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**Progress Report for the Division
of Safeguards and Security:
January-June 1977**

December 23, 1977

Monsanto

MOUND FACILITY

Miamisburg, Ohio

operated by

MONSANTO RESEARCH CORPORATION

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for the

U. S. DEPARTMENT OF ENERGY

Contract No. EY-76-C-04-0053

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R. P. Ratay, Editor

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Foreword

Under the sponsorship of the ERDA Division of Safeguards and Security, Mound Laboratory is responsible for studies to advance nuclear safeguards technology in the public interest.

This report is submitted by W. T. Cave, Director, Nuclear Operations, and R. E. Vallee, Manager of Technology Applications and Development, from contributions prepared by members of the Applied Physics Section: W. H. Smith, Manager.

Summaries

DEVELOPMENT OF NONDESTRUCTIVE ASSAY TECHNIQUES

Prediction of Calorimeter Equilibrium Development of the calorimeter equilibrium prediction program has been completed, and tests of the software program have been conducted on calorimeters ranging from 1 to 6 in. (2.5 to 15 cm) in diameter and power inputs between 0.1 and 8.3 W. Equilibrium values have been predicted with relative accuracies of better than 0.1% (95% confidence) and with an average time improvement factor of 2.1, which represents a 52% time saving, for every test case.

Computer Modeling A computer code is being developed by the University of Cincinnati to describe the steady-state heat transfer in a resistance bridge calorimeter. The computer program which is based on the theory of heat transfer analysis by finite differences has been written and is being checked against experimental data. Following the development of the steady-state code, work will commence on the transient heat flow analysis.

Gamma-Ray Spectroscopy Development The effect of R. Gunnink's new plutonium branching ratios on previous plutonium isotopic ratio measurements is demonstrated. For measurements relative to plutonium-239 the biases change from between 2 and 7% low to between 2 and 13% high. For measurements relative to plutonium-241 the changes in bias are within the statistical uncertainties normally reported for these peaks.

SYSTEMS DEVELOPMENT

Automated Plutonium Assay System In preparation for the proposed demonstration tests on the Automated Plutonium Assay System (APAS), reliability tests on the system hardware have been conducted and appropriate modifications have been made. The development of the computer software for controlling the system and conducting the tests has continued and the experimental plan outlining the tests has been devised.

Development of nondestructive assay techniques

Prediction of calorimeter equilibrium

C. L. Fellers and P. W. Seabaugh

The various facets of the prediction-of-equilibrium study [1,2,3] have been incorporated into a single operating program. Tests on a variety of calorimeters show that equilibrium values can be predicted with a relative accuracy of 0.1% (95% confidence) and with an average time improvement factor of 2.1. The equilibrium prediction program is applicable to any calorimeter (or analytical instrument) which responds exponentially, and requires no subjective evaluation on the part of the operator.

Calorimeter output data collected at equally spaced time intervals are used to evaluate the system time constant, the equilibrium values, and the point at which the prediction process can be terminated. Following the placement of a source of heat in the calorimeter, the operator enters three parameters: the uncertainty in the individual data points, the baseline bridge potential, and an estimate of the system time constant. The calculator or minicomputer then determines the rate at which data should be collected, smooths the raw data using a nonlinear convolution technique, calculates the system time constant, predicts equilibrium values, and terminates the process when the optimum accuracy has been achieved. The level of desired accuracy can be altered but the program is currently written to predict values within $\pm 0.1\%$ relative or $\pm 6 \mu\text{V}$ (95% confidence) of the true equilibrium value, whichever is greater.

The accuracy in the final predicted value is a function of the system time constant, the uncertainty in the individual data points, the magnitude of the calorimeter response, and the time interval between the three points used in the prediction equation. This functional relationship is represented mathematically in Equations 1 and 2.

$$\sigma_a = \frac{1.96ue^{-\lambda\epsilon}}{A_c(1 - e^{-\lambda\epsilon})^2} \quad (1)$$

$$S = \sigma_a A_c \quad (2)$$

where: σ_a = relative uncertainty in the predicted value
 S = absolute uncertainty in the predicted value
 u = uncertainty in the individual data points
 A_c = equilibrium value (A) corrected for the baseline (B)
 λ = reciprocal of the system time constant
 ϵ = time interval between data points used in the calculation of the equilibrium value

