

STR – 368 (Revision 1.1)

STR

**International Target Values
for Measurement Uncertainties
in Safeguarding Nuclear Materials**

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IAEA

International Atomic Energy Agency



***"ITVs are related to measurements
and therefore measurement
scientists have to be involved".***

E. Kuhn (first IAEA Safeguards
Technical Report, March 1993)

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1. Abstract

The objective of nuclear material accountancy (NMA) and its keystone, material balance evaluation (MBE), is to detect and deter the diversion of nuclear material by evaluating the nuclear material balances established through the State's systems of accounting for and control of nuclear material (SSAC) against the corresponding uncertainties and by verifying the correctness of the States' declaration through independent measurements. The effectiveness of NMA thus strongly depends upon the quality of both the facility operator's declarations and the inspector's verification measurements. In order to assess and compare the quality of bulk measurements (weight, volume), non-destructive assays (NDA) and destructive analyses (DA), and in continuation of the pioneering work of the European Safeguards Research & Development Association (ESARDA), a set of international target values (ITV) was first issued by the International Atomic Energy Agency (IAEA) in 1993 and is regularly updated and expanded, in collaboration between the IAEA and international experts.

The IAEA maintains a unique database of verification measurements taken over more than 30 years, paired with State declared values from nuclear fuel cycle (NFC) facilities under safeguards over the world. The database records undergo exploratory and statistical analysis and the IAEA evaluators use them to calculate initial random and systematic measurement uncertainty estimates. Based on these estimates, a network of international consultants from State or regional safeguards authorities (SRA), safeguards laboratories and NFC facilities reach a consensus on a revised set of ITV, i.e. measurement uncertainties that should be achievable under nominal conditions by typical NFC facility laboratories or during safeguards inspections. The role of all international stakeholders in understanding the importance of ITV in safeguards and providing expertise is therefore fundamental to warrant their quality, representativeness and achievability. The current ITV review was conducted with a particular focus on the role of international subject matter experts (SME) with the extension of the international SME network and the creation of a collaborative virtual ITV platform

2. Introduction: The Role of International Target Values (ITV) in Safeguards Accountancy and Verification measurements

One of the missions of the International Atomic Energy Agency (IAEA) in promoting the peaceful use of nuclear energy is to implement safeguards measures to detect and deter the misuse of nuclear material and technology. In the framework of the continued IAEA efforts to increase the ability of the safeguards system to provide independent and credible assurances that States are honouring their safeguards obligations, the critical role of the data collected by inspectors is re-affirmed as it provides an independent and authentic source of information resulting from the IAEA's unique access to nuclear fuel cycle facilities and related sites. In the framework of State-level approaches, the generic safeguards objectives for States with comprehensive safeguards agreements (CSA) are described as follows [1], [2]:

- to detect any **diversion of declared nuclear material** at declared facilities or locations outside facilities (LOFs);
- to detect any **undeclared production or processing** of nuclear material at declared facilities or LOFs where nuclear material is customarily used; and
- to detect any **undeclared nuclear material or activities** in the State as a whole.

The first generic objective, i.e. the detection of diversion of declared nuclear material, has long been and remains the core technical objective of safeguards implementation procedures under a CSA as described in § 28 of INFCIRC/153 (Corrected), Part II [3] : *The agreement should provide that the objective of safeguards is the timely detection of diversion of significant quantities of nuclear material from peaceful nuclear activities to the manufacture of nuclear weapons or of other nuclear explosive devices or for purposes unknown, and deterrence of such diversion by the risk of early detection.*

As is further described in § 29 and § 30 of [3], the objective of diversion detection is pursued by applying NMA to all declared *nuclear material*, i.e. any special fissionable or source material as defined in Article XX of the IAEA Statute [4]. Based on this definition, nuclear material includes material containing ^{239}Pu , ^{233}U , uranium enriched in ^{233}U or ^{235}U (fissionable material) as well as uranium containing the mixture of isotopes occurring in nature, uranium depleted in ^{235}U and thorium in all physical and chemical forms above specific concentrations¹ (source material). The principle of NMA and its keystone, material balance evaluation (MBE), is to detect and deter diversion by evaluating the nuclear material balances established through the SSAC against the corresponding uncertainties. Nuclear material inventories in nuclear fuel cycle (NFC) facilities and movements between NFC facilities are declared to the IAEA by the State through State reports for all material balance areas (MBA) within facilities and at regular intervals called material balance periods (MBP). The credibility of the IAEA's conclusions regarding the absence of diversion can only be warranted if the State's accounting declarations are verified by the IAEA's inspectors through independent observations and quantitative measurements.

¹ As the Board of Governors shall from time to time determine, Article XX, IAEA Statute.

Every year, thousands of days are spent by IAEA safeguards inspectors in NFC facilities around the world. A large portion of this time is used for carrying out in-field measurements by various non-NDA techniques and/or for taking DA samples for analysis by the safeguards analytical laboratory (SAL) in Seibersdorf, Austria or other IAEA certified laboratories of the IAEA network of analytical laboratories (NWAL). The effectiveness of the IAEA's verification strongly depends upon the quality of both the facility operator's declarations and the inspector's measurements (Fig. 1). A commonly agreed reference system is therefore needed to assess and compare measurement qualities. Paragraph 55 of INFCIRC/153 (Corrected), Part II [3] stipulates: *The Agreement should provide that the system of measurements on which the records used for the preparation of reports are based shall either conform to the latest international standards or be equivalent in quality to such standards.*

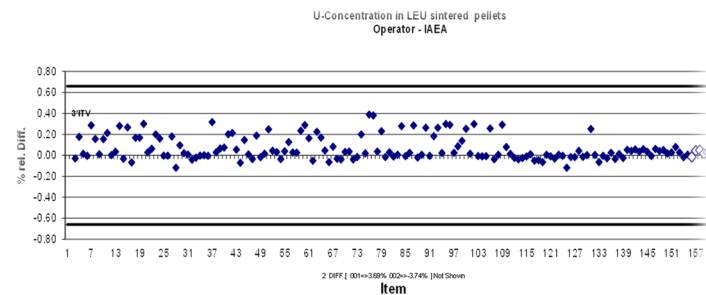


Fig. 1: Due to the negligible sampling uncertainty associated with the stable and homogeneous material in sintered pellets, pellet samples are regularly taken for DA to monitor the quality of the operators' measurement system at fuel fabrication plants.

In the 1970's, the IAEA established a set of international standards of nuclear material accountability defined as values of the measurement uncertainty δ_E (relative standard deviation) expected for closing a material balance, for different types of facilities [5]. Presently, the δ_E values can still be used as global limiting criteria in the field of material balance at material balance area (MBA) level. However, they are values reflecting measurement performance achievable by NFC operators several decades ago. They have not been revised and, importantly, they do not provide the reference framework needed for assessing the measurement uncertainties associated to specific measurands, specific DA or NDA methods and specific material types encountered in NFC facilities. For these reasons, a different approach was initiated in the late 1970's, leading to the introduction of International Target Values (ITV). The history and evolution of ITV is summarised in Section 3. Section 4 calls attention to the reasons to actively engage all stakeholders in the ITV 2020 revision process and describes the actions that were taken to this end. Section 5 describes the progress made in the field of uncertainty quantification and, in particular, in reconciling the IAEA statistical error model-based uncertainty quantification approach with the Guide to the expression of uncertainty in measurement (GUM).

3. What are ITVs?

ITVs are values of uncertainties, expressed as relative random and systematic error standard deviations associated with a single determination, which should be achievable under nominal conditions by a typical NFC facility laboratory on one sample analysis or as the result of an NDA measurement performed on a single item. It is important to note that ITVs should not be interpreted as uncertainties that can only be achieved under ideal laboratory conditions (Fig. 1 State of the Art). Likewise, they do not represent regulatory requirements, but rather a motivating goal – as their name “target values” suggests - and a reference for laboratories and safeguards inspectors and evaluators.

ITV estimates are derived from the operator-inspector paired measurements database maintained by the IAEA. Initial estimates are obtained by the IAEA analysts using a methodology based on Analysis of Variance and are subsequently reviewed by SME. By methodological design, ITV are intended to encompass all sources of measurement errors, including sources which remain unknown or are difficult to quantify.

They are applicable to measurements taken for safeguards purposes under nominal conditions (Fig. 1 State of Practice), with instrument specifications and measurement procedures equivalent to the respective entries in “Appendix II: Description of Measurement Methods listed in the ITV tables”. Under the above assumptions, ITV users include, but are not limited to: analysts from safeguards laboratories, NDA experts and practitioners, safeguards inspectors, safeguards evaluators, ...

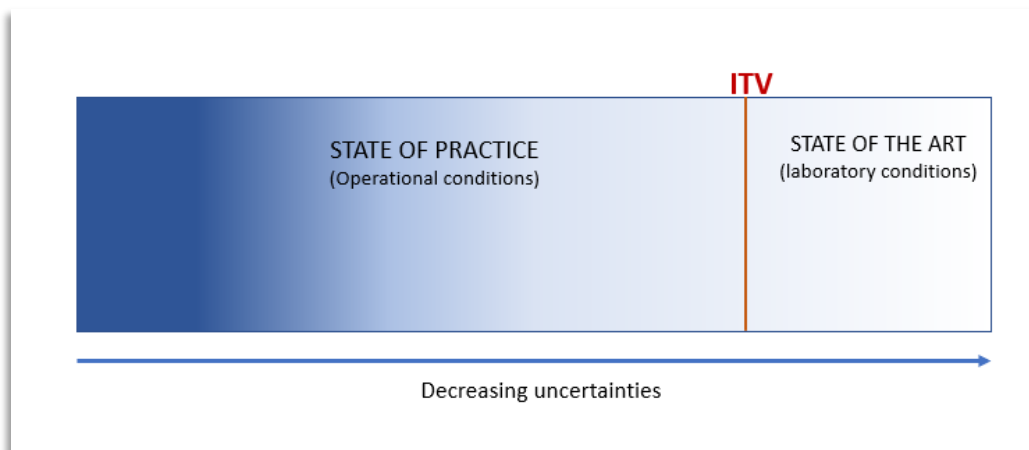


Fig. 2: International Target Values are measurement uncertainties that should be achievable under nominal conditions by typical NFC facility laboratories or during safeguards inspections, but should not be interpreted as uncertainties that can only be achieved under ideal laboratory conditions.

4. History of ITVs

In 1979, the Working Group on Techniques and Standards for Destructive Analysis (WGDA) of ESARDA opened the way by presenting a list of “Target Values” for uncertainty components in destructive analysis (DA) methods” to the IAEA and EURATOM [7]. After extensive consultations with NFC facility operators’ laboratories and safeguards organizations, these first target values were revised in 1983 [8]. In 1987, the ESARDA WGDA in collaboration with the Institute of Nuclear Materials Management (INMM) published attainable random and systematic uncertainties in elemental and isotopic analysis for the most significant material types encountered in the NFC using common DA methods. [9].

In the 1988 edition [10], the ESARDA WGDA and INMM agreed to define the values of the random error uncertainty resulting from sampling for elemental assays.

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

Specialists from four continents took part in the discussion of the proposed concept. The result was the publication of an IAEA Safeguards Technical Report (STR) in March 1993, entitled *1993 International Target Values for Uncertainty Components in Fissile Isotope and Element Accountancy for the Effective Safeguarding of Nuclear Materials* [11]. Articles in the ESARDA Bulletin [12] and in the Journal of the INMM [13] widely publicized the IAEA technical report. The report itself was translated into Japanese [14]. In 2000, international experts reviewed the experience gained with the use of the 1993 ITVs and the progress made in accountancy and safeguards verification measurements. Subsequently, an IAEA STR entitled *International Target Values 2000 for Measurement Uncertainties in Safeguarding Nuclear Materials* [15] was published in April 2001 in the ESARDA Bulletin [16] and in the Journal of the INMM [17].

Each issue of ITVs was related to a given year, reflecting a recognition that the quality of measurements may change, that new methods and instruments may be developed and implemented and that others may become obsolete. The ITVs also reflected the current understanding of the structure of the uncertainty components in nuclear material accountancy measurements.

The current version titled *International Target Values 2010 for Measurement Uncertainties in Safeguarding Nuclear Materials* was published in 2010 as STR-368 [6].

5. The Role of International Stakeholders

The participation of all parties involved in the use of ITVs warrants their quality, representativeness and achievability, ensuring that they can be used as a credible reference by the widest possible range of safeguards practitioners. As an early follow-up of a methodological debate initiated during the ITV-2010 CGM meeting (see Section 4), representatives were identified from the safeguards communities who can provide the different sources of expertise represented in Fig. 3, i.e. analytical laboratories, NDA laboratories, safeguards data evaluators from national and international inspectorates, NFC facility operators and State or regional authorities (SRA).

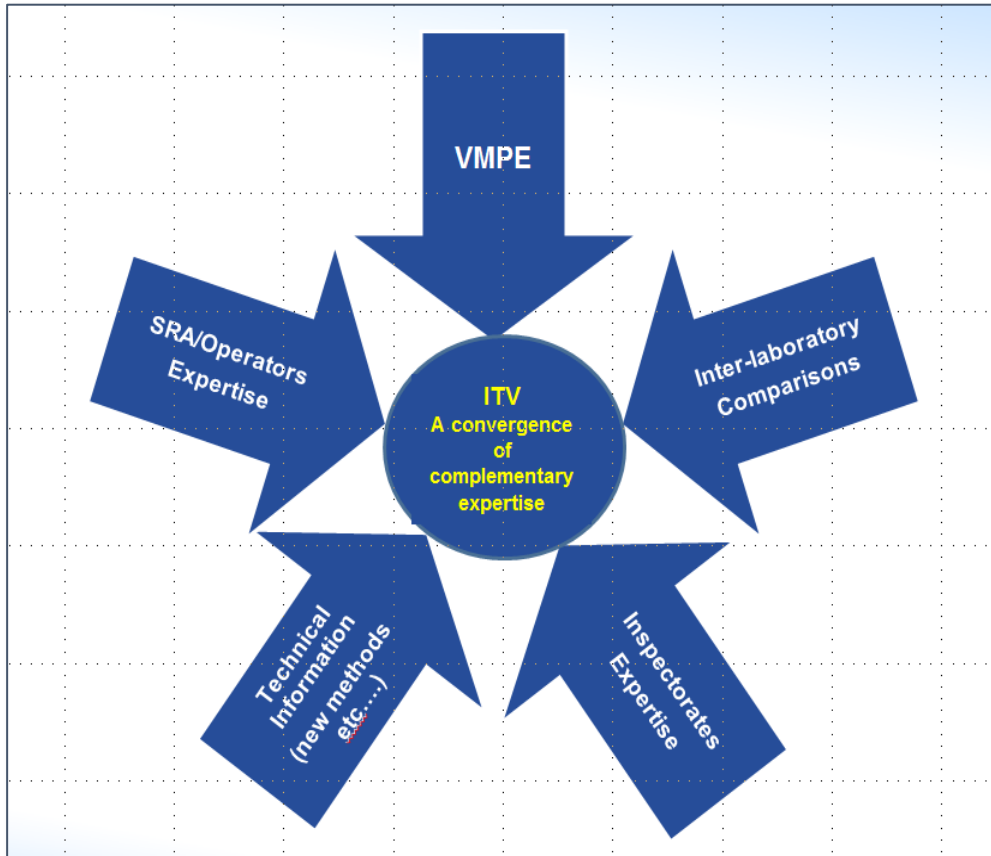


Fig.3: Bringing together different sources of expertise and recognizing their synergy and complementarity is essential to ensure the quality and usability of ITV. VMPE stands for Verification Measurement Performance Evaluation, i.e. the process through which IAEA analysts estimate measurement uncertainties from operator-inspector paired data.

In preparation of the ITV-2020 review, extensive outreach was organized by the IAEA from 2019:

Date	Outreach event
13 May 2019	ESARDA DA/NDA Working Group meeting, 40 th ESARDA Symposium, Stresa, Italy.
19 June 2019	Meeting with NMCC ITV experts in Tokyo, Japan.
17 July 2019	INMM ITV Panel, 60 th INMM Meeting, Palm Desert, CA, USA.
4 February 2020	GT35 (Echantillonnage, mesures, incertitudes) Meeting CETAMA, Paris.
12 July 2020	Joint Meeting of INMM ASC N15 and N15-5.1 (by WebEx).
2-6 November 2020	4 th International Technical Meeting on Statistical Methodologies for Safeguards.
18 November 2020	ESARDA DA/NDA Working Group meeting
16-18 February 2021	International Workshop on Uranium and Plutonium Isotopic Analysis by Non-Destructive Assay Techniques for Nuclear Safeguards.

In order to organize and coordinate the ITV-2020 project, several groups were created corresponding to different Areas of Expertise (AoE), each coordinated by a member of the IAEA ITV team:

Area of expertise (AoE)	IAEA Coordinator
ITV initial values production and review	K. Krzysztozek
Destructive Analysis (DA)	D. Amaraggi
Non-Destructive Assays (NDA)	A. Berlizov and L. Bourva
NDA spent fuel	M. Mayorov
Bulk - mass	K. Zhao and C. Portaix
Bulk - volumes	S. Richet, R. Binner and A. Berlizov
COMPUCEA	E. Bonner
Cristallini	S. Michalak & E. Bonner
Project management and STR narratives	C. Norman (and measurement method SME)

The IAEA coordinators were in charge of:

- Organizing outreach in their area of expertise and liaising with international experts;
- In collaboration with the ITV Scientific Secretary, K. Krzysztozek:
 - organizing the review of the ITV-2010 tables with the SME to review their structure, identifying obsolete entries and identifying the need for new entries and/or tables;
 - Organizing the review of the initial values established by SGIM-IFC² based on historical data and the addition of prospective values based on technical expertise;
 - Collecting and reviewing components of the ITV-2020 report narratives in their area of expertise (description of measurement methods);
- Preparing the 2021 Consultant Group Meeting (CGM).

Sub-groups were created within the different AoE, each with a sub-group leader appointed from the SME:

NDA and Bulk Measurements Sub-	Subject Matter Experts Leader
NDA-1 COMPUCEA	Magdalena Toma (JRC Karlsruhe, EC)
NDA-2 U/Th Content	Nedereh St Amant (CNSC, Canada)
NDA-3 U-235 Enrichment	Markku Koskelo (Aquila Group, USA)
NDA-4 Pu Isotopics	Duc Vo (LANL, USA)
NDA-5 U/Pu Mass by Gamma Techniques	Ram Venkataraman (ORNL, USA)
NDA-6 U/Pu Mass by Neutron Techniques	Martyn Swinhoe (LANL, USA)
NDA-7 Weighing	Ke Zhao and Chris Portaix (IAEA)
NDA-8 Volume and Density	Sebastien Richet (IAEA)
DA Sub-Group	Subject Matter Experts Leader
DA-I Titration & Mass Spectrometry	Evelyn Zuleger (JRC Karlsruhe, EC)
DA-II Spectro(photo) metry, XRF, K-edge	Mike Brisson (SRNL, USA)
DA-III Cristallini & sampling	Olivio Pereira (CTMSP, Brazil)
DA-IV Eastern Time Zone	Georges Duhamel (TRO, IAEA)

² The Nuclear Fuel Cycle Information Analysis Section of the Department of Safeguards Division of Information Management

Kick-off meetings with guidance information were organized by the IAEA:

- on December 16th 2020 for the NDA and bulk measurement AoE, in preparation of the International Working Group on Non-destructive Assay Techniques (IWG-NDA) which took place during the week of 15-19 February 2021;
- on March 22nd 2021 for the DA AoE.

After a short reminder about the definition of ITVs and the way their initial values are estimated by the IAEA, the role and objective of sub-groups were outlined in terms of two interactive tasks: the review of the tables structure, possibly including the addition of new tables, and the review of the IAEA initial estimates based on historical paired data. In case historical data is insufficient or not available, this review also considers the inclusion of technically based uncertainties provided by SME, that are incorporated in the table as prospective ITV, until there is enough paired data acquired to establish ITVs based on implementation data. Target attainment levels were set as:

- **Minimum goal (must have):** update of the ITV-2010 methods/applications and initial ITVs for currently existing methods/application for which enough data is available;
- **Higher goal:** minimum + improved resolution with respect to methods (e.g. software) and application (e.g. concentration) related factors; include ITV *prospective methods/applications* based on technical assessment, e.g. based on bottom-up uncertainty quantification (UQ);
- **Long-term goal:** fill in the “high resolution bottom-up” table when enough observed data is obtained, through a continuous *ITV review in collaboration with SME* (proposed evolution of the ITV process).

Sub-group leaders were in charge of coordinating the work, defining the workflow and agenda in cooperation with the IAEA, scheduling meetings, leading technical discussions, producing meeting minutes and taking the lead in the preparation of progress and final reports to be presented at the CGM.

The final report from sub-groups included:

- The proposed ITV values given as random, systematic and total error standard deviations, organized in structured tables;
- The technical basis as well as the basis for the proposed ITV, for instance experimental data evaluation (data sources to specify), uncertainty quantification for a technique/method, or expert judgement;
- Comments on the main assumptions and factors influencing the proposed ITV values.
- Narratives describing the methods listed in the ITV tables as well as the factors influencing the associated uncertainties.
- Publications and presentations used to support the sub-group work.

The sub-group reports were presented and discussed at the CGM which was held as a virtual meeting from 31 May to 17 June 2021.

A virtual ITV Expert Network platform (Fig. 4) was deployed to facilitate communication between SME and serve as a portal to access information and resources such as discussion boards, action lists, a library of common resources (reference documents, practical guides) as well as the CGM schedules and WebEx access links and participant contact information. This platform is being further developed to provide access to the ITV tables and open resources to a more general audience of safeguards practitioners, e.g. NFC operators, safeguards inspectors, safeguards laboratory staff, SRA. The SME will continue to access a restricted domain to collaborate on a continuous review of the ITV which was proposed during the CGM closing session to replace the 10-year review cycle.

Fig.4: The ITV-2020 Consultant Group Meeting virtual platform

6. ITV Methodology: The IAEA Statistical error model and the Guide to the Expression of Uncertainty in Measurements (GUM) – a reconciliation.

During the ITV-2010 CGM, a passionate discussion took place between the participants about the consistency between ITVs and the recommendations of the *Guide to the Expression of Uncertainty in Measurement* (GUM) re-issued in 2008 as JCGM 100:2008 [18]. In summary, the objective of the GUM is to provide measurement laboratories with a standardized, methodical approach to determining a quantitative measurement uncertainty associated with a measurement result. Its application also ensures that laboratory results are transparent and traceable and can be compared. The GUM uses a modelling approach based on establishing a functional relationship between the measurand and a set of input quantities and on determining the combined uncertainty for a measurement result using error propagation algorithms. One of the main advantages of this procedure is to ensure that laboratories identify all significant contributors to their uncertainties, i.e. establish a reliable *uncertainty budget*. In 2010 the GUM had been widely adopted by safeguards laboratories. Since the GUM is essentially concerned with associating a single measurement result with a reliable uncertainty (expressed as a total standard deviation multiplied by a coverage factor), some CGM members questioned the fact that ITVs are expressed in terms of two components: the random error standard deviation and the systematic error standard deviation. Safeguards evaluators explained that this distinction is essential when propagating uncertainties to the sums of measurement results that form the components of material balances and that the methodologies used for MBE rest on a statistical model of measurement error which is fit for purpose and is not in contradiction with the GUM. A consensus was reached that an additional column labelled *ITV* would give the total uncertainty, combined from its random and systematic components. However, this discussion revealed the need for a constructive methodological and terminological communication between different safeguards communities involved with measurements and measurement uncertainties and drew the participants' attention on the common benefits of opening communication channels and increasing exchanges between these communities before the next ITV issue (ITV-2020).

Numerous interactive meetings and seminars took place on a yearly basis since the ITV-2010 debate, *inter alia* in the framework of ESARDA working groups and the biennial IAEA Technical Meeting on Statistical Methodologies for Safeguards created in 2013, to review and compare uncertainty quantification concepts and methodologies as well as the related terminology and the way uncertainties are expressed according to their field of application. The initial objectives of this liaison effort were to identify commonalities and differences to promote a mutual understanding and distinguish between terminological differences and conceptual or methodological divergences. In order to provide a dynamic context and maintain the momentum towards these goals, it was agreed to describe the result of the exchanges and the methodological reconciliation efforts in a common paper. The different stakeholder communities contributed different sections of this paper which is now referred to as the *Reconciliation Paper* [19].

