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Comparison of Calorimetry and Destructive Analytical Measurement Techniques for Excess Plutonium Powders

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Pacific Northwest National Laboratory, Richland, Washington, USA

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COMPARISON OF CALORIMETRY AND DESTRUCTIVE ANALYTICAL MEASUREMENT TECHNIQUES FOR EXCESS PLUTONIUM POWDERS

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ABSTRACT

In December 1994, International Atomic Energy Agency (IAEA) safeguards were initiated on an inventory of plutonium-bearing materials, originating from the U.S. nuclear weapons complex, at vault 3 of the United States Department of Energy’s Plutonium Finishing Plant (PFP) on the Hanford Site. It was anticipated that the diverse and heterogenous forms of the plutonium would challenge the target precision and accuracy of verification methods employed by IAEA: destructive analyses (susceptible to sampling error) and coincident neutron measurements (requiring knowledge of material form, purity, and appropriate calibration materials). Because of the diversity and heterogeneity of the plutonium, plant operators increasingly have used calorimetry for accountability measurements. During the recent commencement of IAEA safeguards at vault 3, destructive (electrochemical titration) methods were used to determine plutonium concentrations in subsamples of inventory items with widely ranging chemical purities. The concentrations of plutonium in the subsamples were determined and the contribution of heterogeneity to total variability was identified. Measurement results, gathered by the PFP and IAEA laboratories, showed total measurement variability for calorimetry to be comparable with or lower than those of sampling and chemical analyses.

INTRODUCTION

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 (PFP) on the United States Department of Energy (USDOE) Hanford Site in Washington State. The PFP is operated for the USDOE by the Westinghouse Hanford Company (WHC). 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 (WHC) and the IAEA to determine and verify the quantities of plutonium present in the safeguarded inventory. Sampling and analysis of a variety of plutonium-bearing items from the IAEA-safeguarded inventory were performed during the initial physical inventory verification (IIV). Calorimetric measurements were performed for each of the sampled items. Additional items, not under IAEA safeguards at the time of sampling, have been measured (sampling and chemical analysis along with calorimetry) since the IIV. The results of these analyses are statistically evaluated in this report.

THE SAFEGUARDED PLUTONIUM

The dry, powdered plutonium materials present at PFP originated from many processes and sources and thus 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. Though the operator has thorough knowledge of process history and origin of Hanford site-generated material, information on the genesis and composition of scrap or material received from offsite is less exact or detailed. It was anticipated that the diversity of the
chemical forms and the heterogenous physical forms of this inventory would challenge the precision and accuracy of quantitative destructive analytical (DA) techniques.

The purest materials, which would be closest to the stoichiometric PuO₂ composition, originated from calcination of plutonium oxalate and burning of plutonium metal. Though these materials are both nominally PuO₂, the burned metal was heated to higher temperatures than the oxalate and has correspondingly larger sintered particle sizes, higher plutonium concentration, and less (surface adsorbed) water. Residual unburned metal also may be present as a source of both higher plutonium concentration and material heterogeneity. These materials are at or near product quality.

Less pure scrap materials present in the safeguarded inventory originated from many sources. For example, hydrofluorination of PuO₂ was used to produce PuF₄, that then underwent calciothermic reduction in MgO crucibles to form plutonium metal. The presence of PuF₄, CaO, CaF₂, and MgO in addition to non-stoichiometric PuO₂ and plutonium metal in some scrap materials therefore is possible. Other pyrochemical operations to purify plutonium involved molten halide salts and plutonium metal. Scrap from these processes may contain salts (e.g., NaCl), beads of plutonium metal, and other plutonium compounds (e.g., PuOCl). Process glovebox sweepings, another scrap material, contain spilled plutonium powders and also include other dusts and glovebox debris. Scrap ash arises from incineration of gloves, filters, rags, plastic, and paper. The ash residues can contain Al, B, Ca, Cl, Cr, F, Fe, Pb, Mg, Si, Ti, and Zn as well as the Pu.

The plutonium powders are packaged in a nested set of three metal cans. The inner cans are 9 cm in diameter and can be either 9 or 18 cm high. The cans are generally filled to leave approximately 1.5 cm of space at the top of the can.