To obtain the desired accuracy in the minimum time, the interval between points (ϵ) must be optimized. By increasing ϵ , the predicted values are improved, but more time is required to achieve the results. Solving Equation 1 for ϵ yields the optimum interval. However, the uncertainty in the individual data points (u), a characteristic of the calorimeter and data acquisition system, is the only known parameter of Equation 1. The equilibrium value A_c and the uncertainty in the predicted value S are unknown and the system time constant is an estimate made by the operator. A more refined estimate of the system time constant and a rough estimate of equilibrium value are needed before the optimum time

interval can be determined. This information can only be derived from the calorimeter output data.

Data are collected on an interval equal to 3% of the operator's estimate of the system time constant (C). A refined estimate of the time constant is then calculated using Equations 3 and 4. The three data points used in this calculation are separated by an interval (K) equalling 33% of the operator's time constant estimate.

$$V_i = \frac{K}{\ln X_i} \quad (3)$$

$$X_i = \frac{Y_{(i+2K)} - Y_{(i+K)}}{Y_{(i+K)} - Y_i} \quad (4)$$

The best estimate of the time constant is taken as the average of the five successive calculated values whose slope changes sign with respect to that of the previous five values. This can be expressed as follows:

$$\text{Slope } (l-1) = f(V_{l-5}, V_{l-4}, V_{l-3}, V_{l-2}, V_{l-1})$$

$$\text{Slope } (l) = f(V_{l-4}, V_{l-3}, V_{l-2}, V_{l-1}, V_l)$$

$$\text{If } |\text{slope } (l) + \text{slope } (l-1)| \neq |\text{slope } (l)| + |\text{slope } (l-1)|$$

$$\text{then} \quad \text{Time Constant} = \frac{\sum_{m=4}^0 V_{l-m}}{5}$$

In addition, the three data points used to calculate the last time constant estimate (V_l) are used in Equation 5 to provide a rough estimate (A_{est}) of the predicted equilibrium value corrected for the baseline (B):

$$A_{est} = \frac{Y_{(l+K)} - Y_l X_l}{1 - X_l} - B \quad (5)$$

By setting S equal to $0.05A_{est}$ or $3 \mu V$ (whichever is greater), Equations 2 and then 1 can now be solved for ϵ , the optimum time interval between the points used in predicting an equilibrium value.

Starting with the observed data point Y_i ($i = l$), equilibrium values are predicted using Equations 6 and 7:

$$A_i = \frac{Y_{(i+\epsilon)} - Y_i Z_i}{1 - Z_i} \quad (6)$$

$$Z_i = \frac{Y_{(i+2\epsilon)} - Y_{(i+\epsilon)}}{Y_{(i+\epsilon)} - Y_i} \quad (7)$$

A new value is predicted with the reading of each new data point observed at intervals equal to $0.03C$.

A plot of the predicted values, which are representative of the true equilibrium versus time, shows that the predicted values oscillate about a line with zero slope. By monitoring the slopes of successive sets of ten predicted values for inflection points, the final predicted value can be obtained. The accepted equilibrium value is taken as the average of the predicted values which lie between two successive inflection points if this average meets the criteria that it is composed of a least four predicted values and that the standard deviation of the mean is less than S. The predicted value is quoted with 95% confidence limits equal to $1.96S$.

Tests have been conducted on a variety of calorimeters and the results of these tests are presented in Table 1. An average improvement factor with respect to analysis time of 2.1 has been achieved and equilibrium values with relative accuracies of $\pm 0.1\%$ or $\pm 6 \mu V$ (95% confidence) have been predicted in every case.