The conclusion of the paper is an agreement by all parties that the GUM and the statistical model used by safeguards data evaluators are not contradictory but complementary and fit for different purposes. Importantly, their mathematical equivalence was demonstrated and a glossary of terms used in the field of measurement uncertainties was established with a view to clearly distinguishing terminological and conceptual differences. Moreover, opportunities for mutual learning and methodological synergies were

identified and the IAEA is now exploring hybrid uncertainty quantification methods that combine elements from complementary approaches. It was further recognized that a comparison between GUM-based uncertainties and the top-down estimates obtained through the IAEA VMPE process, which supports the estimation of initial ITV values from historical data, can help to identify unaccounted sources of measurement errors and to build more complete measurement models, especially in the field of NDA. This approach has been employed in the ITV-2020 context by leveraging the experience of SME to document prospective uncertainty values from technical expertise or experimental data analysis when not enough historical data were available. The comparison between these prospective values and future historically based data may provide useful information both on the effect of the implementation procedures and conditions of a measurement method and on the completeness of the associated measurement model. Last but not least, closing the communication gap between ITV stakeholders and fostering mutual understanding facilitated collaboration during the preparation of the ITV-2020 review and is expected to promote partnership and participation of all stakeholders in the prospective continuous ITV review process.

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State	ITV Point of Contact
Argentina	Leonardo Pardo Eduardo Gautier
Brazil	Marcos Grund Marco Dutra
Canada	Chris Cochrane
China	Lixia He
EC	Zdenka Palajova Evelyn Zuleger
France	Marielle Crozet Danielle Roudil
Germany	Irmgard Niemeyer
Japan	Shinji Hara
The Netherlands	Frodo Klaassen
Republic of Korea	Haneol Lee Yun Hwang
Russian Federation	Ivan I. Davidov
USA	Joseph Carbonaro

- the IAEA team of evaluators, AoE coordinators, CGM organisers, CGM platform designers and IT support staff;
- Rafael Grossi, Director General of the IAEA, Massimo Aparo Deputy Director General of the IAEA Department of Safeguards, Jacques Baute, Director of the IAEA Department of Safeguards' Division of Information Management for their essential support to the ITV-2020 project; and
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Appendix I: ITV tables

MEASUREMENT METHOD/INSTRUMENT CODES

Method/Instrument Code	Technique
ALPH	Alpha Spectrometry
ANCC	Active Neutron Coincidence Counter
AWCC	Active Well Coincidence Counter
CALO	Calorimetry
COMPUCEA	Combined Procedure for Uranium Concentration and Enrichment Assay
COUL	Coulometry
EBAL	Electronic Balance
ECGS	Electrically Cooled Germanium System
EFCB	Euratom Fast Collar for BWR
EFCP	Euratom Fast Collar for PWR
ELTM	Electromanometer
ENMC	Epithermal Neutron Multiplicity Counter
FNCC	Fast-Neutron Coincidence Counter
FNCL	Fast-Neutron Coincidence Collar
FRSC	Fuel Rod Scanner
GBAS	Glove Box Assay System
GRAV	Gravimetry
GSMS	Gas Source Mass Spectrometry
GYRO	Gyroscopic Scale
HBAS	Hopper Glove Box Assay System
HEPC	High Efficiency Passive Counter
HKED	Hybrid K-Edge/K-XRF Densitometer
HLNC	High Level Neutron Coincidence Counter
HM-5	Spectrometric Gamma Hand-Held Monitor
HRGS	High Resolution Gamma Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
IMCC	Inspector Multichannel Analyzer with CZT detector
IMCG	Inspector Multichannel Analyzer with Ge detector
IMCL	Inspector Multichannel Analyzer with LaBr detector
IMCN	Inspector Multichannel Analyzer with NaI detector
INVS	Inventory Sample Verification Counter
ISOCS	In Situ Object Counting Software
KEDG	K-Edge Densitometer
LCBS	Load-cell Based Weighing System
LRGS	Low Resolution Gamma Spectrometry
LMCA	Laboratory Multichannel Analyzer (HRGS)
LMCN	Laboratory Multichannel Analyzer (NaI detector)
MC-ICPMS	Multi-Collector Inductively Coupled Plasma Mass Spectrometry
MMCC	Mini-Multichannel Analyzer with CZT Detector
MMCG	Mini-Multichannel Analyzer with Ge Detector
MMCL	Mini-Multichannel Analyzer with LaBr Detector
MMCN	Mini-Multichannel Analyzer with NaI Detector
MRGS	Medium Resolution Gamma Spectrometry
PCAS	Plutonium Canister Assay System
PNCC	Passive Neutron Coincidence Counter

PNCL	Plutonium Neutron Coincidence Collar
PNMC	Plutonium Neutron Multiplicity Counter
POLA	Polarography
PSMC	Plutonium Scrap Multiplicity Counter
PUSP	Pu-VI Spectrophotometry
SBAS	Super Glove Box Assay System
TIMS	Thermal Ionization Mass Spectrometry
TITR	Titration
TNCC	Thermal Neutron Coincidence Counter
UNCL	Uranium Neutron Coincidence Collar
USP	Uranium Spectrophotometry
VTDM	Vibrating Tube Density Meter
WDAS	Waste Drum Assay System
XRF	X-ray Fluorescence

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(1) Bulk

TABLE 1 - Mass

Instrument Code	Instrument Type	Mass Range kg ^{1/}	Uncertainty Component % rel., k=1		ITV (% rel., k=1)
			u(r)	u(s)	
EBAL	Electronic balance	0 to 20	0.02	0.02	0.03
LCBS	Platform load cell ^{2/}	0 to 15000	0.05	0.05	0.07
	Crane-mounted load cell ^{3/}	1000 to 15000	0.05	0.05	0.07
GYRO	Gyroscopic scale	1000 to 5000	0.02	0.02	0.03
		6000 to 15000	0.01	0.01	0.01

^{1/} The assumption is that the range of the scale is chosen based on the approximate mass of the item being measured such that the gross mass is within 50 % to 100 % of the full range of the scale.

^{2/} The platform load cell performance can vary with the number of load cells contained in the weighing equipment.

^{3/} The crane mounted load cell uncertainties can be increased by a factor of two if being used outdoors.

TABLE 2 – Volume and Density

Measurement	Instrument	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
		u(r)	u(s)		
Volume ^{1/}	ELTM (Accountability tanks)	0.05	0.1	0.12	<u>2/</u>
	ELTM (Process tanks; high concentration)	0.2	0.2	0.28	<u>3/</u>
	ELTM (Process tanks; low concentration)	1	1	1.4	<u>4/</u>
	ELTM (Accountability tanks)	0.3	0.2	0.36	<u>5/</u>
	ELTM for calibration conditions (Accountability tanks)				<u>7/</u>
Density	ELTM (Accountability tanks)	0.05	0.05	0.07	<u>6/</u>
	ELTM (Process tanks; high concentration)	0.1	0.1	0.14	
	ELTM (Process tanks; low concentration)	0.7	0.7	1	
	VTDM	0.05	0.05	0.07	
	Density for calibration conditions				<u>7/</u>

1/ Volume determinations are made on the basis of level pressure, density and temperature measurements. The volume measurement uncertainties are highly dependent on the homogeneity of the liquid, the quality of the density measurements and of the calibration equation determined in the calibration process. The volume measurements may also involve an absolute error component which has to be taken into consideration when determining the overall uncertainty of volume measurements.

2/ For accountability tanks in newly built large-throughput facilities, uncertainties of 0.05% for u(r) and 0.1% for u(s) at full volume are achievable if: i.) A carefully designed calibration procedure has been implemented under well-controlled environmental and stable temperature conditions; and ii.) Measurements, using high precision electro-manometers, are performed on a well-characterized and homogenized liquid.

3/ Process tanks for high Pu concentration solutions are generally also equipped with high precision electro-manometers, however, the calibration effort and tank design specifications may be lower.

4/ Equipped with standard electro-manometers, lower calibration effort.

5/ The values apply to older facilities where the tank design was not driven by optimized ELTM volume measurement capabilities.

6/ The same comments as given for the volume measurements apply; one additional important calibration parameter is the determination of the probe (dip tube) separation.

7/ Placeholder for ITV still under review.

(2) Destructive Analysis

TABLE 3 – Sampling Uncertainties** for U and Pu Element Mass Fraction and U and Pu Isotope Ratio Measurements

Material	Uncertainty Component (% rel., k=1)				Recommended Sample Size ^{1/}
	U and Pu element mass fraction measurements		U and Pu isotope ratio measurements		
	u(r)	u(s)	u(r)	u(s)	
DUF ₆	0.1	*	1 ^{2/}	1 ^{2/}	5 to 10 g
NUF ₆ , LEUF ₆ ^{6/}	0.05	*	0.1 ^{6/}	*	5 to 10 g
U-oxide Powder	0.2	*	0.05	*	10 to 20 g
U-oxide Pellets	0.05	0.05	0.05	0.05	1 pellet
U-oxides (with Gd)	0.2	*	0.05	*	1 pellet
U Scrap (clean) ^{3/}	1	*	1	*	30 g
U Scrap (dirty) ^{4/}	10	*	10	*	2 x 30 g
Reprocessing Input Solution	0.3	0.2 ^{5/}	0.05	*	2 x 1 mL
High Active Liquid Waste	5	5	*	*	2 x 1 mL
U Nitrate Solution	0.1	*	0.05	*	10 mL
Pu, U and Pu Nitrate Solution	0.2	*	0.05	*	10 mL
Pu-oxide	0.1	*			2 x 1 g
FBR MOX	0.2 (Pu)	*	0.1	*	2 x 1 pellet, or 2 x 2 pellet
	0.1 (U)	*			
LWR MOX	0.7 (Pu)	*	0.1	*	2 x 1 pellet, or 2 x 5 g
	0.1 (U)	*			
MOX Scrap (clean)	1	*	1	*	2 x 5 g
MOX Scrap (dirty)	10	*	10	*	2 x 10 g
HEU Metal and Alloys	0.2	*	0.05	*	1 - 5 g

* Values have not yet been defined.

**Values listed in this table are indicative of the sampling uncertainties that are dependent on the homogeneity of samples.

^{1/} According to STR-69 (Destructive Analysis and Evaluation Services for Nuclear Material Accountancy Verification, STR-69, Rev. 5, IAEA, Vienna, 2004).

^{2/} Additional sampling uncertainties are expected for DUF₆ when samples are taken from the gas phase of a non-homogenized cylinder.

^{3/} Scrap with low impurity content and suitable for recycling.

^{4/} Sampling uncertainties can vary widely depending on material heterogeneity and sample size.

^{5/} Sampling uncertainties of systematic nature, resulting from airlift effects (evaporation).

^{6/} Proposed Prospective Crystalline ITV: Root mean square error = $[u(r)^2 + u(s)^2]^{1/2} = 0.038\%$ (based on limited data i.e. 16 pairs LEU samples, and subject to change as more samples are collected and analysed).

TABLE 4a – Uranium Element Mass Fraction Measurements (DA)

Method	Material	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
		u(r)	u(s)		
GRAV	U (pure compounds)	0.05	0.05	0.07	<u>1/</u>
	U (with Gd)	0.1	0.1	0.14	<u>10/</u>
TITR ^{2/}	U (pure compounds)	0.1	0.1	0.14	<u>11/</u>
	U Alloys	0.2	0.2	0.28	
	Mixed U/Pu	0.1	0.1	0.14	
POLA	U (with Gd)	0.1	0.1	0.14	
IDMS ^{8/}	Hot Cell Conditions	0.2	0.2	0.28	<u>3/ 4/</u>
		0.3	0.3	0.42	<u>3/ 5/</u>
	Glove Box Conditions	0.15	0.1	0.18	<u>3/ 4/</u>
		0.2	0.2	0.28	<u>3/ 5/</u>
KEDG	U and U/Pu Solution	0.2	0.2	0.28	<u>6/</u>
HKED	Spent Fuel Solution	0.2	0.2	0.28	<u>6/</u>
XRF	U Solution (low conc.)	2	2	2.8	<u>7/</u>
USP	U Solution (low conc.)	2	2	2.8	<u>7/ 9/</u>

1/ Materials containing non-volatile impurities at less than 1000 µg/g; ITVs could be higher for powder samples depending on additional sources of uncertainties (oxidation during storage, balance resolution).

2/ Davies & Gray Method.

3/ For all materials typically encountered in the nuclear fuel cycle.

4/ Using gravimetrically prepared and/or using commercially available non-diluted spikes (such as Large Size Dried Spikes), spiking non-diluted samples.

5/ Using samples and/or spikes after dilution, using artificial spikes or high abundant spikes with variable spike to sample amount ratios.

6/ U concentration above 150 g/L with a U/Pu mass ratio above 80 and measurement times in the range of 3 x (1000-3000) s. The uncertainty for dissolved mixed-oxide (MOX) fuel pellets and powder is higher than these ITVs, because of lower U concentration and lower U/Pu ratio.

7/ Uncertainties related to XRF performance and for samples in solution in the range of U concentrations from 1 g to 50 g/L (HKED system).

8/ Refers to ID-TIMS and ID-MC ICPMS.

9/ For process analysis under hot cell conditions.

10/ The range of Gd content is less than 10 %.

11/ Including U with Gd.

TABLE 4b – Plutonium Mass Fraction Measurements (DA)

Method	Material	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
		u(r)	u(s)		
GRAV	Pu Oxide	0.05	0.05	0.07	<u>1/</u>
TITR	Pu Oxide and Nitrate	0.15	0.15	0.21	<u>2/</u>
	U/Pu Oxide and Nitrate	0.2	0.2	0.28	
COUL	Pu (pure compounds)	0.1	0.1	0.14	<u>3/</u>
IDMS ^{11/}	Hot Cell Conditions	0.2	0.2	0.28	<u>4/ 5/</u>
		0.3	0.3	0.42	<u>4/ 6/</u>
	Glove Box Conditions	0.15	0.1	0.18	<u>4/ 5/</u>
		0.2	0.2	0.28	<u>4/ 6/</u>
KEDG	Pu and U/Pu Solution	0.3	0.3	0.42	<u>7/</u>
HKED	Spent Fuel Solution	0.8	0.5	0.94	<u>8/</u>
XRF	Pu Solution (low conc.)	2	2	2.8	<u>9/</u>
PUSP	Process Solutions	2	2	2.8	<u>10/</u>
ALPH	Waste Solution (low conc.)	7	7	10	

1/ Materials containing non-volatile impurities at less than 1000 µg/g.

2/ Techniques such as the MacDonald and Savage and the AgO titration methods.

3/ Recommended Pu mass range for aliquots is 4 mg to 15 mg; for samples containing Fe/Pu mass fraction greater than 25 µg/g, an iron correction or a chemical separation is required to minimize uncertainty; uranium is not an interference when the U/Pu mass fraction is 100 or less.

4/ For all materials typically encountered in the nuclear fuel cycle.

5/ Using gravimetrically prepared and/or using commercially available non-diluted spikes (such as Large Size Dried Spikes), spiking non-diluted samples.

6/ Using samples and/or spikes after dilution, using artificial spikes or high abundant spikes with variable spike to sample amount ratios.

7/ Pu concentration above 40 g/L with a U/Pu mass ratio below 0.1 and measurement times in the range of 3 x (1000-3000) s.

8/ U concentration of 150 g/L to 250 g/L with a U/Pu mass ratio of 80 to 150 and measurement times in the range of 3 x (1000-3000) s.

9/ Uncertainties related to XRF performance and for samples in solution in the range of Pu concentrations from 1 g/L to 50 g/L; counting precision greater than > 1 % if Pu concentration less than 1 g/L (HKED system)

10/ Pu concentration of greater than 0.1 g/L.

11/ Refers to ID-TIMS and ID-MC ICPMS.

TABLE 5a - $^{235}\text{U}/^{238}\text{U}$ Isotope Ratio Measurements (DA)

Method	Material	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
		u(r)	u(s)		
GSMS	DUF ₆ & NUF ₆	0.1	0.1	0.14	<u>1/</u>
	LEUF ₆	0.05	0.05	0.07	
TIMS, MC-ICPMS ^{2/}	DU (0.3 % ^{235}U)	0.5	0.5	0.7	
	U (0.3 % < ^{235}U < 1 %)	0.2	0.2	0.28	
	LEU (1 % < ^{235}U < 20 %) ^{3/}	0.1	0.1	0.14	
	HEU (> 20 % ^{235}U)	0.05	0.05	0.07	

1/ Sampling uncertainty may be dominating if cylinder contents are not homogenized.

2/ For single collector instruments (SF ICPMS and Q ICPMS), recent publication on uncertainties obtained using CRM indicates that corresponding ITV's should be higher than ITVs for TIMS/MC-ICPMS.

3/ Recent studies of measurement data on pellets (LEU) indicate that uncertainty changes with enrichment. A follow-up study to establish enrichment ranges and associated measurement uncertainties is on-going.

TABLE 6a - Plutonium Isotope Ratio Measurements in Pu and U/Pu Materials (DA)

Material	Isotope Amount Ratios	Typical Value for Ratio (*100)	Method		
			TIMS, MC-ICPMS ^{1/}		ITV
			(% rel., k=1)		(% rel., k=1)
			u(r)	u(s)	u(c)
High-Burnup Pu	$^{238}\text{Pu}/^{239}\text{Pu}$	1.7	1.5	1	1.8
	$^{240}\text{Pu}/^{239}\text{Pu}$	43	0.1	0.05	0.11
	$^{241}\text{Pu}/^{239}\text{Pu}$	13	0.2	0.2	0.28
	$^{242}\text{Pu}/^{239}\text{Pu}$	8	0.2	0.3	0.36
Low-Burnup Pu	$^{238}\text{Pu}/^{239}\text{Pu}$	0.02	10	10	14.1
	$^{240}\text{Pu}/^{239}\text{Pu}$	6	0.15	0.1	0.18
	$^{241}\text{Pu}/^{239}\text{Pu}$	0.2	1	1	1.4
	$^{242}\text{Pu}/^{239}\text{Pu}$	0.05	2	2	2.8

1/ $^{238}\text{Pu}/^{239}\text{Pu}$ by alpha spectrometry/mass spectrometry combination and chemical separation before mass spectrometric measurements.

(3) COMPUCEA

TABLE 8a – Uranium Element Mass Fraction (COMPUCEA)

Physical Form	Chemical Form	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
		u(r)	u(s)		
Powder	UO ₂ , U ₃ O ₈	0.2	0.2	0.28	<u>1/</u>
Pellet	UO ₂	0.2	0.1	0.22	
Solution	UNH	0.15	0.15	0.21	<u>2/</u>
Other Solid	UF ₆	0.1	0.1	0.14	<u>3/</u>

Uncertainty categories:

ITV: are presented in black font

Prospective Values: are presented in blue font

 1/ Including pure ADU, AUC, UO₃ powders

 2/ Concentration from 150 gU/L to 400 gU/L. The ITVs are also applicable to UO₂F₂ solutions in the same concentration range

3/ Assuming no material degradation (e.g. due to moisture intake) during or after sample taking

TABLE 8b – ²³⁵U Enrichment (COMPUCEA)

Physical Form	Chemical Form	²³⁵ U Enrichment	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)	Notes
			u(r)	u(s)		
Powder	UO ₂ , U ₃ O ₈	DU (²³⁵ U < 0.69 %)	0.6	0.4	0.72	<u>1/</u>
		NU (0.69 % < ²³⁵ U < 0.73 %)	0.4	0.3	0.5	<u>1/</u>
		LEU (0.73 % < ²³⁵ U < 20 %)	0.25	0.25	0.35	<u>1/</u>
		HEU (²³⁵ U > 20%)				<u>3/</u>
Pellet	UO ₂	NU (0.69 % < ²³⁵ U < 0.73 %)	0.35	0.25	0.43	
		LEU (0.73 % < ²³⁵ U < 20 %)	0.2	0.2	0.28	
Solution	UNH	DU (²³⁵ U < 0.69 %)	0.6	0.5	0.78	<u>2/</u>
		NU (0.69 % < ²³⁵ U < 0.73 %)	0.4	0.45	0.6	<u>2/</u>
		LEU (0.73 % < ²³⁵ U < 20 %)	0.28	0.35	0.45	<u>2/</u>
Other Solid	UF ₆	DU (²³⁵ U < 0.69%)	0.4	0.2	0.45	
		NU (0.69 % < ²³⁵ U < 0.73 %)	0.25	0.17	0.3	
		LEU (0.73 % < ²³⁵ U < 20 %)	0.2	0.15	0.25	
		HEU (²³⁵ U > 20 %)				<u>3/</u>

Uncertainty categories:

ITV: are presented in black font

Prospective Values: are presented in blue font

 1/ Including pure ADU, AUC, UO₃ powders

2/ Concentration 150 gU/L to 400 gU/L

3/ Placeholder for future ITV

(4) Non-Destructive Assay

TABLE 4c – Uranium and Thorium Mass Fraction (NDA)

Method	Technique	Instrument	Material (homogeneous)	Element	Mass fraction range	Uncertainty (% rel., k=1)			Notes
						u(r)	u(s)	u(c)	
HRGS	T1	XMCG	Powder, solution, slurry	NU	1 % to 25 %	2.0	3.0	3.6	DX, IT, KE, LZ, EG=185.7, WT=5, LT=1000, FLD, HY
MRGS	T1	XMCL	Powder, solution, slurry	NU	1 % to 25 %	2.0	3.5	4.0	IT, KE, LZ, EG=185.7, WT=5, LT=1000, FLD, HY
		XMCC	Powder, solution, slurry	NU	1 % to 25 %	5.0	4.0	6.4	IT, KE, LZ, EG=185.7, WT=5, LT=1000, FLD, HY
HRGS	T1	XMCG	Powder, solution, slurry	DU	1 % to 25 %	14	3.5	14	D1, D2, IT, RE, LZ, EG=185.7, ISO-LO, WT=1, LT=1000, FLD, HY
		XMCG	Powder, solution, slurry	NU	1 % to 25 %	7.4	3.4	8.2	D1, D2, IT, RE, LZ, EG=185.7, ISO-LO, WT=1, LT=1000, FLD, HY
		XMCG	Powder, solution, slurry	LEU	1 % to 25 %	5.7	3.7	6.8	D1, D2, IT, RE, LZ, EG=185.7, ISO-LO, WT=1, LT=1000, FLD, HY
		XMCG	Powder, solution, slurry	DU	1 % to 25 %	18	3.2	18	D2, D3, IT, RE, LZ, EG=185.7, ISO-HI, WT=1, LT=1000, FLD, HY
		XMCG	Powder, solution, slurry	NU	1 % to 25 %	17	3.0	17	D2, D3, IT, RE, LZ, EG=185.7, ISO-HI, WT=1, LT=1000, FLD, HY
		XMCG	Powder, solution, slurry	LEU	1 % to 25 %	14	3.0	14	D2, D3, IT, RE, LZ, EG=185.7, ISO-HI, WT=1, LT=1000, FLD, HY
	T2	XMCG	Powder, slurry, sludge	NU	> 25 %	2.0	0.7	2.1	DX, TS, KE, SM=2, EG=185., LT=1000, TD
		XMCG	Solution (organic and aqueous)	NU	1 % to 25 %	1.8	0.8	2.0	DX, TS, KE, SM=10 mL, EG=185.7, LT=1000, TD
		XMCG	Powder	LEU	> 25 %	1.4	0.4	1.5	D2, TS, RE, SM=50 to 100, EG=1001, ISO-LO, LT=2000, TD
		XMCG	Powder, ore	Th	0.1 % - 1 %	5.0	4.0	6.4	D2, D3, TS, SM=10, EG=583/911, LT=3000, FLD, TD
		XMCG	Powder, ore	Th	> 1 wt%	1.5	1.0	1.8	D2, D3, TS, SM=10, EG=583/911, LT=1000, FLD, TD
	T3	XMCG	Powder, ore	Th	0.1 % - 1 %	0.8	4.4	4.5	D2, D3, TS, RE, EG=583/911/2614, PA-911=30,000, LAB, TD
		XMCG	Powder, ore	Th	> 1 %	0.6	1.5	1.6	D2, D3, TS, RE, EG=583/911/2614, PA-911=30,000, LAB, TD
		XMCG	Powder, solution	LEU	> 25 %	13.0	3.0	13.3	D2, D3, TS, RE, SM>=150, EG=185.7/1001, LT=1000, FLD, TD
	XRF	T4	ED-XRF	Powder, solution	U/Th	0.1 % - 25 %	3.0	3.0	4.2
WD-XRF			Solution (organic and aqueous)	U	1 % - 25 %	3.0	3.0	4.2	Refinery Plant Report
T5		WD-XRF	Powder, ore	Th	0.1 % - 10 %	3.3	6.0	6.8	SGAS-NML Measurements

Uncertainty categories:

Prospective Values are presented in **blue font**

Assumptions:

- 1 Only homogeneous material (no sampling error included)
- 2 For method T1, systematic uncertainty includes sample matrix, calibration, wall thickness determination and detector specific spectrum fitting components

Method categories:

- HRGS High Resolution Gamma Spectrometry
- MRGS Medium Resolution Gamma Spectrometry
- XRF X-Ray Fluorescent Analysis

Technique categories:

- T1 Concentration meter principle (in-situ measurements)
- T2 Absolute gamma-spectrometry calibrated against reference materials (on-site laboratory setup)
- T3 In-situ absolute gamma-spectrometry calibrated mathematically (e.g. by Monte Carlo or ISOCS) in situations when no appropriate calibration standard is available
- T4 XRF using a calibration curve
- T5 XRF using fundamental parameters

Notes:

- EG Assay gamma ray energy (keV)
- IT Infinitely thick samples (≥ 5 mfp at the assay gamma ray energy)
- TS Thin samples ($\ll 1$ mfp at the assay gamma ray energy)
- KE Material with known enrichment (e.g., NU at concentration, refinery and conversion plants)
- LZ Low-Z matrix, known contents of high-Z additives
- RE Radioactive equilibrium. In the case of uranium, enrichment can be measured using a complementary peak ratio method (MGAU, FRAM or similar)
- SM Sample mass (g)
- WT Container wall thickness (mm)
- LT Measurement live time (s)
- ISO-LO Low-energy isotopic analysis (60 keV to 250 keV)
- ISO-HI High-energy isotopic analysis (120 keV to 1010 keV)
- PA-XXX Number of counts (area) of the peak at energy XXX keV
- LAB Laboratory measurements
- FLD Field measurements

Instrument categories:

- XMCG HPGe detectors, including both electrically or LN2 cooled, coupled to any MCA
- XMCC CZT, active volume ≥ 1500 mm³, collimator: \varnothing 15 x 5 mm, any MCA
- XMCL LABR, crystal \varnothing 50 x 13 mm (or larger), collimator: \varnothing 44 x 20 mm, any MCA

XMCG types:

- D1 Energy resolution < 750 eV @ 122 keV. Active area > 500 mm². Thickness > 10 mm. Typical detectors in XMCG-D1 category: planar and semi-planar detectors (GL1015, BE1015, BE2830, BE3830, BE5030, Profile S/SP, GLP).
- D2 Energy resolution < 1.0 keV @ 122 keV and < 2.0 keV @ 1332 keV. Relative efficiency $> 15\%$. Typical detectors in XMCG-D2 category: semi-planar and small volume coaxial detectors (BE2830, BE3830, BE5030, Profile S/SP/C/M, GMX, Falcon, Aegis).
- D3 Energy resolution < 1.5 keV @ 122 keV and < 2.3 keV @ 1332 keV. Relative efficiency $> 15\%$. Typical detectors in XMCG-D3 category: portable electrically cooled coaxial detectors and large volume coaxial detectors (Micro-Detective (ECGS), Aegis, GC9023, Profile C/M, GMX).

