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NEAR-REAL-TIME VERIFICATION APPROACHES FOR THE PROCESS AREA OF REPROCESSING PLANTS

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Abstract

Adoption of near-real-time accountancy in large reprocessing plants will necessitate more timely verification. We discuss techniques and instruments that are suitable for on-site verification of input, output, waste streams, and in-process inventory estimation of tanks, solvent extraction contactors, and concentrators. Calculations show that estimates of solvent extraction contactor inventories may make an insignificant contribution to the total uncertainty of the material balance, relative to the contributions by transfer and process tank inventory measurements.

1. Introduction

Near-real-time accounting (NRTA) is under consideration for improving accounting timeliness in reprocessing plants. For both conventional accounting and NRTA, the operator must measure all transfers into and out of the facility, and the inspector must verify these measurements.

The major difference between NRTA and conventional accounting is that for NRTA the in-process inventory must be measured or estimated monthly rather than annually. Therefore, at least on a monthly basis, all nuclear material in process tanks, solvent extraction contactors, and concentrators must be estimated or measured by the operator, and these measurements must be verified by the inspector.

Performing on-site verification measurements will certainly improve timeliness. Such measurements may also be inherently more accurate, particularly if the instrument is on line and sample-taking can be avoided. The criteria we used for choosing measurement verification techniques included:

- necessary inspector skill or training,
- time required for measurement (actual time as well as inspector's time) and sample throughput,
- ability to automate data acquisition and interpretation, and
- precision and accuracy.

In many cases, we have selected instruments and techniques that are already available to the IAEA or that are currently under test.

We considered techniques that may be applicable to input, output, waste streams, and in-process inventory estimation.

2. The Verification Problem

The data available to the inspector for verifying a materials balance (MB) may include the operator's declared measurement results, the inspector's independent measurements, or usually a combination of the two measurements. Verification using independent measurements is the desired method because the state must be viewed as a potential adversary. When verification through independent measurement is not possible, the inspector must authenticate the operator's measurements. Likewise, it is important that the inspector verifies all components of the MB (i.e., input, output, and inventories). Lack of independent information will result in inspectors dependence on the operator's data and may lead the IAEA to an incorrect conclusion or none at all.

With limited resources, the inspector must decide which measurements are most significant and therefore require the most verification resources.

Certainly all input transfers into the chemical processing area and all plutonium product outputs must be verified; for waste streams, the answer is not as unequivocal. If liquid waste streams have been verified to contain consistently low concentrations of plutonium (such as <0.01 g/L) and the volumes are within normal bounds, a statistically derived random attribute measurement by the inspector may suffice. In the measurement of leached hulls, the inspector must authenticate the operator's measurement.

For process inventory verification, the inspector should perform independent measurements on the contents of large buffer storage tanks in the process as well as on product storage tanks. For smaller tanks, especially those that consistently contain only small amounts of plutonium, a statistically derived attribute measurement may suffice. It is difficult to sample or measure concentrators/evaporators; however, if these are operated in batch mode, samples and measurements can easily be taken from the concentrated solution in the catch tank that follows the concentrator. Verifying the in-process inventory of solvent extraction contactors is related to significance of the contactor inventory relative to other inventory and facility throughput.

3. Verification of Transfers

Input.

For light-water reactor (LWR) fuel, the input dissolver solution will contain ~0.2-1% of plutonium relative to uranium in a total concentration of 100-300 g/L with associated fission products and transuranics. Therefore measurements must be made in a high gamma-neutron field, or appropriate separations must be effected. Separation may be accomplished by ion exchange, for example, on resin beads, by liquid chromatography, or by solvent extraction.

Some possible rapid verification techniques for input samples are summarized in Table I.

TABLE I. Verification Techniques for Input Samples

Method	Separation	Precision	Ref
IDMS	Resin bead	~1%	1-6
Gamma ray	Resin bead, other?	?	7-10
XRF-densitometry	None	<1%	11-12

Product

Product samples are concentrated to 100-300 g/L of plutonium, free of fission products and uranium. Although analysis is easier than for input material, one must be careful that time between sampling and analysis is not so long that concentration changes from solvent radiolysis. Some possible verification methods are summarized in Table II. The blind chemical analysis[22] has been applied to input samples for fast breeder fuels but has not been demonstrated for LWR fuels.

TABLE II. Verification Techniques for Plutonium Product

Method	Precision	Ref
K-edge External source	Pu 0.6%	12-16
K-edge Internal source	?	17
Gamma-ray absorption	Pu 6%	18
Passive neutrons	Pu 0.5%	19-21
Blind chemical analysis	Pu <0.5%	22

Wastes

High-level liquid wastes generally are collected as a batch and analyzed chemically by the operator. The inspector can accept operator declared values (plutonium content is low relative to product) or verify random samples using his own measurements.