**SAMPLING, ANALYSIS, AND RESULTS**

The sampling procedure used in this investigation was designed to (1) provide a representative sample of the item for destructive analysis and (2) determine the degree of heterogeneity within the item. The first step in the sampling procedure was to obtain a core sample from each of five randomly selected locations within the item’s inner can. Sampling locations were determined by a perforated circular template that was placed on the top of the item container. The perforations were centered at five randomly selected points on the template.

For the items selected for heterogeneity testing, each core sample was crushed, mixed, and then poured into a cone-shaped pile on the sampling platform for further splitting. The cone-shaped pile was flattened with a spatula and divided into halves. One of the halves was added to a composite of the core samples for each item. The other half was divided into two equal portions, each portion was placed in a separate vial and sent to the PFP Analytical Laboratory for analysis. The splitting scheme for each core sample is shown in Figure 1. This cone-and-quarter technique, which is used in soil and ore analysis, is designed to produce representative samples of materials (such as soils) having a range of particle densities and sizes. Each of the ten aliquots (five locations, two samples per location) obtained by the cone-and-quarter method was then analyzed using amperometric titration and each titration was performed twice.

For the items not selected for heterogeneity testing, a composite sample was prepared for each item using the material obtained from each of the five core samples. Each composite sample was crushed, mixed, and then split according to the scheme shown in Figure 2 (cone-and-quarter technique). Two quarter-sample aliquots were further divided in half (eighths). Two of these subsamples were analyzed in duplicate using amperometric titration and mass spectrometry by the PFP Analytical Laboratory. Aliquots were prepared from the remaining one-eighth subsamples for IAEA and USDOE analysis. An archive sample was also prepared from a portion of the item’s composite sample. The archive sample is available for analysis to resolve possible differences between the PFP Analytical Laboratory chemical analyses and the IAEA chemical analyses. Any remaining material (i.e., not placed in sample vials for chemical analysis) was returned to the container from which it was originally taken.
Seventeen inventory items were selected by the IAEA for sampling during the IPIV; eleven items were product quality PuO$_2$ materials; six items were considered scrap material. Because of time constraints during the IPIV, only one of the seventeen items was actually sampled for heterogeneity testing. Six additional items, five of which are now under IAEA safeguards, were selected from the PFP inventory and sampled by WHC for heterogeneity testing. Samples from these six additional items were not taken for IAEA destructive analyses.

**Item Selection for Heterogeneity Testing**

The objective of item selection for heterogeneity testing was to include items representative of the broad range of material properties under IAEA safeguards. The IAEA-safeguarded inventory consists of over 500 plutonium oxide and and scrap items. Based on the WHC inventory declaration, the plutonium materials ranged in concentration from 35 to 92 wt% plutonium (vs. 88.2 wt% for stoichiometric plutonium dioxide, PuO$_2$). The item net weights ranged from 200 grams to 2000 grams. From 5 to 17 wt% of the total plutonium in each item was present as $^{240}$Pu. The seven items selected for heterogeneity testing, listed in Table 1, span this range of material properties.

**TABLE 1. ITEMS SAMPLED FOR HETEROGENEITY TESTING**

<table>
<thead>
<tr>
<th>Item ID</th>
<th>Stratum</th>
<th>Pu Concentration (wt. %) $^a$</th>
<th>Item Mass (grams)</th>
<th>$^{240}$Pu Isotopic Concentration (wt. %) $^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Item SC4</td>
<td>Scrap</td>
<td>75.98</td>
<td>1195</td>
<td>6.492</td>
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<tr>
<td>Item A</td>
<td>Scrap</td>
<td>35.71</td>
<td>966</td>
<td>5.355</td>
</tr>
<tr>
<td>Item B</td>
<td>Scrap</td>
<td>78.47</td>
<td>675</td>
<td>5.899</td>
</tr>
<tr>
<td>Item C</td>
<td>Product</td>
<td>87.21</td>
<td>1545</td>
<td>12.150</td>
</tr>
<tr>
<td>Item D</td>
<td>Scrap</td>
<td>60.75</td>
<td>400</td>
<td>8.012</td>
</tr>
<tr>
<td>Item E</td>
<td>Product</td>
<td>86.50</td>
<td>1000</td>
<td>16.336</td>
</tr>
<tr>
<td>Item F</td>
<td>Scrap</td>
<td>82.87</td>
<td>307</td>
<td>14.321</td>
</tr>
</tbody>
</table>