Method used to derive ITVs:

- TD Top-down
- BU Bottom-up
- HY Hybrid

TABLE 5b – ²³⁵U Enrichment (NDA)

Method	Technique	Instrument	Material	Grade	Shielding Material	Shielding Thickness mm	Uncertainty Component		ITV (% rel., k=1)	Notes
							(% rel., k=1)			
							u(r)	u(s)	u(c)	
LRGS	T0	XMCCN	UF ₆	DU	Fe	16.0	20.0	5.0	20.6	
			UF ₆	NU	Fe	16.0	10.0	3.0	10.4	
			UF ₆	LEU	Fe	12.5	5.0	2.0	5.4	
			UOX	NU	Fe	1.5	5.0	3.0	5.8	
			UOX	LEU	Fe	1.5	3.0	2.0	3.6	
	T2	XMCCN	UO ₂	LEU	n/a	n/a	3.0	1.0	3.2	pellets, rods
	T1	HM-5	UF ₆	NU	Fe	16.0	13.0	3.0	13.3	
			UF ₆	LEU	Fe	12.5	6.0	3.0	6.7	
			UOX	NU	Fe	1.5	10.0	4.0	10.8	
MRGS	T1	XMCL	UF ₆	LEU	Fe	12.5	4.0	1.0	4.1	
			UOX	NU	Fe	1.5	4.0	1.0	4.1	
			UOX	LEU	Fe	1.5	3.0	1.0	3.2	
	T2	XMCL	UO ₂	NU	n/a	n/a	3.0	1.0	3.2	pellets, rods
			UO ₂	LEU	n/a	n/a	2.0	1.0	2.2	pellets, rods
	T1	XMCC	UF ₆	NU	Fe	16.0	10.0	2.0	10.2	
			UF ₆	LEU	Fe	12.5	3.0	2.0	3.6	
			UF ₆	HEU	Ni	6.5	1.5	1.5	2.1	
			UOX	DU	Fe	1.5	13.0	3.0	13.3	
			UOX	NU	Fe	1.5	4.0	2.0	4.5	
UOX			LEU	Fe	1.5	2.0	2.0	2.8		
HRGS	T1	XMCCG	UF ₆	DU	Fe	16.0	20.0	5.0	20.6	
			UF ₆	NU	Fe	16.0	10.0	2.0	10.2	
			UF ₆	LEU	Fe	12.5	5.0	2.0	5.4	
			UOX	LEU	Fe	1.5	3.0	2.0	3.6	
	T1	ECGS	UF ₆	VDU	Fe	16.0	20.0	3.0	20.2	
			UF ₆	DU	Fe	16.0	10.0	3.0	10.4	
			UF ₆	NU	Fe	16.0	6.0	1.0	6.1	
			UF ₆	LEU	Fe	12.5	2.5	1.0	2.7	
			UOX	DU	Fe	1.5	4.0	3.0	5.0	
			UOX	NU	Fe	1.5	3.0	1.0	3.2	
			UOX	LEU	Fe	1.5	2.0	1.0	2.2	
	T3-LE	XMCCG	Arbitrary	DU	Fe	1.5	8.2	0.7	8.2	mU ≥ 100 g
			Arbitrary	NU	Fe	1.5	5.1	1.5	5.3	mU ≥ 100 g
			Arbitrary	LEU	Fe	1.5	3.0	1.0	3.2	mU ≥ 100 g
			Arbitrary	HEU	Fe	1.5	2.1	1.9	2.8	mU ≥ 100 g
	T3-HE	XMCCG	Arbitrary	DU	Fe	10.0	6.1	1.1	6.2	mU ≥ 100 g
			Arbitrary	NU	Fe	10.0	5.8	0.5	5.8	mU ≥ 100 g
Arbitrary			LEU	Fe	10.0	4.8	0.4	4.8	mU ≥ 100 g	
Arbitrary			HEU	Fe	10.0	3.1	0.8	3.2	mU ≥ 100 g	

Uncertainty categories:

ITV: are presented in black font

Prospective Values: are presented in blue font

Assumptions:

1 Only homogeneous material (no sampling error included)

Method categories:

LRGS Low Resolution Gamma Spectrometry

MRGS Medium Resolution Gamma Spectrometry

HRGS High Resolution Gamma Spectrometry

Technique categories:

T0 Enrichment meter principle, absolute intensity of the 186 keV gamma-rays, infinite thickness geometry calibrated against reference material, using 2 ROI

T1 Enrichment meter principle, absolute intensity of the 186 keV gamma-rays, infinite thickness geometry calibrated against reference material, using NaI(GEM)/GEM

T2 Fixed geometry measurement, absolute intensity of the 186 keV gamma-rays, calibrated against reference material of similar shape, physical form and chemical composition

T3 Peak ratio technique, intrinsic efficiency calibration, arbitrary sample geometry, radioactive equilibrium

LE Low energy range analysis ($E < 270$ keV)

HE High energy range analysis ($E > 120$ keV)

Instrument categories:

XMCG HPGe detector (BE-1015 / GL1015) coupled to any MCA (InSpector-2000, MCA-527 etc.)

ECGS Electrically cooled HPGe spectrometer (Micro-Detective, 50 mm x 30 mm, coaxial), collimator dia 50 mm

XMCC CZT, ≥ 1500 mm³ (all material grades), ≥ 500 mm³ (HEU), coupled to any MCA, collimator dia 15 mm (CZT/1500) or 10 mm (CZT/500)

XMCL LaBr₃ detector, thickness ≥ 12.7 mm, diameter ≥ 50.8 mm, coupled to any MCA, collimator diameter = 44 mm

Uranium grade categories, ²³⁵U isotope mass fractions:

VDU ²³⁵U range from 0.1 % to 0.2 %

DU ²³⁵U range from 0.2 % to 0.6 %

NU ²³⁵U nominal enrichment of 0.71 %

LEU ²³⁵U range from 2 % to 5 %

HEU ²³⁵U range of > 20 %

TABLE 6b – Pu Isotopic Abundances (NDA)

Method	Technique	Instrument*	Material	Burnup	Shielding	²³⁹ Pu		²⁴⁰ Pu		²⁴⁰ Pueff		Specific power		Notes
						(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	(% rel., k=1)	
						u(r)	u(s)	u(r)	u(s)	u(r)	u(s)	u(r)	u(s)	
HRGS	T1, LE	XMCG-D1	Pu	L	L	0.1	0.1	1.5	0.8	1.5	0.8	1.0	0.2	1
			Pu	H	L	0.6	0.3	1.2	0.4	0.9	0.3	0.6	0.3	1, 3
	T2, LE	XMCG-D1	Pu	H	L	0.7	0.3	1.2	0.6	1.5	0.5	0.6	0.3	1, 3
	T1, LE	XMCG-D1	MOX	H	L	1.0	0.4	2.6	0.8	2.6	0.5	1.0	0.7	6
	T1, HE	XMCG-D2	Pu	L	L/H	0.6	0.2	6.2	1.7	6.2	1.7	1.0	0.4	2
			Pu	H	L/H	2.1	0.6	6.2	1.7	5.6	1.6	1.5	0.9	2
	T1, HE	XMCG-D3	Pu	L	L/H	1.7	0.2	16.1	3.1	15.7	3.0	2.4	0.4	7, 11
			Pu	H	L/H	4.9	4.1	15.2	9.9	11.3	6.7	3.3	0.7	7, 11
		XMCG-D3	MOX	H	L	2.6	1.9	6.7	6.0	3.4	2.7	2.7	3.1	10
MRGS	T3/T4, HE	XMCC	Pu	L	L/H	2.5	1.5	20	15	N/A	N/A	N/A	N/A	4, 8
			Pu	H	L/H	3.5	2.5	15	10	N/A	N/A	N/A	N/A	4, 8
	T3/T4, HE	XMCL	Pu	L	L/H	2.5	2.0	20	15	N/A	N/A	N/A	N/A	5, 9
			Pu	H	L/H	3.5	3.0	15	15	N/A	N/A	N/A	N/A	5, 9

N/A: not applicable

Uncertainty categories:

ITV: are presented in black font

Prospective Values: are presented in blue font

Assumptions / considerations:

- LANL data, FRAM v.6.1 analysis with parameter set GePlnr_Pu_060-230
- LANL data, FRAM v.6.1 analysis with parameter set GeCoax_Pu_180-1010
- Additional source of data from IAEA NML obtained using FRAM v.5.2 / MGA (10 analyses) 129 keV = 50000 cnts, OCR=22 kcps, LT=600 s. The prospective values were established based on both, LANL and IAEA dataset.
- PAT analysis, MRGS Intercomparison exercise - XMCC: 413 keV = 2000 cnts (HBPU) and 20000 cnts (LBPu), OCR=10 kcps, LT=600 s.
- PAT analysis, MRGS Intercomparison exercise - XMCL: 413 keV = 10000 cnts (HBPU) and 50000 cnts (LBPu), OCR=10 kcps, LT=600 s.
- IAEA field data, FRAM v.6.1 analysis with parameter set GePlnr_Pu_060-230, 129 keV = 30000 cnts, achievable with standard IAEA XMCG-D1 setup at OCR=10 kcps for LT=600 s
- LANL data obtained using two 1st generation Ortec Detective instruments. Data measured in 2005 and 2016. 28 data sets of 14 sources. 539 spectra. FRAM analysis with parameter set detective_pu_120-460_220210. Normalized to 600 s LT.
- LANL data, FRAM v.6.1 analysis with parameter set CZT500_Pu_120-500 with correlated ²⁴⁰Pu option turned on in addition to correlated ²³⁸Pu and correlated ²⁴²Pu. 9 data sets, 299 spectra. Normalized to 600 s LT.
- LANL datam FRAM v.6.1 analysis with parameter set LaBr_Pu_200-750 with correlated ²⁴⁰Pu option turned on in addition to correlated ²³⁸Pu and correlated ²⁴²Pu. 21 data sets, 530 spectra. Normalized to 600 s LT.

- 10 IAEA field data, FRAM v.6.1 analysis with a modified parameter set "det_coax_120_800_2_ecgs" (interferences with Cs-137 and Ag-110 m), very high BUPu (^{239}Pu isotope mass fraction < 55 %, 2-3 y after chemical purification, DU/Pu = 10), 129 keV = 5000 cnts, 208 keV = 700000 cnts, achievable with standard IAEA XMCG-D3 setup at OCR=5 kcps for LT=1000 s (DT < 20%)
- 11 EC-JRC data, steel filter up to 16 mm, lead filter up to 4 mm, 1400 spectra in 40 sets, analyzed with FRAM v.5.2 and parameter set Pu_Cx_180-1010.

Method categories:

- HRGS High Resolution Gamma Spectrometry
MRGS Medium Resolution Gamma Spectrometry

Technique categories:

- T1 Peak ratio, intrinsic efficiency calibration, "known" ^{242}Pu
T2 Peak ratio, intrinsic efficiency calibration, ^{242}Pu by isotopic correlations
T3 Peak ratio, intrinsic efficiency calibration, ^{238}Pu and ^{242}Pu by isotopic correlations
T4 Peak ratio, intrinsic efficiency calibration, ^{238}Pu , ^{240}Pu , and ^{242}Pu by isotopic correlations
LE Analysis in the low energy range < 230 keV
HE Analysis in the high energy range > 120 keV

Instrument categories:

- XMCG HPGe detector coupled to any MCA, including portable equipment. Electrically or LN2 cooled.
XMCC CZT, $\geq 500 \text{ mm}^3$, coupled to any MCA
XMCL LABR, thickness $\geq 12.7 \text{ mm}$, diameter $\geq 25.4 \text{ mm}$, coupled to any MCA

Material categories:

- Pu PuO₂ clean powder, long time after chemical separation > 1 year
MOX Mixed oxide clean powder based on reactor grade Pu (RGPu), pellets, rods, assemblies:
10 < NU-DU/RGPu < 100

Geometries (Shielding) categories:

- L Low < 3 mm steel (or equivalent, intense X-ray peaks)
H High > 10 mm steel (or equivalent, no/low intensity X-ray peaks) and < 5 mm lead or equivalent

Burnup categories:

- L Low ^{239}Pu isotope mass fraction > 80 %
H High ^{239}Pu isotope mass fraction $\leq 80 \%$

XMCG types:

- D1 Energy resolution < 750 eV @ 122 keV. Active area $\geq 500 \text{ mm}^2$. Thickness $\geq 10 \text{ mm}$. Typical detectors in XMCG-D1 category: planar and semi-planar detectors (GL1015, BE1015, BE2830, BE3830, BE5030, Profile S/SP, GLP).
- D2 Energy resolution < 1.0 keV @ 122 keV and < 2.0 keV @ 1332 keV. Relative efficiency $\geq 15 \%$. Typical detectors in XMCG-D2 category: semi-planar and small volume coaxial detectors (BE2830, BE3830, BE5030, Profile S/SP/C/M, GMX, Falcon, Aegis).
- D3 Energy resolution < 1.5 keV @ 122 keV and < 2.3 keV @ 1332 keV. Relative efficiency $\geq 15 \%$. Typical detectors in XMCG-D3 category: portable electrically cooled coaxial detectors and large volume coaxial detectors (Micro-Detective (ECGS), Aegis, GC9023, Profile C/M, GMX).

TABLE 7a – Total Mass of ²³⁵U and Pu by NDA Gamma-Ray (NDA)

Method	Instrument	Material	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)
			u(r)	u(s)	
HRGS + Computed Efficiency	ECGS - IMCG - MMCG / ISOCS	LEU MTR Fuel Assembly (U alloy); ²³⁵ U, ²³⁸ U			
		HEU MTR Fuel Assembly (U alloy); ²³⁵ U			
		HEU Targets (U metal); ²³⁵ U			
		MTR Fuel Plate/Rod (U alloy); ²³⁵U, ²³⁸U	6.0	3.0	6.7
		Uranium Fuel Rods (U oxide); ²³⁵ U, ²³⁸ U			
		MOX Fuel Rods; ²³⁵ U, ²³⁸ U, ²³⁹ Pu			
		U Oxide Powder; ²³⁵ U, ²³⁸ U			
		Pu Oxide Cans; ²³⁹ Pu			
	ECGS - IMCG - MMCG / ISOCS	LEU Hold-up (ventilation) (U Oxide); ²³⁵ U, ²³⁸ U			
		LEU Hold-up (Process) (U Oxide); ²³⁵ U, ²³⁸ U			
		HEU Hold-up (U oxide); ²³⁵ U, ²³⁸ U			
		LEU Filters (U Oxide); ²³⁵ U, ²³⁸ U			
		Scrap Powder (U Oxide/other); ²³⁵ U, ²³⁸ U			
		Waste (small items; U/Pu Oxide); ²³⁵ U, ²³⁸ U			
		Waste Drums; U-Pu Oxide, metal, other; ²³⁵ U, ²³⁸ U, ²³⁹ Pu			
		Scrap Drums; U-Pu Oxide, metal, other; ²³⁵ U, ²³⁸ U, ²³⁹ Pu			
	ECGS - IMCG - MMCG / ISOCS	Waste Crates; U-Pu Oxide, metal, other; ²³⁵ U, ²³⁸ U, ²³⁹ Pu			
		DU Bulk Containers (Oxides/Catalysts); ²³⁵ U, ²³⁸ U			

1. The ITV are available only for 1 row of the ITV table, namely MTR Fuel Plate/Rod (U alloy), measured using HRGS and employing ISOCS mathematical efficiency calibration method.
2. The ITV Table 7a will be updated with the top-down values, when they are available.
3. Generation of prospective values for the range materials specified in Table 7a is in progress. It will be based on a Monte Carlo bootstrapping approach.

TABLE 7b – Uranium and Plutonium Mass by Neutron (NDA)

Method	Instrument*	Chemical Form	Material	Uncertainty Component (% rel., k=1)		ITV (% rel., k=1)
				u(r)	u(s)	
PNCC	HLNC	Pu Oxide	Can	1.0	0.5	1.1
		MOX	MOX (Pu mass fraction > 10 %)	2.0	0.5	2.1
			MOX (Pu mass fraction < 10 %)	4.0	1.5	4.3
			MOX Rod	2.0	1.0	2.2
			FBR MOX Assemblies	2.0	1.0	2.2
			MOX (Clean Scrap)	5.0	2.0	5.4
PNCC	INVS	MOX	MOX (Pu mass fraction > 10 %)	2.5	1.5	2.9
			MOX (Pu mass fraction < 10 %)	3.5	2.5	4.3
TNCC	GBAS/ HBAS/SBAS	Pu Oxide	Glove Box	10.0	5.0	11.2
		MOX		10.0	5.0	11.2
PNCC	WDAS	Pu Oxide	Waste Drum	10.0	5.0	11.2
ANCC	AWCC	Uranium Alloy	LEU Fuel Assemblies	4.2	3.0	5.2
			HEU Fuel Assemblies	3.0	2.0	3.6
		Uranium Metal	HEU ingots, billets, etc.	6.0	3.0	6.7
ANCC (Thermal)	UNCL	Uranium Oxide	HEU FFA	1.0	1.0	1.4
		Uranium Oxide	LEU FFA	4.0	2.0	4.5
			LEU FFA with NP	4.0	2.0	4.5
ANCC (Fast)	EFCP/EFCB	Uranium Oxide	LEU FFA	2.0	1.5	2.5
			LEU FFA with NP	2.0	1.5	2.5
FNCC (Fast)	FNCL	Uranium Oxide	LEU FFA	2.0	1.5	2.5
			LEU FFA with NP	4.0	1.5	4.3
PNMC	PSMC	Pu Oxide	Can	1.0	0.5	1.1
		MOX	MOX (Clean Scrap)	4.0	1.0	4.1
			MOX (Dirty Scrap)	5.0	1.0	5.1
PNMC	ENMC	MOX	MOX (Pu mass fraction > 10 %)	2.0	0.5	2.1
			MOX (Pu mass fraction < 10 %)	3.0	1.0	3.2
PNCC	HEPC	Uranium Oxide	large LEU/NU Items	3.0	1.0	3.2
PNCC	PNCL	MOX	LWR MOX Assemblies	1.0	3.0	3.2
PNCC	PCAS	MOX	MOX (Pu mass fraction > 10 %)	2.0	0.5	2.1

Uncertainty categories:

ITV: are presented in black font

Prospective Values: are presented in blue font

Appendix II: Using the ITV Tables

In preparation

Appendix III: Safeguards Accountancy and Verification Measurements

In preparation

Appendix IV: Evaluation of Safeguards Accountancy Verification Measurements

In preparation

Appendix V: Description of Measurement Methods Listed in the ITV Tables

(1) Bulk

TABLE 1 – Mass

In preparation

TABLE 2 – Volume and Density

In preparation

(2) Destructive Analysis

TABLE 4a – Uranium Element Mass Fraction Measurements (DA)

TABLE 4b – Plutonium Mass Fraction Measurements (DA)

NOTE: Description of the HKED, KEDG and XRF methods are applicable for Table 4a and Table 4b.
Descriptions of other methods applicable for Table 4a and Table 4 b are in preparation.

Method/measurement system (including software): [HKED / KEDG / XRF](#)

Application: [Measurement of Uranium and Plutonium in Solutions](#)

<p>Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)</p>
<p>K-Edge Densitometry (KEDG), X Ray Fluorescence (XRF), and their combination, Hybrid K-Edge (HKED), are techniques used to determine uranium and plutonium mass fractions or concentrations in dissolver, product, secondary flow, and waste solutions related to fuel reprocessing. A single measurement system, comprising of two High-Purity Germanium (HPGe) detectors, an X ray generator, shielding, collimators, data acquisition electronics, and computer, is typically deployed and capable of all three methods [1]. All three methods are rapid, non-destructive assay techniques, though they are typically listed among destructive assay techniques due to the low uncertainty of KEDG (< 1%). Samples are typically assayed with three (3) replicate measurements, each for a time of 1000 to 3000 s.</p> <p>The KEDG method measures the X ray transmission discontinuity across K-edge energies for an absolute elemental concentration determination in highly concentrated uranium and plutonium solutions (> 50 g U per liter or > 50 g Pu per liter).</p> <p>The XRF method determines the absolute elemental concentrations in pure or mixed solutions of uranium and plutonium in the range of 1 to 50 g per liter by quantifying the fluorescent X rays emitted due to excitation by the generator X rays.</p> <p>The HKED method determines simultaneously uranium and plutonium in solutions where the uranium has higher concentrations, as in the case of dissolver solutions. The XRF portion of HKED quantifies the uranium to plutonium (U/Pu) ratio, and the KEDG portion measures the uranium concentration. The combination of two methods determines the absolute plutonium concentration. Typical uranium concentrations and U/Pu ratios are in the ranges of 150 to 250 g U per liter and 80:1 to 150:1, respectively.</p> <p>HKED systems are installed primarily in on-site analytical laboratories associated with spent fuel reprocessing facilities.</p>
<p>Factors affecting the method performance</p>
<p>The main factors impacting measurement precision and bias of all three methods include the following [1]: counting statistics, X ray generator stability, detector and associated electronics stability, sample homogeneity, sample matrix, sample isotopic abundances, sample container dimensions, sample positioning, and sample temperature.</p> <p>Some of these factors, such as sample temperature and sample isotopic abundances, are corrected for directly in the analysis software. X ray generator stability and sample positioning are typically monitored to be within certain control criteria for every assay or on a periodic basis. Detector electronics stability is also monitored and corrected for by using an in-situ energy calibration check source for each</p>

measurement. The radioactive decay of certain check sources, such as Cd-109, can also affect background evaluation in some KEDG algorithms over time. Its decay rate at the time of installation and sample measurement should be considered during the measurement evaluation. Sample container dimensions are typically measured for each container or monitored by a random sampling of a batch of containers.

Some factors, such as homogeneity and matrix effects, are specific to certain sample types and methods. For instance, high concentration Pu samples measured by KEDG should be performed as quickly as possible due to bubble formation and changing concentration from radiolysis effects. Samples containing suspended particles such as high active liquid waste need homogenization and correction factors, as the XRF calibrations are typically made with clean solutions. The XRF calibrations are also typically made with pure uranium or pure plutonium solutions, and mixed samples may need correction factors to account for attenuations of both the interrogating and fluorescent X rays. Similarly, hybrid-XRF calibrations are typically made with mixed solutions containing no fission products. When present in dissolver solutions, fission products can change the background subtraction analysis and induce additional fluorescence not present in the calibration measurements. Corrective measures such as subtracting an additional passive spectrum measurement without the generator X rays or performing improved background and peak fitting algorithms may be employed.

For uranium assay using KEDG and HKED for the typical three replicate measurements (each of counting time between 1000 s and 3000 s), the ITV values are only valid for concentration levels above 150 g U per liter. When using the HKED technique for dissolved MOX pellets and powders, the uncertainties are typically higher than the ITV values. This is due to lower U/Pu ratios and lower uranium concentrations compared to dissolver solutions. Elemental concentrations below 1 g per liter can be measured by XRF, though with longer counting times and uncertainties larger than the ITV values.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the "Short description" and the "Factors affecting the method performance".

