface to the calorimeter through the keyboard. Output data is sent to the built-in 14" VGA color monitor and can optionally be sent to a Panasonic KX-P1091i dot matrix printer for hard copy output and/or to the 3-1/2" floppy disk drive for archival storage or off-line analysis. All software is on hard disk and backup software is provided on 5-1/4" floppy disk.

E. Sample Preheating Chamber

Five SPCs are included as part of the calorimeter system to increase the sample throughput. A preheater is shown in Figure 3. Each SPC has an AC temperature controller (Dowty Model 72A), digital temperature meter (Omega Model 400B), and top panel, operator adjustable, temperature control. Observed performance parameters are:

1. Optimum operating temperature 45.40°C.
2. Precision +/- 0.02°C.
3. Warm-up time 30 minutes.
4. Temperature adjustment 0.4°C/turn.

Operating temperature of the SPC is set to 45.40°C for optimum performance with the calorimeter. The temperature must be set with the chamber empty to prevent the linear thermistor, connected to the meter, from being influenced by the presence of a heat-producing sample. Rotation of the temperature control clockwise increases the temperature approximately 0.4°C for each turn. A series of control thermistors supply average wall temperature information to the controller. To prevent overheating if the controller malfunctions, a temperature limit switch opens at approximately 55°C and unlatches a relay in series with the line voltage. To restart the SPC after shutdown due to overheating, the reset button must be pressed to latch the relay.

F. Software

BPAC10C software is written in Borland's Turbo Pascal version 5.0, a high level language that has a fast compiler and a debugging program to facilitate program development. The system software package is menu driven and "user friendly". A description of the menu selections is given in Appendix B.

The software runs in a multi-tasking environment and consists of three separate tasks (or windows). The tasks run simultaneously under Quarterdeck DESQview 386, Version 2.26, including QEMM 5.0 (Quarterdeck Extended Memory Manager). The three tasks are described as follows:

1. The I/O Task. This task runs the program BPAC-IO whose sole function is to be concerned with the housekeeping chores of analog and digital I/O. This task is a "service task" to the control and DAS tasks. Most of its time is spent reading the ADC and supplying voltage, power, and temperature data to the other tasks. It also includes diagnostic display of ADC and DAC functions.

2. The Control Task. This task runs the BPAC-CTL program whose function is to perform the PID control of the T0...T3 cylinders. The program receives error, temperature, and power data from the I/O task. It returns digital control correction information to be applied to the specific control modules based on the control algorithm.

3. The DAS Task. This task runs the BPAC-DAS program whose features include the following functions:

   a. Prediction of equilibrium for sample and base line measurements.
   b. Optional equilibrium measurement following prediction.
   c. Decay of sample isotopic data.
   d. Calculation of sample effective specific power (ESP).
   e. Calculation of sample power.
   f. Sample plutonium content calculation.
   g. Propagation of error calculation.
   h. Automated electrical calibration check.
   i. Diagnostic display of ADC and DAC functions.
   j. Update of calibration parameters.
   k. Date, time, and ambient temperature with each measurement.

G. Printer

An 80 column impact dot matrix printer (Panasonic model KX-P1091i) is included with the calorimeter for hard copy output. The printer is in a drawer at the top of the instrument rack (see Figure 2.). Refer to the printer manual for instruction on installing paper, changing the ribbon cartridge, etc.

III. PERFORMANCE TESTS

A. Measurement Requirements

1. Sample power measurement range of 0.1 to 10 watts.

2. Measurement precision of 1% or better (1 σ) for 1 watt to 10 watts samples.

3. Measurement precision of 10 milliwatts or better (1 σ) for a heat range of 0.1 watt to 1 watt.

B. Heat Standard Calibration

BPAC10C was calibrated at LANL with certified heat standards at 17 points over the range of 0.0273 watts to 10.431 watts. Crumpled aluminum foil was used to prevent the heat sources from moving in the sample canister during transfer from the preheater to the measurement chamber. All standards were preheated overnight before measurement. A total of 46 data points, including replicate measurements, were used to determine a linear calibration function and calculate the associated random and systematic error using the method given by Jaech. (See Reference 3 on page 16 of Appendix.)

The calibration function is given in the following equation:

$$X = [Y - (-0.0098 \pm 0.0019)]/(0.99858 \pm 0.00041)$$

where: Y = Base line (PB) - Assay (PA).

The base line measurement is made with an empty preheated canister and the assay measurement is made with the preheated sample. Plots of typical assay and base line data are shown in Figures 3 and 4.

The random error (Jaech method) calculated from the calibration data is:

s(r) = 0.0100 watts.

The systematic error s(sys) is a function of the measured power and is given for selected values in Table II.

Table II. Systematic Error

Sample Power	s(sys)
0.0	0.0020
1.0	0.0017
5.0	0.0019
7.0	0.0026
10.0	0.0038

S(sys) is negligible compared to s(r) for low power samples and s(sys) does not increase the overall error significantly at 10 watts.

C. Performance Test Data

Performance tests of BPAC10C were made with typical samples encountered at the TA55 Plutonium Processing Facility at LANL. Sample measurements were compared to measurements made with the Facility's water-bath calorimeters. An empty container with similar packaging and preheating was also measured twice a day as a base line. All sample and base line measurements were made using the prediction of equilibrium algorithm. The performance test data is given in Table III. Measurement time shown in Table III does not include the time required for the base line measurement. Average base line measurement time was 17.2 minutes. Measurement time for the water-bath calorimeter was the time to first prediction, but the measurement data are the equilibrium values.

Table III
Comparison of Air Chamber and Water-Bath Calorimeter Measurements

Sample ID	Air Chamber Watts	Time Min	Water Bath Watts	Time Min	Diff Watts	% Diff
SWPPMB93	0.1553	21	0.1565	150	-0.0011	-0.73
PMA5118	0.2528	18	0.2529	132	-0.0001	-0.05
PMA1419	0.3356	17	0.3422	102	-0.0066	-1.93
JUEOX0123	0.3598	15	0.3505	162	0.0093	2.66
FFPFOX015	0.5055	16	0.4959	180	0.0096	1.94
DBSP545	0.5817	23	0.5886	180	-0.0069	-1.17
PMA8359	0.6632	24	0.6738	162	-0.0106	-1.58
"	0.6616	17	*	*	-0.0121	-1.80
XBLS32	0.8350	16	0.8291	150	0.0058	0.71
XBS2353	0.8796	15	0.8568	348	0.0228	2.66
STDSRP6-3	0.8407	15	0.8609	138	-0.0202	-2.35
XBSOX131	1.2066	18	1.2053	198	0.0013	0.11
MSE5RR	1.3022	17	1.2934	151	0.0088	0.68
"	1.2885	25	*	*	-0.0049	-0.38
"	1.2845	21	*	*	-0.0089	-0.69
PMA6364	1.5473	19	1.5536	180	-0.0063	-0.40
PMA8360	4.2748	17	4.2692	156	0.0056	0.13
JOF0026	4.3502	14	4.3181	308	0.0321	0.74
"	4.3043	24	4.3107	300	-0.0064	-0.15
"	4.3225	21	4.3109	300	0.0116	0.27
PMA1420	5.1755	19	5.1758	240	-0.0003	-0.01
PMA11030	5.6697	24	5.6538	198	0.0159	0.28
"	5.6624	15	*	*	0.0086	0.15
PMA3373	7.2891	20	7.2908	558	-0.0016	-0.02
RFM7203CP	9.8298	23	9.8120	300	0.0179	0.18
"	9.8305	28	*	*	0.0187	0.19
Average Time =		19		177		

AVE DIFF $\bar{D}$ = 0.0031
STD DEV S(D) = 0.0119
$S(D)/\sqrt{23}$ = 0.0025
t = 1.2400

* Measurements were repeated on the air chamber calorimeter only.

Data from Table III, for sample power less than 1 watt, are plotted in Figure 5. The data shows that the difference between air chamber and water-bath measurements are all less than 3 sigma (3 x 0.010). Data for sample power greater than 1 watt, plotted in Figure 6, shows that these measurements agree within the required 1% uncertainty. Comparison of time for prediction, for the samples shown in Table III, indicates that a factor of 9 improvement in average measurement time can be expected with the BPAC10C system.

The critical value of t (Student's t) for 26 degrees of freedom at the 95% confidence level is 2.056. Therefore, the difference of 0.003 watts in Table III is not statistically significant at the 95% confidence level.