Table 1 - EQUILIBRIUM PREDICTION TEST RESULTS

Test	Equilibrium Value (μ V)	Uncertainty in Predicted Value (95% Confidence)		Error in Predicted Value		Time Improvement Factor
		$\pm\mu$	$\pm\%$	μ V	%	
1	7779.8	7.6	0.10	0.3	0.004	1.81
2	75874.0	75.0	0.10	19.7	0.26	2.85
3	22843.9	22.6	0.10	4.3	0.19	2.09
4	7421.1	7.4	0.10	-1.7	-0.02	2.12
5	2591.9	5.3	0.20	-1.3	-0.05	1.34
6	3725.7	6.0	0.16	-2.2	-0.6	1.95
7	77453.2	76.5	0.10	-10.8	-0.01	1.96
8	1524.3	5.9	0.39	-1.8	-0.12	2.23
9	2585.9	5.9	0.23	0.6	0.02	1.99
10	75934.9	75.0	0.10	35.1	0.05	2.37
11	36855.9	36.4	0.10	-18.2	-0.05	2.09
12	77412.4	76.5	0.10	8.7	0.01	2.54
13	125908.5	124.3	0.10	-28.6	-0.02	2.38
14	20637.5	20.3	0.10	9.0	0.04	2.13
15	25871.0	25.5	0.10	-9.1	-0.03	2.09
16	1647.5	5.7	0.37	1.0	0.06	1.92
17	3655.9	5.9	0.16	5.5	0.15	1.71
18	9179.0	9.0	0.10	0.1	0.001	2.31
19	9003.1	8.9	0.10	-1.6	-0.02	2.06

Computer modeling

C. L. Fellers

A joint effort between Mound Laboratory and the University of Cincinnati Nuclear Engineering Department has been initiated to develop a computer program which describes the heat transfer in a resistance bridge calorimeter. Initially, the computer modeling will be restricted to steady-state heat flow, but will ultimately encompass the transient state as well. This code will allow for rapid analysis of calorimeter designs without the time and cost required to fabricate and test new instrumentation.

A general-purpose resistance bridge calorimeter design is the basis for the development of the mathematical model. The calorimeter is modeled as a series of 25 radial rings and 75 axial segments, and a separate conductor equation is associated with each of the 1875 (25 times 75) control volumes.

An iterative solution scheme is employed by the digital computer to solve the conduction equations for all of the volumes simultaneously. The temperature of any one of these control volumes can be given in the steady-state condition. However, rather than providing merely a printout

of the temperature of all volumes, the program gives a graphical display (matrix) of temperature of selected volumes, primarily of those volumes which correspond to various interfaces in the calorimeter. Although the program input subroutine minimizes the amount of data the user must supply, the operator has the flexibility to vary all of the calorimeter dimensions, material properties, amount, and location of heat generation.

The computer program has been written and debugged, and its validity was established by checking special problems with manual calculations. Code results were also compared to experimental calorimeter measurements. These comparisons indicated the need for revising the model design and some of the numerical techniques used. Following these revisions, good agreement was obtained between the calculated and experimental differentials across the thermal resistance gap in the calorimeter.

Following the development of the steady-state code a report documenting this portion of the work will be prepared and work on the transient version of the code will be initiated.

Gamma-ray spectroscopy development

J. F. Lemming

Gamma-ray spectroscopy is an accepted method for determining the relative isotopic composition of plutonium materials [4]. It has a unique advantage over traditional chemical techniques because it is non-destructive and can be applied to bulk samples.

This method relies on the branching intensities and half-lives of the isotopes

being measured for converting the ratios of gamma-ray peak intensities to atomic ratios and/or for accounting for sample self-absorption effects.

The following data have been collected to quantify the effects on gamma isotopic measurements of both the recent changes in these branching ratios and also their uncertainties.

In 1973 Haas and Jarvis [5] calculated the conversion coefficients for a series of peak pairs using the branching intensities reported by Cline [6] and those reported by Gunnink and Morrow [7]. On the basis of the comparison of their analysis to chemical analysis for small metal samples, they chose to use Gunnink's branching intensities. These branching intensities have given consistently favorable results over a 3-yr period on a variety of both aliquot and bulk samples [8]. A summary of their results is shown in Table 2. The gamma-ray spectroscopy measurements differ from the chemically determined ratios by less than 7%.

In 1974 Parker and Reilly [9] calculated conversion coefficients for a set of peak pairs using Gunnink's branching intensities. However, they did not use the calculated conversion coefficients since the values were not consistent with their measurements on National Bureau of Standards standard reference materials NBS-SRM 946 and 948. Parker and Reilly also used Gunnink's branching intensities to correct for sample self-absorption effects. In this case they found it necessary to adjust some of the published branching intensities by as much as 10%.