Source of bottom-up value (only for prospective ITV not based on historical data)

N/A

Method references

[1] H. Ottmar and H. Eberle, "The Hybrid K-Edge/K-XRF Densitometer: Principles – Design –Performance," Report KfK 4590, Karlsruhe, 1991.

Prepared by: Dante Nakazawa

Date: Rev0 2022-06-21 (initial draft)

Date: Rev1 2022-06-24 (comments from Andrey Bosko and George Duhamel)

Date: Rev2 2022-06-28 (comments from Kamil Krzysztozek)

TABLE 5a - $^{235}\text{U}/^{238}\text{U}$ Isotope Ratio Measurements (DA)

In preparation

TABLE 6a - Plutonium Isotope Ratio Measurements in Pu and U/Pu Materials (DA)

In preparation

(3) COMPUCEA

TABLE 8 - COMPUCEA

Method/measurement system (including software): (COMPUCEA)**Application:** On-Site Uranium Elemental Assay and ^{235}U Enrichment Determination

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The COMPUCEA (COMbined Procedure for Uranium Concentration and Enrichment Assay) is a transportable analytical measurement system that combines L-edge transmission and γ-ray spectrometry methods for high-accuracy (bias defect) on-site uranium concentration and enrichment assays of various uranium bearing materials (pellets, powders, scrap, liquids) and compounds (UO_x, ADU, AUC, UF_6, UNH, UO_2F_2). A solid sample with a typical mass of 5 g is quantitatively transferred into a uranyl nitrate solution. A multistep procedure, involving dissolution in 8 M nitric acid and subsequent dilution to target 3 M acidity and target 190 gU/l concentration is employed. In the case of a concentrated liquid sample, a dilution step may be required, to bring the uranium concentration inside the L-edge linearity interval (<200 gU/l). The obtained solution is characterized for its density and temperature. Aliquots taken from the prepared sample solution are analyzed by L-edge absorption densitometry for uranium concentration assay and passive gamma-ray counting for enrichment determination. The L-edge densitometer is based on a high-resolution Peltier-cooled Si drift detector (e.g. 10 mm² x 0.45 mm SDD), a miniature 30 -50 kV X-ray generator, and a 2 mm pathlength flow-through quartz cell fixed inside a Ti shield. The gamma-ray spectrometric part consists of two $\varnothing 2.5'' \times 0.5''$ LaBr₃(Ce) detectors, arranged inside tungsten shields. A 10 mL aliquot of solution to be analyzed is loaded into a polystyrene container and counted in a fixed geometry. Three replicates of each measurement type are performed resulting in 3 x 1000 s transmission and 3 x 2000 s gamma-ray spectra. The sample preparation and radiometric measurement data are combined, to evaluate the uranium mass fraction (uranium factor) and the ^{235}U mass fraction (enrichment). In the case of neutron poison additives presence, the measured enrichment is corrected based on the effective Gd_2O_3 content, which is determined from the extrapolated and non-extrapolated fits of the L-edge spectrum. The method is field-calibrated using certified reference material (e.g. CRM-125A) and well-characterized working standards in the form of sintered UO_2 pellets. Daily quality check (QC) measurements are performed to verify the stability of the calibrations in the course of a measurement campaign.</p>
Factors affecting the method performance
<p>The uncertainties shown in Tables 8a and 8b are typical values for cases that are not affected by the factors listed in this section. The uncertainties are normally higher for clean powder samples than for sintered UO_2 pellets, due to moisture uptake, inhomogeneity, and other reasons. This is true for any analytical technique that is accurate enough to see the influence of these factors. However, COMPUCEA is the first technique for which this is explicitly indicated in the ITV tables. Moreover, measurements of low concentrated materials that significantly deviate from the optimal concentration (190 gU/l) would suffer from the low counting statistics in both transmission and gamma spectrometric channels. The absorption measurements of low concentrated solutions may benefit from the use of a longer (e.g. 10 mm) pathlength transmission cell. A significant deviation of the solution acidity from the reference value (3M) will bias the results of the concentration measurements. Large quantities of undissolved residue material (e.g. in the case of dirty scrap) will increase the uncertainty of the uranium factor determination.</p>

In the case of materials containing burnable additive (Gd, Er, etc.), the measured enrichment can be negatively biased by up to 0.5%, if respective correction is not applied. If the correction is applied, it tends to increase variability of ^{235}U enrichment results. Thus, a somewhat larger random uncertainty for ^{235}U enrichment is to be expected in this case. Note that materials containing burnable additives are not considered in Table 8b. Analysis of compounds that are not readily dissolved in nitric acid (e.g. UF_4 , uranium silicide) would require development of dedicated sample preparation procedures.

Source of bottom-up value (only for prospective ITV not based on historical data)

ITVs for UNH solutions (150-400 gU/l): ITVs were derived from the ITVs for solids by increasing the systematic component to reflect the effect of dilution (from 400 gU/l to 190 gU/l) and increasing the random component to reflect the reduced counting statistics at <190 gU/l. The random and systematic uncertainty components for the enrichment determination were adjusted accordingly to account for the increased uncertainty components of respective concentration measurement.

ITVs for UF_6 powder: ITVs were derived from the combination of the UF_6 validation study³ and historical data.

Method references

[1] Erdmann N, Amador P, Arbore P, Eberle H, Luetzenkirchen K, Ottmar H, Schorle H, van Belle P, Lipcsei F, Schwalbach P, Gunnink R (2009) COMPUCEA: a high-performance analysis procedure for timely on-site uranium accountancy verification in LEU fuel fabrication plants. ESARDA Bull 43:30-39.

[2] A. Berlizov, A. Schachinger, K. Roetsch, N. Erdmann, H. Schorle, M. Vargas, J. Zsigrai, A. Kulko, M. Keselica, F. Caillou, V. Unsal, A. Walczak-Typke, Feedback from Operational Experience of on-site Bias Defect Analysis with COMPUCEA, Journal of Radioanalytical and Nuclear Chemistry, Vol. 307(4) (2016) 1901-1909.

[3] M. Toma, H. Schorlé, M. Vargas Zúñiga, J. Zsigrai, A. Berlizov, A. Schachinger, S. Richter, J. Truyens; Validation of the preparation method and analysis of uranium hexafluoride samples with the COMPUCEA system, JRC Karlsruhe, 2017, PUBSY No. 105447

Prepared by: A. Berlizov (IAEA SGTS-TVT), E. Bonner (IAEA SGIM-IFC), K. Zhao (IAEA SGVI), M. Toma (JRC/Karlsruhe), J. Zsigrai (JRC/Karlsruhe)

Date: 2022-06-06

(4) Non-Destructive Assay

TABLE 4c – Uranium and Thorium Mass Fraction (NDA)

Method/measurement system (including software): XRGs / XMCX / GEM**Application:** In-Situ Measurements of Uranium Content in Non-Equilibrating and Aged Bulk Materials with Known Enrichment

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the concentration meter principle [1] that derives the U content from the count rate in the 185.7 keV peak of ^{235}U in a gamma-spectrum measured under <u>quasi-infinite thickness</u> conditions. The method is applicable for low-U materials with contents <25 wt%. The spectrum processing and derivation of the U content are performed using the IAEA's GEM (General Enrichment Measurements) software [2]. The detector types include NaI(Tl), LaBr₃(Ce), CZT, and HPGe. The method requires one-point calibration in the case of fixed container wall thickness or two-point calibration at arbitrary container wall thickness. The method requires <u>knowledge of the ^{235}U enrichment</u>, e.g. as declared by the facility operator or defined by a technological process. The IAEA applies the method for in-situ measurements of in-process materials, low-concentrated sub-products, scraps and wastes at NU conversion and purification plants. A typical measurement system includes a collimated detector, an integrated or stand-alone digital MCA, and an instrumentation laptop. The employed detector arrangements are as follows: (i) electrically cooled HPGe with $\varnothing 50$ mm \times 30 mm crystal and $\varnothing 50$ mm \times 8 mm collimator dimensions; (ii) ≥ 1.5 cm³ CZT with $\varnothing 15$ mm \times 7 mm or larger collimator; (iii) $\varnothing 50.8$ mm \times 12.7 mm LaBr₃(Ce) or NaI(Tl) with $\varnothing 44$ mm \times 20 mm collimator. Typical measurement live times are 500-1000 s.</p>
Factors affecting the method performance
<p>The method assumes low-Z material. Presence of unknown amounts of high-Z elements may bias the measured U content to low values. Other constraints: fulfillment of the quasi-infinite thickness geometry conditions and material homogeneity. The measurement uncertainty may degrade at high ambient radiation background, e.g. when measurements are performed in process or storage areas. Analysis of low-U samples taken from waste streams of the UOC refining process may suffer from the elevated contents of Ra-226 and other decay products of uranium isotopes.</p>
Method references
<p>[1] J.L. Parker, T.D. Reilly, The enrichment meter as a concentration meter, in: Nuclear analysis research and development, Program Status Report, September-December 1972, G. Robert Keepin Comp., Los Alamos Scientific Laboratory report LA-5 197-PR (1972), p. 11.</p> <p>[2] A. Berlizov, GEM: A Next Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopics Analysis for Nuclear Safeguards, February 2021, Vienna, Austria.</p> <p>[3] A. Berlizov, U. Repinc, M. Sturm, K. Henderson, S. Baumann, V. Mayorov, Validation of the Concentration Meter Principle for Nondestructive Assay of Uranium in Low-Concentrated Materials, Proceedings of the International Workshop on U/Pu Isotopics Analysis for Nuclear Safeguards, February 2021, Vienna, Austria.</p>

Source of bottom-up value (only for prospective ITV not based on historical data)

ITVs were evaluated based on the results of the validation study [3], which was performed in the IAEA Safeguards Analytical Laboratory.

Prepared by: A. Berlizov (IAEA SGTND)

Date: 2021-05-17

Method/measurement system (including software): HRGS / XMCG / GEM-FRAM- MGAU**Application:** In-Situ Measurements of Uranium Content in Aged Bulk Materials

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the concentration meter principle [1] that derives the U content from the count rate in the 185.7 keV peak of ^{235}U in a gamma-spectrum measured under <u>quasi-infinite thickness</u> conditions. As the method requires the knowledge of ^{235}U enrichment, it is combined with one of the peak-ratio methods (FRAM [2] or MGAU [3]) to measure the ^{235}U enrichment using the same or separately acquired gamma-spectrum. The method is applicable for low-U materials with contents <25 wt%. The spectrum processing and derivation of the U content are performed using the IAEA's GEM (General Enrichment Measurements) software [4]. The IAEA applies the method for in-situ measurements of in-process materials, low-concentrated sub-products, scraps and wastes at conversion and fuel fabrication plants. A typical measurement system includes a collimated HPGe detector (planar type with $\geq 1000 \text{ mm}^2$ active area or coaxial type with rel. efficiency > 10%), an integrated or stand-alone digital MCA, and an instrumentation laptop. Typical measurement live times are 500-1000 s.</p>
Factors affecting the method performance
<p>The method assumes low-Z material. Presence of unknown amounts of high-Z elements may bias the measured U content to low values. Other constraints: fulfilment of the quasi-infinite thickness geometry conditions, material homogeneity, radioactive equilibrium (materials must be aged for ≥ 4 months after chemical separation or purification). The measurement uncertainty may degrade at high ambient radiation background, e.g. when measurements are performed in process or storage areas. Analysis of low-U samples taken from waste streams of the UOC refining process may suffer from the elevated contents of Ra-226 and other decay products of uranium isotopes.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
ITVs were evaluated based on the results of the validation studies [5] and [6].
Method references
<p>[1] J.L. Parker, T.D. Reilly, The enrichment meter as a concentration meter, in: Nuclear analysis research and development, Program Status Report, September-December 1972, G. Robert Keepin Comp., Los Alamos Scientific Laboratory report LA-5 197-PR (1972), p. 11.</p> <p>[2] T.E. Sampson, T.A. Kelley, PC/FRAM: a code for the nondestructive measurement of the isotopic composition of actinides for safeguards applications, Appl. Radiat. Isot. Vol. 48 (1997) 1543-1548.</p> <p>[3] R. Gunnink, W.D. Ruhter, P. Miller, J. Goerten, M. Swinhoe, H. Wagner, J. Verplancke, M. Bickel, S. Abousahl, MGAU: A new analysis code for measuring U-235 enrichments in arbitrary samples, LLNL Report UCRL-JC-114713, January 1994.</p> <p>[4] A. Berlizov, GEM: A Next Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopics Analysis for Nuclear Safeguards, February 2021, Vienna, Austria.</p>

- [5] A. Berlizov, U. Repinc, M. Sturm, K. Henderson, S. Baumann, V. Mayorov, Validation of the Concentration Meter Principle for Nondestructive Assay of Uranium in Low-Concentrated Materials, Proceedings of the International Workshop on U/Pu Isotopics Analysis for Nuclear Safeguards,
- [6] D. Vo, M. Yoho, Optimization and Verification of FRAM 6.1 Parameter Sets, LANL Report LA-UR-19-30676, 2019-10-21, 53 p.

Prepared by: A. Berlizov (IAEA SGTND)

Date: 2021-05-17

Method/measurement system (including software): HRGS / XMCG / GRASS2**Application:** Uranium Content Determination in Freshly Separated Materials

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The method is based on the gamma-spectrometric assay of sampled material in a fixed “thin-layer” geometry [1] and requires knowledge of the ^{235}U enrichment (e.g., as declared by operator or known from the technological process). A quantitatively taken ~2 g powder is homogenized and then pressed into a uniform disk (~1 mm thick) using a special stainless-steel container with a screw-cap plug. In the case of liquids, a 10 mL aliquot is loaded into a polystyrene container using a fixed volume pipette. The taken sample net mass is determined by container weighing before and after loading. Gamma-spectrometric measurements are performed in a well-defined geometry, which is pre-calibrated in SGAS-NML using secondary standards. Spectrum evaluation is done using the IAEA’s GRASS2 (Gamma-Ray ASSay of Thorium and Uranium) software [2]. The software extracts count rate in the 185.7 keV (^{235}U) peak from a measured spectrum, evaluates material density based on the measured sample layer thickness / volume and sample net mass, and solves a non-linear equation for U content. The solution is aided by on-line Monte Carlo computations of the detector efficiency.

The method is applied by the IAEA at refining and conversion facilities that operates with Nu feed. Typical field setup includes an electrically cooled HPGe detector (active area $\geq 1000 \text{ mm}^2$, thickness $\geq 15 \text{ mm}$), radiation shielding with a sample positioning cavity (sample to detector end cap distance ~10 mm), a digital MCA, and an instrumentation laptop. Spectrum counting live times: 1000 s (>50 wt% powder, >200 g/L solution), 2000 s (20-50 wt% powder, 50-200 g/L solution), 3000 s (<20 wt% powder, <50 g/L solution). Additional equipment includes an electronic balance with ≥ 2 -digit precision, control weights and a 5 mL fixed volume pipette.

Factors affecting the method performance

Unknown contents of high-Z elements in sample matrix may lead to an underestimation of the measured U content. Material inhomogeneity may bias measurement results. Coincidence summing effects are reduced by the use of stainless-steel containers that effectively absorb coinciding low-energy gamma and characteristic X-ray emissions. Weighing and aliquoting inaccuracies. Reproducibility of sample positioning. High ambient radiation background, e.g. when measurements are performed in a process or in a storage area. Freshly separated products do not contain Ra-226 in quantities that could pose a bias. Analysis of low-U samples taken from waste streams of the UOC refining process may suffer from the elevated contents of Ra-226 and other decay products of uranium isotopes.

Method references

- [7] A. Berlizov, Gamma-Spectrometric Assay of Uranium in Support of Partial Defect Verification of In-Process Material, Technical Procedure, SG-TP-15270, 2019-07-08, 39 p.
- [8] A. Berlizov, F. Rorif, V. Unsal, A. Bopape, J. Million, On-Site Gamma-Spectrometric Assay of Uranium and Thorium in Support of Safeguards Verifications at Bulk Handling Facilities, RANC-2016, April 10-16, Budapest, Hungary.

Source of bottom-up value (only for prospective ITV not based on historical data)

ITVs were evaluated by the IAEA using top-bottom analysis of the operator-inspector differences and results of the method's performance testing at SGAS-NML. The analyzed sets contained 42 data points for clean UO_2 , U_3O_8 , UO_3 , UF_4 UOC powders with $C_U=70-80$ wt% and 45 data points for solutions with $C_U=40-570$ gU/L.

Prepared by: A. Berlizov (IAEA SGTND)

Date: 2021-05-16

Method/measurement system (including software): HRGS / XMCG / GRASS2- FRAM-MGAU**Application:** Uranium Content Determination in Aged Materials

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the gamma-spectrometric assay of sampled material in a fixed “thin-layer” geometry [1]. A quantitatively taken 50-100 g powder is homogenized and then quantitatively loaded up to the top of a 30 mL polystyrene container. In the case of liquids, a 30 mL aliquot is loaded. The sample net mass is determined by container weighing before and after loading. Gamma-spectrometric measurements are performed in a well-defined geometry, which is calibrated in the field using secondary standards. Spectrum evaluation is done using the IAEA’s GRASS2 (Gamma-Ray ASSay of Thorium and Uranium) software [2]. The software extracts the count rate in the 1001 keV ($^{238}\text{U}/^{234\text{m}}\text{Pa}$) peak from a measured spectrum, evaluates material density based on the sample mass and volume, and solves a non-linear equation for U content. The solution is aided by on-line Monte Carlo computation of detector efficiency and determination of the ^{235}U enrichment using peak ratio method (MGAU/FRAM) from the same spectrum. The method is applied by the IAEA at fuel fabrication plants. A typical measurement setup includes a shielded uncollimated HPGe detector with $\geq 20\%$ relative efficiency, a holder for reproducible sample positioning at a fix distance from the detector (usually 10-15 cm), a digital MCA and instrumentation laptop. Spectrum counting live time: 2000 s. Additional equipment includes electronic balance with ≥ 2-digit precision, control weights and a 5 mL fixed volume pipette.</p>
Factors affecting the method performance
<p>The method is applicable to materials aged for >4 months since last chemical separation or purification. Non-equilibrium materials will be subject to biasing to higher U contents. Unknown amounts of high-Z elements in sample matrix may lead to an underestimation of the U content. The material inhomogeneity and coincidence summing effects are significantly reduced due to sufficiently large sample-to-detector distance. Other factors include weighing and aliquoting inaccuracies, reproducibility of sample positioning, high radiation background, e.g. when measurements are done in a process or storage area.</p>
Method references
<p>[9] A. Berlizov, Gamma-Spectrometric Assay of Uranium in Support of Partial Defect Verification of Scrap Material, IAEA, Department of Safeguards, Technical Procedure, SG-TP-13872, 2015-11-17, 25 p.</p> <p>[10] A. Berlizov, F. Rorif, V. Unsal, A. Bopape, J. Million, On-Site Gamma-Spectrometric Assay of Uranium and Thorium in Support of Safeguards Verifications at Bulk Handling Facilities, RANC-2016, April 10-16, Budapest, Hungary.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>ITVs were derived by the IAEA using top-bottom analysis of the operator-inspector differences. The analyzed set contained 20 field measurements of homogeneous LEU scrap powders with uranium contents from 17 wt% to 88 wt%.</p>

Prepared by: A. Berlizov (IAEA SGTND)

Date: 2021-05-16

Method/measurement system (including software): HRGS / XMCG / GRASS2**Application:** Thorium Content Determination in Powders and Liquids

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the gamma-spectrometric assay of sampled material in a fixed “thin-layer” geometry [1]. A quantitatively taken 10 g powder is homogenized and then pressed into a uniform disk using a special stainless-steel container with a screw-cap plug. 10 mL liquid samples are loaded by volume into a polystyrene container using a fixed volume pipette. Measurements are done in a well-defined geometry, which is pre- or field calibrated using secondary standards. Spectrum evaluation is done using IAEA’s GRASS2 (Gamma-Ray ASSay of Thorium and Uranium) software [2]. The software extracts background corrected count rates in the 583 keV (^{208}Tl) and 911 keV (^{228}Ac) peaks. Material density is evaluated based on the sample layer thickness / volume and sample mass. A system of two non-linear equations is solved for Th content. The solution is aided by Monte Carlo. Coincidence summing corrections are implemented as function of sample fill height. Evaluation modes include analysis of untreated ore or aged materials, analysis of non-equilibrated materials with unknown separation date, and analysis of materials with known separation date. Typical field setup includes an electrically cooled HPGe detector (active area $\geq 1000 \text{ mm}^2$, thickness $\geq 15 \text{ mm}$), radiation shielding with a sample positioning cavity (sample to detector end cap distance $\sim 10 \text{ mm}$), a digital MCA, and an instrumentation laptop. Spectrum counting live time: 1000 s ($> 20 \text{ wt}\%$), 2000 s ($1-20 \text{ wt}\%$), 3000 s ($< 1 \text{ wt}\%$). Additional equipment includes an electronic balance with ≥ 2-digit precision, control weights and a 5 mL fixed volume pipette.</p>
Factors affecting the method performance
<p>Unknown content of high-Z elements in sample matrix may lead to an underestimation of the measured Th contents. At low Th contents ($< 1 \text{ wt}\%$), the unknown matrix properties may lead to increased systematic uncertainties. Material inhomogeneity may bias measurement results. Presence of radioactive equilibrium is checked and taken into consideration by the GRASS2 software. Therefore, no significant effects on the measurement accuracy from the absence of radioactive equilibrium is expected.</p>
Method references
<p>[1] A. Berlizov, Gamma-Spectrometric Assay of Thorium Using Electrically Cooled Gamma-Spectrometer (ECGS), Technical Report, SG-RP-13429, IAEA, Department of Safeguards, ver. 1, 2015-03-02, 31 p.</p> <p>[2] A. Berlizov, F. Rorif, V. Unsal, A. Bopape, J. Million, On-Site Gamma-Spectrometric Assay of Uranium and Thorium in Support of Safeguards Verifications at Bulk Handling Facilities, RANC-2016, April 10-16, Budapest, Hungary</p>

Source of bottom-up value (only for prospective ITV not based on historical data)

ITVs were evaluated based on the laboratory tests performed using reference standard materials with known Th content in the range from 0.05 wt% to 100 wt%. The dataset of >60 spectra taken from >20 samples was evaluated by ANOVA to yield systemic and random measurement uncertainties for the Th content intervals: <1 wt%, 1-20 wt%, and >20 wt%. The obtained ITVs are expected to be applicable to both powders and liquids.

Prepared by: A. Berlizov (IAEA SGTND)

Date: 2021-05-15

Method/measurement system (including software): HRGS / XMCG / ISOCS**Application: Thorium Content Determination in Powders**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the measurement of powdered material using a laboratory gamma-spectroscopy system equipped with an ISOCS [1] characterized high purity germanium detector inside a 10 cm thick lead shield. Prior to each measurement 20 g of homogenized thorium containing powder is loaded in a 25 mL glass vial and weighed using a high-precision laboratory scale (≥ 0.001 g precision). The sample is then measured in close proximity to the detector using a well-defined counting geometry. The counting time is selected to ensure good counting statistics ($\sim 30,000$ counts) in the 911 keV peak of ^{228}Ac for all samples. Following the measurement, activity of ^{232}Th is determined by averaging measured activities of ^{228}Ac and ^{208}Tl, which are assumed to be in secular equilibrium with ^{232}Th. Thorium concentration in the sample is then calculated by dividing measured thorium mass, which corresponds to the observed ^{232}Th activity value, by the total sample mass.</p> <p>Spectra evaluation is performed using a general gamma-spectroscopy software, e.g., Canberra's Genie 2000. During spectrum evaluation the efficiency calibration is performed using the Canberra's ISOCS calibration software. For each measurement the final efficiency calibration is optimized through several iterations using benchmarks available directly from the measured spectra. The benchmarks include the line activity consistency evaluation for several individual peaks of ^{228}Ac (911, 969, and 338 keV) and ^{208}Tl (583 and 2615 keV), as well as comparison between measured and ISOCS modelled thorium concentration.</p>
Factors affecting the method performance
<p>Material inhomogeneity may bias measurement results. Presence of radioactive equilibrium is assumed, which can be confirmed by comparing measured activities of ^{228}Ac and ^{208}Tl. If not properly corrected for, significant ($>10\%$) bias due to true coincidence summing (TCS) effect can be observed for ^{228}Ac and ^{208}Tl measured activity values when samples are measured close ($<5\text{cm}$) to the detector end-cap.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>ITVs were evaluated based on the laboratory tests performed using reference standard materials with known Th content in the range from 0.1 wt% to 88 wt%. The dataset of 184 spectra taken with 16 samples was evaluated by ANOVA to yield systematic and random measurement uncertainties for the Th content intervals: 0.1 - 1 wt% and >1 wt%.</p>
Method references
<p>[1] Venkataraman R, Bronson F, Atrashkevich V, Field M, and Young B; Improved Detector Response Characterization Method in ISOCS and LabSOCS; Journal of Rad. Nucl. Chem., Vol. 264, No.1, 213-219, 2005.</p>

Prepared by: A. Bosko (IAEA SGAS)

Date: 2021-06-14

Method/measurement system (including software): HRGS / XMCG / A-ISOCS**Application:** Uranium Content Determination in Powder, Scrap.

