

Editorial

The 13th issue of the Bulletin aims to give more information about ESARDA and its organization. Two tables containing a block diagram of the ESARDA structure and the list of relevant names in ESARDA replace similar tables in issues No. 1 and No. 5 respectively. The full list of ESARDA partners is also included in this issue. We are glad to welcome two new partners which will soon join ESARDA officially. A calendar of ESARDA meetings is added and will be published regularly in future issues of the Bulletin.

Scanning the issue, the reader will first find comments on the 9th ESARDA Symposium held in London on 12-14 May 1987, then some important news from the ESARDA Working Group on Destructive Analysis. The publication of the 1988 Target Values for Random Uncertainties and Element Assay replaces and extends a similar paper published in issue No. 6 on the 1983 Target Values. An accompanying paper by Mr. Deron refers to examples in the Operator-Inspector differences on the use of the values.

Several other papers are included in this issue. A paper on cooperation between the Italian Committee (ENEA) and the Commission of the European Communities (JRC-Ispra) deals with R&D for Safeguards purposes. A paper from the Euratom Safeguards Directorate deals with the use of the shipper/receiver differences in the facility accountancy. The outcome of a training course organized at JRC-Ispra for IAEA and Euratom inspectors are also reported.

Finally two papers which should have been presented at the ESARDA Symposium of London and, which for various reasons arrived too late, are included in this issue.

The issue is closed by a mournful note. It concerns the premature death of the ESARDA secretary, J. Ley, which affected us all. We will miss him very much.

L. Stanchi

News about ESARDA

ESARDA is an association of European organizations formed to advance and harmonize research and development of safeguards. It also provides a forum for the exchange of information and ideas between nuclear facility operators and safeguarding authorities.

Its partners as of 1st January 1987 were :

- The European Atomic Energy Community
- The Kernforschungszentrum Karlsruhe (KfK) - F.R. of Germany
- The Centre d'Etude de l'Energie Nucléaire - Studiecentrum voor Kernenergie (CEN/SCK) - Belgium
- The Comitato Nazionale per la Ricerca e per lo Sviluppo dell'Energia Nucleare e delle Energie Alternative (ENEA) - Italy
- The Stichting Energie Onderzoek Centrum Nederland (ECN) - Netherlands
- The United Kingdom Atomic Energy Authority (UKAEA) - United Kingdom
- The Energistyrelsen (ENS) - Denmark
- The Commissariat à l'Energie Atomique (CEA) - France
- British Nuclear Fuels plc (BNFL) - United Kingdom

The accession of two new partners has been recently approved by the Steering Committee:

- Centro de Investigaciones Energéticas Medio Ambientales y Tecnológicas (CIEMAT) - Spain
- Kernforschungsanlage Jülich (KFA) - F.R. Germany

Calendar of recent and future ESARDA meetings

Steering Committee : Ispra, Italy, 8 October 1987

Board : Brussels, Belgium, 15 September 1987

Coordinators : Ispra, Italy, 23-24 September 1987

NDA WG : Karlsruhe, February 1988 (combined with RIV)

DA WG : Sellafield, U.K., 10-11 June 1987

Karlsruhe, F.R. Germany, 2 May 1988

C/S WG : London, U.K., 11 May 1987

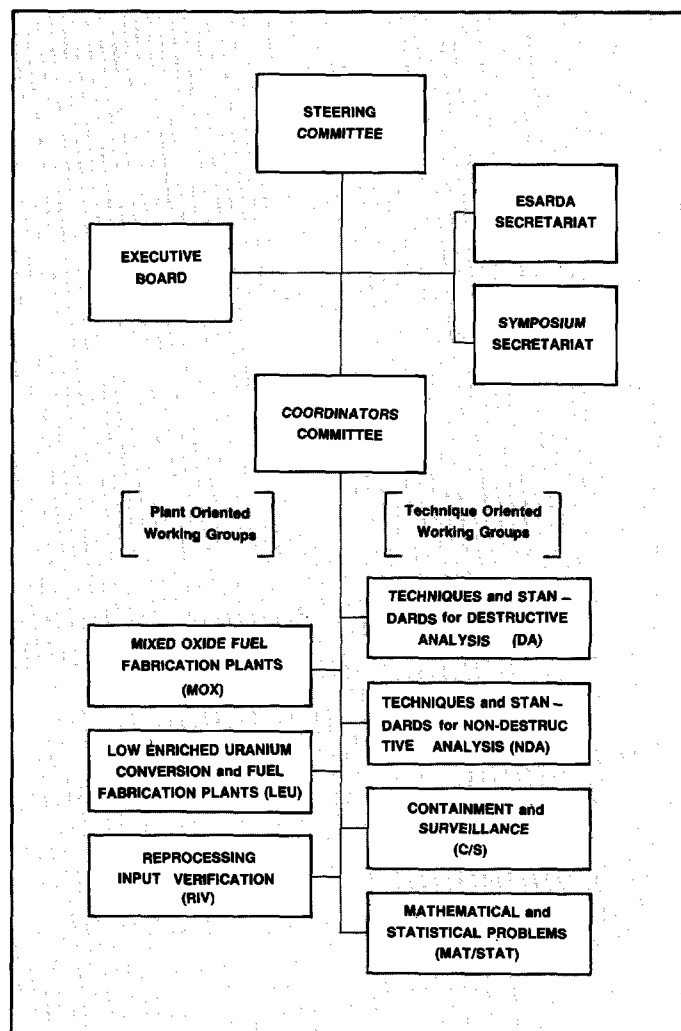
Petten, Netherlands, 3-4 November 1987

LEU WG : Salamanca, Spain, 8-9 September 1987

RIV WG : Karlsruhe, February 1988 (combined with NDA)

* Workshop on Computerized Information System :
Luxembourg, 8-9 Sept. 1987

* 10th Annual ESARDA Meeting (restricted participation) :
Karlsruhe, F.R. Germany, 3-5 May 1988



Retirement

We wish to thank Mr. E. Bastrup-Birk who left ESARDA because of retirement. He has been the Danish member of ESARDA since its beginning. Everybody remembers his lovely character, his organizing capability and his always constructive attitude. We will miss him.

Who's Who in ESARDA ?

(as of 1st September 1987)

Chairman 1987 *R.H. Kroebel*, KfK Karlsruhe, F.R. Germany
 Appointed chairman 1988 *G. Stiennon*, CEN/SCK Mol, Belgium
 Secretary to be appointed
 Permanent Symposium
 Scientific Secretary *L. Stanchi*, CEC, JRC-Ispra, Italy

ESARDA Steering Committee

C. Beets, Belgium
J.M. Boudier, EDF, France
F. Brown, Dept. of Energy, U.K.
M.J.C. Charrault, CEC Brussels, Belgium
M. Cuypers, CEC, JRC-Ispra, Italy
S. Finzi, CEC Brussels, Belgium
C. Fizzotti, ENEA Casaccia, Italy
P. Frederiksen, Risoe, Denmark
R. Gerstler, BMFT, F.R. Germany
W. Gmelin, CEC, Safeguards Directorate, Luxembourg
B.W. Hooton, UKAEA, Harwell, U.K.
K.L. Huppert, DWK Hannover, F.R. Germany
R. Kroebel, KfK, Karlsruhe, F.R. Germany
J.M. Leblanc, Belgonucléaire, Belgium
G. Le Goff, CEA Paris, France
B. Lerouge, CEA Fontenay-aux-Roses, France
R.D. Marsh, BNFL Risley, U.K.
A. Petit, COGEMA, France
F. Pozzi, ENEA Saluggia, Italy
G. Stein, KFA Jülich, F.R. Germany
G. Stiennon, CEN/SCK Mol, Belgium
P. Vanni, ENEA DISP, Italy
W.L. Zijp, ECN Petten, Netherlands
V.H. Braatz, VDEW, F.R. Germany (Permanent observer)

ESARDA Board

S. Finzi, CEC Brussels, Belgium
C. Fizzotti, ENEA Casaccia, Italy
P. Frederiksen, Risoe, Denmark
B.W. Hooton, UKAEA Harwell, U.K.
R. Kroebel, KfK, Karlsruhe, F.R. Germany
B. Lerouge, CEA Fontenay-aux-Roses, France
G. Stiennon, CEN/SCK Mol, Belgium
W.L. Zijp, ECN Petten, Netherlands
 Secretary, to be appointed

ESARDA Coordinators

W.G. Bahm, KfK, Karlsruhe, F.R. Germany
R. Carchon, CEN/SCK Mol, Belgium
M. Cuypers, CEC, JRC-Ispra, Italy
F.V. Frazzoli, University of Rome, Italy
P. Frederiksen, Risoe, Denmark
P. Guillot, CEA Fontenay-aux-Roses, France
R. Haas, CEC, Safeguards Directorate, Luxembourg
R.J.S. Harry, ECN Petten, Netherlands
B. Patrick, UKAEA Harwell, U.K.

Working Group Convenors

Techniques and Standards for Non-Destructive Analysis (NDA)

R.J.S. Harry, ECN Petten, Netherlands

Destructive Analysis (DA)

P. De Bièvre, CEC, JRC-Geel, Belgium

Reprocessing Input Verification (RIV)

C. Foggi, CEC, JRC-Ispra, Italy

Containment and Surveillance (C/S)

F.J. Walford, UKAEA Harwell, U.K.

Low-Enriched Uranium Conversion/Fabrication Plants (LEU)

P.P.A. Boermans, FBFC, Belgium

Mathematical and Statistical Problems (MATH/STAT)

M. Franklin, CEC, JRC-Ispra, Italy

MOX Fuel Fabrication Plants (MOX)

M. Cuypers, CEC, JRC-Ispra, Italy

ESARDA Bulletin Editor

L. Stanchi, CEC, JRC-Ispra, Italy

*Mr. R. Kroebel, present chairman of ESARDA, was invited to make a presentation at the plenary session of the 28th Annual Meeting of the INMM, Institute of Nuclear Material Management, Newport Beach, California, USA, on 13 July 1987.
The abstract of the paper is reported below.*

The Role of ESARDA and its Current R&D Efforts

R. Kroebel, Chairman of ESARDA

KfK, Karlsruhe, F.R. Germany

W. Bahm, Convenor of ESARDA Coordinators

KfK, Karlsruhe, F.R. Germany

M. Cuypers, CEC, JRC-Ispra, Italy

Abstract

The peaceful use of nuclear energy has substantially increased worldwide during the last decade. At present about 400 power stations are connected to the grid providing a total capacity of 280 GWe. According to present day projections a further increase is to be expected.

The generation of electricity by nuclear energy is inherently linked to the production of plutonium, presently worldwide about 75 t per year. Some countries already have or are going to recycle plutonium commercially in thermal and fast reactors. The related reprocessing capacity in countries of the European Community will increase from its current value of 400 t heavy metal per year

to more than 3000 t within the next decade.

This situation will result in a challenge for safeguards mainly for two reasons. Firstly, its recourses are not likely to be increased accordingly, therefore more emphasis has to be put on cost/effectiveness. Secondly, new techniques such as long term storage and remote handling are supposed to be applied requiring adequate safeguards measures. Therefore the present day status of safeguards techniques will need to evolve in order to master all future safeguards aspects.

In this connection ESARDA plays the role of an important platform for exchange of technical information and initiation as well as

coordination of international R&D efforts. The ESARDA Symposium in London through 12-14 May 1987 proved to be very successful in that sense.

The activities of the ESARDA working groups are mainly focussed on specific questions arising from safeguards practice such as estimation of measurement errors to be expected and comparison exercises.

An outline of current R&D efforts of the working groups as well as a report on the London Symposium is presented in this paper.

Some Impressions of the London Symposium 1987

R. Kroebel, W. Bahm

KfK, Karlsruhe

L. Stanchi, CEC, JRC-Ispra

The 9th Annual Symposium on Safeguards and Nuclear Materials Management was held in London from 12th to 14th May 1987 at the Queen Elizabeth II Conference Centre in the heart of London. Two hundred attendees enjoyed the advantages of the wonderful new and modern building and the direct view to the venerable Westminster Abbey. Beyond that a variety of buildings and monuments invited the participants to enjoy historical sights.

The structure of ESARDA-symposia has been changed by the Steering Committee insofar as international-open symposia alternate with internal symposia where essentially only the ESARDA-community meets to stimulate cooperation within the association. The London symposium was the first open one after a two years period following the Liège symposium in 1985. No specific topic inside safeguards has been put as headline, so practically any topic could be addressed within 12 sessions and 2 poster sessions.

The meeting opened with three invited papers dealing with present and future safeguards aspects and R&D topics. It was noted that substantial contributions to R&D are elaborated at JRC-Ispra, but also by other JRC installations and by cooperation within the EC members through ESARDA working group activities. Besides that PERLA will offer a European training laboratory for inspectors and interested customers. Regarding new R&D topics the future importance of C/S measures for long term storage and large bulk handling facilities has been emphasized. Some consideration was given to the introduction of long term criteria by the IAEA and it was stressed that this will be an important development in the next 10-15 years enhancing the inclusion of safeguards features in the design of new plants. On the IAEA side it was explained that these criteria are intended to improve efficiency, effectiveness and transparency of IAEA safeguards. In connection with cost/effectiveness of safeguards the possibilities of random sampling of safeguards activities and increased use of unannounced inspections was put forward by the IAEA. This appears to be an interesting concept which is not entirely based on detection power but also takes into account the deterrence

generated by such kind of inspections.

The second session was devoted to the theme "Safeguards Perspectives". On the US-DOE side the importance of further R&D both for national and international safeguards was stressed and an integrated system was suggested. The increasing use of MOX fuel, storage of fuel assemblies and laser enrichment are seen as present and future safeguards concerns. An interesting comparison of the role of NDA and C/S for safeguards was made in the German contribution emphasizing practical advantages of C/S for example in dry storage facilities. Furthermore difficulties of independent interpretation of NDA-measurement data and impact on plant operation as well as safety risks were elaborated. Taking into account other papers dealing with C/S measures it seems that the role of C/S will be subject to further technical and political discussions. Another interesting paper was that of the IAEA on the development of manpower and number of inspections from 1980 to 1986. It turned out that an increase of 70% and 100% respectively has occurred. The paper closed with the suggestion that the IAEA's experience in establishing and operating an international inspection scheme could also be utilized within the framework of arms control.

Four sessions were devoted to various measurement techniques and their application to safeguards covering areas such as:

- trace analysis of uranium and plutonium
- error sources in Pu-determination
- inter-laboratory comparisons
- Pu-determinations with Ge-detectors, whereby in particular questions related to a) data evaluation methods for Pu-spectra, b) branching ratio evaluation, needed for further Pu-measurements, and c) performances of gamma spectrometers in in-field use
- calibration of neutron measurement systems
- a new technique of laser-induced breakdown spectroscopy
- passive gamma-ray assay of ^{235}U solutions
- advanced software system for measurement data evaluation, and
- NDA applications in enrichment plants.

Related to the latter subject five papers

were presented considering various measurement concepts. In conclusion the experiments carried out have demonstrated that confirmation of LEU presence in the product streams of centrifuge enrichment plants poses plant specific problems requiring specific solutions.

The papers presented in the session on Near-Real-Time Accountancy (NRTA) demonstrated that its successful application depends on many parameters, which in some cases are even counterproductive, such as detection probability, timeliness, false alarm probability, measurement errors and diversion scenarios. Therefore there exists no optimal test but an adequate combination of several tests seems to be a practicable concept. For the application of NRTA a computer program has been developed which enables sensitivity studies to be carried out. Examples of applying specific statistical tests to model plant data were shown. In another paper on the basis of a plant model it was demonstrated that NRTA is superior to conventional accountancy. In the last contribution to this session an intelligent knowledge-based system was proposed in order to accumulate experience gained in reprocessing campaigns and by this way to assist resolving anomalies.

From the papers presented it can be concluded that NRTA is a promising method and substantial R&D effort in various establishments are undertaken in order to make a tool out of it ready for routine application.

The session on "Data Evaluation Methodology" covered a wide range of quantitative problems. It appears that quantification of safeguards effectiveness is still a difficult question. Besides more generic subjects such as evaluation of verification data and Shipper/Receiver differences and modelling of NDA measurements reports on measurement control of k-edge data and on calibration procedures for the PHONID instrument were presented.

In the last session on "Material Control, Accounting and Information System" examples of specific accounting systems implemented in individual plants were described and experience in routine application was reported. Furthermore contributions dealing with stratification of nuclear material in a material balance area, transit control and code verification were

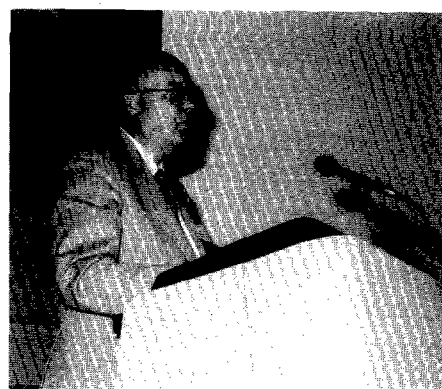


The start of the symposium.

From left to right : N. Tuley, R. Kroebel, J.P. Contzen, D.I. Morphet, A. von Baeckmann, L. Stanchi



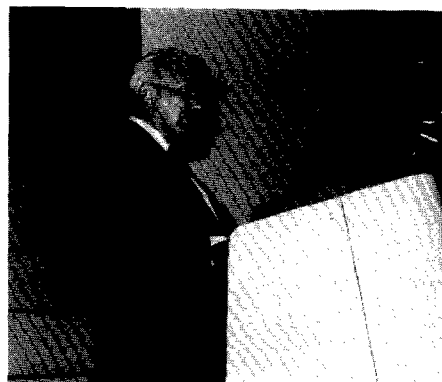
A view of the Congress Hall



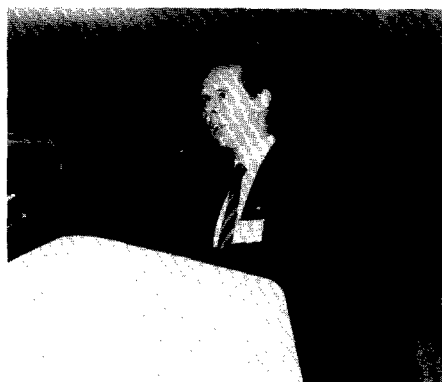
Dr. R. Kroebel, chairman of ESARDA, opening the Symposium



Mr. J.P. Contzen, Director General of the Joint Research Centre of the Commission of the European Communities



Dr. A. von Baeckmann, IAEA, Vienna



Mr. D.I. Morphet, Head of the Atomic Energy Division of the U.K. Department of Energy, Member of the IAEA Board of Governors

presented.