$^a$ Data are WHC declared mass and isotopic values, decay-corrected to 1 Nov 94

$^b$ Item not under IAEA safeguards

**Item Heterogeneity**

Materials in each of the seven selected items were sampled to determine the heterogeneity of their plutonium concentrations. The amperometric data are illustrated (by location) in Figures 3 through 9, respectively. These data were used to estimate the degree of heterogeneity of the plutonium concentration of each item (sampling variability) and determine its significance relative to the underlying analytical variability.

Analytical and sampling variability components were estimated using a suitable analysis of variance (ANOVA) model. The analytical variability component was estimated from the duplicate titrations performed for each sample. The two sample aliquots obtained from each location were used to estimate the within-sample location variability component. Samples taken at the five locations were used to estimate the between-sample location variability component. Estimates of the between-sample location, the within-sample location, and the analytical variability for each item are listed in Table 2.

A statistical F test was performed to determine whether the within-sample location variability is significantly different from zero. The F test indicated that the within-sample location variability is significantly different from zero (at the 0.05 level of significance) for all items except for Item E. A similar F test, performed for each item to determine whether the between-sample location variability is significantly different from zero, showed a significant difference (at the 0.05 level of significance) for Items SC4 and A.
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When samples from Item A were destructively analyzed by amperometric titration, a precipitate was observed. This is an indication of a potential interference in the analytical method. In addition, when aliquots from Item A were analyzed by inductively coupled plasma spectrometry, both manganese and chromium were identified. These elements produce a high bias in the amperometric titration method. Dissolved portions from each of the ten samples subsequently were analyzed by a quantitative gamma spectrometric method (solution counter) that does not suffer from these chemical interferences. Variance components for the solution counter measurements of Item A were also calculated and are listed in Table 2.

When samples from Item D were destructively analyzed by amperometric titration, a faint purple color was observed. This is an indication of the presence of manganese, an interference in the analytical method. Again, dissolved portions from each of the ten samples were analyzed by the solution counter. Variance components for the solution counter measurements of Item D were also calculated and are listed in Table 2.

The data in Table 2 show that the analytical variability for amperometric titration ranged from 0.11% to 0.46% (Item A). The precipitation problem, which occurred during the amperometric titration of Item A, was a likely cause for the increased analytical variability.

**TABLE 2. VARIABILITY COMPONENTS BASED ON DA CORE SAMPLE DATA**

<table>
<thead>
<tr>
<th>Item ID</th>
<th>Stratum</th>
<th>Random $\delta_A$ a</th>
<th>Random $\delta_W$ c</th>
<th>Random $\delta_B$ d</th>
<th>Systematic $\delta_S$ e</th>
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<td>1 RSD (%)</td>
<td>1 RSD (%)</td>
<td>1 RSD (%)</td>
<td>1 RSD (%)</td>
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<tr>
<td>Item SC4</td>
<td>Scrap</td>
<td>0.24</td>
<td>1.98</td>
<td>3.12</td>
<td>0.06</td>
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<tr>
<td>Item A g - Titration</td>
<td>Scrap</td>
<td>0.46</td>
<td>0.58</td>
<td>1.15</td>
<td>0.06</td>
</tr>
<tr>
<td>Item A g - Solution Counter</td>
<td>Scrap</td>
<td>0.75</td>
<td>1.22</td>
<td>1.95</td>
<td>0.2</td>
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<tr>
<td>Item B</td>
<td>Scrap</td>
<td>0.11</td>
<td>1.62</td>
<td>0.00</td>
<td>0.06</td>
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<tr>
<td>Item C</td>
<td>Product</td>
<td>0.14</td>
<td>0.25</td>
<td>0.00</td>
<td>0.06</td>
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<tr>
<td>Item D - Titration</td>
<td>Scrap</td>
<td>0.13</td>
<td>1.15</td>
<td>0.35</td>
<td>0.06</td>
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<tr>
<td>Item D - Solution Counter</td>
<td>Scrap</td>
<td>0.75</td>
<td>2.25</td>
<td>2.00</td>
<td>0.2</td>
</tr>
<tr>
<td>Item E</td>
<td>Product</td>
<td>0.17</td>
<td>0.15</td>
<td>0.25</td>
<td>0.06</td>
</tr>
<tr>
<td>Item F</td>
<td>Scrap</td>
<td>0.18</td>
<td>0.28</td>
<td>0.34</td>
<td>0.06</td>
</tr>
</tbody>
</table>