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>Advanced-ISOCS or Advanced ISOCS Uncertainty Estimator (A-IUE) was developed by Canberra Industries for IAEA's use. A-ISOCS is an efficiency adjustment tool. A-ISOCS varies the "not well-known" parameters in the ISOCS model such as the matrix composition, density, fill height etc and adjusts the efficiency calibration by benchmarking the efficiency shape and magnitude to the data available in the analyzed gamma ray spectra. The benchmarks include relative efficiency results from MGA, MGAU, FRAM analysis, or declared isotopics information, the consistency of line activities from a given multiple-line nuclide, the consistency between measured and modeled uranium or plutonium mass, and the consistency of activities from multiple measurements of the same item. The results presented in this spreadsheet are drawn from 2 reports that were submitted by Canberra Industries to the IAEA, at the end of Phase 2 and Phase 3 of the project. For performance testing A-ISOCS, the IAEA provided 18 measured gamma ray spectra to Canberra, some of which were from the field. These spectra spanned a wide range of enrichments as well as uranium masses. The mass and enrichments were blinded to Canberra. Phase 2 results were obtained using the so called "Best Random Fit" approach for optimizing the efficiency. This was based on creating a large number of models by varying the not-well known parameters within specified uncertainty limits, and find the best model that fit the measured data. Phase 3 results were obtained by optimizing the efficiency using downhill simplex routine.</p>
Factors affecting the method performance
<p>The heterogeneity of source matrix and the non-uniformity of source distribution will impact the results. Performing multiple measurements of a given item from different angles/orientations will help mitigate the bias due to non-uniformities.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
<p>ITVs were evaluated using a top-down approach. The A-ISOCS analysis was carried out using 4 different combinations of optimization benchmarks, and 2 different optimization methods. The 4 benchmarks used were: (i) Relative efficiency from MGAU + ratio of modeled/measured U mass, (ii) User-defined isotopics + ratio of modeled/measured mass, (iii) Relative efficiencies from FRAM + ratio of modeled/measured mass, (iv) Line activity consistency. The 2 different methods used were Best Random Fit and Downhill Simplex. Pooling the results from all 8 different analyses, the random uncertainty $u(r)$ was determined as the standard deviation of the relative deviation of U concentration (or U mass) measurement results w.r.t. the corresponding declared values. The systematic uncertainty $u(s)$ was determined as the standard deviation of the average relative deviations for each of the 8 analysis methods.</p>

Method references

- [1] Andrey Bosko, "Advanced ISOCS for the IAEA - Phase 2: A-IUE Geometry Optimization Software Performance Testing Report", Canberra Industries report, November 2010.
- [2] Gabriela Ilie, "Advanced ISOCS for the IAEA - Phase 3: Performance Testing Report of the Downhill Simplex Optimization Option", Canberra Industries report, May 2013.
- [3] R. Venkataraman, A. Bosko, L.C. Bourva, W. Russ, F. Bronson, A. Berlizov, V. Nizhnik, S. Yoon, A. Lebrun, "Advanced Mathematical Methods for Gamma Ray Based Nuclear Safeguards Measurements", Proc. International Symposium on Nuclear Safeguards, Vienna, October 2014.

Prepared by: R. Venkataraman, Oak Ridge National Laboratory, Oak Ridge, TN, USA.

Date: 2021-06-04

Method/measurement system (including software): XRF / T4/ ED-XRF**Application: Uranium and Thorium Content Determination in Powders and Liquids**

Short Description of the method/measurement system and typical measurement conditions (measurement time, etc.)
<p>The method of X-ray fluorescence analysis (XRF) is suitable for rapid non-destructive analysis to detect and measure the concentration of elements in solid and liquid samples. An XRF analyzer consists of four basic components, X-ray tube, X-ray detector, multi-channel analyzer, and a computer. The energy of each fluorescent X-ray is characteristic of the element excited. The energy dispersive XRF (EDXRF) uses a less powerful X-ray tube (of the order of few hundred watts) to excite the sample and the emitted X-rays are viewed by the detector uncollimated.</p> <p>The EDXRF spectrometer used for the measurements was a Jordan Valley EX-3600TEC with a Rh target operated at 40 kV and the data is processed using the nEXt program provided with the instrument. The EDXRF measurements are based on an internal standard method to determine uranium and thorium concentrations. In this method, a known amount of internal standard yttrium (Y) was added to either a pelletized sample or to a liquid solution that is deposited onto a filter paper. The U $L\alpha$, Th $L\alpha$ characteristic lines, and Y $K\alpha$ line were used for obtaining the calibration plots as well as for the determination of U and Th in the samples. Calibration plots and U/Th concentrations can be made by either plotting U/Y, U/Th and Th/Y amount ratios against the respective intensity ratios of Th $L\alpha$, U $L\alpha$ and Y $K\alpha$ or conversely by plotting the intensity ratios of the analytes and the yttrium X-ray lines against the amount ratios.</p> <p>The powder samples are prepared by using a mixture of uranium and thorium oxides and a fixed amount of yttrium oxide pressed into a pellet. The liquid samples are a result of solutions of uranyl nitrate and thorium nitrate mixed with dissolved yttrium oxide in nitric acid. An aliquot of the liquid solution can be transferred onto a filter paper or other absorbent sheets and dried before measurement.</p>
Factors affecting the method performance
<p>Random error can be reduced by averaging results of repeated measurements. Systematic errors can be reduced by calibration and carefully following established measurement procedures. For thicker samples, additional uncertainty sources have to be taken into account for the determination of the total mass per unit area and the potential non-uniform thickness of the sample. The absorption factor is obtained from a set of transmission measurements of each individual sample and a reference target, and the associated uncertainty is obtained from the measurement uncertainties. The EDXRF is a versatile method for measurements requiring small amounts of sample, minimal sample preparation, and short measurement times. Depending on the concentration range, measurement times can vary from 100s up to 1000s.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
<p>ITVs were evaluated based on the laboratory tests performed using Yttrium as an internal standard added to mixed samples with known Th and U content in the range from 0.1 wt% up to 25 wt%. The obtained ITVs are expected to be applicable to both powders and liquids.</p>

Source of bottom-up value (only for prospective ITV not based on historical data)

ITVs were evaluated based on the laboratory tests performed using Yttrium as an internal standard added to mixed samples with known Th and U content in the range from 0.1 wt% up to 25 wt%. The obtained ITVs are expected to be applicable to both powders and liquids.

Method references

1. An EDXRF method for determination of uranium and thorium in AHWR fuel after dissolution. X-Ray Spectrometry, Vol. 38, 2009, 112-116
2. A comparative study on determination of uranium and thorium in their mixed oxides by EDXRF using tube and radioisotope X-ray sources. X-Ray Spectrometry, Vol. 40, 2011, 379-384

Prepared by: N. St-Amant

Date: 2022-03-4

Method/measurement system (including software): XRF / WDXRF / CAL**Application:** Uranium Content Determination in Solutions Using WDXRF

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>Uranium bearing solutions are irradiated by an X-ray tube in a wavelength dispersive X-ray fluorescent X-ray spectrometer (WDXRF). A LiF220 crystal is used to refract and selectively detect the fluorescent X- rays from the sample. The counts are used from the U $L\alpha_1$ ($2\theta = 37.353^\circ$) line for the calibration.</p> <p>The spectrometer used was the PANalytical Philips Magix Pro PW2440 sequential instrument with a single goniometer, which is equipped with the X-ray tube (Rh anode) providing a maximum voltage/current of 60 kV/125 mA at a maximum power level of 4 kW.</p> <p>The samples covered by this method represent high concentration uranium solutions in an acidic matrix. A set of reference uranium solution samples is required to produce a calibration curve. This calibration curve represents a linear dependence between the raw counts observed for uranium in the WDXRF spectra and uranium concentration in reference solutions. Several reference solutions are typically needed in order to generate a calibration curve that covers the range of concentrations up to 50 gU/L. The sample solutions with concentrations within the calibration range can be analyzed directly, while samples with higher concentration require to be diluted.</p>
Factors affecting the method performance
<p>As WDXRF analysis is performed using the very low energy X-ray lines the sample preparation becomes a critical factor affecting the accuracy of the result. Any deviation in sample composition between the reference samples used for generating the calibration curve and unknown samples to be analyzed may affect the measured result.</p> <p>Additional factors include spectral interferences, instrument instability (drift), counting statistics, etc.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>Multiple WDXRF measurements of the quality control samples at one of the uranium refinery facilities were used to evaluate the method performance. The calibration was performed by measuring eight reference uranium solution samples in the range of 5 to 50 gU/L, and using raw counts to produce a linear calibration curve. The control solutions had concentrations of ~100 gU/L and ~400 gU/L, and therefore required ~10 times dilution in order to fall within the calibration range.</p>
Method references
n/a

Prepared by: A. Bosko (IAEA SGAS)

Date: 2022-03-22

Method/measurement system (including software): XRF / WDXRF / FP**Application: Thorium Content Determination in Powders Using WDXRF**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method is based on the measurement of a powdered material using a laboratory Wavelength Dispersive X-Ray Fluorescence Spectrometer (WDXRF). The samples for WDXRF measurements are typically prepared in the form of loose powder, press pellet, or fused glass bead.</p> <p>The WDXRF spectrometer used to evaluate the method performance was the PANalytical Philips Magix Pro PW2440 sequential instrument with a single goniometer, which is equipped with the X-ray tube (Rh anode) providing a maximum voltage/current of 60 kV/125 mA at a maximum power level of 4 kW.</p> <p>Data acquisition and analysis for the purpose of method evaluation were performed with the PANalytical SuperQ v5.3 software using the Omnian “standardless” analysis (fundamental parameters (FP) method).</p> <p>The FP method [1-3] is based upon the theoretical relationship between measured X-ray intensities and the concentrations of elements in the sample. During analysis process the theoretical X-ray intensity for each identified element is iteratively compared to the theoretical value, which is calculated by combining the physical X-ray production processes and the measured sample composition using known fundamental atomic parameters (attenuation/absorption factors, X-ray energies, relative intensities, etc.).</p>
Factors affecting the method performance
<p>As WDXRF analysis is performed using the very low energy X-ray lines the sample preparation becomes a critical factor affecting the accuracy of the result. The following sample parameters will influence the method performance:</p> <ul style="list-style-type: none"> • quality of the sample surface • sample homogeneity • sample thickness (must be “infinitely” thick for the most accurate result) <p>Additional factors include spectral interferences, instrument stability (drift), counting statistics, etc.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>ITVs were evaluated based on the laboratory measurements performed using a set of 10 reference materials with known Th content in the range from 0.05 wt% to 10.2 wt%. The samples for these measurements were prepared by mixing the sample powder with Flux 66-34 and fusing it into a glass bead. The dataset of 5 independent measurement results taken with each of 10 samples was evaluated by ANOVA to yield systematic and random measurement uncertainties for the Th content.</p>

Method references

1. Sherman J. The theoretical derivation of fluorescent X-ray intensities from mixtures. *Spectrochimica Acta*; 7: 283-24 (1955).
2. R. Jenkins, R.W. Gould, and D. Gedcke, *Quantitative X-Ray Spectrometry*, 2nd edition (1995).
3. R. Tertian and F. Claisse, *Principles of Quantitative X-Ray Fluorescence Analysis* (1982).

Prepared by: A. Bosko (IAEA SGAS)

Date: 2022-03-24

TABLE 5b – ^{235}U Enrichment (NDA)**Method/measurement system (including software):** T0 / XMCN**Application:** Measurements of Uranium Enrichment in UF_6 and Pure UO_x

<p>Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)</p>
<p>XMCN is a portable gamma-spectrometry system, which is based on the $\varnothing 2'' \times 0.5''$ NaI(Tl) scintillation detector (typical FWHM $\approx 11\%$ @ 185.7 keV) coupled to an InSpector-2000 Multichannel Analyzer (IMCN) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCN) from GBS Elektronik GmbH.</p> <p>The MCA is typically set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300.</p> <p>The NaI(Tl) crystal is usually seeded by ^{241}Am- isotope for temperature stabilization purposes. The detector's field of view is shaped by a 44 mm diameter by 20 mm length collimator made of lead. The ^{235}U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ^{235}U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF_6 or >2.7 cm for UO_2 powder with $\rho = 2$ g/cm3).</p> <p>Typically, the spectrum is measured for 300 s live time. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the two-regions-of-interest (2ROI) approach [2], e.g., as implemented in WinU235 [3] and IMCA [4] codes. In this approach, two non-overlapping ROIs are defined within and above the 185.7 keV peak and intended for net peak area determination using a linear combination of the gross numbers of counts registered within their limits.</p> <p>The method requires at least two "infinitely" thick standards for calibration. More standards can be employed to achieve better calibration accuracy. Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term. The term includes the container wall thickness, which is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.</p>
<p>Factors affecting the method performance</p>
<p>Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ^{235}U enrichment will impact the results.</p> <p>Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with poorly known uranium content. A negative bias in the measured enrichment should be expected for materials containing burnable poison additives, such as Gd, Er etc. Due to the uncontrolled contribution from the forward scattering into the lower ROI, the 2ROI approach usually performs better in narrow wall thickness intervals. Multiple calibrations are normally prepared to cover a wider wall thickness interval. Additionally, a significant bias should be expected for materials containing reprocessed uranium due to the uncontrolled contribution of ^{232}U decay products to the upper ROI.</p>

<p>Measurements in industrial environments require observation of background conditions for avoiding biasing to higher enrichment values. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. Temperature instabilities of system's parameters may shift energy calibration, thereby leading to an improper definition of the ROIs and biasing the enrichment result.</p>
<p>Source of top-down value and ITV applicability</p>
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.</p> <p>The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.</p>
<p>Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)</p>
<p>n/a</p>
<p>Method references</p>
<p>[1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.</p> <p>[2] D. Reilly, N. Ensslin, H. Smith Jr., S. Kreiner, Passive Nondestructive Assay of Nuclear Materials, United States Nuclear Regulatory Commission, Washington, D.C, NUREG/CR-5550, LA-UR90-732 (1991) 211–214.</p> <p>[3] WinU235 - Program for ^{235}U enrichment measurements with NaI-detectors, Ver. 2.02, GBS Elektronik GmbH, https://www.gbs-elektronik.de/en/downloads/downloads-nuclear-measurements.php</p> <p>[4] G.H. Gardner, M. Koskelo, M. Moeslinger, R.L. Mayer, B.R. McGinnis, The IMCA: A Field Instrument for Uranium Enrichment Measurements, Proceedings of the 37th Annual Meeting of the Institute of Nuclear Materials Management, Naples, FL, USA, 28 July - 1 August 1996, p. 680-685.</p>

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / ECGS**Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

ECGS stands for Electrically Cooled Gamma Spectrometer, which is a portable high-energy resolution HPGe-detector-based system with an integrated MCA, a microcomputer and an electrical cooler (typical FWHM \approx 1.1 keV @ 122 keV). The IAEA's ECGS is represented by the ORTEC Micro-UF₆TM system, a variant of the standard ORTEC Micro-trans-SPEC portable gamma-spectrometer. The system includes a coaxial HPGe crystal with \varnothing 50 mm \times 30 mm nominal dimensions, furnished with a \varnothing 50 mm \times 8 mm front-end tungsten collimator.

The spectrometer is normally set up to acquire a 4k-channel gamma-spectrum with the 0.075 keV/chn gain. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness $>$ 7 mean-free-paths of the 185.7 keV gammas, e.g. $>$ 1.5 cm for UF₆ or $>$ 2.7 cm for UO₂ powder with $\rho = 2$ g/cm³). The gamma-spectrum is typically measured for 300 s live time.

The measurement geometry ensures that the examined material completely (i.e. without voids) fills the space within the detector's field of view. The on-board *UF₆ Enrichment* program [2] determines the 185.7 keV peak area via a linear background subtraction using two non-symmetrical continuum regions-of-interest (ROIs) around the peak ROI.

Other spectrum evaluation algorithms, e.g. smoothed step background subtraction or peak fitting, can be utilized as well. The method relies on one-point enrichment calibration. Owing to the high-energy resolution of the employed detector, the method is barely affected by the effects of the low-angle photon scattering and isotopic interference (e.g. from ²³²U decay daughters in the case of reprocessed uranium).

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction factor with an empirical quadratic term in the exponent that adequately addresses the ECGS's wide solid angle measurement geometry [2]. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results. Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities.

Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or for materials containing burnable poison additives, such as Gd, Er etc. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments.

Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. The ORTEC Micro-trans-SPEC experiences high deadtimes and prone to overloading when measuring

<p>materials with high contents of reprocessed uranium. Therefore, lower performance is to be expected in this case due to the need to use a narrower collimator and significantly deteriorated peak-to-continuum ratio.</p>
<p>Source of top-down value and ITV applicability</p>
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.</p> <p>The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.</p>
<p>Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)</p>
<p>n/a</p>
<p>Method references</p>
<p>[1] [1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.</p> <p>[2] [2] ORTEC Micro-UF6™ Portable UF6 Cylinder Verification System, Software v.2.3 — User's Manual, ORTEC Part No. 932500, 177 p.</p>

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / HM-5**Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

HM-5 is a handheld gamma-spectrometric instrument [1], which is based on the Ø23 mm x 21 mm NaI(Tl) scintillation detector (typical FWHM ≈ 8.5% @ 662 keV). The detector has an internal 5 mm thick tungsten side shielding and a not-thallium-activated rear NaI crystal, serving as a back shielding. The main outward-facing NaI(Tl) crystal is recessed into the internal collimator by 5 mm, thereby determining a wide-solid-angle field of view of the detector.

The spectrometer has a LED-based temperature stabilization of the photomultiplier tube and a built-in ~500 Bq Cs-137 check source for instrument's gain initialization on startup. The HM-5 is set up to acquire a 1k-channel gamma-spectrum with the 3 keV/chn gain. The ²³⁵U enrichment is determined using the "enrichment meter" principle [2], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with ρ = 2 g/cm³). Typically, a spectrum is measured for 120 s (LEU) or 180 s (NU) live time. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the peak fitting approach implemented in the NaIGEM code [3].

The method relies on one-point enrichment calibration. A wide range of container wall thicknesses is covered owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The method accounts for spectrum distortion effects due to decay products of ²³²U in the case of reprocessed uranium.

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or materials containing burnable poison additives, such as Gd, Er, etc.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of top-down value and ITV applicability
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.</p> <p>The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
<p>[1] Safeguards Techniques and Equipment: 2011 Edition, International Nuclear Verification Series No.1 (Rev. 2), International Atomic Energy Agency, Vienna, 2011, 162 p.</p> <p>[2] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.</p> <p>[3] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ^{235}U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997.</p>

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / XMCC**Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCC is a portable gamma-spectrometry system, which is based on a $\geq 1500 \text{ mm}^3$ (for NU/LEU) or $\geq 500 \text{ mm}^3$ (for HEU) cadmium zinc telluride (CZT) semiconductor room temperature detector with typical FWHM $\approx 2.5\%$ @ 122 keV. The detector can be coupled to an InSpector-2000 Multichannel Analyzer (IMCC) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-527 (MMCC) from GBS Elektronik GmbH, or another MCA. The MMCC system operated via the MCA Touch (MCAT) v.2.X software from GBS Elektronik GmbH [1] represents a standard IAEA setup.

In this setup, a CZT detector is arranged inside a tungsten shielding with the collimator opening matching dimensions of the detector's active area, e.g., 15 mm in diameter in the case of a $15 \times 15 \times 7.5 \text{ mm}^3$ hemispherical CZT/1500 from Ritec Ltd. [2]. A typical collimator length is 5 mm. The system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. The ^{235}U enrichment is determined using the "enrichment meter" principle [3], i.e. on the absolute intensity of counts in the 185.7 keV peak of ^{235}U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness > 7 mean-free-paths of the 185.7 keV gammas, e.g. $> 1.5 \text{ cm}$ for UF₆ or $> 2.7 \text{ cm}$ for UO₂ powder with $\rho = 2 \text{ g/cm}^3$).

Typical spectrum measurement live time is 300 s. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using a peak fitting approach implemented in the GEM software [4]. The method relies on one-point enrichment calibration. It covers a wide range of container wall thicknesses owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The latter removes a considerable systematic bias (inherent to the older two-region method) due to the contribution of the low-angle Compton scattering under the full energy peaks. The method accounts for the spectrum distortion effects due to decay products of ^{232}U in the case of reprocessed uranium.

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured. For an unknown compound, the matrix correction can be calculated using the declared or independently measured uranium content. Additional correction is implemented to account for the presence of burnable poison additives, such as Gd, Er etc.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ^{235}U enrichment will impact the results. Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. The container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of bottom-up value (only for prospective ITV not based on historical data)

The prospective ITVs were derived considering different factors contributing to both systematic and random uncertainty components. Various parameters in the applied analytical expressions were determined based on the carried-out laboratory and limited field tests. Details of the approach are presented in the Annex.

Method references

- [1] MCA Touch user manual, GBS Elektronik GmbH, JOPAG/05.12-DOC-99 Joint Program on the technical development and further improvement of IAEA safeguards between the government of the Federal Republic of Germany and the IAEA, Task C.40/A7191, February 2016, ver. 1.8, 61 p.
- [2] ZRF Ritec SIA, CZT/1500, <http://www.ritec.lv/html/czt500.html>.
- [3] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [4] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)

Prepared by: Andriy Berlizov

ANNEX: Descriptions of the bottom-up evaluations of the measurement uncertainties for XMCC

A. Berlizov (IAEA, SGTVT)

1. Random uncertainty

The random component of the measurement uncertainty was calculated using the following formula:

$$u^2(r) = u_{PEAK}^2 + u_{WALL}^2$$

Here, u_{PEAK} is the random component of the relative uncertainty (%) of the 186 keV peak area determination from the measured gamma spectrum; u_{WALL} is the random component of the relative uncertainty (%) of the wall thickness determination using ultrasonic thickness gauge.

Based on the practically achievable accuracy of the wall thickness determination using IAEA's standard equipment, the value $u_{WALL} = 1.5\%$ was assumed in the current evaluations.

The peak area uncertainty was calculated using the following formula:

$$u_{PEAK}^2 = \frac{10^4}{N} \times \left(1 + 2 \frac{B}{N}\right)$$

Here, N is the net number of counts in the 186 keV peak (net peak area), and B is the number of counts in the continuum background under the peak. The intensity of the 186 keV peak in the ambient background was assumed to be zero.

The net peak area was estimated using the following formula:

$$N = Enr \times K^{-1} \times F(x) \times F_M \times LT$$

Where, Enr is the material ^{235}U enrichment (wt %), K is the enrichment calibration constant (= 0.0310 wt %/cps), $F(x)$ is the attenuation correction factor for container wall thickness x (cm); F_M is the matrix correction factor (= 1.0153 for U_3O_8); and LT is the spectrum acquisition live time (s).

The attenuation correction factor was calculated using the following formula:

$$F(x) = \exp(-\mu a(1 - bx)x)$$

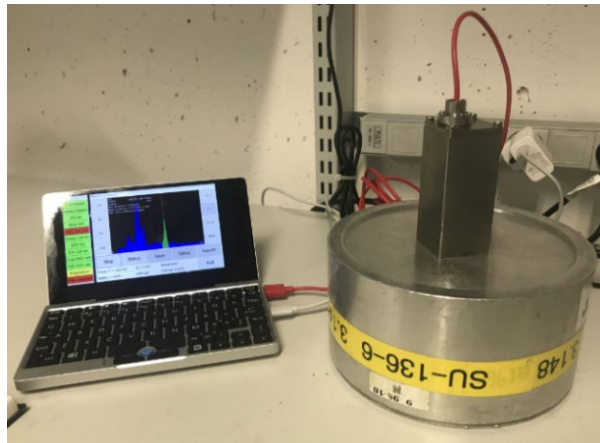
Where, $\mu = 1.21 \text{ cm}^{-1}$ is the linear attenuation coefficient for the 186 keV photons in steel; $a = 1.1136$ and $b = 0.0143 \text{ cm}^{-1}$ are the collimator specific correction constants.

