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T. L. Welsh
L. P. McRae
D. D. Scott
B&W Protec, Inc.

T. F. Moriarty
R. J. Lemaire
V. S. Fotin
International Atomic Energy Agency

R. A. Hamilton
G. A. Westsik
B&W Hanford Company

N. F. Pertzborn
Lockheed Martin Services, Inc.

C. H. Delegard
Pacific Northwest National Laboratory

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**INTERNATIONAL ATOMIC ENERGY AGENCY/HANFORD SITE
SHARED USE OF CALORIMETERS**

T. F. Moriarty, R. J. Lemaire, V. S. Fotin,
International Atomic Energy Agency, Vienna, A-1400, Austria;
L. P. McRae, T. L. Welsh, D. D. Scott,
B & W Protec, Inc., Richland, Washington, 99352-0800, USA;
R. A. Hamilton, G. A. Westsik,
B & W Hanford Company, Richland, Washington, 99352-1200, USA;
N. F. Pertzborn
Lockheed Martin Services, Inc., Richland, Washington, 99352-0950, USA;
C. H. Delegard
Pacific Northwest National Laboratory, Richland, Washington, 99352-0999, USA

ABSTRACT

Hanford Site operators combine gamma ray isotopic and calorimetry measurements for nondestructive plutonium assay. Such measurements offer lower variability (particularly for heterogeneous materials) and decreased radiation exposure, cost, waste, intrusiveness, and material handling compared to destructive analysis. Until now, the International Atomic Energy Agency (IAEA) has relied on destructive analysis to perform the most accurate verification requirements for plutonium stored under safeguards at the Hanford Site. It was recognized that using calorimetry could significantly reduce the need for the IAEA to perform destructive analysis. To authorize the operator's calorimeters for routine IAEA use, however, it was necessary to develop authentication features and perform independent testing. Authentication features include IAEA control of the hardware and calorimeter operating system software, measurement of certified IAEA standards, sealing of calorimeter chambers, and limited destructive analysis of IAEA selected items. A field test of these authentication features was performed at the Hanford Site in June 1997. The field test also was meant to enhance the credibility the IAEA imputes to calorimetry prior to its implementation. Progress in shared use of the Hanford Site calorimeters is reported.

INTRODUCTION

An inventory of about 1,000 containers of plutonium is under IAEA safeguards at the Plutonium Finishing Plant (PFP) at the United States' Department of Energy Hanford Site. These items, originating from the United States' nuclear weapons complex, include relatively pure plutonium oxide powders as well as impure plutonium scrap materials such as those arising from plutonium refining processes, incinerator ash, and sweepings from process glove boxes. The IAEA designates the pure plutonium oxide as the powder (PD) inventory stratum; the less pure material is designated scrap (SC). Each item contains plutonium packaged in a nested set of three metal cans with plutonium held in the innermost can.

As part of its safeguards requirements, the IAEA performs physical verifications of plutonium inventories using three increasingly precise and accurate levels of measurement, known as gross, partial, and bias defect tests, on randomly selected samples of the entire population of items under its safeguards. The results of these independent measurements are compared with the operator's declarations so that safeguards conclusions concerning the entire inventory may be drawn. At the Hanford Site, the IAEA performed bias defect tests by quantitative destructive analysis (DA) on a number of items (8 PD; 12 SC). The DA required onsite sampling and weighing of the selected items, shipment, and offsite electrochemical and mass spectrometric analysis of the samples through the IAEA's Safeguards Analytical Laboratory in Seibersdorf, Austria.

Comparison of Measurement Methods

With IAEA cooperation and participation, parallel nondestructive assay (NDA) and DA measurements of the same and additional similar items (13 PD; 17 SC) were performed by PFP personnel. The NDA was conducted on inventory items by combining gamma spectrometry (to determine the isotopic composition) and calorimeter measurements to determine total plutonium mass. The DA combined weighing, sampling, and amperometric titration and mass spectrometry of the samples to ascertain plutonium mass. Testing for the presence of impurities, which interfere with the chemical analysis, was also performed for selected items. The contributions of material heterogeneity to the total DA analytical measurement variability were determined by multiple sampling and paired, separate, DA of each sample for seven items (2 PD; 5 SC).

The following conclusions were reached in previous studies by analyzing the operator's measurement results and comparing them to IAEA results.

- The IAEA and operator DA results were not significantly different.
- Inventory items, particularly in the SC stratum, were significantly inhomogeneous and sometimes contained interferences to electrochemical DA. It was determined that DA sampling variabilities exceeded chemical analysis variabilities and dominated the total DA (analytical plus sampling) variabilities.
- Combined gamma and calorimeter NDA measurement variabilities were comparable to DA variabilities for PD items and were much lower than DA for SC items.