Leached hulls could be verified using active neutron techniques, such as the ^{252}Cf shuffler[23] or passive neutron or gamma-ray methods.[24]

4. Verification of Process Tanks

For tanks, both the volume and concentration need to be verified. Volume can be verified using an inspector-owned precision pressure transducer connected to operator dip tubes,[25,26] or using other level measuring devices, such as time domain reflectometry or acoustic probes.[27-29]

Concentration can be obtained from density probes[30,31] or from conventional chemical analysis of samples.[32] In-line measurements can be performed using L-edge densitometry,[33] x-ray fluorescence,[34] or in-line spectrophotometry.[35-40] In-line voltametry was demonstrated at Dounreay with a precision of 5%.[41] Using an off-line cell isolated from the flowing solution could improve precision to 1-2%.

5. Verification of Contactor Inventory

A major problem in applying near-real-time accounting for reprocessing facilities arises in estimating or measuring the inventory of solvent extraction contactors. Two techniques have been proposed for determining nuclear material in contactors. One method relies on estimation of the inventory from process operating condition. For example, Beyerlein and Geldard have developed simplified models for estimating the inventory in mixer-settlers[42] and pulsed columns.[43]

Estimation of solvent extraction contactor inventory from process data is being used at the fast breeder fuels reprocessing plant at Dounreay and is the proposed method for the British Nuclear Fuel Limited (BNFL) LWR processing plant, THORP, under construction at Sellafield. Much of the required information for these theoretical models is proprietary, and they are reluctant to give the information to the IAEA.

The second method determines contactor inventory by direct measurement. This was investigated by Ehinger at the BNFP.[44] In this technique, the differential pressure is measured between two probes inserted into the column, one at the bottom of the column (aqueous disengaging section) and the other in the vapor space above the organic disengaging section. In practice, the weight of solution in the column is determined for specific organic to aqueous flow ratios, with only the organic and aqueous solvents present but no uranium or plutonium. Once heavy metal (uranium and/or plutonium) is introduced, the difference between the loaded weight and the "blank" weight is a measure of the heavy metal inventory in the column. For good measurements, the noise from the pulsing action of the columns must be filtered out. This method also may suffer from reluctance on the part of facility operators to make the density probes accessible to the IAEA inspector and

from the inspector's perceived inability to independently verify the concentration derived from the density

We propose considering the use of the process flow-sheet information to determine the amount of plutonium in the solvent extraction contactors in the plutonium purification cycle of a reprocessing facility. The process flow sheet specifies an approximate concentration level of plutonium in each of the four solvent extraction contactors. Each column will have some variability to its plutonium inventory, which is caused by variations in parameters such as organic and aqueous flow rates into the columns and the plutonium concentration entering the 2A column. The plutonium concentration entering the 2A column varies with the type of fuel being processed (that is, whether boiling-water reactor or pressurized-water reactor fuel is being processed) and the burnup of the fuel.

Whether process design information can be applied to the solvent extraction contactors will depend on several parameters. The total uncertainty in measurements for NRTA will be a combination of errors associated with transfers through the process and with errors in measurement or estimation of material in the process. If the material amounts in contactors are small compared with the amount of material in measurable items such as tanks, then the errors in the tank measurements will tend to dominate, and error uncertainties for the contactors may become insignificant. Also, if the material amounts in the contactors are small compared with the throughput of the facility, errors associated with transfer measurements will tend to dominate, and errors associated with the contactor inventory will be relatively small.

The question of whether process design information for the contactors can be used to estimate the plutonium in solvent extraction contactors is then reduced to a question of the relative contribution of errors associated with transfer measurements and the inventory of the contactors and with the other process tanks. The characteristics of the MB equation must be examined on a plant-specific basis to determine the applicability of this approach. The normal operational variation in contactor inventory must be considered.

We have modeled the error contributions from measured throughputs (input and output), measurable inventory in process tanks, and unmeasurable inventory in solvent extraction contactors. The model covered the range of throughputs and inventories in existing commercial reprocessing plants and those plants expected to be in operation by the end of the century.

The total system (transfer and inventories) standard deviation for a 10-day accounting period was calculated as a function of contactor inventory (0-20 kg) and tank inventory (0-200 kg) for facilities of low throughput (5 kg/day, 100 kg/10-day accounting period) and high throughput (50 kg/day).