The background-to-peak count ratio B/N was parameterized as function of material enrichment. The parameterization was based on the set of experimental spectra measured with the 16 mm (the thickest) steel absorber:

$$\frac{B}{N} = 0.958 + \frac{4.3}{Enr}$$

Experimental measurements were performed using the following equipment and experimental setup:

- Detector: CZT 1500 mm³ (RITEC), crystal dimensions = 15 × 15 × 7.5 mm³, collimator dimensions = Ø15 mm × 5 mm (tungsten).
- Multiple channel analyzer: MCA-527, GBS Elektronik
- Software: MCAT 2.0, GEM v.2.2.5
- Working standard materials: SU-135 (19.82 wt %), SU-136 (3.105 wt %), SU-137 (0.708 wt %), SU-138 (0.217 wt %), U₃O₈ powder, quasi-infinitely thick layer = 20 mm, 163 mm diameter, aluminum can with 2 mm thick working window.
- Shielding: 0 mm, 4 mm, 8 mm, 12 mm, 16 mm
- Measurement geometry:



2. Systematic uncertainty

The systematic uncertainty was calculated taking into account the uncertainty of the system enrichment calibration (typical value is $u_{CAL} = 0.6\%$) and the systematic uncertainty of the spectrum fitting. The latter was approximated by the equation: $u_{FIT} = 1.243 + 0.0782 \cdot (B/N)$. The total systematic uncertainty was thus calculated as follows:

$$u^2(s) = u_{CAL}^2 + u_{FIT}^2$$

Method/measurement system (including software): T1 / XMCG**Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCG is a portable high-resolution gamma-spectrometry system, which is based on a liquid nitrogen (LN₂) cooled high-purity germanium (HPGe) detector coupled to an InSpector-2000 Multichannel Analyzer (IMCG) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCG) from GBS Elektronik GmbH. A typical IAEA XMCG is based on the low-energy GL1015 or broad-energy BE1015 planar detector from Mirion Technologies.

The detector has a 1000 mm² x 15 mm HPGe crystal and typical FWHM ≤ 600 eV @ 122 keV. The detector is positioned inside a lead collimator-shielding, recessed by 25 mm with respect to its Ø35 mm x 15 mm opening. The ²³⁵U enrichment is determined using the “enrichment meter” principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an “infinitely” thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with ρ = 2 g/cm³).

The gamma-spectrum is typically measured for 300 s live time. The measurement geometry ensures that the examined material completely (i.e. without voids) fills the space within the detector’s field of view. A modified version of the IMCA program [2] determines the 185.7 keV peak area via a linear background subtraction using two continuum regions-of-interest (ROIs) around the peak ROI. The method relies on one-point enrichment calibration. Owing to the high-energy resolution of the detector, the method is barely affected by the effects of the low-angle photon scattering and isotopic interference (e.g. from ²³²U decay daughters in the case of reprocessed uranium).

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction factor with an empirical quadratic term in the exponent [2]. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic “infinite” thickness geometry assumptions regarding material layer thickness and coverage of the detector’s field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or for materials containing burnable poison additives, such as Gd, Er etc.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. In the case of reprocessed uranium, the measurement performance is expected to deteriorate due to the lower peak-to-continuum ratio.

Source of top-down value and ITV applicability
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.</p> <p>The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
<p>[1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.</p> <p>[2] G.H. Gardner, M. Koskelo, M. Moeslinger, R.L. Mayer, B.R. McGinnis, The IMCA: A Field Instrument for Uranium Enrichment Measurements, Proceedings of the 37th Annual Meeting of the Institute of Nuclear Materials Management, Naples, FL, USA, 28 July - 1 August 1996, p. 680-685.</p> <p>[3] ORTEC Micro-UF6™ Portable UF6 Cylinder Verification System, Software v.2.3 — User's Manual, ORTEC Part No. 932500, 177 p.</p>

Prepared by: Andriy Berlizov and Kamil Krzysztosek

Method/measurement system (including software): T1 / XMCL**Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCL is a portable gamma-spectrometry system, which is based on a $\varnothing 2'' \times 0.5''$ LaBr₃(Ce) scintillation detector (typical FWHM $\approx 5\%$ @ 185.7 keV) coupled to an InSpector-2000 Multichannel Analyzer (IMCL) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-527 (MMCL) from GBS Elektronik GmbH. The LaBr₃(Ce) crystal is not normally temperature stabilized, thus, in this case, the temperature instabilities are compensated by spectrum processing algorithms. The detector's field of view is shaped by a 44 mm diameter by 20 mm length collimator made of lead or tungsten. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with $\rho = 2$ g/cm³). The XMCL system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. Typically, a spectrum is measured for 300 s live time.

The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the peak fitting approach, such as NaIGEM [2], GEM [3] or similar. The method relies on one-point enrichment calibration, which covers a wide range of container wall thicknesses owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The latter removes a considerable systematic bias (inherent to the older two-region method) due to the contribution of the low-angle Compton scattering under the full energy peaks.

The method accounts for the spectrum distortion effects due to decay products of ²³²U in the case of reprocessed uranium. Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured. For an unknown compound, the matrix correction can be calculated using the declared or independently measured uranium content. Additional correction is implemented to account for the presence of burnable poison additives, such as Gd, Er etc.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of top-down value and ITV applicability
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.</p> <p>The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.</p>
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
<p>[1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.</p> <p>[2] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ^{235}U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997.</p> <p>[3] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)</p>

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T2 / XMCN/XMCL**Application: Measurements of Uranium Enrichment in UO_x Pellets and Fuel Rods**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>IAEA inspectors perform in-field measurements of ²³⁵U enrichment of UO_x pellets and fuel rods using a portable low- or medium-resolution gamma-spectrometer. The spectrometer is based on an IAEA standard NaI(Tl) (XMCN) or LaBr₃(Ce) (XMCL) Ø2" x 0.5" scintillation detector with typical energy resolution (FWHM at 185.7 keV) of ≈11% (XMCN) or ≈5% (XMCL). The detector is arranged inside a 10 mm thick lead shielding that is opened from the detector's front-end. The detector is coupled to an InSpector-2000 Multichannel Analyzer (IMCN/IMCL) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCN/MMCL) from GBS Elektronik GmbH.</p>
<p>The measurement system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum. Unlike conventional "enrichment meter" applications, requiring that the measured material completely fills the space within the detector's field of view, the pellet/rod measurements rely on a controlled, reproducible positioning of measured items (normally five pellets or a fuel rod) at a fixed distance from the detector. The high accuracy and reproducibility of positioning are achieved by the use of special pellet/rod holders. The method is calibrated using a standard set of reference pellets or a standard fuel rod measured using the same holder. Field measurements are typically done for 500 s (LEU) or 1000 s (NU/DU) live time. The 185.7 keV peak intensity is determined using a peak fitting approach, such as NaIGEM [2] or GEM [3]. The spectrum fitting accounts for possible presence of decay products of ²³²U in the case of reprocessed uranium.</p>
<p>Data evaluations include application of a matrix correction (including effects of burnable poison additives, such as Gd, Er etc.) and a geometry correction that accounts for possible deviations between the calibration and measured pellets/rods properties (external diameter, inner hole diameter, pellet's height, rod's cladding material and cladding thickness). The external dimensions of pellets/rod are measured/verified in the field using an electronic caliper.</p>
Factors affecting the method performance
<p>Inaccurate measurement geometry setup (e.g., a gap between detector endcap and pellets/rod holder) or inaccurate pellets/rod positioning will bias the measured enrichment. Unknown or inaccurate information about the content of the burnable poison additive will result in a systematic bias. Inaccuracies in the pellets/rods dimension measurements as well as selection of wrong cladding material for a fuel rod (e.g., zircaloy instead of stainless steel) will bias the measured enrichment. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak.</p>
Source of top-down value and ITV applicability
<p>The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random u(r) and short-term systematic u(s) uncertainties.</p>

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in “Short description”, on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the ‘Notes’ column in the table.

Source of bottom-up value (only for prospective ITV not based on historical data)

n/a

Method references

- [1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [2] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ²³⁵U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997.
- [3] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): HRGS/XMCG Peak Ratio/FRAM-MGAU**Application:** ^{235}U enrichment measurements employing the low energy (< 250 keV) or high energy (> 120 keV) region of the spectrum

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The method derives the ^{235}U enrichment from ratios of specific nuclear and detection parameters related to two different isotopes, including the peak area of selected gamma-rays in a high-resolution gamma-spectrum. The method can be used to measure items of arbitrary size, shape and composition. It is applicable to any enrichment level and is useful for in-situ measurements of aged bulk materials with net weight of a few grams and up. A typical measurement system includes a collimated HPGe detector, an integrated or stand-alone digital MCA, and an instrumentation laptop.

For the low-energy (< 250 keV) region uranium analysis, the technique uses the pair of ^{238}U 's daughter ^{234}Th peaks at 92.4 and 92.8 keV to measure the activity of ^{238}U . This requires ^{234}Th to be in secular equilibrium with its parent ^{238}U or the chemical separation date is known so correction can be applied. For the measurement of ^{235}U activity, the peaks at 89 and 93 keV, as well as some other peaks at higher energy, may be used. In order to avoid fitting problems due to poor quality (resolution) spectra, this low-energy region analysis requires detector with resolution < 600 eV at 122 keV Co-57, such as the planar HPGe detector. Spectra with 4K channels are typically acquired, with energy calibration of 0.075 keV/ch. Typical measurement time is 300-1800 s, depending on material amount in the item and container wall thickness (< 1.5 mm Fe). The two software codes employing this technique are FRAM [1, 2] and MGAU [3].

For the high-energy (> 120 keV) region uranium analysis, the technique uses the gamma rays of ^{238}U 's granddaughter $^{234}\text{Pa}/^{234\text{m}}\text{Pa}$ peaks to measure the activity of ^{238}U . This requires ^{234}Pa to be in secular equilibrium with its grandparent ^{238}U or the chemical separation date is known so correction can be applied. The method uses the gamma ray peaks from 120 keV up to 1001 keV. The high energy peaks mean that the measurements are more efficient with high efficiency detector such as a large coaxial or semi-planar HPGe detector. Spectra with 8K channels are typically acquired, with energy calibration of 0.125 keV/ch. Typical measurement time is 300-1800 s, depending on material amount in the item and container wall thickness (< 2 cm Fe). The software code employing this technique is FRAM [1, 2].

Factors affecting the method performance

The present ITV values are for homogeneous materials only. The method based on low energy works well with bare or lightly shielded samples (< 1.5 mm Fe).

The method based on high energy works well with bare samples up to moderately shielded (< 2 cm steel) samples. It can also analyze samples that are heavily shielded up to 5 cm steel, but the biases may be significantly larger. For HEU, the method depends somewhat on the intense gamma ray peaks of $^{232}\text{U} \rightarrow ^{228}\text{Th}$ decay chain to bridge the large gap between the low energy peaks of ^{235}U and the high energy peaks of ^{238}U . The method therefore is less accurate and precise when analysing HEU that does not include any reprocessed uranium. For LEU, the method does not depend on the peaks of $^{232}\text{U} \rightarrow ^{228}\text{Th}$ decay chain and can analyze uranium items that do or do not contain reprocessed uranium equally well.

Both the low-energy and high-energy methods require the uranium to be aged (> 6 months after chemical separation). For fresh uranium, both the MGAU and the FRAM codes allow the user to enter the separation date, if well-known, and the codes will correct the results for the freshness of the material. However, enrichment results with higher uncertainty levels can be observed due to this additional correction. Using HPGe planar detectors with resolution poorer than the recommended level may also compromise the measurement uncertainty. Finally, measurements at too high counting rates

may lead to degraded detector resolution, tailing caused by pulse pileup, and random and coincidence sum peaks, all of which can lead to significant measurement biases.

Source of bottom-up value (only for prospective ITV not based on historical data)

To date, ITV values are from the analysis of lab data using FRAM in the high energy mode and counting time of 600 s. Depending on the source sizes and the measurement distances, the counting rates for the data varied from few kHz up to 50 kHz. For the low energy range, ITV values for DU, NU, and LEU are typically based on field data using MGAU and 300 s as counting time. In any case, only spectra with low to medium counting rates (< 10 kHz) were considered. The ITV values for HEU are from lab data at 300 s counting time.

Method references

- [1] T.E. Sampson, T.A. Kelley, PC/FRAM: a code for the non-destructive measurement of the isotopic composition of actinides for safeguards applications, *Appl. Radiat. Isot.* Vol. 48 (1997) 1543-1548.
- [2] D.T. Vo, "Improvements of FRAM version 6.1," LANL Report LA-UR-19-28449 (2019).
- [3] R. Gunnink, W.D. Ruhter, P. Miller, J. Goerten, M. Swinhoe, H. Wagner, J. Verplancke, M. Bickel, S. Abousahl, MGAU: A new analysis code for measuring ^{235}U enrichments in arbitrary samples, LLNL Report UCRL-JC-114713, January 1994.

Prepared by: Duc Vo and Fabio Dias

TABLE 6b – Plutonium Isotopic Abundances (NDA)

Method/measurement system (including software): HRGS / XMCG / FRAM-MGA

Application: Measurement of Plutonium Isotopic Composition in Pure Plutonium Samples (Low-energy analysis)

<p>Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)</p>
<p>The method determines plutonium isotopic composition by analyzing high resolution gamma spectra acquired with a planar or small semi-planar High-Purity Germanium (HPGe) detector having the energy resolution FWHM < 750 eV @ 122 keV. The method employs the low-energy region (59-230 keV) analysis of the isotopic analysis software codes FRAM v.6.1 (Fixed-Energy Response-Function with Multiple Efficiency [1]) and MGA v.10.0 (Multi Group Analysis Code for Plutonium [2]). The method can be used to measure plutonium items of arbitrary size, shape, and composition. It is applicable to any type of plutonium (aged, fresh, high- and low-burnup samples) and is useful for in-situ measurements of bulk materials with net weight of tens of mg up to several kg.</p> <p>A typical measurement system includes a collimated HPGe detector, a Multi-Channel Analyzer (MCA), and an instrumentation laptop. The MCA is typically set to acquire spectra with 4096 channels and detector gain adjusted to ~0.075 keV/channel. A 1-2 mm cadmium or tin filter is normally used to suppress the 59.5 keV gamma line of ²⁴¹Am to the extent when its amplitude is at about the same level as the intense X-ray peaks in the 100 keV region.</p> <p>Typical measurement time is 300-1800 s, although the presented data were normalized to a live time (LT) of 600 s. The LT of 600 s allows for ~50,000 counts in the reference 129 keV peak of ²³⁹Pu to be acquired with a high-burnup plutonium sample at output count rate (OCR) of ~22,000 cps.</p>
<p>Factors affecting the method performance</p>
<p>High count rate applications, i.e. with DT > 40% (or OCR > 22,000 cps), may broaden the peak energy resolution, alter the peak shapes, and also lead to pulse pileup. This will affect the accuracy and precision of the measurement results.</p> <p>The method generally provides good results when measuring plutonium in a thin wall container (< 3 mm SS (or equivalent)), which does not significantly attenuate the intense X-ray region around 100 keV. Moderately thick and/or heavy absorber (> 10 mm SS (or equivalent)) will significantly suppress the X-ray peaks in the 100-keV region, which can effectively render the technique useless.</p> <p>The ITVs were derived assuming homogeneous samples. The material inhomogeneity, as well as heavy shielding, high ambient radiation background (including presence of fission product emissions, e.g., at nuclear power plants), presence of high-Z elements in material matrix with X-ray emissions close to the Pu X-rays (e.g., such as Th), would worsen the performance of the method.</p>

Source of bottom-up value (only for prospective ITV not based on historical data)

Current evaluations are based on the evaluation of a number of plutonium spectra available at Los Alamos National Laboratory (LANL) and IAEA's Safeguards Analytical Laboratory (IAEA SAL).

LANL analyzed its plutonium data using FRAM v.6.1 with the parameter set GePInr_Pu_060-230. The spectra were measured in lab environments with counting time from 900 to 5400 s and the results were normalized to a counting time of 600 s. The input count rates for the data varied from about several thousand cps up to 50,000 cps. The filters were 0.8-1.6mm cadmium. 67 data sets with 1117 spectra were used in the uncertainty evaluation.

IAEA analyzed its plutonium data using FRAM v.6.1 with the parameter set GePInr_Pu_060-230, and MGA v.10.0. This data included the collection of 111 high-burnup plutonium samples measured over several years in a laboratory environment at IAEA SAL, and 29 good statistics spectra of low-burnup plutonium extracted from the international database. All data were normalized to a LT=600s.

Method references

- [1] D. Vo, "Improvements of FRAM version 6.1," International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 19-30.
- [2] R. Gunnink, MGA: A Gamma-Ray Spectrum Analysis Code for Determining Plutonium Isotopic Abundances, Vol. I, LLNL, Ca. UCRL-LR-103220, 1990.

Prepared by: Duc Vo & Andrey Bosko

Date: 2022-02-16

Method/measurement system (including software): HRGS / XMCG / FRAM-MGA**Application:** Measurement of Plutonium Isotopic Abundances in MOX (Low-Energy Analysis Option)

<p>Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)</p>
<p>The approach is based on the peak-ratio methods implemented in such isotopic analysis codes as FRAM v.6.1 (Fixed-Energy Response-Function with Multiple Efficiency [1]) and MGA v.10.0 (Multi Group Analysis Code for Plutonium [2]). The codes employ intrinsic energy, peak shape and detection efficiency calibrations to derive isotopic abundances from the efficiency corrected full energy peak ratios extracted from a single high-resolution gamma-ray spectrum. A typical measurement system includes a collimated HPGe detector of a planar type with energy resolution FWHM ≤ 750 eV @ 122 keV, a digital MCA, and an instrumentation laptop. A 1-2 mm cadmium or tin filter is used to effectively suppress the intense 59.5 keV gamma-rays of ^{241}Am. The measurements are normally performed at high count rates (5-20 kcps), to allow sufficient statistics of peak counts within practically feasible measurement time. For a standard IAEA XMCG setup involving a collimated HPGe Model BE1015 (Mirion Technologies) with crystal dimensions - $\varnothing 36 \times 15$ mm and collimator opening - $\varnothing 35 \times 15$ mm, the typical 30,000 counts in the reference 129 keV peak of ^{239}Pu from a lightly shielded material can be normally acquired at OCR=10 kcps within the measurement live time of LT=600 s. To allow good spectrum quality, the dead times are typically kept below 40%.</p>
<p>Factors affecting the method performance</p>
<p>High count rate applications, i.e. with DT > 40% (or OCR > 22,000 cps), may broaden the peak energy resolution, alter the peak shapes, and also lead to pulse pileup. This will affect the accuracy and precision of the measurement results.</p> <p>The method generally provides good results when measuring plutonium in a thin wall container (< 3 mm SS (or equivalent)), which does not significantly attenuate the intense X-ray region around 100 keV. Moderately thick and/or heavy absorber (> 10 mm SS (or equivalent)) will significantly suppress the X-ray peaks in the 100-keV region, which can effectively render the technique useless.</p> <p>The ITVs were derived assuming homogeneous samples. The material inhomogeneity, as well as heavy shielding, high ambient radiation background (including presence of fission product emissions, e.g., at nuclear power plants), presence of high-Z elements in material matrix with X-ray emissions close to the Pu X-rays (e.g., such as Th), would worsen the performance of the method.</p>
<p>Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)</p>
<p>Current evaluations are based on limited scope inspector vs. operator paired dataset consisting of ~50 field MOX measurements, obtained with FRAM 6.1 and parameter set "Pu_Plnr_060-230". The evaluations included MOX materials made of naturally enriched Uranium and high-burnup Plutonium in the range of the U/Pu ratios from 10 to 100.</p>

Method references

- [1] T.E. Sampson, T.A. Kelley, PC/FRAM: a Code for the Nondestructive Measurement of the Isotopic Composition of Actinides for Safeguards Applications, Appl. Radiat. Isot. Vol. 48, No. 10-12 (1997) 1543-1548.
- [2] R. Gunnink, MGA: A Gamma-Ray Spectrum Analysis Code for Determining Plutonium Isotopic Abundances, Vol. I, LLNL, Ca. UCRL-LR-103220, 1990.

Prepared by: A. Berlizov & V. Nizhnik (IAEA SGTVT)

Date: 2022-02-07

Method/measurement system (including software): HRGS / XMCG / FRAM**Application:** Measurement of Plutonium Isotopic Compositions (High-energy analysis)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The method determines plutonium isotopic composition by analyzing spectra acquired by a coaxial or large semi-planar HPGe detector with the energy resolution FWHM < 1 keV @ 122 keV and < 2 keV @ 1332 keV. The method employs the high-energy region (180-1001-keV) analysis of the isotopic analysis software code FRAM v.6.1 (Fixed-Energy Response-Function with Multiple Efficiency [1]). The method can be used to measure items of arbitrary size, shape and composition. It is applicable to any type of plutonium (aged, fresh, isotopic homogeneous/heterogeneous, wide range isotopic compositions) and is useful for in-situ measurements of bulk materials with net weight of a fraction of a gram up to several kg. A typical measurement system includes a collimated HPGe detector, an MCA, and an instrumentation laptop.</p> <p>The spectra are normally measured at 8K channels and with energy calibration of 0.125 keV/ch. Typical measurement time is 600-1800 s. A 1-2 mm cadmium or tin filter is used to suppress the 60-keV gamma rays of ²⁴¹Am. The method can analyze spectra of bare plutonium or plutonium that is shielded by up to 0.5 cm lead or 5 cm steel. For plutonium shielded by even thicker absorber up to 2.5 cm lead, the peaks below 300 keV may not be visible and the analysis should be modified to analyze the 300-1001-keV region.</p>
Factors affecting the method performance
<p>The random sum peaks of a source with significant Np-237 and measured at high count rate can interfere with the peak fitting of the 640-keV region. This will affect the accuracy of the measured area of the 642-keV peak of ²⁴⁰Pu. The inaccurate ²⁴⁰Pu fraction would affect the accuracy of all other isotopes.</p> <p>The ITVs were derived assuming homogeneous samples. The material inhomogeneity, as well as heavy shielding, high ambient radiation background (including presence of fission product emissions, e.g., at nuclear power plants) would worsen the performance of the method.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>The ITV values are from the analysis of lab data using FRAM in the high-energy mode. The spectra were measured with counting times from 900-7200 s and the ITV results were normalized to a counting time of 600 s. The counting rates for the data varied from about several kHz up to 50 kHz. In addition to 1-2mm cadmium filter, up to 1.6 mm lead were also used for some data sets. Sixty-eight data sets with 1075 spectra were used in the analysis.</p>
Method references
<p>[1] D. Vo, "Improvements of FRAM version 6.1," International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 19-30.</p>

Prepared by: Duc Vo
Date: 2022-02-16

Method/measurement system (including software): HRGS / XMCG / FRAM**Application:** Measurement of Plutonium Isotopic Abundances in MOX (High-Energy Analysis Option)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>Typical measurement system includes a portable gamma-ray spectrometer with electrically cooled coaxial HPGe crystal and a digital MCA. The spectrometer's energy resolution is $\text{FWHM} \leq 1.5 \text{ eV @ } 122 \text{ keV}$. The IAEA instrument (ECGS - Electrically Cooled Gamma Spectrometer) is represented by the ORTEC Micro-UF6™ system [1], a variant of the standard ORTEC Micro-trans-SPEC portable gamma-spectrometer. The system includes a coaxial HPGe crystal with $\text{Ø}50 \text{ mm} \times 30 \text{ mm}$ nominal dimensions, furnished with the 8 mm thick side shielding and a $\text{Ø}50 \text{ mm} \times 8 \text{ mm}$ front-end tungsten collimator. For materials containing high-burnup Pu, a 1-2 mm Cd or Sn filter is used to suppress the intense 59.5 keV gamma-rays of ^{241}Am.</p> <p>The spectrometer is set up to acquire an 8k-channel spectrum with the 0.125 keV/chn gain. Measurements are performed at $\leq 20 \text{ kcps}$ count rates and dead times $\leq 30\%$, to allow good statistics of counts and sufficient spectrum quality within practically achievable measurement times ($\text{LT} \leq 1000 \text{ s}$). The spectrum evaluation approach is based on the peak-ratio method implemented in FRAM 6.1 (Fixed-Energy Response-Function with Multiple Efficiency [2]) isotopic analysis code. The code employs the intrinsic energy, peak shape and detection efficiency calibrations, to derive isotopic abundances from the efficiency corrected full energy peak ratios, which are extracted from a single high-resolution gamma-ray spectrum.</p>
Factors affecting the method performance
<p>The ITVs were derived assuming isotopically homogeneous samples. The material inhomogeneity, heavy shielding, and high ambient radiation background (including presence of fission product emissions) would worsen performance of the method. The Detective (family) detectors use a special shaping model, about 3 times wider than normal, to process the pulses from the detector crystal. As a result, the dead time can become significantly large for moderate count rate (50% dead time for 20 kHz input rate).</p> <p>The normally large dead time even at low or moderate input rate can greatly increase the sum peaks, broaden the peaks, alter the peak shape, and increase the biases of the results. If the energy resolution of the detector does not meet the required resolution then the bias is likely increased.</p>
Source of bottom-up value (only for prospective ITV not based on historical data)
<p>The ITVs were evaluated based on the top-down approach applied to an inspector vs. operator paired dataset consisting of ~ 25 field verification measurements of MOX fuel assemblies using ECGS. The gamma-ray spectra were acquired for 1000 s live time. They typically contained $\sim 5,000 \text{ cnts}$ in the 129 keV peak of ^{239}Pu and $\sim 700,000 \text{ cnts}$ in the 208 keV peak of ^{241}Pu. The measured MOX fuel was made of depleted uranium and high-burnup plutonium ($^{239}\text{Pu} \leq 55 \text{ wt\%}$, 2-3 years after chemical separation) with U/Pu ratio ≈ 10. The spectrum processing was performed with FRAM 6.1 using a modified version of the FRAM parameter set "det_coax_120_800_1". The modified parameter set included the interfering background peaks of Cs-137 (662 keV) and Ag-110m (658 keV) that impacted evaluations of the ^{240}Pu and ^{241}Am contents.</p>

Method references

- [1] ORTEC Micro-UF6™ Portable UF6 Cylinder Verification System, Software v.2.3 — User's Manual, ORTEC Part No. 932500, 177 p.
- [2] D. Vo, Improvements of FRAM v.6.1, Journal of Nuclear Material Management, 49 (3) (2021) 19-30.

