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The logo for ESARDA, featuring a stylized blue triangle pointing left, followed by the word "ESARDA" in a bold, blue, sans-serif font.

# Bulletin

EUROPEAN SAFEGUARDS RESEARCH AND DEVELOPMENT ASSOCIATION

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This section of the Bulletin has been created in order to encourage our readers to express their views on anything associated with ESARDA activities. All suggestions and comments are very welcome.

# Scientific Papers

## International Standardization In Semiconductor Detector Spectrometry

Please note that this article was written in 1999, therefore  
references to events taking place may be out of date

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### Abstract

The rôle of standards and standardization in providing the unity of measurements is discussed. Special attention is given to international standards in spectroscopy measurements issued by international bodies. The importance of standards for data presentation and instruments is growing as more worldwide programs are implemented. The standards related to spectrometry measurements and prepared by the Technical Committee 45 «Nuclear Instrumentation» of the International Electrotechnical Commission are reviewed. Information on new projects in this field will be discussed.

## 1. Introduction

In the last years the achievements in nuclear science, industry and power have resulted in the use of nuclear technology in various branches of science and industry. Simultaneously, measurement methods to characterize ionizing radiation fields, their sources and radiation interactions with matter were developed. In turn, this nurtured the development of different ionizing radiation measurement methods and improvements in measurement technology and related instrumentation. The wide application of radiation in everyday human activity required corresponding measures to provide the uniformity of terminology and measurements not only on a national level, but also on an international scale. For this reason, international organizations with long-term experience in preparing normative documents began the development of standards, recommendations and guides for describing ionizing radiation measurements and related subjects.

First of all, among them were the International Organization for Standardization (ISO), the International Electrotechnical Commission (IEC), the International Organization of Legal Metrology (OIML), the International Commission on Radiation Units (ICRU), the International Commission on Radiation Protection (ICRP), and the International Atomic Energy Agency (IAEA). The first two mentioned bodies were highly experienced in developing and issuing international standards and the remaining - in preparing recommendations, guides and technical reports. In addition the IAEA has also begun work on international standards. Due to the highly specific content many of the developed documents constitute a major part of measurement quality metrological support.

In the areas of interest to nuclear spectrometry, the ISO standards are prepared by Technical Committee 85 «Nuclear Energy», in IEC - by Technical Committee 45 «Nuclear Instrumentation». The IEC develops the so-called electrotechnical standards, as ISO - the non-electrotechnical ones.

In this report on ionizing radiation spectrometry, standards prepared and issued by these bodies will be discussed. In common spectrometry measurement practice these documents should be followed.

The standards and documents are referenced by their document designations. These designations are constructed as follows:

- first will be the abbreviation of the organization that issued the document (e.g. "IEC"),
- next comes the publication number (e.g. "61435"),
- next is the year of issue in parentheses (e.g. "(1996)"),
- followed by the document full name (e.g. "Nuclear Instrumentation - High purity germanium crystals for radiation detectors").

Therefore the entire reference becomes «IEC 61435 (1996) Nuclear Instrumentation - High purity germanium crystals for radiation detectors».

As in any dynamic field, some documents related to spectrometry are under development in all of the organizations. The designation of the documents in preparation is determined by the certain international organization doing the work and is written in *italic*.

In this report information on standards in force will be given. Information on older standards may be found in references from individual organizations. The IEC maintains a list on the web <http://www.iec.ch/>

It is broadly recognized that compliance with an international standard is a desire to produce and maintain quality work and products. As a rule international standards are the results of discussions of experienced specialists from many countries who have a very good understanding and have worked a long time in the area of the standard's subject. These standards represent the «state-of-the-art» of the technical level in the world. For this reason, some of the descriptions of certain topics in the text of the standard contain complete explanations - like a tutorial on the subject. For example, descriptions would be given for the measurement method and the data processing algorithm. When the reader of the standard has completely read and understood the standard, he will be well informed about the subject of interest. Future engineers, researchers, scientists and students at the beginning of their education and work would be highly recommended to be acquainted with standards because these documents determine the fundamental rules in this area.

## 2. General purpose standards

In this part the main standards relevant to ionizing radiation related areas are reviewed.

### 2.1. Marking

Whenever and wherever work with ionizing radiation is performed, it is necessary to warn people in the area about this. To inform people, a well-known tri-petalled symbol is displayed in such areas. The symbol is described in "ISO 361 (1975) Basic ionizing radiation symbol".

This sign is used world-wide to indicate working places, installations, vehicles, cargo, locations, etc., where ionizing radiation and radioactive objects may be present, used and located.

### 2.2. Quantities and units

To characterize ionizing radiation, phenomena, objects and measurement results, quantities and units are used that are given in

- "ISO 31-0 (1992) Quantities and units - Part 0: General principles".
- "ISO 31-9 (1992) Quantities and units - Part 9: Atomic and nuclear physics".
- "ISO 31-10 (1992) Quantities and units - Part 10: Nuclear reactions and ionizing radiations".
- "ISO 1000 (1992) SI units and recommendations for the use of their multiples and of certain other units".

Some specific quantities and units for ionizing radiation measurement (mainly related to dosimetry) are given in ICRU documents.

### 2.3. Terms and definitions

To provide better understanding and communication among specialists, the IEC and ISO issued international dictionaries. These dictionaries contain terms and definitions related to nuclear physics, energy and nuclear instrumentation. Examples are:

- "ISO 921 (1997) Nuclear energy - Vocabulary",
- "IEC 50(393) International Electrotechnical Vocabulary Chapter 393: Nuclear Instrumentation: Physical Phenomena and Basic Concepts",
- "IEC 50(394) International Electrotechnical Vocabulary Chapter 394: Nuclear Instrumentation: Instruments".

Nuclear industry and power are dynamic and are constantly expanding the technology. As they make progress, new meanings and terms appear frequently. Thus, the work on updating the vocabularies is done in IEC and ISO working bodies continuously. These updates are distributed by regularly issuing vocabulary addenda.

These dictionaries are quite large, so for convenience of the user of an individual standard, many standards include a section with specific terms and definitions relevant to the standard.

### 2.4. Measurement uncertainty

Often in spectrometry one has to work with the measurement results, summarize them, calculate the uncertainty and make conclusions on how well the results describe the actual physical situation. Due to the different ways of thinking, researchers, scientists and engineers expressed the final results and uncertainties in the manner based on the common way of representing measurement results in their own special field. An agreed approach in this subject did not exist, so

it became difficult to compare results among these groups. In 1993 ISO issued a very important and useful document - "ISO (1993) Guide to the expression of uncertainty in measurement" prepared by 7 international bodies: IEC, ISO, OIML, International Bureau of Weights and Measures, International Federation of Clinical Chemistry, International Union on Pure and Applied Physics, International Union on Pure and Applied Chemistry.

This document describes the way to express measurement uncertainty and reconsiders some earlier established approaches in describing measurement errors.

### 3. Standards related to Ionizing Radiation Spectrometry

In the next section, a brief review of international standards and documents that have been issued or are still being prepared is given. For convenience, the standards are grouped by topics.

#### 3.1. Spectrometer metrological parameters

In many instances, manufacturers with low technical ability may misstate certain spectrometer parameters. This can increase the product cost without providing any improvement in operation or results obtained. For example, the spectrometer technical documentation may be given in the words of a standard, but may not be measured according to the standard. The last statement is very important when it is necessary to verify and calibrate the spectrometer at the work place. From the metrology point of view this means that the manufacturer must have and use the same radiation sources, measurement means and measurement procedures as the user. Thus allowing them to achieve reproducible and confident spectrometer parameter values.

For this purpose IEC developed a guide reflecting state-of-the-art metrologically supported Alpha, Gamma and X-ray spectrometers with different detectors

- "IEC 1276 (1994) Nuclear Instrumentation - Guidelines for selection of metrologically supported nuclear radiation spectrometry systems."

This document contains tables with technical parameters for

- precision spectrometers,
- spectrometers intended mainly for scientific research,
- general purpose spectrometers (less expensive than previous ones),
- other types of spectrometers ( for application in process control, education, etc.).

#### 3.2. Test methods

To determine parameters of spectrometers, detectors used in them, multichannel analyzers, spectrometry amplifiers and other equipment, various measurement procedures for tests and calibration are used. They are stated in the following:

- "IEC 61452 (1995) Nuclear Instrumentation - Measurement of gamma-ray emission rates of radionuclides - Calibration and use of germanium detectors",
- "IEC 60759 (1993) Standard test procedures for semiconductor X-ray energy spectrometers",
- "IEC 60759 (1991) Amendment #1",
- "IEC 60333 (1993) Nuclear Instrumentation - Semiconductor charged particle detectors - Test procedures",
- "IEC 61342 (1995) Nuclear Instrumentation - Multichannel pulse height analyzers - Main characteristics, technical requirements and test methods",
- "IEC 61151 (1992) Nuclear Instrumentation - Amplifiers and preamplifiers used with detectors of ionizing radiation",
- "IEC 60741 (1982) Multichannel amplitude analyzers: Standards for time-to-amplitude converters",
- "IEC 60830 (1987) Test methods for multichannel analyzers as multichannel scalers".

This year a standard will be issued based on document prepared in IEC Sub-Committee 45B

- "IEC FDIS 45B/209 prj. 61275 *Measuring of discrete radionuclides in the environment- In situ photon spectrometry system using a Ge detector*".

#### 3.3 Detection limits

In conducting measurements and processing results in spectrometry, one often has to make conclusions about low radionuclide (or in XRFA - element) content in the measured object. Different criteria were used by different groups to estimate the lowest detectable activity. To make the results from different groups easy to compare, the ISO is preparing a standard. The document is in the final stage of development.

- "ISO/DIS 11929-3 *Determination of the lower limits of detection and decision for ionizing radiation measurements - Part 3: Fundamentals and application to counting measurements by high-resolution gamma spectrometry without the influence of sample treatment*".

In accordance with the ISO TC 85 working plan it is expected that the standard will be issued in 1999 and will be widely used in interpreting spectrometry measurement results.

### 3.4. Ge detectors

The improved results in spectrometry measurements mainly depend on the parameters of the Ge detector. The test methods for these detectors are presented in

- "IEC 973 (1989) Test procedures for germanium gamma-ray detectors".

This document described how to determine the peak area, background distribution, peak position, energy resolution, peak/Compton ratio, relative efficiency, etc. for a Ge detector spectrum. The unified dimensions of the re-entrant (Marinelli beaker) are also given so that geometries other than point source can be used.

### 3.5. Sample containers

In IEC TC 45 the document describing the container dimensions for solid, liquid and gaseous samples will be soon finished:

- "IEC 45/429/FDIS prj. 61428 Sample containers for gamma-ray spectrometry with Ge detectors".

This document describes two container types:

- cylindrical placed on the cryostat cap,
- re-entrant (Marinelli beaker) surrounding the cryostat cap.

### 3.6. Cryostat end-caps dimensions

Gamma spectrometers with different Ge detectors are widely used in modern measurement practice. Specific and controlled size samples are used for measurements because of the necessity of calibration. To reduce the cryostat variety and to optimize the measurement geometry a standard was issued that sets the detector end caps dimensions:

- "IEC 60937 (1988) Cryostat end-caps dimensions for germanium semiconductor detectors for gamma-ray spectrometers".

### 3.7. Low background Ge detectors

Spectrometers with Ge detectors are widely used to measure very low activities. This requires the measurement to be done with the cryostat of the Ge detector places in a shield to decrease the impact of external Gamma radiation background. Many companies are delivering special detectors with low intrinsic background. But a unified measurement method and a description of such detectors did not exist. Now in IEC a method is under development that allows to characterize the gamma background in measurements with Ge detectors

- "IEC 45/430/CD prj. 61976 Nuclear Instrumentation-Spectrometry-Test methods for spectrum background determination in HP Ge nuclear spectrometry".

### 3.8. Germanium crystal

In manufacturing high purity Ge detectors it is essential to know some characteristics of the initial semiconductor material. These characteristics significantly influence the detector quality. For a long time a list of detector grade germanium characteristics and their measurement methods did not exist. Now this disadvantage has vanished after the issuing of the standard

- "IEC 61435 (1996) Nuclear Instrumentation - High purity for radiation detectors".

### 3.9. Spectrometry data exchange

After completing spectrometry measurements the information has to be either stored or transmitted for additional processing or supplied to other organizations. To provide the completeness and full understanding of measured spectra data a standard was developed:

- "IEC 61455 (1995) Nuclear Instrumentation - MCA histogram data interchange format for nuclear spectroscopy".

### 3.10. Other standards related to spectrometry

Some standards not directly related to spectrometry with semiconductor detectors are:

- "IEC 61239 (1993) Nuclear Instrumentation - Portable gamma radiation meters and spectrometers used for prospecting - Definitions, requirements and calibration",
- "IEC 61134 (1992) Airborne instrumentation for measurement of terrestrial gamma radiation",
- "IEC 61335 (1997) Nuclear Instrumentation - Bore-hole apparatus for X-ray fluorescence analysis".

In addition to these standards more or less directly related to spectrometry, the IEC alone has published more than 150 standards on nuclear instrumentation. Some of them indirectly affect spectrometry but are often used in it. These standards are related to modular systems (NIM, CAMAC, FASTBUS), low and high voltage connectors and other items.

## 4. Conclusion

This information illustrated by spectrometry applications, proves that standards reflect and fix the achieved technical level. The standards are widely used in industry and science. The standard's nomenclature is constantly growing and their contents are improving. The established IEC and ISO procedure is to periodically review (usually in 5 years) all documents - this is called the standards maintenance procedure. During the review process, the decision is made:

- to continue the use of a standard or
- to withdraw it or
- to update it in accordance with new industry and/or trade requirements, trends or achievements in science and technology.

Of course in the near future, due to the needs of industry and new knowledge gained in industry and science, existing standards will be updated and improved and new ones will be developed.

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### *Acknowledgement*

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# Sample Analysis Methods for Accountancy and Verification

## A Compendium of Currently Applied Analytical Methods

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### 1. Introduction

Reliable measurements of the amount of nuclear material form the backbone of any nuclear material accountancy system. Accurate element and isotope assay is required for establishing declarations of amounts of nuclear material. Independent verification of such declarations relies also on accurate measurements. The requirements for selection of appropriate analytical methods are derived from economic aspects (investment costs, running costs), operational aspects (speed of execution, ease of manipulations) and scientific aspects (accuracy and traceability of results).

Essentially, any analytical method needs to demonstrate its fitness for purpose, which means it has to demonstrate that the analytical goal is achievable with a reasonable effort. With time, the emphasis of the different aspects has shifted, reflecting the concentration of reprocessing into a few large facilities. Consequently, more emphasis is put on the achieved uncertainty and on cost effectiveness. This has led to the modification and optimisation of existing methods and also to the development and implementation of new methods.

### 2. Historical Background

From the early days after discovery of nuclear fission, chemical methods were used to separate and identify the elements involved. Radiometric measurements were used in a non-discriminatory way to identify on a qualitative bases the radioactivity (half-life of the separated fission or neutron capture product). Over time, the chemical methods and the radiometric methods were further developed and could be used in a more quantitative way.

However, following the introduction of accountancy and verification measurements, chemical methods were essentially used for element assay and thermal ionisation mass spectrometry was used for isotope assay. Chemical analysis allows the qualitative and quantitative determination of a sample through the measurement of its chemical or electrochemical property [1]. Radiometric analysis is based on the radiation emitted from the nucleus or the electron shell of the element under investigation and have profited from the recent revolution in computerisation and informatics. From these considerations of the historical development of the analytical methods, it appears evident to consider chemical methods as being destructive and most radiometric methods as non-destructive. This, however, is an over simplification.

According to the IAEA safeguards glossary [1], non-destructive assay (NDA) is defined as: "Measurement of the nuclear material content or element or isotopic concentration of an item without producing significant physical or chemical changes to the item".

The IAEA safeguards glossary does not give an explicit definition of destructive analysis. However, the above quoted definition of NDA implies a definition of DA. This can be formulated as follows: "*Destructive analysis – Measurement of the nuclear material content or element or isotopic concentration involving a measurement technique, which is operated in a way that the sample being measured is not returned to the batch it was taken from, hence introducing a significant change.*"

The ESARDA Working Group on Destructive Analysis (WGDA) has been working in the spirit of this definition for a long time. A first attempt at phrasing such a definition was made at the ESARDA Symposium in Helsinki, 1998 [2].

The Working Group consequently undertook to establish a compendium of analytical methods reflecting the range of measurement techniques currently applied in nuclear analytical laboratories for accountancy or for safeguards verification purposes. The information contained in this document has been condensed from data provided by the laboratories represented in the Working Group. For the purpose of this compendium it is not necessary to identify individual laboratories,

their working methods or their claimed uncertainties. Therefore, the information was carefully screened and no references are provided that could reveal the origin of the data in the tables below.

Analytical methods used for process control or for measuring properties related to the quality of the products were not included in the compendium. Analytical techniques used for High performance trace analysis (HPTA), for environmental sampling or for wide area monitoring will be the subject of a separate compendium.

### 3. Analytical Methods Overview

The following tables summarise the information compiled by the working group. The information is structured according to material type. Together with information on the method itself, the tables provide estimates on the typically achieved total uncertainty under routine conditions as determined by the laboratories. These uncertainty values reflect the range of applications, from routine measurements on large numbers of samples to reference measurements carried out as carefully as possible. The uncertainty estimates requested from the laboratories were expanded uncertainties ( $k=2$ ) so covering in essence the complete uncertainty arising from the measurements. Information on the reference materials used for calibration and for method validation is also given in the tables.

#### 3.1 Measurement of Uranium Oxide and Nitrate Solution

Laboratories from plant operators, safeguards authorities and research institutes provided information. Although the number of analytical methods applied for element and isotope assay in pure uranium materials (oxides or nitric solutions) is rather limited, the handling and implementation of the methods in the various laboratories may be different.

From the information compiled above, the following observations are worth noting:

- Both chemical and radiometric methods are applied
- Chemical methods generally show a lower uncertainty than radiometric ones
- The uncertainties as declared by the laboratories vary (even for nominally the same method) by a factor of two to three
- The reference materials being used originate from three suppliers: IRMM, NBL and AEA Technology

Method	Estimated Uncertainty [%]		Comment	Reference Material
	U content	<sup>235</sup> U abundance		
Davies & Gray Titration	0.1 0.2 0.15 0.06		Method applied in different modifications	EC-NRM-101, EC-NRM-110, IRMM-106, UKU-1, UKU-2, NBL materials
Gravimetry	0.09 0.05 0.11		ISO 7476	NBS materials
X-ray fluorescence Thermal Ionisation Mass Spectrometry	0.2	0.2 0.25 0.004 0.4 0.1 0.13 0.2	Measurement of isotopic abundances: total evaporation as well as conventional techniques are applied	NBS materials, IRMM 183 - 187
COMPUCEA	0.2	0.5		IRMM tailor made materials

Table 1 Methods used for element assay and <sup>235</sup>U molar isotope abundance (enrichment) determination

### 3.2 Measurement of Spent Fuel Solution

Input solutions consist of a chemically very complex matrix due to the high amount of “impurities” (i.e. fission products, activation products and other actinides) accompanying the elements of interest, uranium and plutonium. In addition, phenomena such as radiolysis and post-dissolution precipitation may complicate the analysis of spent fuel solution. Independent of the problems affecting the analysis of a sample, the homogeneity within the input accountancy tank and the actual sample taking may limit the representativity of the sample. The discussion, however, is limited to the analytical methods.

The number of methods applied is small, due to the complexity of the material and the difficulties in handling.

Method	Estimated Uncertainty [%]				Comment	Reference Materials
	U content	<sup>235</sup> U/U	Pu content	<sup>240</sup> Pu/ <sup>239</sup> Pu		
Isotope Dilution	0.4		0.4		Different spikes and U/Pu/f.p separation methods used	CRM-137 CRM-U-005,010 MP2, MU1, NBL-126, 112a, 116 IRMM 046b, 290, 183-187 home made spikes calibrated against primary Ref. material
Mass Spectrometry	0.32	0.4	0.4	0.1		
	0.34	0.4	0.58	0.2		
	0.1	0.13	0.2	0.13		
Hybrid K-edge	0.2	0.5			Measurement time 3000 sec.	IRMM tailor made solutions

Table 2 Methods used for U and Pu assay in spent fuel solution

Basically, only two (fundamentally) different methods are applied; an active radiometric method and a chemical method (IDMS). Isotope Dilution Mass Spectrometry is applied in various modifications by the laboratories.

The following observations should be noted:

- Thermal ionisation mass spectrometry is used as measurement technique for IDMS
- Significantly different uncertainty statements are quoted for IDMS between the various laboratories
- The quoted uncertainty using LSD<sup>1</sup> spikes varies by a factor of three
- Four different chemical separation methods are applied (DOWEX<sup>®</sup> or Bio-Rad<sup>®</sup> Anion exchange, UTEVA<sup>2</sup> column extraction chromatography, TOPO<sup>3</sup> column extraction chromatography, TBP<sup>4</sup> (CCL<sub>4</sub> liquid/liquid extraction)
- <sup>238</sup>Pu is generally determined by alpha spectrometry

The potential of the IDMS technique has certainly not been exploited to its limits; most laboratories are evidently guided by the “fitness for purpose” principle.

On the other hand, the difficulties in handling spent fuel samples as required for IDMS should not be underestimated.

### 3.3 Measurement of Plutonium Oxide and Nitrate Solution

Plutonium nitrate solutions and plutonium oxide are the products of spent fuel reprocessing. The latter is the starting material for production of MOX fuel. Since pure plutonium is of strategic importance, its accurate and timely assay is a primary requirement of the analytical laboratory. Consequently, the measurement techniques applied should provide results with sufficiently low uncertainties and within reasonably short delays. A variety of measurement techniques is applied for Pu assay. Chemical methods require the plutonium to be in solution, hence PuO<sub>2</sub> needs to be dissolved. Problems affecting the uncertainty of the final result arise from the sampling and handling of PuO<sub>2</sub> powder, due to humidity uptake. Concentrated Pu solutions may suffer from evaporation effects, radiolysis or polymerisation.

<sup>1</sup>Large Size Dried spike

<sup>2</sup>UTEVA<sup>®</sup> Resin manufactured by Eichrom Industries

<sup>3</sup>TOPO, Tri-octyl-phosphine-oxide

<sup>4</sup>Tri n-butyl-phosphate

Method	Estimated Uncertainty [%]		Comment	Reference Material
	Pu content	$^{240}\text{Pu}/^{239}\text{Pu}$		
Titration	0.2 0.17 0.28 0.3		Method applied in different modifications	CRM 137 NBS 136e Cetama MP2 UK Pu3-6 NBL126
Gravimetry	0.17		ISO 8300	
Isotope Dilution Mass Spectrometry	0.52 0.13 0.2			UKPuI-80990 EC-NRM 201, 210 IRMM-041 Home made spikes
Coulometry	0.28 0.08 0.065		Method applied in different modifications	UKPu1 EC-NRM 201 Cetama MP2
Product hybrid K-edge	0.2			Home-made working standards
Thermal Ionisation Mass Spectrometry  COMPUCEA		0.1 0.07 0.13 0.07 0.2 0.2	Total evaporation and conventional techniques are applied	

**Table 3 Methods used for Pu assay in plutonium products**

Despite the variety of methods, the scatter in the individual uncertainty statements is not larger than for uranium assay. The titrimetric methods for assaying the Pu element content are of different nature: Davies & Townsend, McDonald and Savage, AgO method. Coulometry is applied in the different laboratories in different modes: controlled potential or constant current. For determination of the isotopic composition of Pu, thermal ionisation mass spectrometry (TIMS) is normally complemented by alpha spectrometry for  $^{238}\text{Pu}$  determination in order to minimise the risk of undetected isobaric interference from  $^{238}\text{U}$ . Due to the poor stability of concentrated Pu solutions, no appropriate reference materials are available for the product hybrid K-edge (PKED). Working standards need to be prepared and calibrated against primary reference materials using a primary method of measurement. Using this approach, traceability of results for the PKED can be established. Another approach that is taken in PKED measurements consists in the addition of a known amount of uranium as internal standard. Then a simple ratio measurement (unknown Pu content relative to known U content) by X-ray fluorescence provides a sufficiently accurate and traceable result. Undissolved  $\text{PuO}_2$  samples are increasingly measured by radiometric methods like passive neutron coincidence counting or by calorimetry.

### 3.4 Measurement of Mixed U/Pu Oxides

The main use of separated plutonium is the production of U/Pu mixed oxide (MOX). The analysis of MOX involves the determination of the uranium component and that of the plutonium component. Complementary, the content of  $^{241}\text{Am}$  is measured. This serves essentially to determine the date of the last Pu separation and it serves the analyst as a consistency check on the stoichiometry (adding all major metallic elements, i.e. U+Pu+Am). Usually the same analytical techniques are applied as for the pure products, uranium and plutonium, provided the individual method does not suffer from interference of the other element. For instance,  $\text{Ag}^+$  titration, Davies and Gray titration, Coulometry and IDMS may be applied to MOX equally well as to pure uranium or plutonium solutions.

## 4. Conclusion

The analytical methods used in nuclear laboratories for accountancy and safeguards verification measurements range from traditional chemical techniques to recent implementations of radiometric methods. Many laboratories still rely on chemical methods because of their superior accuracy, their traceability and the transparency of the uncertainty statement. Radiometric techniques are being increasingly used in the laboratories. However, they are often applied in a “destructive” way, i.e. requiring the sample to be optimised in chemical and physical form in order to achieve the best possible result.

Reference materials are essentially provided by four different suppliers: the New Brunswick Laboratory (NBL), the Institute for Reference Materials and Measurements (IRMM), AEA Technology and the Commission d'Établissement de Méthodes d'Analyses (CETAMA). A separate document on the availability of reference materials is under preparation and will be made available soon. Special reference materials have been prepared upon request for some of the radiometric techniques.

It has to be noted that only few laboratories follow in their analytical procedures the ISO standards that are available for a number of analytical techniques. The Working Group intends to promote a better exchange of information between the respective ISO working groups and the WGDA.

Throughout the compendium we did not specifically identify instrument manufacturers or suppliers. The intention of this document is not to provide a market survey of instrumentation, but to establish an up-to-date picture of the methodologies presently applied for the assay of U and Pu in samples of nuclear material from key points in the fuel cycle and to record the range of reported expanded uncertainties by the different laboratories.

## 5. References

[1] *AEA Safeguards Glossary, 1987 Edition, IAEA/SG/INF/1 (Rev.1)*

[2] *K. Mayer, Destructive Analytical Measurement Techniques in Nuclear Material Safeguards, Seminar on Modern Verification Regimes: Similarities, Synergies and Challenges, 12 – 14 May 1998, Helsinki, Finland; Report EUR 18681EN, p. 189 - 196*

# The Application of the Guide to Uncertainty in Measurements in Safeguards

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## Introduction

Since the 'Guide to the Expression of Uncertainties in Measurements' (GUM) was published in 1993 /1/, quickly followed by a more detailed explanation for chemical analysis applications by Eurachem /2/, the application of the new uncertainty concept has spread widely in the analytical chemistry community. Its acceptance in the nuclear field in general has been more hesitant however, despite a long history of accurate measurements in this area.

Measurement results in the field of Safeguards and Nuclear Materials Control (NMC) can have extremely important consequences. Initial advances in the field included the concepts of International Target Values and the introduction of rigorous external quality control programmes. However, the new concept of uncertainty has not been completely adopted in Safeguards and NMC, whereas other areas of analytical chemistry are moving ahead with applying the new methods of treating measurement results.

Traditionally, the concepts of random and systematic errors have up to now been commonly applied for measurements in NMC and Safeguards. These, although included in the GUM, are no longer an important part of the concept of measurement uncertainties for reasons explained below. The term 'uncertainty' was introduced in the GUM at least partly to move away from 'random' and 'systematic' errors and to bring a new clarity into the measurement process.

The definition of uncertainty given in the GUM, is associated with the measurement or the measurement process and recognises that the uncertainty of a measurement is an integral part of the measurement result. The estimation of this uncertainty, which although not always easy to carry out, is ultimately very satisfactory to the analyst, who instinctively knows the 'worth' of a measurement.

We find a change in emphasis in the GUM away from evaluation of measurement errors through repeated measurements at the same or different laboratories, towards the evaluation of a single measurement by the compilation and addition (by the propagation of uncertainties) of the individual uncertainty components in the measurement process.

## Problems with concepts of 'Random' and 'Systematic' errors

Controversy has always dogged the concepts of 'random' and 'systematic' errors. By 'random error', the assumption is made that repeated measurements can reduce the estimation of this error. When a set of meas-

urements is made, however, it is often not clear if only random error contributions (in practice not a common occurrence) or whether other sources of error are present. The measurements usually contain systematic contributions which cannot be reduced by repetitive measurements. The lack of a widespread awareness of this problem becomes much more apparent and important when dealing with results produced by modern instrumentation, which can make hundreds or even thousands of repeated measurements and produce extremely small standard deviations of the mean. Any analyst with experience of measurements will be – correctly - suspicious of very low relative errors.

The concept of 'systematic error' is often made synonymous with 'bias', whereby this model of measurement considers that after removal of the random component, what is left is a measure of the systematic component which can then be quantified. This concept is also found in the GUM: 'systematic error' is defined as the difference between the 'true' and measured value. This is however of little help to the analyst, who can rarely know the 'true' value of measurand (fundamentally speaking, the 'true value' can never be known, only approximated to). In the field of safeguards especially, where one measurement result is compared directly with a second, the concept of a 'true' value is at best a hazy idea.

Of course, if the magnitude and direction of a bias can be estimated (inter-laboratory comparisons can be a valuable tool for identifying biases) the end result can be corrected for this bias. This correction is a part of the calculation of the value of the result and not of the uncertainty, although a bias correction will also introduce an uncertainty component, which has to be included in the final uncertainty calculation. (It should be added at this point that elimination of a perceived bias is much to be preferred over post-hoc correction).

With the publication of the GUM, the vocabulary for dealing with measurements and their uncertainties has been defined. The use of the terms 'systematic' and 'random' errors are avoided as far as possible. Much vagueness has now been removed and this in turn gives more confidence in the final measurement results.

## Measurement

In Nuclear Safeguards, the definition of a measurement can be defined as a comparison between the value measured and the value from a measurement of a standard defining the unit. Such a standard we would call a Reference Material (RM) and as such could be a direct measure of the unit, sometimes called a 'Primary Reference Material'. A RM could also be calibrated against

this Primary RM. So, for example in another field of measurement, we could measure a time period with a clock. This is calibrated by a more accurate clock, which again may be calibrated against the atomic standard. Each calibration is a measurement which includes the uncertainty of the previous stages, up to and including the primary RM.

Again, by example, the mass of a material is measured by weighing against calibrated weights, which themselves are calibrated back through a chain of comparisons to the SI kilogram in Sèvres, Paris. Each step in the chain adds to the accumulated uncertainty of the measurement of the mass.

## Uncertainty

The present concept, as described in the GUM, accepts that a measurement process involves many different sources of uncertainty, which together can be combined to yield the combined uncertainty of the measurement. The uncertainty is a consequence of the measurement process; the measurement process therefore always results in a measured value and an uncertainty. The measured value is the best estimate of the measurand and the uncertainty expresses the range within which the measurand value is expected to be found.

The uncertainty of the measurement is calculated from the expression that defines the measurand. This can include constants, factors, measured values etc. The uncertainty of each component is combined by the standard rules of uncertainty propagation.

An approximative method, which has proven very useful, is also given in the Eurachem Guide /2/. This method lends itself to spreadsheet type calculations and is easy to implement. One advantage of using the spreadsheet approach is that the contribution of each uncertainty source can be readily expressed as percent of the total uncertainty. This makes it clear which sources of uncertainty have to be reduced in order to reduce the overall uncertainty.

The final uncertainty is then expressed as the '**standard combined uncertainty**'  $u_c$ . A coverage factor,  $k$ , is then applied, which is normally a value between 1 and 3 to yield the 'expanded uncertainty',  $U=k \cdot u_c$ . The Eurachem Guide recommends using a factor  $k=2$ . This has the advantage, for most cases, of being roughly equivalent to the previous '95% confidence limit'.

The big change, however, is that **all** uncertainty sources are included in the value of the final combined uncertainty, including uncertainties associated with fundamental constants, uncertainties which can be quantified in a straightforward manner, and also uncertainties which rely on the experience of the experimenter to quantify them.

This last point is not easily accepted by analysts who have learned that what cannot be quantified using statistical methods cannot be included in an uncertainty calculation. This new approach places more responsibility in the hands of the experimenter, who often knows his measurement apparatus very well and is in a good position to quantify uncertainties that cannot be obtained by repeated measurements.

Because the final estimate of uncertainty of a measured value expresses the range within which the analyst is confident the value of the measurand is to be found, we should always try to be realistic in the estimation of uncer-

tainties. It is very dangerous to attempt to reduce the value of uncertainties to make the end result appear better. It is also undesirable to have deliberate overstatements of uncertainties, which could lead to unnecessary and costly re-measurements.

Experience shows that most sources of uncertainty contribute very little to the overall uncertainty and that the final measurement uncertainty in practice is often defined by the uncertainty from only one or two sources.

Following the GUM, there are two distinct approaches to the evaluation of uncertainty, named simply 'Type A' and 'Type B' evaluations.

## Type A and type B evaluated uncertainties

Uncertainties evaluated from repeated observations are defined as 'Type A'. In other words, it will in most cases cover the term 'random errors'. This definition places the responsibility on the analyst to identify such uncertainty sources and to quantify them by well designed experiments. Most of these sources of uncertainty are clearly recognised and are relatively straightforward to deal with. The values for the uncertainty contributions from these sources are the standard deviations of the means in each case. However we must be cautious: if we suspect components present which we would not expect to be reduced by repeated measurements, i.e. cannot be treated by a type A evaluation, then we should separate out such components and evaluate them as type B.

Uncertainties evaluated by other means are called 'type B' evaluated uncertainties. The contributions to the combined uncertainty from type B evaluated uncertainties can be considerable, and are often the most difficult to quantify. The acceptance of the existence of type B evaluated uncertainty places the whole responsibility of the measurement back in the hands of the analyst. Type B evaluated uncertainties do not necessarily have a different origin to the type A: the difference between them is only the way the uncertainties are evaluated. Type A uncertainties are evaluated using repeated measurements and type B's evaluated using available knowledge. As an example, the uncertainty quoted from a certificate is a type B evaluation. The accepted uncertainty on a physical constant (for instance a half-life) is another. An important source of uncertainty, which most likely has to be evaluated as type B, arises from factors which are neutral as far as the end result is concerned, but which nevertheless contribute to the total uncertainty. As an example, when measuring isotopic ratios on a mass spectrometer with multiple collectors, the between-cup calibration factor may be assumed to be 1.0, but the factor has an uncertainty that contributes to the uncertainty of the final result and needs to be estimated.

## Uncertainty Budgets

The final uncertainty of a measurement value is presented in an **uncertainty budget**. This is a table of all contributions to the final uncertainty. In essence, an uncertainty budget is the proof that all sources of uncertainty have been taken into consideration, and it provides transparency for the user of the result. It has also the advantage that the relative contributions to the final uncertainty can be made clear. It is often instructive to see the relative impor-

tance of the various uncertainty contributions. It also makes clear that certain, perhaps most of the uncertainty contributions are in fact small. However, the fact that they are listed and have a value is a proof of the completeness of the analysis.

Uncertainty budget examples are given in Annexes 1, 2.

## Traceability

Traceability is a subject which is closely bound up with the concept of uncertainties. The accumulation of uncertainties, which arise from inclusion of all uncertainty sources, including the Reference Materials used, is essential for traceability to the SI. The result of a measurement is traceable to the SI if there is a clear link to a SI base unit and all measurement results in this link have stated uncertainties. So each measurement result is expressed with an uncertainty which comes from the measurement procedure itself and from stated references such as the certified value of a Reference Material.

The final uncertainty is therefore most likely to be smallest when the overall number of steps back to the SI is small and the uncertainty of the measurement itself is also small. These considerations arise from a consistent view of the measurement process which is the basis of the new model, the GUM approach. It can have considerable implications for Safeguards measurements in general, although this will not be discussed here.

## Methods of calculation

The basis of all calculations is the formula specifying the result in terms of the component parameters. This is a good place to make the equation as complete as possible, recognising that each parameter in the equation must have an uncertainty.