Besides these sessions the symposium also involved poster presentations many of them accompanied by an exhibit of real instruments, devices or PCs. Therefore, poster presentations should remain an integral part of following symposia, gathering more and more their own momentum. They should no longer be regarded as second-hand information, but as particularly suitable for maximum impact on a qualified part of the participants.

The poster sessions extended the presentations in some areas, i.e. Containment and Surveillance; Data Evaluation Methodology; Material Control, Accountancy and Information Systems; and Measurement Techniques. The allocation of papers to oral or to poster presentations was decided by the technical programme committee in which all the ESARDA working groups were represented. Some papers were attributed to poster sessions on explicit request of the authors. The first three areas of the poster sessions were an extension of the corres-

ponding oral sessions and mostly contained papers dealing with particular solutions for which direct contact between authors and participants was considered highly suitable. The session on Measurement Techniques was assembled in three parts, Destructive Analysis, Scrap and Waste, and Non-destructive Assay and was aimed to group together presentations on similar subjects in order to better stimulate discussions and reciprocal acquirement of knowledge.

Besides the scientific part of the conference which was organized very excellently by our British colleagues and the ESARDA secretariat, the social part was taken care of as well. In particular the reception in the Banqueting Hall was an event which certainly was very enjoyable for all participants.

In conclusion it can be stated that the London symposium in the spirit of ESARDA was an excellent forum for exchange of information and views on all safeguards areas.

Activities of the ESARDA Working Groups

Destructive Analysis

The ESARDA Working Group on Techniques and Standards for Destructive Analysis held its Annual Meeting at Sellafield (U.K.) on 10-11 June 1987.

As usual, the meeting lasted 1.5 days only with 0.5 day for site and laboratory visits. Because of the remoteness of the site 2 travel days had to be added requiring almost 1 week in total. Despite this, the meeting recorded the highest attendance ever in the history of the W.G. : 31 with all major nuclear analytical laboratories of the EC represented plus several observers.

The agenda carried several important items all of which were brought to an end

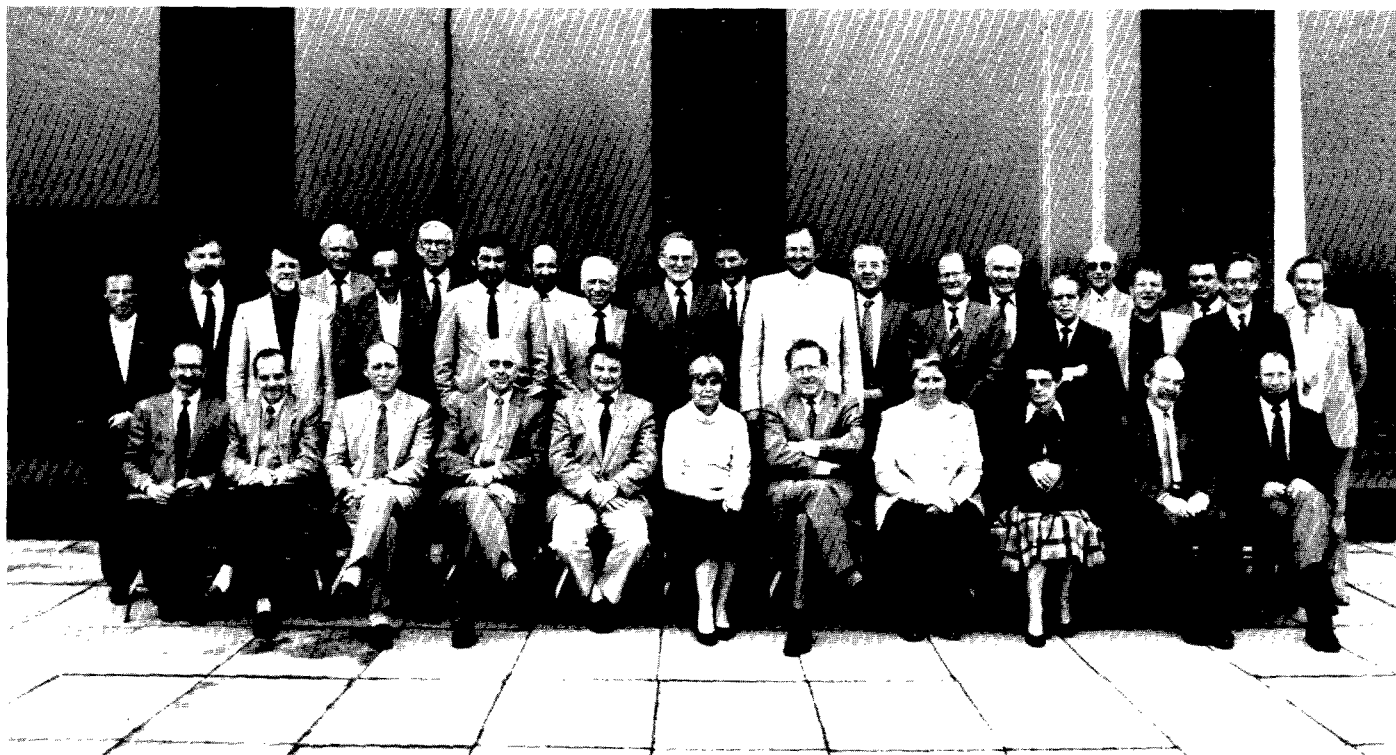
or to conclusions :

- achievement of a document on "Target Values for random uncertainties for sampling and assay of nuclear materials" which is expected to help to set goals for EC and IAEA safeguards verification measurements;
- results of the "Regular European Interlaboratory Measurement Evaluation Programme" (REIMEP) for UF_6 ;
- results of REIMEP for PuO_2 ;
- a new (U.K. introduced) concept on "Blind Analysis as a Safeguards Verification Technique" was discussed on its potential to revolutionize present

verification measurement schemes;

- both Euratom Safeguards Directorate and IAEA presented experience with the 1987 Target Values for measurement uncertainties which the Group had established in its last meeting.

Two Spanish nuclear analysts attended the meeting for the first time (Spain joined ESARDA recently). Former ESARDA President, Brian Hooton, British member of the Steering Committee, also followed the meeting throughout and participated in the discussions.



1987 Annual Meeting of ESARDA WG DA, Sellafield (U.K.), 10-11 June 1987

Random Uncertainties in Sampling and Element Assay of Nuclear Materials. Target Values 1988

Achievable Uncertainties when Nuclear Materials are Sampled for Element Content and Assayed by Destructive Analytical Methods

P. De Bièvre, CBNM, CEC, JRC-Geel, Belgium

S. Baumann, ALKEM, Hanau

R. Schott, COGEMA, Marcoule

J.C. Dalton, BNFL, Sellafield

R. Berg, WAK, Karlsruhe

P. De Regge, SCK/CEN, Mol

T. Görgenyi, NUKEM, Hanau

M. Höflich, NUKEM, Hanau

S. Deron, IAEA, Vienna

E. Kuhn, IAEA, Vienna

E. Mainka, KfK, Karlsruhe

R.P. Bradshaw, BNFL, Springfield

C. Pietri, US-DOE, Chicago

endorsed by the Working Group on Techniques and Standards for Destructive Analysis of the European Safeguards and Development Association (ESARDA) and the Committee on analytical chemistry and laboratory measurement control of the Institute of Nuclear Materials Management (INMM Committee 5.1 - USA), and written on behalf of these groups.

Table 1 - Analytical Methods and Typical Amounts of Sample Needed to Perform one Measurement taken from Ref. /2/

Code	Method	Typical amount of element required for a single measurement (excluding sample preparation) (a)			Unit
		U	Th	Pu	
01	Titrimetry	20 - 100	50 - 100	5 - 50	mg
02	Coulometry	2 - 20	—	2 - 10	mg
03	Gravimetry	2 - 20	0.5 - 1	0.2 - 3	g
04	X-ray fluorescence	0.1 - 30	0.1 - 30	0.1 - 30	mg
05	Isotope Dilution Mass Spectrometry	10 - 1000	10 - 1000	1 - 1000	µg
06	Spectrophotometry	20 - 500	5 - 250	(b)	µg
07	Fluorometry	2 - 500	—	—	ng
08	Alpha Counting	2 - 250	—	0.1 - 1	µg
09	K-edge Densitometry	0.3 - 1	—	0.3 - 1	g
21	Gas Mass Spectrometry	20	—	—	mg
22	Thermal Ionization Mass Spectrometry	1 - 1000	10 - 1000	1 - 1000	µg
23	Gamma Spectrometry	0.1 - 1	—	—	g
24	Alpha Spectrometry	—	—	0.1 - 1	µg

Abstract

A list of "1988 Target Values for Random Uncertainties in Sampling and Element Assay" has evolved gradually from discussions within the ESARDA Working Group on Techniques and Standards for Destructive Analysis and from within the committee on analytical chemistry and laboratory measurement control of the Institute of Nuclear Materials Management (INMM Committee 5.1 - USA). The magnitudes of the random sampling and measurement uncertainties which may be encountered in fissile element assay, and their impact on the total random uncertainties have been estimated.

The paper considers eleven different materials of particular relevance to trade and safeguards, encountered as feeds or products in spent fuel reprocessing and fuel fabrication.

For each material, a table is presented, specifying the relevant Target Values, the minimum amount of sample for which the values apply, and the major precautions to be taken in sampling by the facility operator and the safeguards inspector.

(a) The sample preparation is not considered except for the spiking and chemical treatment in isotope dilution analysis (method 05)

(b) Spectrophotometry:

- Direct measurement at 830 nm of Pu (VI) 0.1 - 10 mg
- Colorimetry 1 - 100 µg

The code identification of the methods are the same as in Ref. /2/.

Table 2 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of LEU-UO₂ Powders, Nuclear Grade

Target Value for the "Total Random Uncertainty": $TTR = \sqrt{TRS^2 + TRM^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.15	10-30	0.20	0.25	10	0.20	0.25
Coulometry (02)	0.13	10-30	0.20	0.25	10	0.20	0.25
Gravimetry (03)	0.10	10-30	0.20	0.22	10	0.20	0.22

Each table must be considered as an indivisible entity. The Target Values for sampling will not be attained unless the recommended minimum amount of material is used for the assay and the precautions specified in the table are taken.

All users of this document are therefore strongly cautioned never to quote a Target Value for sampling without referring simultaneously to the underlying prerequisites stated in the relevant table.

In order to complete its examination of the uncertainties in the accountability measurements, the working group may attempt at some point in the future, to assay the problem of uncertainties in bulk measurements and for the systematic component of the sampling uncertainties.

The authors welcome any comment or suggestion in this respect. Especially plant-specific Working Groups and Committees are expected to examine these problems and let the authors know about their conclusions or proposals.

1. Introduction

The Working Group on Techniques and Standards for Destructive Analysis (WGDA) of the European Safeguards Research and Development Association (ESARDA), consisting presently of representatives of 37 nuclear analytical laboratories, has long been concerned about defining realistic performance characteristics of destructive analysis techniques.

One of the goals of the Working Group is: "to evaluate and recommend criteria for destructive analysis of nuclear materials for use by plant operators and Safeguarding Authorities".

In 1986 contact was taken up with INMM President, Mrs. Y. Ferris, in order to associate USA laboratories to the examination of the abovementioned problems.

Some of the most important and most badly needed criteria are those to be used for evaluating the results of quantitative measurements of fissile isotopes and elements. The Working Group and INMM Committee have recognized and discussed this problem at several meetings and decided that it was appropriate to set reasonable levels of performance as "targets" for nuclear analytical laboratories working under routine conditions.

A first result of these discussions were the "1983 Target Values for Uncertainty Components in Fissile Element and Isotope Assay" /1/. This list was intended to provide

The sample should be taken preferably with a proportional sampling device during the filling of the drums. Alternatively, samples may be drawn from the drums using a sampling thief.

The sample should be transferred to a glass bottle which can be capped air tight and has a stable tare. The free volume above the powder must be kept as small as possible. If the sample cannot be analyzed immediately, a weight control must be carried out which provides an accurate control and correction of the chemical changes which may occur between sampling and analysis. An effective procedure involves weighing the tare and gross mass at the time of sampling, and weighing the gross mass again before analysis commences.

Generally the O/U-ratio in sinterable UO_2 - powder is adjusted between 2.06 and 2.15. If the ratio is lower the TRS-value is possibly larger.

Table 3 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of LEU- UO_2 Sintered Pellets

Target Value for the "Total Random Uncertainty": $\text{TTR} = \sqrt{\text{TRS}^2 + \text{TRM}^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.15	7-20	0.05	0.16	7-20	0.05	0.16
Coulometry (02)	0.15	7-20	0.05	0.16	7-20	0.05	0.16
Gravimetry (03)	0.05	7-20	0.05	0.07	7-20	0.05	0.07

The UO_2 - pellets are sintered at high temperature ($\sim 1600^\circ\text{C}$ in H_2 -atmosphere) so the O/U-ratio is between 2.00 and 2.01.

To achieve the TRS value in the case of pellets smaller than 7 g, it is recommended that more than one pellet is taken and is analyzed independently.

Table 4 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of HEU- UO_2 Powders, Nuclear Grade

Target Value for the "Total Random Uncertainty": $\text{TTR} = \sqrt{\text{TRS}^2 + \text{TRM}^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.15	10	0.20	0.25	2x(0.5-1.0)	0.30	0.34
Coulometry (02)	0.15	10	0.20	0.25	2x(0.5-1.0)	0.30	0.34
Gravimetry (03)	0.10	10	0.20	0.22	2x(0.5-1.0)	0.30	0.32

Preferably the sample should be taken with a proportional sampling device during the filling of the drums. Alternatively samples may be drawn from the drums using a sampling thief.

The sample should be transferred to a glass bottle which can be capped air tight and has a stable tare. The free volume above the UO_2 - powder must be kept as small as possible. If the sample cannot be analyzed immediately, a weight control must be carried out which provides an accurate control and correction of the chemical changes which may occur between sampling and analysis. An effective procedure involves weighing the tare and gross mass at the time of sampling, and weighing the mass again before analysis commences.

If the inspector's samples are not larger than 0.5 - 1 g because of shipment restrictions, they must be taken directly into the vessel in which they will be dissolved quantitatively without further transfer at the verification laboratory. They should be weighed with an accuracy of 0.5 mg or better at

estimates of the capability which could reasonably and realistically be expected at that time from analytical laboratories. A revised list, known as "The 1987 Target Values for Uncertainty Components in Fissile Isotope and Element Assay", has now been published /2/ reflecting the experience gained in the use of the concept and the progress observed in analytical performance since 1983.

It is, however, universally recognized that uncertainties associated with the sampling operation, can also contribute significantly to the overall uncertainty of a measurement process. These considerations led to the preparation of a combined list of "Target Values for Random Uncertainties in Sampling and Element Assay". These Target Values which are presented in this paper, are valid from 1988 onwards and should be updated as is warranted by changes in the state of the practice.

This paper attempts to quantify these sampling uncertainties and their impact on the total random uncertainty of the element assay. As in the earlier paper /1,2/ the intention is to describe the state-of-the-practice which should be reasonably achievable under routine conditions.

Sampling errors caused by the heterogeneity of a material are expected in the long run to be a random effect, provided that the sampling process is random /3/. The experimental data which were available to the Working Group provide estimates of random sampling uncertainties only. To estimate the systematic errors in sampling and their fluctuations, one should have available reference materials in large amounts and of well known homogeneity (or heterogeneity) with respect to the quantity under investigation, and submit them to the sampling procedure and devices actually used. No such experimental evidence was known to the Group so that the systematic components of sampling uncertainties are not included in the present discussion. However the Group recognizes that systematic errors may exist in sampling and are likely to be the major problem as the sampling process becomes less random. This matter might be a topic of the next deliberations of the Working Group and Committee.

The sampling errors in isotopic analyses will be addressed at another occasion because, while their impact is negligible for most bulk materials in the fuel cycle, the same cannot be assumed when analyzing dissolver solutions, scraps, wastes or solid materials. Sampling errors may be observed especially in isotopic analyses of solutions because of stratification or poor mixing when dissolving and blending materials of different isotopic composition in the same tank.

the plant immediately after sampling. They are taken in duplicate in order to verify repeatability of subsampling and treatment. When received at the Safeguards laboratory, they are totally dissolved, the total uranium content is determined and related to the mass of the sample measured at the plant.

The O/U-ratio should be between 2.06 to 2.15. If the ratio is lower the TRS-value is possibly larger.

Table 5 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Uranyl Nitrate Solutions, Pure

Target Value for the "Total Random Uncertainty": $TTR = \sqrt{TRS^2 + TRM^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.15	30-40	0.10	0.18	10	0.10	0.18
Coulometry (02)	0.15	30-40	0.10	0.18	10	0.10	0.18
Gravimetry (03)	0.10	30-40	0.10	0.14	10	0.10	0.14
X-Ray Fluorescence (04)	0.50	30-40	0.10	0.51	10	0.10	0.51
K-Edge (09)	0.20	30-40	0.10	0.22	10	0.10	0.22

The uranyl nitrate solution in the tank is carefully homogenized before taking the samples. Its homogeneity and that of its replicate samples should be checked by precise density measurements ($\pm 10^{-4}$ g/cm³) at a stable temperature ($\pm 0.1^\circ\text{C}$). The inspector's sample must be issued from a **primary sample** of at least 30 g which is itself carefully homogenized. The sample should be filled into a polyethylene bottle which can be capped very tightly and has a very stable tare. If the sample cannot be analyzed immediately a weight control must be carried out which provides an accurate control and correction of the potential evaporation of the sample between sampling and analysis. An effective procedure involves weighing the tare and gross mass at the time of sampling, and weighing the gross mass again before analysis commences.

