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AT A MIXED-OXIDE FUEL FABRICATION FACILITY

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A SIMULATED PHYSICAL INVENTORY VERIFICATION EXERCISE
AT A MIXED-OXIDE FUEL FABRICATION FACILITY

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Abstract

A physical inventory verification was simulated at a mixed-oxide fuel fabrication facility. Safeguards inspectors from the International Atomic Energy Agency (IAEA) conducted the PIV exercise to test inspection procedures under "realistic but relaxed" conditions. Nondestructive assay instrumentation was used to verify the plutonium content of samples covering the range of material types from input powders to final fuel assemblies. This paper describes the activities included in the exercise and discusses the results obtained.

1. Introduction

A physical inventory verification (PIV) was simulated at the mixed-oxide (MOX) fuel fabrication facility operated by the Westinghouse Hanford Company (WHC) in Richland, Washington. The goal of this exercise was to simulate, insofar as possible and under "real but relaxed" conditions, all the activities of an actual PIV, including records audit, inventory stratification and sampling, and verification measurements. The exercise was developed under the auspices of the US Program of Technical Support to IAEA Safeguards (POTAS) and organized through the cooperative efforts of WHC, the International Atomic Energy Agency (IAEA), and Los Alamos National Laboratory. Personnel from Argonne National Laboratory and Mound Laboratory provided considerable assistance in developing and conducting the exercise, especially with the calibration and operation of the calorimeter.

PIV exercises were conducted in November 1983 and September 1984 and are expected to continue in the future. An extensive calibration exercise was conducted before each PIV exercise.

2. Description of Activities

The host facility, WHC, made available a nuclear material inventory containing over 88 kg of plutonium in a variety of forms as shown in Table I. Many of these materials were from the Fast Flux Test Facility program. To allow a records audit, WHC also prepared a complete material history including ledgers, shipping and receiving documents, and internal measurement and transfer documents.

The PIV exercise lasted 9 days. Two days were used for an introduction to the facility and for instrument setup, five days were used for actual PIV activities, and two days were used for data analysis to arrive at an overall conclusion regarding the plant inventory. Two teams of four IAEA inspectors conducted independent inspections for each exercise. The available measurement instrumentation included the High-Level Neutron Coincidence Counter (HLNC), Universal Fast Breeder

TABLE I

STRATIFICATION OF INVENTORY

Stratum No.	Description	Inspector ID	
		Team A	Team B
1	PuO ₂ powder >500 g	PP02	A1
2	PuO ₂ powder <500 g	PP01	A2
3	MOX powder - HEU	MOP0H	B1
4	MOX powder - LEU	MOP0L	B2
5	MOX pellets - HEU	MOPEH	B4
6	MOX pellets - LEU	MOPEL	B3
7	MOX fuel pins	pins	P
8	MOX assemblies	assy	A
9	In-process material	MOPI	F1
10	Scrap	MOPS	C1
11	HEU	HEU	A3

Reactor Fuel Assembly Counter (UFBR), Inventory Sample Coincidence Counter (INVS), 8k multichannel analyzer (SLNC), Plutonium Isotopic Analysis Microprocessor (PIAU), Bulk Calorimeter (CALR), and a variety of weighing instruments. Hewlett-Packard HP-85 computers were used to collect and analyze data from the neutron coincidence counters. Most verification measurements involved the combination of ²⁴⁰Pu(eff) measured by a neutron coincidence counter and plutonium isotopic information measured with a gamma-ray spectroscopy system. Outliers from these measurements were checked using the combination of calorimetry and gamma-ray spectroscopy. The calorimeter also was used to help with the initial calibration of the HLNC instruments.

3. Instrument Check-Out and Calibration

All instrumentation was calibrated and tested before each exercise. This section describes the calibration exercise performed during July 9-13, 1984. The measurement data from this exercise have been assembled and are available to any interested laboratory or person.

High-Level Neutron Coincidence Counter (HLNC)

The PIV exercise provided a unique opportunity to calibrate the HLNC for a number of different material categories. The IAEA decided to generate calibration curves for each category, both with and without multiplication corrections. This resulted in 15 calibration curves for each of 3 detectors. Each HLNC was connected to an HP-85 computer programmed to choose the appropriate curve for the item being measured.

