

LA-UR-95-2276

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*Title:*

**EVALUATION AND DEVELOPMENT PLAN OF NRTA MEASUREMENT METHODS FOR THE ROKKASHO REPROCESSING PLANT**

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*Submitted to:*

**INMM 36th Annual Meeting  
Palm Desert, CA USA  
July 12-19, 1995  
(FULL PAPER)**

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# EVALUATION AND DEVELOPMENT PLAN OF NRTA MEASUREMENT METHODS FOR THE ROKKASHO REPROCESSING PLANT\*

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## ABSTRACT

Near-real-time accounting (NRTA) has been proposed as a safeguards method at the Rokkasho Reprocessing Plant (RRP), a large-scale commercial boiling water and pressurized water reactors spent-fuel reprocessing facility. NRTA for RRP requires material balance closures every month. To develop a more effective and practical NRTA system for RRP, we have evaluated NRTA measurement techniques and systems that might be implemented in both the main process and the co-denitration process areas at RRP to analyze the concentrations of plutonium in solutions and mixed oxide powder. Based on the comparative evaluation, including performance, reliability, design criteria, operation methods, maintenance requirements, and estimated costs for each possible measurement method, recommendations for development were formulated. This paper discusses the evaluations and reports on the recommendation of the NRTA development plan for potential implementation at RRP.

## I. INTRODUCTION

Japan Nuclear Fuel Limited (JNFL) is constructing a large-scale commercial boiling water and pressurized water reactors spent-fuel reprocessing facility at Rokkasho-mura, Aomori prefecture at the Shimokita peninsula of the northern end of Honshu, Japan. The Rokkasho Reprocessing Plant (RRP), is planned to start operation sometime after the year 2000 with a reprocessing capacity of 800 tons of uranium per year. The description of the RRP processes has been given in Coulter's paper in this Proceedings.<sup>1</sup> To ensure that an effective and efficient safeguards system is applied at RRP, national and international safeguards agencies require near-real-time

accounting (NRTA). This requires material balance closures every month in the main chemical separation area and the uranium-plutonium co-denitration process area of RRP.

We reviewed and evaluated NRTA measurement methods that might be implemented at RRP to analyze the concentrations of plutonium in solutions and mixed oxide (MOX) powder in both the main process area and the co-denitration process area. For each measurement application, possible methods were identified and compared. Candidate techniques have been developed at Los Alamos and other safeguards R&D laboratories, such as those in the European community. New measurement techniques that are currently under development were also considered.

Based upon evaluated performance, reliability, design criteria, operation methods, maintenance requirements, and estimated costs for each possible measurement method, we recommended potential methods for various measurement points in the main process area and the co-denitration process area.

This paper discusses recommended NRTA measurement methods for input, intermediate, and output solutions in the main chemical separation process; input, MOX production materials, and holdup in the co-denitration process; and unattended verification systems.

## II. MAIN CHEMICAL PROCESS

### A. Input Dissolver Solution

Clarified dissolver solutions are the input to the main chemical process area where separation and purification occurs. Accountability measurements on input dissolver solutions are essential for effective safeguards. Dissolver solutions contain high levels of fission products and uranium (to 250 g/L). Plutonium concentrations are generally about 1% of uranium concentrations.

\*This work is supported by the US Department of Energy, Office of Nonproliferation and National Security, International Safeguards Division, and by Japan Nuclear Fuel Limited.

**1. Isotope Dilution Mass Spectrometry (IDMS).** IDMS is the well accepted method most widely applied to the determination of isotopic content and total concentration of plutonium in dissolver input solutions.

ESARDA's target values<sup>2</sup> for random precision for IDMS determination of plutonium concentrations in dissolver solutions are 0.3% and 0.5% for sampling and measurement respectively. IAEA values<sup>3</sup> for random and systematic error are 0.4% and 0.2% respectively. Much operating experience from various reprocessing facilities suggests that values on the order of about 0.5% relative are attainable for dissolver solutions.