In a continuing effort to shorten the time necessary to obtain atomic ratios by

Table 2 - COMPARISON OF PLUTONIUM ISOTOPIC RATIOS DETERMINED
BY GAMMA-RAY SPECTROSCOPY AND CHEMICAL TECHNIQUES

<u>Isotopic Ratio</u>	<u>Gamma Aliquot/Chemical</u>	<u>Gamma Can/Gamma Aliquot</u>
$^{238}\text{Pu}/^{239}\text{Pu}$	0.965	1.053
$^{240}\text{Pu}/^{239}\text{Pu}$	0.934	1.004
$^{241}\text{Pu}/^{239}\text{Pu}$	0.992	0.998

gamma-ray spectroscopy, Haas et al. [10,11] had extended their work to different energy regions using branching ratios reported by Gunnink and Morrow. Their preliminary results using NBS-SRM 946, 947, and 948 show biases of less than 10% for all isotopic ratios.

Recently Gunnink et al. [12] published a reevaluation of the gamma-ray energies and absolute branching intensities of uranium-237, plutonium-238, plutonium-239, plutonium-240, plutonium-241 and americium-241 partially in support of isotopic abundance measurements. In the following discussions the effect of the new branching ratios on the published conversion factors is demonstrated.

Table 3 lists the peak pairs (in keV) and conversion factors reported by Haas and Jarvis [5]. The changes in the conversion factors will change the bias between the chemical measurement and the gamma-ray measurement shown in Table 2 from between 2 and 7% low to between 2 and 13% high.

Table 4 compares the branching intensities chosen by Parker and Reilly [9] for relative efficiency corrections to both sets of Gunnink's branching intensities [7,12]. Although the new branching ratios do remove some differences, others have been introduced. Before reliable relative

efficiency corrections can be made these discrepancies should be resolved.

In Table 5 the conversion factors and the peak pairs chosen by Parker and Reilly [9] are compared to those calculated using Gunnink's new branching intensities. These peak pairs essentially cover the extension of the work by Haas et al. [10], and their results are shown in Table 5. The agreement between the old and new branching ratios is better for these peak pairs, and the changes are within the statistical uncertainty normally reported for these peak pairs.

Because the uncertainties in conversion factors are larger than those permitted if a calorimetric assay with overall uncertainty of 1% is to be performed, a series of measurements has been designed to experimentally determine a best set of conversion factors. This experiment will use NBS-SRM 946, 947, and 948 in conjunction with unknown plutonium samples which will be analyzed by traditional chemical techniques at two laboratories.

Table 3 - COMPARISON OF CONVERSION FACTORS FOR
ISOTOPIC RATIOS RELATIVE TO PLUTONIUM-239

Isotopic Ratio	Peak Pair	Conversion Factors		Δ^a (%)
		Old	New	
$^{238}\text{Pu}/^{239}\text{Pu}$	153/144	1,010.3	1,075.0	-6.40
$^{240}\text{Pu}/^{239}\text{Pu}$	642/648	148,332	178,281	-20.19
$^{241}\text{Pu}/^{239}\text{Pu}$	148/144	852.7	908.6	-6.56
$^{241}\text{Pu}/^{239}\text{Pu}$	203/208	623.7	630.8	-1.14
$^{241}\text{Pu}/^{239}\text{Pu}$	662/659	487.5	477.3	+2.09

$$^a \frac{(\text{Old}-\text{New})}{\text{Old}} \times 100$$

Table 4 - COMPARISON OF BRANCHING INTENSITIES
USED FOR RELATIVE EFFICIENCY CORRECTIONS

Energy (keV)	LASL ^a	Relative Intensities (Gunnink)		Δ^d (%)	Δ^e (%)
		Old ^b	New ^c		
129.28	0.0062	0.00620	0.00626	0	0
144.19	0.000325	0.000286	0.000283	12.0	12.92
161.45	0.000125	0.000130	0.000120	-4.0	+4.0
171.34	0.00013	0.000109	0.0001105	3.54	2.21
179.17	0.0000628	0.0000639	0.0000658	-1.75	-4.78
189.34	0.0000872	0.0000776	0.0000830	11.01	4.82
195.65	0.000107	0.000107	0.0001064	0	0.56
203.52	0.00056	0.000560	0.000560	0	0
255.33	0.0000803	0.0000803	0.0000805	0	-0.25
297.43	0.000050	0.0000500	0.0000502	0	-0.4
344.96	0.000561	0.000561	0.0005592	0	0.32
325.02	0.00158	0.00158	0.001570	0	0.63
413.69	0.00151	0.00151	0.001489	0	1.39

^aReference 9.

