Comparison of NDA and DA Measurement Techniques for Excess Pu Powders at the Hanford Site: Operator and IAEA Experience

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ABSTRACT

Quantitative physical measurements are necessary components of the International Atomic Energy Agency (IAEA) nuclear material safeguards verification regime. In December 1994, IAEA safeguards were initiated on an inventory of plutonium-bearing oxide and scrap items in Vault 3 of the 2736-Z Building of the Plutonium Finishing Plant on the United States Department of Energy's (USDOE) Hanford Site. The materials originated in the United States nuclear weapons complex. The diversity of the chemical form and the heterogenous physical form of the plutonium in this inventory were expected to challenge the target precision and accuracy of methods employed by IAEA: quantitative destructive analytical techniques (which are susceptible to sampling error) and quantitative coincident neutron measurements (which rely on knowledge of the material's chemical form and purity). Because of the diverse and heterogenous nature of plutonium-bearing scrap, plant operations increasingly have adopted calorimetric techniques both for item inventory measurements and for verification purposes. During the recent advent of IAEA safeguards at Vault 3, a set of destructive and non-destructive methods were applied to a number of inventory items (cans of plutonium-bearing powders) with widely ranging chemical purities. Results of these measurements, gathered by the operator's and IAEA's laboratories and instruments as well as by instruments from Pacific Northwest Laboratory and USDOE's Los Alamos National Laboratory (LANL), are presented and statistically compared.

INTRODUCTION AND OBJECTIVES

In December 1994, international nuclear materials safeguards were initiated by the International Atomic Energy Agency (IAEA) on an inventory of over 500 plutonium-bearing oxide and scrap items at Vault 3 of the 2736-Z Building of the Plutonium Finishing Plant (PPF) on the USDOE's Hanford Site in Washington State. These materials, originating from the United States nuclear weapons complex and offered under a United States presidential initiative to promote nuclear weapons nonproliferation, represent the first plutonium placed under IAEA safeguards at a former weapons site.

A variety of nuclear materials measurement techniques were employed by the facility operator (Westinghouse Hanford Company (WHC)), the State system (the USDOE), and the IAEA to determine and verify the quantities of plutonium present in the safeguarded inventory. In support of the IAEA goals, the Los Alamos National Laboratory (LANL) provided an advanced plutonium measurement system (Three-Ring Multiplicity Counter [3RMC]) whose operation is based on detection and counting of multiple coincident neutron events.

Plutonium measurement objectives and results obtained by the operator, State, IAEA, and LANL, pursuant to the initial submission are presented and compared. The relative performances of destructive and non-destructive analyses for the operator/USDOE/IAEA/LANL systems are described.

THE SAFEGUARDED PLUTONIUM

The plutonium materials offered for IAEA safeguards originate from many processes and possess a variety of textures (particle size and homogeneity), chemical forms, and impurities. Because the physical and chemical properties can affect the reproducibility and accuracy of certain sampling and measurement methods, care must be taken to select robust methods appropriate to the material diversity. In many cases (e.g., scrap or material received from off-site), the operator has incomplete knowledge of the process history and origin of the inventory materials.

The plutonium (Pu) materials in the initial offering for IAEA safeguards ranged in concentration from 40 to 92 wt% (compared with 88.2 wt% for stoichiometric plutonium dioxide [PuO₂]). No discrete Pu metal items were in the initial offer.

The purest materials, which would be closest to the stoichiometric PuO₂ composition, originated from calcination of Pu oxalate and from burning of Pu metal. Less pure (scrap) materials in the safeguarded inventory, containing compounds in addition to PuO₂, originated from various sources in the weapons complex: Pu metal production (containing PuO₂, PuF₄, MgO, CeO); pyrochemical refining (Pu metal, PuOCl, halide salts); glovebox sweepings (PuO₂, rust, debris); and ash from incineration of Pu-bearing materials (filters, rags, gloves). Analyses have shown that ash residues can contain Al, B, Ca, Cr, Fe, Pb, Mg, Si, Ti, and Zn besides Pu.