10 ml of a typical uranyl nitrate product solution contain approximately 3-4 g of uranium.

Table 6 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of PuO₂ Powders, Nuclear Grade

Target Value for the "Total Random Uncertainty": $TTR = \sqrt{TRS^2 + TRM^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.20	10-20	0.10	0.22	2x0.5	0.30	0.36
Coulometry (02)	0.20	10-20	0.10	0.22	2x0.5	0.30	0.36
Gravimetry (03)	0.20	10-20	0.10	0.22	2x0.5	0.30	0.36
Spectrophotometry (06)	0.50	10-20	0.10	0.51	2x0.5	0.30	0.58

2. Uncertainty Components in Material Accounting

Material accounting measurements of operators and safeguards inspectors involve three major steps (Fig. 1):

- measuring the volume or the mass of an item or a batch of material
- taking representative samples
- measuring the fissile isotope or element content of the samples.

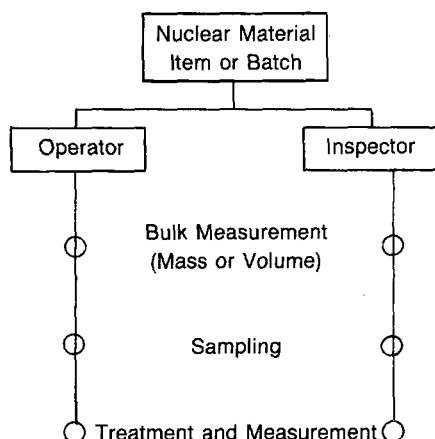


Fig. 1 - Steps in Nuclear Material Accounting Measurements

Each step includes both random and systematic uncertainties for which separate estimates should be made.

Reference /2/ presents Target Values for the random and systematic uncertainties which should be **achievable in the element assay** of the samples including the uncertainties due to the treatment as well as to the measurement.

The present paper dwells upon the **random uncertainties in sampling and their combination with the random uncertainties in element assay only.**

The target value for sampling is reached only by following a very strict sampling procedure to avoid the effects of moisture pick up or loss:

- The dew-point in the plant must be lower than -30°C .
- The sampling is done during the filling of the PuO_2 -containers, with a proportional sampler or with another appropriate sampling device in the process line.
- The total size of the primary sample is about 1% of the total batch (shipper, receiver and reference).
- Handling and subsampling of the primary sample are performed in a dry glove-box with a dew-point lower than -30°C .
- Potential pick up or loss of moisture during the subsampling operations must be monitored.
- The inspector's samples are usually about 0.5 g only because of shipment restrictions. They are taken in duplicate in order to control the repeatability of subsampling and treatment.
- The subsamples to be analyzed are weighed into a stainless steel container or directly into a vessel in which they can be dissolved quantitatively without further transfer.

The following paper was particularly useful in the preparation of this section:

- /4/ PATIGNY, P.: Contrôle de la représentativité de l'échantillon et de l'exactitude des résultats d'analyse de bilan sortie oxyde de plutonium, 6th ESARDA Symposium, Venice 1984, p. 417.

Table 7 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Plutonium Nitrate Solution, Pure

Target Value for the "Total Random Uncertainty": $\text{TTR} = \sqrt{\text{TRS}^2 + \text{TRM}^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.20	5	0.20	0.28	1-5	0.20	0.28
Coulometry (02)	0.20	5	0.20	0.28	1-5	0.20	0.28
Gravimetry (03)	0.10	5	0.20	0.22	1-5	0.20	0.22
X-Ray Fluorescence (04)	0.50	5	0.20	0.54	1-5	0.20	0.54
Spectrophotometry (06)	0.30	5	0.20	0.36	1-5	0.20	0.36
K-Edge (09)	0.20	5	0.20	0.28	1-5	0.20	0.28

Before taking the samples, the solution is carefully homogenized and its homogeneity is checked by highly precise density measurements ($\pm 10^{-4} \text{ g/cm}^3$ or by equivalent means).

In the plant procedure normally 3-4 replicate samples are taken and analyzed.

The inspector's sample is issued from a primary sample of at least 5 g, which itself is carefully homogenized before subsampling.

To limit the effects of outgassing (especially for Pu(VI) solutions), the inspector samples must be accurately weighed and diluted with a measured mass of 3 M nitric acid so that the diluted sample which is shipped contains less than 50 g/l of plutonium. Screw-capped polyethylene bottles of stable tare are recommended because they will not leak even during air-transport, if they are tightly capped properly. Also dangerous inner gas pressure will not build up.

The following discussion papers were particularly useful in the preparation of this section:

- /5/ DALTON, J.C., Mc GOWAN, I.R.: Target Values for Sampling. Discussion paper for the ESARDA/WGDA-Meeting, September 1983, Fontenay-aux-Roses
 /8/ BAUMANN, S., BERG, R., LAVERLOCHERE, J.: Target Values for Sampling Plutonium Nitrate. Discussion paper for the ESARDA/WGDA-Meeting, October 1984, Jülich

3. The Target Values for Random Uncertainties in Sampling

Random as well as systematic uncertainties in sampling depend on numerous factors such as the nature of the material, its homogeneity and stability, the size of the batch or item, the technique of sampling, the size of the sample, the mode of handling the samples, etc. The stability and homogeneity of the material are themselves a function of the fabrication process, the conditions and time of storage.

To simplify its task, the WG and the Committee decided therefore to address first the cases considered of particular relevance in trade and safeguards, namely the sampling of feed and product materials in spent fuel reprocessing and fuel fabrication plants. This covers the solutions of uranium or plutonium nitrate, spent fuels, the powders of uranium and/or plutonium oxide, and the pellets of uranium dioxide and mixed (U,Pu)O₂ oxides.

The WG and the Committee based their discussions mainly on the experimental data accumulated by plant operators. The relevant data were either obtained from the literature /4/ or from the records of the laboratories of the members of the WG and the Committee or of their correspondents. Some of these contributions are recorded in the minutes of the meetings of the WG /5, 6, 7, 8, 9, 10, 11/. At the time the present task was undertaken, the Working Group ISO/TC85/SC5/WG3 of the International Standardization Organization was also discussing the methods of sampling feeds and products at spent fuel reprocessing plants. The recommendations of the ISO WG were taken into account in the present work /12, 13, 14/. The ESARDA/ WGDA also relied on the work of the ESARDA WG on Low Enriched Uranium Conversion/ Fabrication Plants which had examined the sampling of uranium oxide powders and pellets /15/.

Reference /16/ described the special conditions under which IAEA inspection samples are taken. It outlines the additional steps and precautions which are necessary to make the shipment of these samples possible while assuring an adequate control over the stability of the sample even during transport over long distances.

4. Technical Aspects of Sampling

Plant operators apply different sampling procedures depending on the type of materials, fabrication process and analytical requirements. A common (and necessary) feature, however, is to perform first a careful homogenization of the bulk material, whenever possible. Subsequently one or more of three sampling strategies is normally employed:

- a) A series of samples is taken at random (random sampling) /17/ or in succession (periodic systematic sampling) /17/ from a batch of material which may consist of several drums of powder, trays of pellets or a single tank; the individual samples are analyzed separately to obtain a measure of the innate heterogeneity of the material even after homogenization; the number and the size of the individual

Table 8 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Mixed U/Pu-Oxides, LWR

$$\text{Target Value for the "Total Random Uncertainty"} : \text{TTR} = \sqrt{\text{TRS}^2 + \text{TRM}^2}$$

Plutonium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.50	25-50	0.30	0.58	2x(5-10)	0.70	0.86
Coulometry (02)	0.20	25-50	0.30	0.36	2x(5-10)	0.70	0.73
Spectrophotometry (06)	0.50	25-50	0.30	0.58	2x(5-10)	0.70	0.86

Uranium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.30	25-50	0.20	0.36	2x(5-10)	0.20	0.36
Coulometry (02)	0.30	25-50	0.20	0.36	2x(5-10)	0.20	0.36
Gravimetry (03)	0.10	25-50	0.20	0.22	2x(5-10)	0.20	0.22

As much as 25-50 g is required to obtain a representative sample, especially when the mixed oxide is prepared by mechanical blending. The operator obtains this total amount by taking 3 to 5 samples of approx. 8 g each and analyzing them individually. Alternatively, he takes a composite sample of 25-50 g, which is carefully homogenized to enable the subsampling of smaller, but still representative, portions for analysis.

The TTR-value given for the plant procedure applies to the composite sample only. The TRS-value for Pu will be lower if the mixed oxide is prepared by co-precipitation. The subsampling should be performed under the same conditions as the sampling. The subsamples to be analyzed are weighed into a stainless steel container or directly into the vessel in which they can be dissolved quantitatively without further transfer. The inspector's samples are taken in duplicate in order to control the repeatability of subsampling and treatment.

The following discussion papers were particularly useful in the preparation of this section:

- /9/ PELCKMANS, E.: Mixed Oxide (LWR) Sampling Errors. Discussion document for the ESARDA/WGDA, October 1985, Saluggia
- /10/ DALTON, J.C.: Contribution to the Evaluation of Sampling Uncertainties for Mixed Oxide and Spent Fuel Input Solutions. Discussion paper for the ESARDA/WGDA, October 1985, Saluggia.

samples are sufficiently large so that their mean composition can be expected to equal the average composition of the bulk material.

- b) Alternatively a composite sample (bulk or gross sample) may be taken by collecting together samples drawn from

individual items or by using a proportional sampling device in the process line.

- c) A primary sample of large size is taken in the process or storage area.

The latter two types of samples are then transferred to the laboratory where they can

be homogenized and subdivided into smaller samples (multistage sampling) /17/. In these approaches one aims at producing representative samples which eliminate the innate or residual heterogeneity of the material.

The objective of the Working Group and Committee was to estimate the magnitude of the random uncertainties which may still be observed after these processes and which should be attributed to the sampling step.

Whatever approach is chosen to take analytical samples, it is essential that the sampling procedures be well qualified and that these qualified procedures be enforced. The procedures of the plant operator are evidently most effective in this respect because cumulative experience has tailored them into the specific features of the local process.

The WG and the Committee recommend accordingly that the safeguards samples should preferably be taken following the local plant procedures provided that these are compatible with the goals of the inspection. The plant operators are expected to keep a record of the qualification of their sampling procedures, which are available for audit by safeguards inspectorates.

5. Meaning and Presentation of the 1988 Target Values for Sampling and Element Assay

The Working Group and the Committee attempted, for each type of material of interest, to express the achievable repeatability of its sampling by a single "Target Value". To do this, it was found necessary to specify in each case the minimum amount of sample and the major precautions to be taken in carrying out these operations.

Occasionally different Target Values still had to be assigned to the Operator's and Inspector's procedures. A typical example is the sampling of mechanical blends of U/Pu mixed oxides prepared for the fabrication of LWR fuels: a minimum amount of about 40 g of such a material is taken by the operator to obtain a representative sample and to keep the random sampling uncertainty below 0.3%. The inspector will in practice not ship samples larger than 5 to 10 g, so that the sampling uncertainties may be as large as 0.7% for the inspector.

This leads to the presentation of eleven separate tables (Tables 2 through 12), one table for each type of material.

Each table gives estimates of three relative standard deviations (in %):

- TRM is the 1987 Target Value for the

Table 9 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Mixed U/Pu-Oxides, FBR

$$\text{Target Value for the "Total Random Uncertainty": } \text{TTR} = \sqrt{\text{TRS}^2 + \text{TRM}^2}$$

Plutonium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.20	6-10	0.30	0.36	2x(1-2)	0.40	0.45
Coulometry (02)	0.20	6-10	0.30	0.36	2x(1-2)	0.40	0.45
X-Ray Fluorescence (04)	0.50	6-10	0.30	0.58	2x(1-2)	0.40	0.64
Spectrophotometry (06)	0.50	6-10	0.30	0.58	2x(1-2)	0.40	0.64

Uranium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.30	6-10	0.20	0.36	2x(1-2)	0.20	0.36
Coulometry (02)	0.30	6-10	0.20	0.36	2x(1-2)	0.20	0.36
X-Ray Fluorescence (04)	0.50	6-10	0.20	0.54	2x(1-2)	0.20	0.54

As much as 6-10 g is required to obtain a representative sample, especially when the mixed oxide is prepared by mechanical blending.

The operator usually takes 3 to 10 samples of 1-2 g each and analyzes them individually. Alternatively he may take a composite sample of 6-10 g, which is carefully homogenized to enable the subsampling of smaller, but still representative portions for analysis.

The TTR-value given for the plant procedure applies to the composite sample only.

The TRS-value for Pu will be lower, when the mixed oxide is prepared by co-precipitation

The subsampling of powders must be performed under the same conditions as the sampling. The inspector's samples are taken in duplicate, weighed immediately and directly in the vessel which can be used for their dissolution. When received at the Safeguards laboratory they are totally dissolved, the total element content is determined and related to the mass of sample measured at the plant. Duplicate samples are needed to verify the repeatability of subsampling and treatment.

The following discussion paper was particularly useful in the preparation of this section:

/10/ DALTON, J.C.: Contribution to the Evaluation of Sampling Uncertainties for Mixed Oxide and Spent Fuel Input Solutions. Discussion paper for the ESARDA/WGDA, October 1985, Saluggia.

random uncertainty in Element Assay or Measurement Assay taken from Reference /2/
TRS is the 1988 Target Value for the

random uncertainty in sampling
TTR is the 1988 Target Value for the total random uncertainty in sampling and element assay.

The 1987 Target Value for the random uncertainty in Element Assay /2/ can depend upon the analytical method which is used. This value is also only valid if the size of the aliquot submitted to the final measurements is equal to or greater than the amount listed in Ref. /2/ and repeated in Table 1. In this table as in all other ones, gravimetry refers to the method involving calcination to a reproducible oxide, without prior purification, but with impurity analysis on the calcined oxide.

TRS is the relative standard deviation expected when taking replicate but independent samples (see par. 4 (b), (c)) or replicate but independent groups of samples (see par 4. (a)), drawn at the same time under the same conditions from one item or batch of industrial material. A separate estimate is given for the operator and for the inspector. This should clearly point out where the sampling procedures, which the inspector must apply, could yield significantly larger uncertainties than may be expected using the operator's procedures. The table also specifies the minimum amounts of material and the major precautions which must be taken respectively by the operator and the inspector to achieve the proposed Target Values.

When the operator prefers the approach (a) (see par. 4) and takes several but smaller samples as is recommended in the table, the total amount of material collected must still be equal to or larger than the recommended amount. In such instances (Tables 8, 9) the TRS-value should be equal to the relative standard error /17/ to be expected for the random uncertainties when taking small individual samples. Thus, if RS is the relative standard deviation in the taking of p small individual samples, one should have

$$TRS = RS / \sqrt{p} \quad (1)$$

TTR represents the relative standard deviation of single analysis of replicate samples of the same material, taken at the same time and under the same conditions. The values presented in Tables 2 through 12 are calculated according to equation (2):

$$TTR = [TRS^2 + TRM^2]^{1/2} \quad (2)$$

assuming that only one sample is drawn and that it is measured only once as may be done under approach (b) or (c) of par. 4. When p small individual samples are taken instead and analyzed separately (approach (a) of par. 4) the relative standard deviation expected for the total random uncertainty TR will be smaller than TTR if the number and size of the individual samples are large enough for equation (1) to be true.

Then indeed,

$$\begin{aligned} TR &= 1/\sqrt{p} [RS^2 + TRM^2]^{1/2} = \\ &= [TRS^2 + (TRM^2/p)]^{1/2} < TTR \end{aligned} \quad (3)$$

Table 10 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Reprocessing Input Solution of Spent HWR/LWR Fuel

Target Value for the "Total Random Uncertainty": $TTR = \sqrt{TRS^2 + TRM^2}$

Plutonium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
X-Ray Fluorescence (04)	1.0	1-5	0.30	1.1	1-5	0.30	1.1
IDMS (05)	0.50	1-5	0.30	0.58	1-5	0.30	0.58
Spectrophotometry (06)	1.0	1-5	0.30	1.1	1-5	0.30	1.1

Uranium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.30	1-5	0.30	0.42	1-5	0.30	0.42
X-Ray Fluorescence (04)	0.50	1-5	0.30	0.58	1-5	0.30	0.58
IDMS (05)	0.50	1-5	0.30	0.58	1-5	0.30	0.58

The input solution must be clarified by filtration through a 5-20 μm filter or by centrifugation prior to being transferred into the accountability tank.

Before taking the samples, the solution in the tank is carefully homogenized, and its homogeneity is checked by highly precise density measurements ($\pm 10^{-4}$ g/cm³) or by equivalent means. The solution of the tank must circulate through the sampling device during the entire homogenization process.

The sample of concentrated input solution must be transferred to a hot cell of the analytical laboratory of that plant and treated or analyzed within 24 hours.

Whenever a smaller size is quoted for the inspector sample than for the operator sample the inspector's TRS value will usually be larger than the operator's value (Tables 4, 6, 8, 9). Otherwise (Tables 5, 7, 12), the table specifies that the inspector samples are actually subsamples issued from a larger primary or composite sample after a careful multistage sampling operation /17/. Here too the size of the original primary or composite sample should be equal to or greater than the size recommended for the operator sample.

The size recommended for the inspector sample represents the minimum amount of material that can be shipped to the Safeguards verification laboratory without losing adequate control over the potential changes in its composition during transport. Yet the target values TRM and TTR will not be achieved by the inspector unless special precautions specified in the tables are taken in the conditioning and handling of inspector's samples.

In conclusion, each table of Target Values for Sampling and Element Assay

must be considered as an indivisible entity. The Target Values for sampling will not be achieved unless the recommended minimum amount of material and the precautions specified in the table are taken. All users of the present document are therefore strongly cautioned never to quote a Target Value for sampling defined in it, without referring simultaneously to the underlying prerequisites stated in the relevant table. Hopefully, the user will find that the selected format is a useful one to promote the proper utilization of the proposed Target Values.