The data shows that for the low inventories (both tanks and contactors) the errors are throughput dominated for low and high throughput. At high throughput the throughput errors dominate at even high inventories. For the low throughput case and a tank inventory $\geq$40 kg, contribution from uncertainties in contactor inventory become significant (>0.5 kg) only for contactor inventories $\geq$10 kg, and then only for contactor inventory uncertainties >50%.

Similar analyses assumed uncertainties in contactor inventory of 25% rather than 10%. In the high-throughput case, uncertainties still are throughput dominated. In the low-throughput case, contactor inventory uncertainty can become significant (>0.5 kg) for the case of low tank inventory when contactor inventory exceeds ~5 kg.

The study indicates that for many cases, the uncertainty in plutonium content of solvent extraction contactors is small compared to uncertainties in measured transfers and measurable inventory. It is suggested that in those cases, contactor inventory can be inferred from operators' data and need not be verified by measurement during process operation.

This type of approach can be used to assess the significance of contactor inventory uncertainty to overall system measurement uncertainties for any facility design. If the contribution from contactor inventory uncertainty is small relative to throughput and measured tank inventory, the need for independent inspector verification of these measurements becomes questionable. From verification of plant design, the inspector may be able to assume declared flow-sheet values for contactor inventory, provided that the operator cannot divert within the inventory uncertainties. Application of this approach will be facility specific for any design, and the facility should be modeled before conclusions can be drawn on its applicability.

5. Verification of Concentrators

The plutonium from the final plutonium purification cycle generally is concentrated to 250-350 g/L for storage or shipment to a plutonium oxide conversion facility. The concentrator can be operated in batch mode or continuously. For batch operation, MBs can be drawn when the evaporator is empty, as has been suggested in studies of NRTA for the Tokai facility [45]. In this case, independent verification of the concentrator contents is not necessary except to verify by attributes measurements that the concentrator is empty. This can be accomplished with, for example, a simple neutron detector.

For continuous operation, the problem is more complicated. For a plant such as Wackersdorf, in which the concentrator inventory is small relative to the inventory in the remainder of the facility, we propose acceptance of operator-declared inventory for steady-state operation because uncertainties in the overall MB equation would be dominated by uncertainties in the throughput and in tank inventories. Therefore, verification considerations would be as proposed for contactors. If the inspector is sure that the operator cannot divert within the measurement uncertainties

Alternatively, if continuous evaporator operation is used and it is necessary to measure evaporator concentration, an approach like that at Dounreay could be used. A recycle-loop is installed on the evaporator with a gamma-ray absorptiometer on the loop. The absorptiometer triggers withdrawal of concentrate when a 350-g/L concentration is detected. Control of nitrate product to ±5-10 g/L (1.4% to 2.8%) is claimed. By participating in the various aspects of the measurement control program for the absorptiometer, the inspector should be able to verify concentrator concentration.

7. Summary and Conclusions

Near-real-time accounting is under consideration as a technique for improving the timeliness of accounting in reprocessing plants. For verification of a NRTA system, the inspector should be able to verify transfers into and out of the process and in-process inventory during a 30-day accounting period.

For transfers, we consider the significant verification problem to be at the input accountability tank and the product output. For high-throughput facilities, these measurements will dominate in-process inventory measurements in terms of contribution to overall measurement uncertainties. The most precise verification of the input accountability tank will consist of shipping samples to an IAEA laboratory for isotope-dilution mass spectrometric analysis. More timely analyses can be performed on site using quadrupole mass spectrometry, x-ray absorption-edge densitometry, and x-ray fluorescence or gamma-ray spectrometry. High-precision verification of plutonium products can be performed by shipping samples to an IAEA laboratory for chemical analysis. Timeliness can be improved by x-ray absorption-edge densitometry or gamma-ray densitometry on site.

Process tank inventory can be verified by a combination of volume measurement (using dip-tubes and inspector-owned electromanometers) and concentration measurements, either inferred from density probes (measured in-line by x-ray absorption-edge densitometry) or in-line concentration measurements under operator control but with inspector participation in measurement control programs.

Solvent extraction contactor inventory could be measured by dip-tubes. In most cases, it may be adequate to estimate inventory from process operating data because the contribution of inventory uncertainty may be insignificant to overall measurement uncertainty over a 30-day accounting period.

Concentrator inventory may be estimated from process operating data measured by gamma-ray densitometry or, for batch-operated concentrators, determined analytically when the concentrator is emptied.

We do not attempt to recommend the optimum verification techniques for a reprocessing plant. This will depend to a large degree on process design, operating considerations, national laws, and inspector needs and capabilities.

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