MEASUREMENT PERSPECTIVES AND OBJECTIVES

The operator, State system, and IAEA have different perspectives and objectives that influence their selections of Pu measurement methods. Operator Pu measurement interests are in criticality safety, material production and quality, and nuclear material control. The USDOE and IAEA, as regulatory bodies, are concerned with verifying the accuracy of declared nuclear material inventories.

From the operator's perspective, scrap items rarely warrant the radiation exposure and expense of chemical analysis. Appreciable radiation exposure occurs in item retrieval, repackaging, and sampling. The heterogenous scrap materials are impossible to sample accurately without intense homogenization and splitting. Non-
destructive analyses (NDA; including gamma ray isotopic and calorimetric measurements) of entire inventory scrap items are employed instead. Weighing and precise destructive analyses (DA; by mass spectrometry and amperometric titration) generally are reserved for pure product materials.

The domestic (State system) safeguards verify, using NDA, inventory item quantities stated by the operator. Most measurements are made with the High-Level Neutron Coincidence Counter (HLNC) on randomly selected items. These measurements are not independent, but are based on multiple material-specific calibration curves obtained from repeated measurements of inventory items. Verification of samples taken from inventory items by weighing and by DA to determine Pu isotopic compositions and chemical concentrations also may be performed by other USDOE laboratories.

The IAEA must independently verify the accuracy of nuclear material inventory declarations provided by the State for its facilities. To perform this task uniformly over the various facility and material types to the satisfaction of the world community, the IAEA has created a verification system that includes a statistical sampling logic, a hierarchy of increasingly precise verification measurements and associated instrumentation, and a process of systematic data analyses. The results produced by the IAEA are quantitative statements on the operator’s declared inventory, the inventory as verified by the IAEA (based on IAEA measurements of inventory items), the differences of the two inventory values, and the significance of the difference.

IAEA physical verifications of bulk Pu containers begin with on-site tests of inventory items, and include:

1. Item counting (assuring the presence of the items)
2. Qualitative NDA checks of material radiological properties (e.g., gamma spectra or neutron emission)
3. Quantitative NDA to determine Pu mass (i.e., high-resolution gamma spectrometry [HRGS], in conjunction with coincident neutron NDA); the HLNC is used to measure large items, whereas the Inventory Sample Counter (INVS) is used to measure small items or subsamples.

Because of the high safeguards significance of separated Pu, quantitative DA of Pu mass also is required for selected items. The DA tests begin with on-site measurement of total item mass (using IAEA balances or operator balances validated by the IAEA). Samples are sent to the IAEA’s Network of Analytical Laboratories (NWAL) through the Safeguards Analytical Laboratory in Seibersdorf, Austria, where more precise and accurate Pu isotopic and chemical concentration analyses are performed.

PLUTONIUM SCRAP MEASUREMENT APPROACHES

The diverse and heterogenous nature of Pu-bearing scrap has caused the operator increasingly to adopt calorimetric techniques, in combination with Pu gamma isotopic analysis, for shipper/receiver and item inventory measurements. The operator, State system, and the IAEA have found coincident neutron NDA of scrap inventory items using the HLNC to be unreliable because knowledge and assurance of sample purity (e.g., as pure PuO₂) are required (Ensslind). Nevertheless, USDOE and IAEA NDA measurements for Pu-bearing powders are centered on the HLNC, with DA sampling and analyses being necessary complementary measures. IAEA verification methods do not currently include calorimetry.

For IAEA purposes, improved verification of Vault 3 inventory requires development of quantitative NDA techniques for impure as well as heterogenous Pu-bearing scrap. The IAEA used two neutron based Pu scrap measurement devices, the INVS and the 3RM, to complement the HLNC during the initial verification.

Measurements with the INVS were performed to determine if the limitation due to the unknown composition, and resulting neutron self-multiplication, of the scrap could be overcome by use of a sufficiently small sample. Measurements by INVS also were performed to determine if sufficient accuracy could be obtained to reduce DA requirements for the IAEA.