At the IAEA's request, all calibration data were fit to an equation of the form

$$R = a + b \cdot m + c \cdot m^2 \quad (1)$$

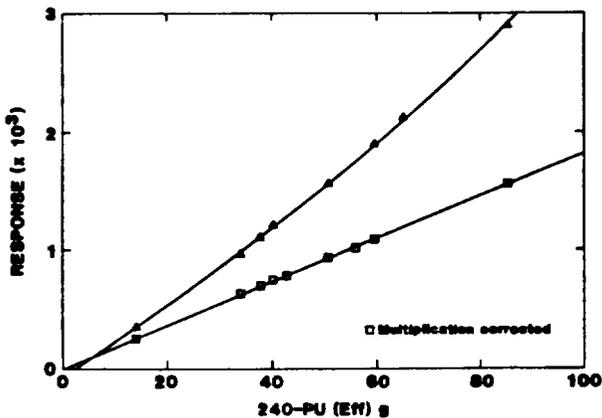


Fig. 1. HLNC calibration curve - MOX pellets.

where R = net coincidence response,
 m = $^{240}\text{Pu}(\text{eff})$ mass, and
 a, b, c = constants.

Figure 1 illustrate a typical calibration curve. There is still discussion among data analysts as to which functional form is the best, some analysts prefer a power law, and whether a nonzero intercept ($a \neq 0$) is physically reasonable.

The IAEA requested that all calibration points be within 2% of the final calibration curve. This requirement ensured that the systematic uncertainty due to the calibration would be less than 2%. A number of points were rejected because of this requirement. Out of 45 standards representing all material categories, 4 were rejected when the data were analyzed without multiplication correction and 9 were rejected when the data were analyzed with multiplication correction.

The physical causes for the deviations are being studied. Calorimeter values for the rejected samples indicate that the declared plutonium masses are correct. The most likely cause for the deviation is an anomalous (α, n) source from light element contamination, incorrect americium content, or additional moisture.

The INVS and UFBR were calibrated using the same procedure used for the HLNC.

Bulk Calorimeter - (CALR)

The IAEA bulk calorimeter⁴ is an isothermal instrument with an internal electrical calibration. Before the July 1984 calibration exercise, the calorimeter was calibrated over the power range of 0.9 to 6.3 W using four certified ^{238}Pu heat standards. The heat standards were measured in different positions to analyze the best distribution error in the calibration. The calorimeter power measurement exhibited a standard deviation of ± 0.018 W.

When combined with the plutonium isotopic composition measurement from the gamma-ray spectroscopy system, the calorimeter yields a measurement of total plutonium mass that is completely independent of operator data⁵. This measurement can be used to calibrate the HLNC. The accuracy of the plutonium mass determination by calorimetry and gamma-ray spectroscopy (CALR/

SLNC/PIAU) was tested during both the calibration exercise and the PIV exercise. On 13 samples containing more than 9 kg of plutonium, the average relative difference or bias between the CALR/SLNC/PIAU measurement and the declared mass was 0.05%. The precision of the CALR/SLNC/PIAU measurement was 0.71%, with the major component being the precision of the isotopic measurement.

Plutonium Isotopic Composition Measurement System (SLNC/PLAU)

This system contained a high-purity germanium detector coupled with a Silena Cicero 8k multichannel analyzer for collecting high-resolution plutonium spectra. These spectra were transferred to the PIAU, which analyzed the peaks for isotopic composition. The analysis technique is based on an intrinsic calibration and requires no additional calibration information. Gamma-ray spectra were collected in two or three separate systems that were identical except for count rate and count time.