Discussion and Conclusions

(1) Despite the diversity of plants, industrial processes and sampling procedures, it was possible for the major feed and products materials under safeguards to ascribe a single value to the random uncertainty which should be expected respectively from the operator's and inspector's samplings, under routine industrial and inspection conditions. This undoubtedly reflects the efforts made both by the operators and the Safeguards authorities to optimize their sampling procedures.

(2) These "1988 Target Values for Random Uncertainty Components in Sampling" should be achievable or need to be achieved in 1988 and onwards under routine conditions.

(3) The Target Values presented for the sampling uncertainties are defined for a specific material and are applicable only if the minimum amount of material and major sampling precautions specified in the Tables are taken. **They should never be quoted without reference to the underlying prerequisites given in the relevant table.**

(4) The Inspector and Operator are expected to achieve the same Target Values for Sampling, except when the Inspector samples are smaller than the minimum representative sample size, because of shipment restrictions.

(5) In the case of UO_2 pellets and spent fuel solutions and only the Operator's procedure, the measurement uncertainties are expected to be greater than the uncertainties due to sampling. The opposite is true for UO_2 powders and FBR U/Pu mixed oxides. In the other cases measurement and sampling are expected to have similar uncertainties.

(6) In the case of PuO_2 powders, sampling uncertainties may be minimized by sampling under controlled dry atmosphere and using inline proportional samplers.

(7) Efficient clarification of spent fuel dissolver solutions to remove particulate

Table 11 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of Reprocessing Input Solution of Spent FBR Fuel

$$\text{Target Value for the "Total Random Uncertainty": } TTR = \sqrt{TRS^2 + TRM^2}$$

Plutonium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.30	1-5	0.30	0.42	1-5	0.30	0.42
X-Ray Fluorescence (04)	1.0	1-5	0.30	1.1	1-5	0.30	1.1
IDMS (05)	0.50	1-5	0.30	0.58	1-5	0.30	0.58
Spectrophotometry (06)	1.0	1-5	0.30	1.1	1-5	0.30	1.1

Uranium Assay

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (g)	TRS (%)	TTR (%)	Sample Size (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.30	1-5	0.30	0.42	1-5	0.30	0.42
X-Ray Fluorescence (04)	0.50	1-5	0.30	0.58	1-5	0.30	0.58
IDMS (05)	0.50	1-5	0.30	0.58	1-5	0.30	0.58

The input solution must be clarified by filtration through a 5-20 μm filter or by centrifugation, when being transferred to the accountability tank.

Before taking the samples, the solution in the tank must be carefully homogenized and its homogeneity must be checked by highly precise density measurements ($\pm 10^{-4}$ g/cm³) or by equivalent means. The solution of the tank must circulate through the sampling device during the entire homogenization process.

The sample of concentrated input solution must be transferred to a hot cell of the analytical laboratory of that plant and treated or analyzed within 24 hours.

matter is an important condition to minimize the uncertainties in sampling input solutions.

(8) The ESARDA-WGDA and INMM Committee 5.1 expect other ESARDA Working Groups, especially the plant specific Working Groups as well as appropriate USA Committees to define Target Values for the bulk measurements and for the systematic component of the sampling uncertainties, in order to complete the examination of the uncertainties in the

accountability measurements and let the authors know.

(9) The use of density could be useful to estimate experimentally the magnitude of the systematic uncertainties in the sampling of solutions /19, 20/.

(10) Safeguards authorities should consider to use the "1988 Target Values for Random Uncertainty Components in Sampling" and to publish comparisons between the

"Target" and actual "Performance" values observed in their inspection activities.

(11) Periodical comparisons of "Targets" and "Performance" should help operators as well as safeguards inspectorates to identify the aspects of the measurement systems which may be critical.

(12) Such comparisons are needed as experimental evidence to update periodically the "Target Values" in view of improved performance, changes in accountability standards or safeguards criteria.

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Table 12 - 1988 Target Values for the Random Uncertainty in Sampling and Element Assay of LEU-UF₆

Target Value for the "Total Random Uncertainty": $TTR = \sqrt{TRS^2 + TRM^2}$

Analytical Method (Code)	TRM (%)	Plant Procedure			Inspector Procedure		
		Sample Size (a) (g)	TRS (%)	TTR (%)	Sample Size (a) (g)	TRS (%)	TTR (%)
Titrimetry (01)	0.20	4-8	0.20	0.28	4-8	0.20	0.28
Coulometry (02)	0.20	4-8	0.20	0.28	4-8	0.20	0.28
Gravimetry (03)	0.15	4-8	0.20	0.25	4-8	0.20	0.25

(a) The sample size of 4-8 g refers only to the "subsample" or final sample which is collected in a small PTFE-tube and sent to the analytical laboratory. This sample is actually issued from a "Field-sample" or primary sample of 0.3 to 0.5 kg.

The TRS value combines the uncertainties coming from both the field sampling and the subsampling operations.

Before taking the "Field-Sample" the bulk container must be heated to 95°C so that all the UF₆ it contains is in the liquid phase. The same must be done with the Field-sample before taking the subsample for elemental assay.

A useful reference is:

- /18/ Procedures for Handling and Analysis of Uranium Hexafluoride, Vol. 1, Inspection, Weighing and Sampling of Uranium Hexafluoride Cylinders, ORO-671-1, April 1972, USAEC, Oak Ridge, TN, USA

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How to Use the ESARDA Target Values for Sampling and Element Assay

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1. Introduction

How does the actual performance of safeguards verifications compare with the capability of the systems expected from the 1988 Target Values ? (ref. 1) To make such a comparison it is necessary to know the structure of the sampling and measurement systems. The impact of sampling and measurement errors will indeed differ greatly depending upon the actual structure of these systems, which also varies from plant to plant. This structure is unfortunately rarely well known to the Safeguards inspectors. For the sake of the discussion we chose therefore to examine four cases, which are schematized in Fig. 1. We tabulated also the standard deviation of the operator-inspector (OP-IN) difference which should be expected in each one of these four situations, and compared it to typical values actually observed in safeguards inspections (Table I). The target value corresponding to the most likely real situation is printed in bold character in the table.

The present paper deals only with random uncertainties as reference [1] does.

To evaluate the effect of fluctuating systematic errors, it would be necessary to define Target Values for the systematic uncertainties in sampling and to know how many items or batches carry the same systematic error. It is not seen how that could be done at this point in time.

2. Case 1:

One Sample/One Analysis per Item

In the first case we assume that the operator and the inspector each take independently one sample of the same item or batch, and perform each a single measurement of the sample. The achievable standard deviation of the random fluctuations of OP-IN differences should in this case be given by equation (1):

$$SD_i^2 = TTR_o^2 + TTR_i^2 = TRS_o^2 + TRS_i^2 + TRM_o^2 + TRM_i^2 \quad (1)$$

where the subscripts o and i identify the Target Values of the operator's and inspector's systems respectively. This case is the simplest one, but it corresponds rarely to the reality.

Table I. Achievable and observed random uncertainty component of operator-inspector differences (relative standard deviation, in %)

Material	Target (c)				Observed
	Case 1 (n = 1; p = 1)	Case 2 (n = 2; p = 1)	Case 3 (n = 2; p = 5)	Case 4 (n = 2)	
LEU-UO ₂ powder	0.35	0.32	0.25	0.15	0.51
LEU-UO ₂ pellet	0.23	0.32	0.17	0.15	
LEU-UO ₂ 2 (a)	0.09	0.09	0.07	0.05	0.05
HEU-U oxide powder	0.42	0.39	0.33	0.15	0.38
UN nitrate solution	0.25	0.21	0.18	0.15	0.22
PuO ₂ powder	0.42	0.41	0.34	0.20	0.56
PuN nitrate solution	0.40	0.35	0.27	0.20	0.26
Pu in LWR-MOX	1.04	0.91	0.81	0.50	0.61
U in LWR-MOX	0.51	0.41	0.32	0.30	0.22
Pu in FBR-MOX	0.58	0.54	0.45	0.30	
U in FBR-MOX	0.51	0.41	0.32	0.30	
Pu in LWR input (b)	0.82	0.66	0.51	0.50	0.98
U in LWR input (b)	0.82	0.66	0.51	0.50	0.52
Pu in FBR input (b)	0.82	0.66	0.51	0.50	
U in FBR input (b)	0.82	0.66	0.51	0.50	

(a) gravimetry; (b) IDMS; all other results by titrimetry.

(c) p, number of replicate samples of the same item or batch of material; n, number of replicate treatment-measurement combinations of the same sample (see Fig. 1).

3. Case 2 :

One Sample/n Replicate Analyses per Item

Usually the operator and the inspector will both do several independent analyses of their sample. If they do, say, each n measurements, the variance of the difference between their means should have a magnitude given by equation (2) :

$$SD_i^2 = TRS_o^2 + TRS_i^2 + 1/n (TRM_o^2 + TRM_i^2) \quad (2)$$

The sampling component stays the same but the measurement component of the variance is divided by n. Compare the values obtained for this case, when n = 2, (Table I, case 2) with the values expected in case 4, which illustrates, as we shall see below, a situation where sampling errors are

the same for OP and IN and do not affect the difference; the sampling errors may double the variance of the OP-IN differences in all cases examined, except for the assay of UO₂ pellets, U and Pu nitrate solutions and spent FBR solutions, where according to the Table I the impact of the sampling errors should be smaller.

In reality case 2 applies usually only to uranyl and plutonium nitrate solutions. The observed values are in these instances in agreement with the computed values.

4. Case 3 :

p Samples/n Replicate Analyses per Item

Very often the operator will not report the results of analyses of individual samples but the mean of his analyses of p samples of the

batch of material of interest. On the other side the inspector receives only one sample of this batch or item. If here again the operator and inspector perform each n independent analyses of each sample, the differences between the operator's data and the mean result obtained by the inspector on one sample should have a variance of random fluctuations within

$$SD_3^2 = 1/p \text{ TRS}_o^2 + \text{TRS}_i^2 + 1/n [1/p \text{ TRM}_o^2 + \text{TRM}_i^2] \tag{3}$$

The random component of the operator's sampling errors are expected to become negligible and the "Inspector's" sampling errors would be expected to be the dominating factor (Table I, case 3).

Case 3 is probably very close to the situations which are encountered most often in actuality.

The observed values are within or close to the computed targets in the case LEU- UO_2 pellets, HEU-U oxide powders, Pu and U in LWR-MOX. They are on the average quite larger in the case of LEU- UO_2 and PuO_2 powders, although analyses of duplicate inspector's samples of PuO_2 powders have typically a relative standard deviation of 0.26 % only.

5. Case 4 :
Operator and Inspector Use the Same Sample

The fourth case we wish to present illustrates the actual situation encountered until now in particular in safeguarding input solutions of spent fuel reprocessing plants. There, the operator and the inspector receive, and analyze, one or two subsamples of the same primary sample. The operator and the inspector, in such a situation, must have the same sampling error. Neither this sampling nor its fluctuation contributes to the OP-IN difference.

In this case the variance of the difference measures only the contribution of the measurement errors. The random component should then be within

$$SD_4^2 = 1/n (\text{TRM}_o^2 + \text{TRM}_i^2) \tag{4}$$

The U elemental assays appear to meet the computed targets. However the Pu elemental assays are on the average still far from fulfilling the expected performance and this despite the significant progresses which could be achieved in the last years.

6. Conclusions

- 1. To use the Target Values for sampling errors it is necessary to know how they propagate with the uncertainties arising in the elemental assay. For this purpose the operator and the inspector must state what their sample and analysis procedures are and must adhere to these statements.
- 2. Target Values for random uncertainties in Sampling and Elemental Assay have been combined according to four simple cases illustrating situations typically encountered. The Target Values for OP-IN differences deduced from these cases were compared to typical performances.
- 3. According to the IAEA experience the performance of DA verification measurements is still not reaching the level expected from the 1987 Target Values (ref. 1) in the case of LEU- UO_2 powders, PuO_2 powders and for the Pu element assay in spent fuel solution.

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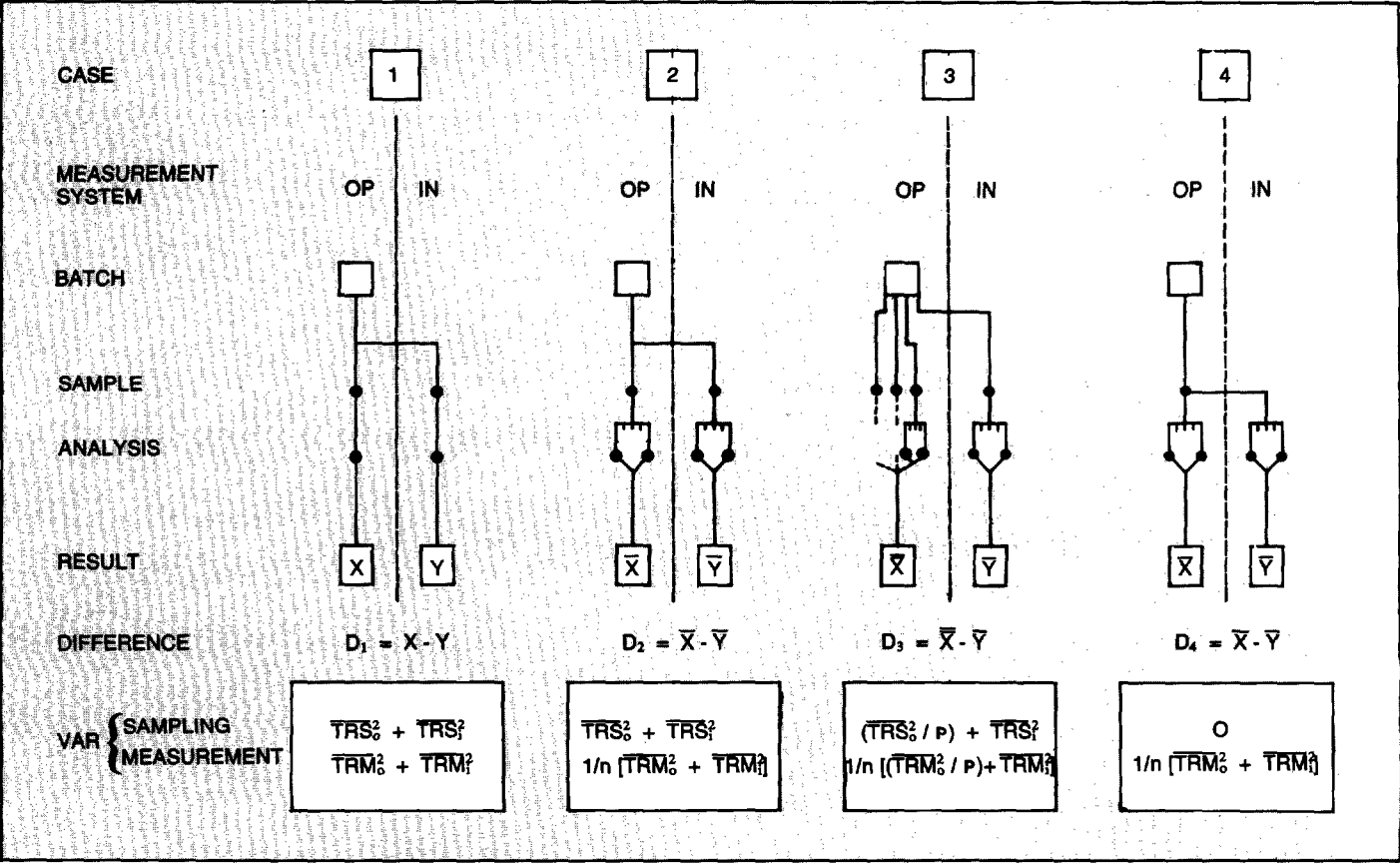


Fig. 1 - Structure of measurement systems and propagation of sampling and measurement errors

Cooperation Agreement between ENEA and JRC-Ispra for R & D on Techniques and Methods for Safeguards Purposes

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Introduction

The Joint Research Centre (JRC, Ispra Establishment) of the Commission of the European Communities and ENEA, the Italian Nuclear and Alternative Energy Commission, have been involved, for many years, in R&D activities for safeguards implementation.

Specifically JRC's R&D programme is devoted to assisting the Euratom Safeguards Directorate and the European plant operators in safeguards implementation in the frame of Euratom and Non Proliferation Treaties.

In this context JRC is setting up a performance evaluation and training laboratory (called PERLA) whose goal is to make available an infrastructure for testing NDA and C/S devices in near field conditions.

As far as ENEA is concerned, the new five year plan 1985-1989 foresees a considerable effort aimed at improving R&D activities in safeguards field, particularly for the most sensitive area, namely fuel fabrication and reprocessing plants.

Both organisations have offered to the IAEA a technical support programme for safeguards implementation. The Commission's programme started in 1981.

The Support Programme for Safeguards Implementation offered to the IAEA by ENEA in 1985 has started in January 1986. This support programme mainly concerns the field of fuel cycle, with particular emphasis on the back end activities.

ENEA and JRC have been already involved in joint R&D activities in safeguards field before deciding to formalize such a common effort.

After some technical meetings between JRC and ENEA specialists in order to focus on and to define the area of common interest, the formal commencement date for this cooperation agreement was fixed in October 1986.

Aim of the Agreement

The cooperation between ENEA and JRC is aimed at improving the comprehension of

safeguards needs and at allowing a better exchange of information on respective programmes.

Such a joint effort in the R&D activities will be realized through the harmonization of the programmes, in order to avoid needless duplications and to improve activities in areas of high priority.

The agreement covering, in this first stage, the years 1987-1989 will be devoted, at the beginning, to activities concerning measurement technology, data acquisition and evaluation methodology and support to PERLA.

It is also foreseen, in the near future, to deal with other relevant safeguards techniques, namely containment and

surveillance (C/S) techniques, simulation studies for near real time accountancy (NRTA), mathematical models for measurement error analysis and measurement techniques for irradiated fuel assemblies.

