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ISPO TASK F.9
DEVELOPMENT AND APPLICATION OF
ISOTOPE CORRELATION TECHNIQUES

ISOTOPIC SAFEGUARDS TECHNIQUES

JUNE 1978

USA

IAEA

**PROGRAM FOR
TECHNICAL ASSISTANCE
TO IAEA SAFEGUARDS**

Department of Energy
Safeguards & Security

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ISOTOPIC SAFEGUARDS TECHNIQUES

to
International Safeguards Project Office
Program for USA Technical Assistance to the
International Atomic Energy Agency

by
C.L. Timmerman

June 1978

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ISOTOPIC SAFEGUARDS TECHNIQUES*

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The purpose of this paper is to explain and illustrate the idea and uses of isotopic safeguards techniques. The paper maintains a generalized, simple approach to facilitate understanding of the techniques. Once understood, the application, demonstration, and implementation of isotopic safeguards techniques becomes much easier.

Introduction

As uranium is burned in a nuclear reactor, plutonium is produced. Natural laws control this process, yielding simple relationships between the beginning and ending isotopic concentrations of the nuclear fuel. That is, the amount of plutonium produced is related to the amount of uranium remaining. The concentrations of many of the isotopes of both elements (U and Pu) can also be used to form useful relationships. These relationships, called isotopic functions, have become a prime tool in developing a reliable, straightforward method for verifying measurements of the total amount of plutonium and its isotopes produced in the nuclear fuel. The need to verify the amount of plutonium and other isotopics is illustrated by Figure 1. The fact that this technique would fill the material accounting safeguards gap in the fuel cycle between the fuel fabricator and the reprocessor is potentially its greatest asset. Isotopic safeguards has developed into a proven, reliable technique to verify the spent fuel content at the head end of a reprocessing facility.

The fact that the aforementioned relationships exist has long been recognized.(1,2) Both burnup experiments and calculational burnup codes have been used to study the transmutation of uranium to plutonium. The transmutation of isotopes is principally governed by simple first-order differential equations.(3) However, the coefficients of these equations depend on numerous core details and on the reactor operating history. The

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FIGURE 1. Why Isotopic Safeguards Are Important

complexity of the core models, the cross sections of the isotopes, and the approximations needed to solve the equations has obscured the simplicity of the relationships.

Similar relationships have been found in measurements of isotopic concentrations in batches of dissolved spent fuel at chemical reprocessing plants. These measurements represent random samples of large amounts of irradiated fuel forming batches as large as one tonne. Observations of these functional relationships provide the best accuracy obtainable because they represent actual spent fuel measurements coupled with predictions from the theory associated with burnup calculations.

The isotopic functions have been investigated under programs principally funded by the U.S. Arms Control and Disarmament Agency in the past and currently by the Department of Energy through the International Safeguards Project Office which is managed by the Brookhaven National Laboratory. The purpose of the programs has been to provide the International Atomic Energy Agency (IAEA) with a practical verification technique for safeguarding the amount of plutonium input to chemical reprocessing facilities. The programs are known as Isotopic Safeguards Techniques.

Isotopic Functions and Ratios

An isotopic function is defined as a functional relationship between the measured isotopic concentrations of uranium and plutonium, or as the total elemental Pu/U mass ratio, for a given dissolution batch. Examples of such functions include Pu/U versus ^{235}U , ^{236}U versus ^{235}U , and $(^{239}\text{Pu})^2$ versus ^{235}U . In each example the Pu/U, ^{236}U , and $(^{239}\text{Pu})^2$ are the dependent or y variables and the ^{235}U is the independent or x variable.