Calibration of HLNC with Calorimetry and Gamma-Ray Spectroscopy (CALR/SLNC/PIAU)

The HLNC calibration curves for this exercise were calculated using operator data for plutonium mass and isotopic composition. It would have been possible to use calibration curves calculated from CALR/SLNC/PIAU plutonium mass data. Tables II and III illustrate this procedure. In Table II, an HLNC calibration curve is generated using calorimetry plus gamma-ray isotopics. In Table III, the HLNC calibration curve is used to verify unknown inventory items. The agreement in this example for the total of six items was $0.2\% \pm 2\%$. This illustrates that good results can be obtained using a calibration that is completely independent of operator data, albeit at the cost of longer measurement times.

4. PIV Exercise

The activities performed during the two PIV exercises were:

1. Calibration verification
2. Records and reports audit
3. Receipt of preliminary physical inventory listing
4. Establishment of inventory population
5. Stratification of inventory and establishment of sampling plan
6. Verification measurements
7. Calculation of operator-inspector difference
8. Discussion of anomalies with operator.

Calibration Verification

At the beginning of the PIV exercise, the participants were asked to verify the calibration curve for PuO_2 powder by remeasuring six items. The September HLNC responses agreed with the July values to better than 1%. Responses were then run through a Deming fit program to derive calibration parameters. The calibration verification enabled the inspectors to test the instruments and measurement procedures, learn the fitting

TABLE II
PLUTONIUM OXIDE CALIBRATION DATA, HLNC vs CALR

Mass Pu (g) ^a		²⁴⁰ Pu(eff) ^b (%)	HLNC-II Response (R) (Counts/s)
WHC	CALR		
346.8	349.7	11.96	1079.4 ± 5.4
443.9	447.5	19.22	2439.8 ± 15.0
556.1	562.4	11.83	1812.4 ± 7.7
573.9	577.6	18.85	3095.2 ± 15.3
641.3	645.5	19.44	3701.8 ± 17.7
666.0	666.3	12.27	2522.3 ± 12.6
852.9	855.0	12.10	3187.5 ± 13.5
922.6	925.0	11.70	3381.8 ± 12.9
1076.7	1080.0	12.07	4168.9 ± 16.2

^aTotal plutonium mass in grams. WHC is operator-declared value. CALR is value determined by combining calorimeter heat measurement with PIAU determined isotopic composition.

^bThe effective ²⁴⁰Pu fraction in per cent as measured by the PIAU. This is multiplied by the CALR mass to give effective ²⁴⁰Pu mass (²⁴⁰m).

RESULTS OF DEMING FIT PROCEDURE:

$$R = a_1(240_m) + a_2(240_m)^2$$

$$a_1 = 23.80 \pm 1.55$$

$$a_2 = 0.0589 \pm 0.0159$$

$$\text{cov} = -0.0236$$

$$r^2 = 0.9803$$

$$S = 553.7$$

TABLE III
PLUTONIUM OXIDE CALIBRATION TEST RESULTS

Declared Mass (g)	Measured Mass ^a (g)
49.9	47.3 ± 3.7
98.7	97.5 ± 6.8
218.2	216 ± 13
218.2	217 ± 15
650.5	665 ± 29
867.7	855 ± 35
2103.2	2098 ± 50

^aTotal measured plutonium mass determined by dividing the effective ²⁴⁰Pu mass measured by the HLNC-II (using CALR plus PIAU calibration) by the effective ²⁴⁰Pu fraction measured by the PIAU.

procedures, and gain confidence in the calibration curve.

Records and Reports Audit

Two activities were required to audit the records prepared by WHC. The first was to compare a previous IAEA report with the facility ledger for the period June 1-30, 1984; the comparison was made to verify that facility records had not been altered after an earlier inspection. The second was to audit the facility books for the period August 1-September 9, 1984. The ledger (Inventory Change Report format) was compared with the relevant source documents. The available source documents included the DOE-741 (US material transfer document) and internal WHC shipping and receiving reports. Results were summarized on a log sheet and any discrepancies were discussed with the operator.

The inspectors consider this audit to be an important part of a PIV and they perform this activity carefully.

Receipt of Preliminary Physical Inventory Listing

The physical inventory listing (PIL) is given to the inspectors by the operator declaring

the facility inventory. It usually is presented when the inspectors begin the records and reports audit.