The variance of the difference, V(D), is the sum of the variances $[s(D_1)]^2$ and $[s(D_2)]^2$ for the air chamber and water-bath measurements.

$$V(D) = [s(D)]^2 = [s(D_1)]^2 + [s(D_2)]^2$$
$$= (0.0119)^2 = (0.0100)^2 + [s(D_2)]^2$$

$$s(D_2) = 0.0064 \text{ watts.}$$

The average uncertainty for the water-bath calorimeter measurements calculated from the variance of the differences is in the range of estimates from replication of standards which shows that the uncertainty varies from 0.0018 at 1 watt to 0.0059 at 7 watts. Therefore, the observed errors are consistent with expected values for both calorimeters.

Table III includes repetitive measurements of 5 of the samples including low, medium and high power samples. The average standard deviation calculated from the repetitive measurements is 0.0078 watts or 0.29%.

The calorimeter exchange standard (CALEX) was measured 5 times to determine the precision of the measurement at less than 1 watt and to verify the accuracy of the calibration. The CALEX data is given in Table IV.

Table IV

Replicate Data For CALEX Heat Standard

MEASURED VALUE (WATTS)	EXPECTED VALUE (WATTS)	DIFF (WATTS)
0.9889	0.9838	0.0051
0.9870	0.9838	0.0032
0.9838	0.9839	-0.0001
0.9850	0.9839	0.0011
0.9807	0.9840	-0.0033

$\bar{D} = 0.0012$

$S(D) = 0.0032$

$S(D)/\sqrt{5} = 0.0014$

$t = 0.8571$

The critical value of t for 5 degrees of freedom at the 95% confidence level is 2.571. Therefore, the difference of 0.12% in Table IV is not statistically significant at the 95% confidence level. The standard deviation, 0.0032 watts, is less than the required value of 0.010 for sample power less than 1 watt.

IV. SYSTEM OPERATION

Set up and operation of BPAC10C is covered in the Detailed Procedures included as Appendix A.

V. CONCLUSIONS

The test and calibration data indicates that the calorimeter meets or exceeds the measurement requirements for measurement time and precision in all sample power ranges. The observed measurement errors are consistent with the error model used in the sample power propagation of error calculation given in section 4.1 of the Detailed Procedures in Appendix A. Comparison of measurements of typical samples in the air chamber calorimeter to measurements in the water-bath calorimeter verified that the systematic error due to the calibration function is negligible compared to the random error.

The throughput of the ANL BPAC10 Series Calorimeter has been demonstrated to be 2 to 3 measurements an hour with overnight preheating. Therefore, 10-12 samples in an 8 hour day should be possible including measuring the base line at the beginning of the day and repeating the base line measurement after 2-3 samples are measured. Additional preheaters will be required to obtain the maximum throughput, but preheaters can be added for less than $10K each. More experience will be required to determine the minimum sample preheating time for each type of sample. However, preliminary data gathered in this test indicates that preheaters emptied early in the shift can be refilled with samples for measurement near the end of the same 8 hour shift.

The precision, accuracy, and measurement time of BPAC10C is well matched to that of gamma-ray isotopic measurements that are typically used to determine the ESP (effective specific power) needed to calculate the plutonium content of the sample. In fact the gamma-ray isotopic measurements typically take longer than BPAC10C power measurements. For sample power greater than 1 watt, the measurement uncertainty of the ESP will usually be greater than or equal to the uncertainty of the BPAC10C power measurement.

A BPAC10 Series Calorimeter takes up much less laboratory space than the equivalent number of water-bath calorimeters that would be required to measure the same number of samples. The time required for measurement of measurement control samples each week has a much smaller impact on sample throughput for BPAC10 (1/50 compared to 1/8) which significantly improves productivity.

Addition of the BPAC10 Series Calorimeter to the array of NDA measurement equipment in use at a large plutonium facility can reduce the backlog of samples waiting for calorimetric assay. The instrument can also be used to measure some samples that are currently measured by faster (than water-bath calorimetry) methods such as neutron or gamma counting that are subject to matrix effects and interferances from other isotopes that can produce large biases. This should improve the uncertainty in the material balance and reduce the down time during physical inventory.