**2. Hybrid K-Edge/X-Ray Fluorescence Densitometry (HKED).** The Hybrid KED/XRF Densitometer<sup>4</sup> measures the uranium element concentration by using K-edge densitometry (KED) and the plutonium element concentration by using x-ray fluorescence (XRF). The instrument was pioneered at KfK, Karlsruhe. Similar systems have been designed and installed at Sellafield, LaHague, Los Alamos, and Tokai. The KfK-designed HKED is in routine use for input verification measurements at La Hague.<sup>5</sup> For input dissolver solutions from typical light-water reactor fuels, the measurement uncertainties of the hybrid system are currently estimated from paired comparisons of inspector-operator results to about 0.2 to 0.3% for uranium and to about 0.6 to 0.7% for plutonium. Kurosawa et al.,<sup>6</sup> developed a HKED composed of an x-ray generator, detectors, collimators, and flow-type cells. The instrument was compactly designed and installed in a shielded cell at the Tokai Reprocessing Plant (TRP). They reported the precision for determining uranium concentration (approximately 180 g/L) by KED to be 0.2% for a 1000-s count and the precision for determining plutonium (approximately 1.5 g/L) by XRF to be 1.7% for a 3000-s count. They suggested the larger uncertainty for plutonium may be due to the distance between the x-ray tube and the measurement cell and the geometry of the cell and collimator. In the most recent publication, TRP<sup>7</sup> reported the results of improvement on the precision for determining plutonium concentration by XRF to be 0.98%.

**3. Isotope Dilution Gamma-Ray Spectrometry (IDGS).** IDMS has long been the most accepted technique for determining the plutonium content of the input dissolver solution in reprocessing plants. However, IDMS is time consuming, sample preparation is lengthy, the equipment and operation

are costly, and the final sample is not always representative of the batch content. It would, therefore, be desirable to have an alternative technique that can provide timely, less expensive, and simpler on-site verification of the plutonium in the input dissolver solution to avoid IDMS's disadvantages and to complement the HKED measurement of concentrations. Such a novel technique, IDGS,<sup>8</sup> has been recently developed by using low-energy gamma-ray spectrometry. It has been successfully demonstrated in measuring plutonium isotopic composition and elemental concentrations of spent-fuel dissolver solutions with burnups up to 28 000 MWD/T. Precision for plutonium concentration measurement is better than 1% with a bias between IDGS and IDMS of less than 0.2%.

Using IDGS to analyze dissolver solutions for plutonium isotopic compositions, the precision is approximately 0.5% for the <sup>240</sup>Pu /<sup>239</sup>Pu ratio and 0.2% for the <sup>239</sup>Pu (weight percent) within a 1-h count time. The agreement between IDGS and IDMS for dissolver solutions is very good, especially so for the <sup>240</sup>Pu /<sup>239</sup>Pu ratio (average IDGS/IDMS ratio is 0.997) and the weight percent of <sup>239</sup>Pu (average IDGS/IDMS ratio is 0.999), which are important for determining the total plutonium concentration. Los Alamos and Tokai Reprocessing Plant of Power Reactor-Nuclear Fuel Development Corporation (PNC) are continuing to develop the IDGS technique for the simultaneous measurements of concentrations and isotopic compositions for both plutonium and uranium in highly irradiated spent-fuel dissolver solutions at a reprocessing plant.

Based on the above discussions, HKED (concentration only) or IDGS (concentration and isotopic composition), or both, are possible methods for routine analyses of input dissolver solutions. However, a small portion of samples (<10%) will be analyzed by IDMS.

## **B. Intermediate Process Solutions**

The Pu(VI) spectrophotometric method for total plutonium determinations is applicable to intermediate process solutions. RSDs for plutonium determination by Pu (VI) spectrophotometry, including both sampling and assay, range from 0.3% for pure plutonium nitrate solutions to 1.0% for dissolver solutions. Thus, we expect RSDs of about 0.5% for intermediate process solutions by Pu (VI) spectrophotometry.

However, the required measurement uncertainties for intermediate process solutions are 2.0% for random errors and 1.0% for systematic errors. We

suggest nondestructive assay (NDA) methods for rapid analysis of intermediate process solutions. Intermediate process solutions normally containing 2 to 10 g/L of plutonium with more than 94.7% of the fission products removed can be analyzed using the HKED or IDGS technique. Solutions from the process containing 6.0 g/L of plutonium and 0.2% fission products can be measured by L-edge densitometer (LED), HKED, or IDGS. Solutions from process vessels containing 10 to 30 g/L can be analyzed by XRF or KED.