The 3RM, like the HLNC, measures single- and two-neutron coincident events but can also measure three-neutron coincident events (triples). Because the 3RM can measure triples, neutron self-multiplication factors can be determined for each item and knowledge of the Pu chemical form or purity is not required, thus allowing impure and heterogenous Pu scrap to be measured (Stewart et al.). A neutron multiplicity counter, similar to the 3RM, has been used successfully by the IAEA to verify containers of impure PuO₂ at a plutonium-uranium mixed-oxide fuel fabrication plant. At the request of the IAEA, the USDOE and LANL provided a 3RM for the initial physical inventory verification at Vault 3.

MEASUREMENT PERFORMANCE

To evaluate the performance and utility of the various analytical techniques, a set of comparative measurements was made by the operator, the USDOE, LANL, and the IAEA on 17 inventory items (11 consisted of pure PuO₂ materials and were designated stratum PD by the IAEA; six were considered scrap material and designated stratum SC). These items were selected from the initial inventory offer of over 500 items. The measurement methods and the number of items measured by each analytical method are listed in Table 1.

The measurement results, in terms of relative difference, are listed in Table 2. The IAEA calculates relative differences as follows:

\[
\text{Rel. Diff. (\%)} = \frac{(\text{Declared} - \text{Measured})}{\text{Declared}} \times 100.
\]

Possible discrepancies in item declarations are identified by the IAEA based on comparison of these relative differences with the variability estimates derived from IAEA measurement experience for the same techniques and similar materials. Results from weighing and isotopic analyses generally agreed between the operator and the IAEA and will not be considered explicitly here.
TABLE 1. VERIFICATION METHODS APPLIED TO THE SAFEGUARDED INVENTORY.

<table>
<thead>
<tr>
<th>Measurement Method</th>
<th>Measurement Source (number of samples)</th>
<th>IAEA</th>
<th>WHC</th>
<th>USDOE</th>
<th>LANL</th>
</tr>
</thead>
<tbody>
<tr>
<td>DA Weight</td>
<td>EBAL * (17)</td>
<td>EBAL (17)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Isotopic</td>
<td>TIMS * (13)</td>
<td>Mass spec. (17)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical</td>
<td>Titration * (13)</td>
<td>Titration * (17)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sol'n. counter * (1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NDA Gamma</td>
<td>HRGS (17)</td>
<td>HRGS (17)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Neut. Conc.</td>
<td>HLNC (11)</td>
<td>HLNC (10)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>INVS (5) 3RM C (16)</td>
<td>3RM C (16)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calorimetry</td>
<td>Calorimeter * (17)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Notes:
* EBAL: Facility Electronic Balance
* TIMS: Thermal Ionization Mass Spectrometer
* Macdonald and Savage
* ASTM C698
* Solution counter: gamma spectrometric Pu isotopic and concentration analysis
* ANSI N15.22-1987

COMPARISON OF RESULTS

Identification of a Discrepant Inventory Item

As shown in Table 2, the declared mass value stated for item PD1 was inconsistent with measurements by the operator DA, the operator calorimeter, the IAEA DA, the USDOE HLNC, and the LANL/IAEA 3RM C. Each of the five separate measurements identified the bias and all closely agreed. Operator inspection of supporting records showed the inconsistency arose from inappropriate use of neutron-well NDA confirmation measurement data for items PD1 through PD8. Shipper data ordinarily are used in this situation. The results based on shipper data for the items PD1 through PD8, indicated within the ( ) in Table 2, generally showed lower relative differences for all measurement techniques.

Comparison with the Operator DA Measurements

Destructive analyses, normally considered the most accurate and precise for quantitative Pu measurements, were performed for all 17 items by the operator laboratory. These results were compared with those obtained by the IAEA NWAL and by the various NDA methods. The comparisons were made by calculating the relative differences as in equation (1) but substituting the operator DA values for the declared values. The results are illustrated in Figure 1 for the PD stratum items and in Figure 2 for the SC items.

Operator DA vs. IAEA DA

The relative differences of the thirteen items measured by both operator and IAEA DA ranged from -0.2% to 0.3% for the PD stratum and from -2.3% to 3.5% for the SC items. The average relative difference for the eight PD items is 0.04% with a standard deviation of 0.19%; for the five SC items, the average relative difference is 1.68% with a standard deviation of 2.58%. Though the operator and IAEA DA results are similar, the higher variability of the SC materials is evident and is attributed to material heterogeneity (Welsh et al. 7).