Figure 1.
BPAC10 Series Bulk Plutonium Assay Calorimeter

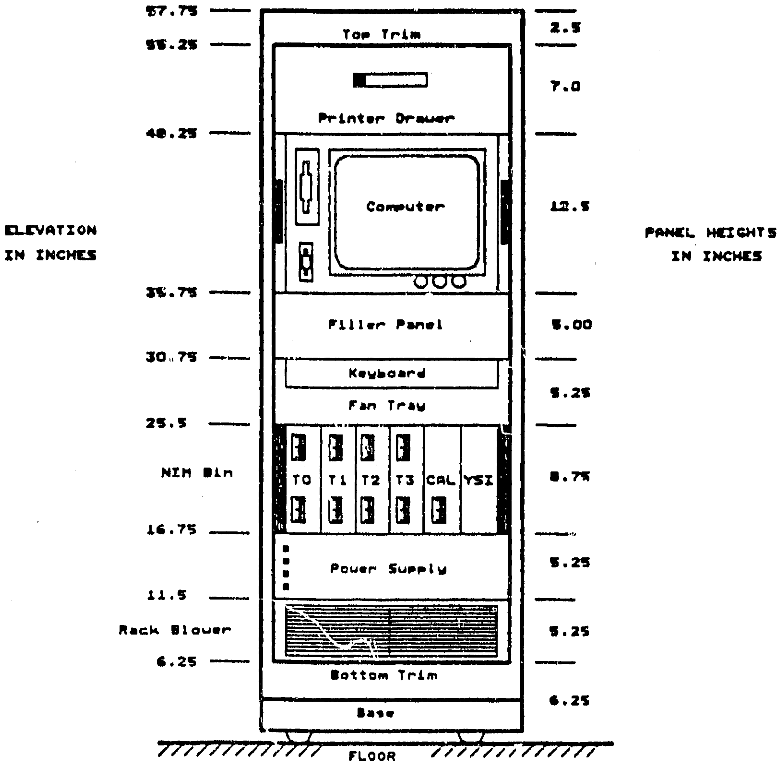

Figure 2.
Instrument Rack Layout

Figure 3.
Preheater

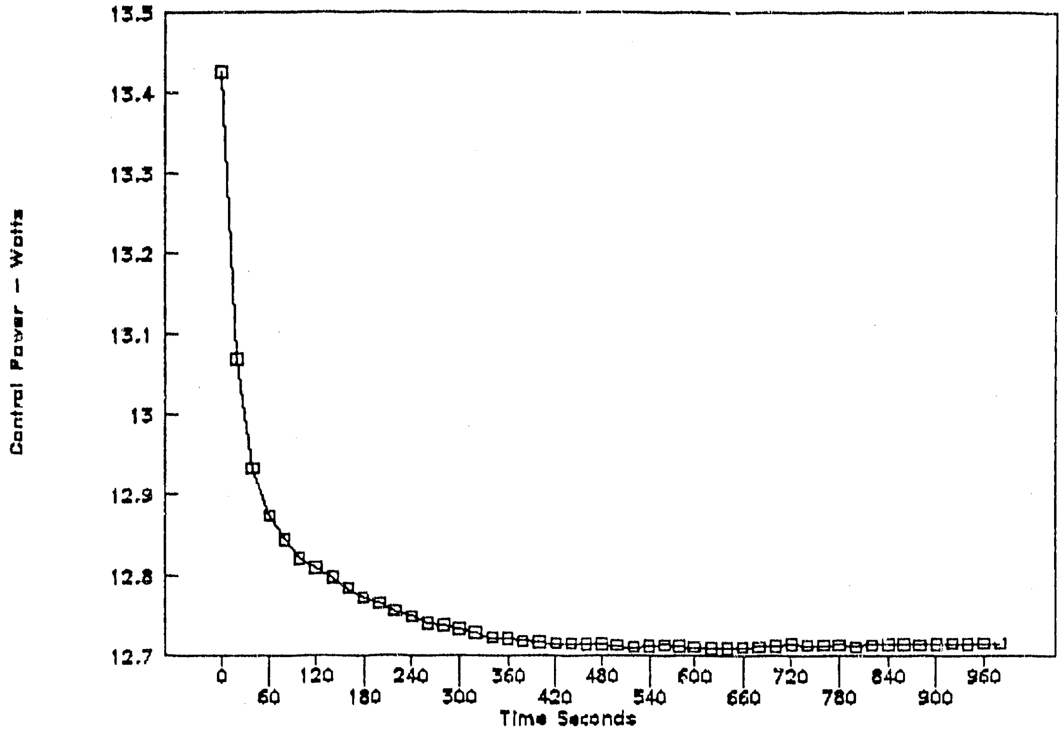

Figure 4.
BPAC10C Sample Assay Data

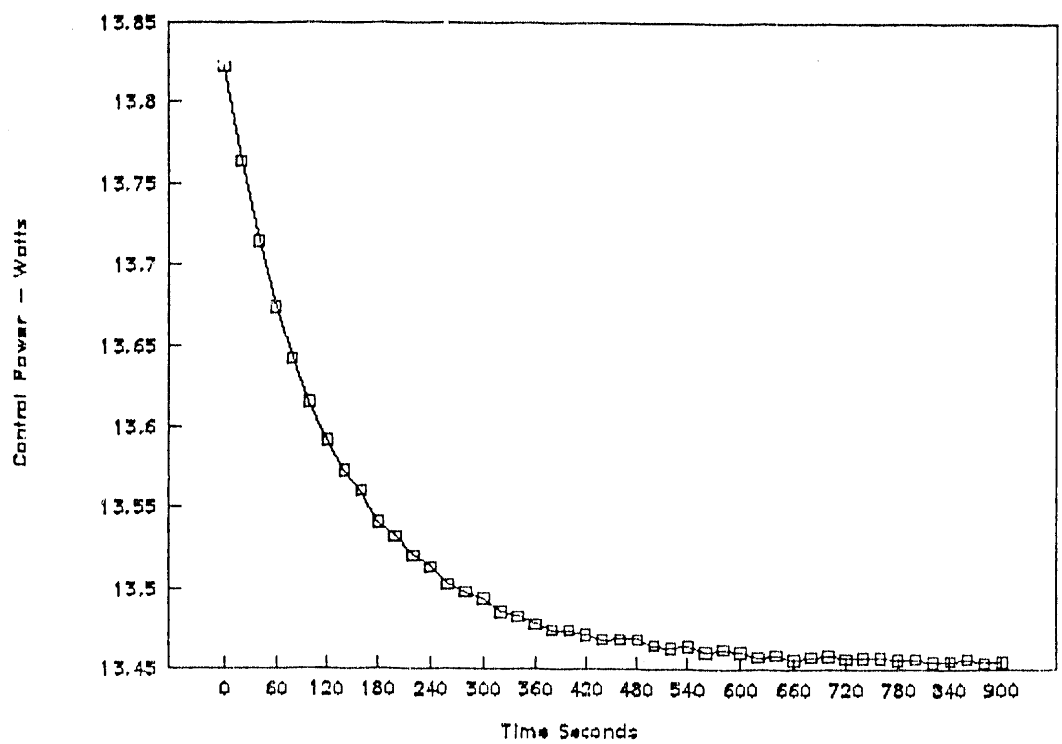

Figure 5.
BPAC10C Base Line Assay Data

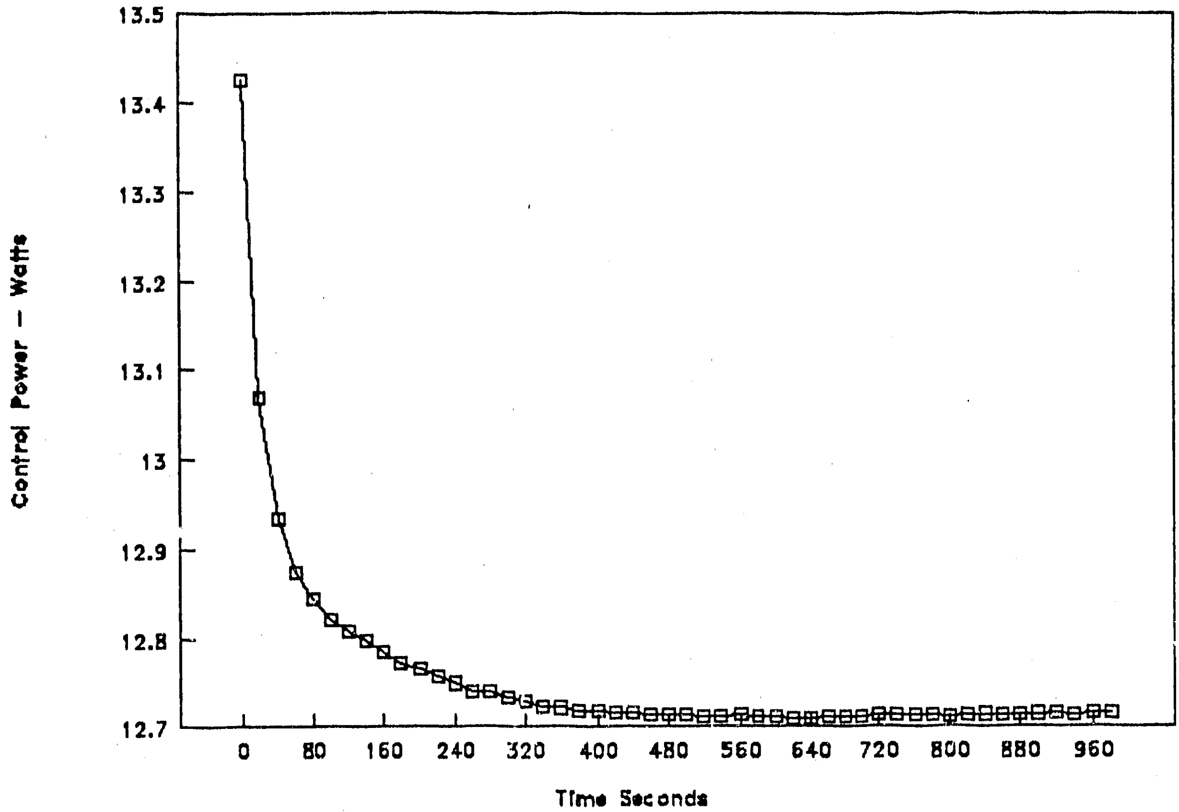

Figure 6.
Comparison of Measurements < 1 Watt

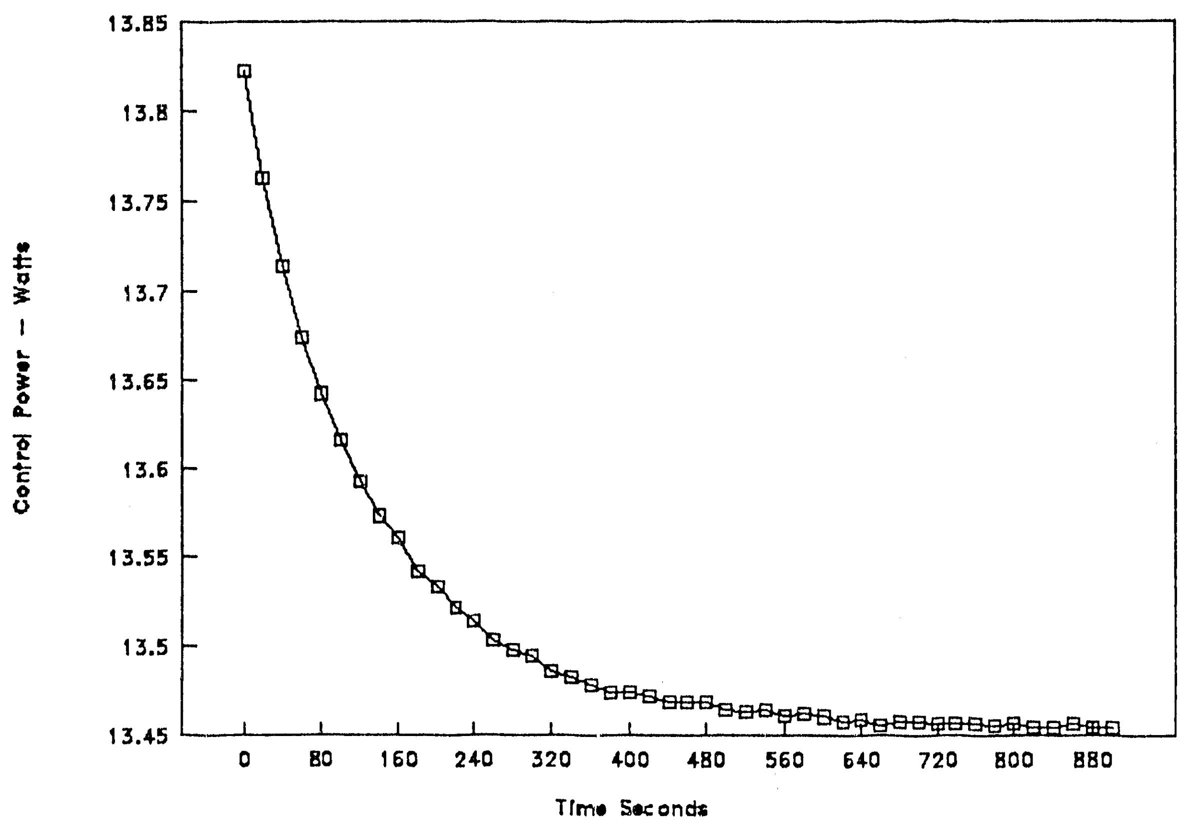

Figure 7.
Comparison of Measurements > 1 Watt

VI. ACKNOWLEDGMENTS

Individuals with expertise in various disciplines were required for the design, construction and testing of the calorimeter. Most notable of these are:

Chamber, cylinder and canister fabrication. .... ANL Central Shops

Coil construction and chamber assembly. .... G. T. Hicks, SSD/EL

Instrument rack construction. .... S. A. Borkowski, SSD/EL
J. A. Bulka, SSD/EL
R.G. Schuessler, SSD/EL

Printed circuit board CAD work. .... R. J. Deland, SSD/EL
G. T. Hicks, SSD/EL

Quality Assurance Program. .... R. J. Voogd, SSD/EL

VII. REFERENCES

[1] The Bulk Assay Calorimeter, Parts 1 and 2, ANL-NDA-9.

[2] ANSI N15.22-1987, "Calibration Techniques for the Calorimetric Assay of Plutonium-Bearing Solids Applied to a Nuclear Materials Control."

[3] Statistical Methods in Nuclear Materials Control by John L. Jaech, 1973, TID 26298.

APPENDICES

APPENDIX A

DETAILED PROCEDURE

APPENDIX A

DETAILED PROCEDURE

1.0 STATEMENT OF PURPOSE

1.1 Objective and Purpose of the Procedure

This procedure describes how to use the BPAC10C bulk calorimeter in combination with plutonium isotopic data or specific power data to determine the mass of plutonium in plutonium-containing materials.

1.2 Typical Measurement Performance

BPAC10C is capable of producing measurements with a precision of < 1% for sample power in the range 1 to 10 watts. In range 0.1 to 1 watt the precision is < 10 milliwatts. The above performance is based on tests using prediction of equilibrium and a variety of sample types normally encountered at TA55 and repetitive measurement of the CALEX sample. Measurement time for the test data was less than 20 minutes for samples preheated overnight.

2.0 SYSTEM REQUIREMENTS

2.1 Checklist of Required Equipment

Table V lists the components and accessories.

Table V. Bulk Calorimeter System Components and Accessories

Quantity	Description of Item	
1	Calorimeter Measurement Chamber (CMC)	____
1	Control Cabinet (CC)	____
5	Sample Preheating Chamber (SPC)	____
5	Sample Canisters	____
1	CMC A.C. Cable	____
1	Multiwire cable for calorimeter CC to CMC	____
1	5-1/4 inch BPAC10C Back-up software disk	____

2.2 Description of Equipment

The bulk calorimeter is designed to measure the thermal power emitted by plutonium-containing samples packaged in 8802 Vollrath cans. The sample power range of the BPAC10C instrument is 0.1-10.0 watts. This system operates on 110/120 V 60 Hz AC and requires less than 20 amps.

The calorimeter consists of five temperature controlled concentric cylinders. Their operating temperatures and other control parameters are measured and displayed by the temperature control program as shown in the example given in Table VIII (Section 3.4).

2.3 Description of Software

The system software runs in a multitasking environment under Quarterdeck's DESQview and MS-DOS 4.01. Three tasks are required to run the calorimeter, BPAC-IO, BPAC-CTL and BPAC-DAS. All tasks and the operating system are loaded automatically on power-up. All software is contained on a hard disk drive. Backup software is available on 5-1/4" floppy disk. Software is menu driven and operator input is via the keyboard. The descriptions of the Main Menu subroutines are in Appendix B.

3.0 SYSTEM INSTALLATION

The calorimeter is designed for use in a measurement laboratory environment with ambient temperature in the range of 68° to 75° F.

3.1 Setup of Measurement System and Checkout

1 Unpack components and check against Table V. Check for any physical damage.
2. Locate components in a low gamma background area if isotopic measurements are to be made.
3. Make certain that electrical power 115 V 60 Hz and at least 20 amps is available. Do not connect to building power at this point.
4. Hook up components as indicated in Table VI. The cable connectors allow only correct connections. Please observe precautions listed in Table VI.

Table VI. System Hookup

Component/Component	Connector
CMC/CC	Multiwire
CMC/AC Power	AC Power*
SPC/AC Power	AC Power
CC/AC Power	AC Power**

* 115 V AC power for the CMC is supplied by the CC power distribution panel inside the rear door of the CC. DO NOT PLUG INTO BUILDING MAINS.

** Before plugging in CC/AC power cord, make sure the main power switch (see 3.1-8) is in the off position.

5. Install paper in the printer and roll paper up so that the perforation is even with the black bar above the print head.

6 Remove the shipping disks from both drives.

7. Turn power switch to ON on SPCs. The SPCs warm up to a temperature of 45.4°C in approximately 30 minutes. Adjust as necessary (0.4°C/turn).

8. Turn on the system (switch is inside the rear door of the CC on the power distribution panel).

9. After boot-up, the DAS will display the following:

"PLEASE LOG ON...."

"Operator:"

"Facility/MBA:"

Hard Copy Device is ENABLED automatically if the printer is ready. If the printer is not ready the following message will appear:

"Printer is not connected or ready. Do you want to attempt to correct this condition? Y/N"
If the response is yes and the printer is enabled the following prompt will appear:

"Be SURE that the paper is set to Top-Of-Form. (Roll up paper to top of sheet by turning knob on platen."

"Hit any key when ready.."

NOTE: The program checks status of printer and will not attempt to print if printer is off, not connected, or out of paper.

The next message to appear is:

"Want floppy disk enabled? (Y/N)" <Y>
A "Y" response produces the following message on the screen:

"An attempt will be made to initialize drive B:. In case of an error, a possibility exists that DOS may cause the program to abort. In such case, check the floppy for proper formatting and sufficient free space. A minimum of 25K is needed per sample."

"Insert FORMATTED disk in DRIVE B: Ready (Y/N)?"

Insert formatted disk in B drive, close the door and respond <u>Y</u> to the prompt.

3.1.1 If the system fails to boot-up on the hard drive after several tries, turn main power switch off and on, put the back-up disk in the <u>A</u> drive and reboot by pressing the button on the front panel of the computer.

3.1.2 After the requested information is entered, the Main Menu is displayed as shown below:

Data Processing Procedures: Main Menu

A: Assay Sample (PA)

B: Base Line Assay (PB)

C: Calculate Sample Pu Mass

D: Date and Time Change: - - - - - - 1990-02-01 at 10:30:00

E: Electrical Calibration Check

F: Floppy Disk Status: - - - - - - READY

H: Hard copy Toggle: - - - - - - - - ON LINE

O: Operating Instructions

R: Restart Program

X: eXit Program F1: Switch to IO Task F2: Switch to CTL Task

Enter Selection:

3.1.3 Check date and time displayed at menu selection "D"; correct if necessary by pressing <D> and entering correct date and time.

NOTE: Enter time in the 24 hour format. The current date is used in decay calculations, so entering the correct date is important.

3.1.4 Check the status of the printer displayed at menu selection "H". If status is "OFF LINE" press <H>. If status does not change, check to see if printer is turned on, paper is in the printer, and printer is in on-line mode. (If the printer will not function, all output data can be copied from the screen. Data is also stored on disk and can be printed later.)

3.1.5 Verify that the printer is working by pressing <Print Screen>.

3.1.6 Press <O> to view a description of each "Main Menu" selection (see Appendix B).

3.1.7 Check front panel meters as follows:

1. Error meters should indicate cold (left).
2. Power meters should indicate high power input (right).
3. Press F2 to check all system temperatures.
4. Temperatures should be above ambient and slowly increasing.

3.1.8 Return to Main Menu after system warm-up check by pressing <F3> and press <E> to start Electrical Calibration Check.

3.1.9 Respond <N> (No) to prompt "Begin this calibration immediately?" and enter date and time for calibration to start, allowing 5 hours minimum for warm-up. The electrical calibration takes approximately 60 minutes. If the calorimeter is not needed until the following day, the start time should be set for the next morning.