Establishment of Inventory Population

All items in the vault were checked against the PIL. The PIV-exercise inventory contained approximately 150 items, a relatively small inventory compared to the inventory present in some facilities inspected by the IAEA. Some teams chose to check the mass information on the tag against the PIL listing. This check was thought to be a more complete verification but it was noted that time constraints may not permit this activity in larger facilities.

After all the items were checked, the inventory was sealed. Working papers were provided to record the item check results and sealing activities.

Stratification of Inventory and Establishment of Sampling Plans

The teams stratified the inventory as shown in Table I. They then determined sample sizes for the following three cases.

- Gross defect: 50-100% of an item missing. Use HLNC and operator-declared gamma-ray isotopics.
- Medium defect: 10-50% missing. Use HLNC and inspector-measured gamma-ray isotopics.
- Small defect: 1-10% missing. Use calorimetry and gamma-ray isotopics or sample for chemical analysis.

The Inspectors selected the individual items from the PIL using a random number generator.

Verification Measurements

The verification measurements continued for 5 days. Initially the teams used the operator-declared isotopics to analyze the neutron data. Later they reanalyzed these data with gamma-ray isotopics.

Team A made their gamma-ray measurement through the walls of the HLNC so that they could collect neutron and gamma-ray data simultaneously. This procedure limited the precision of the isotopic determination because the sample-to-detector distance limited the available count rate. Team B used a separate measurement system to collect gamma-ray data in a geometry that allowed them to obtain a higher count rate than Team A. Both teams limited the total count time to 20-30 min, which is the time available during actual inspections.

A third gamma-ray spectroscopy system measured spectra through the walls of the calorimeter preheater. Because the calorimeter measurements take 3 to 4 h, it was possible to count these samples for a much longer time. The samples were counted long enough to obtain a 1% precision on the specific power value; count times of 1-1/2 h (3600 s, live time) were sufficient.

The calorimeter was used for the small defects measurements and to resolve anomalies that

might occur between HLNC measurements and the operator's declared mass. The HLNC results are influenced by the presence of certain impurities, which cause abnormal (a, n) emission, and by the presence of abnormally high moisture content. If an HLNC measurement differed from the operator's value by more than a few per cent, that item was measured in the calorimeter to verify the discrepancy. Table IV is a tabulation of such outlier results compared to subsequent calorimetry verification. This table shows four cases where the calorimetry measurement resolved an apparent discrepancy from an erroneous HLNC measurement. In this way an inspector can make a number of measurements with the faster HLNC (20 min per sample) and check anomalies with the slower (3-4 h), but more accurate, calorimeter. The combination of the two NDA methods based on different principles gives high credibility to final value.

Using the HLNC calibration curves determined in July 1984, the two teams measured a total of 66 items. Table V summarizes the results one team obtained measuring 11 items from a PuO₂ powder stratum. The team decided item 46982 was an outlier and measured it in the calorimeter to resolve the anomaly. If item 46982 is not included, the average mass difference is $-0.22 \pm$

TABLE IV

CALR MEASUREMENTS TO RESOLVE
HLNC-OPERATOR OUTLIERS

ID	Pu Total (g)		
	Operator	HLNC	CALR
46982	666	706 ± 9	667 ± 5
61	561	491 ± 17	561 ± 9
21182	562	731 ± 13	524 ± 6
12340	518	452 ± 15	524 ± 6

TABLE V

HLNC MEASUREMENT RESULTS

ID	Plutonium Mass (g)		
	Operator	Inspector ^a	Inspector ^b
5123	647	650	658
5108	1086	1091	1025
5110	655	635	584
5114	764	770	762
56421	857	863	813
605902	556	557	539
46982	666	706	750
53491	610	618	573
5596	875	875	882
20242	833	835	771
5598	873	880	842
	<u>8422</u>	<u>8480</u>	<u>8199</u>

^aHLNC response plus operator-declared isotopic composition.

^bHLNC response plus inspector-measured isotopic composition.