The slope, or isotopic ratio, of two variables is defined as the ratio of the dependent variable's irradiated value minus its initial value divided by the independent variable's irradiated value minus its initial value. The following example is provided for illustration:

$$\text{Isotopic Ratio} = \frac{(^{236}\text{U}) \text{ irradiated} - (^{236}\text{U}) \text{ initial}}{(^{235}\text{U}) \text{ irradiated} - (^{235}\text{U}) \text{ initial}}$$

The above relationship is stated more simply as $\Delta ^{236}\text{U} / \Delta ^{235}\text{U}$ for nomenclature convenience. Because the term $\Delta ^{235}\text{U}$ is used so frequently it has been further simplified to ^{235}D to indicate the depletion of the ^{235}U isotope.

Obtaining a consistency for the slope of any function is of primary importance to isotopic safeguards techniques. By having a constant slope, the functions derived will be of a linear form. The linearity provides a functional consistency of the isotopic relationship over a large exposure or burnup range. This

functional consistency is necessary for an isotopic function to be of value to the technique and is a basic criterion for determining the merit of various isotopic functions. A graphical example of the linearity of the isotopic function ^{236}U versus ^{235}U is provided in Figure 2. This example illustrates the functional consistency for various enrichment groups of three different reactors.

An isotopic ratio can be used as a consistency check for batch-to-batch measurements in a reprocessing campaign. That is, the isotopic ratios formed from measurement data should be relatively constant for batches dissolved from the same reactor fuel lot. The ratio can also be used as a verification tool. Historical data from the same reactor, or a similar type reactor, can be used to form the isotopic ratio, which is compared (within accuracy limits) to incoming batch measurements from a present reprocessing campaign. In this way previous data can be used to verify present data through the consistency of isotopic ratios.

When historical data have been accumulated which highly characterize a particular fuel, these values may be applied to future similar fuels from the same reactor (assuming of course that controlling irradiation conditions are the same). The use of historical data to check successive discharges from the same reactor is illustrated in Table I for the Yankee Rowe reactor. All the Yankee fuel in this example is stainless steel clad and the same design. Reactor lots processed (10-20 tonnes U) at the chemical plant differ only in the enrichment and exposure. Two features of the empirical correlations are illustrated in Table I. First is the constancy of Pu/U versus ^{235}D for material of the same initial enrichment and design for a PWR. Second is the empirically derived expression relating the term $(\text{Pu/U})/^{235}\text{D}$ to the initial enrichment. Each batch reported by the operator is tested by forming the ratio between Pu/U and ^{235}D . If all data check within statistical limits of the historical values and within direct IAEA measurements of input and product samples, the Pu/U ratio of input to the campaign is verified.

The case of no historical data but a high degree of consistency is illustrated by the data in Table II.(4) In this case, advantage is taken of the consistency of the data over a wide range of exposure, e.g., the Pu/U varies widely but the isotopic ratio is nearly constant. The general verification procedure outlined above is used. Each batch reported by the operator is tested to see if it differs from the majority of the data. If no significant differences are found, the verification of specified isotopic functions has been shown to a given accuracy.

Pu/U Ratio Method(5)

There are presently no adequate safeguards measures for checking the content of nuclear fuel from the fabrication facility through the reactor to input at a reprocessing plant.

FIGURE 2. Uranium-236 Weight Percent as a Function of ^{235}U Weight Percent

Table I
Comparison of Plutonium Results by Empirical
²³⁵U Depletion Method to Measured Values
Yankee Rowe Fuel

<u>No. of Dissolver Batches</u>	<u>²³⁵U₀ Wt. %</u>	<u>²³⁵D</u>	<u>Grams Pu* Tonne U ²³⁵D</u>	<u>Empirical Method Grams Pu Tonne U</u>	<u>Measured Grams Pu Tonne U</u>	<u>% Difference</u>
16	3.404	.7981	5,727	4,571	4,530	0.91
14	3.406	1.0349	5,726	5,926	5,982	-0.94
11	4.101	1.5252	5,376	8,200	8,257	-0.69
7	4.101	1.4276	5,376	7,675	7,628	0.62
11	4.935	1.9820	4,957	9,824	9,899	-0.76
12	4.935	2.1594	4,957	10,703	10,692	0.10
11	4.941	2.0536	4,954	10,173	10,099	0.73