3.1.10 Confirm that the calorimeter is warming up by periodic checking of the temperatures by pressing <F2>.

3.2 Instrument Calibration Check

After the delay period, the system automatically enters an internal electrical calibration sequence where the DAS determines the base line power necessary to maintain thermal equilibrium in the absence of a sample. The system also determines the incremental powers necessary to obtain a six-point electrical calibration. Both the applied powers and the system responses to these powers are measured; this procedure takes approximately 60 minutes. A linear least squares fit is performed by the DAS on the six-point data set, and the slope (a_1), intercept (a_0), and their uncertainties (s_1 and s_0) are determined and stored for subsequent sample measurements. The intercept (a_0) is equal to the base line power (BP). The DAS prints the results of the electrical calibration in the format shown on the next page and stores the results in disk file CALIBRAT.XXX in the same format if the drive is ready.

TABLE VII.

Electrical Calibration Output

Report No. ____

Period __ / __ / __ to __ / __ / __

Facility/MBA :

Operator :

ELECTRICAL CALIBRATION of T3 MEASUREMENT CIRCUIT for BPAC10C

DATA POINT			T3 COIL			CAL COIL		TOTAL	
Dp #	# of Msmts	Time (Sec)	Amp Err (Volts)	Power (Watts)	+/- (Watts)	Power (Watts)	+/- (Watts)	Power (Watts)	+/- (Watts)
1	2	90	0.00096	12.4870	0.0004	0.0000	0.0000	12.4870	0.0004
2	4	90	0.00085	10.3904	0.0005	2.0966	0.0000	12.4870	0.0005
3	4	90	0.00069	8.2916	0.0004	4.1949	0.0000	12.4865	0.0004
4	4	90	0.00107	6.1933	0.0080	6.2925	0.0000	12.4858	0.0003
5	5	90	0.00072	4.0963	0.0002	8.3897	0.0000	12.4859	0.0002
6	5	90	0.00085	2.4865	0.0001	9.9998	0.0000	12.4863	0.0001

Calibration started on: 1990-02-01 at 13:11:40
completed on: 1990-02-01 at 14:11:00

Electrical Calibration Parameters :

Intercept = 12.4870 +/- 0.0002
Slope = -1.0001 +/- 0.0000

Slope OK? Yes ___ No ___

Problems:

3.2.1 Accept the electrical calibration if the calculated slope is equal to -1.0000 +/- $3*s_1$ and the intercept equals the zero input calibration step +/- $3*s_0$. If these conditions are not met, repeat the electrical calibration.

3.2.2 If an extra copy of the CALIBRATION is needed, press <H>. Press <M> to return to menu.

3.3 Measurement Control

1. After the calorimeter has reached stable operating temperature (minimum of five hours), press F2 and view the screen shown in Table VIII. Verify that actual zone temperatures agree with the nominal temperatures.

2. Verify that the SPCs are set at 45.4°C.
   Adjust the temperature as required using the temperature control on the SPC. (0.4°C/turn)

3. Verify that the temperature error meters in the CC read zero.

Table VIII

BPAC10C CONTROL PARAMETER DIAGNOSTIC DISPLAY 1990-02-21 at 08:42:46.2
Loop Time 0.77

	TA	TB	T0	T1	T2	T3	CAL
BASE ERROR:			0.0001	-0.0081	-0.0001	-0.0001	
DELTA ERROR:			-0.0001	0.0002	-0.0004	0.0002	
INTGL ERROR:			-0.0002	0.0001	0.0001	0.0000	
GAINS:			4	4	0	0	
CORR FACTOR:			0.9969	1.0003	0.9994	1.0001	
DAC DATA:			5.8146	5.4077	5.7553	7.3693	0.0000
PREAMP DACS:					5.0000	5.0000	
DESIGN TEMP:	Amb.	31.5	35.4	38.5	41.2	45.4	45.4
(Actual)	21.84	31.69	35.40	38.49	41.15	45.42	45.42
POWER:			3.4075	3.2532	3.7514	13.5025	0.0000

Press: F1: Switch to I/O Task, F3: Switch to DAS Task, eXit Program,

Hardcopy of / Redraw this screen..

4.0 DATA COLLECTION

4.1 Preparation of Samples

1. Load a plutonium sample into the sample canister and lock the lid in place.
2. Return the loaded canister to the preheater and preheat for a minimum of 4 hours or overnight.

4.2 Power Measurement

4.2.1 Quickly transfer a preheated loaded sample canister from the preheater to the calorimeter and replace the cover. (Preheat time should be as long as practical, but not less than 4 hours.)

4.2.2 Start the "Assay Sample Procedure" by pressing <A> with the "Main Menu" displayed and respond to the prompts displayed on the screen as shown below:

ENTER SAMPLE I.D. 0.4 watt sample
DO YOU WISH TO STOP AFTER A PREDICTION? (Y/N) <Y>

4.2.3 The starting time is displayed and measurements are displayed on the screen every 20 seconds until a prediction is reached.

4.2.4 The result is displayed and sent to the printer as shown below:

```
Assay started on:  1990-02-13 at 16:39:35,   TA + 25.84 Deg C
    completed on:  1990-02-13 at 16:58:01

  Assay Duration:    18 Min 26 Sec     Delta TA = -0.56 Deg C

       Sample ID:  0.4 watt sample

             Sample Time Constant : 30.9740 Min

Predicted Equilibrium System Power : 12.0892 +/- 0.0004

               System Assay Power: 12.0892 +/- 0.0010
```

4.2.5 The measurement data and the result shown above are stored in disk file SAMPLE.XXX where the XXX represents a number from 000 to 999 assigned sequentially by the program if the drive is ready.

4.2.6 Press <M> to return to menu.

NOTE: The predicted power is an extrapolated value of the sample power based on the measurement time up to this point. Occasionally, the DAS fails to predict the power and the measurement continues to equilibrium. In this case, the DAS prints out the equilibrium power.

4.3 Base Line Power Measurement

4.3.1 Remove the loaded sample canister from the CMC, insert a preheated empty canister and replace cover.

4.3.2 Start the "System Base line Assay" procedure by pressing <B> with the "Main Menu" displayed.

4.3.3 Respond as follows:

"Do you wish to stop after a prediction?" (Y/N) <Y>

4.3.4 On completion of the base line power measurement, the DAS will print,

```
Assay started on:  1990-02-13 at 17:00:49   TA = 23.53 Deg C
    completed on:  1990-02-13 at 17:18:52   TA = 23.99 Deg C

  Assay Duration:  18 Min  3 Sec      Delta TA = -0.45 Deg C

Predicted Equilibrium System Power : 12.4817 +/- 0.0007

     System Base line Power: 12.4817 +/- 0.0010
```

4.3.5 The System Assay Power (PA) and Base line Power (PB) are retained in memory for use in the sample assay calculations.

4.3.6 The measurement data and results shown above are stored in disk file BASELINE.XXX if the drive is ready.

4.3.7 Press <M> to return to menu.

4.4 Sample Plutonium Mass Calculation

4.4.1 Start the "Calculate Sample Pu Mass" procedure by pressing <C> with the Main Menu displayed.

4.4.2 Screen I below allows the operator to recheck the sample ID and:

1. Enter a value for ESP and its uncertainty to calculate only sample power and plutonium mass after <C> is pressed.

2. Leave the value of 1.0000 for ESP in which case only the sample power will be calculated after <C> is pressed.

3. Enter 0.0000 for the value of ESP which will signal the program to bring up Screen II for isotopic data input for calculation of ESP after <C> is pressed.

SCREEN I

PLUTONIUM MASS CALCULATION

Stratum/Sample ID/Type: EXAMPLE 1
Operator : Ronald Perry
Facility/MBA : ANL/SPM

The most recently measured values of calorimeter power are:

Type	Power		Uncertainty	
Sample ESP	3.8992 W/Kg	+/-	0.0068	W/Kg
Assay (PA)	12.0892 W	+/-	0.0010	W
Base (PB)	12.4817 W	+/-	0.0010	W

NOTE: To Calc sample Pwr & Pu content : Enter ESP +/- uncertainty.
To Calc Sample Pwr only : Leave ESP = 1.0000
To Enter Isotopic data : Enter ESP = 0.0000

Press: ENTER - select/change values, C - continue, M - Main Menu.