Take for example the simple case of a calibration solution made by dissolving a weighed amount of a substance and dissolving in a volume of solution:

$$\beta = 1000 \cdot m \cdot P / V$$

where  $\beta$  is the result (concentration expressed as weight dissolved material per volume solution),  $m$  the weight of the substance,  $P$  its specified purity and  $V$  the volume of the solution. The uncertainties of all the values entered into this equation are evaluated as type B. The uncertainty budget in detail is given in Annex 1. The relative contributions of the three terms in the equation are roughly equal in this case.

A second more complicated example is given with full details in Annex 2. The concentration of Pu in a sample solution is measured by mass-spectrometric isotope dilution (IDMS) using a  $^{242}\text{Pu}$  certified spike. The analysis of the individual terms contributing to the combined uncertainty of the sample concentration shows that the measurement of the mass-discrimination factor,  $K_{242/239}$  contributes the largest uncertainty (63.7%), followed by the uncertainty from the certified value of the spike (25.8%) and the measurement of the blend (7.0%). All other uncertainty contributions are 1% or less of the total  $u_c$ .

## Methods of calculation

Once we have the equation which defines the result in terms of the input parameters, we can calculate the uncer-

tainty of the result by assigning individual uncertainties to all terms in the equation and propagating these to the final result. The error propagation can be time-consuming and in some cases difficult to handle because of the complicated equations from deriving the partial derivatives.

An alternative method, mentioned previously, was given in the Eurachem document /2/. In this method, sometimes called the 'spreadsheet method', the result is calculated by varying the value of each input parameter in turn by one uncertainty. The percent differences are then added together quadratically to give the final uncertainty. The method has the advantage of being relatively straightforward to carry out and as a bonus shows the relative contribution of each parameter to the final uncertainty.

Thirdly, commercial programs are becoming available, which use a numerical differentiation method and one of these programs /3/ was used in calculating the examples in the Annexes.

Each of these methods can be used; each has advantages and disadvantages. Tests have shown that they are all capable of estimating the final uncertainty of a result.

## Consequences of GUM

The acceptance of the GUM is high, not only in the analytical chemistry field, but wider, as the implications of having a well-founded basis for chemical measurements is appreciated. As an example, accreditation (EUR-25000) for an analysis laboratory requires the consequent use of uncertainties as defined in the GUM.

However, the practical analyst will also appreciate the advantages of the new system.

- It allows an objective comparison of measurement results
- It provides greater openness and transparency of measured results

It requires that the analyst

- Understands his or her measurement process and can evaluate uncertainties to all contributions of the measurement process
- Always quotes results together with an uncertainty, stating the coverage factor
- Prepares an uncertainty budget as proof of the uncertainty calculations

## References

- /1/ Guide to the Expression of Uncertainties in Measurements, BIPM, ISO, 1993
- /2/ Quantifying Uncertainty in Analytical Measurements, Eurochem, ISBN 0-948926-08-2, 1995
- /3/ 'GUM Workbench', Danish Technological Institute, 1999 (<http://www.gum.dk>)

## Annex 1: Example of simple uncertainty budget - Preparation of a calibrated standard

A calibration standard is prepared from a highly purified material (reference material) with a concentration of about 1000 mg·L<sup>-1</sup>.

Model Equation:  
 $\beta = 1000 \cdot m \cdot P / V;$

**Symbol List:**

Symbol	Unit	Definition
$\beta$	mg·L <sup>-1</sup>	concentration of the calibration standard
m	mg	mass of the highly purified material
P		purity of the material
V	mL	volume of the solution (flask)

$\beta$ :  
 Result

m:  
 Type B normal distribution  
 Value: 248.0 mg  
 Expanded Uncertainty: 0.13 mg  
 Coverage Factor: 1  
 The value is the reading of the balance and the uncertainty was evaluated during the qualification of the balance.

P:  
 Type B rectangular distribution  
 Value: 0.999  
 Limits:  $\pm 0.001$   
 The purity was given by the producer of the material as 99.9% with a limit of 0.1%.

V:  
 Type B normal distribution  
 Value: 250 mL  
 Expanded Uncertainty: 0.13 mL  
 Coverage Factor: 1  
 The value is the nominal volume of the flask. The uncertainty was evaluated during its qualification.

**Uncertainty Budget:**

Quantity	Value	Standard Uncertainty	Degrees of Freedom	Sensitivity Coefficient	Uncertainty Contribution	Index
m	248.00 mg	0.13 mg	50	4.0	0.52 mg·L <sup>-1</sup>	31.3 %
P	0.99900	580·10 <sup>-6</sup>	$\infty$	990	0.57 mg·L <sup>-1</sup>	38.0 %
V	250.00 ml	0.13 mL	50	-4.0	-0.52 mg·L <sup>-1</sup>	30.8 %
$\beta$	991.01 mg·L <sup>-1</sup>	0.93 mg·L <sup>-1</sup>	260			

**Result:**

Quantity:  $\beta$   
 Value: 991.0 mg·L<sup>-1</sup>  
 Expanded Uncertainty:  $\pm 1.9$  mg·L<sup>-1</sup>  
 Coverage Factor: 2.0  
 Coverage: 95.45%

**Annex 2:**

Plutonium sample uncertainty budget for a single measurement

Model Equation:  
 $C_{239PuX} = C_y \cdot m_y / m_x \cdot (R_y - R_b \cdot K_{242/239Pu}) / ((K_{242/239Pu} \cdot R_b - R_{242/239PuX}) \cdot (1/R_y));$

{amount abundances in the sample}

$$f_{238\text{PuX}} = R_{238/239\text{PuX}}/\Sigma R_{\text{PuX}};$$

$$f_{239\text{PuX}} = 1/\Sigma R_{\text{PuX}};$$

$$f_{240\text{PuX}} = R_{240/239\text{PuX}}/\Sigma R_{\text{PuX}};$$

$$f_{241\text{PuX}} = R_{241/239\text{PuX}}/\Sigma R_{\text{PuX}};$$

$$f_{242\text{PuX}} = R_{242/239\text{PuX}}/\Sigma R_{\text{PuX}};$$

$$f_{244\text{PuX}} = R_{244/239\text{PuX}}/\Sigma R_{\text{PuX}};$$

$$\Sigma R_{\text{PuX}} = R_{238/239\text{PuX}} + 1 + R_{240/239\text{PuX}} + R_{241/239\text{PuX}} + R_{242/239\text{PuX}} + R_{244/239\text{PuX}};$$

{observed ratios corrected for mass-discrimination factors}

$$R_{238/239\text{PuX}} = K_{238/239\text{Pu}} \cdot R_{238/239\text{Puox}};$$

$$R_{240/239\text{PuX}} = K_{240/239\text{Pu}} \cdot R_{240/239\text{Puox}};$$

$$R_{241/239\text{PuX}} = K_{241/239\text{Pu}} \cdot R_{241/239\text{Puox}};$$

$$R_{242/239\text{PuX}} = K_{242/239\text{Pu}} \cdot R_{242/239\text{Puox}};$$

$$R_{244/239\text{PuX}} = K_{244/239\text{Pu}} \cdot R_{244/239\text{Puox}};$$

$$C_{\text{PuX}} = C_{239\text{PuX}}/f_{239\text{PuX}}.$$

**Symbol List:**

<b>Symbol</b>	<b>Unit</b>	<b>Definition</b>
$C_{239Pu}$	mol/g	amount content of $^{239}Pu$ in sample
$CP_{ux}$	mol/g	amount content of Pu in sample
$C_y$	mol/g	amount content of cPu in spike
$f_{238Pu}$		amount abundance of $^{238}Pu$ in Pu
$f_{239Pu}$		amount abundance of $^{239}Pu$ in Pu of sample
$f_{240Pu}$		amount abundance of $^{240}Pu$ in Pu of sample
$f_{241Pu}$		amount abundance of $^{241}Pu$ in Pu of sample
$f_{242Pu}$		amount abundance of $^{242}Pu$ in Pu of sample
$f_{244Pu}$		amount abundance of $^{244}Pu$ in Pu of sample
$K_{238/239Pu}$		correction factor for $R_{238/239Pu}$
$K_{240/239Pu}$		correction factor for $R_{240/239Pu}$
$K_{241/239Pu}$		correction factor for $R_{241/239Pu}$
$K_{242/239Pu}$		correction factor for $R_{242/239Pu}$
$K_{244/239Pu}$		correction factor for $R_{244/239Pu}$
$M_{238Pu}$	g/mol	atomic mass for $^{238}Pu$
$M_{239Pu}$	g/mol	atomic mass for $^{239}Pu$
$M_{240Pu}$	g/mol	atomic mass for $^{240}Pu$
$M_{241Pu}$	g/mol	atomic mass for $^{241}Pu$
$M_{242Pu}$	g/mol	atomic mass for $^{242}Pu$
$M_{244Pu}$	g/mol	atomic mass for $^{244}Pu$
$M_{P_{ux}}$	g/mol	molar mass of plutonium in sample
$m_x$	g	mass of sample in preparing blends
$m_y$	g	mass of spike in preparing blends
$R_{238/239Pu_{ox}}$		uncorrected amount ratio $R_{238/239Pu}$ in sample
$R_{238/239Pu}$		corrected amount ratio $R_{238/239Pu}$ in sample
$R_{240/239Pu_{ox}}$		uncorrected amount ratio $R_{240/239Pu}$ in sample
$R_{240/239Pu}$		corrected amount ratio $R_{240/239Pu}$ in sample
$R_{241/239Pu_{ox}}$		uncorrected amount ratio $R_{241/239Pu}$ in sample
$R_{241/239Pu}$		corrected amount ratio $R_{241/239Pu}$ in sample
$R_{242/239Pu_{ox}}$		uncorrected amount ratio $R_{242/239Pu}$ in sample
$R_{242/239Pu}$		corrected amount ratio $R_{242/239Pu}$ in sample
$R_{244/239Pu_{ox}}$		uncorrected amount ratio $R_{244/239Pu}$ in sample
$R_{244/239Pu}$		corrected amount ratio $R_{244/239Pu}$ in sample
$R_b$		measured ratio of an individual sample
$R_y$		certified ratio $R_{242/239Pu}$ in spike
$\Sigma R_{P_{ux}}$		sum of ratios for Pu in sample

## Uncertainty Budget:

Quantity	Value	Standard Uncertainty	Degrees of Freedom	Sensitivity Coefficient	Uncertainty Contribution	Index
$C_{239\text{Pu}}$	$1.5057 \cdot 10^{-3}$ mol/g	0.074 %				
$C_y$	$465.71 \cdot 10^{-6}$ mol/g	0.038 %	50	4.2	$740 \cdot 10^{-9}$ mol/g	25.8 %
$K_{238/239\text{Pu}}$	0.99994	0.020 %	50	$10 \cdot 10^{-6}$	$2.0 \cdot 10^{-9}$ mol/g	0.0 %
$K_{240/239\text{Pu}}$	1.00006	0.020 %	50	$360 \cdot 10^{-6}$	$73 \cdot 10^{-9}$ mol/g	0.2 %
$K_{241/239\text{Pu}}$	1.00012	0.040 %	50	$53 \cdot 10^{-6}$	$21 \cdot 10^{-9}$ mol/g	0.0 %
$K_{242/239\text{Pu}}$	1.00018	0.060 %	50	$-1.9 \cdot 10^{-3}$	$-1.2 \cdot 10^{-6}$ mol/g	63.7 %
$K_{244/239\text{Pu}}$	1.0003	0.10 %	50	0.0	0.0 mol/g	0.0 %
$M_{238\text{Pu}}$	238.0495525 g/mol	$920 \cdot 10^{-9}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{239\text{Pu}}$	239.0521556 g/mol	$920 \cdot 10^{-9}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{240\text{Pu}}$	240.0538065 g/mol	$870 \cdot 10^{-9}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{241\text{Pu}}$	241.0568444 g/mol	$870 \cdot 10^{-9}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{242\text{Pu}}$	242.0587359 g/mol	$870 \cdot 10^{-9}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{244\text{Pu}}$	244.0641970 g/mol	$2.0 \cdot 10^{-6}$ %	50	0.0	0.0 mol/g	0.0 %
$M_{\text{Pu}}$	239.34110 g/mol	$61 \cdot 10^{-6}$ %				
$m_x$	1.008500 g	$5.7 \cdot 10^{-3}$ %	$\infty$	$-2.0 \cdot 10^{-3}$	$-110 \cdot 10^{-9}$ mol/g	0.6 %
$m_y$	3.016000 g	$1.9 \cdot 10^{-3}$ %	$\infty$	$650 \cdot 10^{-6}$	$38 \cdot 10^{-9}$ mol/g	0.1 %
$R_{238/239\text{Puox}}$	$6.707 \cdot 10^{-3}$	1.0 %	50	$1.5 \cdot 10^{-3}$	$110 \cdot 10^{-9}$ mol/g	0.5 %
$R_{240/239\text{Puox}}$	0.241738	0.035 %	50	$1.5 \cdot 10^{-3}$	$130 \cdot 10^{-9}$ mol/g	0.8 %
$R_{241/239\text{Puox}}$	0.035332	0.18 %	50	$1.5 \cdot 10^{-3}$	$96 \cdot 10^{-9}$ mol/g	0.4 %
$R_{242/239\text{Puox}}$	0.023796	0.15 %	50	$3.6 \cdot 10^{-3}$	$130 \cdot 10^{-9}$ mol/g	0.8 %
$R_{244/239\text{Puox}}$	0.0	0.0	50	0.0	0.0 mol/g	0.0 %
Rb	0.94670	0.019 %	50	$-2.1 \cdot 10^{-3}$	$-380 \cdot 10^{-9}$ mol/g	7.0 %
Ry	451.9	0.72 %	50	$9.1 \cdot 10^{-9}$	$30 \cdot 10^{-9}$ mol/g	0.0 %
$\Sigma R_{\text{Pu}}$	1.30760	0.011 %				
$c_{\text{Pu}}$	$1.9688 \cdot 10^{-3}$ mol/g	0.074 %	100			

Result:

Quantity:  $c_{\text{Pu}}$ Value:  $1.9688 \cdot 10^{-3}$  mol/gExpanded Uncertainty:  $\pm 2.9 \cdot 10^{-6}$  mol/g

Coverage Factor: 2.0

Coverage: 95.45%

# ESARDA activities

## Results of the ESARDA "REALS" Prediction Benchmark Exercise

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### Abstract

Within the ESARDA Non Destructive Assay Working Group (NDA WG) a benchmark exercise was launched for the evaluation of different techniques for the prediction of the REALS count-rate. The different techniques make use of the Monte Carlo code MCNP to generate the necessary data for their interpretation models. Mainly two different interpretation techniques are used. The first type of techniques relies on a point-model, which is modified to include the space dependence of different model parameters.

The second kind of technique is based on the analog use of the Monte Carlo code MCNP. The results of these prediction techniques are compared with measurements performed in JRC-Ispra at PERLA. This article reports on the results of the different techniques used in the benchmark exercise. It also gives an idea about the performance of these REALS prediction techniques by means of a brief sensitivity study of the most relevant parameters in the MCNP input file.

### 1. Introduction

The measurement of the REALS count-rate via coincidence analysis is a commonly used technique in safeguards for the assay of plutonium bearing samples. To determine the amount of plutonium present in the sample, the measured REALS count-rate is often related to the unknown plutonium-mass by means of a calibration. Similarly, active interrogation techniques are used to measure of the fissile uranium mass. The calibration is generally determined by using calibration standards. To reduce the need for physical standards, prediction techniques based on Monte Carlo simulations can be applied. One of the very popular Monte Carlo simulation techniques is the MCNP code [1].

The MCNP code has been benchmarked on many occasions for both applications in reactor physics as in safeguards and nuclear waste management. Three years ago the ESARDA NDA Working Group launched an MCNP REALS Prediction Benchmark Exercise to confront diffe-

rent approaches to REALS evaluation with each other, which were all based on the Monte Carlo simulation code MCNP. This article will summarise the main characteristics of the experimental set-up and the different configurations considered in the exercise. Also the key-points of the different techniques used in the exercise will be highlighted and the results of the different methods discussed.

### 2 Measurement set-up

For the Benchmark Exercise the following reference geometry is considered: a PERLA PWR UO<sub>2</sub> fuel assembly mounted inside the cavity of a neutron coincidence collar [2]. The assembly together with the neutron coincidence collar was placed in the PERLA copy of the Unattended Measurement System (PUMS) [2]. On the same PUMS structure, four groups of background measurement chains are mounted in order to monitor during the measurements the fluctuations in the neutron background.

The design drawings together with additional information related to the fuel assembly and the neutron coincidence collar were used to prepare the input files for the MCNP4a code. The input was then modified by each participant in order to enable the extraction of the necessary data needed in the algorithm developed for the REALS prediction. The development of a simulation model must be based on the understanding of the working of the Shift Register which is used during the measurements to process the incoming pulses and convert this data to REALS (R). The scope of the exercise is to extract from the simulation code MCNP the information necessary to estimate the parameters R. Then the measurements results can be compared with the simulation results.

### 3 Configuration and settings

For the benchmark exercise a total of six different configurations were defined grouped in two groups of three.

The difference between the two groups is the presence of cadmium liners in the second group of configurations (indicated with a letter a at the end of the configuration name), where they are absent in the first group. A description of the different configurations is given in Table 1. A more complete description was distributed amongst the participants [2] along with the MCNP files.

Configuration	Absorber	Poison	Cadmium-liner
ESARDA1			
ESARDA1a			X
ESARDA2	X		
ESARDA2a	X		X
ESARDA3		X	
ESARDA3a		X	X

**Table 1:** Characteristics of the different configurations used in the benchmark exercise

For every configuration a measurement of thirty minutes is performed using a pre-delay of 4.5  $\mu$ s, a window of 64 $\mu$ s and a long delay of 1024 $\mu$ s between windows.

For every configuration, an active and passive measurement is performed. For the active measurement, an Am-Li interrogation source with a source strength of 8.7 10<sup>4</sup> n/s is used [3]. By subtracting the passive REALS count-rate from the active REALS count-rate, the net active REALS count-rate is obtained. This net active REALS count-rate can then be compared with the simulation results predicted by the proposed REALS interpretation techniques.

## 4 Proposed REALS interpretation techniques

Six different participants started in the benchmark exercise: Euratom (EC), JRC-Ispra (EC), Harwell Instruments Ltd (UK), SCK-CEN (BE), BNFL (UK), University of Rome (IT). During the exercise, Euratom and University of Rome withdrew from the project and JRC-Ispra changed their method. In the following, the main principles of the four remaining proposed techniques are given.

### 4.1 Generalised autocorrelation technique (SCK-CEN) [AC technique]

Neutron coincidence counting techniques for the determination of the REALS count-rate R are commonly based on the following form of Böhnel's expression based on a single point model:

$$R = \frac{N_{SF} \varepsilon^2 M_2(p)}{2}$$

where  $M_2(p)$  stands for Böhnel's generalised second moment. In the case of this benchmark exercise this expression cannot immediately be implemented to predict the REALS due to several reasons which are explained below:

- The detection efficiency  $\varepsilon$  and the induced fission probability  $p$  will strongly vary as a function of the source loca-

tion. Therefore different zones in the geometry will have to be identified to minimise the bias due to the use of a global value for the efficiency and multiplication.

- Different source terms have to be considered : The number of induced fissions produced by the interrogating Am-Li source  $F_{Am-Li}$  can be evaluated as the product of the Am-Li neutron source strength  $S_{Am-Li}$  and the probability  $k^{IF}$  for a source neutron to induce a fission in the fuel assembly:

$$F_{Am-Li} = k^{IF} \cdot S_{Am-Li}$$

The total number of induced fissions in all fission chains can be expressed by:

$$F_{IF} = p \frac{\nu_1 F_{Am-Li}}{1 - p\nu_1}$$

where  $\nu_1$  is the first moment of the secondary induced fission prompt neutron multiplicity distribution.

- Böhnel's expressions, accounting for multiplication in the sample, are only valid for 'instantaneous' multiplication. Hence Böhnel's expressions can only be used to describe the fast multiplication in the sample, whereas thermal multiplication is not adequately modelled.

Since Böhnel's description of the multiplication process cannot be used, we have chosen for an alternative approach commonly applied in reactor correlation techniques [4]. Instead of separating the detection and multiplication process, which relies on the validity of Böhnel's expressions, we tally the detection probability per starting neutron including multiplication resulting in a detection probability function  $D_i(t_0, p)$ .

The probability to detect a first neutron of a group of  $\nu$  neutrons emitted by a fission source  $i$  at  $t_0$  equates to:

$$\nu D_i(t_0, p) dt_0.$$

The probability to detect a second neutron of this group of  $\nu$  neutrons emitted by the fission source  $i$  at  $t_0 + t$  equates to:

$$(\nu - 1) D_i(t_0 + t, p) dt_0.$$

The REALS count-rate R can then be calculated as the sum over all the different source terms as specified above and over the different multiplicities, which yields:

$$R = \sum_i F_i \sum_\nu P_i(\nu) \int_0^\infty \int_p^{p+W} \nu (\nu - 1) D_i(t_0 + t, p) dt_0 dt$$

After rearrangement of the different summations and defining the autocorrelation function

$$C_i(t, \nu) \int_0^\infty D_i(t_0 - p) D_i(t_0 + t, p) dt_0$$

we finally obtain:

$$R = \sum_i F_i \nu_2^i \int_p^{p+W} C_i(t, p) dt$$

where  $\nu_2^i$  denotes the second moment of the neutron multiplicity distribution of reaction  $i$ .

## 4.2 Böhnel's equation modified for space dependence (Harwell Instruments) [BE technique]

In this approach, the prediction of the net active REALS count-rate  $R$  is based on Böhnel's expression which was modified for space dependence defining the 264 fuel pins (i) each split into 15 axial segments (j):

$$R = \sum_{i=1}^{264} \sum_{j=1}^{15} [k_{2,j}^{IF} S_{Am-Li} (\epsilon M)_{i,j}^2] f \frac{v_2^A}{2} \left( 1 + (M-1) \frac{v_1^A}{v_2^A} \frac{v_2}{v_1-1} \right)$$

where  $f$  is the coincidence gate utilisation factor,  
 $v_1^A$  is the first moment of the primary induced fission prompt neutron multiplicity distribution,  
 $v_2^A$  is the second factorial moment of the primary induced fission prompt neutron multiplicity distribution.  
 $M$  is the leakage multiplication factor.

Two separate MCNP runs are required to generate the necessary data:

1. Fission rate run: calculation of the primary induced fission probability  $k_{i,j}^{IF}$  in the assembly per Am-Li source neutron from  $^{235}\text{U}$  and  $^{238}\text{U}$  separately.
2. Efficiency run: calculation of the product ( $\epsilon M$ ) of the detection efficiency and the self-multiplication factor. This run is intended to calculate the detection efficiency for induced fissions.  
 Since the majority of these will be in  $^{235}\text{U}$ , a thermal induced  $^{235}\text{U}$  prompt fission neutrons spectrum is specified.

The fission rate run needs to be carried out without tracking any induced fission neutrons (using the MCNP "NONU" option). This is because the secondary induced fission neutrons are already tracked in the efficiency run.

Both the detection efficiency and the induced fission rate are likely to exhibit large axial variations. For this reason, the fuel rods are conceptually split into a number of axial segments for the purpose of the present modelling. The induced fission rate and the detection efficiency may then be assumed as approximately constant throughout each segment. The fission rate and detection efficiency can then be calculated for each segment (15) and for each pin position (264).

In order to determine the co-incidence gate utilisation factor  $f$  another series of MCNP calculations were made. The ( $\epsilon M$ ) result, averaged over all pin positions at mid-height, was split into a number of time bins representative of the experimental parameters. The coincidence gate utilisation factor  $f$  is equal to the ratio of the REALS response (4.5 - 68.5  $\mu\text{s}$ ) to the total time integrated REALS response [5]. It was found that  $f$  did not show a significant axial variation, so it is justified to take the mid-height value. However,  $f$  was found to be different for the no cadmium and cadmium configurations.

Results for configurations 1 and 1a yield values of  $f=0.5412 (\pm 0.0016)$  and  $f=0.6323 (\pm 0.0019)$  respectively. These values were adopted for the other configurations as well.

## 4.3 Analog Monte Carlo pulse train generation (BNFL) [AM technique]

The computer code MCNP was used to produce a pulse train arising from (n,p) reactions within the  $^3\text{He}$ -detectors by performing the following steps:

1. Implicit capture was turned off using the PHYS card;
2. If during a history an (n,p) reaction occurred in any of the detectors, the history number and the time between source particle creation and interaction were logged using a PTRAC card. A particular history may have more than one interaction logged;
3. The resulting file of selected histories was then processed as follows. The time spacing between source particles was determined randomly using a Poisson distribution (reducing the emission rate of the source by the ratio of histories with (n,p) reactions over the total number of histories). The times to the (n,p) reactions within the history are added to the cumulative running time to produce a pulse train.

The resulting pulse train can then be processed using a software version of the shift register. This method has the advantage of not requiring modifications to the geometry or source specification in the model. However, it does require many histories to be generated and the processing of large files.

The standard version of the code MCNP does not model the fission process perfectly. The number of neutrons created in a fission event is determined from a tabulation of the mean number of neutrons as a function of energy. The integer above and below is then chosen randomly in the correct proportion. Thus events with more or less neutrons than these two numbers are not generated. Hence, the sampling of the second moment of the neutron multiplicity distribution will not be performed correctly.

## 4.4 Analog Monte Carlo pulse train generation with modified MCNP (JRC-Ispra) [MAM technique]

To overcome the problems associated with an inaccurate sampling of the neutron multiplicity distribution in MCNP, JRC inserted an additional module into the MCNP code. Every time a fission occurs, this module is called from MCNP and then the entire neutron multiplicity distribution for the specific nuclide is sampled.

This approach assures that all the relevant neutron transport processes including fission are modelled accurately and hence an accurate prediction of the actual neutron pulse train is obtained. The neutron pulse train is then passed through a software implementation of a Shift Register to obtain the simulated REALS.

## 5 Results of the Benchmark Exercise

The different configurations identified in paragraph 3 were measured both in passive and active mode. In order to obtain the REALS count-rate induced by the interrogation Am-Li source, the passive count-rates were subtracted from the (active + passive) ones. This way any inaccuracy in the modelling of the passive correlated signal is eliminated. These active REALS count-rates corrected for the passive background can then be compared with the simulation

	Measurement	BE technique (Harwell Instruments)		AC Technique (SCK-Mol)		AM Technique (BNFL)		MAM Technique (JRC)	
		REALS	REALS	Dev (%)	REALS	Dev (%)	REALS	Dev (%)	REALS
ESARDA1	472	495	+5	396	-16	394	-16	486	+3
ESARAD1a	34	34.9	+3	27.7	-18	22.9	-33	34.6	+2
ESARDA2	303	309	+2	299	-1	259	-15	321	+6
ESARDA2a	27	27.0	+0	25.0	-7	20.8	-23	27.2	+1
ESARDA3	385	398	+3	351	-9	329	-15	405	+5
ESARDA3a	33	32.8	-1	27.1	-18	25.9	-22	32.1	-3
Average Deviation			+2.0		-11.5		-21		+2.3

**Table 2:** Measured and simulated REALS count-rates for the considered configurations obtained with the different interpretation techniques.

results obtained with the different interpretation techniques. Table 2 lists the REALS count-rate ( $s^{-1}$ ) and the relative deviation (%) to the measured value of the different techniques for the configurations considered.

Table 2 shows that for method BE and MAM an average overall deviation of about 2% exists and individual deviations of 5% are reported. These results prove that the REALS count-rate can adequately be predicted in an absolute sense from first principles, by means of an interpretation technique making use of MCNP for the generation of the necessary data.

The results given by the AC-technique have larger reported deviations than BE and MAM. Since BE and AC both rely on a model which essentially consists of including the space-dependence into Böhnel's point-model, both models can produce similar results. The discrepancy in the reported values results mainly from a different detail in axial zone partitioning. For the simulations with BE, 15 different axial zones were considered, whereas for AC, only three

axial zones were identified. Because of the strong axial profile of the REALS count-rate, the use of large zones will lead to biased results (AC). These results also point out the sensitivity of the simulation results to the choice of partitioning the entire fuel assembly into smaller zones.

The results obtained with the AM-technique show the largest deviations from the experimental values. Since the AM-technique and the MAM-technique are the same apart from the fact that MAM includes an additional subroutine to sample the neutron multiplicity distribution, the observed bias with AM can be attributed to this fact.

From these results, one might conclude that a prediction of the actual REALS count-rate can be performed with an accuracy of about 5%. To investigate whether it is realistic to expect an accuracy of 5%, a brief sensitivity study with regard to the most relevant parameters in the MCNP input file was performed.

The results of this sensitivity study performed using the MAM approach only are described in Table 3.

	Measurement	ORIGINAL		ASSEMBLY		MODIFIED		SPECTRUM	
		REALS	Dev	REALS	Dev	REALS	Dev	REALS	Dev
ESARDA1	472	486	+3	482	+2	482	+2	489	+4
ESARAD1a	34	34.6	+2	37.6	+11	31.2	-8	33.2	-2
ESARDA2	303	321	+6	325	+7	330	+9	331	+9
ESARDA2a	27	27.2	+1	27.9	+3	25.2	-7	27.2	+1
ESARDA3	385	405	+5	406	+5	401	+4	407	+6
ESARDA3a	33	32.1	-3	31.6	-4	30.2	-8	31.2	-5
Average Deviation			+2.4		+4.1		-1.3		+2.0

**Table 3:** Simulated REALS count-rates for the different configurations and different modifications to the MCNP input file.

The MCNP input file labelled ORIGINAL is the one distributed to all of the participants for their estimation of the REALS count-rate. The ASSEMBLY input file is substantially identical to ORIGINAL. The main difference is constituted by a new way of describing the fuel assembly.

The MODIFIED input file contains changes in polyethylene density and  $^3\text{He}$ -pressure. To analyse the influence of the spectrum of the Am-Li source, in the SPECTRUM input file a new spectrum (an evaluation based on measurements by Tagziria) replaces the Geiger and van der Zwan spectrum in the MODIFIED input file (no other changes are introduced).

Table 3 shows that individual changes of more than 10% are possible from model to model. Although the average deviation for the different modified input files remains less than 5%, it is more realistic to put forward a value of 10% as a performance value for this approach.

This analysis emphasises that in order to have confidence in calculated response functions one must take great care to ensure that the physical model of the experimental set up is faithful to reality.

The benchmark exercise also illustrates that both an analog Monte Carlo technique and one based on a modified form of Böhnel's point-model are capable of obtaining the desired accuracy. From a methodological point of view, an analog Monte Carlo technique is preferred since it models the different physical and instrumental processes as they occur during a measurement.

When using a technique based on Böhnel's point-model, a sufficient discretisation in space is required to obtain accurate results. Since it is not known in advance which degree of discretisation is needed, it remains difficult to determine this optimum level. Therefore, if computation time is not a limiting factor, analog Monte Carlo which includes the sampling of the neutron multiplicity distributions is the preferred technique with a 10% performance value.

## 6 Conclusion

Within the ESARDA NDA Working Group a benchmark exercise was launched for the evaluation of different techniques for the prediction of the REALS count-rate. The dif-

ferent techniques make use of the Monte Carlo code MCNP to generate the necessary data for their interpretation models. At the end four different laboratories participated to the benchmark exercise.

Two main types of interpretation techniques were used. The first type relies on a point-model, which is modified to include the space dependence of different model parameters. The second kind is based on analog use of the Monte Carlo code MCNP.

The results of the benchmark exercise prove that both an analog Monte Carlo technique (including neutron multiplicity sampling) and one based on a modified form of Böhnel's point-model are capable of obtaining results with an accuracy less than 5%.

A sensitivity study with regard to the most relevant parameters in the MCNP input file has shown that a performance value of 10% for these REALS prediction techniques is more realistic.

From a methodological point of view, an analog Monte Carlo technique is preferred since it models the different physical and instrumental processes as they occur during a measurement. The techniques based on a point-model modified for space-dependence need a sufficient level of space discretisation to obtain accurate results and this takes a combination of experience and experimentation to optimise. However this process can also be valuable for the modeller because it demands that the key aspects of the problem are properly considered.

## 7 References

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# Analysis of the outcome of the ESARDA/INMM Workshops on “Science and Modern Technology for Safeguards”

By the ESARDA Scientific Council & Co-ordination Board

This paper has been presented at the IAEA Symposium on International Safeguards, Vienna, 29 October-02 November 2001.

## Executive Summary

Nuclear safeguards (also strengthened and integrated safeguards) strongly rely upon Science and Technology, in order to ensure effectiveness of application, transparency and objectivity of conclusions.

The new challenges posed by integrated safeguards: ensuring correctness and completeness without cost increase, may require that new techniques are employed, existing techniques are modified, to cope with the new requirements. The developments of the nuclear fuel cycle towards large and automated facilities will also, to a certain extent, require the application of new technologies.

Conscious of these new scenarios, ESARDA decided to have a thorough overview of Science and Technology aspects, looking in particular to the possibility of identifying new techniques not yet applied in Safeguards, which could help in increasing efficiency/effectiveness, at the same cost.

To that purpose ESARDA decided to organise, together with the INMM, a series of Workshops on “Science and Modern Technology for Safeguards”, whose main aim was *“to inform the safeguards community about selected science and advanced technologies that are currently available or that will become available in the next few years and that could be used to support needed advances in international safeguards”* and to *“stimulate interchange amongst experts in the various technologies and in safeguards”*.

Three Workshops have been held, the first in Arona (I), in October 1996 [1]; then at Albuquerque (NM-USA), September 1998 [2] and the third one in Tokyo, on November 2000 [3]. With similar purposes, i.e. in order to identify possible similarities and synergies with technologies employed in other verification conventions, ESARDA dedicated the 1998 “Annual Meeting” to that topic, holding at Helsinki (Fin), a Seminar on “Modern Verification Regimes: Similarities, Synergies and Challenges” (May 1998) [4], inviting representatives of other nuclear and non-nuclear regimes (CTBT, Chemical and Biological Conventions) to gather together and confront ideas and exchange information with nuclear safeguards specialists.

ESARDA/INMM Workshops were attended typically by approximately 100 participants (115 in Arona), presenting and discussing about 40 papers in each workshop.

The Helsinki Seminar was attended by 139 participants, with 67 papers presented. The papers covered a

wide range of scientific/technical and non-technical aspects, but almost all were dealing with innovative or future applications for nuclear safeguards and other verification regimes.

The ESARDA Co-ordinators decided then to perform an analysis of the outcome of the ESARDA/INMM Workshops and of the Helsinki Seminar, with the purpose of establishing whether the aims of ESARDA in deciding the workshops were achieved, analysing the status of the development of those techniques and methods presented, which may have an application for Safeguards and suggesting future directions for the ESARDA activities and for Safeguards R&D. Along with the analysis, Co-ordinators collected and successively distributed recommendations and suggestions to the ESARDA WGs, when some needs emerged which could be fulfilled by the technical and scientific ESARDA structure itself.

Following the main format given to the Workshops, the Co-ordinators’ analysis has been structured along the following areas:

1. “hard” sciences (instruments, C&S, Sensors, etc);
2. “soft” sciences (data and information treatment, knowledge building);
3. non technical (or socio-political) aspects;
4. role of the Regional Systems of Accountancy and Control (RSAC) and of the State Systems of Accountancy and Control (SSAC).

Within these areas, the Co-ordinators examined whether any technique emerged that could be applicable in safeguards and which techniques deserve attention from ESARDA for further analysis in working groups or in topical Seminars, Workshops and Symposia

The main conclusions reached by Co-ordinators were:

In **Hard Sciences**, classical analytical (DA, NDA) techniques will continue to be the basis of the NMA “leg” of Integrated Safeguards. In sensitive areas, reprocessing, spent fuel assay, waste and spent fuel repositories, further improvements in performances are needed. Developments in these traditional areas are largely reported in regular Symposia (INMM, ESARDA, IAEA). Therefore Co-ordinators do not suggest special seminars or workshops for them.

• **HPTA (High Performance Trace Analysis) / ES (Environmental Sampling)**

R&D and application studies are requested for new areas for strengthened and Integrated Safeguards: wide-area and site specific HPTA/ES are areas where still R&D is needed. Co-ordinators feel that is now time to evaluate the performances of those techniques (bulk and particle analysis, for inside facility and WAES). Co-ordinators recommend as well a cost evaluation of the techniques with respect to their application in safeguards.