### C. Product Solution

Final product solutions from the main process area can contain 200 to 350 g/L of plutonium in  $\text{HNO}_3$ . The target value at RRP is about 250 g/L. Large quantities of impurities are not present in product solutions. However, there may be very small quantities of uranium, neptunium, americium, and impurities, such as chromium, nickel, and iron, from stainless steel vessels and piping. Inventory accountability requires accurate and precise measurements of total plutonium at this location.

**1. Constant Potential Coulometry (CPC).** CPC is an accepted procedure to account for the total plutonium in product solutions.<sup>9</sup> ESARDA's target value for the total random uncertainty for pure plutonium nitrate solutions, when determined by coulometry, is 0.28% with a systematic error of 0.2%. The IAEA expects a random (not including sampling) and systematic error of 0.15% each for plutonium output solutions.

The coulometric method lends itself to remote operation. Various degrees of automation, such as developed at the TRP,<sup>10</sup> have been implemented. Using an automated CPC instrument, researchers at Tokai reported within-run precisions of less than 0.1% and between-run precisions of about 0.2% for standards.

**2. Titrimetry.** Titrimetry can be applied to the determination of plutonium in product solutions. Reported precisions and biases are generally less than 0.5% and 0.1% respectively, which are adequate for inventory purposes.

Expected precision and bias for titrimetric determination of plutonium in the product solution are about the same as for CPC. ESARDA's target value for the total random uncertainty for the titrimetric determination of plutonium in pure plutonium nitrate solutions is 0.28% with a systematic error of 0.2%.<sup>2</sup> The IAEA expects a random (not including sampling)

and systematic error of 0.15% each for plutonium product solutions.<sup>3</sup>

**3. K-Edge Densitometry (KED).** K-edge densitometry has been thoroughly tested at several facilities for the assay of plutonium and uranium product solutions. A great deal of work was done at the Tokai reprocessing plant. The earlier papers<sup>11</sup> report the average reproducibility of replicate measurements on plutonium product solutions to be between 0.25% and 0.3% RSD and an average difference between KED and titration results of -0.36% rel. In the most recent publication,<sup>12</sup> data are given that reflect routine use of the KED in reprocessing campaigns to provide a precision of 0.7% RSD for fresh samples and 1.0% for aged samples. The bias with respect to coulometry results varied from +0.28% to -0.81% rel. for fresh samples and was -0.22 rel. for aged samples.

K-edge densitometry, x-ray fluorescence, and titration results agreed well in tests at KfK.<sup>4</sup> The precision due to counting statistics was 0.22% RSD; other random variables contributed an additional 0.14% RSD.

ESARDA's target value for random uncertainty for the KED determination of plutonium in product solutions is 0.2% with a systematic error of 0.15%.<sup>13</sup> The IAEA expects a random and systematic error of 0.2% each for plutonium product solutions.<sup>3</sup>

The required random and systematic errors for product solutions at reprocessing plants are 0.2%. The IAEA recommends the MacDonald and Savage titrimetric method or CPC and KED.<sup>14</sup> The EURATOM laboratories use potentiometric titration and K-edge absorption for plutonium nitrate samples.<sup>15</sup> For NRTA measurement at RRP, we suggest that all plutonium nitrate product solutions are to be measured by K-edge densitometry for concentrations and by high-resolution gamma-ray spectrometry (HRGS) for isotopic compositions (if they are required). A small number of samples (<5%) will be analyzed by the titrimetric method or by the coulometric method.

## III. CO-DENITRATION PROCESS AREA

### A. Input and Intermediate Process Solutions

Input to the co-denitration process is the product from the main process area. Therefore, the same measurement methods (K-edge densitometry, coulometry, or titrimetry) can be used to measure

solutions from the input. These measurement methods also can be applied to the intermediate process where solutions contain about 150 g/L of plutonium mixed with uranium.

## **B. MOX Product Canister**

The verification of the MOX powder contained in storage canisters requires both gamma-ray and neutron instrumentation. The high-resolution gamma-ray spectroscopy is needed for the plutonium isotopic measurement. The passive neutron coincidence counter (NCC) is used to measure the spontaneous-fission rate from the plutonium for the  $^{240}\text{Pu}$ -effective mass determination. When this is combined with the plutonium isotopic compositions, the plutonium mass is determined.