- **a** Analytical variability
- **b** Relative standard deviation, (standard deviation/mean) x 100%
- **c** Within-sample location variability
- **d** Between-sample location variability
- **e** Systematic analytical variability (estimated from analyses of blind standards)
- **f** Statistically significant at the 0.05 level of significance
- **g** Item not currently under IAEA safeguards
- **h** Results based on solution counter counting statistics

**Composite Sample Analysis**

Analytical and sampling variability components were estimated from the composite sample data with the aid of a suitable ANOVA model. The analytical variability component was estimated from the duplicate titrations performed for each sample. Because two samples were obtained from each composite sample, a within-composite sample variability component could be estimated. Estimates of
the within-composite sample and the analytical variabilities by material type are listed in Table 3. The analytical and within-composite sample variabilities in Table 3 are similar to the analytical and within-sample variabilities shown in Table 2.

TABLE 3. VARIABILITY COMPONENTS BASED ON DA COMPOSITE SAMPLE DATA

<table>
<thead>
<tr>
<th>Data Set</th>
<th>Random $\delta_A$ a (1 RSD %)</th>
<th>Random $\delta_W$ b (1 RSD %)</th>
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<tr>
<td>Product Items  (13)</td>
<td>0.15</td>
<td>0.12</td>
</tr>
<tr>
<td>Scrap Items    (10)</td>
<td>0.33</td>
<td>2.40</td>
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</tbody>
</table>

a Analytical variability  
b Within-composite variability

The average plutonium content for each item sampled for heterogeneity testing was determined in two ways: from the core sample DA results and from the composite sample DA results. The mean plutonium concentration for each item and the standard deviation associated with each mean are listed in Table 4. A hypothesis test comparing the two means was performed for each item. The hypothesis of equality of means was not rejected at the 0.05 level of significance for each of the items listed in Table 4. This is taken as evidence that the WHC composite sample methodology provides material representative of the average plutonium content of the sampled item.

TABLE 4. COMPOSITE SAMPLE MEAN versus CORE SAMPLE MEAN

<table>
<thead>
<tr>
<th>Item ID</th>
<th>Core Sample Mean (wt% Pu)</th>
<th>Standard Deviation associated with the Core Sample Mean (wt% Pu)</th>
<th>Composite Sample Mean (wt% Pu)</th>
<th>Standard Deviation associated with the Composite Sample Mean (wt% Pu)</th>
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</tr>
<tr>
<td>Item SC4</td>
<td>77.52</td>
<td>1.19</td>
<td>78.11</td>
<td>1.66</td>
</tr>
<tr>
<td>Item A a</td>
<td>46.12 (33.60) b</td>
<td>0.26 (0.33)</td>
<td>46.49 (33.58)</td>
<td>0.25 (0.30)</td>
</tr>
<tr>
<td>Item B</td>
<td>82.12</td>
<td>0.42</td>
<td>81.90</td>
<td>0.16</td>
</tr>
<tr>
<td>Item C</td>
<td>85.28</td>
<td>0.07</td>
<td>85.32</td>
<td>0.11</td>
</tr>
<tr>
<td>Item D</td>
<td>66.76 (62.22) b</td>
<td>0.27 (0.72)</td>
<td>67.04 (61.75)</td>
<td>0.11 (0.59)</td>
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<tr>
<td>Item E</td>
<td>86.90</td>
<td>0.11</td>
<td>86.82</td>
<td>0.09</td>
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<tr>
<td>Item F</td>
<td>80.71</td>
<td>0.15</td>
<td>80.26</td>
<td>0.19</td>
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</table>

a Item not under IAEA safeguards  
b Values in parentheses represent solution counter results