4.4.3 The output for Example I is as follows:

SAMPLE OUTPUT RESULTS for BPAC10C Date: 1990-2-13
Sample ID/Type : Example I
Operator : Ronald B. Perry
Facility/MBA : ANL/SPM

MEASUREMENT DATA			Msmt Duration
Effective Specific Power (W/kg)	=	3.8992 +/- 0.0068	
Calorimeter Base Line Power (W)	=	12.4817 +/- 0.0010	18 Min 3 Sec
Calorimeter Assay Power (W)	=	12.0892 +/- 0.0010	18 Min 26 Sec
Heat Standard Calib. Intercept a	=	0.0000 +/- 0.0000	
Heat Standard Calib. Slope b	=	1.0000 +/- 0.0000	
Sample Power (W) [(BP-AP)-a]/b	=	0.3925 +/- 0.0014	
Sample Pu Mass (kg)	=	0.1007 +/- 0.0026	

4.4.4 The output for Example II has the same information as above except that the first and last line under MEASUREMENT DATA are omitted because the ESP was not entered.

4.4.5 Screen II is displayed when <C> is pressed after entering zero for the value of ESP in Screen I.

SCREEN II

SAMPLE PARAMETER TABLE

Enter Pu Mass and Isotopic Data:

Pu/Isotope	Mass/Wt %	Uncertainty
Pu (Kg)	0.1200 Kg,	+/- 0.0010 Kg
Pu 238	0.1298 Wt %,	+/- 0.0005 Wt %
Pu 239	87.6970 Wt %,	+/- 0.0630 Wt %
Pu 240	10.6350 Wt %,	+/- 0.0720 Wt %
Pu 241	1.2988 Wt %,	+/- 0.0033 Wt %
Pu 242	0.1391 Wt %,	+/- 0.0005 Wt %
Am 241	0.5320 Wt %	+/- 0.0018 Wt %

NOTE: Americium content MUST BE Weight % of Pu mass, NOT % of sample weight!

4.4.6 Enter the declared value for the plutonium content of the sample in order to decay-correct the value to the date of calorimeter measurement. (Pressing the <ENTER> key will advance the cursor to the next data entry field.)

4.4.7 Enter mass spectrometric isotopic data or gamma ray isotopic data.

4.4.8 Press <C> when all data has been correctly entered. Use backspace key to correct errors. Step through a second time to reedit entries.

4.4.9 Enter the dates of analysis.
NOTE: Analysis dates do not appear until <C> has been pressed following isotopic entries.

4.4.10 Press <C> after dates have been entered to obtain results.

4.4.11 The results will be printed as shown on p. 15.

SAMPLE OUTPUT RESULTS for BPAC10C Date: 1990-02-13

Sample ID/Type : Example 1
Operator : Ronald Perry
Facility/MBA : ANL/SPM

OPERATOR'S DECLARED DATA

Original Pu mass & isotopic %			Updated Pu mass and isotopic %		
Item	Value	Uncertainty	Item	Value	Uncertainty
Pu (kg)	0.1200	+/- 0.0010	Pu (kg)	0.1199	+/- 0.0010
Pu 238 %	0.1299	+/- 0.0005	Pu 238 %	0.1289	+/- 0.0005
Pu 239 %	87.7850	+/- 0.0061	Pu 239 %	87.8443	+/- 0.0631
Pu 240 %	10.6457	+/- 0.0721	Pu 240 %	10.6520	+/- 0.0721
Pu 241 %	1.3001	+/- 0.0033	Pu 241 %	1.2354	+/- 0.0031
Pu 242 %	0.1392	+/- 0.0005	Pu 242 %	0.1393	+/- 0.0005
Am 241 %	0.5320	+/- 0.0018	Am 241 %	0.5923	+/- 0.0018

Pu Analysis Date: 1989-02-01 ESP calculated from: Facility isotopics
Am Analysis Date: 1989-03-01

MEASUREMENT DATA

			Msmt Duration
Effective Specific Power (W/kg)	=	3.8992 +/- 0.0068	
Calorimeter Base Line Power (W)	=	12.4817 +/- 0.0010	18 Min 3 Sec
Calorimeter Assay Power (W)	=	12.0892 +/- 0.0010	18 Min 26 Sec
Heat Standard Calib. Intercept	a =	0.0000 +/- 0.0000	
Heat Standard Calib. Slope	b =	1.0000 +/- 0.0000	
Sample Power (W) [(BP-AP)-a]/b	=	0.3925 +/- 0.0014	
Sample Pu Mass (kg)	=	0.1007 +/- 0.0026	

Relative Std. Dev.	s =	2.5664 %
Relative Difference	d =	16.0557 %
Ratio Diff/(Std. Dev.)	d/s =	6.2561

Isotopics OK? Yes ___ No ___

Problems:

4.4.12 The output data will be stored in disk file B:CALCS.XXX if the drive is ready.

4.4.13 Press <H> if an additional copy of the data is needed or press <M> to return to menu.

4.5.14 Repeat steps 4.1 to 4.4.13 for subsequent samples.

5.0 DATA EVALUATION AND REPORTING

With the plutonium content and its standard deviation determined and printed, the measurement of the Pu content of the sample is essentially complete.

5.1 Error Propagation

The error in determining sample plutonium mass by calorimetric assay is calculated by the DAS by combining the error in the sample effective specific power with the error in the sample power. This section describes the error model. The model is based on ANSI Standard N15.22-1987 "Calibration Techniques for the Calorimetric Assay of Plutonium-Bearing Solids Applied to Nuclear Materials Control."

5.1.1 Effective Specific Power Error Calculation

The effective specific power (ESP) is defined as the weighted sum of the isotopic specific powers. The standard deviation in this value is dependent upon the errors in both the isotopic mass fractions and their specific powers.

$$ESP = P_{eff} = \Sigma_i F_i P_i$$

$$\sigma^2 (P_{eff}) = \Sigma_i [F_i P_i]^2 [\sigma^2 (F_i)/F_i^2 + \sigma^2 (P_i)/P^2_i],$$

where

F_i	=	mass fraction of the radioisotope i relative to total Pu
$\sigma(F_i)$	=	standard deviation in F_i
P_i	=	isotopic specific power (W/kg)
$\sigma(P_i)$	=	standard deviation in P_i (W/kg)
P_{eff}	=	effective specific power (W/kg)
$\sigma(P_{eff})$	=	standard deviation in P_{eff} (W/kg)

i	Isotope	$P_i \pm \sigma(P_i)$ (W/kg)
1	238Pu	567.57 ± 0.26
2	239Pu	1.9288 ± 0.0003
3	240Pu	7.0824 ± 0.002
4	241Pu	3.412 ± 0.002
5	242Pu	0.1159 ± 0.0003
6	241Am	114.2 ± 0.42

In general, there is a negligible increase in the uncertainty reported in the isotopic mass fraction as a result of radioactive decay. This is not the case for 241Am, however.

$$\sigma^2_t(241Am) = [1.034t(L^{Pu}_{241} - L^{AM}_{241})\ \sigma_{t(0)}\ (241Pu)]^2 + \sigma^2_{t(0)}\ (241Am),$$

where t(0) = 241Am analysis date,
t = calorimeter power measurement date, and
L = the decay constants.

5.1.2 Sample Power Error Calculation

The inner sample chamber is held at a constant temperature of 45.4°C. When no heat-producing source is present, the calorimeter supplies a constant base line power, PB, 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 45.4°C drops to a lower assay power, PA. Using the effective specific power, P_{eff}, the plutonium mass is determined by:

$$M = P_S/P_{eff},$$

where the sample power P_S is given by:

$$P_S = \alpha + (PB - PA)\ /\beta$$

where α and β, the intercept and slope of the calibration line, have the values given in the following equation.

$$P_S = - (0.00981 \pm 0.0020) + (PB - PA)/(0.99858 \pm 0.00041) \quad (1)$$

The uncertainty in measurements made with the calibration function have a random and systematic error component. The uncertainty in a value of P_S determined from the calibration curve given in equation (1) was calculated by the method outlined in reference (3). The systematic error, s(sys), due to the calibration line is a function of the measured power as shown in Table II.

The prediction program also calculates an error in the fit for PA and PB. This value is usually less than 0.001 and is included in the above error estimate. However, the fitting error is occasionally larger due to the statistical procedure used to determine when to stop the iteration.

The error model for BPAC10C is:

$$S^2(P_S) = [S^2(PB) + S^2(PA) + S^2(r)]/N + S^2(HDE) + S^2(sys)$$

Where $S^2(PB)$ is the variance of the base line measurement, $S^2(PA)$ is the variance of the sample assay measurement, $S^2(r)$ is the variance of the measured sample power, N is the number of replicate measurements, S(HDE) is the heat distribution error, and $S^2(sys)$ is the calibration error.

Error Values

S(r) = 0.010 watt from replicate measurements
S(HDE) = 0.0000(PB-PA) from heat standard measurements
S(sys) = 0.0.0020 watt from calibration with heat standards.