Operator DA vs. Calorimetry

The operator has adopted non-destructive calorimetry for routine analyses of inventory items due to its accuracy, precision, and elimination of the expense and exposure required for destructive sampling and analysis. Destructive analysis also suffers from sampling variability caused by the heterogeneity of Pu-bearing materials. This variability, particularly for scrap, can be significantly larger than the variability associated with the DA method itself (Welsh et al. 7).

On this basis, it is useful to compare the operator DA results to the calorimeter assay results for each item. The relative differences between the operator DA results and the calorimetry results were calculated for each item and are listed in Table 2. The relative differences for 14 of the 17 items were less than 1%; 13 were less than 0.5%. These data are well within the variability associated with the measurement methods. The average relative difference for the 11 PD items is 0.16% with a standard deviation of 0.35%. For the five SC items (eliminating item SC6) the average relative difference is 1.47% with a standard deviation of 1.90%. Again, the SC results show higher variability than the PD results.

Cr and Mn Interferences for Item SC6

When samples from item SC6 were destructively analyzed by the operator (no IAEA NWAL DA was performed), Cr and Mn interferences were identified that produce a high bias in the amperometric titration method (ASTM C698 4). Dissolved samples of this item subsequently were analyzed by a quantitative gamma spectrometric method (solution counter, see Tables 1 and 2) that does not suffer from chemical interferences. The solution counter results have a smaller relative variability, particularly for scrap, that is attributed to material heterogeneity (Welsh et al. 7).

Operator DA vs. Neutron NDA

Relative differences between the operator DA and the HLNC-USDOE, HLNC-IAEA, 3RM C-LANL/IAEA, and INVS-IAEA also were calculated. As expected, the PD materials have lower relative differences than the SC materials.

HLNC-USDOE: The relative differences for the ten items measured by both the operator DA and the HLNC-USDOE ranged from -1.2% to 7.1% for the PD stratum and -15.8% to 5.7% for the SC stratum. The average relative difference for the seven PD items is 0.87% with a standard deviation of 2.84%; for the three SC items, the average relative difference is -2.20% with a standard deviation of 11.80%.
HLNC-IAEA: The relative differences for the eleven items measured by both the operator DA and the HLNC-IAEA ranged from -3.3% to 5.2% for the PD stratum; HLNC-IAEA did not measure the SC items. The average relative difference for the eleven PD items is 0.38% with a standard deviation of 2.71%.

3RMC-LANL/IAEA: The relative differences for the sixteen items measured by both the operator DA and the 3RMC-LANL/IAEA ranged from -3.6% to 3.2% for the PD stratum and -34.6% to 8.2% for the SC stratum. The average relative difference for the ten PD items is -0.38% with a standard deviation of 2.08%; for the six SC items, the average relative difference is -9.06% with a standard deviation of 14.29%. If the result for SC3 is not used, the average relative difference is -5.94% with a standard deviation of 7.68%.

INVS-IAEA: An INVS sample was obtained from five of the 17 items. The four PD items had a relative difference of approximately -4%. The single SC item had a relative difference of approximately -20%.

CONCLUSIONS

INVS Results

The initial results for the INVS measurements, based on a limited amount of data (five samples), were not promising. Verification by INVS still requires subsampling and weighing. Unless future tests indicate an improvement in performance, the use of the INVS is not recommended either as a replacement for NDA of whole items or for partial replacement of DA.

DA and Calorimetry

As shown in this and the related paper (Welsh et al.), if the variability due to heterogeneity is incorporated with the DA analytical variability, the DA uncertainty is no better than the uncertainty in the calorimetric measurements. Destructive analyses of scrap also are subject to the effects of interferences, the presence of which are generally unknown. Without prior purification steps (which themselves contribute to measurement variability), chemical assay can give unreliable results for impure items. For highly impure and heterogenous scrap materials with possible electrochemical interferences (e.g., Mn, Cr, V, Pb), dissolution and plutonium solution counting, by gamma spectrometry, may be attractive. However, sampling problems would still exist.