Survey of Contents of ENEA-JRC Cooperation Agreement

The joint programme is subdivided into three major task areas, identified from among the existing common activities and the respective R&D programmes.

The subject matter treated in each area under the individual task is summarized in Table I.

Table I - Cooperation Agreement between ENEA and JRC for Safeguards Implementation

Measurement Methods for Liquid Phase System

- Volume and/or mass determination in accountancy vessels by tracer techniques
- Performance evaluation of measurement methods and techniques (DA and NDA) for safeguards control in reprocessing plants
- Calibration and volume determination in accountancy vessels (instruments performance evaluation and error sources investigation)

Non Destructive Techniques

- Pu isotopic analysis by gamma spectrometry
- Passive neutron measurements of plutonium samples from reprocessing (Pu nitrate solutions) or conversion plants (MOX powders)
- Passive neutron measurements of MOX fuel pins
- Active neutron measurements of bulk Pu samples and uranium or Pu contaminated waste.

Although the formal commencement date for the agreement was fixed in October 1986 some activities are already in an advanced state of development. Hereafter the paper will report the goals and the main actions as well as the progress on the individual tasks.

Measurement methods for liquid phase systems

The first task of this area deals with the

development and demonstration of tracer techniques for the determination of volume and/or mass in accountancy vessels of reprocessing plants. In this framework, the research activities are aimed at the development of tracer addition, tracer homogenization and sampling procedures, radiochemical separation procedures and definition of mass spectrometry measurement conditions and their

application in hot conditions.

Such a task is divided into two subtasks to be carried out at ENEA-ITREC and ENEA-EUREX pilot reprocessing plants.

An experimental activity, named ITITEX (ITREC input tank experiment) has been carried out using lutetium as a tracer, measuring the volume of cold U + Th solutions. Results obtained by the tracer technique were compared with conventional measurement techniques and expected values. The accuracy achieved with the tracer method was better than 0.5% /1/.

The topic of the second task is the study of different measurement methods and techniques, both DA and NDA, devoted to nuclear material assay for safeguards purposes in reprocessing plants.

The aim of the task is a performance assessment and reliability estimation of the measurement methods and evaluation of the implication on normal plant operations of both NDA instruments installation and in loco DA analysis execution by the inspectors.

As far as the third task is concerned, the goal is the investigation of error sources in volume determination and the performance evaluation of different instruments for level and density measurements.

The activity will be carried out through the execution of a series of calibration runs on an input accountancy tank (Fig. 1) using different instrumental systems. In the meantime

a simulation program for input tank volume measurements, called SPRIT /2/, is being tested and demonstrated in order to evaluate the influence on the measurement procedure of the variation of different physical parameters.

Nondestructive techniques

In the framework of the agreement, a considerable effort is devoted to joint activities aimed at developing and testing nondestructive techniques, namely gamma spectrometry and neutron measurements.

Pu isotopic analysis by gamma spectrometry is a well known technique which can be considered to be in an advanced level of development both for the hardware and for the software. However some problems still exist as far as high burn up samples and high count rates are concerned.

The task deals with the evaluation of Pu spectra made available both by JRC and ENEA. The goal is to assess the performance of different software packages in terms of accuracy and precision.

As far as neutron measurements are concerned, three tasks have been defined dealing with MOX sample assay, fuel pin assay and active neutron measurements for bulk Pu samples and Pu or uranium wastes.

The first task foresees measurements of

input Pu solutions and output MOX samples coming from EUREX-UMCP plant, the ENEA pilot conversion plant (Fig. 2). During the experiments, which will be carried out with a HLNCC II type measuring head, the influence on coincidence measurements due to moisture and to the presence of slight impurities will be studied.

The second task on neutron measurements, whose goal was to develop and test a nondestructive technique for the assay of fuel pins coming from the plutonium fabrication plant at ENEA, Casaccia, is now completed /3/. A measuring head, named ARIANE I and shown in Fig. 3, has been built and tested. Such a measuring head, coupled with shift register electronics, allows the measurement of one MOX fuel pin at a time and will be applied for the characterization of PEC fuel pins.

The PHONID system (Fig. 4), a topic of the last task of this area, based on the active interrogation of fissile material, is already operational for ^{235}U nondestructive assay /4/. The focus of the task will be on the possibility of employing the same system for the assay of bulk Pu samples as well as uranium or plutonium contaminated wastes. In this context Monte Carlo neutronic calculations will be used to predict the response of the system as a function of the fissile content, the sample physical form and the geometric characteristics of the instrument.

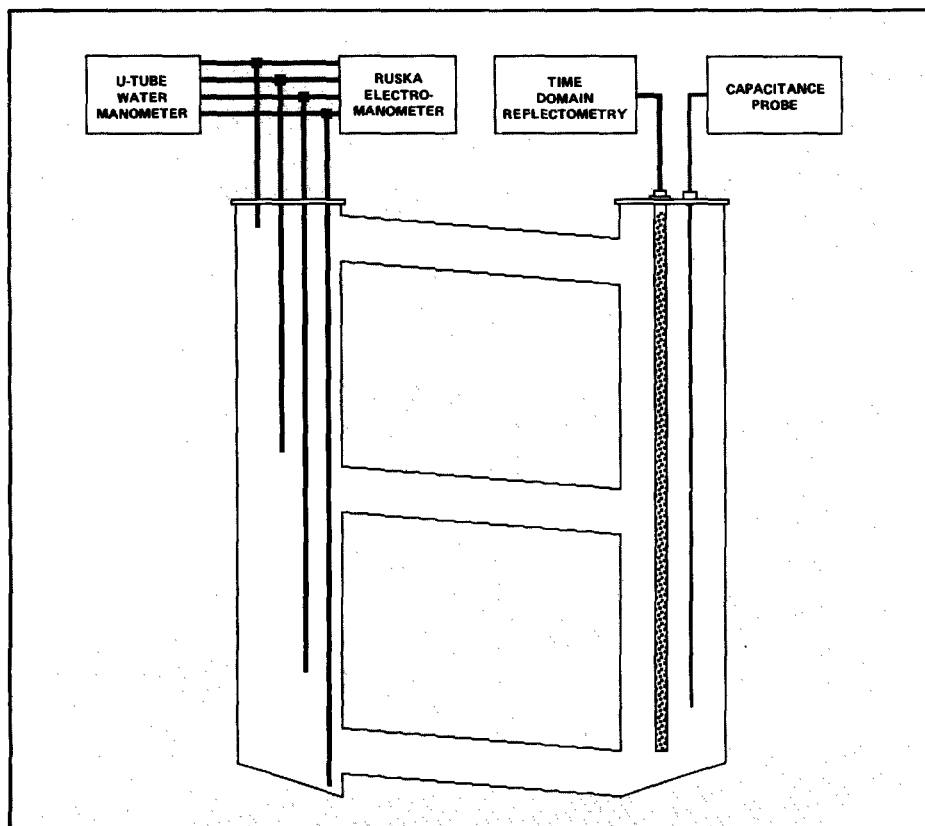


Fig. 1 - Input accountancy tank (PERSPEX model)

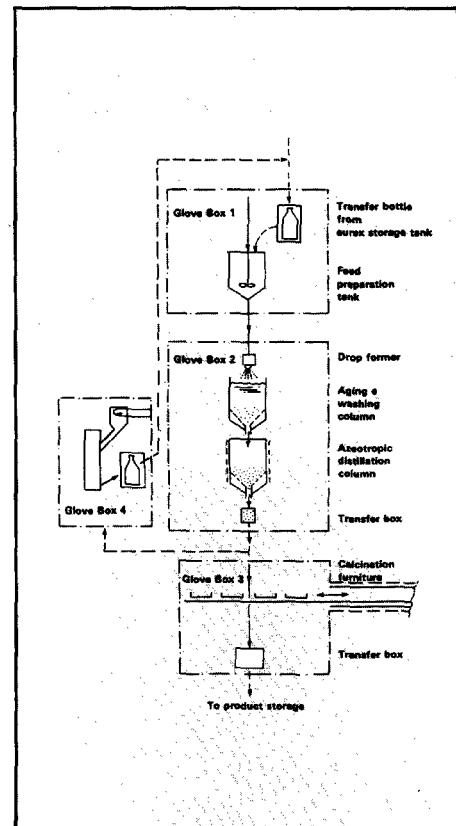


Fig. 2 - UMCP equipment flowsheet

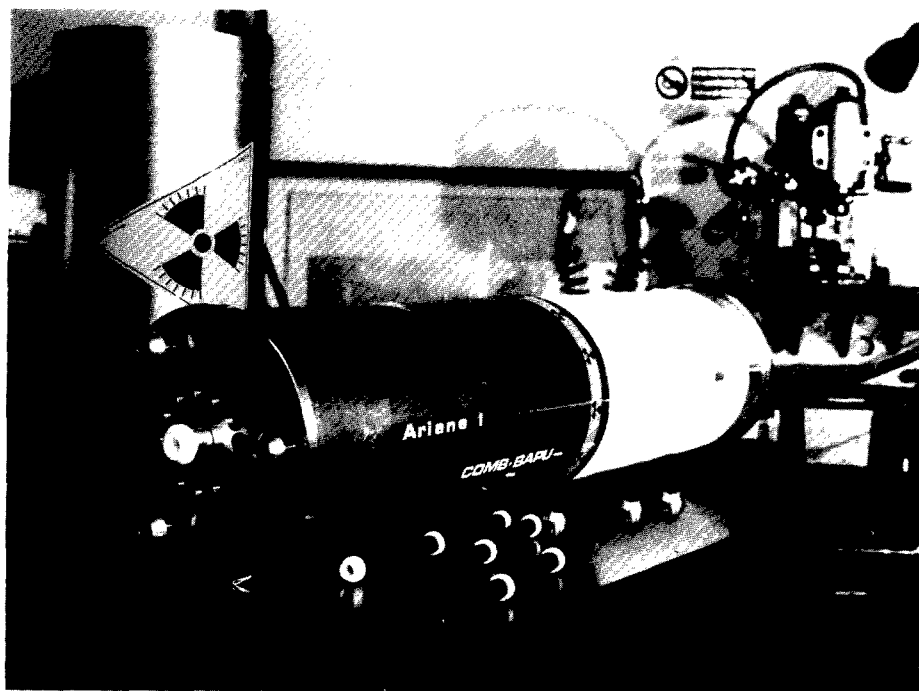


Fig. 3 - ARIANE 1 : Measuring head

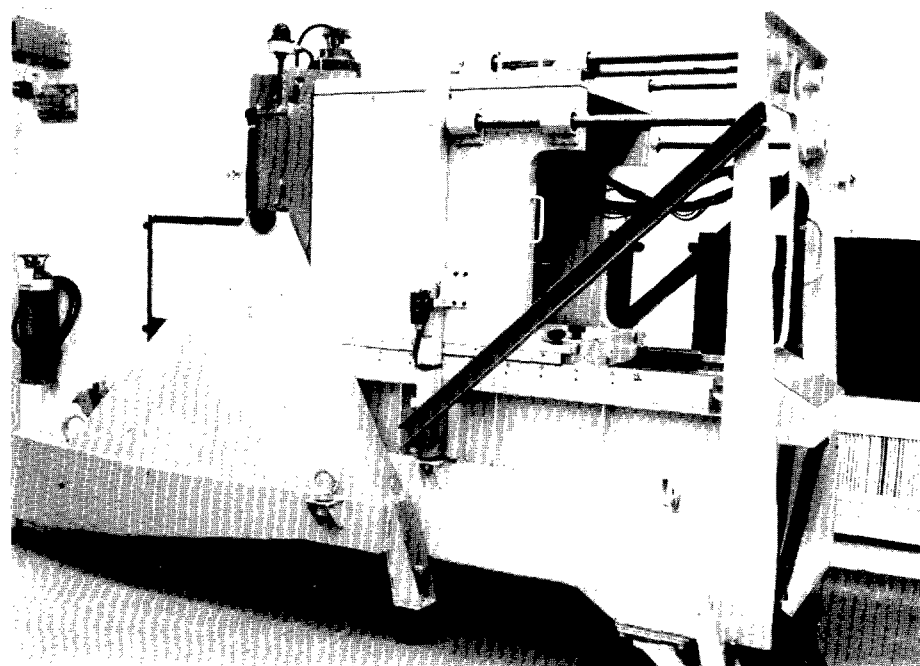


Fig. 4 - View of PHONID 3

Conclusions

The results in the ongoing tasks have been obtained mainly at the Casaccia, EUREX and Rotondella laboratories. It is expected that a number of new experiments will start early in 1988 also at JRC-Ispra in the PRE-PERLA laboratories, which became operational on June 1st, 1987.

The experiments will include characterization of PERLA standards and intercalibration of NDA instruments.

The formal agreement which has started in 1986 provides a clear framework for R&D cooperation in areas of mutual interest and has increased substantially the exchange of information between the two organizations.

It also simplifies the planning of joint experiments and the monitoring of their progress.

Due to the emphasis which has been put on development, test and performance assessment of techniques and instruments for fuel fabrication and reprocessing facilities, it is expected that plant operators and safeguards authorities will directly benefit by the results obtained in the framework of this cooperation.

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The Use of the Shipper/Receiver Difference in the Facility Accountancy and the Safeguards Evaluation

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Abstract

The Shipper-Receiver Difference (SRD) is one of the accountancy elements of major interest for the evaluation of the transfer declarations among facilities. After an introductory part dedicated to the interpretation of the SRD definition, the study provides for the elements of a procedure to be used in the evaluation process. Specific cases of misuse of SRD declarations, of hidden SRDs and of SRDs without safeguards significance are illustrated. Techniques to be used in examining the safeguards value of single SRDs and sets of SRDs are suggested and discussed.

Shipper/Receiver Difference (SRD) Definition

1. In /1/ and /2/ the SRD is defined as follows:

"Shipper/receiver difference" means the difference between the quantity of nuclear material in a batch as stated by the shipping material balance area and as measured at the receiving material balance area.

2. This definition uses two terms which need some interpretation; they are :

- the **statement** of the shipper
- the **measurement** of the receiver.

3. The **statement** of the shipper is his declaration concerning the shipped batch, based on his best knowledge of the relevant nuclear material. This knowledge is obtained either by direct instrumental measurement (of a quality level compatible with the international standards) or by "continuity of knowledge" of previous measurements. This "statement" must be supplied to the receiver /1/.

4. The **measurement** of the receiver is a comprehensive word indicating all the results of the quantitative observations, made on the received batch, relevant to the quantitative declarations of the shipper. Obviously there are parameters, like the "number of items", which in most of the cases are easily and rapidly checked, while others, like the amount(s) of nuclear material(s) or the U enrichment require complex measures or, in some cases, are impossible to be measured without tampering with the transferred items (e.g. fuel assemblies).

5. This interpretation of the definition implies that the receiver shall enter in his records:

- first: the registration of the receipt of a batch together with the statement of the shipper (RD, RF or RN repeating the batch name and all the relevant qualitative and quantitative data as stated by the shipper). The "measurement basis" code N (or F) is used in this entry to indicate that the recorded values do not have origin in the receiving MBA;

- then: if quantitative determinations (according to par. 4 above) are performed, the results of this batch measurement(s), which possibly differ from those of the shipper's statement.

The possible differences between these two sets of numerical parameters are, collectively, the SRD defined above.

6. In principle the receiver is authorized to use "shipper's values" as long as the batch received maintains its own integrity. When quantitative determinations on the received batch are not performed no SRD occurs and the receiver accepts full responsibility of the quantities stated by the shipper as soon as the batch physical integrity is lost or changes (e.g. by irradiation). But when accountancy measurements are performed on the received batch, the ascertained difference, even if zero, should be recorded in the accountancy and reported to the Safeguards Authorities.

Detection of an SRD by the Inspectors

7. If the rules mentioned above are followed, the inspectors become aware of an SRD

- during inspections,
 - . by means of a direct communication from the operator or
 - . by a record auditing; or
- at the headquarters,
 - . during the ICRs review or
 - . by means of a special report.

8. However, in practice, there are cases of SRD which are not treated by the operators along the above mentioned lines, but are hidden by other types of Inventory Changes. Attention must be paid to such cases, the most common of which are described in paragraphs 9 to 15.

Case of Hidden SRDs

A. Discrepancy in the item number

9. When the first rapid check of a receipt shows to the receiver an item number different from that stated by the shipper, the receiver may be lead - wrongly - to issue an RD (or RF or RN) entry containing the number of items he has identified. In the absence of any other piece of information the inspectors may become aware of this hidden SRD when the transit control system will detect such inconsistency.

10. This type of inconsistency is still more difficult to detect in the absence of a real shipper's report, e.g. in cases of RN and RF. In such cases inconsistencies may be detected only through the audit of the vouchers and of similar records.

B. Transformation of an SRD into a Correction

11. This case occurs when, after measuring a receipt, the receiver corrects his accountancy by means of the Deletion/Addition (D/A)

technique and communicates his results to the shipper who, in turn, agrees to correct his transfer records by the same D/A technique. This operation implies that the shipper, from the safeguards point of view, is ready to consider the receiver's measures as correct and uses then in his accountancy. With such a decision he takes full responsibility for the stated figures, as if the boundary of his MBA would be extended up to the receiver's KMP. A possible error in the stated figures is eventually mirrored, with opposite signs, both in shipper's and receiver's MUFs. If the original shipper's data are considered as provisional the SRD no longer exists. If there is no shipper's explicit agreement the SRD must be restored (or, at least, evaluated as such).

12. Commercial agreements between the parties may stipulate that the quantity transferred is determined by the receiving MBA. But these agreements lay outside the scope of safeguards, which is called to decide on the Material Balances of the single MBAs, drawn in accordance with the techniques declared in the BTC and verified by the inspectors. As mentioned in the previous paragraph, these agreements may be regarded as within the scope of safeguards only if explicitly the shipper accepts backward his part of responsibility for the measurement performed by the receiver. However, in such a case, the inspectors would miss the redundant information contained in the SRD statement (two measures of the same batch) and possibly should increase the verification at this (unique) KMP between two MBAs under different managements.