Prepared by: A. Berlizov (IAEA SGTVT)

Date: 2022-02-08

Method/measurement system (including software): HRGS / XMCG / FRAM**Application:** Measurement of Plutonium Isotopic Compositions using the Ortec Detective Family (medium- and high-energy analysis)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The method determines plutonium isotopic composition by analyzing spectra acquired by a very large coaxial or the electrical cooled HPGe detector made by Ortec (Detective family) with the energy resolution FWHM < 1.5 keV @ 122 keV and < 2.3 keV @ 1332 keV. The method employs the mid- and high-energy regions (120-460-keV and 180-1001-keV) analysis of the isotopic analysis software code FRAM v.6.1 (Fixed-Energy Response-Function with Multiple Efficiency [1]). The method can be used to measure items of arbitrary size, shape and composition. It is applicable to any type of plutonium (aged, fresh, isotopic homogeneous/heterogeneous, wide range isotopic compositions) and is useful for in-situ measurements of bulk materials with net weight of a fraction of a gram up to several kg. A typical measurement system includes an instrumentation laptop together with the Detective/Transpec or an LN2 cooled HPGe detector connected to an MCA.

Depending on the detector system, the spectra are measured at either 8K channels with 0.125 keV/ch energy calibration (Transpec and LN2 cooled HPGE system), 8K channels with 0.366 keV/ch energy calibration (Detective), or 16K channels with 0.183 keV/ch energy calibration (Detective X). A 1-2 mm cadmium or tin filter is used to suppress the 60-keV gamma rays of ²⁴¹Am. The method can analyze spectra of bare plutonium or plutonium that is shielded by up to 0.5 cm lead or 5 cm steel. For plutonium shielded by even thicker absorber up to 2.5 cm lead, the peaks below 300 keV may not be visible and the analysis can be modified to analyze the 300-1001-keV region.

Factors affecting the method performance

The random sum peaks of a source with significant Np-237 and measured at high count rate can interfere with the peak fitting of the 640-keV region. This will affect the accuracy of the measured area of the 642-keV peak of ²⁴⁰Pu. The inaccurate ²⁴⁰Pu fraction would affect the accuracy of all other isotopes. The Detective (family) detectors use a special shaping model, about 3 times wider than normal, to process the pulses from the detector crystal. As a result, the dead time can become significantly large for moderate count rate (50% dead time for 20 kHz input rate). The normally large dead time even at low or moderate input rate can greatly increase the sum peaks, broaden the peaks, alter the peak shape, and increase the biases of the results. If the energy resolution of the detector does not meet the required resolution then the bias is likely increased.

Source of bottom-up value (only for prospective ITV not based on historical data)

The LANL spectra were lab data and measured with real counting times of 900 s and the ITV results were normalized to a live time of 600 s. The counting rates for the data varied from about 2kHz up to 20 kHz. In addition to 1-2mm cadmium filter, up to 8 mm steel were also used for some data sets. Twenty-eight data sets with 539 spectra were used in the analysis.

The JRC spectra were lab data measured using two sets of samples. Combinations of cadmium filters of up to 2 mm, steel filters of up to 16 mm, and a lead filter of 4 mm thickness were used (the lead filter was used without any other filter). There was a total of 1440 spectra, grouped into 40 categories for the ANOVA analysis. For the first set of samples, the count rate varied between about 2 kHz and 16 kHz, and spectra of 900 s true time were used for the analysis. For the second set, the count rate varied between about 0.2 kHz and 4kHz, and spectra of 7200 s true time were used. In this way, the total number of counts in the spectra was approximately between $1e6$ and $1e7$ counts for all samples. The full JRC dataset is available for download at <https://data.jrc.ec.europa.eu/dataset/d4df018b-7ad6-476c-bd6c-781c24bdc836>

Method references

- [1] D. Vo, "Improvements of FRAM version 6.1," International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 19-30.

Prepared by: Jozsef Zsigrai and Duc Vo

Date: 2022-02-17

Method/measurement system (including software): MRGS / XMCC-L / FRAM-PAT**Application:** Determination of ^{239}Pu and ^{240}Pu Abundances Using Medium Resolution Gamma-Ray Spectrometers

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

Utilization of the MRGS for Plutonium isotopic measurements is an emerging application area in nuclear safeguards. It has been promoted recently as a result of the multilateral effort on U/Pu Isotopic Analysis Inter-Comparison Exercise by MRGS [1], which was coordinated by the IAEA. Two technologies identified as representing viable spectrum analysis options were subsequently implemented in the isotopic analysis codes – FRAM 6.1 (Fixed-Energy Response-Function with Multiple Efficiency [2]) and PAT (Plutonium Attribute Test [3]). Both codes contributed to the current evaluations of the prospective (bottom -up) ITVs for CZT and LaBr_3 based gamma-ray spectrometers. Both codes are based on the peak ratio method with application of iterative intrinsic calibration approaches for energy, peak shape and detection efficiency.

For technique 3 category, the FRAM code determines the ^{239}Pu , ^{240}Pu , ^{241}Pu , and ^{241}Am abundances from the spectrum analysis, while isotopic correlations are used to calculate the abundances of minor Pu isotopes – ^{238}Pu and ^{242}Pu . For technique 4 category, the FRAM code at first determines the $(^{241}\text{Pu} + ^{241}\text{Am})/^{239}\text{Pu}$ ratio from spectrum analysis. It then calculates the $\text{Pu}240/^{239}\text{Pu}$ ratio using its correlation with the $(^{241}\text{Pu} + ^{241}\text{Am})/^{239}\text{Pu}$ ratio and then the $^{238}\text{Pu}/^{239}\text{Pu}$ ratio using its correlation with the just determined $\text{Pu}240/^{239}\text{Pu}$ ratio. Finally, it calculates the ^{242}Pu fraction using its correlations with other Pu isotopes, either measured or calculated from correlations.

To determine the ^{239}Pu and ^{240}Pu abundances, the PAT applies correlations of these isotopes vs. $^{239}\text{Pu}/(^{241}\text{Pu} + ^{241}\text{Am})$ ratio, which is derived from the fitting of the 230-470 keV spectral region.

Typical measurement systems applied by the IAEA are composed of a shielded CZT ($\geq 1.5 \text{ cm}^3$) or LaBr_3 ($\text{Ø}2'' \geq 0.5''$) detector, a digital MCA, and an instrumentation laptop. A 1-2 mm cadmium or tin filter is used to effectively suppress the intense 59.5 keV gamma-rays of ^{241}Am . Using these spectrometers at $\text{OCR}=10 \text{ kcps}$ and measurement live time $\text{LT}=600 \text{ s}$, one can normally acquire 2,000/20,000 counts (CZT) and 10,000/50,000 counts (LaBr_3) in the reference 413 keV peak of ^{239}Pu from a lightly shielded High/Low burnup Pu material. The quality of respective spectra will be sufficient to warrant the performance of the isotopic analysis by FRAM and PAT at the level of the stated ITVs.

Due to the large uncertainty estimates found in the bottom up approach these methods should be limited to plutonium attribute at this time.

Factors affecting the method performance

Material inhomogeneity, heavy shielding, high ambient radiation background, spectral interferences (e.g., with emissions of fission products). Time since the spent fuel is removed from the reactor and the Americium removed from the plutonium can significantly affect the results from the ratio method (technique 4 category).

Source of bottom-up value (only for prospective ITV not based on historical data)

The FRAM code evaluations are based on measurements with a variety of materials with known (certified) isotopic compositions under laboratory conditions. 299 CZT spectra in 9 sets and 530 LaBr₃ spectra in 21 sets were used in the analysis. The PAT code was evaluated against sets of spectra collected within the framework of the inter-comparison exercise [1]. The ITVs represent approximate average performance values obtained by the both codes.

Method references

- [1] A. Berlizov, M. Koskelo, B. McGinnis, J. Carbonaro, "The Inter-Comparison Exercise on Uranium and Plutonium Isotopic Analysis with Medium Resolution Gamma-Ray Spectrometers," International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 94-107.
- [2] D. Vo, "Improvements of FRAM version 6.1," International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 19-30. [3] R. Gunnink, A. Berlizov, PAT, a Program for Determining the Category of Plutonium Samples, International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, IAEA, February 2021, Journal of Nuclear Materials Management 2021 Volume XLIX No. 3: 84-93.

Prepared by: D. Vo (LANL) & A. Berlizov (IAEA SGTND)

Date: 2021-05-24

TABLE 7a – Uranium and Plutonium Mass by Gamma (NDA)

Method/measurement system (including software): HRGS/ISOCS**Application:** ^{235}U measurement of fresh UO_2 in MTR fuel Plates or Rods

<p>Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)</p>
<p>HRGS/ISOCS measurements can be performed by either liquid nitrogen cooled high resolution gamma-ray spectrometry systems (IMCG) or electrically cooled systems (ECGS) [1]. The collected high resolution gamma-ray energy spectra are analysed using computed photopeak detection efficiencies obtained from the In-Situ Object Calibration Software [2]. This allows to determine the activity (Bq) of radionuclide present in the measured item for a given item description/measurement geometry. For this application, ISOCS geometry models are based on available geometry templates describing a pre-set fixed geometry measurement. Mechanical support frames are usually used to assist inspectors in the field in reproducing the assumed measurement geometry. Description of the measured item is based on declared physical and chemical characteristics of the MTR fuel plates or rods.</p> <p>A measurement of ambient level of gamma-ray background radiation is performed first. This allows for the stripping the measured background spectrum from the item spectra. The ISOCS characterized measurement systems used for these measurements are typically Broad Energy (BeGe) detectors with relative efficiencies between 20 % and 50 %. Typical measurement times of 300-s for HEU items and 600-s for LEU items are used. Data acquisition is typically performed with gain setting corresponding to 0.075 keV per channel over a 16,382 channels spectrum. This allows the measurement of ^{234}U, ^{235}U and $^{238}\text{U}_{\text{eq}}$ photo-peaks up to 1,228 keV. Once a spectrum is collected, an automated spectral analysis, including peak search, peak area fitting, efficiency correction (from ISOCS), MGA-U [3] analysis and LACE steps, is performed using a predefined Analysis Sequence File (ASF). Using the specific activities of the key uranium (^{234}U, ^{235}U, $^{234\text{m}}\text{Pa}$) the activity quantification is reported in terms of mass of radionuclides. Isotopic mass ratios can also be derived (i.e. ^{235}U wt.%), although subject to the impact of potential $^{234\text{m}}\text{Pa}/^{238}\text{U}$ disequilibrium. Selection of the ISOCS efficiency calibration data is performed at the time of the execution of the ASF.</p> <p>Comparison of the ISOCS ^{235}U weight fractions with values derived by the MGA-U isotopic peak ratio analysis code is also performed to further evaluated the consistency of the ISOCS efficiency data/mass results, as well as visual confirmation of the consistency of the ^{235}U, $^{234\text{m}}\text{Pa}$ mass results using the Linear Activity Consistency Evaluation (LACE) plots [4].</p>
<p>Factors affecting the method performance</p>
<p>Uncertainty associated with the positioning of the measured item is limited by using mechanical holders. Measurements are usually performed in a relatively low background environment. Availability/reliability of information used in the development of the ISOCS model may impair on the measurement accuracy. This usually is the largest source of systematic error. Consistency checks between modelled and quantified results (e.g. quantified vs. modelled U masses), ISOCS based FRAM/MGA-U enrichments, and LACE provide means to gain confidence on the reliability of the ISOCS mass results.</p> <p>Quantification of ^{238}U via $^{234\text{m}}\text{Pa}$ peak intensities requires for the material to have reached equilibrium.</p>

Method references
<ol style="list-style-type: none"> 1. https://www.ortec-online.com/products/radiochemistry-health-physics-research-industrial/gamma-spectroscopy/all-in-one-spectrometers/micro-trans-spec 2. Venkataraman, R., Bronson, F., Atrashkevich, V., Young, B. M., Field, M. "Validation of in-situ object counting system (ISOCS) mathematical efficiency calibration software. Nucl. Instr. and Meth. 1999, A 422. 3. R. Gunnink, W.D. Ruhter, P. Miller, et al., "MGAU: A NEW ANALYSIS CODE FOR MEASURING ²³⁵U ENRICHMENTS IN ARBITRARY SAMPLES", UCRL-JC-114713, Livermore, 1994.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
<p>The reported ITV values originate from a very limited set of both field measurements and inspection results. These estimated performance values have been propagated to generate paired data with operator values assumed to carry only small systematic and random uncertainties as the manufactured of fuel pins usually follows very high level of quality controls/quality check analytical measurements.</p>

Prepared by: Ludovic Bourva

TABLE 7b – Uranium and Plutonium Mass by Neutron (NDA)

Method/measurement system (including software): AWCC**Application:** Measurement of LEU/HEU fresh fuel plates and assemblies

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The Active Well Coincidence Counter (AWCC) is a Safeguards verification NDA measurement system coupled with a multiplicity shift register (JSR-15). It can perform active neutron interrogation of LEU/HEU fresh fuel plates and assemblies to derive the fissile (^{235}U) content of the measured item(s). The counter is made of a main High-Density Polyethylene (HDPE) hollowed body. Two HDPE ‘plug’ can be inserted and secured at the bottom and top of the hollowed body to form a measurement cavity in the central part of the counter. Each plug has an inserts to hold one (α, n) neutron interrogation sources, such as $^{241}\text{AmLi}$. Sources with neutron output typically between 3×10^4 and 5×10^4 neutrons per second.</p> <p>Forty-two CO_2 quenched, 4 atmospheres pressurized, 20” long, 1” diameter ^3He proportional counters are embedded in the cavity walls, forming 2 concentric rings. Charge collection resulting from neutron interaction in the ^3He gas is performed through a wire anode and initial stage amplification of the collected signals are performed through AMPTEK A111 pre-amplifiers mounted within an electronics enclosure located above the counter main HDPE body. [1]</p> <p>For the measurement of fresh fuel plates and assemblies, the AWCC cavity is tilted into a horizontal position and specific HDPE inserts, embedding the two $^{241}\text{AmLi}$ sources, are used rather than the bottom and top plugs. Cadmium liners can be placed around the measurement cavity wall, although reducing the overall detection efficiency of the counter. That results into an harden neutron interrogation flux, thus improving the depth of penetration the neutrons, limiting self-shielding in denser samples like metallic fuel plates [2].</p> <p>^{235}U quantification is performed by interpreting the measured doubles neutron rate registered in the multiplicity shift register. Although randomly time correlated (α, n) neutron detections are vetoed by the shift register, their presence impacts the overall precision of the measurements. AWCC measurements are therefore only well suited for the measurement of ^{235}U masses above 50 grams. Because they mainly release neutrons below the ^{238}U fission energy threshold (≈ 1 MeV), the use of $^{241}\text{AmLi}$ interrogation sources limits the dependency of the measurement results to the enrichment of the measured item. Although empirical calibrations have been performed, calibration of the AWCC is also mainly performed by using benchmarked Monte Carlo calculations.</p> <p>Typical measurement times range from 600 to 1000 s.</p>
Factors affecting the method performance
<p>The positioning of the fuel plates/assemblies in the measurement insert/cavity needs to be reproducible and similar to the one used during calibration/modelling.</p> <p>Calibrations established for HEU material tend to show some biases when measuring LEU fuels. This is mainly due to changes in the material composition, density and to a lesser extend enrichment.</p>

Method references

1. H.O Menlove, "Description and Operation Manual for the Active Well Coincidence Counter", Los Alamos Scientific Laboratory, LA-7823-M, Manual ISPO-66, May 1979.
2. H.O Menlove, G. E. Bosler, "Application of the Active Well Coincidence Counter (AWCC) to High Enrichment Uranium Metal", Los Alamos National Laboratory, LA-8621-MS, ISPO-121, August 1981.

Prepared by: Ludovic Bourva

Method/measurement System (including software): ENMC**Application: Measurement of Pu in oxide and MOX**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The method for the Epithermal Neutron Multiplicity Counter is the same as described for the PSMC (q.v.). The ENMC [1,2] has 121 polyethylene-moderated 10-atmosphere ^3He tubes and 27 preamplifiers giving a neutron detection efficiency of 64%. The detection efficiency includes a contribution from epithermal neutrons because of the high pressure of the tubes. The high pressure and close separation of the tubes gives a short dieaway time of 19 μs, which improves the statistical precision.</p> <p>The measurement time is typically 600 seconds.</p> <p>The isotopic composition used in the analysis is obtained from mass spectrometry.</p>
Factors affecting the method performance
<p>The data is typically processed with point model equations with a multiplication bias correction for large multiplication items [3]. One assumption is that the (α,n) neutron energy is the same as the spontaneous fission neutron energy. This is a very good approximation for (α,n) neutrons from oxygen, but less good for other potential (α,n) emitters.</p> <p>The counting statistics on the Triples become poor at higher counting rates and for items with high "alpha" values ($\alpha > 10$).</p>
Method Reference
<ol style="list-style-type: none"> 1. J. E. Stewart, H. O. Menlove, D. Mayo, K. Kroncke, F.A. Duran "Epithermal Neutron Multiplicity Counter (ENMC): Faster Plutonium Assays by Factors up to 20" Nuclear Materials Management XXVII July 1999 and Los Alamos National Laboratory Report LA-UR-99-3791 (1999). 2. H. O. Menlove, C. D. Rael, K. E. Kroncke and K. J. DeAquero "Manual for the Epithermal Neutron Multiplicity Detector (ENMC) for Measurement of Impure (MOX) and Plutonium Samples" Los Alamos National Laboratory Report LA-14088 (2004) 3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement system (including software): FNCL**Application:** ^{235}U measurement in LWR fresh fuel assemblies with/without burnable poisons

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The verification of the fissile mass of ^{235}U in fresh fuel assemblies with the Fast Neutron Coincidence Collar (FNCL) is performed by measuring both passive and active neutron coincidence rates with the fuel assembly centered within the FNCL measurement cavity. Typically, 4 min and 11 min are used for the respective passive and active measurements. Neutron interrogation by epi-cadmium (Fast Mode) neutrons is provided by ^{241}Am-Lithium sources with total neutron output around 1×10^5 neutrons per second. The FNCL uses 12 fast neutron liquid scintillation detectors to perform both neutron and gamma-ray detection.</p> <p>A dedicated FNCL assay software supports the operation of the equipment and provides analytical results. Data acquisition is performed using 500 MHz digitizers and the net coincidence rates are derived within the FNCL software after several data filtering steps, including gamma-neutron pulse shape discrimination, pile up rejection and neutron energy filtering.</p> <p>The standard FNCL can be used to assay all commercial LWR fuel types, including PWR, BWR, VVERs. The FNCL measures the ^{235}U mass/cm of the fuel assembly. An active length verification (for example with an HM5) is necessary to verify the active length of the fuel assembly.</p>
Factors affecting the method performance
<p>The FNCL relies on the detection of fast neutrons above 0.5 MeV induced from epi-cadmium interrogation neutrons. The measured neutron coincidence rates from the FNCL therefore show little to no dependency on the neutron poison loading of the fresh fuel assemblies. No neutron poison correction based on operator declared data is therefore used in the FNCL evaluation of the assembly fissile mass, unlike assays conducted with the UNCL in thermal mode.</p>
Method references
<ol style="list-style-type: none"> 1. Beaumont J., et al. "A fast-neutron coincidence collar using liquid scintillators for fresh fuel verification" <i>Journal of Radioanalytical and Nuclear Chemistry</i> 314.2 (2017): 803-812. 2. Tomanin A., et al. "Inter-Comparison Exercise for the Safeguards Verification of PWR Fresh Fuel Assemblies using Fast and Thermal Neutron Coincidence Collars" <i>Proceedings of ESARDA 41st Annual Meeting Symposium on Safeguards and Nuclear Material Management, 14-16 May, 2019 Stresa (VB), Italy</i> pp. 378-388. 3. Beaumont J., et al. "Field testing of a fast-neutron coincidence collar for fresh uranium fuel verification." <i>Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment</i> 962 (2020): 163682.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
<p>The reported ITV values originate from a limited set of field measurements performed in conditions similar to those encountered in the field. These estimated performance values have been propagated to generate paired data with operator values assumed to carry only small systematic and random uncertainties as LWR assemblies are manufactured to very high level of quality controls.</p>

Prepared by: Ludovic Bourva

Method/measurement system (including software): GBAS/SBAS/HBAS**Application:** Plutonium hold-up in glove-box and process equipment

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
The GBAS (Glove Box Assay System), and iterative instrument developments such as SBAS (Super Glove Box Assay System) and HBAS (Hopper Glove Box Assay System) are neutron counting system used to evaluate the plutonium hold-up in glove boxes [1]. These systems use relatively large neutron moderator slabs with ^3He proportional counters to measure Doubles neutron count rates when positioned around the glove box. The full glove box sides are scanned using pairs or up to four slabs (mSBAS) [2] to record the Doubles neutron rate at several heights along the faces of glove boxes. A mathematical model based on solid angle calculations are used to infer the normalized rates, accounting for effects like neutron reflection from the floor.
Factors affecting the method performance
Plutonium hold-up determinations by Doubles neutron counting are challenging measurements, for the recorded count rates depend on the relative position of the source/detectors and the importance of the neutron scattering in any given measurement situation (glove box surrounding, internal structure of the glove box/hopper, position of the source material...). The technique therefore represents a semi-quantitative approach based on extensive characterization of the detector response for any specific measurements case.
Method references
<ol style="list-style-type: none"> 1. T. Asano, H. Kobayashi, S. Takahashi <i>et al.</i>, "Development of improved hold up measurement system at plutonium fuel production facility", Proceeding of the 38th INMM annual meeting, Phoenix AZ, 1997. 2. H. Tanaka, H. Nakamura, T. Hosoma, "Design of geometrical detector arrangement for extensive hold up measurement", Proceedings of the Symposium on International Safeguards, Vienna, Austria, 29 Oct. 02 Nov. 2001, IAEA-SM-367/14/08.
Source of bottom-up value (only for prospective ITV not based on historical data)
The reported ITV values originate mainly from operator measurement.