In a collaboration between the IAEA and various national and international safeguards organizations and for effective safeguarding of nuclear materials, International Target Values (ITV) for uncertainty components in fissile isotope and element accountancy have been defined (Deron et al. 1994). The IAEA also maintains a continually updated database containing estimates of the random and systematic measurement variabilities for the various material types and measurement techniques encountered in their safeguard activities in facilities worldwide. Table 5 lists the combined variability (sampling and analytical) as determined from this recent sampling event at PFP, the
relevant ITV for fuels-grade PuO₂ and plutonium–uranium mixed oxide (MOX) scrap, and the IAEA measurement experience statistics. As can be seen from Table 5, the PFP Analytical Laboratory measurement variabilities agree with those experienced by the IAEA. For comparison purposes, Table 5 also includes the variability associated with the PFP Analytical Laboratory measurements.

TABLE 5. VARIABILITY ESTIMATES

<table>
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<th>Analytical Technique</th>
<th>PFP Experience Random/Systematic</th>
<th>IAEA Experience Random/Systematic</th>
<th>ITV a Random/Systematic</th>
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<td></td>
<td>PuO₂ Product</td>
<td>PuO₂ Scrap</td>
<td>PuO₂ Pure</td>
</tr>
<tr>
<td>DA - Titration</td>
<td>0.2 b/0.1</td>
<td>2.4 b/0.1</td>
<td>~3/~1.5</td>
</tr>
<tr>
<td>DA - Solution Counter</td>
<td>0.8/0.2</td>
<td>3.0 b/0.2</td>
<td>0.15/0.10</td>
</tr>
<tr>
<td>Calorimeter</td>
<td>0.34/0.1</td>
<td>0.64/0.1</td>
<td>5.1/0.5</td>
</tr>
</tbody>
</table>

a Deron et al., 1994
b Based on analyses of composite samples

As expected, all variability estimates for scrap are greater than those for product quality PuO₂. In addition, the PFP measurement experience shows that the variability of the PFP calorimeter technique is comparable with the total DA variability for product quality materials and is clearly superior to the total DA variability for scrap.

COMPARISON OF RESULTS

To evaluate further the performance of the calorimeter, the calorimetric measurements were compared to the PFP and IAEA destructive analysis results of the respective items. Thirteen product quality PuO₂ items and ten scrap items were compared. The measurement results, in terms of relative difference with respect to the PFP destructive analysis results, are listed in Table 6. Relative difference is defined as follows:

\[
\text{Relative difference (\%) } = \frac{\text{PFP DA - Measured}}{\text{PFP DA}} \times 100
\]

where Measured is either the IAEA DA result or the PFP calorimeter result. The relative differences are illustrated in Figures 10 and 11 for product and scrap materials, respectively.

PFP Destructive Analysis versus IAEA Destructive Analysis

The relative differences of the eight product items measured by both PFP and IAEA DA ranged from -0.22% to 0.30%. The average relative difference for the eight product items is 0.05% with a standard deviation of 0.19%. The relative differences for the five scrap items measured by both PFP and IAEA ranged from -2.28% to 3.83%. The average relative difference for the five scrap items is 1.66% with a standard deviation of 2.56%. The PFP and IAEA DA results are similar. The higher variability of the scrap materials is evident and is attributed to material heterogeneity.

PFP Destructive Analysis versus PFP Calorimetric Measurements

The relative differences of the thirteen product items measured by both PFP DA and PFP calorimetry ranged from -1.29% to 0.93%. The average relative difference for the thirteen product items is 0.03% with a standard deviation of 0.52%. The relative differences for the ten scrap items...
# TABLE 6. MEASUREMENT RESULTS