The neutron detector will be designed for an effective efficiency of ~8% to achieve a maximum counting rate of 900 000 counts/s. The electronics can accommodate counting rates about twice as high for cases of very high burnup and high  $^{241}\text{Am}$  content. For the MOX can, plutonium analyses for both random and systematic errors are within 1.0%.

The canister counter will include three side holes for simultaneous neutron and gamma-ray measurements of the plutonium isotopics using the HRGS system. The holes will line up with the center of each of the MOX cans and three HPGe detectors will be positioned in front of each hole.

## **C. In-Process Inventory and Holdup in the Calcination, Reduction, and Blending Glove Boxes**

**1. The Quantitative Determinations Of Plutonium In-Process Inventory and Holdup in Glove Boxes.** Both gamma-ray and neutron coincidence measurements have been considered for the quantitative determinations of plutonium inventory in the calcination, reduction, and blending glove boxes at RRP. The RRP neutron coincidence measurement design is very similar to the PFPF Glove-Box Assay System (GBAS).<sup>16</sup> The measured uncertainties in the gamma-ray assays of in-process plutonium inventory at PFPF were between 25 and 30% ( $1\sigma$ ) compared to uncertainties closer to 5% ( $1\sigma$ ) for the neutron coincidence assays of the same material. Most of the large error in the gamma-ray results is caused by large, uncertain equipment attenuation effects. Therefore, neutron coincidence measurements of in-process plutonium inventory are recommended for the calcination, reduction, and blending glove box equipment at RRP.

Previous experience with measured holdup quantities correlated with material-balance data shows that inventory differences that grow with process operation time can be explained by the accumulation of holdup on the substantial surfaces of process equipment in high-throughput bulk-processing facilities. Such deposits cover the inner surfaces of glove boxes, ventilation ducts and transfer lines. Although these are relatively thin deposits (typically varying from 1 to 10 mg SNM/cm<sup>2</sup> between cleanouts), the surfaces of large glove box or a long ventilation duct can accumulate near-kilogram quantities over extended periods of operation time. On-line measurements of plutonium by neutron coincidence counting with slab detectors are not well suited to quantitative determination of these thin deposits of holdup on the surfaces of the containment enclosures, particularly when significant plutonium inventory may reside inside of the processing equipment within the containment enclosures. However, these surface holdup deposits are amenable to quantitative assay by portable gamma-ray detector systems.

We are designing the RRP holdup measurement systems based on the PFPF GBAS with some modifications to reduce assay uncertainties associated with MOX buildup on glove box surfaces and floors and with cross talk from the neighboring boxes. The Monte Carlo Neutron Photon calculations will be used in the design of the neutron slab detectors to minimize the uncertainties in the plutonium in-process inventory that arise from the effects of surface holdup, cross talk and other sources. We are also recommending the addition of an automated portable gamma-ray system for quantitative assay of thin deposits of holdup on the glove box surfaces.

**2. Determination of Plutonium Isotopic Distributions in Glove Boxes.** The ability of neutron coincidence counting to give accurate (~5%) measurements of plutonium in-process inventory is largely dependent on a known  $^{240}\text{Pu}$  isotopic fraction. Although a nominal  $^{240}\text{Pu}$  isotopic fraction of 24% is quoted for spent fuel of the "standard specification" by JNFL, a relative standard deviation of ~20% might be expected in this value. Therefore, the relative uncertainty may exceed 20% ( $1\sigma$ ) in the RRP in-process inventory of plutonium determined by neutron coincidence counting interpreted by the "standard specification"  $^{240}\text{Pu}$  isotopic fraction. It is recommended that the neutron coincidence counting be supplemented by high-resolution gamma-ray measurements of plutonium isotopic composition to minimize this source of uncertainty.

#### IV. UNATTENDED VERIFICATION SYSTEMS

The MOX and UO<sub>3</sub> product measurement systems will be designed for continuous operation in an unattended mode to monitor and assay the plutonium and uranium leaving the plant. The UO<sub>3</sub> product measurement system is not discussed in this paper because it is not an NRTA measurement. It is assumed that the unattended systems would have requirements similar to those of other facilities under IAEA inspection.<sup>18,19</sup> The IAEA would visit the facility at approximately 30-90 day intervals and the systems would need to collect and store data continuously over that interval. The inspector, upon arriving at the facility, would obtain data from each of the systems and review the data to determine when and how much material had moved through the detectors. The equipment could be under IAEA seal when the inspector was not present.