The relative accuracy and precision of calorimeter-based NDA, compared to DA, are known by the PFP operator and make it the PFP method of choice for accountability measurements of plutonium materials in storage.

Calorimetry is also more rapid, less expensive, produces less waste, requires less item handling, and inflicts less personnel radiation exposure because items do not need to be opened and repackaged. In addition, sample shipment and laboratory residue disposal are avoided.

IAEA Use of Operator Calorimeters for International Safeguards

The sampling, shipping, analysis, and waste disposal operations required for DA are also costly in time, money, and radiation exposure for the IAEA. These factors, and the comparative analytical findings, persuaded the IAEA to consider replacing part of their quantitative DA requirements with NDA measurements using the PFP calorimeters and PFP gamma isotopic analysis confirmed by IAEA gamma measurements. To draw valid safeguards conclusions, however, the IAEA must be able to authenticate the results of the calorimeter assay measurements made with operator equipment. Various methods the IAEA could use to assure validity of calorimeter results were proposed and evaluated.

No single approach was found to be sufficiently robust and practical for routine IAEA use. Instead, a combination of authentication measures was judged to provide adequate assurance that genuine calorimeter measurements could be obtained for IAEA purposes from PFP equipment. The selected measures are:

- sealing of the calorimeter sample measurement well and the pre-equilibration bath;
- use of authenticated tamper-indicating and -resistant software for calorimeter operation, stored on a removable hard drive, and kept under IAEA control;
- calorimeter measurement of standard items previously verified and certified by DA; and
- the possibility of DA of selected items following calorimeter measurements.

The acceptance of shared use of the PFP calorimeters for IAEA safeguards depends on the successful application of these authentication measures and satisfactory demonstration of measurement activities through field testing at PFP.

AUTHENTICATION MEASURES

Authentication measures are required by the IAEA to permit use of the PFP calorimeters to draw appropriate safeguards conclusions. These measures are described.

Maintaining Continuity of Knowledge Over the Material

Calorimeter measurements at PFP require 4 to 7 hours. Measurement times are minimized by thermally adjusting items to be measured to the calorimeter operating temperature in a

pre-equilibration bath (known as a PE bath). To increase measurement throughput, unattended overnight calorimeter operation is also possible. In such circumstances, continuous inspector presence would be impractical. Consequently, to maintain continuity of knowledge over the material and to reduce the potential for possible tampering with the measurements, the calorimeter wells and the PE bath can be sealed with electronic IAEA seals. The PE bath also can be used as a convenient storage location for items that have been removed from the vault for measurement.

Software Authentication

The PFP calorimeters are controlled, and the data gathered, analysed, and reported, using a PC-based computer system, a Windows 3.1¹ operating system, and a specialized application package named WinCal. WinCal was developed for the PFP calorimeters to provide improved servo control and endpoint detection procedures and increased graphical, data logging, and diagnostic capabilities on an easily upgradable computer platform. Special features were added to WinCal to address IAEA requirements arising from shared use and to permit IAEA verification of the software function. One feature is a keyboard password system that locks out almost all keyboard entries and mouse operations unless authorized by a password, chosen by the IAEA inspector and kept resident only in memory. The password serves as a tamper indicator because it will disappear if the program is stopped. A second feature is a checksum routine that monitors software components controlling program operation. The checksum can be queried at any time during operation and is provided with each measurement report. WinCal also provides a training mode that operates on any standalone computer.

The real-time measurement performance data (e.g., temperatures, powers, calorimeter operating bridge potentials) provided by the software offer further assurance that the calorimeter measurements are genuine by being intrinsically tamper-indicating (e.g., a discontinuity in reaching equilibrium may indicate potential tampering).

The software developers provided the IAEA with complete documentation of the WinCal modules, the source code of WinCal, and the necessary commercial software required to compile an executable version of the code from the source code. The IAEA compiled and loaded the executable code to a removable hard drive owned by the IAEA. The process of compiling the software and loading it to a removable hard drive provides the IAEA with confidence that it has complete control over the executable version of the code. The IAEA thus can ensure that the reviewed software source code is the same as that used for the IAEA's calorimeter measurements at the facility. The source code and operating media will remain under the control of the IAEA and can be further reviewed if required (e.g., in the case of a software upgrade). Software changes require concurrence of the operator and the IAEA.