13. This case of hidden SRD differs from the case of a real correction, which may occur, e.g. when the shipment is made on provisional data, eventually corrected when the definitive (shipper's) data are available. When correcting his own data the shipper informs the receiver of the improvement so that the latter may record, in his books, the new shipper's statement by the D/A entries. The observation of the accounting and original dates should allow the discrimination between the two cases without resorting to the operational or accounting records.

C. Use of NM Code instead of DI

14. This is the case of a batch, received and recorded in the books with the correct procedure, which is measured by the receiver after a certain time (even after a PIT event). The possible difference is recorded as an NM, instead of being recorded as an SRD, using the code DI.

15. This case is more difficult to detect through reports, in spite of the fact that, whenever the batch name for the NM is that of the received batch, the "measurement basis" should indicate if such a batch was or was not previously measured in the MBA. If what was described in par. 14 occurs, the RD shall carry the measurement basis **N** (or **F**) and the batch in the PIL should be coded **L** (or **H**) and not **T** (or **G**). This type of hidden SRD should be detected in inspection during the accounting verification. The evaluation of the IC will follow the same pattern of a normal SRD. If considered important an action to correct the accountancy must be undertaken.

The Evaluation Process

16. As soon as an inspector is aware of an SRD a first evaluation step shall be performed to decide whether or not the difference is compatible with the combined measurement errors of the shipper's and receiver's measurement systems (including item counting). In case a **decision** of "non compatibility" is taken follow-up actions may be envisaged as mentioned in § 23 and 42 to 48.

17. In most of the cases the decision on the compatibility is taken on the basis of the results of appropriate statistical tests. Note, however, that what suggested in the following should not hamper a **professional responsible judgement which transforms a mathematical result into a safeguards decision**.

SRD on the Number of Items

18. One case, which in general requires a follow-up, is that of the difference in the item number. The first thing to be examined is the amount and the quality of the material involved. The case of an apparent **gain** in transfer has also to be investigated, but it creates a lower safeguards concern than a **loss**.

19. A discrepancy in the item number should be explicable, in principle, by the involved parties including, if necessary, the carrier(s) and the intermediaries. The Community has imposed /1/ specific obligations on these persons, so that the Euratom inspectors may extend the enquiry on the transfers, in case of suspected anomalies.

20. In the case where a shipper realises he has shipped a number of items lower than that originally stated, the SRD will be resolved by a set of D/A statements (both in the shipper's receiver's books), originating at the shipping facility.

21. In the case where no satisfactory explanation is obtained and if the nuclear material involved is more than X Eg* the investigation shall mandatorily be continued, involving the carrier(s) and the intermediaries.

22. In the case where the quantities involved are lower than the above threshold, the case may be dismissed, but a note will be maintained for five years in the safeguards records of the facilities and carriers involved. The case cannot be dismissed in case of recidive.

23. For larger quantities (or in case of recidive) a comprehensive report must be prepared to provide the Safeguards Authorities with all the information to decide the appropriate follow-up actions and/or the appropriate actions. A similar follow-up action is required also if the SRD is resolved as mentioned in §20, when the suspicion exists that the origin of the SRD amended was not due to innocent causes.

SRD on Nuclear Material Quantities

24. The most common SRD case is that concerning the amounts of material contained in a batch. This case is considered in general terms. Special cases (e.g. transfers from reactor to reprocessing, non availability of all data — some RF or RN cases —, etc.) will be dealt with further on.

25. An SRD pertinent to one batch originates from the measurements of this batch in the shipping and receiving MBA. Each MBA uses its own measurement system, the general characteristics of which are known to and have been approved by the Safeguards Authorities. Such measurement system should comply with or be equivalent in quality to the international standard as laid down in art. 9 and art. 55 of /2/.

*) The current values of X used by Euratom Safeguards is 50 Eg.

26. The following notations are used:

Q_S = quantity stated by the shipper

Q_R = $Q_S + D$ = quantity measured by the receiver

D = Shipper/Receiver Difference ($D \neq 0$ in case of apparent loss in transit).

These measurements are affected by measurement errors, expressed in terms of variances ($\text{var } x = \sigma^2$) both of random (r) and systematic (s) origin. Subscript t stays for total, S for shipper, R for receiver:

$$\sigma_{tS}^2 = \sigma_{rS}^2 + \sigma_{sS}^2$$

$$\sigma_{tR}^2 = \sigma_{rR}^2 + \sigma_{sR}^2$$

27. The evaluation of D will be carried out in steps making use, when appropriate, of the statistical test for the significance of the difference between the means of two distributions with known variances.

28. In the execution of the tests the **false statement risk α is taken equal to 0.05**. Obviously, if it is felt by the Safeguards Authority that a different level of risk has to be taken, α shall be chosen accordingly.

Step 1

29. The first test is carried out to judge if D is or is not compatible with the random measurement errors of the two involved systems. The null hypothesis

$$H_0 = \{|D| = 0\}$$

will be tested against

$$H_1 = \{|D| \neq 0\}$$

30. With obvious meaning of the symbols the null hypothesis H_0 will be accepted if $|D|$ is lower than the critical value

$$C_1 = u(\alpha) \sqrt{\sigma_{rR}^2 + \sigma_{rS}^2}$$

(For a double sided test $u(0.05) = 1.96$.)

31. If H_0 has not to be rejected, it is suggested to decide that D is not significant for safeguards and that all the relevant reports may be judged trustworthy. The evaluation process for the individual D considered is then stopped.

32. If $|D| > C_1$ a second test will be carried out to establish if D is or is not compatible with the combination of all the quoted measurement errors. H_0 and H_1 are as in §29, but now the critical value is

$$C_2 = u(\alpha) \sqrt{\sigma_{tS}^2 + \sigma_{tR}^2}$$

33. If H_0 has not to be rejected, D is still not significant from the safeguards point of view.

34. However a $|D|$ laying in the interval C_1, C_2 :

$$C_1 < |D| < C_2$$

is an indication of a possible systematic difference between the two measurement systems involved. A long term follow-up may be envisaged and therefore a **note for the safeguards records** will be made as for the case mentioned in §22 above. The evaluation process for the individual D considered is then stopped.

35. If $|D| > C_2$ the inspectors shall try to establish the reason(s) for this anomalous difference. However, before passing to the "enquiry" another two tests will be performed to situate the magnitude of D in respect of the current knowledge of possible biases between laboratories. Therefore steps 3 and/or 4 will be executed: at this point of the evaluation process a statement along which "D is not compatible with the measurement errors of the two systems" it is still somewhat risky. In fact D could be due to a high relative bias between the two systems.

Step 3

36. If some of the random or systematic variances used for the test are smaller than the **international standards*** a new critical value C_3 will be computed. The subscript T in the formula stays for "target value".

$$C_3 = u(\alpha) \{ \text{Max}(\sigma_{rS}^2, \sigma_{rT}^2) + \text{Max}(\sigma_{sS}^2, \sigma_{sT}^2) + \text{Max}(\sigma_{rR}^2, \sigma_{rT}^2) + \text{Max}(\sigma_{sR}^2, \sigma_{sT}^2) \}^{1/2}$$

37. If $|D| < C_3$ the same conclusions and actions indicated in §33 and 34 above are suggested.

Step 4

38. A last test is recommended before passing to Step 5, in fact the possible involvements of unknown biases is a quite delicate matter. Many interlaboratory comparisons indicated that differences much larger than the claimed errors may exist between pairs of laboratories /4/. Indicating with B the larger discrepancy (absolute value) between laboratories in an intercomparison relevant to the same type of measurement in question, another critical value C_4 will be considered:

$$C_4 = B + C_1$$

39. In cases $|D| > C_4$ one shall pass to step 5.

40. In case $|D| < C_4$ the possibility of an innocent bias between the two measurement systems cannot be excluded. A record will be prepared including all the pieces of information relevant to steps 1 to 4 (obviously including the sign and size of D) and, if existing, the historical dossiers of the SRD for the two facilities in question (ref. §22, 34 above and §41, 47, 48 below).

41. In case $-X \text{ Eg} < D < Y \text{ Eg}^*$, it is left to the inspectorate the choice either to dismiss the cases (which nevertheless will be recorded for long term evaluation) or to continue the procedure.

Step 5

42. At this level of the procedure the **discrepancy D** cannot be considered explicable by measurement errors only. If $D < -X \text{ Eg}$ or $D > Y \text{ Eg}$ or if so decided (see §41 above) an enquiry shall be opened at the shipper's and receiver's facilities to understand the origin of the abnormal discrepancy.

*) In Euratom Safeguards the ESARDA (ECSAM) target values /3/ are currently used for these purposes.

*) The values of X and Y currently used in Euratom Safeguards are 50 Eg and 200 Eg respectively.

43. Obviously a positive D is less worrying than a negative. It cannot cover an attempt of diversion, but it is rather the indication of a possible error in the measurements and/or accounting operations. Such a positive D may be due to an overestimation of the receiver, an underestimation of the shipper or both, eventually appearing as a negative MUF (loss of material) in one or both Material Balances. The discovery of the possible error will be, in this case, welcomed by all the involved parties. A negative D could, in turn, cover a diversion attempted by the shipper or the receiver and is therefore a major safeguards concern.

44. A critical review must be made of all available operating and accounting records (of both facilities) relevant to the transfer in question. The inspectors shall, as far as possible, try to establish their own independent estimate of the quantity of transferred material. This may be very difficult to achieve because one should be in the position to check if the batch, already handled by the receiver, is physically the **whole** batch dispatched by the shipper.

45. The enquiry shall provide information on the performance of the measurement systems and of the technique of accounting for nuclear materials, and take into account any other fact pertinent to the SRD in question, that the skillfulness of the inspectors may note.

46. For transfers between facilities within the territory of the European Community the enquiry may be extended to the carriers and the intermediaries. The enquiry will include the examination and the evaluation of the records that these persons or undertakings must keep according to the European law (see arts. 32 to 34 of I/1).

47. If one of the parties (or both) rectifies his declaration, so that IDI becomes lower than one of the previous thresholds (C_1 to C_4)

- a) the enquiry shall be considered concluded,
- b) the accountancies of the two facilities must be rectified in accordance with the corrections,
- c) possible sanctions will be applied on the basis of the report on the enquiry,
- d) a note for the records must be prepared if so required (see, e.g., §34).

48. If the difference remains unresolved, the report on the enquiry will be used to take a decision on the appropriate follow-up, including the proposal to the Commission for the application of possible sanctions.

Particular Cases

49. There are cases for which the evaluation scheme described above is not appropriate or cannot be implemented. The most common are examined in the following.

Lack of data

50. In cases of materials imported (RF) or received from non safeguarded activities (RN) only batch data are stated by the shipper. In most of the cases the shipper's measurement system characteristics and the measurement errors on the shipper's stated quantities are unknown to safeguards. This hampers carrying out the evaluation process as mentioned above.

51. In such cases the (unknown) shipper's errors will be supposed equal to those of the international standards of measurements. In such conditions steps 1 to 4 may be executed.

52. Step 5, if required, makes much less sense, being impossible to carry out an enquiry at the shipper's facility. However, for RF involving shipping facilities safeguarded by the IAEA, a cooperation with that organization will be asked for; for RN a similar cooperation may be requested to the competent authorities of the relevant member state.

53. Similar solutions will be taken for RFs, with significant SRDs, coming from third states with whom the Community has a cooperation agreement.

54. The RFs and RNs usually require an attentive verification because the redundant verifiable information at the shipping facility is, normally, not available. These verifications are aimed at the confirmation of the receiver's measurements. Once the inspectors are satisfied with the truthfulness of the amounts of materials **entered** under the Euratom responsibility, possible SRDs, for RFs and RNs, have no impact in the safeguards of the Community stocks, but have only the marginal interest of an apparent loss or gain at the Community's boundary.

55. A possible greater interest in such matters will exist when art. 91 of I/2/ will be practically implemented (transfer of responsibility in imports/exports with third states).

Transfers of spent fuel to reprocessing

56. A special case of SRD is that concerning the spent reactor fuel undergoing reprocessing. The first accurate physical measurement of the nuclear material contained in a spent fuel assembly (or element) may be made only after its dissolution at the head-end of a reprocessing plant. Before this measurement the last accurate determination of the nuclear material in the assembly is made at the fuel fabrication facility: the spent fuel data, issued by the reactor, are values calculated on the basis of its original (fresh) content and on the irradiation history of the assembly. This "measurement" is often inaccurate and therefore the SRD in question has only the sense of an adjustment of the books in which the rough reactor estimates are recorded as shipper's values.

57. In such circumstances the general approach for the SRD evaluation makes no sense. For such transfers the main preoccupation of safeguards shall be that of verifying that **all and only** the spent fuel leaving the reactor enters into the dissolver and that the correct measurements pertinent to such material enter in the accountancy.

58. The historical trend of the SRDs for material of single reactors has, nevertheless, an interest for all the parties involved. The detection of possible systematic errors in the reactor calculations may help their improvement. This will allow a better estimate of the quantities of safeguarded material waiting for reprocessing.

SRD, which are not SRD

59. Attention must be paid to the physical process used for SRD determination. In fact, sometimes, quantities of material not measured or difficult to measure are called SRDs by the operators. It is not unlikely that, for the sake of simplicity, such a practice is accepted by safeguards but it is important that the origins of such declarations are known in order that a wrong evaluation is not carried out leading to wrong conclusions. Examples of such cases follow.

60. The measurements of the UF_6 amounts in large cylinders of natural or LEU cylinders are usually obtained by subtracting the tare weight from the gross weight of the same cylinder before its

emptying. This is perfectly correct in the hypothesis that the tare of the empty cylinder is made when it is "completely" empty. But, in practice, some heels which are difficult both to extract from the cylinders and to measure, remain in the "empty" cylinders. This obviously influences a possible apparent SRD.

61. When the costly determination of the residuals or the cylinder cleaning is not a normal operation practice it is usual to allow the improper SRD declaration, which includes the residuals in the containers.

62. If later on the "empty" cylinders are cleaned, it is important to know the amounts recovered (or, at least, estimate of these mean residual quantities). This knowledge shall not be used to impose any SRD retroactive corrections, but it is important for the long term SRD evaluation. If the recovered amounts are not taken into account, the evaluation process may indicate a (non existing) bias between the two facilities involved in the UF₆ cylinder transfers.

63. Another possible misuse of the reporting code DI is relevant to nuclear transformations occurred in the shipping facility but accounted for in the receiving facility and reported as SRDs. There may be reasons for which such anomalous use of the DI code has been tolerated, or even agreed upon with the Safeguards Authorities. What is important is that those called to evaluate the reports are aware of this DI conventional meaning and deal with these entries as with NT entries.

64. Attempts should be made, whenever possible, to stop this practice, possibly introduced because of incorrect declarations by the shipping facility: no declaration of NT and shipment of irradiated material as fresh.

Serials of SRDs between Pairs of facilities

65. It often happens that one facility is normally fed with nuclear material of a typical form and category by another facility. If for such transfers SRDs are established on a routine basis, it is useful to perform a comprehensive SRD analysis for these pairs of facilities.

66. Such analysis is meaningful only if the receiving facility declares regularly an SRD for each received batch, even if the detected values are positive (apparent gain in transit) or zero (no difference detected in receiver's measurements).

67. This analysis will normally be performed after the closure of the receiver's MB period and will cover such a period. It will cover "homogeneous" SRD data sets. The homogeneity of a set requires that for each SRD of the set the following five identifiers are the same:

- (1) Shipping facility
- (2) Receiving facility
- (3) Category of nuclear material
- (4) Shipper's error path
- (5) Receiver's error path.

68. This analysis will not be mandatory in case each SRD of the set has been considered (at the moment of the single SRD evaluation) to be compatible with the random errors of the two facilities (see §30: $ID_1 < C_1$).

69. Attention must be paid if a marked prominence of one sign (+ or -) is noted in the set of SRD under examination. A test should be performed in this case to explore if this may be the result of a systematic difference between the involved measurement systems.

70. In the following the same notations used in §26 will be implemented, the subscript *i* indicating the *i*-th batch of the set. It follows that now

$$D = \sum D_i$$

is the total relative (with sign) difference between the receiver's and the shipper's measurements pertinent to the considered set. Moreover use will be made of the **relative** standard deviation, δ , which is linked to the **absolute** standard deviation, σ , pertinent to the (absolute) amount *Q* by the relationship

$$\delta = \sigma/Q.$$

71. Once again it is suggested to take the false alarm risk, α , equal to 0.05. For a double sided test this means that $u(\alpha) = u(0.05) = 1.96$. Obviously if the inspectors feel that a different level of risk has to be taken $u(\alpha)$ shall be chosen accordingly.

72. The path along which each SRD set will be evaluated is similar to that followed for single SRD values. The objectives of the analysis are :

- a) to establish, by means of a significance test, if the zero hypothesis $\{D = 0\}$ may be accepted;
- b) to establish, in case of rejection, an estimate of the possible relative bias between the two involved measurement systems.

Step 1

73. A first test is performed to assess whether or not the overall difference *D* is compatible with the sole random measurement errors of the two systems involved. The null hypothesis

$$H_0 = \{D = 0\}$$

will be tested against the alternative hypothesis

$$H_1 = \{D \neq 0\}$$

74. The critical value, V_1 , of this test is

$$V_1 = u(\alpha) \sqrt{\text{var}_r D}$$

where

$$\text{var}_r D = \delta_{IS}^2 \sum Q_{Si}^2 + \delta_{IR}^2 \sum q_{Ri}^2.$$

75. If H_0 cannot be rejected the process is stopped and the possible difference *D* is accepted as a product of the random errors of the measurement systems considered.

76. If H_1 cannot be rejected it will be concluded that the two measurement systems present a relative measurement bias. The analysis will continue with step 2.