^bReference 7.

^cReference 12.

$$^d \frac{(\text{LASL}-\text{Old})}{\text{LASL}} \times 100$$

$$^e \frac{(\text{LASL}-\text{New})}{\text{LASL}} \times 100$$

Table 5 - COMPARISON OF CONVERSION FACTORS
IN ISOTOPIC RATIOS RELATIVE TO PLUTONIUM-241

Isotopic Ratios	Peak Pair	Conversion Factors		
		Parker & Reilly ^a	Haas et al. ^b	New Gunnink ^c
$^{238}\text{Pu}/^{241}\text{Pu}$	153/148	1.227	1.135	1.183
$^{240}\text{Pu}/^{241}\text{Pu}$	160/164	45.14	50.7	44.91
$^{239}\text{Pu}/^{241}\text{Pu}$	208/203	1569	1603	1585
$^{239}\text{Pu}/^{241}\text{Pu}$	129/125	0.0281	0.027875	0.027510

^aReference 9.

^bReferences 10 and 11.

^cReference 12.

Systems development

Automated plutonium assay system

*C. L. Fellors, J. H. Birden, M. F. Duff
and J. R. Wetzel*

The Automated Plutonium Assay System (APAS) [13,14] which demonstrates calorimetric assay in an automated mode of operation is scheduled to be tested during July and August 1977. In preparation for this test an experimental test plan has been devised, further modification to the system hardware has been incorporated, and development of software programs for controlling the system has continued.

The experimental tests are being performed to determine the precision and accuracy of the overall system, to evaluate system reliability, and to demonstrate the timeliness of the measurement collection and feedback. The samples to be used in the tests are comprised of FFTF-type mixed oxide powder blended to a ratio of 70%

UO_2 to 30% PuO_2 . Four kilograms of this material has been ordered from HEDL.

The mixed oxide will be tested for homogeneity (and reblended if necessary) before being loaded into six sample containers. Aliquots of the powder will be taken for isotopic characterization. Since the bulk material was blended homogeneously, the six sample cans will contain isotopically homogeneous material but each can will be filled with differing quantities (see Table 6).

These six cans plus three cans containing plutonium-238 heat standards (used for calibrating the calorimeters) and one empty can will constitute the total sample set for the APAS tests. Each sample will be passed through the system repeatedly, and each time it is analyzed it will be considered a unique sample.

The tests will be divided into two operating modes (container verification and material accounting), each lasting approximately

Table 6 - APAS SAMPLES

Can No.	Nominal Net Weight (g)
1	100
2	300
3	500
4	800
5	800
6	1000

15 days. The container verification mode will simulate an application where the isotopics of a batch of material and the quantity of plutonium in each sample container are known from a previous measurement. Each sample moving through the system will be calorimetered and a plutonium content will be calculated using batch isotopics. This value will then be compared to the previously determined quantity. Also, the ratio of the plutonium-239 to plutonium-241 will be measured on randomly selected samples by gamma-ray spectroscopy. This ratio will be compared to the previously determined batch isotopics. Each of the six mixed oxide samples will be measured in one of two calorimeters 90 times and analyzed by gamma-ray spectroscopy 60 times at a throughput rate of one sample per hour per instrument. In addition, 60 measurements on the plutonium-238 heat standards and 30 measurements on the empty can will be made in each calorimeter. At the conclusion of each sample analysis in the calorimeter or in the spectrometer a report will be generated giving the analysis results and a comparison of the measured and known characteristics of the sample.