5.1.3 Plutonium Mass Error Calculation

The standard deviation in the plutonium mass is calculated by the DAS by the following equation:

$$\sigma^2(M) = M^2[\sigma^2(P_s)/P_s + \sigma^2(P_{eff})/P_{eff}]$$

and is printed with the plutonium mass value.

APPENDIX B

DAS OPERATING COMMANDS

APPENDIX B

DAS OPERATING COMMANDS

The procedures described in these instructions are accessed from the MAIN MENU. In order to display the MAIN MENU press <M> when the prompt is displayed at the bottom of the screen.

Note: In the instructions, Operator Responses are enclosed in <..>. Only data entry requests must be terminated by the <ENTER> key. All other one-key responses, e.g., (y/n), require only a touch of the appropriate key.

TYPE <A>

ASSAY OF SAMPLE PROCEDURE

The operator may input information about the sample and decide if the assay will be performed using the default options. (See BASE LINE ASSAY PROCEDURE) for a description of the data collection.

TYPE <Y> ... for Defaults

The DAS will monitor the electrical power applied to the measurement chamber and predict the final settling point. This value and its uncertainty will be displayed and stored for later calculations.

TYPE <B>

BASE LINE ASSAY PROCEDURE

This procedure measures the empty chamber power required to maintain the calorimeter at the operating temperature. This base line power is used to calculate the sample power and should be measured after each sample measurement for best precision. During the power measurement, data is taken every 0.5 seconds. The mean and standard deviation of the mean of 40 new data points is displayed every 20 seconds. These mean values are used by the prediction/equilibrium recognition program to determine the predicted and/or equilibrium steady state base line power. Preliminary prediction calculations are displayed so that the operator can see how the measurement is progressing. Final results are displayed, stored in memory, and output to disk.

The operator may elect to continue data acquisition after a predicted value of the final power is obtained in order to obtain an equilibrium value for comparison. Type <Y> in response to start-up question for normal operation.

TYPE <C>

SAMPLE PU MASS CALCULATION

This procedure updates the isotopic data and the operator's declared value to the date of calorimetric assay and calculates the effective specific power (ESP). The ESP is then used to calculate the sample Pu mass.

The DAS asks the operator to recheck the sample identification and correct if necessary. The values of the BASE LINE & ASSAY POWER and the data field for entering a known value for the ESP (effective specific power) are displayed. Enter the ESP and its uncertainty in the correct position. Terminate entries with the <ENTER> key. Use the back space key for error correction. Move the cursor back to any field to re-enter data. When all entries are correct, press <C> to obtain assay results. No entries are required on this screen if the ESP is not known. To calculate the ESP press <C> to continue to the next screen. Enter the isotopic data and uncertainties in the proper fields. Corrections are made as above. Press <C> to continue after all data has been correctly entered. Enter the dates of analysis in the format shown. (If any errors in data entry have been made up to this point, press <M> to return to the Main Menu and press <D> to resume procedure. All data has been preserved and errors can still be corrected.) Press <C> to continue. The normal response to the question "Do you want to decay the isotopic to today's date?" is <Y>. The sample power is calculated from the difference in BASE LINE & ASSAY POWERS corrected for the measurement circuit calibration. The sample Pu mass is determined from the sample effective specific power (W/kg) and the instrument Pu calibration. The results are output to the printer and stored on disk.

TYPE <D>

SET DATE AND TIME

Setting the current date and time with this procedure is only necessary if the calorimeter is moved across time zones, because the computer has a battery-backed clock. However, it is important to check the date and time because these values are used by the program for calculating the isotopic decay to the date of measurement.

TYPE <E>

INTERNAL ELECTRICAL CALIBRATION CHECK

This procedure checks the electrical calibration of the power measurement circuits against the independently calibrated calibration circuits. Any change in calibration will be detected by a change in the slope or intercept of the calibration line. Intercept changes are canceled when the base line is subtracted from the assay power; therefore, only intercept changes are of consequence to measurement accuracy. Recalibration of the measurement system with a certified voltage reference source of the type used for calibration of digital voltmeters is required to correct intercept errors.

The Calorimeter must be in an equilibrium base line condition when this routine is initiated. If this condition is not true, the operator must delay data acquisition by selecting a future start time. (If the Measurement Chamber is already empty, a delay of 30 min is adequate.) The DAS will sequentially apply power increments to the Calibration Coil while monitoring the system response. The measurement circuit slope and intercept are calculated and stored for use in calculating sample thermal power. The results are output to the printer and stored on disk.

TYPE <F>

FLOPPY DISK DRIVE STATUS

This procedure checks the status of the B (lower) disk drive to determine if it is ready to receive data. Pressing <F> changes the status flag from "ON LINE" to "OFF LINE" if disk output is not needed. The status flag is changed from OFF to ON if <F> is pressed and the drive is ready. If the drive is not ready, the status flag will not change. Not ready conditions include no disk in drive, disk not formatted, drive lever open, and disk full.

TYPE <H>

TOGGLE HARD COPY

Checks status of printer and changes from off to on if printer is ready. Turns printer option off if printer option is on. If printer is not ready, no change will occur.

TYPE <O>

OPERATING INSTRUCTIONS

The file you are now reading is the SYSTEM OPERATING INSTRUCTIONS file.

TYPE <R>

RESTART PROGRAM

This procedure allows the operator to restart the program and log on, with or without reinitializing, without going out of the program.

TYPE <F1>

SWITCH TO I/O TASK

Switching to the I/O task allows the operator to view I/O data as it is updated every few seconds to confirm that the I/O task is running. There are no operator usable functions in the I/O task.

TYPE <F2>

SWITCH TO CTL TASK

Switching to the CTL task allows the operator to view the BPAC10C CONTROL PARAMETER DIAGNOSTIC DISPLAY. The diagnostic display gives the temperature of the controlled regions in the calorimeter which is useful for monitoring the warm-up when the calorimeter is turned on. This procedure also displays error voltage measurements and correction factors at key test points in the calorimeter temperature control circuits. This information may be useful in diagnosing malfunctions and pinpointing which module is causing the problem. The information is not needed for normal operation and is not intended for operator use.

TYPE <F3>

SWITCH TO DAS TASK

Switches back to the DAS task from the CTL or I/O task. Switching from task to task does not interrupt the function currently being performed by any task.

TYPE <X>

EXIT PROGRAM

Immediate exit from calorimeter program. All data not saved to disk or HARD COPY is lost. Control of the computer is returned to DOS (Disk Operating System). To resume calorimeter program operation type "BPAC-DAS". Use of EXIT in orderly shut down of the equipment is recommended starting with the DAS task. After exiting the program, the DESQview window should be closed and the process is then repeated for the CTL and I/O program. After the window for the I/O program is closed, QUIT DESQview. The computer is now back in single-user DOS mode and the power can be turned off to the system. Refer to the DESQview manual for additional information.

APPENDIX C

CALIBRATION OF BPAC10C

APPENDIX C

CALIBRATION OF BPAC10C

Calibration of the electrical power vs the thermal power due to radioactive decay of plutonium samples requires the use of certified heat standards. Calibration is performed only by persons qualified and authorized to do so. This calibration procedure is only intended for authorized use for initial calibration against certified heat standards.

1.0 Configuration

1.1 Heat Source Requirements
Certified heat sources (Pu 238) covering the operating range of the calorimeter up to 10 watts are required. Three or more standards that can be used in combination to produce 5 to 7 points covering the range should be adequate.

1.2 Set up and checkout the measurement system as instructed in the detailed procedure. (3.1)

2.0 Calibration Procedure

2.1 Sample Preparation
Center the heat source midway between the center and bottom of the canister by filling the bottom of the canister with crumpled aluminum foil. The heat source should be placed in a shot filled can to prevent the source from moving during handling. Fill the void space with loosely crumpled aluminum foil.

2.2 Preheat the canister overnight at 45.4°C and transfer the canister to the calorimeter.

2.3 Start the Assay procedure. (See Detailed Procedure.)

2.4 Remove the sample canister when assay has been completed and start Base Line procedure.

2.5 Subtract the Assay result from the Base Line result to obtain the uncorrected instrument response.

2.6 Repeat 2.1-2.5 for 5-7 points covering the range of the calorimeter (1-10 watts).

3.0 Fitting data to Calibration Curve

3.1 Press <Alt> <SysRq> to display the DESQview MENU and select Open Window.

3.2 Select the DEMMING PROGRAM (DEM)

3.3 The Demming least squares program for a linear function can be used to calculate the calibration parameters and error terms needed. Select the equal error in x and y option and enter the power given for the heat standard decayed to the date of CALORIMETRY as the independent variable (X) and the power difference from step 2.5 as the dependent variable (Y).