Step 2

77. If $|ID| > V_1$ a second test will be performed to assess if the overall difference is compatible or not with the random and systematic errors characterizing the two measurement systems. The total variance of *D* may be well approximated by :

$$\text{var}_t D = \text{var}_r D + \delta_{SS}^2 (\sum Q_{Si})^2 + \delta_{SR}^2 (\sum Q_{Ri})^2$$

so that the critical value for the second test will be

$$V_2 = u(\alpha) \sqrt{\text{var}_t D}.$$

78. If the realised $|ID|$ is smaller than V_2 (but larger than V_1) it is possible to conclude that a systematic difference between the

performances of the two measurement systems may explain such a D value. No further action may be envisaged because the δ_s have been considered already at BTC verification level as compatible with the international standards. However it is useful that the operators are informed of the results of these evaluations.

Step 3

79. The value D is the best available indicator of the systematic difference between the measurement systems. The relative bias is $D/\Sigma Q_{Ri}$ for the receiver and $-D/\Sigma Q_{Si}$ for the shipper. Because usually D is significantly lower than each of the two sums, the absolute value of the relative biases is very similar and we will indicate such a value with

$$b = |D| / \Sigma Q_{i(S \text{ or } R)}$$

the standard deviation of b being $\sqrt{\text{var}_1 D / \Sigma Q_i} = \delta_b$.

80. The value b will be compared with the relevant international standard that will be indicated here with b^* . The following scheme is proposed:

- i) $b < \text{Max}(2b^*, 2\delta_b)$ no action
- ii) $b > \text{Max}(2b^*, 2\delta_b)$ submission of a standard for analysis to both installations for re-checking their individual performances. The evaluation of this verification may lead to imposing a corrective action to one or both laboratories, as necessary.

81. A corrective action seems to be necessary when the possible difference between the laboratory determination and the standard value (unknown to the laboratory) is larger than twice the overall laboratory uncertainty (at 1σ level) or twice the international standard (at 1σ level), whichever greater. (It is expected that a corrective action on at least one laboratory has to be undertaken whenever $b > 4b^*$.)

Warning !

82. Before any evaluation process is initiated it is vital to be assured that the analysis is made on **real SRDs**: attention must be paid to the particular cases mentioned in §56 to 64 above for which the above evaluation pattern is not pertinent or appropriate. In cases similar to those of the example of §59 to 62 the evaluation may still be done once the **real SRDs** have been re-established.

Treatment of "Flagged" SRDs

Item differences (§22)

83. The case has already been dealt with in §23 above.

Quantity differences (§34, 37 and 41)

84. The evaluation pattern design for single SRDs indicates three cases in which anomalous SRDs have to be recorded (**flagged**) for possible further consideration. The term anomalous, in this context, means that the declared amount is not consistent with the random errors of the two measurement systems involved. The three cases considered are listed in the following, together with a score, in increasing order of safeguards seriousness.

Case	Score*
$C_1 < D < C_2$	A
$C_2 < D < C_3$	B
$C_3 < D < C_4$	C

85. The flagged records will be kept "per facility" (both shipper's and receiver's) and, when applicable, "per error path". Whenever a case is flagged, its score is accumulated in the two indicators of the involved facilities.

86. Whenever a flagged SRD has been included in the evaluation of one SRD set (see §65 to 82) the relevant score will be subtracted from the indicators of the relevant facilities. However a historical record of the flagged SRDs will be maintained for any further possible use.

87. When the indicator of one facility (all flag mixed) becomes equal or larger than Z^* an action must be initiated to clarify the situation. This action shall be carried out, when applicable, along the pattern indicated for serials of SRD evaluation. If the available evidence is not sufficient for a statistical evaluation the BTC of the involved measurement system must be reviewed. If an analytical laboratory is involved, a calibrated sample unknown to the laboratory will be submitted to it for analysis in view of checking its actual performances (see for reference §80).

The Status of the Art in Euratom Safeguards

88. It is well-known that the safeguards reports received by the Euratom Safeguards Directorate from all the facilities in the European Community are (when necessary) transformed in computer readable form and stored for any further use.

89. It follows that the mechanical part of the SRD analysis may be performed automatically as soon as the appropriate software is available. On the basis of the procedures above described, a system has been designed capable of :

- detecting from the ICRs all the SRD declarations;
- tracing the relevant receipt and shipment declarations;
- computing the critical values C_1 to C_4 to be used for the evaluation of each SRD.

90. The relevant software has been written and at present its performances, together with the available inputs, is in the testing and implementation phase. All SRDs declared during the last years are being screened.

91. After the completion of this phase, it is planned to extend the software so that the evaluation of SRD sets may be added to the evaluation of single SRDs.

92. The results of these evaluations and the subsequent judgements will be the object of reports to the IAEA, in accordance with art. 32(b) and (c) of /2/.

*) The Euratom Safeguards is studying the possibility of using for A,B,C and Z scores of 1,2,3 and 6 respectively.

References

/1/ Commission Regulation (Euratom) No. 3227/76 concerning the application of the provision on Euratom Safeguards
/2/ INFCIRC 193 (Euratom-IAEA Safeguards Agreement)
/3/ ESARDA Target Values may be found in ESARDA Bulletin No. 6 (April 1984) and in paper IAEA-SM-293/21 presented at the IAEA Symposium (10-14 Nov. 1986) by De Bièvre et al.
/4/ Maximum interlaboratory discrepancies may be deduced from laboratory intercomparisons: JEX-70, IDA-72, IDA-80.

Symbol List

SRD Shipper Receiver Difference
MB Material Balance

MBP Material Balance Period
KMP Key Measurement Point
IC Inventory Change
ICR Inventory Change Report
PIL Physical Inventory Listing
PIT Physical Inventory Taking
D/A Deletion/Addition technique used for reporting corrections in ICRs
BTC Basic Technical Characteristic of facilities to be provided by the facility operator to the Safeguards Authority - Similar to Design Information /2/

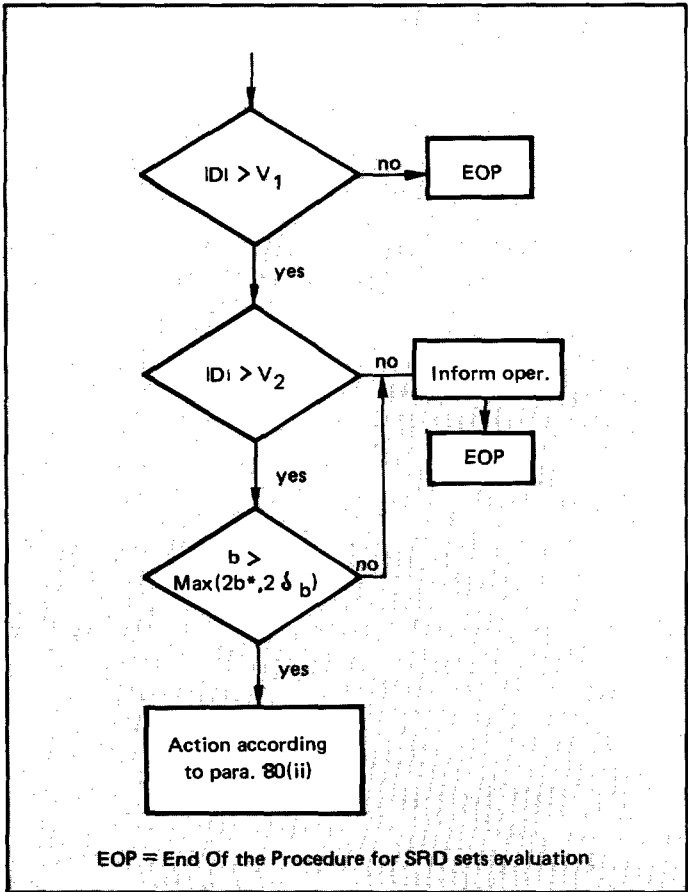
IC codes to be used for reporting purposes

RD Receipt Domestic
RF Import (Receipt Foreign)
RN Receipt from Non-safeguarded activity
DI SRD direct declaration

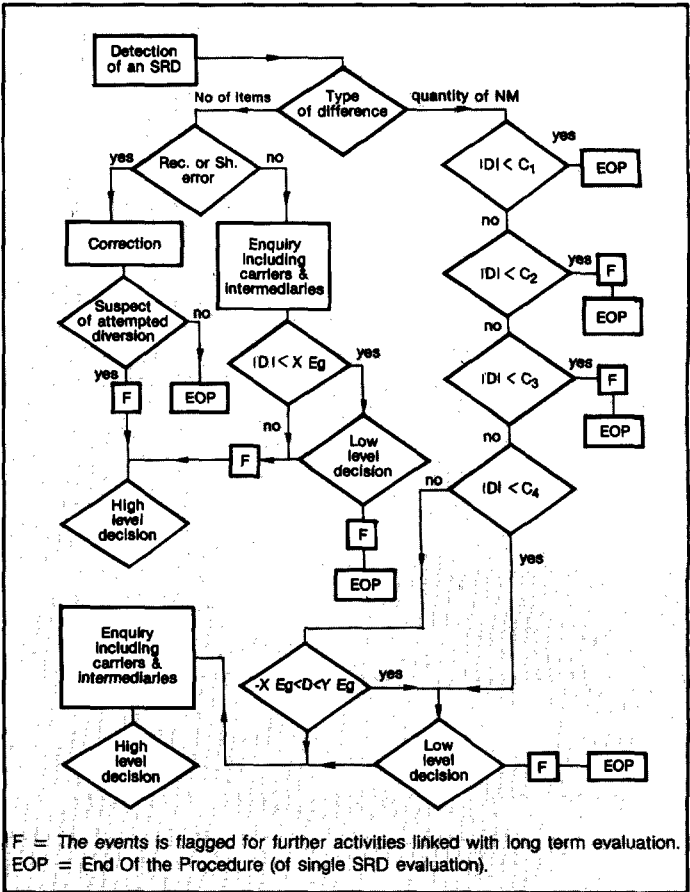
NM New Measurement. Difference between a new and a formerly measured nuclear material quantity, when such a difference is not an SRD or a correction.
NT Nuclear Transformation. Equivalent to Nuclear Loss (LN) and Nuclear Production (NP) used under INFCIRC 153 Agreements

Measurement Basis codes to be used for reporting purposes

N Not measured in the reporting MBA
T Tagged, because already measured and reported in the reporting MBA
L Labelled, because already reported as measured in another MBA
F As N, but estimated
G As T, but estimated
H As L, but estimated.



Evaluation Scheme for SRD Sets



A Training Course for IAEA Inspectors at JRC-Ispra

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Introduction

From the 6th - 17th July 1987 the Ispra Establishment of the Joint Research Centre of the Commission of the European Communities (CEC) was the site of an integrated auditing course for IAEA and Euratom inspectors. This was the first training course of this kind for IAEA inspectors staged under the CEC technical support programme to the IAEA. The course, which took the form of a two week case study of verification activities in a reference Highly Enriched Uranium (HEU) plant, was prepared under a collaboration between US Department of Energy (DoE) and the CEC. For the US DoE the course came under the Programme of Technical Assistance to IAEA Safeguards as directed by the International Safeguards Project Office (ISPO) at Brookhaven National Laboratory. This HEU physical inventory verification exercise (HEU PIV) was based on an earlier HEU PIV exercise created by Los Alamos National Laboratory (LANL) for IAEA inspectors which took place in September 1985. The technical content of the Ispra HEU PIV exercise was prepared in collaboration by Los Alamos National Laboratory, IAEA, the Euratom Safeguards Directorate of the CEC, NUKEM GmbH in the F.R. Germany and the Ispra Establishment of the JRC.

Course Objectives

The HEU PIV exercise was a case study which included all inspection activities related to accountancy and verification of physical inventory at an HEU fabrication facility. The exercise was designed as a course for experienced inspectors and integrated three main elements. The first element was the logic of the plant accounting system, the computation of MUF and the measurement standard deviation of MUF. The second element was verification planning (sampling theory and selection of measurement approach) and verification measurements for a typical variety of material types. The third element covered data processing to evaluate operator inspector differences, material balance evaluation in the form of MUF-D and completion of inspection reports.

At the planning stage the specific objectives of the course were identified as

- provide safeguards inspectors with experience and training in NDA techniques developed for the verification of HEU inventories at bulk handling facilities,
- provide an opportunity to test and evaluate different PIV procedures and strategies,
- provide safeguards inspectors with experience and training in record auditing, accountancy and material balance evaluation procedures (MUF-D),
- provide an opportunity for the exchange of information on PIV procedures between different IAEA operational sections and between IAEA and Euratom,
- provide an opportunity to test and evaluate the use of NDA equipment in conditions similar to those found during actual inspections,
- assess the usefulness of current computer programs for analysis of data.

Twelve inspectors (eight IAEA, four Euratom) participated in the course. They were divided into three teams (two IAEA, one Euratom) of four inspectors. Each team worked through the case study in parallel with the others.

Course Content

It is impossible, in a brief description, to do justice to the complexity of the course contents. The contents were briefly as follows. The first phase was a familiarisation with the measurement instruments and their calibration. The instruments were the Active Well Coincidence Counter (AWCC), the Davidson MCA with both NaI and Ge detectors, the Enrichment Meter, PHONID, the Ispra Gamma Scanner, the ultrasonic thickness gauge and balances.

The second phase focused on the safeguards features of an HEU plant, the operators accounting system, MUF and MUF standard deviation. This phase included a detailed presentation by the NUKEM staff member responsible for safeguards, of the operating records and audit

trail which form the basis for verification activities. NUKEM provided realistic accounting declarations for MUF and MUF standard deviation computations. These were computed during the course by means of the DBMS based software SASTIS which is available at IAEA.

The third phase covered verification planning applied to the ending inventory of the material balance declarations supplied by NUKEM. Each team applied stratification and sampling methodology to these data. The participants had the opportunity to discuss planning methodology and in particular the newer statistical formalisations which take account of the integration of sampling plan and measurement approach.

The fourth phase consisted of verification measurements of the samples of material selected for verification. The material measurements which formed this phase of the course are shown in Table 1 which also shows the instruments available for each type of material. The measurement phase was carried out in the Pre-PERLA laboratory facilities using the HEU material available at Ispra.

Table 1 - Allocation of Material and Instruments to Teams on Days 4-7

Day	Team		
	1 IAEA	2 IAEA	3 EUR
4	A $\alpha + \beta$	B α	C $\alpha + \gamma$
5	D $\alpha + \delta$	A $\alpha + \beta$	B $\alpha + \epsilon$
6	C $\alpha + \gamma$	D $\alpha + \delta$	A $\alpha + \beta$
7	B α	C $\alpha + \gamma$	D $\alpha + \delta + \epsilon + \beta$

Material	A = UO ₂ powders
	B = Particles, platelets (Bildchen), metal
	C = MTR assemblies and plates
	D = Scrap, UF ₆
Instruments	α = AWCC + PMCA + EBAL
	β = PHONID
	γ = Gamma-scanner
	δ = PMCG
	ϵ = Enrichment Meter

The fifth phase was evaluation of the NDA data generated during the course. The participants tested statistically the differences between operator declarations and their own measurements and went on to evaluate the performance of the different instruments on the various material types.

The sixth phase was the creation of a D statistic for the verified inventory. This was based on DA measurements which were simulated for the course. The participants then computed some components of the MUF-D analysis. For this purpose the participants had available an extended version of the SASTIS software providing the testing of the D statistic, the sampling distribution of the D statistic, inspector estimation of inventory, etc. One of the interesting consequences of this data analysis was that it allowed participants to get a feeling for the effect on the overall statement of the fact that Destructive Analysis (DA) verification measurements are not normally available for some stratum of material. The same analysis was then carried out using NDA data for the strata lacking DA data. In addition participants were able to carry out the analysis using entirely their own NDA data. These analyses and the discussions they provoked helped participants to understand the relationship between measurement quality and detection sensitivity for a variety of diversion scenarios.

Course Preparations

The design and preparation of the course was a collaboration between LANL, IAEA, Euratom Safeguards Directorate Luxembourg, NUKEM and JRC-Ispra. It involved a significant work effort for a total of about 25 staff members of these organisations. A group of four coordinators (one for each of the organisations) was created to instigate and coordinate the preparations which included:

- preparation of a course manual covering all aspects of HEU verification,
- preparation and documentation of measurement procedures,
- data evaluation software development,
- transportation of instruments,
- a two week calibration exercise (June 1-13, 1987) to create calibration curves

for each combination of instrument and material type.

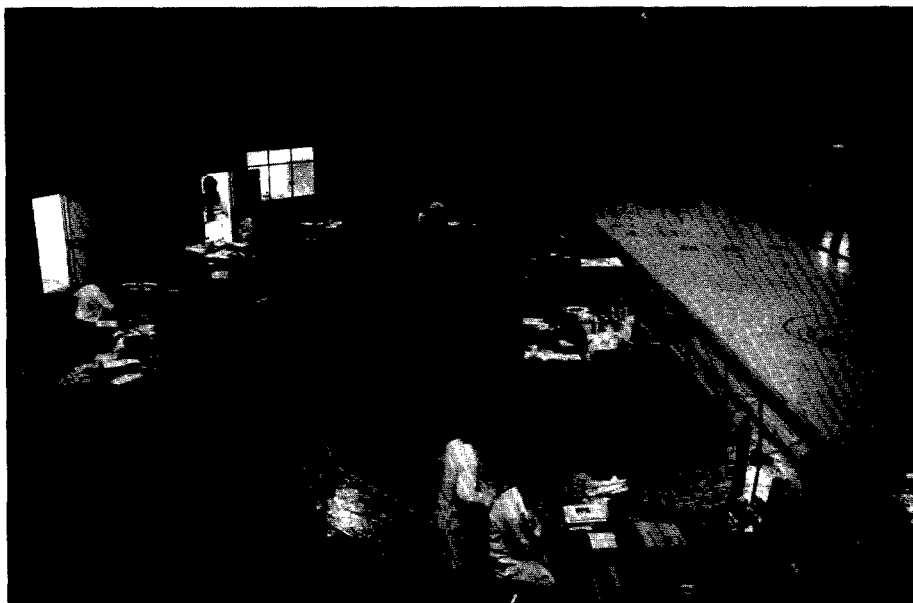
It is normal in a training course as ambitious as a PIV exercise, that the designers attempt to add to what has been achieved by earlier PIV exercises. This course was no exception to this pattern. The innovations which gave it its own particular flavour included:

- the participation of NUKEM providing a wealth of detail on plant operating records as well as realistic data sets for MUF evaluation and inventory verification;
- the availability of SASTIS software for MUF, standard deviation of MUF, MUF-D, etc.;
- the participation in the course of instruments developed by Ispra for Euratom inspectors (Enrichment Meter, PHONID, Gamma-Scanner). This allowed inspectors compare and contrast the operating characteristics of a wide variety of instruments.