*Obtained by following equation:

$$\frac{\text{Grams Pu/Tonne U}}{^{235}\text{D}} = .7439 - .503 (^{235}\text{U}_0)$$

Table II (4)
Example Of Pu/U And Istopic Consistency
Yankee Rowe Core V Data

Raw Data

Input Batch #	Exposure MWd MTU	Total Pu g/TU	<u>Uranium Isotopics</u>		<u>Plutonium Isotopics</u>			
			²³⁵ U Wt. %	²³⁶ U Wt. %	²³⁹ U Wt. %	²⁴⁰ Pu Wt. %	²⁴¹ Pu Wt. %	²⁴² Pu Wt. %
1	9,700	4,968	3.176	0.2209	85.05	10.29	4.01	0.36
20	13,900	7,104	2.796	0.2997	79.35	12.53	6.65	0.78
10	15,800	7,930	2.675	0.3253	77.57	13.47	7.37	1.04
22	19,700	8,863	2.471	0.3589	75.14	14.54	8.29	1.34
18	21,400	9,794	2.254	0.3993	72.69	15.46	9.18	1.75
16	25,600	11,119	2.046	0.4454	69.31	16.34	10.85	2.50

Isotopic Ratios

Input Batch #	Exposure MWd MTU	Total Pu g/TU	<u>Pu/U</u>		<u>Pu/U</u>		<u>²⁴⁰Pu</u>	
			²³⁵ D %	Dev.	Δ ²³⁶ U %	Dev.	²³⁹ Pu (100 - ²³⁹ Pu)	Dev.
1	9,700	4,968	5,371	-0.52	27,463	-0.01	7.748 E-3	2.76
20	13,900	7,104	5,448	0.91	27,334	-0.48	7.387 E-3	-2.04
10	15,800	7,930	5,417	0.33	27,516	0.19	7.486 E-3	-0.72
22	19,700	8,863	5,441	0.78	27,792	1.19	7.519 E-3	-0.28
18	21,400	9,794	5,306	-1.72	27,258	-0.75	7.535 E-3	-0.07
16	25,600	11,119	5,413	0.26	27,427	-0.14	7.567 E-3	0.35
			$\bar{X} = 5,399$		$\bar{X} = 27,465$		$\bar{X} = 7.540 E-3$	
			$\sigma = 0.98\%$		$\sigma = 0.67\%$		$\sigma = 1.58\%$	

Where: $^{235}D = \text{wt. \% } ^{235}U \text{ final} - \text{wt. \% } ^{235}U \text{ initial}$
 $\Delta ^{236}U = \text{wt. \% } ^{236}U \text{ final} - \text{wt. \% } ^{236}U \text{ initial}$

The need to verify the measured amount of plutonium in spent fuel at the input to a reprocessing plant is the basic reason for developing the isotopic safeguards concept. The plutonium-to-uranium ratio method is used to independently determine the plutonium content of a reprocessor's dissolver batch. The amount of plutonium at the input of the reprocessing plant is derived by the following equation:

$$\text{Plutonium at Input} = \text{Pu/U ratio} \times (\text{Initial Uranium} - \text{Burnup}).$$

Measurements from three sources, two of them independent of chemical reprocessing, are used in the above equation. The initial uranium measured at fabrication must be measured accurately for the technique to be valid. Data(6) have proven this accuracy to be true, for the data have shown the fabricator's measurements to have a 0.15% relative precision and a 0.05% relative standard deviation of the systematic error. Correcting the initial uranium for burnup introduces the second independent source of information, the amount of heat produced by the uranium during irradiation. This calculated burnup is generally $\pm 5\%$ of the true value on an assembly level.(7) This error is not very large when one considers that only 2 to 5% of the initial uranium, depending on the burnup, is burned during irradiation in a reactor core. The third source of information is the Pu/U ratio obtained from the reprocessing plant. The accuracy of the Pu/U ratio as shown by our statistical studies is within 2% or less depending on the reactor and enrichment group. Using this ratio in conjunction with the final total uranium, obtained from the initial uranium and predicted burnup, a value for the total plutonium can be derived as stated in the above equation.