Co-ordinators point out that, in the analysis, also the burden onto the operator should be taken into account, since previous experiences show that this aspect may be not at all irrelevant.

DA and NDA ESARDA Working Groups should have the above issues on their agenda.

Co-ordinators also recommend that actions are taken to promote the development of the ES analytical capability in additional laboratories in Europe and to attempt to evaluate the worth of ES analysis, based on five years of implementation experience within facilities, embracing the limitations and successes of the analytical technique, its cost and its overall contribution to safeguards effectiveness.

• **Satellite Imagery**

The use of satellite imagery still needs technical developments, which are expected from commercial companies. New commercial satellites are being launched with higher resolution power and the prices for imageries is going down, which makes this technique more available for safeguards application. However the safeguards community should outline criteria for interpretation and application, should define how to integrate the results given by the technique with other safeguards measures and information. Also costs should then be considered (for being considered within Integrated Safeguards, Containment and Surveillance and Back-end of the Fuel Cycle Working Groups).

• **Unattended Monitoring/Remote Data Transmission** will be much more used in the future Safeguards. Here also cost/benefit analysis with respect to the application is required. In this context Co-ordinators recommend a thorough assessment of the reliability of C/S devices, since experience suggests that the weakest "ring" of an unattended system might be the local C/S device.

Encryption/authentication of data is required.

Concerning **Soft Sciences** Co-ordinators noted that the volume of data collected for future integrated safeguards will be very huge and that the data often might vary greatly in reliability and potential relevance; there is not only the need of collecting, storing and interpreting large heterogeneous data sets from diverse sources, but as well to deal with various kinds of uncertain information, like contradictory, incomplete, fragmentary, vague, biased (purposely or not) or deficient pieces of information. It is necessary to make this information contribute to better knowledge and to facilitate decision making.

Open source information is a kind of information that the safeguards technical world has never dealt with; we

may need specialists and special sessions for discussing these completely new aspects. Co-ordinators suggest also that fuzzy techniques could be of great help in the area of non technical (or socio-political) aspects, to approach non-quantifiable aspects with fuzzy probability numbers. Co-ordinators acknowledge that fuzzy techniques are very much developed and applied in several areas: one aspect of the difficulties encountered in applying them to Safeguards, may be due to a lack of specifications and formulation of the problem by R&D and by customers. Co-ordinators suggest that customers together with R&D developers, select a few specific examples, formulate questions, specify the problem, develop solutions and show the practical advantages of the fuzzy techniques. To this challenging goal, ESARDA has to provide reflection and actions.

**Non technical** (or socio-political) aspects are dominating the scene for new safeguards: Co-ordinators recommend providing opportunity for discussing those aspects in special workshops and sessions in ESARDA Symposia. Technical aspects, such as innovative criteria, should be discussed in the ESARDA Integrated Safeguards WG.

ESARDA should make efforts to diffuse to the wide public the issues that are discussed within the Organisation and in the Seminars and Symposia: specialised and general press should always be informed and/or be invited to large open Safeguards meetings, as for instance, the Symposia.

**Final consideration:** Co-ordinators believe that the series of workshops has provided a very good insight into possible, available scientific techniques. Arona, Albuquerque and Tokyo Workshops were very important to set the scenario of the techniques for the future safeguards, within the above framework.

The Helsinki Seminar allowed a good evaluation of the similarities and possible synergies with other verification conventions.

The formula chosen of having a broad spectrum of technological areas explored, without substantial limitations and having as well non technical areas (socio-political, Regional Systems of Accountancy and Control) discussed, has shown to be very fruitful, since several interesting new and innovative aspects came up in both (technical and non technical) domains. The Workshops in fact gave a very positive contribution to evidence and discuss non technical (socio-political) aspects and to underline the essential contribution the Regional System of Accountancy and Control give to non-proliferation.

After Tokyo more targeted workshops/seminars should follow, making use of the outcome of the wide-scope workshops so far held. Therefore for the future thematic events, seminars or special sessions in Symposia should be held, with the aim of discussing new technologies and approaches, their adaptation or improvement for (new) safeguards applications. Co-ordinators recommend that new technologies entering the field of Safeguards, should be evaluated with respect to their performances in Scientific/Technical terms, but also analysing their costs with respect to their application in Nuclear Safeguards and Non Proliferation. One excellent example of the above strategy of focussed

workshops is the recent ESARDA Dresden Seminar [5], that came just at the right time and that extensively addressed future Integrated Safeguards: the outcome was judged to be very positive.

#### **Recommended Topics for Future Special Sessions.**

A list of possible topics, derived from the analysis and the outcome of the Workshop on "Science and Modern Technology for Safeguards", follows. It is understood, as said in the previous chapter, that "classical" NMAC measures and techniques will continue to be treated in regular meetings and Symposia.

Therefore the following recommendations regard only those aspects which emerged as relatively or completely new areas and deserve special sessions or particular attention in this phase of rapid changes Safeguards is experiencing.

1. Integrated Safeguards and Application of the Additional Protocol. This topic is still the most urgent and important; in particular aspects of application of the Additional Protocol and Integrated Safeguards are to be addressed urgently.

Several of the following topics are linked with, if not contained within, the more general area of Integrated Safeguards:

2. Information Evaluation and Knowledge Building. This is probably the most challenging technical area, since future safeguards will be much more "information oriented".
3. Techniques and Application of Environmental Sampling/HPTA Evaluation of achievements/capabilities/costs.
4. Use and cost of Satellite Imagery
5. Use of Unattended/Remote Monitoring
6. General Economical Aspects of IS
7. Non-technical (Socio-Political) aspects and use of Regional Systems of Accountancy and Control. These areas deserve further and continuous follow up, being th non-technical aspects which will drive future safeguards.

#### **Note: Definitions**

**Nuclear Material Accountancy Safeguards** refers to the INFCIRC 153 safeguards measures implemented for detection of a diversion of declared nuclear material. They include the safeguards strengthening measures, which can be implemented under the existing legal authority of a Safeguards Agreement.

**The Additional Protocol** measures are the safeguards measures to detect undeclared nuclear material and activities in a state as provided by the Additional Protocol (INFCIRC 540 (corrected)).

**Strengthened Safeguards** refers to measures provided by nuclear material accountancy safeguards to detect a diversion of declared nuclear material and, in addition, the measures provided by the additional protocol (INFCIRC 540 (corrected)) to detect undeclared nuclear material and activities in a State.

**Integrated Safeguards** is defined as the optimum combination of all safeguards measures available to the IAEA under comprehensive safeguards agreements and Additional Protocol, which achieves the maximum effectiveness and efficiency within the available resources in fulfilling the Agency's right and obligation in paragraph 2 of INFCIRC 153 (corrected).

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# THE ESARDA WORKING GROUP FOR LOW ENRICHED URANIUM CONVERSION AND FABRICATION PLANT

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## I INTRODUCTION

It is generally felt that safeguards of Low Enriched Uranium (LEU) facilities has reached a high level of quality and reliability because of improvements to the verification schemes and the surveillance and measurement techniques. In particular, the ESARDA parties have recognized that the LEU Working Group has successfully completed its tasks and achieved the objectives laid down in its Terms of Reference (TOR). Accordingly, it was decided to discontinue the Working Group.

The aim of this document is to give a survey of the major achievements of the Working Group since its constitution in 1978.

## II CONSTITUTION - TERMS OF REFERENCE

The ESARDA LEU WG is a plant specific working group and was formed to provide an European forum where representatives from plant operators, research centres and safeguards inspectorate could meet to discuss the problems of safeguarding LEU conversion and fabrication plants and to propose acceptable solutions to these problems. Since the beginning, the work of the group has taken into account the systems already existing in the plants for the development of safeguards systems; this approach has always been followed until now, and was possible because since its constitution, the main European organizations have always been represented in the group.

The Terms of Reference have been modified and adapted in function of new needs and changes in the various facilities and also in function of the evolution of the safeguards approaches. The last version approved in 1997 by the ESARDA Executive Committee is as follows.

## TERMS OF REFERENCE OF THE ESARDA WORKING GROUP FOR LOW ENRICHED URANIUM CONVERSION AND FUEL FABRICATION PLANTS

### Objective

To provide the Safeguards Community with expert advice on methods, procedures and to exchange and demonstrate experience gained in nuclear material management and application of Safeguards in LEU facilities by the members of the working group.

### Tasks

As a general task, the Group promotes activities for development of current Nuclear Material Management and application of Safeguards taking into account the requirements of both plant operators and safeguards authorities.

In particular, the Group will:

1. *Promote the exchange of information and experience between Facility Operators and Safeguards Authorities*
2. *Maintain a list of methods and procedures suitable for accountancy and verification purposes and recommend the implementation of new ones*
3. *Recommend the participation of the members to the intercomparison exercises for nuclear material measurement techniques, for example REIMEP, scale system evaluation etc....*
4. *Promote the use of common and correct terms for the definition of materials and methods*
5. *Maintain a direct channel of information with Safeguards Authorities in order to be informed on the evolution in general terms of the LEU plants safeguards approach*
6. *Identify the issues, which are of safeguards relevance in the LEU plants and assess the different safeguards approaches particularly in terms of their impact on the facility operations*
7. *Identify and co-operate with the appropriate ESARDA Working Groups or Forum to address the above issues.*

As derived from the TORs, issues always included in the agenda of the WG meetings can be grouped under the following topics:

- System Studies
- Intercomparison: Destructive Analysis ( DA ) and Non Destructive Analysis (NDA )
- NDA measurements techniques Furthermore, "experience gained during recent PIVs and perspectives" was a basic issue and always included in the agenda of the WG meetings.

### III MEETINGS AND CONVENORS

Meetings were organised at regular intervals to evaluate actions going on and to decide upon new actions. An historical survey is given in the following table.

ESARDA LEU Meetings.

1. JRC-Ispra	1977
2. DCS-Luxembourg	June 27, 1978
3. BNFL-Springfields	September 26-27, 1978
4. JRC-Ispra	January 25-26, 1979
5. CEN-FBFC Mol	June 12-13, 1979
6. JRC-Ispra	October 8-9, 1979
7. RBU-Hanau	March 6-7, 1980
8. BNFL-Springfields	September 27-28-29, 1982
9. JRC-Ispra	June 7-8, 1983
10. FN-Bosco Marengo	October 25-26, 1983
11. RBU-Hanau	June 19-20, 1984
12. FBFC-BCMND-Dessel	January 23-24, 1986
13. RNL-Copenhagen *	May 13-14-15, 1986
14. ENUSA-Salamanca	September 8-8, 1987
15. KFK-Karlsruhe*	May 3-4, 1988
16. FBFC-Valence	September 28-29, 1989
17. Como*	May 15-16-17, 1990
18. ANF, Lingen	October 15-16, 1991
19. Salamanca *	May 7, 1992
20. BNFL-Springfields	September 13-14-15-16, 1993
21. Gent*	May 17-18-19, 1994
22. Aachen*	May 8, 1995
23. Bath*	May 15, 1996
24. Montpellier*	May 12, 1997
25. Helsinki *	May 11, 1998
26. Seviglia*	May 3, 1999

\* meetings held during the week of the ESARDA Annual Meetings.

The 1977 meeting was not a LEU WG meeting proper but was organized to examine the possibility of establishing such a group.

The first meeting proper of the LEU WG was in June 1978 and attracted the support of all the LEU fuel fabricators in the EEC.

The list of the Convenors is given in the following table.

1978	B. Love, Safeguards Directorate, CEC, Luxembourg
1979	M. Bresesti, JRC Ispra, Italy
1982	A.G. Hamlin, NMACT Harwell, UK
1983	R. Stewart, BNFL Springfields, UK
1983	P. Boermans, FBFC Dessel Belgium
1995	F. Mousty, JRC Ispra, Italy

### IV SYSTEM STUDIES

The aim of this activity was to contribute to the solution of the basic questions relative to safeguards of LEU facilities taking into account the point of view of both operators and inspectors. Different aspects together with adopted solutions are briefly described.

#### IV.1 Verification Schemes.

When considering safeguards approaches for LEU plants, one has to take into account the differences between the different facilities in size, complexity and operating procedures. Also the implementation of new techniques is a sensitive factor. However, it was possible to consider basic schemes applicable to whatever plant.

A first scheme is based on the verification of input and output and on the verification of the physical inventory once a year. In such a scheme, the knowledge of the details of the plant operations is not imperative.

The second verification scheme is based on an optimised utilisation of a Fast Response Computerised Accountancy System, together with statistically based sample verification. However, it requires a detailed knowledge of the plant operations, which leads to an intrusive system but allows a significant reduction of the detection time.

The third approach considers an extensive use of containment and surveillance techniques (C/S). This system seems difficult to use in existing plants and more attractive when it can be built in the conception of the facility. For example this was the case of the LEU facilities where new buildings were needed for the production of MOX assemblies.

## IV.2 Computerised Nuclear Material Management Systems

Significant and very fast progress in this field has been made over the last 20 years so that many WG meetings were dedicated to this topic. At the beginning near real time nuclear materials management systems were developed and later on were improved to become real time management systems. A system was developed and implemented at BNFL Springfields under a Joint Collaboration Contract between BNFL and CEC-JRC ISPRA. Another system was developed and implemented at FBFC DESSEL while a real time multi-user system, connected directly to the material areas was developed and used by RBU in HANAU.

## IV.3 Nuclear Material Statistical Accountancy Systems

Different packages were developed to be used as a tool for the analysis of materials balance information. Examples of such systems are:

- NUMIS (Nuclear Materials Information System) developed at BNFL with some assistance of CEC-JRC ISPRA
- NUMSAS (Nuclear Material Statistical Accountancy System) developed by JRC ISPRA and used on a routine basis by the EURATOM Safeguards Directorate in most of the plants of the E.C,
- MADES (Material Accountancy Data Evaluation System) which relies exclusively on the ICRs and PILs as reported under EURATOM regulation 3227/76,
- MACSSA is a fully interactive driven software for the analysis and the evaluation of material balance information of fabrication facilities
- SAMBA is a computer code for the statistical evaluation of MUF and LEMUF developed by CEC-JRC ISPRA during the years 90s.

## V INTERCOMPARISON EXERCISES

### V.1 The weighing scale exercise.

Different series of measurements went on for many years starting in 1981 after the calibration and certification of a set of 7 weights (from 1kg to 50 kg) prepared by CEC-JRC ISPRA. The results of each series of measurements were treated by using different procedures. First, an estimation method based on the use of constrained regression where residual analysis can be exploited in order to single out aberrant instruments. Later on, an iterative technique elaborating the information obtained in both the usual unconstrained procedure and the constrained one was preferred. Of course, the experimenters arranged the experimental design in a common way. The last exercise took place at the beginning of the 90s. These procedures proved to be useful because they enable the users to control the accuracy and the precision of their scale system without making use of expensive calibration procedures.

### V.2 Inter-laboratory experiments for the determination of uranium.

The LEU WG decided to carry out inter-laboratory analytical experiments to give the participating laboratories the opportunity to intercompare their capability and to evaluate any systematic uncertainty present in the determination of U in sintered UO<sub>2</sub> pellets or in solutions and to adapt, if necessary, the routine procedures. The CEC-JRC GEEL was generally in charge of the organisation of the different tests, included material preparation, certification, distribution and evaluation of the results. Later on, to avoid duplication of these tests, the WG members were requested to participate to the REIMEP exercises when LEU material was to be analysed. When organised by the WG, the measurement technique was imposed to the participants while REIMEP leaves the participant free to select the preferred measurement technique.

Usually, the exercises involved analytical laboratories from all European LEU fuel fabricators and from certain Research Centres.

The results were generally good and conform to international standards.

## VI NDA MEASUREMENTS

Fuel assemblies constitute the output of the LEU plant, as well as the input into the reactor, and their verification is important from the safeguards point of view. The neutron collar is a plant specific instrument that relies on the counting of coincidence neutrons originating in either spontaneous or induced fission processes allowing the determination of uranium (235 or 238) concentration in LEU fresh fuel assemblies and of plutonium in MOX fuel. Since many factors (traces of

234 or 236U, presence of absorbers or poison rods,...) can influence the quality of the measurements, many measurement campaigns were organised in different plants to improve reliability, precision and accuracy of the measurements.

In addition, the PHONID devices were extensively tested on request of the WG. The PHONID family constitutes a series of non-destructive assay instruments designed and built by CEC-JRC ISPRA. These instruments have been and are used for inspections of LEU facilities and are based on neutron measurements.

The last exercise based on PHONID device was organised in response to the publication of the IAEA International Target Values for Uncertainty Components in 1993 ( ). The NDA and LEU WGs agreed to carry out some practical measurements to assess the sampling error, due to the heterogeneity of the material, associated with the determination of the <sup>235</sup>U content of bulk uranic materials. The measurements were carried out by NDA techniques (PHONID and MGA-U) on bulk material followed by destructive analysis of samples of the same material. Measurements were performed at two LEU facilities, at FBFC Dessel and BNFL Springfields.

## VII LIST OF PUBLICATIONS ON BEHALF OF/IN COLLABORATION WITH THE WG

### 1. Publications in the ESARDA Bulletin

- A.G. Hamlin, NMACT, Harwell: Activity of the LEU WG, Bul. No. 3
- F. Brunelli, L. Olivi, CEC JRC ISPRA: Aberration diagnosis for a weighing scale system , Bul. No. 6
- R. Stewart, Convenor LEU WG, A.A. Musto: Achievements of the ESARDA LEU WG, Bul. No. 6
- P. Boermans, FBFC DESSEL, V. Verdingh, CBNM GEEL: Activity of the LEU WG, Bul. No.10
- P. Boermans, FBFC DESSEL, S. Guardini, EC, JRC ISPRA, R. Ingels, Belgonucléaire, DESSEL, B. Richter, Jülich: Report on the Joint Meeting of ESARDA WGs (LEU, MOX, C/S, NDA) on Unattended/Integrated Safeguards Systems, Bul. No. 26

### 2. Publications in the Proceedings of the Annual Symposia

- S. Saiger, RBU HANAU: Activity of ESARDA Working Group on Safeguarding Low Enriched Uranium Conversion/Fabrication Plants. Symp. No. 2, Edinburgh, 1980
- V. Verdingh et al. CBNM GEEL: Characterization of Uranium Dioxide Reference Material for In-plant Use, Symp. No. 5, Versailles, 1983
- R. Stewart BNFL SPRINGFIELDS, A.A. Musto BNFL RISLEY: A Survey of the Interaction between EURATOM, the European Research Centres and Plant Operators in the Field of LEU Conversion and Fuel Fabrication, Symp. No. 5, Versailles, 1983
- F. Brunelli, L. Olivi, P. Parisi, CEC-JRC ISPRA: Procedure of Estimation of the Accuracy and Precision of a Set of Weighing Scales, Symp. No. 6, Venice, 1984
- V. Verdingh CBNM GEEL, W.L.Zijp, ECN PETTEN: Repeatability and Reproducibility of Gravimetric Uranium Determinations in UO<sub>2</sub> Pellets, Symp. No. 6. Venice, 1984
- Symp. No 8, Copenhagen, 1986: Contribution of the WG
- F. Brunelli, L. Olivi, P. Parisi, CEC-JRC ISPRA: Conclusion on the Intercomparison Weighing Scale Exercise, Symp. No. 9, London, 1987
- P. Boermans, FBFC DESSEL, V. Verdingh, CEC JRC GEEL: Safeguards Characteristics of Fabrications in LEU Facilities. Symp. No 10, Karlsruhe, 1988
- P. Boermans et al: Achievements of the ESARDA LEU WG, Symp. No. 11, Luxembourg, 1989
- Symp. No 12, Como, 1990: Contribution of the WG
- V. Verdingh et al: Interlaboratory Comparison Exercise for the Determination of Uranium by Potentiometric Titration (First Phase): Symp. No. 13, Avignon, 1991
- F. Mousty, CEC-JRC ISPRA, P. Boermans, FBFC DESSEL: NDA on Wastes in LEU Facilities, Symp. No 14, Salamanca, 1992
- Symp. No. 16, Gent, 1994: Contribution of the WG
- P. Boermans et al: An Investigation of Sampling Error at Low Enriched Uranium Fabrication Facilities, Symp. No. 19, Montpellier, 1997

## VIII CONCLUSIONS

The ESARDA Working Group has, as was intended, provided an excellent forum for discussions between plant operators, research centres and Safeguards Authorities. Useful results were obtained that have contributed to the improvement of safeguards within the European Community. However, discontinuing the Group does not mean that LEU issues will not be discussed in ESARDA; in the future, any challenges that emerge applicable to LEU facilities will be addressed at other for a within ESARDA or by direct communication between interested parties.

# ESARDA Working Group for Non Destructive Assay Nuclear Techniques: Rôle and Developments

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## 1. Abstract

Nuclear Safeguards are experiencing substantial modifications, determined first by policy changes required after the Iraqi crisis, but also by the evolution of the fuel cycle towards large automated facilities, by the requirement of a more cost/effective use of human resources and by the advent of new available technologies.

Aware of the above situation and of the fact that Safeguards R&D must not follow, but, to a certain extent, drive the changes, ESARDA has launched, together with INMM, an action consisting in the organization of a series of topical meetings on "Science and Modern Technology for Safeguards" [1, 2, 3] and has dedicated the last two "Internal Meetings" to arguments that are oriented at individuating synergies with other verification regimes (Helsinki, 1997) [4] and look into the requirements for the future Safeguards (Dresden, 1999) [5]. The main aim of the Workshops was "to inform the safeguards community about selected science and advanced technologies that are currently available or that will become available in the next few years and that could be used to support needed advances in international safeguards" and to "stimulate interchange amongst experts in the various technologies and in safeguards".

All the above considerations will essentially change the scientific and technical requirements on ESARDA and particularly on the ESARDA Working Groups.

The ESARDA Working Group on Techniques and Standards for Non Destructive Analysis (NDA) has started to discuss the future of R&D needs required by the NDA laboratories in the future Safeguards [6], while continuing to carry on its "statutory" short term activities.

This paper reports the current R&D activities of the NDA WG and discusses the rôle of NDA techniques in the future Integrated Safeguards, R&D needs, priorities, technical developments required, applications and interfaces with other areas, as for instance fighting nuclear smuggling and safeguarding nuclear materials coming under safeguards from the military cycle, not so far considered as issues to be discussed in the "traditional NMA Safeguards".

## 2. Introduction

The main tasks of the ESARDA Working Group on TECHNIQUES AND STANDARDS FOR NON-DESTRUCTIVE ANALYSIS, as described in its terms of reference, are the following:

- facilitating circulation of information and technology transfer;
- defining needs for procedural standards and for reference materials;
- designing and coordinate the production and characterization of reference materials;
- assessing and contribute to improving the performances of NDA techniques;
- assisting operators and Safeguards Authorities in their duty of Safeguards implementation;
- setting up and maintaining a list of NDA instruments and methods currently used for Safeguards purposes.

The group is presently composed of members from EU countries and observers from USA (LLNL, LANL, BNL), Hungary, Ukraine, IAEA and ABACC.

Members and observers represent plant operators, the nuclear industry, R&D laboratories, NDA instrument developers and National, Regional and International Safeguards Control Authorities: this selected composition contributes to the good and independent scientific outcome of the WG activities.

Special topical meetings are held frequently on items of specific interest, such as NDA on nuclear waste materials [7], passive neutron assay and quality of NDA data [8, 9].

The NDA Working Group is at the present managing, or has just completed, several international projects which are listed here:

- |            |   |
|------------|---|
| Project 1  | Database of NDA Instruments                                 |
| Project 2  | Uranium Intercomparison Exercise                            |
| Project 3  | ESARDA Spectra Data Bank                                    |
| Project 4  | Influence of Nuclear Data                                   |
| Project 5  | Plutonium Intercomparison Exercise                          |
| Project 6  | Monte Carlo Calculations Round Robin                        |
| Project 7  | Monte Carlo Simple Case                                     |
| Project 8  | Preparation and Characterisation of Pu Waste Drum Standards |
| Project 9  | Pu Waste Round Robin  |
| Project 10 | NDA Performance Values List                                 |
| Project 11 | International Workshop on Quality of NDA Results            |

Other projects, long since completed, are mentioned in this paper, but are neither listed above, nor reported in detail in this paper.

Some milestones have been reached recently by the group:

- the Neutron Coincidence Collar “reals” count rate evaluation and the Monte Carlo round robin (Project 6) has been completed;
- the Uranium Enrichment International Round Robin Exercise (Project 2) has been completed, providing a detailed picture of the capabilities of the Safeguards Laboratories around the world in determining  $^{235}\text{U}$  abundance with different techniques;
- the Pu bearing waste drum standards (project 8) have been constructed and characterized: they are available for the round robin amongst laboratories that will start in 2001.

Future activities comprise, besides issues still relevant for “traditional Safeguards”, technical problems linked with the new challenges posed by new regimes of safeguards and non-proliferation.

Indeed, when the integration process between classical NMAC measures and the new (INFCIRC 540) measures will reach the steady state, it is expected that traditional safeguards verification measures could be reduced, in particular for less sensitive materials and facilities.

However, for direct use materials and in sensitive facility types, traditional measures of NMAC will remain at the basis of the verification activities: they must take profit of new technologies as data transmission tools for remote assay monitoring, operation of devices in unattended mode, which will be required by the evolution of the fuel cycle towards large automated facilities.

Other aspects of integrated safeguards will call for modifications and implementation of new NDA techniques, as we will discuss in the next sections: wide area environmental sampling [10] and nuclear signature analysis [11] to detect proliferation indicators, are two examples.

NDA will, however, be largely required also for counteracting nuclear smuggling, to verify nuclear materials coming from the dismantling of weapons, for verifications and accountability under the CTBT and Fissile Material Cut-Off Treaty.

### 3. Activities of the NDA Working Group

In the last years the main scientific/technical activities of the NDA Working Group have been concentrated in three broad areas:

- Reference Materials and Quality Control;
- Assessment of NDA Performance Values and Nuclear Data;
- Databases for Service Activities.

The following sections present a summary of the above activities conducted by the group, without giving all details of the experiences and of the results, since most of them have been reported in scientific literature and at Seminars and Symposia.

#### 3.1 Reference Materials and Quality Control

This is the area that the NDA Working Group has always considered of primary importance, since ESARDA, which includes representatives of R&D laboratories from all over the world, control Authorities and central reference laboratories (like JRC-IRMM) is a suitable forum, both to

achieve the requirements of accuracy and representativeness that a reference material must have and, on the other side, to reach that level of general scientific consensus that is required to conduct an International Intercomparison Exercise properly.

The latest experiences managed by the NDA Working Group reported here, are:

- The Uranium Enrichment Intercomparison Measurement Exercise and its follow ups (Projects 2, 3, 4)
- The Plutonium Intercomparison Exercise, so called Pu-2000 (Project 5)
- The waste reference drums and the subsequent round robin in preparation (Projects 8,9)
- The workshop on the “Quality of NDA Results” organized by the NDA Working Group (Project 11).

#### 3.1.1 Uranium Enrichment Intercomparison Measurement Exercise

The latest example of an international round robin just completed is the intercomparison of gamma spectrometry techniques for the determination of  $^{235}\text{U}$  abundance in low and highly enriched uranium samples.

This exercise was proposed to production, research and safeguards laboratories using X- and  $\gamma$ -ray spectrometry for determining uranium enrichment 14 laboratories from EU countries and from US, Hungary and Russia, participated to the measurement campaigns.

The participants applied one or more of the currently used gamma spectrometry methods, employing the traditional infinite thickness approach, or the more recent approaches that do not require external calibrations.

The reference samples with different matrices and enrichments were prepared and certified by the Institute of Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC) at Geel (Belgium). BNFL Springfields also provided samples, which were then characterized by IRMM.

Samples were then measured by participants and results were collected at the IRMM laboratories.

Results have been analyzed and reported [12,13] by LNHB (Laboratoire National Henri Becquerel) of the CEA Saclay (France).

The outcome was a thorough overview of the capabilities of Safeguards laboratories over the world, to measure  $^{235}\text{U}$  abundance for nuclear material accountability purposes.

Results show that methods employing the traditional “infinite thickness” approach (type “a” methods in fig 1), determine the  $^{235}\text{U}$  abundance with uncertainties that, with few exceptions, are of the order or lower than 1%. Other recent methods which may have several advantages (room temperature detectors as CdTe, or not requiring external calibration as MGA-U), still need improvement to reach an equivalent accuracy.

#### 3.1.2 Pu 2000

A similar exercise is now under execution for plutonium samples, the so called “ESARDA Pu-2000 Exercise”, with the aims of:

- comparing different spectra evaluation codes,
- evaluating the influence of instrumentation and nuclear data on measurement results,

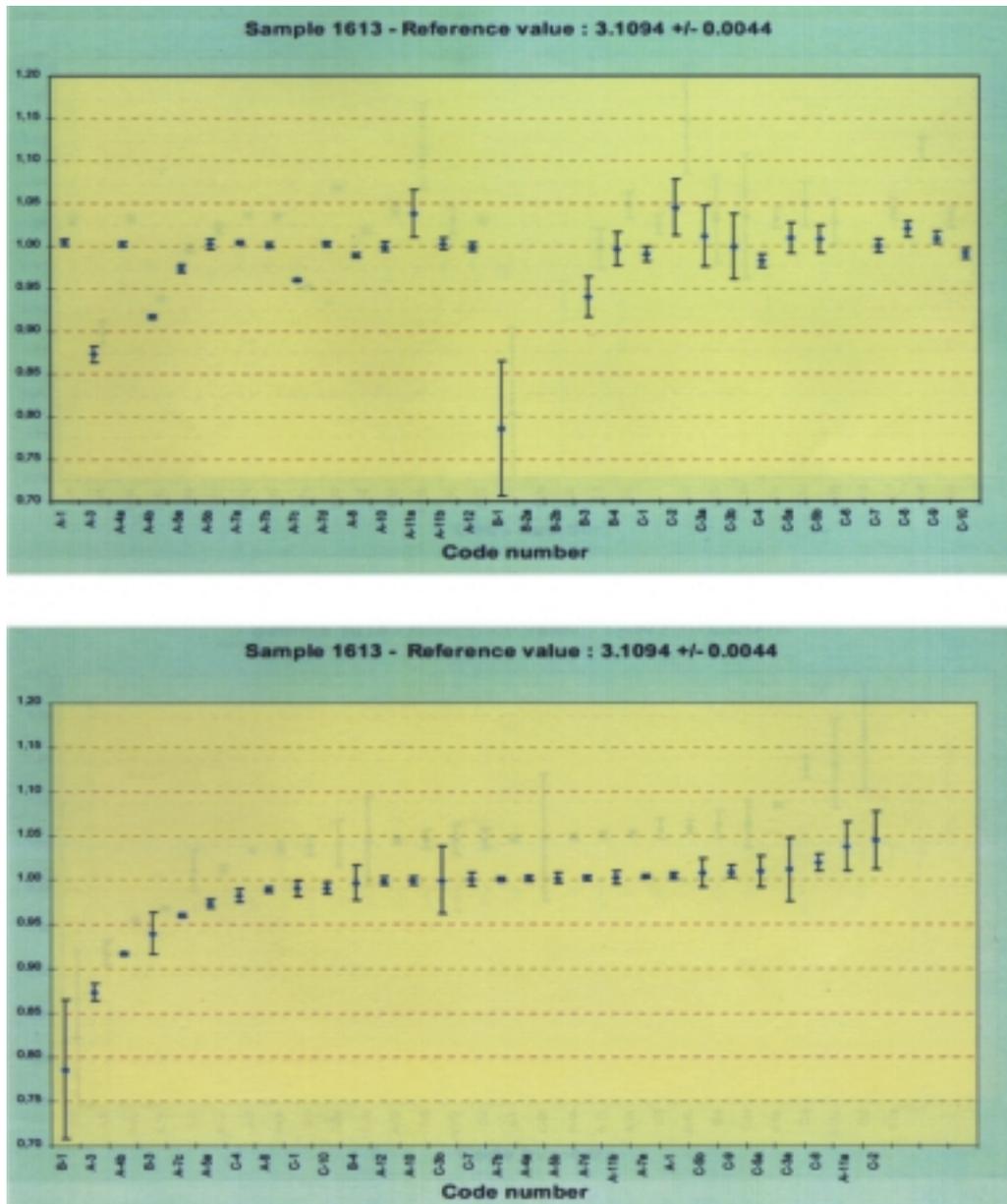


Figure 1 (from Ref.13)

- offering a proficiency testing exercise to participating labs,
- drawing conclusions on the general status and necessary improvements of the performance of plutonium isotopic measurements with respect to safeguards requirements.

The WG has managed the round robin in a similar way as for the U exercise: samples were prepared and characterized by IRMM, the participating laboratories perform the measurements at Geel and CEA Saclay (LNHB) will analyze the results.

The measurements are carried out on a variety of Pu samples:

- PuO<sub>2</sub> sealed sources <sup>239</sup>Pu: 64-98%;
- MOX pellets, PuO<sub>2</sub> powders, Pu metal (1000 mg Pu);
- Quality Control Samples, <sup>239</sup>Pu: 61-93%;
- Unconventional ("strange") samples: 100% <sup>239</sup>Pu;
- Test Samples 100% <sup>241</sup>Am

Results will be available by Autumn 2001.

The group is planning to give the same follow-up to the Pu-2000 as for the Uranium Exercise, ie to insert reference spectra into the ESARDA Spectra Database (see sect. 3.3.2) and to carry on a study on the influence of the used nuclear data set on the results (next sect. 3.1.3).

### 3.1.3 Nuclear Data

During the Uranium Exercise above described, it appeared that different sets of nuclear data had been used by the participants in their measurements. Therefore it was decided to carry on an analysis of the influence of the nuclear data on the results: with that purpose laboratories, which participated to the exercise, were requested to introduce different nuclear data sets in their computer codes in order to check the effects of these input data on the enrichments results obtained. Two participants were willing to cooperate: Lawrence Livermore National Laboratory and the Laboratoire National Henri Becquerel.

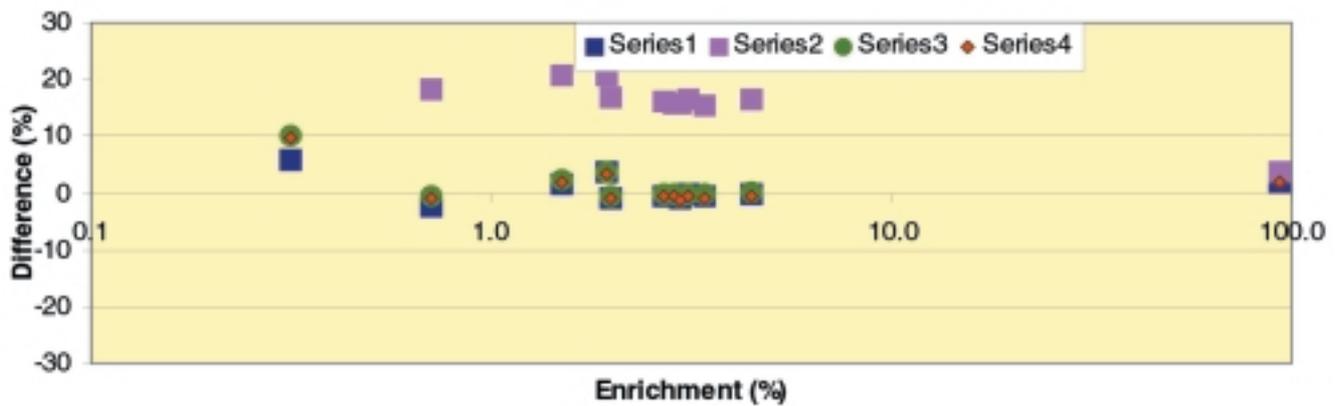


Fig. 2

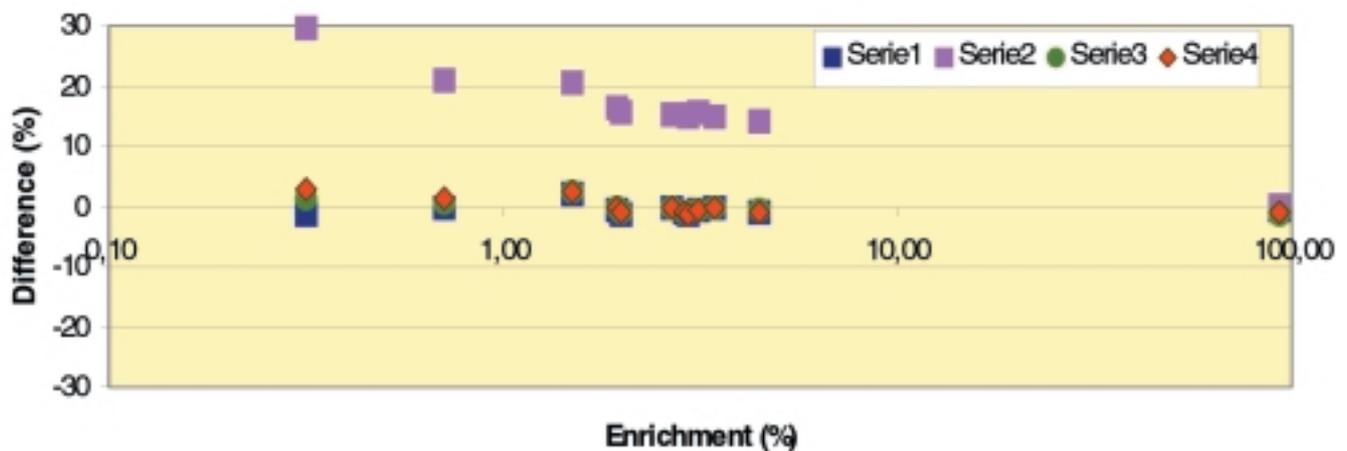


Fig. 3 (From Ref. 14)

The results obtained in the analysis show that a difference up to 20% could be expected only due to the different nuclear data sets used. Three of the four sets gave consistently similar results, the fourth gave important differences, as indicated in figs. 2 and 3. Results were presented to the 23rd ESARDA Symposium, May 2001 [14].