Although comprehensive analysis of the WinCal source code is a considerable task, the IAEA has the potential to exhaustively authenticate the code. Even with a modest commitment of limited IAEA resources, however, significant characteristics of the code have been reviewed

by IAEA software and NDA experts through discussions with the WinCal software developers. The first step in demonstrating that the software functioned correctly was to confirm that the password protection was genuine and effective. The checksum routine, which ensures the executable program has not been altered during use at PFP, was also reviewed. As IAEA understanding of the code is developed, particular aspects of the code may be selected for closer scrutiny. Such an approach is analogous to the ongoing design information verification activities performed by the IAEA at safeguarded facilities. Software authentication is a relatively new activity to the IAEA. Provision of the WinCal source code has given the IAEA an excellent opportunity to develop expertise in this area.

After the WinCal operating software had been verified, controlled, and compiled by the IAEA, it was loaded on a 600 megabyte removable hard drive also controlled by the IAEA. The IAEA uses the loaded hard drive to operate the PFP calorimeters for verification purposes. The PFP calorimeters' computer system was modified to accommodate the removable hard drive by means of a chassis slide framework. The framework contains interfaces between the computer and the hard drive, a cooling fan, and a combined locking device and power switch to allow hard drive changes without interrupting power to the computer. Holes were drilled in the framework and computer case to accommodate IAEA cup wire sealing of the hard drive to the computer/controller console. A hard drive similar to that of the IAEA is dedicated to PFP facility use.

Use of Standards

Confirmation of the correct operation of the calorimeters can be performed by measuring inventory items whose contents have been verified independently by the IAEA through weighing, sampling, and DA.

Twenty inventory items are present in PFP's IAEA safeguarded storage which have previously been measured by IAEA DA. Of these, 12 items have been certified as IAEA standards. Conclusions about the validity of the calorimeter assay can be reached during the inspection by IAEA calorimeter measurement of these standard items and comparison of the results to the known IAEA DA values.

Destructive Analysis

The IAEA retains the right to weigh, sample and DA items in the safeguarded inventory. This right may be exercised to confirm correct calorimeter operation of IAEA-selected items after calorimeter measurement. This action provides a degree of assurance to the IAEA of genuine calorimeter operation because a potential diverter cannot anticipate whether an item or items may be selected by the IAEA for DA.

FIELD TESTING

Authorization for IAEA use of the calorimeter system at the PFP required the successful completion of a field test. The field test, performed in June 1997, established three objectives for success:

- maintenance of continuity of knowledge over the material by effective sealing of the calorimeter wells and PE bath;
- satisfactory operation of the calorimeter hardware and WinCal software; and
- satisfactory calorimeter measurement results for IAEA certified inventory items.

The field testing successfully achieved all the stated objectives. The results of combined IAEA-confirmed PFP gamma NDA and IAEA shared use calorimeter measurements of plutonium mass for eight certified IAEA standards are presented in Table 1. The results are compared with inventory values declared by the operator to the IAEA and results of prior IAEA weighing/sampling/DA of the same items. All values have been decay-corrected to the date of the June 1997 measurements.

For the four certified standards tested from the PD (pure powder) stratum, relative differences between the declared plutonium mass and the IAEA DA ranged from -0.4% to +0.2%. Relative differences between the declared and IAEA gamma/calorimeter values showed a similar range, from 0.0% to +0.7%. The similarity in DA- and calorimeter-based measurement variabilities for PD materials was observed in previous studies. For the SC (scrap) stratum, relative differences between the declared masses for the four tested certified standards and the IAEA DA ranged from -2.6% to +2.0%. Such variabilities are high for bias defect verifications required by IAEA safeguards. However, the relative differences between declared and IAEA gamma/calorimeter values only ranged from -0.2% to +0.2%. These results confirm the difficulty, described in previous studies, of achieving low measurement variability by DA methods for impure and heterogeneous plutonium materials and demonstrate the merits of calorimeter measurements.

Table 1. Item Measurement Data Comparison.

Item ID	Relative Difference (%) [*] IAEA DA	Relative Difference (%) ^{**} IAEA Calorimeter
PD20	-0.4	0.0
PD21	0.2	0.6
PD22	0.2	0.7
PD23	-0.2	0.4
SC24	-1.3	0.2
SC25	-2.6	-0.2
SC26	2.0	-0.2
SC27	-0.3	0.2

^{*} $100\% \times (\text{IAEA DA} - \text{Declared}) / \text{Declared}$

^{**} $100\% \times (\text{IAEA Calorimeter} - \text{Declared}) / \text{Declared}$

CONCLUSIONS

With the successful completion of field testing, PFP calorimeters are to be approved for routine use for IAEA safeguards plutonium verifications at PFP. This new method decreases measurement variability, cost, time to reach safeguards conclusions, radiation exposure, and waste disposal for the IAEA in the bias-level verification of plutonium-bearing items at PFP when compared with weighing, sampling, and DA.

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D. Schriefer
W. Theis

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