3.4 Select the linear least squares fitting option (2) for the function: $Y = a1(X) + a2$.

3.5 The value of a1, a2, and their errors are given by the program under the heading "FINAL PARAMETERS".

3.6 The random error variance is given by DEMMING under the heading "S/df = variance of an observation of unit weight = $[s(r)]^2$

3.7 Request the "Interpolation on the line" option and "x for all y" with 67% Student's Limits to obtain a table giving the systematic error limits for the calibration line. The systematic error variance $[s(sys)]^2$ is the square of the values given in the table under the heading "x error".

3.8 Calculate the total error variance (V) by summing the random and systematic error variance given in 3.6 and 3.7

3.9 Exit from the DEMMING program

4.0 Entering the calibration parameters.

4.1 Press <Alt> <SysRq> and Open Window and select Parameter Setup (PS) from the DESQview MENU.

4.2 Respond <Y> to obtain list of current default values. Type <H> to print a hard copy.

4.3 Press <ENTER> and respond <Y> to create a new file.

4.4 Enter new slope at prompt and press <ENTER>.

4.5 Enter uncertainty in slope at prompt.

4.6 Enter intercept at prompt. NOTE: Include sign for negative entry.

4.7 Enter uncertainty in intercept.

4.8 Enter total uncertainty s(t) = sqrt(V) from 3.8

4.9 Respond <Y> to continue and repeat step 3.2 to check file for correct entries and make a HARD COPY to keep as a record of the change.

4.10 Respond <N> to prompts to exit program.

4.11 Press <R> to select "Reinitialize" BPAC-DAS program and read in new calibration parameters.

APPENDIX D

TROUBLESHOOTING HINTS

APPENDIX D

Troubleshooting Hints

The computer and calorimeter control electronics are not easily repairable in the field; however, there are some faults that can be corrected by the operator. Do not attempt to open the equipment enclosures without the assistance of an electronics specialist. Shock hazards exist inside the enclosures when the line voltage cord is plugged in even when the switch is turned off. If the checks listed below do not correct the problem, do not use the equipment.

DATA ACQUISITION SYSTEM

Symptom: No power to computer.

Check to see that the line cord is plugged into a live outlet, that the proper voltage is present and that the switch on the MIF111 Distribution Box is on.

Next, verify that the switch on the ISOBAR surge suppressor box is on and its indicator light is lit. If not lit press the white RESET button on the ISOBAR to assure that it is not tripped.

Next, verify that the computer/printer plug in strip (located on the left side of the rack as viewed from the rear and just under the top of rack) plugged into the ISOBAR the switch is on and the reset is not tripped.

Finally verify that the computer and printer cords are plugged into the outlet box.

If the computer is plugged in and all switches are turn on and computer still does not come on, try turning power off and back on with the switch on the computer/printer plug in strip.

Symptom: Computer fails to boot on start-up.

Check the floppy disk drives to make sure that the shipping disks have been removed and that the "A" drive is empty.

Press the reset button on the front panel (lower right) and see if the system boots.

If the system still fails, try turning the power off for a few seconds and back on again.

If the system still fails, insert the backup disk into the "A" drive (5 1/4" drive) and restart.

Symptom: Program is hung-up and will not respond to keyboard commands.

Be sure that the program is in the command mode and is monitoring the keyboard. Read any prompt that may be displayed on the screen to see if the program is waiting for a particular key response from the operator.

If the cursor is being displayed with a drive letter, the program has aborted. If a run time error message is being displayed, record the error message, error address, and restart the program by typing BPAC-DAS <ENTER>.

Try unplugging the keyboard cable from the computer for a few seconds and plug in again.

If the above checks do not solve the problem, re-boot the system by pressing the reset button on the front panel.

If the problem is not corrected, turn the power to the computer off for a few seconds and back on. Switch off power switch on outlet strip located at top of cabinet above printer drawer at rear. This turns off printer and computer.

PRINTER

NOTE: SEE PRINTER MANUAL FOR ADDITIONAL INFORMATION.

Symptom: Red POWER light off.

Ensure that the line cord is plugged in at both ends and the power switch is on (right side near rear when facing printer from front of cabinet).

Check fuse (see manual).

Symptom: Red PAPER OUT light on.

Load paper into the printer. The PAPER OUT light will go out.

Symptom: Printer does not respond to computer.

Ensure that the POWER light is on and the PAPER OUT light is off. If this is not the case, refer to error conditions above.

Verify that the printer is operational by running the self test as follows: Hold down the LF button while turning on power. Printer should continuously print the full character set until stopped by turning off power.

Check the data cable connection at the rear of the printer to see if the connector is in straight, is well seated, and both connector clips are engaged.

Symptom: Paper does not feed properly.

Remove the paper from the printer and discard any that is crumpled.

For fanfold paper, verify that it can travel freely without catching, and that the right side pinwheel is adjusted correctly for the width of the paper.

Symptom: Print quality is poor, rows of dots are missing, or carriage moves but produces no print.

Lift top cover and inspect ribbon cartridge to see that it has not been dislodged.

Turn knob on top of cartridge in direction of arrow to see if ribbon is jammed. If ribbon moves easily, turn knob several times to bring fresh ribbon under print head.

Run self test as described above. If print is light or blank, try replacing ribbon cartridge.

CONTROL CABINET

Symptom: No power to CC.

Check line cord connection and see that it is connected to a live outlet and the proper voltage is present.

Check switch position on the MIF111 Distribution Box to see that it is on.

Check fuses on MIF111 Power Distribution Box. First unplug the line cord and then remove fuses to avoid possible shock hazard.

Symptom: Calorimeter fails to warm-up.

Check to see that the AC power cable is connected between the CC and the CMC. (Caution this cable should be plugged into MIF111 Box, not into the ISOBAR.)

Check to see if the lamps on the CMC base are on. Check to see that AC cord is firmly plugged into the socket on the CMC base. If still not on, unplug AC power cord and check fuses on CMC base.

Check the coil volt meters on the T0, T1, T2 and T3 control modules to see if voltage is being applied to all coils.

Check the temperatures on the Control Parameter Diagnostic Display (Press <F2>). If one or more of the temperatures are reading incorrectly or read negative, the fault may be in the temperature measurement circuit. This does not necessarily mean that the calorimeter is not warming up. After a minimum of 5 hours warm-up, check the error meters on each control module. If the meters read zero, proceed with setup and electrical calibration and make a note of the problem with the temperature readout. If the meters are not reading zero and the temperatures are not correct, the calorimeter should not be used.

PREHEATER

Symptom: No power to preheater on start-up.

Check to see that the line cord is plugged into a live outlet.

Check to see that switch on preheater is turned on.

If preheater still fails to come on, check circuit breakers on top panel

Symptom: Preheater temperature reads negative.

Negative temperature reading indicates an open temperature measurement circuit. Unplug line cord and remove screws from cover and lift preheater from case. Check for a broken wire or loose connection at temperature meter. Meter connections can be checked by removing plate on meter where wires enter meter.

Symptom: Preheater shuts off during operation.

Overheating will cause the preheater to shut down and not restart. Test the preheater without a sample in the chamber by pressing the restart button and watching the temperature as it warms up. If the temperature rises above 45°C and the heater lamp does not start to blink, try turning the temperature control counter clockwise. If the heater stays on full until the power shuts down, the controller is malfunctioning and the preheater should not be used. If the lamp starts to blink or goes off, attempt to set the control for the proper operating temperature.

If the preheater functions properly without a sample, the sample power may be too high. Sample power should not exceed 10 watts.

ABBREVIATIONS

ADC	Analog to Digital Converter
ANL	Argonne National Laboratory
BPAC10C	Bulk Plutonium Assay Calorimeter 10 (Watt) (unit) C
CALEX	Calorimeter Exchange
CC	Control Cabinet
CMC	Calorimeter Control Cabinet
DAC	Digital to Analog Converter
DAS	Data Acquisition System
DOE-OSS	Department of Energy, Office of Safeguards and Security
DVM	Digital Volt Meter
ESP	Effective Specific Power
I/O	Input/Output
LANL	Los Alamos National Laboratory
NDA	Nondestructive Assay
NIM	Nuclear Instrumentation Module
PA	Assay Power
PB	Base Line Power
PID	Proportional Integral Differential
RSD	Relative Standard Deviation
SD	Standard Deviation
SPC	Sample Preheating Chamber
TA55	Tech Area 55 (Los Alamos Plutonium Facility)

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