### 3.1.4 Standard Waste Drums for Calibrating Waste Assay

Nuclear waste assay is becoming one of the most important issues in the fields of safeguards and waste management and disposal. Nuclear waste management is constantly in the public eye. From a technical point of view it is one of the most challenging issues for R&D laboratories. Waste drums can only be assayed by NDA techniques prior to being disposed of, or accounted for by Safeguards Authorities. Measurement problems are linked with the low Special Nuclear Materials (SNMs) content to be assayed and to the absence of well characterized, highly certified

reference materials, representative of the waste population currently produced in the EU.

Therefore the NDA Working Group decided to manage a project oriented to design, produce and certify waste drum standards, with variable Pu mass contents, which will be used for calibrating, assessing and improving the NDA techniques currently used to assay the Pu content of waste drums.

It was decided to procure and certify about 100 sealed Pu sources, with Pu content from 5mg to 10g, welded in 35 pins that can be rearranged in different positions inside the drum.

Pu sources were characterized by the JRC-IRMM and welded into pins by CEA-Cadarache. The drums and drum matrices were provided and characterized by BNFL-Sellafield.

Eight 100-I and eight 200-I reference drums are now ready and available for assay. The drums will be used for a blind round robin to assess performances of waste assay, but the drums will then be kept as international reference materials for any further purpose [15,16].

Pu characterization accuracy	Matrix characterization
0.5% on Pu mass fraction	Matrix #1: 0.15 g/cm <sup>3</sup>
0.1% on <sup>239</sup> Pu abundance	Matrix #2: 0.15 g/cm <sup>3</sup> +pvc
0.2% on <sup>241</sup> Am abundance	Matrix #3: 0.4 g/cm <sup>3</sup>

Table 1: Pu and matrix characterization

In Table1 the main drum and source parameters.  
In fig. 4 the eight 100-l drums are presented.



Fig. 4

### 3.1.5 International Workshop on Quality of NDA Results

The management of nuclear materials, their national, regional, international safeguarding as well as the R&D background activities that underpins the Nuclear Material Accountancy and Control (NMAC) are more and more requested to guarantee the quality of the measurement results involved. All aspects of NMAC, are concerned with guaranteeing the quality of the results: i) the management and accounting of nuclear materials by the plant operator; ii) the auditing and verification of nuclear material stocks and flows carried out by control authorities iii) finally R&D is an area of challenging study on QC/QA, complicated by the objective difficulty to define and quantify the "product" of the research activities.

Among the tasks of the ESARDA NDA Working Group, the assessment and the improvement of the performances of NDA techniques is one of the most important. Following the issuing of tables of performance values currently achievable and achieved by NDA techniques [17,18], the Working Group finally decided to organize an International Workshop on the matter of NDA quality of measurement results, inviting experts coming from EU Companies, laboratories and certification or accreditation organizations.

The specific aims of the Workshop were:

- gaining insight into terms, procedures, norms and legal issues of Quality Assurance;

- getting an overview on the way quality assurance is achieved in different laboratories;
- reviewing the scientific/technical requirements of quality measurements and assessing the most important NDA techniques against those requirements;
- reviewing the formal requirements for quality measurements;
- exchange of experience with laboratories being on their way to or having achieved the implementation of a formal Quality Management System;
- reviewing the specific requirements in the areas of Quality Assessment and Assurance posed by the auditing organizations to development R&D laboratories;
- drawing conclusions and giving recommendations to NDA laboratories.

The workshop was held in November 1998 [9] at the JRC-Ispira, Italy. During three days of presentations and discussions, 25 participants from both purely R&D, from commercial laboratories, from certification/accreditation bodies and safeguards authorities, drafted a set of conclusions and recommendations to NDA laboratories, covering the range of problem areas above outlined.

### Conclusions of the Workshop

The most important conclusion was that, in fact, all participating laboratories exhibit some kind of Quality System, although often not completely documented. It was agreed that this is absolutely necessary to produce reliable measurement results. With respect to official recognition (certification or accreditation) various kinds of opinions were represented, from "not necessary" to "important".

No disagreement on the very high importance of Reference Materials in general and NDA standards in particular: once again, the need of more numerous and/or more accurate/precise standards was expressed.

In detail, the following conclusions and recommendations were drafted:

- No reliable measurement can be done without a quality system. This quality system, can range from very informal (based only on trained and experienced operators) to full accreditation/certification, following the needs.

The implementation of a Quality System with accreditation/certification are tools to ensure and improve quality and are not the goal by themselves. Therefore the Quality System should be tailored to the real needs and/or to the customer requirements.

- The high importance of the expectations of the "customer" for all actions related to QM was strongly emphasized, with view to the fact that often laboratories exaggerate the width and depth of their QS documentation, which leads to higher costs than necessary.
- Performance monitoring represents an integral part of the Quality Control of equipment and must be foreseen in the design of new instrument.
- Calibrations must be performed using certified reference materials, or using other techniques assuring traceability to primary international reference materials.

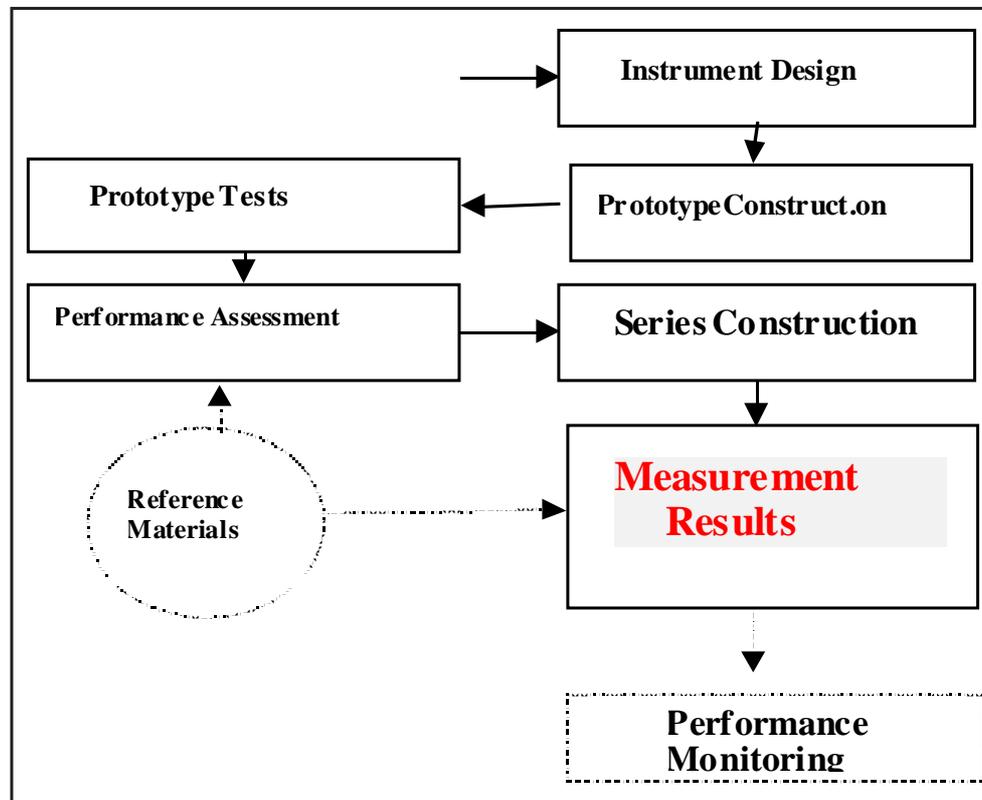


Fig 4: Schematic View of the Process Leading to Measurement Results.

- Where suitable standards do not exist, Monte Carlo simulation can be used. Only those codes which have been validated by relevant experiments should be used.
- It was clearly stated that QC and QA procedures, at any required level, must be applied not only to the final phases of measurement taking, but must be applied to the whole cycle of activities leading to the results, i.e. starting from the instrument design, as indicated in Fig.4.

### 3.2 Performance Assessment

The assessment of the performances of the NDA methods is an area where the WG is permanently and intensively active.

Broadly speaking, two kinds of activities have been carried on under this heading: first (sect. 3.2.1) the overview of different techniques and methods with the aim of evaluating their "typical" performance (in terms of measurement uncertainty) when applied to different material types, in different measurement conditions. This first kind of activity led to the ESARDA NDA Performance Values (EPVs) compilations (Project 10) [17,18].

The second area of assessment concerns experiences specifically designed by the WG to determine the capabilities of a tool: this is the case for instance, of the Monte Carlo round robins (sect. 3.2.2; Projects 6,7). The aim of this last area is also to improve, after having assessed the performances, the capabilities of a technique: this was the case, for instance, of the Project regarding the evaluation and improvement of the  $^{242}\text{Pu}$  content evaluation, developing and using a new algorithm [19] and of the Project conducted together with the LEU Working Group aimed to assess the NDA sampling error [20].

#### 3.2.1 ESARDA NDA Performance Values (EPVs)

The NDA Working Group has been engaged for several years in setting up, maintaining and updating an extensive list of performances regarding NDA techniques applied to the most common material types of the fuel cycle, including waste materials, which are becoming more and more relevant also for safeguards. The evaluation was essentially based on the experience of the WG members, vast literature survey, results of campaigns in which the member laboratories were involved, as well as on evaluations from IAEA and EURATOM inspections.

The outcome of this work has been an extensive compilation of performance values, intended as typical measurement uncertainties, for different NDA methods, applied to the most common material types encountered in the EU fuel cycle. The EPVs have been diffused in documents and papers presented on behalf of the Working Group in various occasions to meetings and Symposia [17,18].

The extensive work and the outcome produced were also used to give an essential contribution to the compilation of the International Target Values list [21], prepared by IAEA with the contribution of several international organizations.

Both ITVs and ESARDA NDA Performance Values are currently under reviewing, being about ten years old: it is expected to have the new versions by early 2001.

#### 3.2.2 Monte Carlo Intercomparison Exercises

One of the activities that in the past have been frequently claimed to be promising, but had not received sufficient attention, is the use of theoretical simulation codes, like Monte Carlo, in modeling experimental configurations,

predicting results and so reducing the number of required reference material standards. Now, under the stimulation of the EURATOM Safeguards Office that has listed this issue in their needs list [22], the NDA Working Group has decided to launch an action to evaluate the potential of theoretical methodologies and in particular of Monte Carlo packages (eg MCNP), to be used as laboratory and field tools.

The first action decided was an assessment of the actual capabilities of theoretical methods in evaluating results from NCC (neutron coincidence counting) methods.

To that purpose the LEU reference PERLA fuel bundle [23] was chosen: participant laboratories were provided with a MCNP input file and evaluated the “reals” rate, as recorded by a standard shift register. The shift register records were collected in controlled “reference” conditions by the JRC-Ispra PERLA laboratory.

Several European Safeguards laboratories participated to the exercise, employing slightly different methods. Results and conclusions of the exercise have been published [24,25] and are not detailed here, but the general conclusion was that one can expect today that even complex geometries in active neutron collar counting can be simulated by theoretical techniques with approximations lower than 10%, probably after the ESARDA exercise and the experience gained, better than 5%.

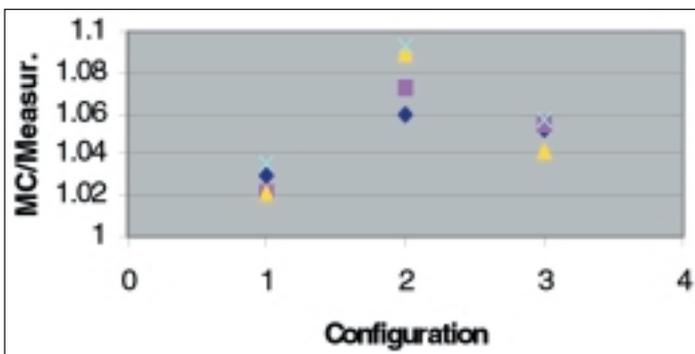
In fig. 5 the overall results from the participants, showing measured/calculated ratios of “reals” count rates, for different geometrical configurations, are given.

In view of the good outcome and promising results of the first exercise, the NDA Working Group decided to start a second round robin, which will require participants to use Monte Carlo packages to evaluate counting rates produced in a simple geometry, consisting of He3 detector tubes, exposed to a neutron flux from an AmLi source, after crossing a polyethylene slab. Participants will be required to model the experimental configuration with their code(s), e.g. MCNP, and to evaluate neutron counting rates.

The final purpose of the exercise is to enable laboratories to set up a computational system capable of field prediction of instrument answers, as well as to limit the number of calibration curves required, therefore also limiting the number of required reference material standards.

### 3.3 Databases

As a service to the Safeguards community, the NDA WG is preparing a number of databases which are addressed to different users: R&D laboratories, Control Authority, nuclear operators.



For this kind of work the group profits from its status of an international forum, where operators and inspectors are sitting together and developing laboratories with certifying laboratories.

This gives to the outcome of the WG's work an informal but substantial “certificate” of scientific quality and independence.

#### 3.3.1 ESARDA Database for NDA Instrument and Methods: ENDA

As outlined in the introduction, one of the main items of the group's terms of reference is keeping a list of NDA instruments, currently applied in the different parts of the fuel cycle.

The group has recently started to discuss and specify this project, deciding to set up not just a list of items, but to “frame” the instruments into facility safeguards schemes. The decision was made to start with a prototype database, which will consider the back/back end of the fuel cycle, i.e. the disposal of spent fuel. The project is in the starting phase and it is being performed by STUK, Finland.

The first prototype, based on Microsoft Access, is now ready and working [26]: instrument data are being inserted into the database.

After the first prototype, all other facility types of the fuel cycle will be considered: fabrication, reprocessing, waste facilities.

#### 3.3.2 ESARDA Spectra Data Bank: ESDB

One of the most important aspects for instrument developers is to find “reference” measurement results data files for testing their prototypes. This avoids that each laboratory reproduces experimental set ups and measurements campaigns and, on the other side, if the test measurement results data sets are “certified”, this gives further validation to the analysis tools.

This is particularly true for gamma spectrometry tools (hardware but also software packages), which need to be tested to evaluate their performances on reference spectra, which should be known and accepted by the international community.

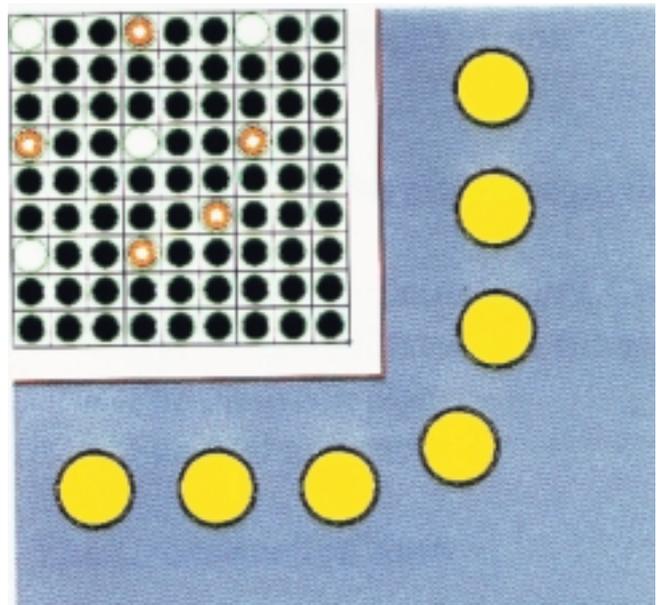


Fig. 5 (from ref. 25)

Aware of these aspects, the NDA WG decided to use the outcome of the Uranium and Plutonium Intercomparison Exercises to set up a bank of "spectra" with the above basic reference characteristics, since they have been taken on certified samples and because the exercise was open to and followed by several international laboratories.

Uranium spectra are being properly formatted and introduced into the DB: by end 2000 the Uranium part will be working and the DB will be put on Internet, to serve the largest possible number of users.

By the end of 2001, the Plutonium section of the DB will also be working.

## 4. Future

The NDA WG will continue the activities that have been outlined in the previous sections, in the framework of exchange of information, technology transfer, Performance Evaluation, international exchanges and round robins.

But the group is aware that nuclear Safeguards are changing: the advent of integrated Safeguards with the implementation of the Additional Protocol will require rational redefinition of goals and criteria for some material types and facilities, possible relaxation of some traditional measures, use of new advanced technologies, modifications of inspection practices and all this will certainly affect the terms of reference and the activities of the NDA WG.

The evolution of the fuel cycles and the new technologies made available by scientific progresses will also impose new technical challenges to international safeguards and non-proliferation.

Following these needs the group started to discuss the technical impacts that the above issues will have on the development and application of NDA techniques under the new requirements. In the following paragraphs some excerpts of this analysis are given.

### *New Design of Instruments for NMAC*

The evolution of the fuel cycle towards large automated facilities and a more cost/effective use of human resources, require new technical approaches, such as the development of newly designed instruments, more automated and remotely controlled.

This topic has recently received quite considerable attention in Safeguards. It is in fact one of the most important directions for future technical developments of field systems. The NDA WG together with other ESARDA WGs, are required to develop technical guide-lines for the future [27].

Modern technology allows these developments: instruments can be designed today so as to permit assay in almost automatic mode: an "integrated system" is capable of a "low level" field decision making processing, allowing simpler decisions to be taken by the field software. Raw data are transmitted, if required, to HQs.

This will require a new generation of Safeguards instruments, much more integrated among disciplines (NDA and C/S, for instance); much more integrated from a logical point of view, with information of different nature (eg from

extended declaration); integrated from an informatic point of view, within networks which should, as far as possible in view of the data confidentiality, make use of modern information society tools.

### *Back end of the fuel cycle*

For safeguards but also for national waste management and decommissioning policies, NDA techniques for waste and scrap assay, for monitoring spent fuels and waste in intermediate/final repositories must undergo substantial improvements, exploring more sophisticated tools as neutron/gamma tomography, calorimetry (for scraps), which could show in future high performances and reliability.

This is an important area, since several EU countries and future incoming States have or are looking to spent fuel repository policies.

### *Excess Materials*

A new application field is represented by the assay of materials coming from the military field. For these materials it is necessary, at least in a first step, to develop or modify NDA methods in order to ensure on the one side the confidentiality of some strategic parameters, but also the auditors' need of quantitative assessment.

### *Illicit Nuclear Traffic*

Fighting nuclear smuggling is an important aspect of protecting the general public from a kind of nuclear threat and preventing disruptive actions, dreadful for the public health and security.

NDA is almost the only technical tool for detecting and, through deterrence, preventing nuclear smuggling. Therefore NDA R&D laboratories and industrial companies will be more and more required to develop, test and implement instruments and tools for fighting illicit nuclear traffic, with required performances which will gradually increase in terms of detection limits.

## 5. Conclusions

NDA will still be an important pillar of the national, regional and international NMAC systems and the perception of auditors and laboratories is for an increased use of non destructive techniques. This is linked to several regulatory and technical characteristics of Nuclear Safeguards, but also to the aspect that verification regimes, by their political nature, are committed, through technological and scientific developments, to "minimize subjectivity" [28] in their conclusions.

However future developments in Safeguards and non proliferation, as well as in other nuclear material control activities (eg: smuggling), require that NDA laboratories and Working Groups pay attention to the new requirements and challenges.

Developments in the nuclear fuel cycles and the appearance on the "market" of new advanced technologies, will also introduce important changes.

Synergies can be found, with benefit for the use of resources, looking at other nuclear verification regimes [29,30].

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# International Target Values 2000 for Measurement Uncertainties in Safeguarding Nuclear Materials

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## Abstract

This issue of the International Target Values (ITVs) represents the fifth revision, following the first release of such tables issued in 1979 by the ESARDA/WGDA. The ITVs are uncertainties to be considered in judging the reliability of analytical techniques applied to industrial nuclear and fissile material, which are subject to safeguards verification. The tabulated values represent estimates of the "state of the practice" which ought to be achievable under routine measurement conditions. The most recent standard conventions in representing uncertainty and reliability data have been considered, while maintaining a format that allows comparison with the previous releases of the ITVs. The present report explains why target values are needed, how the concept evolved and how they relate to the operator's and inspector's measurement systems. The ITVs 2000 are intended to be used by plant operators and safeguards organizations, as a reference of the quality of measurements achievable in nuclear material accountancy, and for planning purposes. The report suggests that the ITVs can be used with benefit for statistical inferences regarding the significance of operator-inspector differences whenever valid performance values are not available.

## 1. Introduction

Safeguarding nuclear material involves a quantitative verification of the accountancy of fissile materials by independent measurements. The effectiveness of these verifications depends to a great extent upon the quality of the

accountancy measurements achieved by both the facility operator and the safeguards inspectorate. For this reason a typical model of Safeguards Agreements<sup>1,2</sup> stipulates that:

*"The Agreement should provide that the system of measurements on which the records used for the preparation of reports are based shall either conform to the latest international standards or be equivalent in quality to such standards".*

Although the above requirement was directed to the facility operators, it indeed applies equally well to the safeguards inspectorates.

In the absence of relevant international standards of measurements, the International Atomic Energy Agency (IAEA) had defined in the 1970s a set of international standards of nuclear material accountancy<sup>3</sup>, which lists the "expected measurement accuracy associated with the closing of a material balance" at five different types of nuclear facilities. However, these values have never been reviewed despite numerous technological changes since their adoption by consensus by a group of experts designated by their Governments. Safeguards officials and evaluators but also plant measurement specialists need more current and informative references regarding the performance capabilities of measurement methods used for the determination of the volume or mass of a material, for its sampling, its elemental and isotopic assays. Such informa-

tion is needed for the various nuclear materials encountered in the nuclear fuel cycle.

The Working Group on Techniques and Standards for Destructive Analysis (WGDA) of the European Safeguards Research and Development Association (ESARDA) pioneered the way in 1979 by presenting a list of "Target Values" for the uncertainty components in destructive analytical methods<sup>[4]</sup> to the safeguards authorities of Euratom and of IAEA. Revised estimates were prepared in collaboration and published as the 1983 Target Values<sup>[5]</sup> after four years of extensive discussion and consultation with and within operators' laboratories and safeguards organizations. The international acceptance of the concept grew further with the next review, which involved, besides the ESARDA/WGDA and IAEA, the active participation of the members of two specialized committees of the Institute of Nuclear Materials Management (INMM). The 1987 Target Values, published as a result of this review<sup>[6]</sup>, defined, like the previous editions, the values of "random" and "systematic" error parameters to be aimed for in elemental and isotopic analyses of the most significant types of materials using common destructive analytical methods. The same groups took a new step when they agreed to define with the 1988 edition<sup>[7]</sup> the values of the random error parameter to be met in the elemental assays as a result of sampling. Unfortunately, it was not possible at this time to include values for sampling uncertainties arising from systematic effects.

Following a 1988 recommendation of the IAEA Standing Advisory Group on Safeguards Implementation (SAGSI), the IAEA convened a Consultants Group Meeting in June 1991 to provide expert advice on international standards of measurements applicable to safeguards data. A concept of International Target Values (ITVs) was proposed on the model of the 1988 ESARDA Target Values and included estimates of the "random and systematic error" uncertainties originating from the measurements of volumes or masses of nuclear materials. The scope of ITVs was also extended to include a consideration of the non-destructive assay methods (NDA) which had won acceptance as accountancy verification tools.

Specialists from four continents took part in the discussion of the proposed concept. The ESARDA/WGDA held joint meetings with the ESARDA Working Group on NDA methods (ESARDA/WGNDA). The IAEA organized a series of Consultants Group Meetings with the participation of a representative from a large European reprocessing plant, of Brazilian and Japanese nuclear national authorities along with representatives of ESARDA, INMM, the International Organization for Standardization (ISO), the European Commission (EC) and IAEA inspectorates. The result was the publication of an IAEA Safeguards Technical Report in March 1993, titled "1993 International Target Values for Uncertainty Components in Fissile Isotope and Element Accountancy for the Effective Safeguarding of Nuclear Materials"<sup>[8]</sup>. Articles in the ESARDA Bulletin<sup>[9]</sup> and in the Journal of the INMM<sup>[10]</sup> widely publicized the IAEA technical report. The report itself was translated into Japanese<sup>[11]</sup>.

International experts and panels have now reviewed the experience gained with the use of the 1993 ITVs and the progress made since 1993 in accountancy and safeguards verification measurements. These include ESAR-

DA/WGDA, ESARDA/WGNDA, the Institute for Nuclear Material Management (INMM), the Japanese Expert Group on ITV 2000, Working Groups of the International Standardization Organization (ISO) dealing with analytical measurements in nuclear fuel industry and the Brazilian/Argentinean Agency of Accountability and Control of Nuclear Materials (ABACC). This report contains the changes made in the presentation of the ITVs and in some of the target values to reflect the latest recommendations of the experts.

An effort was made to bring the nomenclature in line with the latest recommendations of ISO<sup>[12]</sup>, the National Institute of Standards and Technology (NIST)<sup>[13]</sup> and the European Association of Chemical Measurements (EURACHEM)<sup>[14]</sup>. A clear distinction for example is made between the meaning of the term "error" and the term "uncertainty". The ITVs 2000 indeed represent target standard uncertainties, expressing the precision achievable under stipulated conditions. These conditions typically fall in one of the two following categories: "repeatability conditions" normally encountered during the measurements done within one inspection period; or "reproducibility conditions" involving additional sources of measurement variability such as "between inspections" or "between laboratories" variations.

As in earlier publications the values listed in the present document have been derived from an evaluation of actual measurement data. Four sources of information were considered. The most relevant and complete set of measurement data still comes from the information gathered by safeguards inspectorates during the statistical evaluation of the results of the measurements reported by the facility operators and the results of independent measurements performed on the same materials by the inspectors<sup>[15,16]</sup>. This approach will be referred to as the "top-down" approach. These data were complemented and confirmed by "bottom-up" assessments of measurement uncertainty components published by measurement specialists<sup>[17-26]</sup> and derived according to the ISO<sup>[12]</sup>, NIST<sup>[13]</sup> and EURACHEM<sup>[14]</sup> guides. In addition and whenever possible, it was verified that the proposed ITVs were consistent with the results of laboratory intercomparisons<sup>[27-33]</sup> or measurement quality evaluation programmes<sup>[34-48]</sup>. In cases where little or no statistical data was available (particularly for sampling uncertainties), some values were defined on the basis of expert opinion.

The ITVs 2000 bear a date like the ESARDA Target Values and 1993 ITVs issued previously. This reflects the experience that the quality of measurements may improve with the development of newer methods and instruments. ITVs also reflect the current understanding of the structure of the uncertainty components in nuclear material accountancy measurements. Changes can also occur in the future as this understanding improves or varies.

As with the previous lists, the ITVs 2000 should be achievable from today forward under the conditions normally encountered in typical industrial laboratories or during actual safeguards inspections. They do not represent the measurement uncertainties, which would only be achieved under exceptional or ideal laboratory conditions, or with most recently developed methods, which have not yet found wide use for daily and routine measurements.

Significant changes in the application of instruments and techniques have taken place since the previous edition. Measurements with instruments like high level neutron coincidence counters (HLNC), K-edge X-ray absorptiometer and fluorescence analyzers (HKED) are used routinely at the plants by inspectors with great success, not only to detect partial defects but also to verify the flow and balance of nuclear materials. This has allowed to decrease strongly the fraction of items, which need to be verified by chemical analysis. The latter methods are used now mainly for verifying the quality of operators' measurement systems and the absence of small but measurable biases in the closing of the material balances. Here, improvements were also observed with the combined use of Large Size Dried Spikes (LSD) and thermal ionization mass spectrometers with multidetectors and total sample evaporation for the verification of the uranium and plutonium content in spent fuel solutions and U/Pu fuel materials by isotope dilution mass spectrometry (IDMS).

It is expected that the ITVs 2000 will continue to be a motivating goal for beginner laboratories and a reasonable reference for experienced laboratories and safeguards evaluators. With the growing acceptance of modern quality assurance concepts it is suggested that the ITVs 2000 can also constitute a good reference against which analytical laboratories would validate their measurement system.

## 2. Safeguards Accountancy Verification Measurements

As evident from the title of the report and the introduction, the principal application of the ITVs should be in safeguards activities. The safeguards verification data also form the major source of information on which the ITVs are based. A description of the origin of the safeguards data is therefore relevant.

Figure 1 describes the basic measurement scheme followed in safeguards measurement verifications. For each inspection,  $j$ , the inspector selects, in accordance with a random sampling plan, the items or batches of nuclear materials to be verified by an independent measurement. The inspector then compares the result of his verification measurement,  $Y_{ij}$ , to the result,  $X_{ij}$ , which the operator has obtained on the same batch or item  $i$ , and which the operator has declared to the inspectorate. The ability of the inspector to detect whether the difference  $d_{ij}$  is significantly different from zero depends upon the overall uncertainties in the results  $X_{ij}$  and  $Y_{ij}$ . Figure 1 identifies the major steps of the measurement process where uncertainties can arise, although not all steps may be relevant for every method (e.g., several of the steps may be omitted or combined under a single step for NDA methods).

**Step 1** corresponds to the measurement of the volume or mass of the item or batch of material. This so-called "**bulk**" measurement, when needed, takes place in the plant area and involves a calibration procedure.

**Step 2**, the "**sampling**", involves removing, for the purpose of the analytical measurement, a representative portion of the material from the batch or item to be analyzed. This portion may be a complete item in the case of an NDA measurement. This step is also done in the plant area.

**Step 3** concerns the precautions which must be taken in the way the sample is "**conditioned**" and packaged at

the sampling station so that all characteristics to be measured are preserved during its transport to the location or laboratory where the characteristics will be measured<sup>[49]</sup>.

**Step 4**, the "**shipment**", is the transport of the sample to the location where it can be measured. This is rarely a trivial operation even when the movement is very short, as in the case of an NDA measurement, which is often done practically on the spot.

**Step 5**, the "**treatment**", is intended to bring the sample into the most appropriate geometrical, physical and/or chemical form for the measurement. This step is skipped when a complete item is subject to an NDA measurement. The treatment of a sample taken for destructive analysis may involve a sequence of individual steps, such as subsampling, dissolution, dilution, spike or standard addition, chemical treatment or chemical separation, etc.

**Step 6**, represents the "**measurement**" itself. In general terms, a measurement is based on a calibration from which the parameters linking the observed signal and the measurand are determined. Typical examples are HLNC calibration curves for Pu mass determination, calibrations of Gamma Spectrometers for <sup>235</sup>U abundance determination or the determination of the mass-discrimination correction factor for a mass-spectrometer. The standardization of a titrant solution is another example of a calibration, although it is frequently not recognized as such. Calibration functions may be as simple as a single calibration factor (actually representing a straight line through the origin), or may be complex and represented by an empirically determined calibration curve. Calibrations based on recognized references, such as certified reference materials or well-known physical constants, establish the traceability chain between the measurement result and the International System of Units (SI). Calibrations may be valid and used without modifications for long times, repeated on a daily basis or even performed with each individual measurement. Sometimes, calibration is performed in two steps: elaborate calibration exercises for determining the fundamental characteristics of the calibration function are combined with more frequently repeated "normalization" measurements to correct for short-term effects or minor deviations from the overall calibration function at the specific working range.

**Step 7**, the "**calculation**", consists in transforming the results of the physical or chemical measurement obtained in the preceding step into an estimate of the amount of fissile element or isotope in item or batch  $i$ . Particularly when the operator and inspector use DA, this involves estimating the total element content, by combining the result of the bulk measurement  $w_{ij}$  with the elemental concentration  $c_{ij}$ . In the case of Uranium materials, this is combined with the isotope abundances  $f_{ij}$  of the fissile isotope (<sup>235</sup>U or <sup>233</sup>U) to yield a measure of the amount of fissile isotope in item or batch  $i$ , according to equations (1) and (2), respectively.

$$X_{ij} = w(O)_{ij} \cdot c(O)_{ij} \cdot f(O)_{ij} \quad (1)$$

$$Y_{ij} = w(I)_{ij} \cdot c(I)_{ij} \cdot f(I)_{ij} \quad (2)$$

Every stage of the process, starting with bulk measurements must be performed under well-controlled conditions. Hence quality control measures are imperative at every step of the process. Quality control on sampling can be

done by taking replicate samples after different mixing times or taking samples from a number of items of the same batch of bulk materials. Quality control materials or samples can be introduced at specific steps to monitor the quality of the whole process or any part of it, including the conditioning and shipment steps. Figure 1 shows an example where control materials are used independently by the operator and the inspector to check the quality of the processes following the sampling. Quality control measures should be performed in the frame of a documented quality system<sup>[49-53]</sup>.

The uncertainties in the measurements of element concentrations and isotope abundances in the ITVs refer to the combined effects of the uncertainties in Steps 3 to 6 occurring after the taking of the sample in Step 2.

**Step 8**, the "reporting" of the results, is purely clerical but unfortunately it can be a source of errors. Uncertainties arising from such errors are not considered in the ITVs proposed in this document. Yet it is essential that appropriate quality assurance measures be taken to avoid the occurrence of clerical errors.

When NDA is used the attention focuses most on the measurement (Step 6) as the preceding steps have usually less impact or may even be omitted. For example, bulk measurements and sampling are not needed if the NDA method allows direct measurement of the total amount of fissile element or isotope contained in a whole item or batch of nuclear material, as with various neutron counters or calorimeters.

### 3. Evaluation of Safeguards Accountancy Verification Measurements

The statistical terms used in this document are defined in Annex 1 in an effort to promote the understanding of the statistical concepts used here.

The safeguards inspectors examine the operator-inspector paired differences to determine whether these remain within upper and lower limits, which are commensurate with the characteristic uncertainties of the operators' and inspectors' measurement systems. For this purpose, the inspectors need to quantify the major uncertainties of the actual data collected during their verification activities.

Two categories of uncertainties play an important role in planning for inspections and in drawing inferences from inspection data: uncertainties due to repeatability effects, which are of a purely random nature, and uncertainties resulting from systematic effects within a given set of data, corresponding to an inspection period<sup>[54,55]</sup>. These uncertainties will be designated by the symbols **u(r)** and **u(s)**, respectively:

- **random uncertainty components, u(r)**, are due to errors varying in an unpredictable way among individual items or results. Counting statistics or the repeatability of measurements within a short period of time under constant conditions are typical examples for random uncertainty sources. Simply stated, the effects of random uncertainties can be reduced by repeated measurement, sampling and analysis, but it is not possible to correct for random errors.
- **uncertainty components of a systematic character, u(s)**, are due to errors affecting an entire group of

items in the same way, like all measurement results interpreted with the same calibration curve, normalized with the same normalization experiments, or affected by the same background subtraction. But also uncertainties in the certified values of reference materials, nuclear data uncertainties or constant instrument or laboratory biases will appear to have a systematic character. The effects of uncertainties of a systematic character cannot be reduced by repetition under a fixed set of conditions encountered during a given inspection period. The cause of systematic errors may be known or unknown. If both the cause and the value of a systematic error are known, it can be corrected for, but there will still remain an uncertainty component of systematic character, which is associated with this correction.