The material accounting mode of operation will be similar to the container verification mode except that instead of a single

ratio the complete isotopic analyses will be performed by gamma-ray spectroscopy. The same number of samples, heat standards, and empty can measurements will be made in the calorimeter as in the container verification mode. In addition, each of the six samples will be analyzed for total isotopics (except for plutonium-242) by gamma-ray spectroscopy 10 times. An average of the isotopics determined by gamma-ray analysis plus a "stream average" value for plutonium-242 will be combined with each independent calorimeter analysis and the mass of plutonium (in grams) contained in 540 unique samples will be reported.

Data from these tests will be statistically analyzed using ANOVA techniques and calibration curves comparing APAS-determined values (NDA) to plutonium quantities determined by conventional destructive analysis techniques.

Before the system tests are started it is imperative that all of the hardware is operating in a reliable manner. A computer program has been written which alternately moves two sample cans between 10 storage wells in the system and monitors the number of sample moves and the number of times the dual-state pickup device fails to pick up or release a sample. Several problem areas within the system have been identified by running this reliability test, and corrective action has been taken:

1. The relative encoders on each axis of the three-axis transporter were re-initialized once each hour during the reliability test [15]. By reading the zero offset in each encoder immediately before re-initialization, it was determined that two of the encoders (on the y and z axes) were drifting beyond acceptable limits. New encoders were

installed and the original devices are being repaired.

2. In the event of an encoder failure, the transporter could ram the end of the boxline at full speed and possibly damage itself or the can it carries. To minimize the effects of such an occurrence, two self-contained, mini-cushion shock absorbers have been installed at the end of the transporter support frame. Preliminary tests indicate that the shock absorbers satisfactorily protect the transporter from damage.
3. Following several hours of continuous operation, the transporter control unit overheated and resulted in a total loss of power to the transporter. The manufacturer of the control unit analyzed the problem and corrected it by providing a larger power supply and installing it in a well-ventilated section of the control chassis.
4. Various mechanical portions of the transporter are showing signs of wear after extended operation. Glass-filled Teflon bushings which were galling have been replaced with bronze bushings. The pinion gear driving the x-axis stepping motor was replaced because of visible wear. This wear was attributed to a misalignment of the transporter, which has been corrected.
5. One of the two constant-tension devices which maintain tension on the 20-conductor electrical cable as the transporter moves along the x-axis failed during the reliability tests. The device was replaced, and the cause of the failure was identified and corrected.

6. The sample cans occasionally became hung up as they entered a storage well due to the short lead-in guide on the well. Tapered sleeves were installed on each storage well to create an improved lead-in angle for the cans.

The maximum rate of movement of the transporter is fixed, but during the demonstration tests the transporter will operate less than 25% of the time. Only 10 movement cycles will be performed each hour during the demonstration tests although the transporter is capable of making 42 cycles per hour when operating continuously. The reliability test program maintained the transporter at its maximum movement rate. Following the correction of identified problems, the reliability program was run continuously for 140 hr. A total of 5928 can movements was made with no failures of can pickup or release. The test was terminated to allow for the continuation of software development.

As reported earlier [16], a decision was made to purchase and interface a microprocessor between the calorimeters and the central process computer. However, this will not be accomplished because of a lengthy delivery delay of the microprocessor by the manufacturer. As a result, a new programming technique is being used to obtain calorimeter data. Since the digital voltmeter used for collecting calorimeter data cannot be run as an interrupt device, the computer must continually check to see when a reading is to be taken. Therefore, the computer is dedicated to the digital voltmeter during the calorimeter data collection period. The new technique uses a computer executive command to mark time or delay for various time periods. During the mark-time delays, the computer is not dedicated to the calorimeter read

program and is, therefore, available for other operations such as sample transport or gamma-ray spectroscopy sampling. The new calorimeter read program collects data from the calorimeter every 60 sec which it stores in a common data block. These data are then converted from raw binary-coded-decimal bit patterns to floating point values which can be passed to the

FORTTRAN operating programs for storage or data calculations. Each of the two calorimeters is monitored on 1-min intervals but is offset from the other by 30 sec. This means the computer is monitoring a calorimeter every 30 sec. Utilizing the mark-time delay, 95% of the calorimeter read program has been regained for other computer operations.

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