TABLE VI

HLNC MEASUREMENTS USING HIGH-PRECISION
GAMMA ISOTOPIC VALUES
(CALR System)

Item	Operator	Pu Total (g)	
		HLNC-4	HLNC-II
1	556	550 ± 4	562 ± 3
2	651	658 ± 7	648 ± 3
3	868	867 ± 8	868 ± 4
4	924	903 ± 7	915 ± 3
5	1078	1116 ± 14	1138 ± 5
	4077	4094 ± 19	4131 ± 6

1.23% using the operator-declared isotopic composition.

Clearly the short gamma-ray measurements limit the precision of the results shown in the last column of Table V. If it is feasible to count for longer times (1-2 h), a 1-2% precision can be obtained for plutonium mass (see Table VI). For 1/2-h count time and close geometry, 2-4% precision can be achieved.

5. Calculation of Operator-Inspector Difference

Most of the PIV activities are summarized on a working paper shown in Fig. 2. A complete description of the information contained on the form is given in Ref. 3. The form shown in Fig. 2 is illustrative of those produced during the two PIVs. Column 1 shows how the inventory was stratified; column 2 gives the amount of plutonium in each stratum. As can be seen in the third column, 28 items were sampled from the total inventory. The remaining six columns report the measured difference between operator-declared and inspector-measured values and estimate the uncertainty in that difference. With this information, the inspectors can look at each stratum and anomalously large differences. The ANOVA (analysis of variance) table combines the values from the individual strata to test whether they have consistent variances.

The data analysis continues by calculating the total difference for the entire inventory and an estimate of the uncertainty in the difference. In the example shown in Fig. 2, the inspection team found a difference of -56.67 ± 331 g of plutonium out of a total inventory of 74289 g or 0.08%.

The PIV summary shown in Fig. 2 represents a systematic set of inspection procedures starting with stratification, continuing through verification measurements, and finishing with data analysis. It helps the inspector reach a decision on the validity of the operator-declared inventory.

6. Discussion of Anomalies with Operator

At the conclusion of the PIV activities, the two teams met with facility staff to discuss the findings. The inspectors had detected some anomalies with the operator's declaration. A few errors were found in the records and two items

were found to have incorrect values. One had the wrong material type and the second the wrong mass value.

Overall, both teams reached the conclusion that the differences between operator-declared and inspector-measured inventory were neither greater than the IAEA goal quantity nor statistically significant. Therefore, the operator's declared inventory was verified by Independent IAEA inspection.

7. Concluding Remarks

The two PIV exercises have been enthusiastically received by all parties. The instrument developer has the opportunity to observe the operation of NDA instruments under conditions that very closely simulate those found during an actual PIV. The IAEA inspector has the opportunity to test these instruments under actual field conditions and in a relaxed, friendly atmosphere. In such conditions, the inspector can more readily gain confidence in the reliability of his measurement. These exercises offer a unique opportunity for all parties to interact and participate in the process of developing reliable measurement techniques and procedures for safeguards inspections.

Two PIV exercises have been completed; a third is scheduled during the fall of 1985. Similar exercises for high-enriched uranium fuel fabrication and light water reactors are also being conducted under the auspices of POTAS.

The success of these PIV exercises was brought about only by the efforts of many people in all of the participating organizations. W. DeMerschman served as the exercise coordinator for WHC. D. Engle, M. Serier, J. Brunke, B. Woehle, A. Mah, R. Rowley, A. DuPuis, and L. Southam of WHC all helped greatly with the exercise.

R. Perry of Argonne National Laboratory, W. Stroh of Mound Laboratory, and S. Fiarman of the IAEA were responsible for the very successful operation and performance of the bulk calorimeter.

The participation of D. Atencio, W. Atencio, M. Hykel, M. Krick, J. Halbig, P. Rinard, and H. Menlove of Los Alamos National Laboratory is gratefully acknowledged. Their help was essential in all parts of the exercises.

W. Alston served as the exercise coordinator for the IAEA and prepared much of the exercise workbook. J. Fager helped train the participants in instrument operation before they came to Richland. The 15 inspectors who participated in the two exercises were the ultimate audience and actors of the play. The effort spent in preparing and presenting these exercises was rewarded by the inspectors' cooperation and enthusiasm.

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