Course Evaluation

As the final act of the course, the participants were asked to fill out a questionnaire giving their personal evaluation of the course. Judging by their replies the course seems to have been reasonably successful. They all felt that such a course should be offered to inspectors and that to this end it could usefully be repeated. The replies of the participants offered a variety of ideas which could be used to enrich future courses.

The author of this report feels that it is statistically significant that the course ran smoothly and that nothing went wrong on the day. This, however, was not due to chance. It was due to the extreme hard work of the many people in the participating organisations who scorned delights and lived laborious days in making sure that everything worked. It was very enjoyable to be a part of such dedicated teamwork and for this the author is thankful to them all.



General view of Pre-PERLA with the course participants performing NDA measurements

Spent Fuel Measurements at NPP Paks

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Abstract

Gamma spectra of seven WWER-440 assemblies of 1.6% initial enrichment discharged from the core after the first campaign have been taken by using a collimator system built in the wall of the spent fuel pond. Activity ratios of ^{106}Ru , ^{134}Cs , and ^{144}Ce to ^{137}Cs have been evaluated for calculating Pu content, burnup, and cooling time, respectively.

Axial and azimuthal burnup distributions have been evaluated by a microcomputer adapted new code from as many as 420 spectra recorded. Results have been compared with calculated values obtained by the BIPR-5 code.

Introduction

The first 440 MWe PWR unit of NPP Paks went into operation in December 1982. First refuelling took place in 1984, when the fuel assemblies of 1.6% initial enrichment were taken out of the core finally. In May 1985, after about one year cooling, high resolution gamma spectrometric measurements have been carried out on seven assemblies both for reactor operational and safeguards purposes. In this paper we describe the preliminary results of this study.

Experimental

The original positions of the seven assemblies in the core are shown in Fig. 1.

A 120 cm long collimator system built in the concrete wall of the spent fuel storage pond has been used for the reduction of the

gamma-ray intensity. The collimator was constructed similar to the one used in NPP Bohunice, Czechoslovakia /1/. The tube was shut on the water pool side by a 5 cm thick steel plate. The assemblies were transported and kept in the front of the collimator by the refuelling machine for the period of measurements. Translation and rotation for a full scanning was also performed with the machine. The distance of the assemblies from the closing plate of the collimator was 15 cm in water during the measurements. The collimator enabled the detector to "see" the whole cross-section of the assemblies in segments of about 1 cm height.

Gamma spectra have been taken at 11 height positions (see Table 1) and each side of the hexagonal assemblies by a 35 cm³ coaxial Ge(Li) detector, coupled to a 4 K multichannel analyzer (type NTA of EMG, Budapest). The preamplifier was connected to the other part of electronics with 25 m long cables. Because of the high ambient temperature, detector and preamplifier were additionally cooled by compressed air.

Measurement point	1	2	3	4	5	6
Height in mm	543	763	1003	1243	1483	1723
Measurement point	7	8	9	10	11	
Height in mm	1963	2203	2443	2683	2883	

Table 1. Measurement positions along assembly length, counted from lower end of assembly

Most of the measurements lasted for 200 and some for 500 s, but single long term measurements (2000-5000 s) were also carried out for each assembly in order to provide data for calibration purposes. Altogether 420 spectra were recorded and analyzed to take relative axial and azimuthal intensity distributions.

The spectra have been read out from the MCA by a home-made interface, transmitted to a microcomputer, and stored on floppy disks. Evaluation of the spectra was performed by using a microcomputer adapted version of a new code developed by one of the authors (J.S.).

Results and discussion

Activity ratios of $^{106}\text{Ru}/^{137}\text{Cs}$, $^{134}\text{Cs}/^{137}\text{Cs}$, and $^{144}\text{Ce}/^{137}\text{Cs}$ have been established for determining relative Pu content, burnup,

and cooling time, respectively. These data can be evaluated on the basis of the actual operational data (irradiation history), as a result of running the code BETTY /2/. This is under progress now. So far we could, however, evaluate burnup (BU) on the basis of an empirical formula of Golubev /3,4/, as

$$\text{BU}(\text{kg/TU}) = (16.6 \pm 0.9) R$$

where R is the $^{134}\text{Cs}/^{137}\text{Cs}$ activity ratio. BU in MWd/kgU was obtained from this by multiplying with 0.944.

For determining activity ratios from the measured peak areas, besides half-life and gamma yield data taken from the literature, experimental data were also needed for detection efficiencies, and corrections for the relative energy dependence of the gamma beam transmission through the water layer + steel plate combined with that of self-absorption. These were automatically taken into account by making use of the intrinsic calibration method based on the evaluation of ^{134}Cs peak areas in the spectra of long term measurements carried out for each assembly. All the fission products (subjects to this investigation) with their gamma lines of interest, falling into the energy interval between 600 and 800 keV, are listed in Table 2.

Gamma-ray		Fission product	Half-life d
energy keV	yield %		
604.7	97.6	^{134}Cs	752.4
621.9	9.81	^{106}Ru	371.6
661.6	85.0	^{137}Cs	11008.6
696.5	1.34	^{144}Ce	284.9
795.8	85.4	^{134}Cs	752.4

Table 2. Data of fission products measured

On the basis of the evaluated peak areas and the actual irradiation history, by running the code BETTY more precise determination of BU will be possible on one hand, and verification of the cooling time and determination of relative Pu-content in the assemblies on the other hand.

Axial and azimuthal burnup profiles evaluated on the basis of Golubev's formula can be seen on Figs. 2a and 2b. Axial profiles are exemplified here on one side only. On the abscissa of the axial distribution the measurement position is indicated in m, whereas on the ordinate BU is calculated in MWd/kgU, from the $^{134}\text{Cs}/^{137}\text{Cs}$ ratio. On the

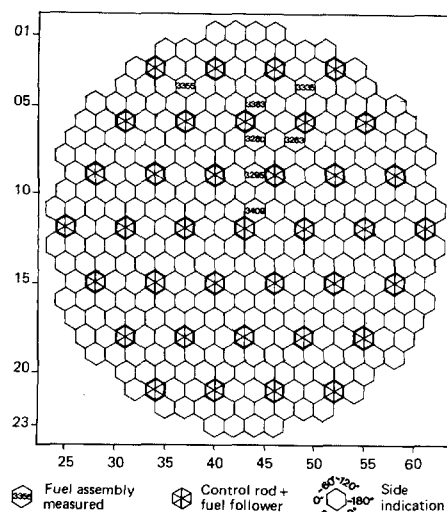


Fig. 1 - Reactor core map of Paks Unit 1 indicating positions of assemblies measured

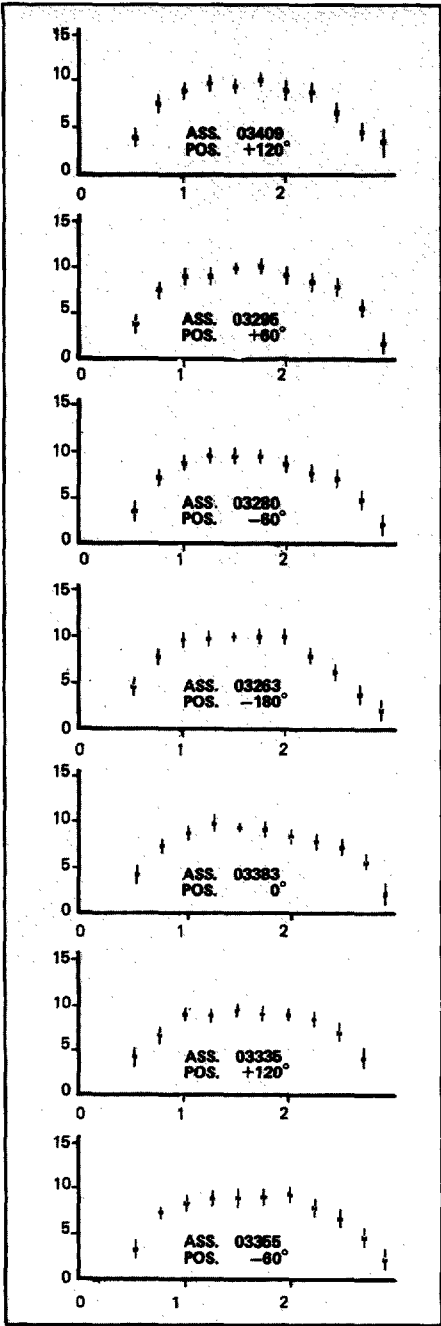


Fig. 2a - Axial BU distributions of the seven assemblies

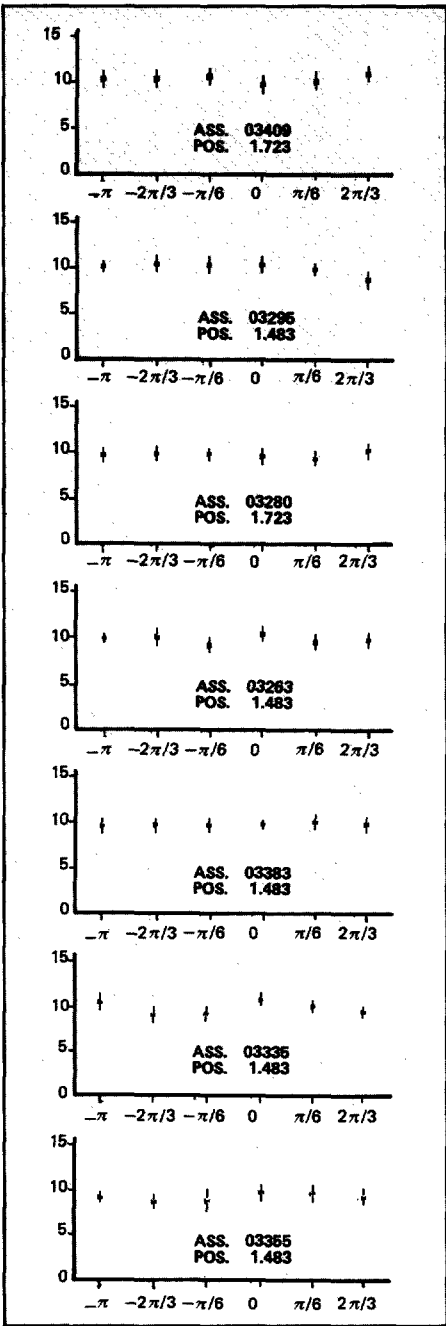


Fig. 2b - Azimuthal BU distributions of the seven assemblies

abscissa of the azimuthal distribution the angular position read on the refuelling machine can be seen, while on the ordinate BU is represented again. Error bars are mainly due to the error of the formula. This source of error will be eliminated by using values provided by BETTY. Azimuthal distributions are shown either in the 5th or

6th height positions. BU values averaged over the six sides of the individual assemblies taken in 5th height positions, as the function of the original assembly positions in the core can be seen in Fig. 3. Table 3 shows a comparison between the mean BU values of Fig. 3 and those provid-

ed by the BIPR-5 code /5/, which considers each assembly as a single point. Agreement was obtained within to 10% in the absolute values, but the trend of the two sets of values is very similar.

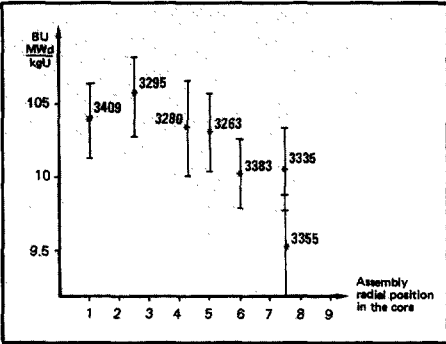


Fig. 3 - Mean BU values at the 5th axial position of single assemblies vs original assembly position

Conclusions

Examination of axial and azimuthal BU distribution proved to be possible with the method described above. Absolute BU values have been determined by using an empirical formula, which is not accurate enough, but provided data which are still consistent in some sense with those calculated by the widely used BIPR-5 code. More exact results are expected by employing computer codes taking into consideration spent fuel history, which makes possible validation of burnup codes, as well as verification of cooling time and Pu content for safeguards purposes at the same time. Radial BU distribution within the core indicate a good qualitative agreement with the one calculated by the BIPR-5 code.

Acknowledgement

This work was supported by the Hungarian Atomic Energy Commission.

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Assembly No.	3409	3295	3280	3263	3383	3335	3355
BU from this work	10.3 ± 0.27	10.55 ± 0.28	10.34 ± 0.33	10.31 ± 0.27	10.03 ± 0.24	10.06 ± 0.28	9.53 ± 0.36
MWd/kgU from BIPR-5 code	11.51	12.10	11.67	11.34	11.09	10.87	10.87

Table 3. Mean BU values as determined in this work and by the BIPR-5 code

Automated Material Control and Accountability for the Chemical Manufacturing Area of a Fuel Fabrication Plant

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Abstract

Control and Accountability for special nuclear material (SNM) within the Westinghouse Commercial Nuclear Fuel Division's Columbia Plant has been completely automated. The enabling computerized systems function within the boundaries of four key facility activities: receipt, chemical processing, mechanical processing, and shipment. SNM received onsite and processed in the Chemical Manufacturing Area is controlled by the Chemical Area System (CHAS); a near real-time system which utilizes the concept of tracking material within discrete, uniquely identified containers. SNM processed through the Mechanical Manufacturing Area, to shipment offsite is similarly controlled by the Rod Accountability and Monitoring System (RAMS).

Introduction

CHAS is an online, near real-time computer system which provides for tracking and controlling of SNM within discrete, uniquely identified items which move from one location to another within the Chemical Manufacturing Area. Accuracy of data entry is essential to maintain compliance with regulatory requirements. The system utilizes a state-of-the-art man-to-machine interface for data entry and retrieval designed to optimize data integrity and to minimize manual data input and material movement errors. The main features of the system include:

- simple, menu driven, operator transaction screens,
- automatic, hand held, scanning laser barcode readers for input of operator, location, and item identification,
- automatic weigh scales that feed measurement data electronically to a control computer,
- industrial grade workstations with modular design,
- detailed editing of transaction data prior to update of database,
- portable barcode readers for collection of inventory data,

- automated inventory data collection software for verification of items and tampersafe seals,
- detailed reporting capability for process, control, and accountability needs.

Background

The United States Nuclear Regulatory Commission under the auspices of Title 10 CFR 70 regulations, requires a system of storage and internal handling controls to be established, maintained, and followed to provide current knowledge of the identity, quantity, and location of all special nuclear material (SNM) contained in discrete items within a nuclear processing plant. Such activities are generally known as "Item Control". Prior to the implementation of CHAS, item control was laborious and time-consuming for it depended to a large extent on the maintenance of manual records. Timely access of the Chemical Area data was not possible and data entry errors along with subsequent corrections resulted in substantial wasted manpower. For this reason, Westinghouse management decided to implement an on-line, near real-time item control system within the Chemical Manufacturing Area to complement the other existing plant systems and thus, provide the basis for a plant wide Material Control & Accountability (MC&A) system. As previously reported /1,2/, accountability of special nuclear material within the Mechanical Processing and product shipment portion of the Columbia facility has been completely automated and is controlled by the Rod Accountability and Monitoring System (RAMS).

System Objective

The primary function of the Chemical Area Computer System is to maintain compliance with regulatory control and accountability of SNM. In order to meet these requirements, software subsystems were

created to achieve compliance with the individual objectives listed below:

- Item Control
- Measurement Control
- Traceability
- Inventory.

Additional functionality was included to enable others to benefit from this new system and includes:

- Production Report Capability
- Process Control Software
- Production Planning & Control Reports
- Customer & Quality Control Support
- Ad-Hoc Inquiry Capability.

Finally, the system was specifically designed to address known inadequacies within existing computer and manual systems by incorporating the following goals:

- minimize data entry errors,
- enhance ergonomic man-to-machine interface,
- utilize industrial grade workstations with modular design,
- utilize modular, menu driven software programs,
- perform front end data validation,
- provide meaningful error messages for operators,
- provide positive operator feedback for each transaction.

Hardware Description

CHAS manages data and provides specific capabilities by utilizing three distinct levels of computer hardware (Fig. 1). At the lowest level, microcomputer based workstations equipped with automatic data entry peripherals (i.e. laser barcode scanners, electronic scales, etc.) provide the user interface that is a major contributor to the success of the entire system. The second level consists of three area mini-computers which contain the transaction based software as well as the system databases. The databases at the area computer level contain information only for those items which currently exist within the Columbia

ESARDA News

10th ANNUAL ESARDA MEETING (Restricted participation)

Karlsruhe, 3 - 5 May 1988

The tenth Annual Meeting will be held at the Kernforschungszentrum Karlsruhe (KfK), F.R. Germany. The attendance will be limited to the ESARDA Steering Committee members, coordinators, working group members and observers.

The title of this internal meeting of ESARDA is :

Medium and Long Term Trends in ESARDA Working Groups' Activities

11th ANNUAL ESARDA MEETING

ESARDA announces that the 11th meeting will be a general **Symposium on Safeguards and Nuclear Material Management** and will be held in spring 1989.

A call for paper will be circulated in 1988.

In Memoriam

Jean Ley

Mr. Jean LEY, secretary of ESARDA died on 20 April 1987 at the age of 50. Since 1978 he ran the secretariat of ESARDA in a manner which was both efficient and friendly.

His total dedication to the job was always accomplished with that great optimism peculiar to him and he never failed to communicate this optimism and enthusiasm to all those who worked with him.

His jovial nature and his willingness to cooperate at any level will remain in the memory of all those who had the privilege of knowing him.