A basic assumption is that  $u(r)$  and  $u(s)$  are characteristics of the type of material, its chemical and physical form and of the method of measurement. A further assumption is that the component of systematic character,  $u(s)$ , is constant for a given inspection period, but that it varies in a random manner from one inspection to another, for both the operator and the inspector.

Consequently, the inspectors group the data pairs originating from one inspection period,  $j$ , by material balance areas (MBA), by strata of materials of similar characteristics and by measurement methods<sup>[56]</sup>. For a given MBA and stratum, call:

$$d_{ij} = (X_{ij} - Y_{ij}) / X_{ij} \quad (3)$$

the operator-inspector difference,  $d_{ij}$ , for item  $i$  in inspection  $j$ , with

$$\begin{aligned} i &= 1, 2, \dots, m_j \\ j &= 1, 2, \dots, K \end{aligned}$$

**Note:** *to simplify the presentation, relative differences are treated here. In practice, absolute differences,  $(X_{ij} - Y_{ij})$ , would be used when the size of the items of a given stratum vary widely.*

The assumed error model is

$$d_{ij} = d + \Delta_j + \varepsilon_{ij} \quad (4)$$

where

- $d$  is the mean difference over the  $K$  inspections,
- $\Delta_j$  is the systematic error of the operator-inspector difference during inspection  $j$ , and
- $\varepsilon_{ij}$  is the random error of the operator-inspector difference for item  $i$  during inspection  $j$ .

The expected values of  $\Delta$  and  $\varepsilon$  are both zero (i.e., they are both centred random variables in a statistical sense). An analysis of variance components of the operator-inspector differences,  $d_{ij}$ , according to this model equation gives estimates of the variance  $s^2(\varepsilon)$  of the random component and of the variance  $s^2(\Delta)$  of the component due to systematic effects within the given inspection period<sup>[56-59]</sup>. In performing this analysis of variance components, it is generally assumed that  $\Delta_j$  and  $\varepsilon_{ij}$  are normally distributed and that the variances of the random error are the same for all inspections. The set of results are therefore screened for outliers prior to performing this evaluation.

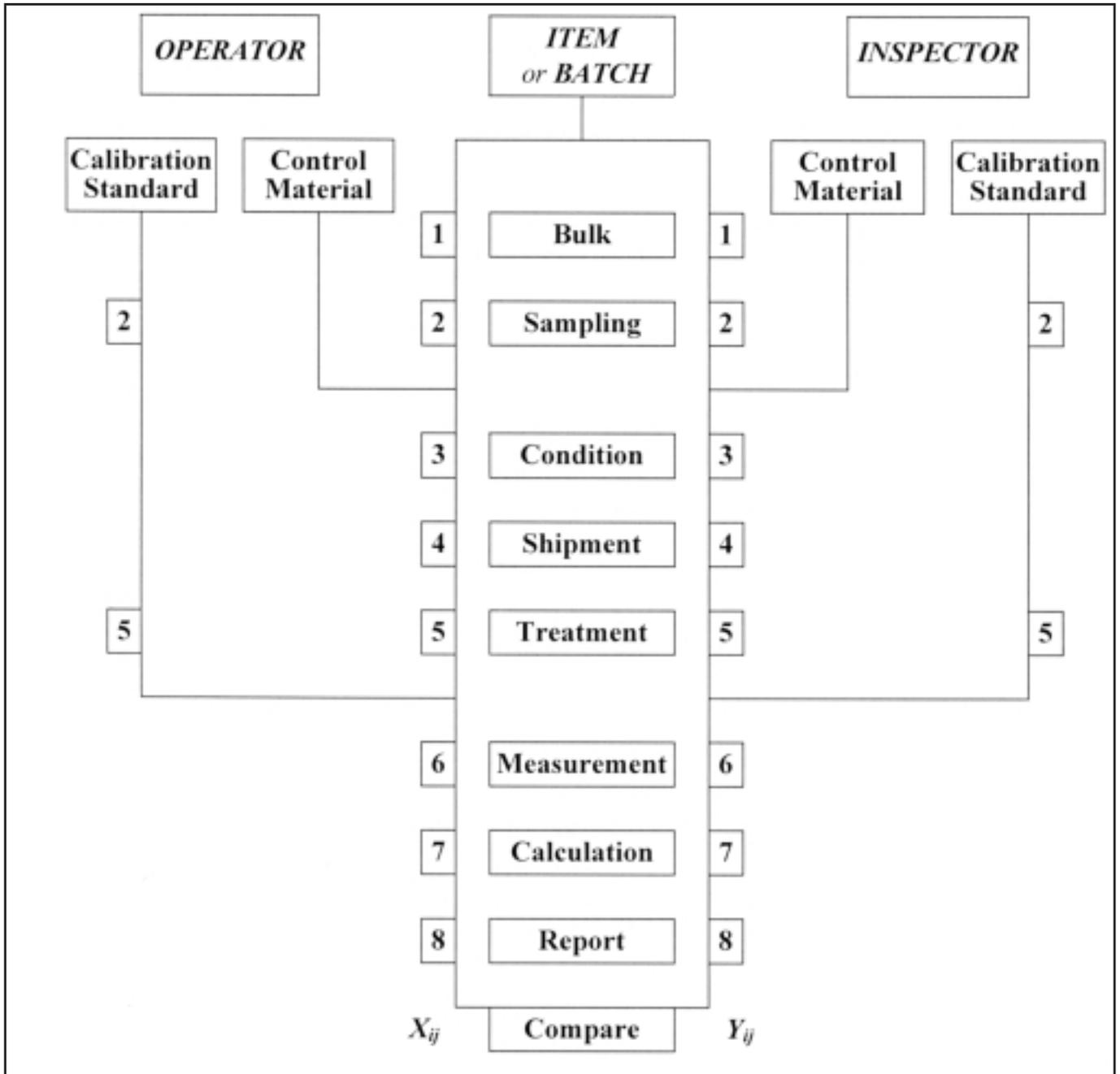


Figure 1: Accountancy and Verification Measurement Scheme for item  $i$  during inspection  $j$

Paired comparisons of this type are done separately for bulk measurements, element concentrations and isotope abundances, as well as for the masses of fissile elements and isotopes. One obtains, for each type of measurement, an estimate of the combination of the actual uncertainty components for the operator's and inspector's measurement systems:

$$s^2(\epsilon) = u^2(r,O) + u^2(r,I) - 2r(\epsilon) u(r,O) u(r,I) \quad (5)$$

$$s^2(\Delta) = u^2(s,O) + u^2(s,I) - 2r(\Delta) u(s,O) u(s,I) \quad (6)$$

where

- $u(r,O)$  and  $u(r,I)$  are the standard uncertainties due to random error components for the

operator and the inspector respectively,

is the Pearson correlation coefficient between the operator's and the inspector's random errors,

- $r(\epsilon)$

and

- $u(s,O)$  and  $u(s,I)$

are the standard uncertainties due to effects of systematic character for the operator and the inspector respectively

- $r(\Delta)$

is the Pearson correlation coefficient between the operator's and inspector's systematic errors.

It can be expected that the uncertainties of operator's and inspector's data have similar magnitudes when both are obtained with similar methods. Under the assumption that the errors of the operator and of the inspector are independent from each other, i.e.

$$r(\varepsilon) = r(\Delta) = 0, \quad (7)$$

the values

$$u^2(r,O) \approx u^2(r,I) \approx s^2(\varepsilon) / 2 \quad (8)$$

and

$$u^2(s,O) \approx u^2(s,I) \approx s^2(\Delta) / 2 \quad (9)$$

provide good estimates of the standard uncertainties.

In other situations operator's DA results may be compared with much less precise and/or much less accurate inspector's results obtained for example by some NDA methods. If, for example

$$u(r,I) \gg u(r,O) \quad (10)$$

and

$$u(s,I) \gg u(s,O) \quad (11)$$

Then,

$$s(\varepsilon) \approx u(r,I) \quad (12)$$

and

$$s(\Delta) \approx u(s,I) \quad (13)$$

i.e., the total fluctuation originates practically solely from the uncertainties in the measurements of one party only, the inspector in this example. In such a case,  $u(r,O)$  and  $u(s,O)$  must be derived from a comparison with inspector's measurements also obtained by DA.

In the IAEA data analysis, various statistical techniques[56] are used to derive separate estimates of the operator's and inspector's uncertainty parameters based on the collection of historical operator-inspector differences. The results of these evaluations are "Performance Values" obtained for each MBA/stratum/measurement method combination. These Performance Values are generally updated once a year as more historical data becomes available for DA and NDA. A similar approach is also applied by other Safeguards organizations<sup>[60-63]</sup>.

Annex 2 describes how the Performance Values are used in planning inspections<sup>[61-64]</sup> and in drawing inferences based on the declared values of the operator and on the measured values of the inspector. There are, however, situations where insufficient historical data is available to derive Performance Values. In these instances ITVs are used until sufficient measurement history is accumulated.

Conversely the most recent and best Performance Values may be used to justify a revision of the ITVs. The relationship between the ITVs and the Performance Values is explained in Section 6 and Figure 2.

## 4. Results of Laboratory Intercomparisons

Laboratory intercomparisons also offer a documented set of relevant experimental data for defining Target Values. The most useful information stems from experiments,

where the participants analyze very well characterized materials or measure well known volumes or masses of nuclear materials in industrial tanks or containers, and where their results are directly compared to the certified composition of the materials or to the certified value of the respective quantities. Permanent or periodic measurement evaluation programmes have a greater value for our present purpose than one-shot intercomparison experiments, because the participants tend to follow more closely their routine measurement procedure when the intercomparison samples are submitted sufficiently frequently.

The Institute for Reference Materials and Measurements (IRMM, Geel), the Commission d'Etablissement des Méthodes d'Analyse (CETAMA) of the Commissariat à l'Energie Atomique (CEA, France) and the New Brunswick Laboratory at Argonne (NBL, USA) administer such programmes in the area of nuclear material measurements. The discussion of the present edition of the ITVs made extensive reference to the reports published on the results obtained in the Regular European Interlaboratory Measurement Evaluation Programme (REIMEP)<sup>[35-41]</sup>, the programme of Evaluation de la Qualité des Résultats d'Analyses dans l'Industrie Nucléaire (EQRAIN)<sup>[34,42-44]</sup>, and the Safeguards Measurement Evaluation Programme (SME)<sup>[45,46,65]</sup> run respectively by these three organizations. The calorimetry Exchange Programme of the Mound Laboratory<sup>[47,48]</sup> and the Waste Drum Measurement Evaluation Programme of NBL are examples of too rare NDA measurement evaluation programmes. Unfortunately also, there exists still no permanent measurement evaluation programme regarding bulk measurements and the quality of sampling procedures.

Mass measurements are rather straightforward, so that actual inspection data probably provide sufficiently reliable estimates of their uncertainties. The measurement of volumes of solutions in industrial tanks using pneumatic level indicators is a more complex procedure and has been the object of several scientific experiments with international participation. The results of these experiments have been reported<sup>[66-69]</sup> and were used in the discussion of the relevant Target Values. The uncertainties to be expected in the use of tracer techniques for volume measurements have been evaluated in the same or similar experiments<sup>[67,70-73]</sup>.

There are numerous references of interest regarding one-shot intercomparisons of the quality of elemental and isotopic assays by DA<sup>[29-33,74-76]</sup>, as well as extensive intercomparisons of non destructive measurements by gamma spectrometry<sup>[27,28,31,39]</sup>.

The evaluations of such one-shot experiments are usually much more elaborate than those of actual inspection data or those of permanent measurement evaluation programmes. They provide, therefore, a better insight into the structure of the sources of measurement uncertainties.

A frequent drawback of interlaboratory comparisons is that they too rarely involve the measurements of actual industrial materials under industrial conditions. The report of the cooperative certification of working reference materials of plutonium and uranium oxides for NDA constitute exceptions<sup>[77-79]</sup>.

The ITVs 2000 were defined to be consistent with the standard uncertainties observed in the most recent interlaboratory comparisons and measurement evaluation pro-

grammes involving the use of current technologies, with due consideration to the comments made above.

## 5. Results of Method Validations and Quality Control Measurements

The experimental validation of measurement methods has become a standard practice for metrological and analytical laboratories required by most quality systems. Consolidated guidelines for the standardized performance of such studies are just emerging, however<sup>[52, 80]</sup>. This sometimes makes it difficult to compare the respective results obtained by different laboratories. The most trustworthy studies of this type are certainly those which identify the basic metrological parameters of the measurement process, estimate the contributions of the uncertainties occurring in these elementary steps, and compare the expected performance with the results of actual measurements of well-known amounts of materials<sup>[17-26, 81-91]</sup>. When the uncertainty propagation model yields larger values than the experimental determinations of the total uncertainty, it is probable that the design of the experiments failed to include one or several sources of potential uncertainties. These cases must receive specific attention. The ESARDA/WGND has undertaken a comprehensive examination regarding the propagation of the uncertainties in NDA measurements for safeguards, and in general to the Quality Control and Quality Assurance aspects of NDA measurements. Workshops<sup>[52, 53]</sup>, discussed QC and QA in the whole process of NDA assay, starting with the instrument design, down to the use in laboratory conditions, in verification measurement, preparation and use of RMs, traceability, qualification and certification of the measurement results.

The reports on the developments of isotope dilution mass spectrometric assay of spent fuel solutions using Large Size Dried (LSD) Spikes<sup>[92]</sup>, metal spike<sup>[93]</sup>, internal standard<sup>[94]</sup> and total evaporation techniques (TET)<sup>[95]</sup> were considered with a particular interest because the analyses of spent fuel dissolver solutions at large reprocessing plants should be of the highest possible accuracy.

The EURACHEM document<sup>[14]</sup> contains model cases for the uncertainty propagation for various types of analytical techniques. The IAEA is preparing a technical report on the propagation of uncertainties in radiochemical measurements and nuclear material analyses<sup>[96]</sup>. The latter report includes examples dealing with uranium and plutonium assays of spent fuel solutions by isotope dilution mass spectrometry (IDMS),  $\alpha$ -spectrometry and X-ray fluorescence analysis (XRFA). These documents describe how the elementary sources of uncertainties in each step of the assay are identified. This leads to the expression of the final result,  $y$ , as a function of the elementary parameters of the assay,  $x_i$ :

$$y = f(\{x_i\}) \quad (14)$$

The combined uncertainty<sup>[12-14]</sup> can be derived according to equation (15) when the  $\{x_i\}$  are actually independent variables:

$$u_c^2 = \sum_i c_i^2 \cdot u_i^2 \quad (15)$$

where  $u_c$  is the combined standard uncertainty for the assay result  $y$   
 $u_i$  the standard uncertainty for parameter  $x_i$

$c_i$  a coefficient of sensitivity defined in equation (16) below.

$$c_i = (\delta y / \delta x_i) \quad (16)$$

Quality control measurements carried out in parallel with the assay of actual samples are a particularly relevant and convenient source of information. These measurements follow as far as possible the same process than the one applied to the samples. The control materials used in these measurements are preferably characterized or certified materials with well documented traceability to the International System (IS) of Measurements. "Type A"<sup>[12]</sup> estimates of several standard uncertainties  $u_i$  can usually be derived from a variance component analysis of the results of the quality control measurements collected over a sufficiently long period of time. Physical data, certificates of reference materials, weights, physical standards and instruments, such as balances, provide "Type B"<sup>[12]</sup> estimates of the other standard uncertainties. This approach is meanwhile applied at several laboratories and has yielded comparative assessments of the respective uncertainties of major nuclear analytical techniques, such as mass spectrometry, IDMS<sup>[22, 97]</sup>, radiometry (HRGS, GS,  $\alpha$ -spectrometry), and the potentiometric titration of uranium and plutonium.

The standard uncertainties obtained from the above studies are grouped in two categories:

- The standard uncertainties of purely random character,  $u(r)_i$ ,
- The standard uncertainties of systematic character,  $u(s)_i$ .

Equation (15), applied to the random uncertainties,  $u(r)_i$ , provides an estimate the combined standard uncertainty of the random effects,  $u_c(r)$ . The combined standard uncertainty of systematic character,  $u_c(s)$ , is calculated similarly by applying equation (15) to combine the contributions of the standard uncertainties  $u(s)_i$ .

Such studies establish the necessary quantification of the traceability of nuclear material analyses, and constitute an essential source of information for the selection of the ITVs 2000.

## 6. Meaning of International Target Values 2000 for Uncertainty Components

The International Target Values 2000 for Measurement Uncertainties (ITVs 2000) are values for uncertainties associated with a single determination result; e.g., this may be the result reported by one laboratory on one sample (independent of the analytical scheme applied internally in the laboratory), or the result of an NDA measurement performed on a single item. The ITVs 2000 take into account actual practical experiences and should be achievable today under the conditions normally encountered in typical industrial laboratories or during safeguards inspections.

The ITVs 2000 were selected on the basis of a critical discussion of the inspectorates' performance evaluations of actual historical data and their comparison with the 1993 ITVs. They are also chosen to be consistent with uncertainty assessments provided by:

- experimental validation of measurement methods and instrumentation,
- interlaboratory measurement evaluation programmes, or
- individual laboratories.

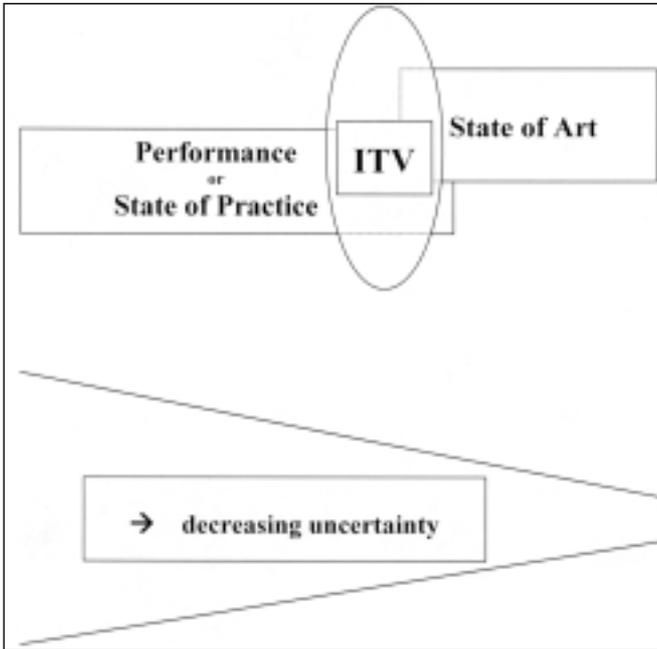


Figure 2: Conceptual Relationship between Performance, State of Art and International Target Values

The ITVs 2000 are applicable to the accountancy data collected by the inspectorates. They do not represent the ultimately achievable performance of a measurement system, which would be obtained under exceptional or ideal laboratory conditions. However, they reflect reasonably well the progress observed during the past several years in the routine performance of measurements done for the purpose of material accountancy and verification.

Figure 2 visualizes the conceptual relationship between Performance Values and ITVs. Performance Values are described by a range of values of the parameters measuring the uncertainties observed during actual industrial operations and safeguards inspections. This range is sometimes said to represent the State-of-the-Practice. The uncertainties achieved under "ideal" conditions by research laboratories or laboratories producing and certifying primary reference materials can be represented by another range of values which may be taken to illustrate the State-of-the-Art in analytical measurements. At a given time, the two ranges of values can overlap to various degrees depending upon the nature of the measurement and the spread of analytical technology advances at that time. The ITV for a given type of measurement is a single value, which has been selected to be a goal of acceptable level, achievable in practice.

The ITVs 2000 intend to take also into account all sources of measurement uncertainties, including sources which may not be apparent in Performance Values resulting from paired comparisons of operator's and inspector's measurements.

## 7. Structure and Content of the ITVs 2000

The presentation of the 1993 ITVs involved 16 different tables. A different format was chosen for the presentation of the ITVs 2000, which include only 7 tables.

- Table 1 provides a list of the codes used to identify the

measurement methods in Tables 2 to 7. The methods used by the IAEA are described briefly in Reference [98].

- Tables 2 to 6 list the ITVs 2000 for bulk and density measurements, sampling, the determination of element concentration, of <sup>235</sup>U isotope abundance, and of plutonium isotope ratios, respectively.
- ITVs for total amount of fissile element or isotope are given in Table 7 for NDA techniques providing a direct measurement.
- Each table identifies separate ITVs according to the type of material and measurement method, as appropriate.
- Two parameters, **u(r)** and **u(s)**, characterize the quality, which should be aimed for in a specific measurement of a given material using a specified method at a single laboratory; **u(r)** and **u(s)** are specific subsets of the combined standard uncertainty comprising all uncertainties arising from random effects and systematic effects, respectively, according to the description in chapter 3.

These parameters should include all uncertainty components, which determine the potential difference between the measured and the true value. For example, the values specified for the element and isotope concentration measurements include all uncertainties generated in steps 3 to 6 of Figure 1 as well as the uncertainties of the calibration measurements, and the uncertainties of the reference data and materials used for the calibration.

- It has not yet been possible to propose ITVs for the term **u(s)** applicable to sampling, except in a few cases, where this parameter was found to be actually measurable. It should also be noted that random sampling errors were frequently not assessed on the basis of experimental data (due to lack of such) and are based on expert opinion and facility experience.

- The combination of the **u(r)** and **u(s)** parameters

$$u_c(t) = [u(r)^2 + u(s)^2]^{1/2} \quad (17)$$

is equivalent to the relative combined standard uncertainty of the measurement, as it is defined in the ISO<sup>[12]</sup>, NIST<sup>[13]</sup> and EURACHEM<sup>[14]</sup> Guides, when it is applied to the measurement of a single laboratory.

- The ITVs in Tables 2 to 7 apply to situations where the measured quantity is large enough so that the relative uncertainty of the measurement remains essentially constant for the given range of measurements.
- The **u(r)** and **u(s)** parameters of bulk measurements, sampling, element concentration and isotope abundance measurements from Tables 2 to 6 must be combined according to equations (18) and (19), in order to obtain the ITVs, **u<sub>c</sub>(r)** and **u<sub>c</sub>(s)**, applicable to analytical data resulting from a given combination of several measurement steps.

$$u_c(r)^2 = \sum_l u_l(r)^2 \quad (18)$$

$$u_c(s)^2 = \sum_l u_l(s)^2 \quad (19)$$

where *l* refers to an individual step of the analytical process,

and  $l = 1, 2, \dots, n$

Examples of such calculations are given in Chapter 8.

Table 1: Measurement Method Codes

Method /Instrument Code	Technique
ANCC	Advanced Neutron Coincidence Counter
AWCC	Active Well Coincidence Counter
CALR	Calorimeter
COMP	Combined Product Uranium Concentration and Enrichment Assay (COMPUCEA)
DIPT	Dip Tube
EBAL	Electronic Balance
FRSC	Fuel Rod Scanner
GRAV	Gravimetry
GSMS	Gas Source Mass Spectrometry
HKED	Hybrid K-Edge/K-XRF Densitometer
HLNC	High Level Neutron Coincidence Counter
HRGS	Infield High Resolution Gamma Spectrometer
IDMS	Isotope Dilution Mass Spectrometry
INVS	Inventory Sample Coincidence Counter
KED	K-Edge Densitometer
LCBS	Load-Cell Based Weighing System
LMCA	Laboratory Multichannel Analyzer/Hi-resolution GS
LMCN	Laboratory Multichannel Analyzer, NaI-detector
PCAS	Plutonium Canister Assay System
PHON	Photon Neutron Interrogation Device
PMCG	Portable Multichannel Analyzer, GeLi-detector
PMCN	Portable Multichannel Analyzer, NaI-detector
PSMC	Plutonium Scrap Multiplicity Counter
TIMS	Thermal Ionization Mass Spectrometry
TITR	Titration
UNCL	Uranium Neutron Coincidence Collar
VTDM	Vibrating Tube Density Meter
WDAS	Waste Drum Assay System

**Note:** Measurement codes for NDA instruments correspond to the codes adopted in the IAEA Safeguards Manual<sup>[39]</sup>

Table 2: Bulk &amp; Density Measurements

Measurement	Instrument	Uncertainty Component (% rel. Std. Uncertainty)	
		u(r)	u(s)
		Mass	LCBS
	EBAL	0,05	0,05
Volume <sup>1/</sup>	DIPT	0,30	0,20
Density	DIPT	0,30	0,20
	VTDM	<0.05	<0.05

1.) Volume determinations are made on the basis of level pressure, density and temperature measurements. The volume measurement uncertainties are highly dependent on the homogeneity of the liquid, the quality of the density measurements and of the calibration equation determined in the calibration process. The volume measurements may also involve an absolute error component which has to be taken into consideration when determining the overall uncertainty of volume measurements. For accountability tanks in large-throughput facilities, uncertainties of 0.05% for u(r) and 0.1% for u(s) at full volume are achievable if: i.) A carefully designed calibration procedure has been implemented under well-controlled environmental and stable temperature conditions; and ii.) Measurements are performed on a well-characterized and homogenized liquid.

Table 3: Sampling Uncertainties for Elemental Concentration and  $^{235}\text{U}$  Abundance

Material	Uncertainty Component (% rel. Std. Uncertainty)				Recommended Minimum Sample Size <sup>5/</sup>
	Concentration		$^{235}\text{U}$ Abundance		
	u(r)	u(s) <sup>1/</sup>	u(r)	u(s) <sup>1/</sup>	
DUF <sub>6</sub>	0,10	nd	1	nd	5-10 g
HEUF <sub>6</sub> & LEUF <sub>6</sub> & NUF <sub>6</sub>	0,05	nd	0,10	nd	5-10 g
U-oxide Powder	0,20	nd	nd	nd	10-20 g
U-oxide Pellets	< 0.05 <sup>2/</sup>	< 0.05	< 0.05	< 0.05	1 pellet
U Scrap (clean) <sup>3/</sup>	1	nd	1	nd	30 g
U Scrap (dirty) <sup>4/</sup>	10	nd	10	nd	2 x 30 g
Reprocessing Input Sol.	0,30	0,20	< 0.05	nd	2 x 1 ml
U Nitrate Sol.	0,10	nd	< 0.05	nd	10 ml
Pu, U/Pu Nitrate Sol.	0,20	nd	< 0.05	nd	10 ml
Pu-oxide	0,10	nd			2 x 1 g
FBR & LWR MOX	0.70(Pu) 0.20(U)	nd	0,10	nd	2 x 1 pellet or 2 x 2 g (FBR MOX) or 2 x 5 g (LWR MOX)
MOX Scrap(clean) <sup>3/</sup>	1	nd	1	nd	2 x 5 g
MOX Scrap(dirty) <sup>4/</sup>	10	nd	10	nd	2 x 10 g
U Metal	0,05	nd	< 0.05	nd	1-5 g
HEU Alloys	0,20	nd	< 0.05	nd	5-10 g

- 1.) Missing values (nd) have not yet been defined.
- 2.) 0.20 for Gadolinium-containing pellets.
- 3.) Scrap with low impurity content and suitable for direct recycling.
- 4.) Sampling errors can vary widely depending on material heterogeneity and sample size.
- 5.) According to STR-69<sup>[100]</sup>

Table 4: Element Concentration

Method	Material	Uncertainty Component (% rel. Std.Uncertainty)				Notes <sup>1/</sup>
		U-Conc.		Pu-Conc.		
		u(r)	u(s)	u(r)	u(s)	
GRAV	U Oxides(pure),UF <sub>6</sub>	0.05	0.05			2/
	Pu Oxide			0.05	0.05	2/
TITR	U Oxides,UNH,UF <sub>6</sub>	0.1	0.1			
	U Alloys	0.2	0.2			
	Pu Oxide, Pu Nit.			0.15	0.15	3/
	MOX, U/Pu Nit.	0.1	0.1	0.2	0.2	3/
IDMS	U & Pu Compounds					4/5/
	Hot Cell Conditions Glove Box Conditions	0.2 0.15	0.2 0.1	0.2 0.15	0.2 0.1	
KED	U in solution	0.2	0.15			6/
	Pu in solution			0.2	0.15	6/7/
	FBR MOX			0.3	0.2	6/
HKED	Spent Fuel Solution, LWR MOX	0.2	0.15	0.6	0.3	8/
COMP	U Compounds	0.2	0.15			2/6/9/
ANCC	Pu Oxide, MOX			0.2	0.2	10/
INVS	Pu Oxide, MOX			2	1.5	11/12/
	MOX Scrap			10	2.5	11/

- 1.) Concentration measurements on powders and solutions require weight change correction because of sample instability.
- 2.) Material containing non-volatile impurities < 1000 ppm
- 3.) Equivalent performance may be expected when applying coulometry
- 4.) Materials typically encountered in the nuclear fuel cycle
- 5.) Under conditions of sufficiently different isotopic compositions of spike and sample and near-optimum sample:spike ratio[83,96,97]
- 6.) Measurement time 1000 sec., adjusted for age of source when necessary
- 7.) For samples in solution with >50 g/l Pu
- 8.) 150 g/l U, 1.5 g/l Pu
- 9.) 200 g/l U
- 10.) For: 2g sample; 4 hour counting time; isotopic determination by mass spectrometry; detector efficiency > 40%
- 11.) Measurement time 300 sec.
- 12.) Isotopic determination by mass spectrometry

Table 5:  $^{235}\text{U}$  Abundance

Method	Material	Uncertainty Component (% rel. Std. Uncertainty)		Notes
		u(r)	u(s)	
GSMS	DUF <sub>6</sub> & NUF <sub>6</sub>	0,1	0,1	
	LEUF <sub>6</sub>	0,05	0,05	
	HEUF <sub>6</sub>	0,02	0,02	
TIMS	DU (< 0.3 wt.% $^{235}\text{U}$ )	0,5	0,5	
	U (0.3% < $^{235}\text{U}$ < 1%)	0,2	0,2	
	LEU (1% < $^{235}\text{U}$ < 20%)	0,1	0,1	
	HEU (> 20 wt.% $^{235}\text{U}$ )	0,05	0,05	
COMP	LEU Compounds	0,4	0,2	1/
LMCN <sup>2/</sup>	LEU Oxides	0,3	0,3	
	HEU Oxides	0,2	0,2	
PMCN <sup>2/3/</sup>	DUF <sub>6</sub>	20	15	4/
	NUF <sub>6</sub>	10	8	4/
	LEUF <sub>6</sub>	5	3	4/
	NU Oxides	5	5	
	LEU Oxides	3	2	
	NU & LEU Scrap (clean) <sup>5/</sup>	5	5	6/
	NU & LEU Scrap (dirty)	15	10	6/
	LEU Fuel Rods	2,5	1	
	LEU Fuel Assemblies	2,5	1	
	HEU Metal	0,5	0,5	7/
	HEU Alloys	1	1	7/
PMCG <sup>3/</sup>	DUF <sub>6</sub>	15	10	4/
	NUF <sub>6</sub>	8	5	4/
	LEUF <sub>6</sub>	4	2	4/
	LEU Oxides	3	2	
	HEU Metal	0,5	0,5	7/
	HEU Alloys	1	1	7/

1.) Measurement time 1000 sec., adjusted for age of source when necessary; see Ref. [21]

2.) For materials not containing reprocessed uranium.

3.) Measurement time 300 sec.

4.) Includes uncertainty component associated with ultrasonic thickness gauge measurement of the UF6 cylinder.

5.) Scrap with low impurity content and suitable for direct recycling.

6.) Uncertainties for scrap represent average performance observed on historical data. Material matrix heterogeneity is the main contributor to the observed uncertainties and can vary widely.

7.) Calibration against reference material certified to 0.3 % or better & uncertainties in the correction of container wall absorption of 0.5 % or less.

**Table 6: Plutonium Isotope Assay  
of Pu Oxide and MOX**  
(% Relative Standard Uncertainties)

Material Type	Isotope Ratio	Typical Value for Ratio (*100)	Method					
			TIMS <sup>1/</sup>		HRGS <sup>2/</sup>		LMCA <sup>3/</sup>	
			u(r)	u(s)	u(r)	u(s)	u(r)	u(s)
High-Burnup Pu	<sup>238</sup> Pu/ <sup>239</sup> Pu	1,7	1,5	1	2	2	1	1
	<sup>240</sup> Pu/ <sup>239</sup> Pu	43	0,1	0,05	1	1	0,7	0,7
	<sup>241</sup> Pu/ <sup>239</sup> Pu	13	0,2	0,2	1	1	0,7	0,7
	<sup>242</sup> Pu/ <sup>239</sup> Pu	8	0,2	0,3				
Low-Burnup Pu	<sup>238</sup> Pu/ <sup>239</sup> Pu	0,02	10	10	10	10	5	5
	<sup>240</sup> Pu/ <sup>239</sup> Pu	6	0,15	0,1	2	2	1,5	1,5
	<sup>241</sup> Pu/ <sup>239</sup> Pu	0,2	1	1	2	2	1	1
	<sup>242</sup> Pu/ <sup>239</sup> Pu	0,05	2	2				

- 1.) <sup>238</sup>Pu/<sup>239</sup>Pu by alpha spec./TIMS combination
- 2.) Measurement time 3 x 100 sec.
- 3.) Measurement time 3 x 1000 sec.; 0.5 g Pu.

Table 7: Total Mass -  $^{235}\text{U}$  & Pu  
by Direct NDA Measurement Techniques

Instrument	Material	Uncertainty Component (% rel. Std.Dev.)				Notes
		$^{235}\text{U}$ Mass		Pu Mass		
		u(r)	u(s)	u(r)	u(s)	
AWCC	HEU Metal, HEU Alloys	5	3			1/
	HEU Fuel Elements	3	2			1/
FRSC	U Fuel Rods	1	1			
PHON	LEU Oxides	2	1			
	LEU Scrap	4	1			
UNCL	U Fuel Assemblies	4	2			
HLNC	Pu Oxide Powder			1	0.5	2/3/
	FBR MOX (> 10% Pu)			2	0.5	2/3/
	LWR MOX (< 10 % Pu)			4	1.5	2/3/
	MOX Scrap			10	3	2/4/
	Pu Fuel Rods			1.5	1	2/3/
	MOX Fuel Rods			2	1	2/3/
	MOX Fuel Assemblies			1.5	1	2/3/
PCAS	FBR MOX			1.5	1	3/
	MOX Scrap			8	2	4/
PSMC	MOX Scrap (clean)			2.5	1	3/5/
	MOX Scrap (dirty)			8	2	4/
WDAS	MOX Waste			8	2	4/
CALR	Pu Oxide and MOX			0.4	0.4	3/6/7/

- 1.) Measurement time 600 sec.
- 2.) Measurement time 300 sec.
- 3.) Isotopic determination by mass spectrometry and alpha spectrometry.
- 4.) Uncertainties for scrap represent average performance observed on historical data. Material matrix heterogeneity is the main contributor to the observed uncertainties and can vary widely.
- 5.) Scrap with low impurity content and suitable for direct recycling
- 6.)  $^{241}\text{Am}$  content determined by gamma spectrometry or alpha spectrometry
- 7.) Lower uncertainties are achievable for materials containing low burn up Pu

## 8. Use of ITVs

ITVs are considered to be achievable in routine measurements involved in the determination of the amount of nuclear materials for materials accountancy and safeguards verification purposes. They are intended to be used as a reference by plant operators, state systems and international safeguards organizations. They should, however, not be normally used in place of values based on actual measurements in estimating the statistical significance of operator-inspector differences or MUF. Analytical laboratories can find it useful to determine experimentally the actual uncertainties of their measurements, and to compare them with the corresponding values, which can be derived from the ITVs 2000.

Safeguards authorities regularly compare the performance values with the current ITVs. They will examine with the relevant authorities and laboratories means of improving the performance, in cases where the performance values are significantly higher than the ITVs, and too high to allow the IAEA to meet its detection goals[101]. When reliable performance values are not available, ITVs may be used instead to calculate sampling plans, to set reject limits and to calculate estimates of the combined uncertainties of inventories, throughputs, MUF and D's, as described in Annex 2.