<table>
<thead>
<tr>
<th>Stratum</th>
<th>Item ID</th>
<th>Grams Plutonium</th>
<th>Relative Difference (%)</th>
<th></th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>PFP DA (^a)</td>
<td>IAEA DA (^b)</td>
<td>PFP Calorimeter (^c)</td>
</tr>
<tr>
<td>Product</td>
<td>PD1</td>
<td>265</td>
<td>265</td>
<td>266</td>
</tr>
<tr>
<td></td>
<td>PD2</td>
<td>470</td>
<td>471</td>
<td>470</td>
</tr>
<tr>
<td></td>
<td>PD3</td>
<td>865</td>
<td>864</td>
<td>862</td>
</tr>
<tr>
<td></td>
<td>PD4</td>
<td>866</td>
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</tr>
<tr>
<td></td>
<td>PD5</td>
<td>861</td>
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<td>1594</td>
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<td>861</td>
<td>NA</td>
<td>859</td>
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<td>269</td>
<td>262</td>
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<td>SC2</td>
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<td>SC3</td>
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<tr>
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<td>SC6</td>
<td>291</td>
<td>NA</td>
<td>273</td>
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<tr>
<td></td>
<td>SC6</td>
<td>271 (^e)</td>
<td>NA</td>
<td>273</td>
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<tr>
<td></td>
<td>A</td>
<td>443</td>
<td>NA</td>
<td>339</td>
</tr>
<tr>
<td></td>
<td>A</td>
<td>320 (^e)</td>
<td>NA</td>
<td>339</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>543</td>
<td>NA</td>
<td>519</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>258</td>
<td>NA</td>
<td>235</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>237 (^e)</td>
<td>NA</td>
<td>235</td>
</tr>
<tr>
<td></td>
<td>F</td>
<td>238</td>
<td>NA</td>
<td>237</td>
</tr>
</tbody>
</table>

\(^a\) PFP amperometric titration (ASTM 1992)  
\(^b\) IAEA titration (Macdonald and Savage 1979)  
\(^c\) PFP calorimeter measurement (ANSI 1987) and NDA isotopics  
\(^d\) NA: not analyzed  
\(^e\) PFP gamma spectrometric (solution counter) result
measured by both PFP DA and PFP calorimetry ranged from -5.94% to 4.42%. The average relative difference for the scrap items is 0.64% with a standard deviation of 2.93%. Solution counter DA results are used for items SC6, A, and D because of the titration interferences known to be present in these items. The data are within the variability associated with the measurement methods. Again, the scrap results show higher variability than the product results.

CONCLUSIONS

Homogeneity cannot be assumed for many of the plutonium powder items at vault 3 of the 2736-Z Building of the USDOE's PFP on the Hanford Site. The analyses have confirmed that part of the material is indeed significantly heterogeneous. This means that precautions should be taken to obtain representative samples for DA. A core sampling and blending technique employed at the IPIV is an effective means to acquire representative samples.

As shown in this paper, 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 heterogeneous scrap materials with possible electrochemical interferences (e.g., Mn, Cr, V, Pb), dissolution and plutonium solution counting by gamma spectrometry is an attractive alternative analytical method. 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 PFP method of choice for accountability measurements of plutonium materials in storage. Use of the PFP calorimeter by the IAEA as an alternative method for quantitative plutonium nondestructive analysis (particularly for scrap) is being developed. Steps are being taken to allow the PFP and IAEA shared access to a calorimeter controller. Means for independent IAEA validation of the PFP calorimeter systems are being developed.

Despite the merits of the various nondestructive analytical methods, sampling and DA provide fundamental assurances of bulk material quantities and thus remain necessary components of the IAEA verification requirements. Verification by DA is also central to the IAEA validation of the PFP calorimeter system proposed for safeguards use.

REFERENCES


FIGURE 1: CORE SAMPLE SPLIT.

Individual Sample → put into composite sample vial (22mL) → put into 2 dram vial for laboratory analysis Sample A

FIGURE 2: COMPOSITE SAMPLE SPLIT.

Composite sample → put into composite sample vial (22mL) → put into 2 dram vial for laboratory analysis Composite Sample A

Made from a section of each of the 5 individual samples

Any remaining material -- put back into original container
FIGURE 5: ITEM B.

FIGURE 6: ITEM C.
FIGURE 9: ITEM F.

FIGURE 10. RELATIVE DIFFERENCES - PRODUCT STRATUM.
Gamma spectrometric (solution counter) results
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