The difficulty, exposure, expense, and unknown interferences of destructive analyses, along with the demonstrated accuracy of calorimetry, make calorimetry the operator’s method of choice for accountability of Pu materials in storage. Use of the operator’s calorimeter by the IAEA as an alternative method for quantitative Pu NDA (particularly for scrap) may warrant further development. Steps are being taken to allow the operator and IAEA shared access to a calorimeter controller. At present, the IAEA has no means for independent validation of the operator’s calorimeter systems.

HLNC Results

The USDOE HLNC gave acceptable results for analysis of the purer PuO₂ materials (stratum PD) and for the few impure scrap items (stratum SC) measured. The IAEA HLNC gave acceptable results for analysis of the stratum PD items; the IAEA did not measure SC items by HLNC.

3RMC Results

For the purer PuO₂, the 3RMC required shorter counting times (about 30 minutes) than the calorimeter (about six hours). For certain highly impure scrap items, however, the 3RMC required longer counting times than the calorimeter without necessarily providing a more accurate result. In the IAEA’s view, the 3RMC was satisfactory in verifying high-quality PuO₂ (PD stratum) and most Pu-bearing scrap (SC stratum).

The IAEA has experience with an instrument similar to the 3RMC. The 3RMC can be readily validated by use of IAEA standard neutron sources. Further improvements in 3RMC performance are desired, particularly for the problematic highly impure scrap (e.g. item SC3).

Importance of DA to the IAEA

Finally, it is emphasized that despite the utility of the various NDA methods, sampling and DA provide fundamental assurances of bulk material quantities and thus remain necessary components of the IAEA verification requirements.

REFERENCES


### TABLE 2. MEASUREMENT RESULTS.

<table>
<thead>
<tr>
<th>Item ID</th>
<th>Declared (Shipper Data)</th>
<th>Relative Differences (%) Between the Declared Values and the Measured Values</th>
<th>Relative Differences (%)</th>
<th>WHC CA a vs. WHC CAL</th>
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</thead>
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<tr>
<td></td>
<td>BS g Pu</td>
<td>AS g Pu</td>
<td>WHC DA</td>
<td>IAEA DA</td>
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<tr>
<td>PD1</td>
<td>250</td>
<td>246</td>
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<tr>
<td></td>
<td>(271)</td>
<td>(266)</td>
<td>(0.4)</td>
<td>(0.0)</td>
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<td>PD2</td>
<td>473</td>
<td>467</td>
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<td>-0.6</td>
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<tr>
<td></td>
<td>(476)</td>
<td>(470)</td>
<td>(0.0)</td>
<td>(0.0)</td>
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<td>PD3</td>
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<td>859</td>
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<td>-0.3</td>
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<td></td>
<td>(870)</td>
<td>(864)</td>
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<td>(864)</td>
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<td>(0.1)</td>
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<td>PD5</td>
<td>882</td>
<td>870</td>
<td>1.0</td>
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<td>(873)</td>
<td>(861)</td>
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<td></td>
<td>(872)</td>
<td>(860)</td>
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<td>(869)</td>
<td>(858)</td>
<td>(-0.3)</td>
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<td>863</td>
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<td></td>
<td>(874)</td>
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<td>SC5</td>
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<td>SC6</td>
<td>284</td>
<td>275</td>
<td>-5.8</td>
<td>0.7</td>
</tr>
</tbody>
</table>

Notes:

- BS: Before item was sampled.
- AS: After item was sampled.
- *: Items measured before the sampling occurred.
- CA: WHC amperometric titration (ASTM C698*).
- CAL: WHC calorimeter measurement (ASTM C698*).
- [(CA-CAL)/CA]*100
- Relative differences using the shipper’s data for the declared value instead of the neutron-well data.
FIGURE 1. RELATIVE DIFFERENCES - STRATUM PD BASED ON OPERATOR WHC DA.

FIGURE 2. RELATIVE DIFFERENCES - STRATUM SC BASED ON OPERATOR WHC DA.
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