Such applications of the ITVs require having a good insight of the measurement and verification systems. It is in particular important to recognize that, because of practical constraints, some measurement steps may be common to the operator and the inspector. It should also not be forgotten that the operator-inspector differences can carry errors which are not related to measurement uncertainties.

The following three examples illustrate how the tabulated ITVs can be used to calculate ITVs for combined uncertainties applicable to practical situations. Further examples are presented in Reference [102].

### Example 1:

#### Target Values for the Determination of the Total Mass of Fissile Element on Independent Samples

Consider a situation where the operator and the inspector determine fully independently the total amount of plutonium in a batch of LWR pellets. The operator measures the plutonium concentration by titration on ten randomly

selected pellets, the inspector by IDMS on an independently selected but single pellet.

The Target Values for the combined relative standard uncertainties applicable to the determination of the total mass of plutonium by the operator are derived from the following equations, respectively for the random errors, the errors of systematic character and their combination:

$$u_c(r, O) = \sqrt{\sum_i u_i^2(r, O)/n_i(O)} \quad (20)$$

$$u_c(s, O) = \sqrt{\sum_i u_i^2(s, O)} \quad (21)$$

$$u_c(O) = \sqrt{[u_c^2(r, O) + u_c^2(s, O)]} = \sqrt{(0.0555 + 0.0425)} = 0.31\% \quad (22)$$

The above values would be used in the calculation of Target Values for the relative standard uncertainties to be expected in the inventory, throughput and MUF declared by the operator.

Similar equations are used to calculate the corresponding values applicable to inspector's measurements,  $u_c(r, I)$ ,  $u_c(s, I)$  and  $u_c(I)$ . The Target Value for the combined uncertainties on the total Pu mass measured by the inspector is equal to:

$$u_c(I) = \sqrt{[u_c^2(r, I) + u_c^2(s, I)]} = \sqrt{(0.5150 + 0.0125)} = 0.73\% \quad (23)$$

Its magnitude is determined essentially by the random sampling uncertainty component. This is also true for the Target Value applicable to the Operator-Inspector difference:

$$u_d = \sqrt{[u_d^2(r) + u_d^2(s)]} = \sqrt{(0.5705 + 0.0550)} = 0.79\% \quad (24)$$

Assuming that the values of Target Values,  $u_c$ 's, given in Table 8 and equations (22), (23) and (24), are effectively achieved, the 95% confidence intervals of the final results of the operator, of the inspector and of their difference, would be respectively equal to:

$$CL(O) = k u_c(O) = 2 \times 0.31 = 0.62\% \quad (25)$$

$$CL(I) = k u_c(I) = 2 \times 0.73 = 1.46\% \quad (26)$$

$$CL(d) = k u_c(d) = 2 \times 0.79 = 1.58\% \quad (27),$$

where the coverage factor  $k$  is 2.

Table 8: Target Values for Total Pu Mass with Independent Samples and DA (Example 1)

	Step	Method Instr.	$n_i$	ITV (% rel. Std. Dev.)			Variance Component		
				$u_i(r)$	$u_i(s)$	Table	$u_i^2(r)/n_i$	$u_i^2(s)$	
O P E R A T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		10	0,70	nd	3	0,0490		
	6- Pu-Conc.	TITR	10	0,20	0,20	4	0,0040	0,0400	
	Sum of variance components							0,0555	0,0425
	Combined Std. Uncertainties, $u_c(r,O)$ and $u_c(s,O)$ , (in % rel.)							0,24	0,21
I N S P E C T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		1	0,70	nd	3	0,4900		
	6- Pu-Conc.	IDMS	1	0,15	0,10	4	0,0225	0,0100	
	Sum of variance components							0,5150	0,0125
	Combined Std. Uncertainties, $u_c(r,I)$ and $u_c(s,I)$ , (in % rel.)							0,72	0,11
D I F F	Variance of Rel. Operator-Inspector Difference							0,5705	0,0550
	Standard Uncertainties of Rel. Diff., $u_d(r)$ and $u_d(s)$ , (in %)							0,76	0,23

**Example 2:  
Target Values for the Determination of the  
Total Mass of Fissile Element on a Common  
Sample.**

In situations where the inspector analyzes a subsample of a homogeneous operator's sample, the sampling errors no longer contribute to the uncertainty of the Operator-Inspector difference. An example of this situation could be

a co-operative effort to identify the existence of biases in the chemical analysis.

Apply these conditions to the first example. In this case, as shown in Table 9, the Target Value for the Operator-Inspector difference and its 95% confidence interval will be:

$$u_d = \sqrt{(0.0675 + 0.0550)} = 0.35 = \% \tag{28}$$

$$CL(d) = k u_d = 2 \times 0.35 = 0.70\% \tag{29}$$

**Table 9: Target Values for Operator-Inspector Difference on Total Pu Mass, with Common Sample and DA (Example 2)**

	Step	Method Instr.	n <sub>i</sub>	ITV (% rel. Std. Dev.)			Variance Component		
				u <sub>i</sub> (r)	u <sub>i</sub> (s)	Table	u <sub>i</sub> <sup>2</sup> (r)/n <sub>i</sub>	u <sub>i</sub> <sup>2</sup> (s)	
OPERATOR	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		1	0,70	nd	3			
	6- Pu-Conc.	TITR	1	0,20	0,20	4	0,0400	0,0400	
	Sum of variance components contributing to Op-In Differ.							0,0425	0,0425
INSPECTOR	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		1	0,70	nd	3			
	6- Pu-Conc.	IDMS	1	0,15	0,10	4	0,0225	0,0100	
	Sum of variance components contributing to Op-In Differ.							0,0250	0,0125
DIFF	Variance components of relative difference							0,0675	0,0550
	Standard Uncertainties of Rel. Diff., u <sub>d</sub> (r) and u <sub>d</sub> (s), (in %)							0,26	0,23

**Example 3:  
Estimation of the Uncertainty of  
Operator-Inspector Differences for NDA  
Sampling Plan Calculations**

Consider a situation where an inspector must calculate a sample size for verifying the content of LEUF<sub>6</sub> containers using a PMCN. The operator declarations for the material are based on DA measurements of <sup>235</sup>U abundance and the stoichiometric value for U-concentration in UF<sub>6</sub>. No historical inspector measurement data is available. Therefore ITVs need to be used to provide an estimate of the uncertainty which may be associated with the operator-inspector difference.

The variance components calculated from the ITVs 2000 are given in Table 10. The standard combined uncertainty associated with the operator-inspector difference in this example is equal to:

$$u_d = \sqrt{u_d^2(r) + u_d^2(s)} = \sqrt{(25.0175 + 9.0075)} = 5.83 \% \quad (30)$$

In the absence of an uncertainty estimate based on historical measurement data, the inspector would thus use the above value calculated from the ITVs for performing sample size calculations and establishing rejection limits. In this example, the relatively large uncertainty associated with the NDA measurement almost entirely determines the overall uncertainty of the operator-inspector difference.

**Table 10: Target Values for Operator-Inspector difference  
(Example 3)**

	Step	Method/Instr.	ITV (% rel. Std. Dev.)			Variance Component		
			u <sub>d</sub> (r)	u <sub>d</sub> (s)	Table	u <sub>d</sub> <sup>2</sup> (r)	u <sub>d</sub> <sup>2</sup> (s)	
O P E R A T O R	1- Bulk	EBAL	0,05	0,05	2	0,0025	0,0025	
	2- Sampling <sup>235</sup> U wt. %		0,1		3	0,0100		
	6- U-Conc.	Stoichiom. Val.						
	6- <sup>235</sup> U wt. %	GSMS	0,05	0,05	5	0,0025	0,0025	
	Sum of variance components						0,0150	0,0050
I N S P E C T O R	1- Bulk	EBAL	0,05	0,05	2	0,0025	0,0025	
	6- U-Conc.	Stoichiom. Val.						
	6- <sup>235</sup> U wt. %	PMCN	5	3	5	25,0000	9,0000	
	Sum of variance components						25,0025	9,0025
D I F F	Variance components of relative difference						25,0175	9,0075
	Standard Uncertainties of Rel. Diff., u <sub>d</sub> (r) and u <sub>d</sub> (s), (in %)						5,00	3,00

## 9. Future Developments

It is intended to keep updating the ITV tables regularly in order to incorporate the latest relevant information. The following activities will be especially important for this purpose:

- Growing emphasis is being placed on reassessing the uncertainties of chemical measurements according to the ISO<sup>[12]</sup>, NIST<sup>[13]</sup> and EURACHEM<sup>[14]</sup> guides. This should be done systematically for the methods in current use. It should become a part of the process of qualification of new measurement methods and instrumentation.
- The inspectorates will continue to update actual performance evaluations.
- It is important that interlaboratory measurement evaluation programmes continue to be conducted, particularly in the area of Pu measurements. Operator and inspector laboratories should participate in such programmes. Their results should be published as it was done in the past.
- Models more specific to the NDA measurement processes are being reviewed by the ESARDA/NDA Working Group to monitor and assess the sources of major uncertainties in actual inspectors' measurements. This will hopefully involve uncertainty assessments in line with the above guides as well as periodical estimates of actual Performance Values and the development of interlaboratory measurement evaluation programmes for NDA.
- Results of experimental qualifications of recommended sampling procedures<sup>[103-112]</sup> should be made available to the inspectorates to substantiate and expand ITVs for the uncertainty components in sampling procedures.
- The IAEA will also follow with the greatest interest developments in bulk measurements and elemental assays of spent fuel solutions and their impact on the accuracy of the accountability of large throughputs and inventories of nuclear materials at large plants now coming under safeguards.

The IAEA will continue its cooperation on the above topics with Euratom, with State authorities and with the expert groups, which were involved in the review of the ITVs 2000. The next revision of the ITVs will also be another opportunity to seek further contributions from more countries and organizations.

### Annex 1 Statistical Terminology

The statistical terminology used in this document is given here in an effort to promote better understanding of the statistical concepts discussed herein. The terminology is divided into four groups as follows:

#### Group 1: Basic terms

(true value, conventional true value, measurand, measurement, measurement result,

measurement error, uncertainty, uncertainty component, expectation, expected value, mean, variance, standard deviation, sample standard deviation, experimental standard deviation, error parameter, sample, estimation, statistic, estimator, estimate).

#### Group 2: Selected sources and classes of error

(bulk measurement error, sampling error, random error of result, random error of measurement, systematic error of result, systematic error of measurement, calibration error, bias, relative error).

#### Group 3: Descriptors

(precision, accuracy, repeatability, repeatability conditions, reproducibility, reproducibility conditions).

#### Group 4: Safeguards specific use of terms

(random sampling, inspection by attribute, alarm level, significant difference, defect, discrepancy, detection probability, false alarm, risk).

The definitions of these terms, as used in this document, are given to the extent feasible, in a way which is consistent with the latest internationally recognized standards or manuals. The relevant source<sup>[14,54-56,114]</sup> of the definition is specified in the following tables.

## Annex 2

### Use of Performance Values for Inspection Purposes and Their Limitations

The Performance Values (see chapter 3) are used in planning inspections and in drawing inferences based on the declared values of the operator and on the measured values of the inspector<sup>[56]</sup>.

From an inspection planning viewpoint, they allow calculation of sample sizes for NDA and for DA verification methods that are optimal with respect to achieving the desired level of defect detection probability with the minimum number of samples.

When evaluating the verification data, they serve first to define item-level alarms, or reject limits, such that if a given item paired difference,  $d_{ij}$ , exceeds the limit  $L$  in absolute value, it is identified as a discrepancy, where  $L$  is defined by the equation:

$$L = z_{\alpha} \left[ u^2(r) + u^2(s) \right]^{1/2} \quad (\text{A2.1})$$

where  $z_{\alpha}$  is the normal probability distribution factor associated with the probability  $\alpha$  of declaring a false alarm. Current practice is to take  $z_{\alpha} = 3$ , which results in a false alarm probability of less than 0.3 %.

The item paired differences are calculated on either an absolute or relative basis, as was mentioned in chapter 3. Of course, for a homogeneous stratum, it makes no difference whether absolute or relative differences are calculated.

In addition to defining attribute test reject limits as just described, performance values are also used in calculating the variances used in material balance evaluations for material unaccounted for (MUF), operator-inspector difference (D), and the inspector's estimate of MUF, (MUF-D).

In a large facility the probability of detection will be driven by the amount of material. Regardless of how accurately and precisely material is measured,  $\sigma$  will be large because the amount of material is large. In such cases, the probability of detecting diversion by means of a material balance evaluation will be small and additional safeguards measures such as near real-time accountancy (NRTA) are called for.

The users of the Performance Values must remain aware of a number of limitations in their meaning or content.

Plant operational or economic constraints may inflate the variance components of the operator-inspector differences significantly compared to the capability of current measurement technology. The safeguards inspector must indeed verify that the uncertainties in the plant measurement system are not deliberately inflated in order to reduce the detection capability of the verification measurements. The latter concern increases with the throughput or material inventory of the plant. There will therefore always be a need for Target Values providing an accepted measure of the capability of current measurement technology under reasonably economic and operational conditions encountered in the industry.

Conversely, paired comparisons do not detect the measurement errors or uncertainties, which are common

to the operator and inspector. For example, if both use the same reference material for calibration, the uncertainty of the certified value of the reference material will appear as a common systematic component in both results. The common component can also be of a random nature; random sampling errors are common, for instance, when the operator and the inspector measure the same sample or separate aliquots of the same sample.

These common components do not affect the uncertainties of the differences between operator's and inspector's measurements on a single stratum. They can, however, mask a potential bias with respect to the true amount of material. Consequently the use of Performance Values can lead to underestimation of the total uncertainties in the operator's declarations or in the material balance differences over the plant. Independent measurement evidence, free from such common mode uncertainties, is hence needed.

The user of the Performance Values must also know that the estimate of the between inspection effects,  $s(\Delta)$ , becomes less precise as the random uncertainty component,  $s(\epsilon)$ , increases. When the inspector's uncertainties are large compared to the operator's values, it becomes difficult to obtain a precise estimate of the operator's uncertainties, and vice-versa. This is frequently the case when the operator's data come from DA measurements while the inspector measures by NDA. The paired comparisons can lead to an overestimation of the random uncertainties of the operator's DA measurements, and, at the same time, to a poor estimate of the between-inspection effects in the inspector's NDA results. As a further complication, estimates of these parameters will be affected when the operator's values are based in part on nominal or average values. A separate evaluation of the performance of individual measurement methods is necessary to guard against such potential problems.

GROUP 1: BASIC TERMS		
Term	Definition	Ref.
true value	Value consistent with the definition of a given particular quantity. <b>NOTES:</b> 1. This is a value that would be obtained by a perfect measurement. 2. True values are by nature indeterminate.	[55] 1.19
conventional true value	Value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose. <b>NOTE:</b> "Conventional true value" is sometimes called <i>assigned value</i> , <i>best estimate</i> of the value, <i>conventional value</i> , or <i>reference value</i> .	[55] 1.20
measurand	Particular quantity subject to measurement.	[55] 2.6
measurement	Set of operations having the object of determining a value of a quantity.	[55] 2.1
measurement result	Value attributed to a measurand, obtained by measurement <b>NOTES:</b> 1. When the term "result of a measurement" is used, it should be made clear whether it refers to: - the value indicated by the measurement instrument - the uncorrected result - the corrected result and whether several values are averaged. 2. A complete statement of the result of a measurement includes information about the uncertainty of measurement.	[55] 3.1
measurement error	Result of a measurement minus a true value of the measurand. <b>NOTES:</b> 1. Since a true value cannot be determined, in practice a conventional true value is used. 2. The quantity is sometimes called <i>absolute error</i> of measurement when it is necessary to distinguish it from <i>relative error</i> .	[55] 3.10
uncertainty (of measurement)	Parameter associated with the result of a measurement, characterizing the dispersion of the values that could reasonably be attributed to the measurand. <b>NOTE:</b> The parameter may be, for example, a standard deviation (in which case the uncertainty is also called the <i>standard uncertainty</i> ), or the width of a confidence interval.	[55] 3.9
uncertainty component	Uncertainty arising from a distinct source contributing to the overall uncertainty. <b>NOTES:</b> 1. If there is correlation between any components then this has to be taken into account by determining the covariance. 2. It is often possible to evaluate the combined effect of several components. 3. Where components whose contribution is evaluated together are correlated, there may be no additional need to take account of the correlation	[14] 2.3.1
expectation, expected value, mean	If $X$ is a continuous random variable having the probability density function $f(x)$ then the expectation (or expected value or mean), if it exists, is $\mu_x = E(X) = \int x \cdot f(x) dx$ (the integral being extended over the intervals of variation of $X$ ).	[54] 1.18
variance	$\sigma^2 = V(X) = E [X - E(X)]^2$ The variance is the expectation of the square of the <i>centred random variable</i> (i.e. a random variable the expectation of which equals zero).	[54] 1.22

GROUP 1: BASIC TERMS		
Term	Definition	Ref.
standard deviation	$\sigma = \sqrt{V(X)}$	[54] 1.23
sample standard deviation, experimental standard deviation	<p>For a series of <math>n</math> measurements of the same measurand, the quantity <math>s</math> characterizing the dispersion of the results and given by the formula:</p> $s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$ <p><math>x_i</math> being the result of the <math>i^{\text{th}}</math> measurement and <math>\bar{x}</math> being the arithmetic mean of the <math>n</math> results considered.</p> <p><b>NOTES:</b></p> <ol style="list-style-type: none"> <li>1. Considering the series of <math>n</math> measurements as a sample of a population, <math>\bar{x}</math> is an unbiased estimate of the mean <math>\mu</math>, and <math>s^2</math> is an unbiased estimate of the variance <math>\sigma^2</math> of that distribution.</li> <li>2. The expression <math>s/\sqrt{n}</math> provides an estimate of the standard deviation of the distribution of <math>\bar{x}</math> and is called the <i>experimental standard deviation of the mean</i> (it is sometimes, but incorrectly, called <i>standard error</i> or <i>standard error of the mean</i>).</li> </ol>	[55] 3.8
error parameter	<p>Synonymous for error variance.</p> <p>In general, the result of a particular measurement, <math>x_i</math>, may be modelled by</p> $x_i = \mu + \varepsilon_{1i} + \varepsilon_{2i} + \varepsilon_{3i} + \dots,$ <p>where <math>\mu</math> is the true value of the measured quantity and the <math>\varepsilon_{.i}</math> are individual errors made during the particular measurement <math>i</math> according to the various potential sources of errors 1, 2, 3, ...</p> <p>If, for example, the error <math>\varepsilon_{1i}</math> is a representation of a centred random variable <math>\varepsilon_1</math> (mean zero): <math>E(\varepsilon_1) = 0</math>, then the variance <math>E(\varepsilon_1^2) = V(\varepsilon_1) = \sigma_{\varepsilon_1}^2</math> is called an error parameter.</p> <p>If the individual sources of error (or fluctuation) are independent from each other, then the overall variance of the measurement results can be calculated by</p> $V(X) = \sigma_X^2 = \sigma_{\varepsilon_1}^2 + \sigma_{\varepsilon_2}^2 + \sigma_{\varepsilon_3}^2 + \dots = \sum (\sigma_{\varepsilon_{.}}^2)$	
sample	<ol style="list-style-type: none"> <li>1. One or more of the individual items into which a population is divided, taken with the intention to provide information on that population.</li> <li>2. A portion of material taken from a larger batch of material with the intention to be representative for that larger batch with respect to the characteristics under consideration.</li> </ol>	various
estimation	The operation of assigning, from the observations in a sample, numerical values to the parameters of a distribution chosen as the statistical model of the population from which this sample is taken.	[54] 2.49
statistic	<p>A function of the sample random variables.</p> <p><b>NOTE:</b></p> <p>A statistic, as a function of random variables, is also a random variable and as such it assumes different values from sample to sample. The value of the statistic obtained by using the observed values in this function may be used in a statistical test or as an estimate of a population parameter, such as a mean or a standard deviation.</p>	[54] 2.45
estimator	A statistic used to estimate a population parameter.	[54] 2.50
estimate	The value of the estimator obtained as a result of an estimation.	[54] 2.51

<b>GROUP 2: SELECTED SOURCES and CLASSES of ERRORS</b>		
<b>Term</b>	<b>Definition</b>	<b>Ref.</b>
bulk measurement error	The measured mass (volume) of an item minus its true mass (volume).	[55] 3.10
sampling error	The true value for the portion of material constituting the sample minus the true value for the larger batch of material for which the sample is intended to be representative.	
random error (of result)	A component of the error, which, among a number of test results for the same characteristic, varies in an unpredictable way. <b>NOTE:</b> The random error of an analytical result cannot be compensated for, but it can be usually reduced by increasing the number of observations.	[54] 3.9
random error (of measurement)	Result of a measurement minus the mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions. <b>NOTE:</b> 1. Random measurement error is equal to measurement error minus systematic measurement error. 2. Because only a finite number of measurements can be made, it is possible to determine only an estimate of random error.	[55] 3.13
systematic error (of result)	A component of the error, which, among a number of test results for the same characteristic, remains constant or varies in a predictable way. <b>NOTES:</b> 1. Systematic errors and their causes may be known or unknown. 2. Under constant measurement conditions, the systematic error is independent of the number of measurements made and therefore cannot be reduced by increasing the number of analyses.	[54] 3.10
systematic error (of measurement)	Mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions minus a true value of the measurand. <b>NOTES:</b> 1. It is important to observe the restriction "under repeatability conditions". The value of the systematic measurement error may remain constant as long as the measurement conditions remain unaltered. However it may vary, in an unpredictable manner, with the changing of the measurement conditions or the settings of the measurement system. The systematic measurement error is therefore systematic (or constant) only with respect to a given set of measurement results, while it is at the same time a random component of the error on a long term perspective. Hence the systematic measurement error possesses a probability distribution (with expectation zero) and can be represented by a random statistical variable over a sufficiently long period of time. 2. The systematic error components under consideration in the present document are all of this dual nature.	[55] 3.14
calibration error	An error associated with a given calibration. Hence, a systematic measurement error component with respect to all measurements performed with the same calibration.	[114] 7.9
bias	The difference between the expectation of the test result and an accepted reference value (conventional true value). <b>NOTE:</b> 1. Bias can also be described as the total of all long term systematic error components; i.e., those components of the error that do not vary even under reproducibility conditions. 2. Like the <i>true value</i> , bias is by nature indeterminate. 3. If the bias is estimated and corrected for, the uncertainty of the correction must still be taken into account.	[54] 3.13
relative error	The absolute error of the measurement divided by the true value of the measurand. Frequently expressed as a percentage value (i.e. multiplied by hundred).	

GROUP 3: DESCRIPTORS		
Term	Definition	Ref.
precision	The closeness of agreement between independent test results obtained under stipulated conditions. <b>NOTE:</b> 1. Quantitative measures of precision depend critically on the "stipulated conditions". Repeatability conditions and reproducibility conditions are particular sets of extreme stipulated conditions. 2. The standard deviation of the test results is one usual quantitative measure of precision. A larger standard deviation represents less precision. 3. Precision depends only on the distribution of random errors and therefore does not relate to deviations from the true value.	[54] 3.14
accuracy (of measurement)	The closeness of agreement between the result of a measurement and a true value of the measurand. <b>NOTE:</b> 1. "Accuracy" is a qualitative concept. 2. When applied to a set of test results, accuracy involves a combination of random error components and a systematic error component.	[54] 3.11 [55] 3.5
repeatability	Precision under repeatability conditions.	[54] 3.15
repeatability conditions	Conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. <b>NOTE:</b> 1. Repeatability conditions constitute one specific and extreme set of conditions. 2. Repeatability relates practically to smallest fluctuation that is reasonably achievable among independent test results.	[54] 3.16
reproducibility	Precision under reproducibility conditions	[54] 3.20
reproducibility conditions	Conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. <b>NOTE:</b> 1. Reproducibility conditions constitute another specific and extreme set of conditions. 2. Reproducibility relates practically to the largest fluctuation that is reasonably encountered among independent test results. 3. Operator - inspector differences are typically generated under reproducibility conditions.	[54] 3.21

GROUP 4: SAFEGUARDS SPECIFIC USE OF TERMS		
Term	Definition	Ref.
random sampling	<p>Random sampling is a method for taking a sample from a group of distinct items. A random sample is selected by a procedure that gives each item a fixed and determinate (usually equal) probability of selection.</p> <p><b>NOTE:</b> When applied to non-distinct things, such as samples of bulk material, the method of selection does not usually indicate probabilities of selection of the samples. In principle, when drawing samples from a larger bulk of material, its content could conceivably be divided into a large number of possible samples of a certain size; some procedure would be used to give each potential sample an equal chance of being selected. The actual sampling method is designed to achieve the same result in a practical way, so that the statistical theory based on the concept of random sampling can be applied.</p>	[56] 2.8
inspection by attributes	<p>In attributes inspection, the item inspected is classified as being either acceptable or not (i.e. a defect) on the basis of the measurement.</p> <p><b>NOTE:</b> <u>Attributes inspection has nothing to do with the quality of measurement.</u></p>	[56] 6.2
alarm level	<p>A synonym for a critical value in the terminology of testing an hypothesis.</p> <p><b>NOTE:</b> In the subject context, the alarm level is the value of an operator-inspector difference which, if exceeded in absolute value, is cause for labelling the item in question a defect during inspection by attributes.</p>	
significant difference, defect, or discrepancy	<p>Three synonyms for an operator-inspector difference that exceeds the alarm level in absolute value.</p>	[114] 8.21
detection probability	<p>The probability that an item with a true operator-inspector difference of a given amount will be declared to be a discrepancy. More generally, when referring to a safeguards index such as MUF or D, it is the probability that the index will be found to differ significantly from its hypothesised value for a given true value of the index.</p>	[114] 5.17
false alarm	<p>In attribute testing, declaring an item to be a defect when the true operator-inspector difference is zero is a false alarm. A similar definition applies to tests on MUF and <math>\hat{D} = 0</math>.</p>	[114] 5.18
risk	<p>A synonym for the probability of reaching the incorrect conclusion in hypothesis testing. Two types of risks are usually considered:</p> <ol style="list-style-type: none"> <li>1. the risk of false alarm, defined above,</li> <li>2. the risk of non-detection, associated with the failure to detect a "true" defect.</li> </ol>	

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# Control of Nuclear Material Holdup In MOX Fuel Fabrication Plants in Europe

By the members of the ESARDA MOX Working Group

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## 1. Introduction

MOX fuel has been produced in Europe for over 30 years using well-established fuel fabrication processes married with mature plutonium management techniques.

European MOX fabrication facilities have evolved from early FBR fuel plants into commercial scale LWR facilities. By the Millennium around half a million mixed oxide fuel rods had been made for thermal reactor fuel corresponding to more than 1000tHM and utilising more than 50t of plutonium. The combined capacity of the current MOX fuel fabrication plants in Europe is over 250tHM/year, all of which is subject, without discrimination, to Euratom Safeguards.

While some protagonists of the nuclear industry suggest that MOX plants are awash with nuclear material, cannot be adequately safeguarded and that material 'stuck in the plant' could conceal clandestine diversion of plutonium, the real situation in Europe is very different: nuclear operators have gone to considerable efforts to deploy effective systems for safety, security and nuclear material control and accountancy, and the safeguards authorities likewise have implemented a hierarchy of safeguards measures enabling them to safeguard MOX plants effectively.

A variety of definitions of material in-process, in-process inventory, holdup or hidden inventory can be found in the published literature (see Appendix). These definitions are not always consistent and can lead to confusion when discussing the concepts involved. This paper will introduce 'definitions' which are consistent with the way that the Euratom Safeguards Office treats nuclear material, especially with respect to access to the material at monthly and annual inventory taking. It describes how work in progress, and in particular material holdup, is controlled, measured and accounted for.

## 2. What is "holdup"?

### 2.1 A typical MOX plant

A typical MOX Fuel Fabrication facility is shown in figure 1. At the very simplest level a MOX plant can be thought of as being split into a number of zones. Some of

these zones will be storage areas, where the material is well defined and where safeguards Confinement/Surveillance (C/S) measures can be applied if required, and the remainder will be process areas. All of the material held in the process areas is considered to be in-process material. The actual in-process inventory at any one time will depend on the operational state of the plant.

Given the premise that all material not in store is defined as in-process inventory then next consider the different processes and nuclear materials which they hold.

The rod handling and fuel assembly areas of a MOX plant deal with individual items whose weight and composition have been determined earlier and are therefore dealt with at an item level. The powder and pellet areas of the MOX plant (up to the point the rods are sealed by welding) are bulk handling zones where material is subject to direct measurement by weighing and chemical analysis. It is in these areas that intrinsic measurement uncertainty lie and which determine the material accountancy performance. The ancillary processes dealing with scraps and residues recycling are also bulk handling zones.

Some nuclear materials are clearly and unambiguously identified, for example the engraved serial number on fuel rods. Some materials are in unique containers and have been weighed (and or counted) such as trays or furnace boats of MOX pellets. Therefore material which is truly holdup and "hidden" inventory only occurs in the bulk handling areas.

We can now draw up two definitions:

- **Process areas** are all areas that are not stores.
- **In-process inventory** is the material in the process areas of the plant. It includes all bulk material in process vessels, as well as material in identified containers such as pellets, or fuel pins. Part of the in-process inventory will be made up of material that is considered to be holdup or "hidden" inventory.

To distinguish between holdup and "hidden" inventory, let us have a look at a glove box.

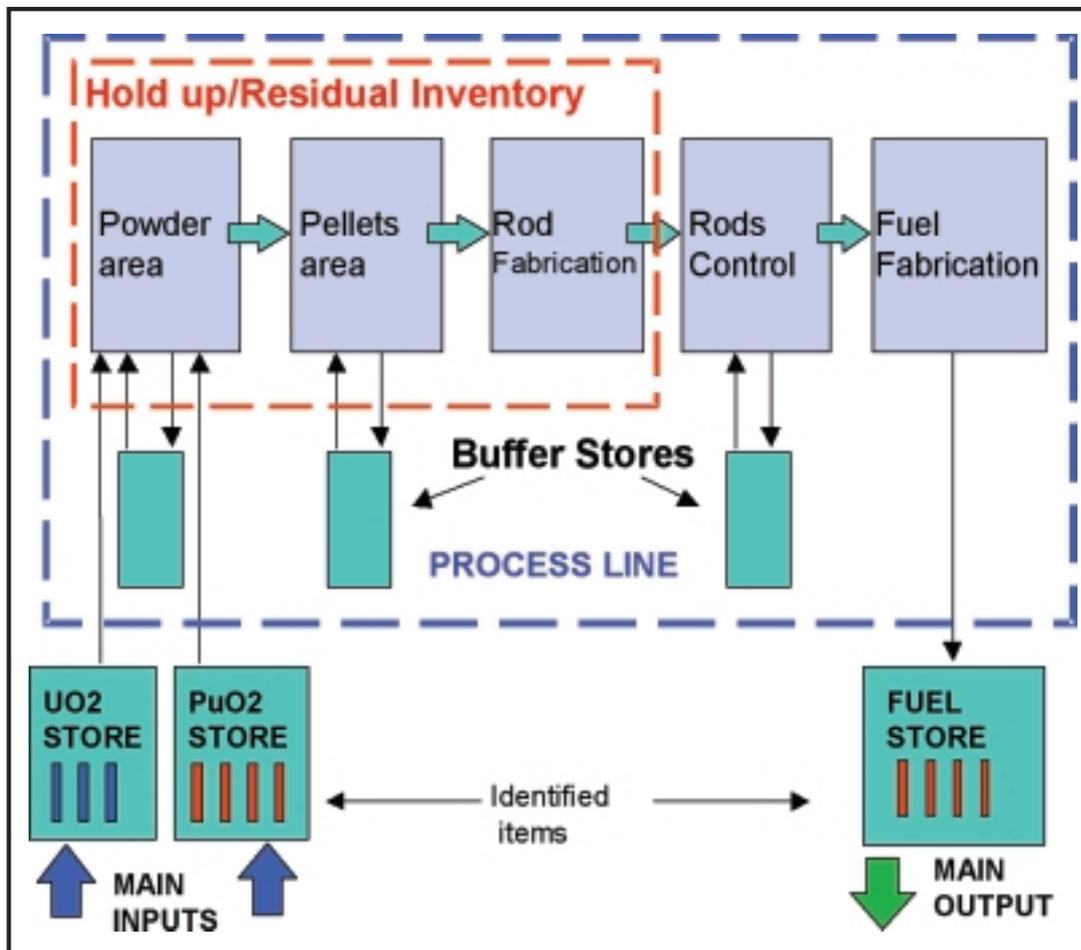


Figure 1 – A typical MOX plant

### 2.2 A glove box at a glance

Consider as an example a simple glove box (see Figure 2) which contains a piece of plant equipment such as a pellet crusher. Trays of pellets of known weight enter the glove box, the pellets are tipped into the grinder and the resultant powder leaves the glove box in containers that are also weighed. A forced ventilation system is attached to the glove box and any airborne dust that may arise as a result of the crushing operation will be collected on filters.

During normal operation of the glove box, all material would be considered as belonging to the in-process inventory of the glove box.

During inactive commissioning an assessment of the amount of material that remains in the crusher, after it has been ‘emptied’, can be made. This reaches equilibrium within a very short time and thereafter remains approximately constant.

This material constitutes the holdup; the quantity is reasonably well known but cannot be directly measured. It remains in the crusher until it is deliberately removed, either for the Interim Inventory Verification (IIV) or, more likely, at Physical Inventory Taking (PIT) /Physical Inventory Verification (PIV).

There will be material on glove box surfaces, on the filters in the glove-box ventilation system and possibly on associated pipe-work. The material on the filters will nor-

mally form the majority of this inventory until such time as the filters are removed for disposal and/or an appropriate measurement is made. This material, which can be recovered during cleaning or maintenance operations, is part of the holdup (although the quantity is difficult to assess apart from feedback from experience). The remaining material in the pipe-work and glove box faces will form part of the “hidden” inventory and will be recovered eventually during the decommissioning and/or dismantling of the equipment or of the plant.

We can now precise the definitions of holdup and “hidden” inventory:

- **Holdup** refers to material that remains in the process area of the plant when the plant has been run down. The amount of material in holdup can be assessed, but may not be directly measurable. This material is recoverable as a result of the periodical thorough cleaning of the plant, including routine filter changing.
- **“Hidden” inventory** refers to material that remains in the plant in small quantities after the periodical thorough cleaning and which cannot normally be estimated or measured. This material is only recoverable as a result of dismantling of whole or parts of the plants, e.g. glove box or pipe-work.

### 2.3 The inventory of plutonium in a MOX plant

MOX fuel generally has total plutonium content of 4 to 10 percent, dependant on the fissile content of the plutoni-

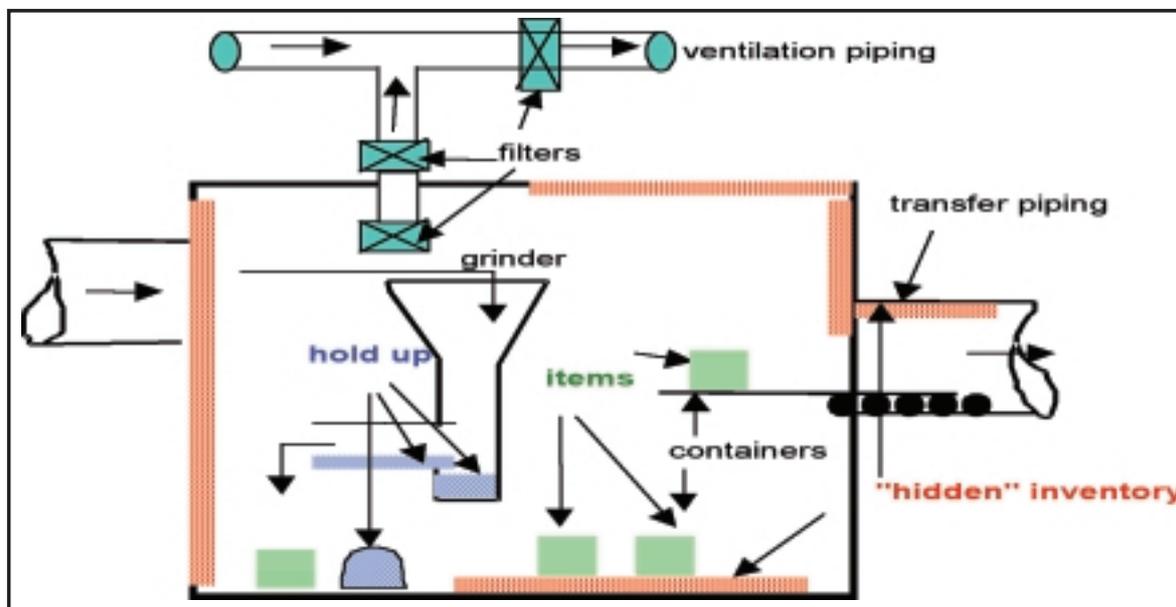


Figure 2 – A glove box at a glance

um. MOX fuel designs call for rods with different plutonium concentrations (up to three in PWR and six in BWR). MOX rods can include some uranium only pellets and a MOX assembly may also contain some uranium only rods. In large plants, high throughputs are obtained by having a number of parallel process lines and this together with the variations of fuel designs and plutonium concentrations lead to significant plutonium stocks.