The unattended systems shown in Fig. 1 would consist of the neutron (NCC) and gamma (HRGS) detectors, some type of ID sample read, two intelligent shift registers (ISR), an intelligent multi-channel analyzer (IMCA), a local computer (PC), and connections to the network via a local operating network (LON). The ISR/PC system would be completely isolated from the network to provide a totally independent backup system. The other ISR and the IMCA would connect directly to the network. Each of the data acquisition electronics (ISRs and IMCA) would run a Monitor-type program. The main functions of the Monitor program would be to collect data from the detector on a continuous basis, temporarily store the data in the battery-backed-up memory, monitor the "health" of the detector and data acquisition electronics, and on request dump information to a Collect program. The Collect program would run on the local PC and also run on a central PC connected to the network. The main tasks of Collect would be to off-load data from the IMCA or ISR and store data on its hard disk. The system would run continuously during the inspection period and data would be accumulating on the local PC disk as well as on the disk of the central PC connected to the network. During the inspection the inspector would have the option to retrieve the backup data from the local PC to compare with the data shipped over the LON network. The local backup system provides the reliability of existing systems while the LON based systems provide the enhanced capability of having all the data centrally stored when the inspector arrives at the plant.

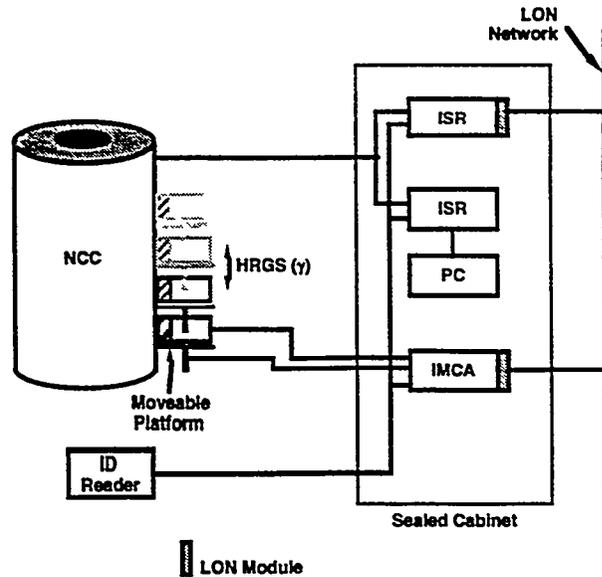


Fig. 1. Layout of NDA station for both neutron and gamma data and its connection to the Local Operating Network. Shown are the redundant ISR/PC combinations, the IMCA/PC combination, the controller, and the reader to provide the sample ID.

#### VII. SUMMARY

A wide variety of destructive assay (DA) and NDA measurement methods for NRTA measurements at RRP have been reviewed and evaluated. Evaluated destructive assay methods included mass spectrometry, alpha spectrometry, IDMS, CPC, titrimetry, spectrophotometry, and inductively coupled plasma spectroscopy. Evaluated gamma-ray techniques included HRGS, solution assay instrument, intrinsic densitometry, KED, LED, XRF, HKED, and IDGS. Neutron assay techniques included NCC and Neutron Multiplicity Coincidence Counter.

Based upon evaluated performance, reliability, design criteria, operation methods, maintenance requirements, and estimated costs for each possible measurement method, we recommended potential methods for various measurement points in the main process area and the co-denitration process area. The minimum measurement method/instrumentation requirement for NRTA measurements at RRP could include the following:

- IDMS for input dissolver solutions (concentration and isotopic abundances),
- Titrimetry (MacDonald and Savage) or CPC for product solution (concentration),

- KED for product solution (concentration),
- HRGS for product solution (isotopic abundances),
- IDGS for input dissolver and intermediate process solutions (concentration and isotopic abundances),
- HKED for input dissolver solutions (concentration),
- NCC/HRGS for MOX product canisters, and
- LED for intermediate process solutions (concentration).

The number of instruments needed will depend on the throughput of RRP. Redundancy and spare parts for each instrument should be considered.

NDA has many advantages over DA, such as timeliness, small sampling errors, less cost, ease of use, less maintenance, and waste minimization, and should be used whenever possible. Automated and unattended measurement systems are also recommended.

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