To obtain an independent value of the plutonium content using the Pu/U ratio method, it must be shown independently that the Pu/U ratios measured by the reprocessing plant are correct. By applying past similar reprocessing data and consistency checks to the Pu/U ratio and other isotopic ratios and functions, isotopic safeguards can provide an important complement and supplement to independent verification of input reprocessing batches.

It has been reported(8) that all of the observed differences are 1.0 to 1.5% or less for measured plutonium input compared to plutonium derived by isotopic safeguards methods. The 1.5% is considered a realistic upper limit, where larger differences on a batch level may require further sampling and investigation. As demonstration of the technique progresses, a definite practical upper limit can be determined.

A main process condition required to use the Pu/U ratio or other isotopic functions is that the input dissolver measurements must come strictly from the spent fuel from which they were derived. This is normally the case for head-end processes without recycle. If the acid which dissolves the spent fuel is recycled

from other sections of the reprocessing plant, corrections are required for such additions of recycle acid. Recycle of any nuclear material before measurement is made at the accountability tank must be taken into account. This procedure of measuring all recycle nuclear material holds for all verification procedures and is not just particular to isotopic safeguards. In general, verification procedures are easier to apply for the case of no recycle because only one actual measurement needs to be made per batch.

Recent Developments

A statistical package has been incorporated into the technique and is used to quantify the properties of various isotopic functions and their associated data. The use of these automated statistical procedures has provided many more meaningful results which can be derived from the isotopic data.

The statistical tools used to analyze the data are namely the paired comparison and regression analysis. A paired comparison results when a sample from a reprocessor's measurement batch is analyzed by two different laboratories. These paired comparisons can be used to compare laboratories for bias, to estimate measurement variances, and to indicate the presence of outlier batch results. The second analysis tool, the linear regression, initially involved the comparison of various linear regression approaches. After much study of these various approaches, a decision to use Deming's approach(9) was reached. Deming's approach is a model in which errors in both variables (x and y) are considered. Both an intercept model, $y = \alpha + \beta x$, and an initial point model, $y - y_0 = \beta(x - x_0)$, are utilized to provide the maximum statistical data from the regression analysis. For example, the initial point model provides a stabilizing line-fitting method for data having a small burnup range, while the intercept model supplies a more accurate fitting to larger ranged data. The decision to use this regression technique with errors in both the x and y variables resulted when it was shown(10) that the random variances for the x and the y variable were approximately equal, or at least that the error variance of y was not substantially larger than the error variance of x . This fact illustrated that errors occurred in both variables.

After choosing the appropriate statistical approach and corresponding models, these statistics are useful in defining the magnitude of the variance components along with identifying anomalous or outlier data. The regression analysis also provides a least squares type linear fitting of the data points grouped by enrichment. This fitting supplies a more accurate estimate of the slope and intercept than was previously possible. However, possibly the most important feature of the statistics is that they specify the accuracy basis for acceptable verification results for each isotopic function.

Rough ideas had been used in the past to identify useful linear functions. Because of the large number of isotopic variables and functions possible, automated decision and identification techniques needed to be incorporated. The resultant program determines the isotopic variables, and eliminates any redundant functions that occur. The pairing of the variables utilizes the idea that the relative change in one variable's concentration over a sample burnup range should be approximately equal (within 0.1%) to the relative change in the other variable's concentration over the same burnup range in order to form a linear function. Computer calculations have greatly improved the efficiency and precision with which linear functions can now be found. The following are examples of beneficial, linear functions which have been studied by this method to date:

$^{240}\text{Pu} \times ^{241}\text{Pu}$	vs	$^{235}\text{U} \times ^{242}\text{Pu}$
^{235}U	vs	$^{239}\text{Pu}^2$
$^{239}\text{Pu} \times ^{240}\text{Pu}$	vs	$^{235}\text{U} \times ^{241}\text{Pu}$
$^{239}\text{Pu}^2 \times ^{240}\text{Pu}^2$	vs	^{240}Pu
$^{239}\text{Pu} \times (100 - ^{239}\text{Pu})$	vs	$\Delta(^{235}\text{U} \times ^{239}\text{Pu}^2)$
^{240}Pu	vs	$\Delta(^{235}\text{U}^2)$
^{240}Pu	vs	$^{239}\text{Pu} \times (100 - ^{239}\text{Pu})$
$^{239}\text{Pu}^2 \times (100 - ^{239}\text{Pu})/^{235}\text{U}^2$	vs	$(100 - ^{239}\text{Pu})$
Pu/U	vs	$(100 - ^{239}\text{Pu})$
Pu/U	vs	^{235}D
^{236}U	vs	^{235}U

Data Base

A substantial data base has been collected to date. A listing of the data sets according to type, reactor, and quantity is provided in Table III. These data have increased the safeguards benefit of isotopic functions by providing a sufficiently large set of data from which to work. Calculated burnup data are included in the data base, which complement the empirical methods, and this has provided greater theoretical insight into the technique. The features of the isotopic functions observed for the measured data have also been observed for the calculated data, thus supplying a high level of confidence in the isotopic functions.

An in-depth analysis of the data base using the statistical package and reactor operator and design information has led to a labeling index as to the qualities and properties of every data point in each data set. A list of the markings used for this

Table III
Isotopic Safeguards Data Base

Reactors	Number of Input Batches		Number Of Burnup Samples	Number of Sets Calculated Data
	Measured	Remeasured		
I. Pressurized Water				
1. Connecticut Yankee				1
2. Diablo Canyon				1
3. Fort Calhoun				1
4. Indian Point 1	20	20		1
5. Point Beach 1				1
6. San Onofre 1			6	3
7. Saxton			67	1
8. Sena	27	29		
9. Trino	30	48	23	3
10. Vver			5	
11. Yankee Rowe	89	82	35	5
12. H. B. Robinson 2			3	
Total	166	179	137	16
II. Graphite Moderated				
1. AGR				1
2. Calder Hall				1
3. Chapel Cross			24	
4. NPR	11			1
5. Windscale AGR	10			
Total	21		24	3
III. Boiling Water				
1. Big Rock Point 1	23			3
2. Browns Ferry 1				2
3. Dodewaard	13	6	13	
4. Uresden 1	62	18		3
5. Garigliano	31		18	
6. Humboldt Bay	20	20		3
7. KRR	15	29		1
8. LaCrosse				1
9. Nine Mile Point 1				1
10. Oyster Creek 1				1
11. VAK	3	4	10	1
12. JPDR-1			30	
Total	167	77	71	16
IV. Heavy Water				
1. NPD (Candu)	9	9	6	1
2. Douglas Point	3	6		1
3. Gentilly 1				1
4. Pickering				1
5. NRU			24	
Total	12	15	30	4
V. Fast Reactor				
1. EBR-11			18	
Total 35 Reactors	366	271	280	39 Sets

quality index is given in Table IV. It must be understood that most of the data falls under the ordinary category, for this is basically an index for marking and labeling unusual and outlier points to provide a clearer understanding into their meaning.

Our data base makes an effective safeguards tool in itself because of the isotopic functions now developed. Measurement histories of particular reactors and/or fuels become inherent as the data bank increases. Perhaps the most important aspect of the data base is that the isotopic functions determined from new reprocessing measurements should be consistent with past results and with properties defined by the data base for that type of reactor fuel. In this way the data base contributes in a positive way to the future analyses of reprocessing input measurement.

Table IV
Quality Index Listing

<u>Property</u>
Normal data point
Exposure averaging
Enrichment averaging
Exposure and enrichment averaging
Fringe effects
Exposure averaging and fringe effects
Cladding differences
Unirradiated assembly
Uranium outlier
Plutonium outlier

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