As described above, front-end stocks of plutonium oxide or nitrate and back end stocks of finished fuel elements in storage are not part of the in-process inventory. Likewise, true in-process inventory does not include intermediate stocks of finished pellets and fuel rods held in secure stores under strict item control.

The table below gives a typical breakdown of inventory in stores and in-process. At Physical Inventory Taking, in process inventory is only made of holdup and "hidden" inventory, and is located in glove boxes.

	During normal operation	At Physical Inventory Taking
In stores	90%	99.8%
In process equipment	10%	0.2%

Table 1 – distribution of plutonium inventory

## 2.4 Holdup and material balance

A material balance cycle starts and finishes with a Physical Inventory Taking (PIT) and is roughly an annual cycle. At PIT the aim is to make all material available to be accounted for in the material accounts and to be verified (PIV) by the safeguards authorities. There should be very little material in the plant not in measurable containers; that which is not is the holdup and "hidden" inventory.

Material that is known to be in certain locations and is defined as holdup should be reduced to the minimum possible, such as by replacing filters before a PIT/PIV when it can be done without adversely impacting operation of the plant, and has to be accounted for at PIT/PIV. This would most probably be by estimates established during commissioning trials or from later plant experience. The material is included in the physical inventory and is not part of the Material Unaccounted For (MUF).

As far as the "hidden" inventory is concerned, it should be reduced to a level as low as possible and the operator can take appropriate actions when possible. "Hidden" inventory which remains will automatically become a component of the MUF that is established at the end of the Material Balance Period (MBP). It will only be recoverable when the plant, or parts of it, is decommissioned.

Two different MUF components can thus be distinguished: the difference between the input and output due to the measurement uncertainties and any "hidden" inventory which may be present in the plant. The MUF (including its "hidden" inventory component) is written off from the books each year. If the component due to the measurement uncertainties has a mean of zero then the Cumulated MUF (CUMUF) should be a measure of the "hidden" inventory, which remains in the plant.

## 3. The Control of holdup in MOX plant

### 3.1 Design features of the plant to minimise holdup

Among other things, MOX fuel fabrication plant and more specifically plutonium plant processes are designed to protect operators from radiation exposure, to prevent criticality events and to meet ionising radiation regulations and also to ensure a high degree of quality control.

Taking into account safety and radioprotection leads to high containment standards consisting of multiple barriers, and emergency breach protection systems. Such measures in effect make plutonium much less accessible both to the operator (diversion more difficult) and to the inspector (calling for tailored safeguard approach). It also translates into several other measures such as:

- Confining plutonium materials to airtight glove boxes, under constant under-pressure;
- Locating glove boxes within fire proof zones with tight barriers;
- Increased automation to give hands-off, remote operation;
- Compact equipment, close process connections;
- Regular and more efficient glove box cleaning to reduce material deposits;
- Optimised maintenance to reduce dose uptake;
- Minimised waste streams.

As far as quality control is concerned, it requires extensive control and tracking features that are also beneficial to nuclear materials accountancy and can be of direct benefit to the safeguards authorities:

- control over homogeneity and plutonium concentrations and therefore extensive efforts to minimise holdup;
- 'bar' coding of containers and components to allow automatic identification of the products either in store or as they are transferred within the plant;
- fully computerised data capture and tracking for product traceability;
- automated inspection (e.g. pellet surface, end cap weld integrity, etc.);
- sophisticated handling and transport systems;
- the introduction of statistical process control and planning techniques.

### 3.2 Design features of glove boxes to minimise holdup

Focusing on glove boxes, a key equipment of the bulk handling part of a MOX fabrication plant, some technological features can participate in reducing the "hidden" inventory. New glove boxes only use stainless steel and lead glass for their construction, and their internal shape is optimised for plutonium recuperation: hence the "hidden" inventory can be kept relatively low. Holdup in process vessels is also dependent on the smoothness of the stainless steel surfaces in the silos, blenders, homogenisers and other process equipment and the free flowing characteristics of the powders.

Glove box extract systems employ high airflow venting through a primary outlet HEPA (High Efficiency Particle Arrester) filter to the vent ducting. This outlet filter traps most of the possible contamination. Ventilation ducting usually terminates at a single or double HEPA filter bank.

### 3.3 Control of holdup during plant operation

On one hand, routine cleaning procedures are carried out during tooling-up between different production campaigns and also after each change of Pu-enrichment. It allows keeping the amount of holdup and "hidden" inventory at a low level. On the other hand, the constant monitor-

ing of accountancy balances gives an indication of the level of holdup and "hidden" inventory. Because of these two features, special Non Destructive Assay measurement for the determination of holdup and "hidden" inventories is not required.

However, since gamma and neutron dose rate measurements are done for radiation protection monitoring at selected points in the process area, they are additionally used to give adequate warning of any apparent increase of holdup and "hidden" inventories. Those measurements are done at each glove box, at each working place and at all locations known to give higher dose rates.

The regularity of cleaning keeps the holdup and "hidden" inventory relatively low and avoids build-up to any "saturation level". Warnings from the radiation monitoring or from the accounting system give rise to additional cleaning measures. An additional thorough cleaning is also required for the annual PIT to minimise holdup and "hidden" inventories and to make those materials available for physical verification; for the same reason, in so far as possible, some maintenance operations such as filter replacement are scheduled before annual PIT.

### 3.4 Holdup evaluation

MOX fuel fabrication is a discontinuous, "batch" process and each production batch (material from the same origin, e.g. from a mixing process) can be individually balanced and followed from process step to process step, from glove box to glove box, from "accounting position" to "accounting position". A running balance of the expected inventory is recorded for each "accounting position", subdivided into cans, boats, trays etc. for each production batch. Routine cleaning is performed between each production campaign.

The differences between input/output-data of each accounting position are allocated to special batches. Following collection and measurement of sweepings from the glove box then the difference is split into two special batches, one for sweepings and one for material holdup (still in the process equipment). If in addition the actual process equipment is thoroughly cleaned then any remaining difference is regarded as "hidden inventory". The determination of the "holdup" and "hidden inventories" is primarily based on weight measurements but is affected by arising of Plutonium Contaminated Materials (PCM). PCM is primarily in the form of organic waste and in the filters, which are measured by NDA methods. Following waste transfers and thorough cleaning the accounts are reset to zero. Of course there would still be some nuclear material present in the form of firmly sticking deposits in the glove box system, which is kept in check by the continuous dose rate measurements and by monitoring the cumulative MUF account.

Periodical cleaning therefore is an important measure on the one hand for the reduction of the holdup and inventory and on the other hand for the adjustment of the book accounts.

### 3.5 Specific safeguards measures

In modern plants where processes are difficult or impossible to access, early consultation between the oper-

ator and the safeguards authorities is vital. During design verification activities the safeguards authorities can gain assurance that there are no undeclared features/access routes. During commissioning the operator's measurement systems and process operations are verified and process norms established for use in future process monitoring. Any holdup values established at this time can be verified by the inspectors.

In some plants, where production can be stopped on a regular basis, the safeguards approach is based on a monthly Short Inventory Verification (SIV). The operator then ensures that the majority of the material is moved into measurable locations, thus reducing in-process inventory to holdup and hidden inventory. In continuously operating plants, a safeguards approach based on frequent inventory verification of a running process has been adopted by Euratom. This requires the in-process inventory to be measured using methods that are as unobtrusive as possible.

## 4. Plant specific experience

This section will provide some examples based on actual operation and experience of European plants. Obviously, it is not intended here to be comprehensive on all features of each of these plants, whether these features are common to all plants or specific to a plant or a process, and the illustrations given below shall not be understood as the only features of those plants.

### 4.1 The Belgonucléaire MOX plant: holdup management for PIT

Production procedures require regular cleaning of production equipment and/or glove boxes. Different levels of cleanings are applied, depending on the purpose: change of Pu-enrichment within a campaign, change of production campaign, and once per year for the PIT.

At the time of the PIT, specific actions are applied:

- all powders and pellets are collected in cans or trays, which can be weighted or measured with NDA methods;
- the scrap material is transferred to storage room after measurement;
- the process equipment that could contain MOX powder or MOX pellet is emptied and cleaned as far as reasonably possible (thorough cleaning).

For instance, a prolonged emptying procedure is applied to the ball mills in order to minimise the remaining holdup quantity (usually, less than 25g of MOX powder), which is then recorded on the Physical Inventory Listing (PIL) as part of the dust estimate of the process area concerned. Likewise, remaining powder after emptying grinding devices (including filters and dust collectors) is recorded on the PIL as part of the dust estimate of the process area concerned. Other process equipment does not usually contain any MOX powder after emptying and cleaning.

The powder transport container are also emptied and weighted; if it is not completely empty, the container is listed on the PIL.

The outlet filters of the glove boxes are measured with a portable neutron device, and Pu amounts are deduced based on a calibration curve.

In the glove boxes, dust sweepings are collected, weighted and measured with NDA methods.

While those are specific measures performed at PIT time, preventive measures are permanently applied, such as splitting the process area in working-units for which balances are drawn in a continuous way (in-out), continuous control of the quantities in-out of the blenders and the ball-mills, or traceability of batches.

As a result of those preventive measures and PIT specific actions, the holdup and "hidden" inventory can be minimised, and experience shows that it remains at a constant level over the years.

### 4.2 The MELOX plant: combining plant design and pro-active material management

As in other MOX plants, the holdup and "hidden" inventory will derive from the use of powders (handling, dosage, milling, homogenisation), the pellets handling (drop of pellet), and the pellet grinding (grinding dust).

In order to limit "hidden" inventory and to track holdup, active measures are implemented. For instance, containers are identified and weighed at the entry and the exit of each production station; the data are sent and processed in the Material Information System (MIS) to determine the material undergoing processing in each production station and detecting any drift. In the powder processing areas, the quantity of material entering and leaving each line is measured and compared to determine the quantity remaining in the line. Dealing with pellet handling, the mass of each container entering a processing station is compared with the mass known when leaving the previous processing station, in order to detect pellets dropping between two stations.

This complements preventive measures incorporated at the design stage. For instance, every docking station for jar drainage or filling is equipped with a cyclone system to prevent dissemination of powder during powder transfers, and jars are equipped with plugs during transfer between stations. A cyclone system is also used in the pellet grinding glove boxes to recover the material in dust pots; and pre-filtration of the extraction from those glove boxes prevents the deposit of dusts in the ventilation ducts. In addition, all glove-boxes and their attachments use stainless steel to facilitate cleaning.

In addition, specific operational actions are taken to minimise the holdup and the "hidden" inventory while participating in product quality and reduction of workers radiological exposition. Regular cleaning is one of those measures: cleaning is performed routinely when the Pu content has to be changed in a given campaign and between campaigns, during the annual inventory, and also during specific maintenance operations. Cleaning operations can also be triggered by the results of regular irradiation measurements performed by the "Radiation Protection and Environment section" of the plant at several points (filters, hoppers in glove boxes, ...).

All those measures, whether design or operation, ensure a minimised quantity of holdup and "hidden" inventory. Holdup and "hidden" inventory are evaluated with a good accuracy by feed back, and by measurement whenever possible. They are then recorded in material balance for each production station, and the MIS can track it in each production station as well as in the entire plant.

### 4.3 The Siemens MOX Facility: feed back of holdup quantification

#### 4.3.1 Handling weight data

The Siemens 2 plant was designed for automatic data capture from identification and weighing systems in all areas which transfer items in "containers" (e.g. trays, boats etc.). In the Powder Treatment Area the transfer data was to be read manually from the weighing systems after having completed a transfer. The accounting system would therefore reflect the results after the completion of transfers. "Dynamic" readings from the continuous weighing systems would be recorded via a "Monitoring System" and used "only" for a plausibility check of the data booked in the accounting system.

Holdup amounts found to lie within the uncertainty of the measurement systems would not be recorded in the accounting system, only significant amounts would be recorded. However even differences that lie within the measurement uncertainties could be evaluated over a longer period of time, if the differences proved to be systematic.

#### 4.3.2 Some holdup levels

- **Ball Mills** - The "machine holdup" was on average about 200g of MOX powder. If the material balance in the mill was higher then mill operation was continued, until this level of run out was reached.
- **Blenders** - For a 200kg blender a constant "machine holdup" lay between 300g and 800g of powder. Before each change of enrichment, the blender was cleaned to avoid inhomogeneities in the final product.
- **Filters** - All exchanged filters in the upstream of the Mixer glove box between 1988 and 1991 (183 filters) from the first step glove box HEPA filter were measured before disposal. The Pu-content of the filters amounted to 504g, e.g. approximately 15kg of MOX powder. The next filter in the down stream ventilation system was found to be normally free of nuclear material or only slightly contaminated. The 15kg of MOX Powder was about 30% - 50% of the amount of material on the filters, the rest was removed mechanically before transfer to waste.
- **The Pneumatic Transport System** - trials with a Pneumatic powder transport system show the receiving station has a constant "holdup inventory" of about 100g, whereas the sending station appears optically to be clean, as long as dry, flowing powder is used. The holdup in the feed pipes depends on the type of the material (Mix, Mastermix, UO<sub>2</sub> etc.) and can amount to 1 - 5g per meter of the system as such.

### 4.4 The Sellafield MOX Plant: prevention is better than a cure

SMP employs the Short Binderless process which is a rapid forced milling system which has short material dwell

times and a compact design, thus minimising the amount of material being processed in order to achieve the desired throughput. The process is arranged in vertical towers. These towers are all individually monitored by near real time accountancy software that employs the continually occurring inventory data to assess statistically the holdup and cumulative differences between tower inputs and output.

Like any MOX plant the activity that tends to generate the most dust tends to be the grinding of pellets. In SMP grinding again occurs in enclosed equipment with dust being removed by force by a high power extract system. This collects material grinding dust onto a re-usable pulsed filter. The pulse blow back force removes material from the filter into a sealed collection vessel, which is weighed and transferred to the residue, recycle system.

All process operations up to the transfer of pellets from boats to trays are to be conducted automatically without manual intervention. A combination of material tracking, direct in-line weighing and process sequence control give the plant a high probability of detecting individual pellet losses.

The plant safety case included a large number of hazard operability and analysis studies (HAZOPS and HAZANS), which have covered the issue of glove boxes and equipment design to prevent unwanted accumulations and to provide a deterministically criticality safe processes (either by plant geometry or capacity limitations). These studies also identified all areas with potential for material to collect and as a consequence engineered the design so that these materials migrate and collect in known places with special collection and retrieval systems.

## Conclusions

Modern MOX fabrication plants allow for a highly efficient accounting and safeguarding of nuclear material, from the design stage up to day to day operation. The design of the plant and of the glove boxes are meant to reduce loss of nuclear material and therefore to reduce material unaccounted for. During operation, control of nuclear material is enhanced by monitoring and regular cleaning and maintenance of the plant: such operations lead to the routine recovery and measurement of most of the material which did not make its way into a MOX pellet.

At PIT/PIV time, holdup can be assessed and is therefore accounted for, while "hidden" inventory is recorded into the MUF because it cannot normally be measured or estimated. Even in the MUF, "hidden" inventory is under control: this small quantity of nuclear material is in the plant and is recovered as a result of dismantling of the plant or parts of the plant (glove boxes, ..).

On one hand, the operators of MOX fabrication plants are dedicated to reducing to the minimum the "hidden" inventory and to providing the best information to assess holdup when it cannot be directly measured; on the other hand the safeguards authorities are implementing a hierarchy of safeguards measures and their inspectors have gained an in-depth knowledge of the plants. Combining those factors enables the safeguards authorities to give safeguards assurances for MOX plants.

## APPENDIX: Some Reference Definitions United States Nuclear Regulatory Commission

### *Regulation 10 CRF part 70 Licensing of SNM*

**70.51 Material in process** means any SNM possessed by the licensee except in unopened receipts, sealed sources and ultimate product maintained under tamper safing.

### **NUREG/BR-0096 Instructions and Guidance for Completing Physical Inventory Summary Reports, October 1992**

**IN-PROCESS HOLDUP** - Process related SM or SNM that has not been drained from its processing equipment at the time of physical inventory. The quantity of any in-process holdup must be included in the physical inventory determination. [NOTE: The term "in-process holdup" should not be confused with the term "residual holdup".]

**RESIDUAL HOLDUP** - Any SM or SNM that remains within processing equipment (including ventilation filters and ductwork) after system drain down and/or clean out. If, at the time of physical inventory, the total quantity of residual holdup is significant, such holdup must be measured (or estimated on the basis of partial measurements and engineering calculations) and included in the physical inventory listing.

### **NUREG/CR-5550, LA-UR-90-732 Passive Non-destructive Assay of Nuclear Materials**

**NUCLEAR MATERIAL HOLDUP** - The term "holdup" refers to the accumulation of nuclear material inside the processing equipment of nuclear facilities. Other common terms for such material are "hidden inventory", "normal operating loss", and "in-process inventory". The choice of terminology depends in part on the application or point of view. For example, the nuclear material that remains in the facility after the runout of all bulk products may be called "in-process inventory". The material that remains after thorough brushing, wiping, acid leaching, and rinsing may be called "fixed holdup".

Because of the high economic value of nuclear material and the need to ensure radiation safety and criticality safety and to safeguard against theft or diversion, it is important to minimize holdup, to measure or model its magnitudes, and to remove it.

IAEA Safeguards Glossary (IAEA/SG/INF/1, Rev.1, 1987, latest version)

**Art. 58: HOLDUP** - nuclear material deposits remaining after shutdown of a plant in and about process equipment, interconnecting piping, adjacent work areas. For plants in operation the holdup is the amount of nuclear material contained in the process.

**Art. 174: MUF observed value** - the value stated by the operator, based on the closing of a material balance for a given period. Whether or not diversion has occurred, the MUF for bulk material will normally be different from zero owing to holdup in process equipment and unmeasured losses.

# REPORT OF THE ESARDA REFLECTION GROUP 2000

**M. Cuypers**

JRC, Ispra

On behalf of the Reflection Group

This paper reflects the report of the ESARDA Reflection Group 2000, presented by the chairman of the Group, M. Cuypers, in a special meeting of ESARDA Members, held at Bruges on May 7, 2000 and discussed at the ESARDA Steering Committee on May 11, 2000.

The Steering Committee has accepted the report and has formulated comments on a few recommendations related to the safeguarding of nuclear waste, the involvement of ESARDA in illicit trafficking, the level of extension to other nuclear areas. Some recommendations for internal use are also being reviewed. These points are being clarified now within different Committees of the Association.

May 6th, 2001

## 1. Background

ESARDA was founded in 1969. In its 32 years of existence, the Association has organized periodically reflections on its policies and objectives.

A thorough analysis was performed in 1988 by the Working Groups during the annual meeting of the Association. The results of this reflection and analysis were never published, but served to re-orientate the activities of the Working Groups, to take into account the prospected evolution of the Nuclear Fuel Cycle in Europe.

In 1993, a "Reflection group on the future of ESARDA in a changing world" was established under the leadership of G. Dean (CEA). The group submitted its final report to the Steering Committee and, after approval, it was presented to the ESARDA 16th Annual meeting at Ghent in 1994. The report has been published in the ESARDA Bulletin n. 25 (June 1995). ESARDA has implemented most of the recommendations of the Reflection Group of 1993 (RG 1993), in view of adapting the activities and structure of the Association to the new challenges. A report on the degree of implementation of the RG 1993 recommendations has been submitted to the Steering Committee on November 29th, 1996.

During the period 1994-1999, important changes took place in nuclear safeguards and non-proliferation, in general. During the same period, new Parties joined the Association (ANPA, SKI, STUK, WKK). At several occasions these new Parties expressed the need and made recommendations to review the structure and working methods of the Association. In particular, STUK, during its presidency, made specific suggestions. The Steering Committee then considered that some conclusions drawn at the RG 1993 needed to be reassessed. At the May 3, 1999 Steering Committee meeting at Sevilla, it was therefore decided to establish the second Reflection Group to study the different issues and the proposals for change made by ESARDA members.

## 2. Establishment of a New Reflection Group

The Steering Committee defined and approved the following terms of reference of the new Reflection Group (RG 2000):

"to review the status of implementation of the decisions taken after the proposals of the RG 1993, analyzing whether further actions are needed and making proposals to the Steering Committee regarding possible improvements in the structure and operations of the Association "

The 1999 Chairman of ESARDA, T. Varjoranta, proposed that M. Cuypers (JRC, Ispra), already member of the RG 1993, would take the lead of this initiative. The following ESARDA partners accepted to participate to the RG 2000 : CEA, DTI, EC-ESO and JRC, FZJ, STUK. The following persons were nominated by their respective organizations: M. Beaman (DTI, London), W. Kloeckner (EC, ESO), Y. Souchet (CEA, IPSN), G. Stein (FZJ, Juelich), M. Tarvainen (STUK, Helsinki). C. Foggi (JRC, secretary of the Association) accepted to act as secretary of the RG 2000.

The group met five times namely on September 6-7, 1999 at Brussels, November 22, 1999 at Ispra, March 9, 2000 at Brussels, April 18, 2000 at Ispra and September 27, 2000 at Ispra.

The main subjects, discussed by the RG 2000 were the following:

- direction of the existing activities of the Association
- future safeguards challenges
- extension of the scope of ESARDA
- organizational structure and administrative issues
- international co-operation
- customers
- information to the public

For each of the subjects mentioned above an analysis was made of the level of implementation of the recommendations of the RG 1993 and new initiatives were proposed, when appropriate.

This report reflects the results of the discussions of the Reflection Group. The ideas presented in this report list a number of areas where further consideration is needed regarding the possibility of expanding the area of interest and improvement of the management of ESARDA's work. The RG recognizes, however, that it would not be feasible for all of these ideas to be taken forward and that decisions will need to be taken on prioritisation.

### 3. Direction of the Existing Activities of the Association

The RG confirmed the fact that ESARDA should primarily remain a forum to discuss safeguards present and future issues, for the exchange of information and experience and for the execution of common projects of safeguards practitioners (plant operators, safeguards authorities and developers). As stated in the ESARDA Agreement the core business of ESARDA is R/D activities and the implementation of the results by plant operators and safeguard authorities.

Existing activities are those presently conducted in working groups, the organization of Symposia and thematic seminars and the Bulletin publication.

At present, ESARDA has six active Working Groups, three of them "discipline oriented" (on "Techniques and Standards for Destructive Analysis (DA)", "Techniques and Standards for Non-Destructive Assay (NDA)" and "Containment and Surveillance (C/S)"), two of them being "plant oriented" (MOX Fuel Fabrication, Back-end of the Fuel Cycle) and finally a working group on Integrated Safeguards. The terms of reference and the activities of the working groups are described in the internal document of the Association called "ESARDA strategic plan". The work plans are to a large extent based on the R&D program and needs list periodically submitted by the EURATOM Safeguards Office (ESO) and on the R&D program available from the IAEA. The actual work plans of the Working Groups also take into account the requests and needs of the plant operators and of other ESARDA members.

#### 3.1. Plant Oriented Working Groups and Integrated Safeguards

The plant oriented Working Groups are addressing the safeguards issues in large industrial facilities of the nuclear fuel cycle. They are related to MOX Fuel Fabrication and the Back-end of the Fuel Cycle.

A third Working Group (LEU Conversion and Fuel Fabrication Plants) has recently terminated its activities.

##### 3.1.1. LEU Conversion and Fuel Fabrication Plants

The activities performed in the framework of this Working Group have been satisfactorily completed. The Steering Committee has therefore decided to close the Working Group. Issues that still need to be addressed in this area could be easily tackled within the MOX Fuel Fabrication Working Group or, if necessary, in ad hoc meetings requested by the organizations directly interested (e.g. plant operators and ESO). The participation to the MOX working group of the former chairman of the LEU conversion and fuel fabrication working group would provide the proper link for future co-operation.

*The RG supports the course of actions taken by the Steering Committee to close the working group and recommends that a final report, collecting the results obtained over the years, be issued by the former chairman of the working group.*

##### 3.1.2. MOX Fuel Fabrication Plants

Issues related to the MOX fuel fabrication facilities are dealt with in a satisfactory way in the relevant ESARDA Working Group.

However it should be considered that, besides the utilization of Pu in MOX for LWRs, different scenarios are being developed for the burning of weapons grade material in high temperature reactors and in accelerator driven systems (ADS). In the development of these reactors and ADS, it is necessary to take into account the safeguards requirements, including those for the fuel cycle associated to these new systems.

*The RG recommends that, for the future, the MOX Fuel Fabrication Working Group may discuss matters related to the safeguarding of fuel fabrication using plutonium (Pu) excess material to the extent that these matters become relevant to the needs of ESARDA members.*

It is however clear that the group should only be involved in the safeguards aspects of the fuel fabrication for these new approaches where these are relevant to the needs of ESARDA members.

The name of the working group should be changed into "Fuel Fabrication Working Group".

##### 3.1.3. Back-end of Fuel Cycle

The terms of reference of the ESARDA Working Group on the Back-end of the Nuclear Fuel Cycle (BFC-WG) are now practically limited to spent fuel storing and disposal. The back-end of the fuel cycle requires special safeguards attention in the future. Several European and other countries have active plans related to the final disposal of spent fuel in geological repositories. Safeguarding of final disposal plants will have to be developed and implemented during the next 20 years.

The final disposal of spent fuel is performed in three separate phases, needing three different plant types. Dry or wet storage installations are used for the interim storage of spent fuel. In conditioning facilities, the spent fuel is subsequently loaded into the final disposal canisters or casks. Finally, the spent fuel will be disposed in geological repositories. For Geological Repositories, three different phases have to be covered, namely the construction, operating and post-closure phase, each of which will require different safeguards measures. The operating phase needs particular attention as, due to ongoing and parallel activities, like excavation and construction of new disposal rooms and corridors, disposal of canisters and subsequent back-filling of the corridors, continuous flow and design re-verification will be required. Safeguarding of closed repositories will have to be based purely on techniques, which make sure that the integrity of the repository has not been violated.

Proliferation and safeguards issues have to be addressed specifically for each disposal concept and each geological matrix.

*The RG recommends to study in detail the possible approaches.*

The effects of strengthening the safeguards system and the Additional Protocol have to be addressed in the approach for the back-end of the fuel cycle.

In fact, the classic safeguards approaches and practices (e.g. timeliness, Significant Quantities) are based on the assumption that the nuclear material is accessible for direct re-verification. Nuclear material disposed in geological

repositories is not designed to be accessible anymore, therefore, safeguarding of disposed material will have to rely on flow and design verification and C/S systems. Completely new safeguards approaches and techniques may be needed. Current plans for a possible later retrieval of the disposed material, discussed seriously in a number of countries, might represent an additional safeguards challenge.

Concerning “waste”, it was noted by the RG that different safeguards approaches have been developed, e.g. by IAEA, EURATOM and Japan.

*This RG agrees that further study of the nuclear waste issue would be worth while, including:*

- definition/classification of waste and a comparison of different approaches to safeguards of nuclear material in waste;
- state-of-the-art in safeguards of waste;
- safeguards of retrievable waste and relevant safety aspects;
- methods to distinguish nuclear material from HLW and MLW.

Regarding the further use of Pu of civil and military origin various options are presently under discussion, including both MOX fuel fabrication and disposal. The non-proliferation issue of the Pu disposal has often been raised. Basic safeguards concerns are valid but with special emphasis due to the high strategic value of separated Pu.

*The RG 2000 proposes that further consideration might be given to expanding the work of the BFC-WG to include also the areas of safeguards of nuclear waste and of Pu disposition.*

In particular, for the Pu disposition, if and when this will happen in the EU framework, also the relevant discipline oriented working groups should be involved. It is also felt that safeguarding of the spent fuel reprocessing still deserves some attention of the BFC-WG. The RG concluded that:

- in the past, back-end of fuel cycle safeguards was not recognized as a priority area by the RG-1994. Now this judgment shall be overturned due to the emergence of new policies, new scenarios and new interested parties.
- it is strongly recommended to continue - and possibly expand – the ESARDA activity in this area.
- by taking into account the needs of all parties involved, it is expected that the Working Group will attract more participation of operators, authorities, Inspectorates and researchers. It was emphasized that the Convenor of the group should be an operator, if possible.

#### 3.1.4. Integrated Safeguards

In May 2000, a Working Group on Integrated Safeguards was established. This topic is discussed with more details in section 4.2.

### 3.2. “Discipline Oriented” Working Groups

The three working groups on “Techniques and Standards for Destructive Analysis (DA)”, “Techniques and

Standards for Non-Destructive Assay (NDA)” and “Containment and Surveillance (C/S)” have a long standing and well defined mission for establishing and maintaining the scientific and technical base of the measurement systems and of containment and surveillance measures applied in nuclear materials management and safeguards. Their operation has been satisfactory and successful and the product of their activities has been diffused through Symposia, Seminars, workshops and the ESARDA Bulletin. Only a few comments are therefore given here. Concerning more specifically the area of C/S, it has been recognised since long time that there are great similarities between technical instruments used for Physical Protection (PP) and C/S measures. In many cases the same instruments can be used for both purposes. It is well known that the US makes little - or no - difference between PP and C/S for safeguards. There is a similar trend in the Russian Federation.

*The RG therefore recommends that the ESARDA Working Group on C/S should increase its interest in the study of the technical characteristics of PP instruments and devices and investigates more deeply possible transfer of technology from the physical protection to the safeguards areas.*

The RG encouraged, in general, the technical discipline oriented Working Groups to pursue their important objectives and maintain their expertise.

*The RG also recommends that a periodic check is performed to assess the relevance of the Working Group activities to the issues described in the following chapters 4 and 5.*

### 3.3. Conferences - Seminars

The organization of Symposia, Seminars and other public events of this kind is one of the activities where ESARDA serves its partners and gets high visibility and universal recognition of excellence. As a result of this recognition, IAEA and the INMM have often asked ESARDA to co-operate with them in the organization of some of their events.

Nowadays, the core of this activity is the Annual Meeting, alternatively consisting of a Symposium on “Safeguards and Nuclear Material Management” and of a topical Meeting (Seminar or Workshop). Additional topical Seminars and Workshops are organised, when needed.

In the years 1998-2000 ESARDA has increased the frequency of Seminar and Workshops, with a view to i) inform the safeguards community on the evolution of the nuclear verification techniques and policies and ii) explore the possible contribution of science, technology and synergies with other verification regimes to the establishment of more advanced safeguards systems without undue aggravation of the costs. The timelessness of these events, some of which were organised in collaboration with INMM, have led to them being judged a considerable success.

It is recognised that there are several types of meetings, which are useful to the safeguards community:

General exploratory workshops, where prospective issues are debated in view of their possible impact on the

development of safeguards (examples: the Oct. 1996 Arona, the Sept. 1998 Albuquerque and the Nov 2000 Tokyo Workshops on "Science and Modern Technology for Safeguards"; the May 1998 Helsinki Workshop on "Modern Verification Regimes: Similarities, Synergies and Challenges")

Topical technical workshops, where the specialists of certain technical areas or disciplines meet to discuss in depth the progress and the perspectives of their domain (Examples: the May 2000 Dresden Workshop on "Strengthening of Safeguards: Integrating the New and the Old", the 1998 Ispra Workshop on the "Quality of NDA Data")

Internal meetings, where the members of the Association discuss about the evolution of the environment in which ESARDA has to operate and make proposal about how to respond to the challenges. (Examples are the Internal Meetings of Copenhagen (1986), Karlsruhe (1988), Como (1990) and Salamanca (1992)).

*The RG recommends that these activities are continued taking available opportunities to hold meetings focused on topical specialist issues of particular interest to ESARDA members.*

## 4. Future Safeguards Challenges

### 4.1. Role of Non Technical Factors in Future Safeguards

Future safeguards concepts and activities will be strongly influenced by non-technical factors (sometimes called inappropriately political factors), such as:

- i) the use of transparency as a means for redistributing inspection efforts without violating the principle of non discrimination,
- ii) the use by the IAEA of the national/regional inspection data, in order to reduce the inspection effort linked to traditional safeguards of INFCIRC/153, and in relation to integrated safeguards. In order to reach this objective it is necessary to apply quality control, quality and integrity assurance measures to the national/regional inspection data to be used by the IAEA.

*The RG recommends that ESARDA studies the impact of these factors on the contents and structure of future safeguards.*

### 4.2. Integrated Safeguards

The political, legal and technical situation of safeguards in the light of strengthening of the safeguards system (SSS), has changed since 1994. The model Additional Protocol has been approved and agreements based on it have been and are now being ratified with individual States and groups of States. Discussions are taking place in many different fora on the practical implementation of this new Protocol and its relation to the traditional safeguards.

Integrated Safeguards is defined by the IAEA as "the optimum combination of all safeguards measures available to the Agency under comprehensive safeguards Agreements and additional agreements, which achieves the maximum effectiveness and efficiency with available

resources in fulfilling the Agency's right and obligations in paragraph 2 of INFCIRC 153 (corr.)". ESARDA has devoted much attention to this issue, organizing several sessions in its Symposia; it has also organized a full Seminar on "Integration of Safeguards" in May 2000 and has created a Working Group on this same subject. Several R&D laboratories are actively involved in the development of related methods and techniques, for instance, in the field of environmental sampling, satellite monitoring, information and knowledge management.

In the next few years, the implementation of the Additional Protocol and its integration with the traditional safeguards will have a major impact on the plant operators and on international inspection.

*The Reflection Group recommends that ESARDA gives a high priority to supporting the development and implementation of Integrated Safeguards.*

ESARDA should facilitate the exchange of information between operators, member states and national, international and regional inspectorates in support of the IAEA's work - helping to prepare ESARDA members for the implementation of additional protocols and also for implementation of Integrated Safeguards (as they affect to ESARDA members) and providing expert views on methodologies and approaches for integrating INFCIRC/153 and INFCIRC/540 measures. Specifically, ESARDA should:

- provide a forum to discuss and encourage consistency in the provision of information under Art. 2 of the Additional Protocol and issues relating to complementary access (e.g. managed access, environmental sampling and unannounced inspections);
- develop and promote approaches to enable the fullest possible use of R/SSACs by the IAEA - in particular, given the unique extent and technical quality of current Euratom safeguards (accountancy and verification) activities, in the implementation of Integrated Safeguards in the European Union;
- develop and promote approaches to enable possible use of SSAC's by the ESO
- assess the consequences for operators and national, regional and international inspectorates of the implementation of the facility-specific Integrated Safeguards criteria;
- evaluate different methodologies and approaches to integrating traditional safeguards and measures arising from the Additional Protocol;
- investigate and co-ordinate of R&D needs for new technologies in Integrated Safeguards and related follow up;
- future activities in the light of strengthening safeguards system and integrated safeguards.

The terms of reference and work-plan of the recently established ESARDA Working Group on Integrated Safeguards reflects the points mentioned above.

### 4.3. Excess Material

By Excess Material is understood nuclear material not anymore needed for defense purposes. In this area, the situation has completely changed since the moment when the previous Reflection Group (RG 1993) made recommendations.

Although politicians are still cautious to discuss the related safeguards issues, the matter is now in the public domain. Nowadays, safeguards of Excess Material is an issue mainly linked to the "Trilateral Agreement" between the USA, the Russian Federation and the IAEA). The IAEA has already started inspection of HEU stocks. Recently the UK has put a substantial amount of its Excess Material (several tons) under EURATOM Safeguards.

Moreover, the European Union is now defining a common policy in respect of Non Proliferation issues, and the issue of Excess Material is one of the components of this policy.

Many points of the safeguards approach to be applied at the different stages of the dismantling process needs still to be clarified, both for U and Pu. The following one is an example of issues to be addressed. From the technical point of view, the stores used for Excess Materials are similar to those used for civil Pu and C/S technologies used for safeguarding them are similar to those applied in classical safeguards. For measurements the situation is however different, since the information barrier prevents to perform measurements which would result in the complete knowledge of the quantity and quality (isotopic composition) of the nuclear material when it is still in a sensitive form. Measurements have therefore limited function, such as of verifying that the material has a certain quality (e.g. is Pu) and that the signal remains stable in time (i.e. there is no change in the material). This is equivalent to say that it is probably not possible to establish the Initial Inventory of Excess Material.

The challenge, in fact, is to ensure that the materials are not diverted without releasing sensitive information. It has to be noted that several presentations have been made on the safeguarding of Excess Material in ESARDA Symposia and Workshops.

The RG recommends that, as and when safeguards methodological and technical issues relating to the disposition process impact on ESARDA members and European nuclear facilities, they can be considered in the relevant ESARDA working groups

#### 4.4. Export/Import Control and Illicit Trafficking

The issue of Export/Import control has gained a new international interest in the framework of the "strengthening of the safeguards" system. Several organizations have expressed the need to have access to complete information on laws and regulations related to the export/import of nuclear material and to the dual use technologies. In this area it is worth noting that:

- the Nuclear Suppliers Group (NSG) has decided to create a web site containing this type of information and the EC (JRC) has been entrusted to create such a web site;
- Canada is actually engaged in creating a similar web site.

In this general framework, some members of the RG proposed that ESARDA takes some initiative to act as a central point in the EU for providing a support to the establishment of a web site in this area. Some members of the RG consider also that ESARDA should enlarge the scope and create a more general web site including, in addition to export/import regulations, also the EURATOM Regulation, the NPT and all attached regulations, national and EU

regulations on safeguards, etc. The issue of Illicit Trafficking is of continuing interest in the framework of the strengthening of the safeguards system. It also has an interest outside the safeguards community for Customs, Police and Health Authorities. It is worth mentioning that some R&D organisations have already contacts with Customs Authorities on technical matters. Some working groups of ESARDA (DA and NDA) could continue to have a role to play, through advice, harmonisation, performance evaluation to national and international Authorities.

The RG considers that this type of R&D initiatives could be co-ordinated at an EU level.

*The RG recommends that, to the extent that the issues concerned are relevant to the main focus of ESARDA activities and the needs of its members and the international safeguards inspectorates, ESARDA pays appropriate attention to this challenging concern.*

#### 4.5. Decommissioning of Nuclear Facilities

The number of nuclear facilities being decommissioned is increasing in several EU countries. Several safeguards aspects during plant decommissioning should be studied, such as:

- i) how to safeguard the material emerging from the plant (e.g. Pu),
- ii) how to make sure that there is no hidden inventory,
- iii) how to make sure that the facilities are not used for unauthorised material processing.

The problems are compounded with the fact that each plant is a specific and different case requiring a specific approach, and that the various plants are now at different stages of decommissioning. This general area is also linked to some of the requirements of the implementation of the Additional Protocol.

*The RG recommends that ESARDA becomes active in this area, by first analysing and describing the existing problems.*

### 5. Extension of the Scope of ESARDA

As stated in section 3, the RG recognizes and emphasizes that ESARDA should primarily remain a forum for the exchange of information and experience and for the execution of common projects of safeguards practitioners. This fact does also imply that ESARDA be acquainted and abreast with techniques and methodologies applied in other areas of verification, in order to be more effective and efficient in the implementation of its main safeguards mission. The potential area of extended interest would include other verification regimes oriented to the control and reduction of weapons of mass destruction, an area which has already been tackled by ESARDA in several sessions of symposia and in the seminar "Modern Verification Regimes : Similarities, Synergies and Challenges" held at Helsinki in May, 1998. Although each verification regime has its own specificity in the implementation its regulations, the general methodologies are often similar and some technologies can be used by different systems; important synergies can therefore be found and exploited. This last point was also put in evidence in a report from the ESARDA Coordinators.

Some experts from ESARDA Parties (e.g. CEA, FZJ, STUK) are already involved in discussions related to "Other Regimes" and the experts taking part in the discussions are often the same which are dealing with nuclear safeguards: internal synergies are therefore easily achieved.

Furthermore, in the framework of the Common Security and Foreign Policy (CSFP), the EC (DG RELEX) is requesting the JRC to provide technical support. The JRC is now studying the possibilities to call on expertise of ESARDA as an European Association and on individual members for providing co-operation and support.

The RG noted that the IAEA and INMM are also increasing their interest to be in direct contact with other verification regimes.

For the possible extension of the interest of ESARDA, a clear distinction is to be made between:

- verification regimes dealing with the nuclear area;
- verification regimes dealing with non-nuclear areas (chemical and biological).

### 5.1. Extension to Other Nuclear Verification Areas

The RG recognized that a number of subjects considered in chapter 4 (Integrated Safeguards, Excess material, Export/Import control and illicit trafficking) are elements within the wider subject of nuclear non proliferation (NPT).

*The RG, therefore, recommends that ESARDA could give further consideration to the role it can play in helping to keep its members abreast of safeguards-related developments in the wider subject of nuclear non proliferation.*

Concerning the Comprehensive Test Ban treaty (CTBT), the RG recognized that there are common areas of interest and some common technology needs, such as environmental sampling and remote monitoring. Experts of CTBT could possibly take part in ESARDA WG activities, provided that the reciprocal advantages of such participation are identified and recognized.

*The RG recommends to establish contacts with the CTBT organization.*

A future Fissile Material Cut-Off Treaty (FMCT) would ban the production of fissile material for nuclear weapons or other nuclear explosive devices. Negotiations for such a Treaty have yet to start, but when they do, it is expected that a major focus will be put on the arrangements necessary to verify the Treaty. It is likely that these arrangements will draw directly on existing experience and expertise in the implementation of safeguards verification measures.

*The RG proposes that, as and when verification issues arise which relate to ESARDA activities and the needs of its members, they could be taken account of in planning future ESARDA activities.*

### 5.2. Extension to Non-Nuclear Verification Regimes

As was mentioned above, important synergies can be found and exploited between nuclear safeguards and non

nuclear verification regimes. Based on the existing experience in designing and implementing an effective and efficient verification system, technology and methodology transfer could take place. Examples are the definition of inspection approaches and inspection schemes, inspection logistics, the application of sealing, surveillance and monitoring systems, data management, environmental monitoring, satellite monitoring.

*The possibility of creating an ad hoc Working Group dealing with similarities and synergies of methodologies and technologies between different Verification Regimes, essentially hosting people from other regimes, was considered by the RG, but no firm proposal is made at this stage.*

This issue should be examined and after that further contacts will be established with technical experts of the other regimes to explore their possible interest in cooperating with ESARDA.

### 5.3. Global approach to verification

Modern society is confronted with a number of technological risks. Particularly challenging are the risks deriving from those technologies which may be used to produce arms (nuclear, chemical, biological, space).

Prioritization of R&D among the various technological proliferation areas requires a thorough analysis. The prioritization will eventually condition the allocation of resources. The scientific society involved in the issue – and ESARDA is part of this society - cannot be insensitive to this aspect of the problem. In particular, the scientific community as a whole has the task of assessing the impact on society and the environment of the various technological risks.

Although the Reflection Group recognizes the importance of the issue for modern society, it is not in a position to make a proposal of whether, and if so how, ESARDA might have a role to play.

The RG acknowledges however that it could be interesting to contact experts in the appropriate scientific fields with a view to investigating what has been done in terms of this kind of study and whether any ESARDA initiative might be appropriate.

## 6. Organizational Structure of ESARDA and Administrative Issues

### 6.1. Management structure of ESARDA

The re-structuring of ESARDA in 1994 was significant, including creation of the Executive Committee and the assignment of certain duties to this body, reduction of the number of meetings of the Steering Committee to one per year only, creation of the Scientific Council and Coordination Board (SCCB), which increases the responsibilities of the Coordinators. As a consequence of the restructuring, the Internal Rules were improved and adapted to the new situation and a Strategic Plan was created.

It was however noted that:

- i) there is still some overlapping between the activities of the Executive Committee and those of the Steering Committee;
- ii) the Secretariat needs to be reinforced;

- iii) some of the Working Groups need more clear policy directions;
- iv) the duties and responsibilities of the Coordinators are already on the increase; Coordinators should be given a more central role in defining and structuring the activities of the Association; this will further increase their workload;
- v) expansion of the activities will raise the problem of financial funding.

It was also noted that:

- i) attendance at the meetings of the Steering Committee is not large;
- ii) in the past few years, the Executive Committee has effectively managed the Association;
- iii) the Coordinators are increasingly and effectively involved in the scientific management of the Association;
- iv) the process for making key decisional processes is too often very slow.

*The RG recommends to partly reshape the management procedures, by making distinction between "strategic issues" - for which decisions should be taken by the Steering Committee - and "operational issues", for which decision could be taken at a lower level (Executive Committee, SCCB).*

An example of such decisions, which have to be taken frequently, is the nomination of observers in working groups.

In order to address the reshaping of the management procedures, it will be necessary to have available for the Association a well defined, approved and periodically updated strategic plan, as the one which was prepared in the past by the SCCB. This plan helps to focus the work of the organization, ensuring, in a transparent manner, that defined and approved objectives are pursued by the different components (e.g. working groups) of ESARDA, through a number of specific and well structured activities. The strategic planning is also a basic tool to measure the global efficiency of the activities of ESARDA.

*The RG recommends to review further the management procedures and, in particular, utilize the strategic plan as a basic tool and to review and precise the role of the Executive Committee and Coordinators, based on the six years of experience after the RG 1993.*

### 6.1.1. Working Groups

It is becoming more and more difficult to find candidate Convenors for the Working Groups, due to the excessive workload put on them. It would therefore be beneficial to modify the working procedures, so as to subdivide the workload among several persons. This can be done by officially creating the post of Deputy Chairman in addition to the post of Chairman (the term Convenor being abolished), both to be appointed by the Executive Committee. The Deputy Chairman would have the responsibility of preparing the minutes and reporting to outside, whereas the Chairman would retain the overall responsibility of guiding the Working Group and convening and chairing its meetings. The Deputy Chairman could substitute the Chairman in case of necessity. At the end of a term, the Deputy

Chairman could be one of the candidates to the position of Chairman. Both the Chairman and the Deputy Chairman should - in the opinion of the Reflection Group - be appointed normally for a period of two years. The BFC working group has already started to implement informally this procedure.

*The RG recommends that the WG convenors examine if the proposed management structure of WG are appropriate for their specific WG.*

*The RG recommends also that ESARDA relies more on temporary "ad-hoc" Working Groups to treat important specific topics.*

The creation of such ad hoc groups should be fast without lengthy procedures for approval by the management bodies of the Association.

### 6.1.2. Parties

In order to streamline the management of the Association it is necessary to review a number of procedures and, in particular, those related to the application of the existing unanimity rule for key decisions and more specifically for the application of the annual rotation of the chair. It is believed that at least for the issue of the rotation of the Chair, it would be necessary to complement Art. 3.7 of the Agreement with a codicil stating that "The Party who is entitled to take the Chair of the Association in a given year shall - at least 6 months before the beginning of his term - announce to the Secretariat his intention to honour this commitment and nominate the Chairperson. The absence of this communication within the established deadline shall be interpreted as a renunciation to take the chair in the year considered."

*The Steering Committee has agreed to modify the Agreement as indicated above and the RG recommends to implement as soon as possible this rule.*

## 6.2. Membership to the Association

### 6.2.1. New Parties from the EU Enlargement Countries

Recommendations of RG-1993 were implemented, since Organisations from Finland and Sweden have in the meantime joined ESARDA.

The need was recognized to open the doors of ESARDA to future EU Member States Organisations, even before their admission to the Union.

*The RG recommends:*

- *considering as a priority the following countries: Czech Republic, Estonia, Hungary, Poland and Slovenia. This approach is also in harmony with the general EU policy for enlargement of the Union.*
- *organising a Seminar to inform the organisations of these Countries of the activities performed by the Association and to explore the possibility of providing them some R&D support related to their current safeguards problems. It has to be noted in this context that the EURATOM Safeguards Office has already taken some initiatives in this direction.*

*The RG recommends to encourage, in addition, Organisations from Norway and Switzerland to join the Association.*

*The RG recommends to study a procedure which gives these prospective Members the possibility to participate to all ESARDA activities, including participation in some of the management activities of the Association, but with the exclusion of the Chair.*

### 6.2.2. Involvement of Organisations from CIS

Recommendations by RG-1994 were followed, but with unsatisfactory results. Participation in Working Group activities, in Symposia and in Internal Meetings was offered to several organizations, with payment of all expenses, but attendance has been limited. Particularly disappointing was the failure of a joint Seminar ESARDA-MINATOM, which was eventually organized by MINATOM together with DOE.

*The RG suggests that ESARDA should leave the door open to new initiatives in the future. Actions should be concentrated on co-operation with relevant Organizations in the Russian Federation.*

It is worth mentioning that an Ukrainian Organization is already regularly participating to the ESARDA NDA Working Group, under the sponsorship of STUK.

*The RG recommends incorporating the activities of the informal group EU-RF into ESARDA.*

This group, which is composed of representatives of the Commission, France (IPSN, EDF, Cogema), Finland (STUK), Sweden (SKI) and UK (DTI and BNFL) meets twice a year. If this proposal is accepted, a Working Group could be created.

### 6.2.3. Individual Membership

Some individuals - formerly affiliated to an ESARDA Party, then having terminated their affiliation – are willing to continue to contribute to the activities of the Association. In many cases their participation could be very beneficial to ESARDA. This possibility is however ruled out by the existing Internal Rules, which only provide for a limited participation of the so-called “honorary members”.

Furthermore, there exist a number of individuals, very knowledgeable of safeguards and non-proliferation, but not affiliated to ESARDA Parties or any public or private Organisation. They could contribute very positively to the activities of the Association, because of their past experience.

*The RG recommends that individuals could participate “ad personam” to ESARDA activities, upon request of the chairman of a working group or by at least two members of the Executive Committee and after final approval by the Executive Committee. The final decision will be laid down in an individual membership letter, defining the rights and duties of the individual member.*

### 6.2.4. Commercial Companies Membership

Regarding the participation of commercial Companies in the activities of the Working Groups, the RG agreed to the procedures proposed by the Co-ordinators.

## 6.3. Communication, Internal and External to ESARDA

Communication to the scientific and technical environment seems to work well. Symposia and Topical Meetings (Seminar and Workshops) have always attracted a large number of participants and papers. They are highly reputed, and their proceedings are issued with a high standard of presentation.

The Bulletin is issued rather irregularly, but its contents are valuable and the presentation excellent; some innovation will however be needed in order to give more visibility to this magazine. Soon, there will be an ESARDA web site.

Communication inside ESARDA has experienced some difficulties: it seems that scientific and administrative information does not reach the Steering Committee timely and completely. Modifications have been recently made to the procedure for transferring scientific information from the Working Groups to the SCCB and to the Steering Committee; the new procedure has however raised objections by the Working Group Conveners.

It was noted that the Steering Committee receives the minutes of the SCCB and of the Executive Committee which, in principle, contain all needed information.

### Internal

It was recognized that communication between members of the Association within the same Party could be improved. Also, there should be more information emerging from the Working Groups (especially periodical reports).

*The RG proposes that the minutes of the Executive Committee be distributed also to the Coordinators and the Conveners of Working Groups.*

### External

It was recognised that the organisation of scientific events (Symposia, Seminars, Workshops, etc.) is one of the most successful activities of ESARDA and give a great visibility to the Association. It was agreed that also the ESARDA Bulletin and the newly born ESARDA web site are important means of communication.

*The RG strongly encourages ESARDA to take any initiative to improve the Bulletin and to increase ESARDA use of and accessibility via the internet.*

*The RG suggests also to prepare a leaflet describing ESARDA and its activities, to be used for large distribution. It was agreed to transform the Bulletin into a two-part journal, one of which fully dedicated to peer-reviewed scientific articles. It was suggested to increase the number of subjects considered by the Bulletin, to include the prospected new areas of interest of the Association.*

*It was also suggested to extend peer reviewing to the papers of the Symposia, but it was concluded that it would be difficult to implement the proposal for the following reasons: i) insufficient time, ii) lack of resources, iii) difficulty to reject certain categories of presentations, even if not scientifically optimal.*

## 6.4. Forecasting

There is a need to periodically (typically once per year) re-examine the status and the perspective of the

Association and not waiting for the creation of a reflection group. This is particularly true if some of the important changes proposed by the present RG will have to be implemented.

*The RG proposes that the Executive Committee organizes once a year a special meeting or introduces as an important point of its agenda a reflection on the future directions of the Association.*

For this reflection it is advisable to enlarge the participation to these meetings to special observers, which could be called from working groups, Coordinators or be some selected experts. The ESARDA chairman should have the privilege for the nomination of these observers.

## 6.5. Financial Issues

The financial coverage, required for the organisation of ESARDA events (Symposia, Seminars, Workshops, Internal Meetings, etc.) has always been covered by the fees of the participants. It is, however, to be noted that the personnel cost of the host Organisation and of the ESARDA secretariat are not covered by these fees. Furthermore, the Agreement foresees that in case of deficit, the Parties will cover this according to a defined key of reimbursement.

*The RG recommends that the internal rules of the Association describe more clearly the procedures on these issues.*

The RG raised also the question whether the EC can provide funds (in the framework of network initiatives) to stimulate some specific activities of the Association. This financial contribution could stimulate a number of members of ESARDA to participate more regularly to ESARDA meetings at the level of Executive Committee and SCCB. Some financial contribution to the working group chairmen and deputy chairmen could also be envisaged for easing the proper management of their respective working groups. Finally some funds are also required to perform small studies of direct interest or relevance to ESARDA activities.

## 6.6. Review of the Agreement

The RG is not in favour to introduce at this stage important changes to the Agreement, a process that could take a very long time and demand extended negotiations among the Parties.

## 7. International Co-operation

ESARDA has always promoted international co-operation with other Organizations related to the Safeguards Community. International co-operation provides opportunities to exchange information and experience on the status of safeguards implementation and its evolution in different countries and as such may contribute to the transparency of safeguards and non-proliferation measures. The long-standing co-operation with INMM for organizing joint seminars and workshops has always been successful and should be continued. The co-operation with the IAEA has been conducted practically since the creation of the Association. This co-operation is performed through the joint organization of IAEA symposia, participation of IAEA experts to ESARDA working groups. The ESARDA symposia

and internal meetings provide also an opportunity to the IAEA to have access to a wide scientific European Community and to present the present status of safeguards and may be more important its evolution (e.g. strengthening and integrated safeguards). The presentation at international meetings of the objectives, scope and practical activities of ESARDA had the objective to present a certain global image of the scientific and technical safeguards community in the EU. This action has also induced an interest in the Russian Federation, South East Asia and to a lesser extent in South America to establish in the respective regions a technical and scientific forum in support to national, regional or international safeguards.

*The RG recommends to actively pursue the existing activities on international cooperation.*

## 8. Customers

One of the important criticism made to the efficiency of ESARDA is the fact that it is not sufficiently "customer oriented". The RG has identified the following customers of ESARDA activities. The RG also identified some "deliverables" for the various categories of ESARDA customers.

Policy Makers: safeguards are a key issue for non-proliferation. Disarmament, management of weapon grade nuclear materials, undeclared nuclear activities, illicit trafficking, are major political issues. Policy makers require an improved knowledge about the technicalities of these issues.

National Authorities: wish to be informed on the state of the art of safeguards methodologies and technologies, on safeguards approaches, on the state of implementation, on the techniques, on the experience gained.

Inspectorates (national, regional, international): they are the main customers for instruments development and approaches development; they wish to be informed about the experience in other Countries, to exchange information and to improve their public relations.

Technical support organizations: they wish to continuously monitor the state of the art of techniques and technologies, the availability of tools, the potential (target values) and the performance (performance values) of the measurement systems.

Plant Operators: need a forum to compare their nuclear material accountancy and control systems and related experience, to discuss matters related to safeguards implementation, advances in technology and the possible application of safeguards instruments and devices in their plant operation.

R&D Organizations: they need to exchange information in order to correctly and efficiently plan their activities and they wish to create and be parties to networks. (common projects, bench marking).

Developers (institutional and commercial): they need to know what the needs of operators and inspectorates are; they can use ESARDA as a forum for exchange of information.

Media (and public): they need general information on the safeguards activities, their rationale and the acceptability of verification measures.

## 9. Information to the Public and non Safeguards Community

This area has not been addressed at all by ESARDA, notwithstanding the recommendations made by RG-1993. The distribution of information has the objective to inform non insiders on the significance of safeguards and non proliferation, the way that safeguards is implemented, its performance, domestic and international structure etc. These actions should provide an increased transparency in the areas of peaceful development of nuclear activities.

The tools which may be operated are, for instance:

- WEB site
- participation to conferences on nuclear energy
- preparation of pamphlets, describing the main elements of the existing system
- preparation of a general file, which could be distributed to media

The Secretariat has created an ESARDA web site, which is installed at the JRC Ispra.

At present, the site only contains general (and static) information on the structure and the activities of the Association. It is planned to add dynamic information on the upcoming ESARDA events (Symposia, Seminars), scientific information originated by the ESARDA Working Groups (scientific and technical reports, reports of activities, data bases, etc.) and reports by ESARDA Committees and data bases produced by working groups.

*The RG recommends that ESARDA seeks advice, if necessary from external experts, in developing an overall communication and publication strategy.*

## 10. References

- "Report of the Reflection Group on the Future of ESARDA in a changing World" G. Dean, ESARDA Bulletin 25, June 1995, pp 7, 11

The following documents and notes have been used during the discussions of the RG 2000:

- Minutes of the different RG 2000 meetings, prepared by C. Foggi
- "Future of ESARDA: Reflection Group Issues", note prepared by M. Tarvainen
- "Safeguards Trends and Items which need Observation ", note prepared by H. Nackaerts
- "Verification Regime for CWC ", note prepared by Y. Souchet
- "ESARDA Reflection Group Issues ", note prepared by B. Burrows
- Proposal of the Co-ordinators for Membership of commercial Companies
- "ESARDA is 30 years old : Young, adult or old " ESARDA symposium paper at Sevilla by M. Cuypers and C. Foggi.
- "Verification at large " note prepared by C. Foggi and G. Stein
- "ESARDA and the Internet " note prepared by C. Foggi
- "Comments by the SCCB on the report of the ESARDA Reflection Group 2000"
- "Comments on draft report of the ESARDA reflection group" by G. Andrew
- "Comments on draft report

## ESARDA Members' contribution

# Handbook of Gamma Spectrometry Methods for Non-Destructive Assay of Nuclear Materials

**P. Mortreau, R. Berndt, Monograph,**  
EUR 19822EN, Ispra, April 2001

Inspector training courses are an element of JRC's support programme for the EURATOM Safeguards Office. In this framework, three gamma spectrometry courses are held with the following topics:

- Basic Physics for Non- Destructive Assay,
- Verification of Uranium Enrichment by Gamma-Ray Spectrometry,
- Plutonium Isotopic Composition Determination by Gamma-Ray Spectrometry.

Up to now, the contents of each course was documented with a manual that specifically concentrated on the relevant topic. It appeared well thought-out to include into the documentation a handbook of gamma spectrometry to summarize and recall basic knowledge as well as technical and nuclear data shared by these different courses. Moreover, the purpose of such a handbook was to provide a manual that is as useful and practical as possible for inspector use in-field by selecting a limited number of essential data.

The first part of the handbook comprises of detailed user instructions for a series of measurement programs in the form of step-by-step procedures. These refer to software and hardware commonly used by the EURATOM or IAEA inspectors. The second part of the handbook contains a glossary of 400 keywords. They define some basic concepts of nuclear physics and gamma radiation measurements, technical data and information concerning the software used in the procedures. The last part of the handbook is a library of spectra (U, Pu, Th, MOX and spent fuel measured with Ge, NaI or CZT detectors, nuclear data useful for some applications of gamma spectrometry, gamma and X-rays for U, Pu and decay products, decay chains, nuclear data for selected nuclides, Compton and backscatter peak energies, photon mass attenuation coefficients, infinite thickness for typical Uranium materials, etc...). There are annexes concerning trouble shooting with measurements and radioprotection.

The use of this handbook during the last training course showed that the participants were better prepared to answer the test questions.

## In memoriam

Paul Ek, who was employed at the Swedish Nuclear Power Inspectorate, SKI, for many years, has died of cancer, at the age of 63. He is survived by his wife, Ann-Marie, and his children, Thomas and Regina and their families.

In 1963, Paul joined the relatively newly formed Swedish Delegation of Atomic Energy Issues and was soon in charge of matters relating to the transport and control of nuclear materials. Paul immediately perceived the potential of this somewhat neglected area and developed the Swedish State System for Accountancy and Control of Nuclear Materials. He became the Director of the Office of Nuclear Materials Control, now called the Office of Nuclear Non-Proliferation. The Office has remained more or less unchanged through a number of reorganizations of SKI, and activities carry Paul's distinctive mark.

Paul was the type of person who rapidly identified areas where work was needed. He was in the vanguard of work within physical protection and safeguards and, thereby, established a strong position for himself and for his colleagues. Paul was a key figure on the international scene and was a driving force in the negotiations concerning the Non-Proliferation Treaty, the Convention on Physical Protection and a number of other important instruments.

His strong position on the international front led to his appointment by IAEA Director General, Hans Blix, to the task of rationalizing the IAEA's inspection activities. Paul was therefore employed from 1983 to 1985 at the IAEA. Shortly afterwards, Paul assumed the position of chairman of the Standing Advisory Group to the Director General of IAEA, SAGSI, and revived the group, turning it into a driving force in the area of international safeguards – the role of the group today. Paul's main contribution was probably that of laying the foundation for the reinforcement of IAEA's safeguards after Iraq's nuclear arms programme was exposed. As chairman of SAGSI, Paul formulated the "93+2" programme, that gave input and basis for the Additional Protocol to the IAEA's safeguards agreement that was approved by the IAEA Board of Governors May 1997. By this the IAEA have got a tool to strengthen its safeguards activities.

Paul had a long-standing co-operation with Joint Research Centre of the European Commission, especially their facilities at Ispra and Karlsruhe, in various issues concerning Non-Proliferation of nuclear material. He was a great inspirer and, among other things, initiated and took part in the on-going TACIS project where JRC Ispra will supply equipment and know-how to strengthen the safeguards activities at the Ulba nuclear fuel fabrication plant in Kazakhstan.

Paul realized the great importance of human resources, like nuclear scientists and engineers, in the work for Non-Proliferation of nuclear materials and weapons. He was one of the initiators of the Swedish support to the International Science and Technology Center (ISTC). He actively supported and took part in ISTC's work to find peaceful tasks for former nuclear weapons scientists and engineers in Russia and Ukraine.

From 1996 Paul worked directly under the leadership of SKI's Director General to give support in the field of Nuclear Non-Proliferation to States in Central and Eastern Europe, an area where Paul, in his typical way, was one of the first to identify the need for reinforcement. Paul was in the midst of this significant work when he all too soon passed away.

During his long career at SKI, Paul made his mark within safeguards, on the national and international front. Through his enthusiasm, initiative and commitment, Paul has made significant achievements, at the same time that he has been controversial. Those of us who worked with him know that he was an excellent leader, always paving the way, while supporting and encouraging his colleagues.

## New from ESARDA

### New Decisions of the ESARDA Steering Committee Appointments and Awards

ESARDA rules allow the assignment of the title "Honorary Member" of the Association to those who have actively contributed for a number of years to the promotion and improvement of the European Research and Development Association.

On the occasion of the retirement of Carlo Foggi, Secretary of the Association for 13 years and in recognition of his leadership and active participation in working groups, the Steering Committee in Bruges on May 11, 2001 decided unanimously to assign the title of "**Honorary Member**" to **Carlo Foggi**. He is the fourth person that has been awarded and follows S. Finzi, L. Stanchi and R.J.S Harry.

In recognition of services which have been provided to the Association and in order to further motivate persons to actively promote the objectives of ESARDA, the Steering Committee in its meeting at Bruges also decided to establish an "ESARDA Distinguished Service Award". This award

will be assigned to persons, who through their strong commitment have contributed significantly

- *to the implementation and fulfilment of the objectives of ESARDA in accordance with the Agreement of the Association.*
- *to the promotion of safeguards and nuclear materials management by innovative and professional input and continuous support.*

The Steering Committee is pleased to assign the first "**ESARDA Distinguished Service Award**" to **Marc Cuypers** in recognition of his outstanding service for more than 30 years of service.

For future symposia of the Association, the Steering Committee discussed also the creation of an award to recognise the excellence of presentations during the open annual symposium.

#### **Marc Cuypers**

Marc Cuypers obtained his degree in Chemistry from the University of Liège (Belgium) in 1960. He presented his PhD in "Sciences Physiques" at the University of Paris (Sorbonne) in 1964. He then spent 20 months as Assistant Professor at the Texas A&M University at College Station, where he was in charge of a project on "the in-situ analysis of the elemental composition of moon rocks by neutron activation analysis".

In 1968 he joined the Joint Research Center (JRC) at Ispra. During the period 1968-1978 he established the Non-Destructive measurements laboratory for nuclear safeguards.

In 1978, he was appointed Program Manager of the nuclear safeguards program of the JRC. In this function he developed a network of international co-operation in the field of nuclear safeguards R&D and negotiated cooperation Agreements with the US Department of Energy (USDOE), the International Atomic Energy Agency (IAEA) and the Japan Atomic Energy Research Institute (JAERI).

In 1989 he was appointed Head of Division of nuclear safeguards. He launched the creation of several test and training facilities, such as PERLA for Non-Destructive meas-

urements, TAME for mass and volume determinations and TEMPEST for the environmental testing of instruments and devices. In 1994 he was strongly involved in the development of a strategy and important cooperation program with the Russian Federation in the field of nuclear material accountancy and control in the framework of TACIS.

In 1995 he launched a new project on the electronic identification of livestock, which will be concluded by the end of 2001. This project is aimed towards the establishment of a reliable traceability system of the EU livestock, which comprise more than 300 million animals.

In addition to his safeguards responsibilities Marc Cuypers was appointed Deputy Director of ISIS in 1997.

Since 1970, he has been involved very actively in the European Safeguards Research and Development Association (ESARDA). He acted for nearly 10 years as Scientific Secretary of the Association. He was for many years a member of the Non-Destructive working group and became the first chairman of the MOX working group. He has been an EC member of the ESARDA Co-ordinators Committee was later appointed EC member of the Steering Committee. He acted twice as chairman of the Association

(in 1994 and in 2000). In his function as chairman of the ESARDA Reflection Group (1999-2001) he was strongly involved in the promotion and adaptation of the structure and activities of the Association to the new challenges in the field of nuclear non-proliferation.

In July, 2000 he was assigned at New Orleans the "Distinguished Service Award" by the US Institute of Nuclear Materials Management (INMM).

When Marc Cuypers retired in March 2001, he was attributed by the European Commission the title of "Honorary Director". His nomination to receive the first Distinguished Service Award of ESARDA in May 2001 is a logical consequence of his professionalism and continuous contribution to the Association.



## *Carlo Foggi*

Carlo Foggi has been the Secretary of ESARDA for nearly 13 years, from November 1988 to September 2001. During this long period he had the opportunity to know - and interact with - most of the people working in the area of nuclear safeguards, therefore his name is familiar to the readers of the ESARDA Bulletin. He retired from work at the end of September 2001, although he still collaborates with the Joint Research Centre of the European Commission on specific projects.

Carlo Foggi was born 66 year ago in Colico – a town located in the heart of the Italian Alps. He carried out his primary and secondary studies in Arezzo, a famous and ancient art town of Tuscany – central Italy. His higher education was carried out at the "Politecnico" University of Milan. There, he won a competition to join the then recently founded branch of Nuclear Engineering, where he completed his studies under the guidance of Professor Stefano Silvestri - one of the fathers of the Italian nuclear industry – and became one of the first 7 Italian nuclear engineers. At that time, three nuclear power stations were already operating in Italy – one BWR (Garigliano), one PWR (Trino Vercellese) and one Gas-Graphite Reactor (Latina) - making this country a very promising land for nuclear industry.

In 1959 Carlo Foggi joined the Agip Nucleare, a branch of the Italian oil Company ENI dealing with nuclear business, where he worked at the neutron design of the core of an advanced gas-graphite reactor with enriched fuel. In 1960 he joined the European Atomic Energy Community (one of the European institutions, now part of the European Union) and there he remained for the subsequent 40 years.

He was attached to the Joint Research Centre, first in Brussels, then in Ispra (Italy).

At the Commission Carlo Foggi's activities were initially related to R&D. Until 1970 he dealt with reactor core neutron design (and related safety aspects) for a number of dif-

ferent concepts that were studied by the JRC: heavy water reactors refrigerated with organic liquid (ORGEL project), PWRs, BWRs, MTRs, research reactors for dynamic excursion tests, naval reactors (in collaboration with the Dutch ECN), etc. At the same time, he developed reactor neutron physics codes and taught reactor neutron calculation methods to technicians of selected European industries.

From 1970 he was involved in the technical developments of the European safeguards system and participated in the establishment of the first "facility attachments" for the European reactors. At the same time he turned his attention to the emerging field of the "isotopic correlations", a technique that studied the relations between the abundance of the various isotopes existing in irradiated fuels (including fission products). This technique can be used for consistency check of declarations made by the operators of the fuel cycle, but also as a means to trace the history of irradiated nuclear fuels. Due to the expertise developed in this area, in 1970 Carlo Foggi was appointed Convenor of the ESARDA Working Group on "Isotopic Correlations". When this Group terminated its activities, ESARDA created the Working Group for "Reprocessing Input Verification", of which Carlo Foggi was appointed Convenor. Under his leadership, the two ESARDA Groups participated in - or organised - many studies and international Exercises, such as: the Mol-IV and the RITCEX experiments at the Belgian reprocessing plant of Mol, the ICE experiment on data of the German reprocessing plant WAK of Karlsruhe, the CALDEX experiment for advanced tank calibration in Karlsruhe, the Isotope Bench Mark experiment on data from the French reprocessing plant COGEMA of Cap de la Hague. During this period he also organised many "Ispra Courses" on safeguards. These were courses of one or two weeks, addressed to nuclear facility operators, national and EURATOM nuclear inspectors, R&D people and national authorities, given at the Commission's Centre of Ispra (Italy). Attendance from the European safeguards community was very large.

From 1980 Carlo Foggi was also involved in non-nuclear areas, namely in the Commission's project "Energy Bus" for energy saving in the small and medium industry, as well as in the development of data bases (then an emerging branch of informatics). At the same time he started dealing with statistical aspects of accountancy and sampling of nuclear materials, as well as in the theory of error propagation in measurements performed at reprocessing plants. In these two areas he published many studies that were to improve practical applications. Also in this period he contributed to the safeguards studies of the German fast reactor KALKAR.

In November 1988 he became the Secretary of ESARDA, and this is the part of the story that the reader of the

Bulletin already knows. In 1994 he also took on the additional tasks of being the Editor of the Bulletin, the Scientific Secretary of the ESARDA Meetings and the Editor of the Proceedings of these meetings (tasks that were previously performed by Mr. Luciano Stanchi). He had a very strong personal dedication to the Association and contributed significantly to increasing its visibility. ESARDA Symposia and Workshops set nowadays a standard of excellence, and top organisations, like the International Atomic Energy Agency and the Institute of Nuclear Material Management, are happy to co-organise with us specific events of theirs.

As the Chairman of ESARDA, it is my pleasure to thank Carlo for all his contributions to the success of our Association and wish him the best for his future.

## A note from the Editor

On the 1st October 2001, Louis-Victor Bril became Secretary of ESARDA taking on all the duties of the ESARDA Secretariat:

He was trained as an engineer.

He has been involved in all aspects of nuclear activities:

- safety system engineer at a Nuclear Power Plant during the building phase; engineer in the commissioning team,
- engineer at a design office, including management of R&D,
- relations with European nuclear industry, participation in the development of European Commission policy in the nuclear industry field,
- nuclear projects management for the European Union Tacis and Phare programmes, dedicated to enhancing the safety of nuclear installations in candidate countries and in the NIS.

He joined the Joint Research Centre of the European Commission two and half years ago. He is currently managing other projects in the field of safeguards and non-proliferation.