Prepared by: Ludovic Bourva

Method/measurement system (including software): HEPC**Application: Measurement of Large LEU/NU items**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
<p>The High Efficiency Passive Counters (HEPC) are neutron coincidence counters design to measure large items (from 1 to 15 kg) of uranium oxide powder canisters placed within the counter cavity. The units developed for measurements at fuel fabrication plants within the European Union (EU) rely on the detection of coincident neutrons emitted from spontaneous fission events in ^{238}U. The neutron measurement is coupled with a separate gamma-ray measurement to provide an evaluation of the measured mass of uranium compound. The counters are operated in standard neutron coincidence mode (4.5 μs pre-delay / 64 μs Gate Width) and achieved a ^{252}Cf neutron detection efficiency of the order of 53 %. The counters operate a large number of proportional ^3He tubes (148) pressurized to 4 atmospheres and arranged in three 'rings' on each of the 6 sides of the prism. Standard coincidence electronics (JSR-12/JSR-15) are used with the measurement system.</p> <p>Standard measurement times for the verifications are 1000-s, which should be consistent with measurement precision of the order of 1-%.</p> <p>Reports on the design, qualification and calibration of these counters can be found in the literatures [1, 2].</p>
Factors affecting the method performance
<p>As for any standard passive neutron counter the HEPC calibration accuracy is subject to measuring pure oxide material similar to the one used to establish the counter calibration. Dependence of the measurement to ^{235}U enrichment was minimized by lining the internal wall of the cavity with Cadmium to limit the effect of thermalized albedo neutrons into the measured sample. Measurement of items in a physical form other than the one used for calibration (i.e. pellet vs. powder) or enrichment close to the upper LEU limit (i.e. 20 wt.%) show systematic biases due to alternative neutron multiplication in the items.</p> <p>Variation in the sample fill height and central positioning within the counter cavity have also been reported to create small relative measurement biases of the order of 1 to 3 %.</p>
Method references
<ol style="list-style-type: none"> Peerani, P.; Jaime, R.; Looman, M.; Ravazzani, A.; "Implementation of a Verification Procedure for large LEU Samples based on Passive Neutron Assay"; Proceedings. 25th Annual Meeting. Symposium on Safeguards and Nuclear Materials Management (ESARDA). Peerani, P <i>et. al</i>; "Development of high-efficiency passive counters (HEPC) for the verification of large LEU samples"; Nuclear Instruments and Methods in Physics Research Section A Accelerators Spectrometers Detectors and Associated Equipment 601(3) 326-332.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
<p>The reported ITV values originate mainly from relatively small set of verification results performed mainly with LEU powder samples covering the ^{235}U enrichment range between 2.5 and 5 wt.%.</p>

Prepared by: Ludovic Bourva

Method/measurement System (including software): HLNC**Application: Measurement of Pu in plutonium oxide and MOX**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The $^{240}\text{Pu}_{\text{eff}}$* content of an item is determined from the total (Singles) and coincidence (Doubles) counting rates of the High-Level Neutron Coincidence counter (HLNC) [1], using a shift register to extract the correlated rate. The HLNC uses 18 4-atmosphere ^3He tubes in a polyethylene moderator to detect the neutrons. The neutron detection efficiency is ~17%. Two analysis methods are used in Passive Neutron Coincidence Counting (PNCC). 1. A passive calibration curve ("PCC") using only the Doubles rate and 2. A method ("known alpha") that combines both Singles and Doubles rates and uses a calculated ratio of (α,n) to spontaneous fission neutrons ("alpha") to correct for item self-multiplication. The data is typically collected and analyzed using the International Neutron Coincidence Counting code [2]. The isotopic composition is needed to convert from $^{240}\text{Pu}_{\text{eff}}$ to total Pu and to calculate "alpha" and is obtained from mass spectrometry. (The uncertainty contribution from the mass spectrometry results is negligible).</p> <p>The measurement time is 300 s. A good measurement of the background counting rates is important for the known alpha technique.</p> <p>These methods are used for many kinds of items that fit in the cavity (17.5 cm diameter and 41 cm tall).</p> <p>* The $^{240}\text{Pu}_{\text{eff}}$ of an item is the weighted sum of the even Pu isotopes.</p>
Factors affecting the method performance
<p>The passive calibration curve method is valid for items that are similar to those used to make the relevant calibration. The results for items with different neutron multiplication (for example, from different shapes or density) will be biased. This method is not very sensitive to small amounts of impurities, especially when the neutron multiplication is small.</p> <p>The known alpha method contains an intrinsic correction for neutron multiplication and so is not sensitive to the size and shape (density) of the item. The result is sensitive to the value used for the (α,n) rate, which is usually calculated from the isotopic composition and chemical composition of the sample assuming it is pure.</p> <p>A good quality control check is to compare the results from the two analysis methods and to understand the reason for the difference between them (if any).</p>
Method References
<ol style="list-style-type: none"> 1. H. O. Menlove and J. E. Swansen "A high performance Neutron Time Correlation Counter," Nuclear Technology 71 497-505 (1985). 2. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): INVS**Application: Measurement of Pu in small samples of plutonium oxide and MOX**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The Inventory Verification Sample counter (INVS) [1] uses the passive calibration curve method - the same as the PCC method in the HLNC (q.v.), but the masses of the measured items are small, around 1g for PuO₂ and 1-5 g for MOX. The detector uses 18 6-atmosphere, polyethylene moderated ³He detectors, giving a neutron detection efficiency of 43%. The cavity size is 4.9 cm in diameter and 15 cm tall. The calibration curve is made with well-characterized reference material. The conventional counting time is 600 s. (The most precise values were obtained when the measurement time was chosen to reach 0.2% statistical error on the Doubles counting rate and this varied from 2 to 30 hours [2].) The data is collected and analyzed using the International Neutron Coincidence Counting code [3]. The analysis uses the plutonium isotopic composition obtained from mass spectrometry.</p>
Factors affecting the method performance
<p>The best results are obtained by defining separate sample types (for example, PuO₂ powder, LWR-MOX powder, FBR-MOX powder and FBR-MOX pellets) and keeping sample sizes consistent. Quantitative corrections are made for sample-to-sample variations such as mass, density, "alpha" and Pu content (and also ²³⁵U enrichment if applicable) using the MCNP[®] code [4].</p> <p>("alpha" is the ratio of (α,n) to spontaneous fission neutron emission)</p>
Method references
<ol style="list-style-type: none"> 1. J.K. Sprinkle, H.O. Menlove, M.C. Miller and P.A. Russo "An Evaluation of the INVS Model IV Neutron Counter" Los Alamos National Laboratory Report LA-12496-MS 1993 2. H. Ottmar, A. Schubert, T. R. Wenz, H. O. Menlove, G. W. Eccleston, U. Blohm-Hieber and M. T. Swinhoe "A Consistent Calibration Approach for Neutron Coincidence Counting of Small PuO₂ and MOX Samples." Proc. 19th ESARDA Annual Meeting Montpellier, France May 1997 3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009) 4. See mcnp.lanl.gov
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): PCAS**Application: Measurement of plutonium in MOX**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>There are several implementations of the PFPF*-Canister Assay System (PCAS) measurement system, which are similar [1]. All use the passive coincidence counting method described under the HLNC detector (q.v.). The PCAS-3 has 16 polyethylene-moderated 4-atmosphere ^3He tubes with 8 preamplifiers, giving an efficiency of 13% and a dieaway time of 52 μs. PCAS are used for the measurement of canisters containing large masses of MOX (up to 12 kg of Pu) resulting in high counting rates. Deadtime corrections are important. The PCAS has a uniform detection efficiency profile over the height of the material in the canister.</p> <p>The isotopic composition used in the analysis is obtained from mass spectrometry.</p> <p>The data is collected in unattended mode [2] and analyzed with INCC [3].</p> <p>*PFPF Plutonium Fuel Production Facility in Tokai, Japan.</p>
Factors affecting the method performance
<p>The performance of the instruments is helped by the fact that the production material measured in the facility is very homogenous with little variation in isotopic composition.</p>
Method References
<ol style="list-style-type: none"> 1. H. O. Menlove, J. Baca, K. E. Kroncke, M. C. Miller S. Takahashi, S. Seki, S. Inose, and T. Yamamoto "PFPF Canister Counter for Foreign Plutonium (PCAS-3) Hardware Operations and Procedures Manual" Los Alamos National Laboratory Report LA-12485-M (1992) 2. D. G. Pelowitz "MIC- Multi Instrument Collect" Los Alamos National Laboratory Report LA-UR-001182 (2000) 3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): PNCL**Application: Measurement of Pu in LWR MOX Fuel Assemblies**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The verification of the plutonium mass of LWR MOX assemblies can be done using the passive analysis methods of the HLNC (q.v.) with a detector that looks like the UNCL (q.v.) [1]. The original UNCL-I was configurable for active measurements of both BWR and PWR fuel, but also had an additional detector side that could make 4-sided passive detector for passive measurement of MOX assemblies [2]. Other detectors have been specifically built for this application. A typical total measurement time of 10 minutes.</p> <p>The data is typically collected and analyzed using the International Neutron Coincidence Counting code [3] (or in unattended mode). The PNCL measures the Pu mass/cm of the fuel assembly. An active length verification (for example with an HM5) is necessary to verify the active length of the fuel assembly.</p>
Factors affecting the method performance
<p>The performance of the method is helped by the uniformity of the items- production MOX assemblies. The amount of Pu in a typical MOX assembly is large and leads to very high counting rates in the collar. This requires careful determination of deadtime correction coefficients.</p>
Method References
<ol style="list-style-type: none"> 1. H. O. Menlove "Passive/Active Coincidence Collar for Total Plutonium Measurement of MOX Fuel Assemblies" Los Alamos Laboratory Report LA-9288 1982 2. H. O. Menlove, J. E. Stewart, S. Z. Qiao, T.R. Wenz and G. P. D. Verrecchia "Neutron Collar Calibration and Evaluation for assay of LWR Fuel Assemblies Containing Burnable Neutron Absorbers" Los Alamos Laboratory Report LA-11965-MS 1990 3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): PSMC**Application: Measurement of Pu mass in oxide and MOX**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The methods described under HLNC (q.v.) have limitations for impure and inhomogeneous materials such as scrap and waste. The passive neutron multiplicity counting technique (PNMC) [1] expands the experimental information provided by passive coincidence counting to give three measured quantities: Singles, Doubles and Triples. This allows quantitative information on spontaneous fission rate ($^{240}\text{Pu}_{\text{eff}}^*$), neutron multiplication and (α, n) rate to be extracted from the measurement data. In this way systematic measurement uncertainties can be greatly reduced, thus making the plutonium assay by PNMC much more reliable and accurate, particularly for poorly characterized and impure materials with a large induced fission component.</p> <p>The method does not require reference materials for calibration, but does require high efficiency detectors. Even so, typical measurement times are longer than for passive coincidence counting to obtain good statistics on Triples (~600 s). One detector used for this method is the Plutonium Scrap Multiplicity Counter (PSMC) [2], which has an efficiency of 55% using 80 polyethylene-moderated, 4-atmosphere ^3He tubes and 19 preamplifiers. The dieaway time is 47 μs. The data is typically collected and analyzed using the International Neutron Coincidence Counting code [3].</p> <p>The isotopic composition used in the analysis is obtained from mass spectrometry.</p> <p>* The $^{240}\text{Pu}_{\text{eff}}$ of an item is the weighted sum of the even Pu isotopes.</p>
Factors affecting the method performance
<p>The data is typically processed with point model equations with a multiplication bias correction for large multiplication items. One assumption is that the (α, n) neutron energy is the same as the spontaneous fission neutron energy. This is a very good approximation for (α, n) neutrons from oxygen, but less good for other potential (α, n) emitters.</p> <p>The counting statistics on the Triples become poor at higher counting rates and for items with high "alpha" values ($\alpha > 10$).</p>
Method References
<ol style="list-style-type: none"> 1. N. Ensslin et al., "Application Guide to Neutron Multiplicity Counting", Los Alamos National Laboratory Report LA-13422-M (1998). 2. H. O. Menlove, J. Baca, M. S. Krick, K. E. Kroncke and D. G. Langner "Plutonium Scrap Multiplicity Counter Operation Manual" Los Alamos National Laboratory Report LA-12479-M (1992) 3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): UNCL thermal mode**Application: Measurement of ^{235}U in LWR Fuel Assemblies with/without burnable poisons**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)
<p>The verification of the mass of ^{235}U in fresh fuel assemblies with the UNCL [1] is performed by measuring both passive neutron and active neutron coincidence rates with the fuel centered in the UNCL cavity. The active measurement uses a single AmLi with source strength typically 4×10^4 n/s to induce fissions in the fuel. The instrument has with 18 4-atmosphere polyethylene moderated ^3He detectors Two UNCL-II detector sizes exist, one for PWR fuel and one for the smaller BWR fuel, although an earlier design, UNCL-I, could be configured to measure both types. Because of the difficulty of making individual detector calibrations (the lack of availability of partially made fuel assemblies) the calibration of these detectors is made as a family – all detectors are related back to one initial collar calibration for PWR and one for BWR fuel assemblies. A typical total measurement time is 10 minutes. The data is usually collected and analyzed using the International Neutron Coincidence Counting code [2].</p> <p>The UNCL measures the ^{235}U mass/cm of the fuel assembly. An active length verification (for example with an HM5) is necessary to verify the active length of the fuel assembly.</p>
Factors affecting the method performance
<p>The measurement in thermal mode is affected by the amount of Gd, or other burnable poison in the fresh fuel assembly. The correction for Gd is significant for large Gd loadings (20% for 12 rods at 5.2% Gd_2O_3 in a PWR assembly) and relies on the operator's declaration. Variability of BWR fuel designs (non-symmetrical pin lattice, variable pin types and enrichments) makes the use of a single mass calibration and neutron poison correction more susceptible to biases. (The effect of Gd is much less when collars are used in fast mode (q.v.). The poison amount can be verified with a measurement in fast mode [1], although this can be prohibitive in terms of effort and measurement time.)</p> <p>The results are biased if the fuel assembly is not centered in the detector cavity.</p>
Method References
<ol style="list-style-type: none"> 1. H. O. Menlove, J. E. Stewart, S. Z. Qiao, T.R. Wenz and G. P. D. Verrecchia "Neutron Collar Calibration and Evaluation for assay of LWR Fuel Assemblies Containing Burnable Neutron Absorbers" Los Alamos Laboratory Report LA-11965-MS 1990 2. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Martyn Swinhoe

Method/measurement System (including software): UNCL fast mode**Application: Measurement of ^{235}U in LWR Fuel Assemblies with/without burnable poisons**

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time etc.)

The verification of the mass of ^{235}U in fresh fuel assemblies with the UNCL [1] is performed by measuring both passive neutron and active neutron coincidence rates with the fuel centered in the UNCL cavity (similar to the UNCL thermal mode q.v.). The active measurement uses a single AmLi with source strength typically 4×10^4 n/s to induce fissions in the fuel. In fast mode a cadmium liner is used to prevent the return of thermal neutrons into the fuel assembly and so the interrogation is done with epi-cadmium neutrons which significantly reduces the effect of burnable poisons on the results.

Fast mode measurements were originally foreseen on the origin collar designs, using removable cadmium liners. Two UNCL-II detector sizes exist, one for PWR fuel and one for the smaller BWR fuel, although an earlier design UNCL-I could be configured to measure both types. However, the counting time required to obtain adequate statistical precision with these detectors in fast mode was at least one hour, making inspection use difficult.

A new design of fast interrogation collars (EFCP/EFCB) [2] with 33 polyethylene moderated 10 atmosphere ^3He detectors for the PWR type and 25 ^3He detectors for the BWR type and a permanently installed cadmium liner allow a total measurement time of 20 minutes.

The data is typically collected and analyzed using the International Neutron Coincidence Counting code [3]. The UNCL measures the ^{235}U mass/cm of the fuel assembly. An active length verification (for example with an HM5) is necessary to verify the active length of the fuel assembly.

Factors affecting the method performance

The correction for Gd relies on the operator's declaration. However, the effect of burnable neutron poison in 17 x 17 PWR fuel for 12 Gd rods at 5% wt Gd_2O_3 is significantly reduced in the fast mode (3.2% compared to 20% for the UNCL in thermal mode). For 9x9 BWR fuel the effect of 6 Gd rods at 5 wt.% Gd_2O_3 is 3.8% in fast mode, compared to 33% in thermal mode. Nevertheless, K_3 corrections should be applied within their calibration range and may be impacted by specific fuel designs.

Method References

1. H. O. Menlove, J. E. Stewart, S. Z. Qiao, T.R. Wenz and G. P. D. Verrecchia "Neutron Collar Calibration and Evaluation for assay of LWR Fuel Assemblies Containing Burnable Neutron Absorbers" Los Alamos Laboratory Report LA-11965-MS 1990
2. L. G. Evans, M. T. Swinhoe, H. O. Menlove, P. Schwalbach, P. De Baere and M. C. Browne "A new fast collar for safeguards inspection measurements of fresh low enriched uranium fuel assemblies containing burnable poison rods" Nuclear Instruments and Methods in Physics Research A 729 (2013) 740-746.
3. W. Geist, J. Longo, M. Krick and W. Harker "INCC Software Users Manual" Los Alamos Report LA-UR-01-6761 (2009)
4. M. T. Swinhoe, H. O. Menlove, P. De Baere, D. Lodi, P. Schwalbach, C. D. Rael, M. Root, A. Tomanin and A. Favalli "A new generation of uranium coincidence fast neutron collars for assay of LWR fresh fuel assemblies" Nuclear Instruments and Methods in Physics Research, A 1009 (2021) 165453

Source of bottom-up value (only for prospective ITV not based on historical data)

The EFCP/EFCB uncertainty estimates come from a limited number of measurements described in a paper [4] describing initial field tests of the BWR and PWR versions of the Euratom fast collar.

Prepared by: Martyn Swinhoe

Method/measurement system (including software): [WDAS](#)**Application:** [Measurement of 200-l/55-Gal Pu Solid Waste Drums](#)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The WDAS (Waste Drum Assay System) measurement systems represent a category of large Passive Neutron Coincidence Counters applied to the quantification of plutonium in solid waste contained in 200-l/55-Gal drums [1]. The passive detection of fission events (either spontaneous or induced) is performed in these counters using a relatively large number (>100) of ^3He proportional neutron counters, usually pressurized to 4 atmospheres. Neutron detection efficiency varies depending on the actual design but values between 18 % and 35 % are common [2, 3, 4]. Signals generated from local pre-amplifiers connected to banks of several ^3He tubes are fed into either shift register or neutron multiplicity electronics to generate coincident or multiplicity count rates (Singles, Double, Triples). Empirical calibration of the system is performed by deriving a Weighted Volume Average (VWA) response of the counter to ^{252}Cf or Pu spontaneous neutrons emitted in air. This is done by measuring a known neutron source at several vertical/radial positions within the volume of the waste container. Derivation of the waste matrix corrections to be applied for different types of waste material is performed by repeating this counter response characterization for a number of waste measurement scenarios (e.g. combustible, metallic, plastics, earth, concrete...). Derivation of these correction effects for 'unknown' waste matrix measurement is usually performed by an 'Add-a-Source' (AAS) interrogation measurement [2, 5]. This entails evaluating the decay corrected counter response of a reference ^{252}Cf source exposed in the vicinity of the waste drum, and comparing this measurement to a reference Reals rate established for an empty drum. Characterizing this AAS perturbation for an equivalent number of waste measurement scenarios allows to establish an AAS matrix calibration. In the case of a waste item with unknown matrix absorption/moderation properties, the measured AAS perturbation can be evaluated and interpreted using the AAS matrix calibration to derive the waste matrix correction to be applied to the passive Doubles rate.

Quantification of the $^{240}\text{Pu}_{\text{Eff}}$ mass present in the item has to be complemented by isotopic information to derive the total plutonium content. These counters can measure from a few milligram quantities up to several 100's grams of plutonium in 200-l drums. Measurement times are usually between 20 and 30 min per assay, including a 3 to 5 min AAS measurement.

Factors affecting the method performance

As for any waste assay system, the mass calibration used to interpret the measured rates is based on a number of assumptions (i.e. Single efficiency, source uniformity, matrix homogeneity...). Real waste items are bound to break some of these assumptions and thus generating biases. AAS or other matrix correction factors introduced in WDAS system correct the raw measured rates, but the accuracy of these corrections can also be affected by effect such as matrix heterogeneity coupled with source non-uniformity.

Method references
<ol style="list-style-type: none"> 1. Menlove, H O, Baca, J; Harker, W; Kroncke, K E; Miller, M C; Takahashi, S; Kobayashi, H; Seki, S; Matsuyama, K; Kobayashi, S; “WDAS operation manual including the add-a-source function”, Los Alamos National Laboratory, Report LA-12292-M, April 1992. 2. Menlove, H O, et al.; “HENC Performance evaluation and Plutonium Calibration”; Los Alamos National Laboratory, Report LA-13362-MS, October 1997. 3. Menlove, H O, Beddingfield, D. H; Pickrell, M M; Davidson, D R; Mc Elroy, R D; Brochu, D B; “The Design of a High-efficiency neutron counter for waste drums to provide optimized sensitivity for plutonium assay”, Proceedings for the Nondestructive Assay and Nondestructive Examination Waste Characterization Conference, Salt Lake City, January 14-16, 1997. 4. Whitehouse, K R; Rackham, J W, Sharpe, R J and Wilson M; “TRU-D: Waste Drum Assay System – The use of passive neutron technologies to accurately categorize between PCM/TRU and Low-Level Waste”; Proceedings of the 25th Annual ESARDA symposium, Stockholm, 13-15 May 2003. 5. Mayo, D R, Menlove, H O, Pecos, J M; “Nondestructive Assay Tests of High-Efficiency Neutron Counter (HENC) for Waste Assay and Possible Diversion Scenario” Los Alamos National Laboratory, Report LA-13418-MS, May 1998.
Source of bottom-up value (only for prospective ITV not based on historical data)
n/a

Prepared by: Ludovic Bourva

Appendix VI: ITV Glossary

In preparation

Appendix VII: List of Participants

ARGENTINA		
ACOSTA, Elizabeth	egacosta@cnea.gov.ar	National Atomic Energy Commission (CNEA)
ACOSTA, Gabriela	gacosta@arn.gob.ar	National Atomic Energy Commission (CNEA)
BAVIO, Marta	mbavio@cnea.gov.ar	National Atomic Energy Commission (CNEA)
FERNANDEZ, Mauricio	mafernan@cnea.gov.ar	National Atomic Energy Commission (CNEA)
GAUTIER, Eduardo	gautier@cnea.gov.ar	National Atomic Energy Commission (CNEA)
PARDO, Leonardo	lpardo@arn.gob.ar	National Atomic Energy Commission (CNEA)
RAMELLA, Jose, Luis	ramella@cnea.gov.ar	National Atomic Energy Commission (CNEA)
ZUBILLAGA, Erica	ericazubillaga@cnea.gov.ar	National Atomic Energy Commission (CNEA)
BRAZIL		
CRISTIANO, Bárbara	barbara.cristiano@cnen.gov.br	National Nuclear Energy Commission (CNEN)
DIAS, Fabio	fdias@abacc.org.br	Brazilian–Argentine Agency for Accounting and Control of Nuclear Materials (ABACC)
DUTRA, Marco	dutra@cnen.gov.br	National Nuclear Energy Commission (CNEN)
FERNANDEZ MORENO, Sonia	sfmoreno@abacc.org.br	Brazilian–Argentine Agency for Accounting and Control of Nuclear Materials (ABACC)
GRUND, Marcos	marcosg@cnen.gov.br	Laboratório de Salvaguardas (LASAL-CNEN)
MOREIRA, Marcos	marcos@abacc.org.br	Brazilian–Argentine Agency for Accounting and Control of Nuclear Materials (ABACC)
PALHARES, Lilia	lpalhares@cnen.gov.br	National Nuclear Energy Commission (CNEN)
PEREIRA, Olivio	oliviojr@ipen.br	Instituto de Pesquisas Energéticas e Nucleares (IPEN-CTMSP)
RENHA, Geraldo	grenha@cnen.gov.br	National Nuclear Energy Commission (CNEN)
SILVA, José	wanderley.silva@cnen.gov.br	National Nuclear Energy Commission (CNEN)
VAZ DE ARAUJO, Ana Maria	araujo@abacc.org.br	Brazilian–Argentine Agency for Accounting and Control of Nuclear Materials (ABACC)
CANADA		
COCHRANE, Chris	chris.cochrane@canada.ca	Canadian Nuclear Safety Commission
HEPPELL-MASYS, Kathleen	Kathleen.heppell-masys@canada.ca	Canadian Nuclear Safety Commission (CNSC)
JOVANOVIC, Slobodan	slobodan.iovanovic@cnscccsn.gc.ca	Canadian Nuclear Safety Commission (CNSC)
MESTER, Zoltan	zoltan.mester@nrc-cnrc.gc.ca	National Research Council Canada (NRC)
ST-AMANT, Nedereh	nadereh.st-amant@canada.ca	Canadian Nuclear Safety Commission (CNSC)
CHINA		
CHENG, Yimei	chengyimei@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
GAO, Xuemei	gaoxuemei74@163.com	State Nuclear Security Technology Center
HE, Lixia	helixia@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
KANG, Haiying	hykang@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
LI, Chun	lichun5678@163.com	State Nuclear Security Technology Center
LI, Dingming	dingminglee@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
LI, Lili	13641368967@139.com	China Institute of Atomic Energy (CIAEA)
LIU, Guorong	liugr@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
LIU, Hongbin	hb660316@126.com	CAEA/CIAE
LV, Xuesheng	xslv@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
WANG, Chen	13717760841@163.com	China Institute of Atomic Energy (CIAEA)
YING, Zhecong	beareye@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)
ZHOU, Hao	zhouhao_ciae@126.com	China Institute of Atomic Energy (CIAEA)
ZHU, Haiqiao	grace@ciae.ac.cn	China Institute of Atomic Energy (CIAEA)

EUROPEAN COMMISSION		
ANCIUS , Darius	darius.ancius@ec.europa.eu	DG ENER
AREGBE , Yetunde	yetunde.aregbe@ec.europa.eu	JRC/Geel
COULAND , Marc	marc.couland@ec.europa.eu	DG ENER
JAKOPIC , Rozle	rozle.jakopic@ec.europa.eu	JRC/Geel
LODI , Dario	dario.lodi@ec.europa.eu	DG ENER
MOREL , Sylvain	sylvain.morel@ec.europa.eu	JRC/ Karlsruhe
PALAJOVA , Zdenka	zdenka.palajova@ec.europa.eu	JRC//Brux
PEDERSEN , Bent	bent.pedersen@ec.europa.eu	JRC/Ispra
TAGZIRIA , Hamid	hamid.tagziria@ec.europa.eu	JRC/Ispra
TOMA , Gabriel	gabriel.toma@ec.europa.eu ;	JRC/ Karlsruhe
TOMA , Magdalena	magdalena.toma@ec.europa.eu	JRC/ Karlsruhe
TOMANIN , Alice	alice.tomanin@ec.europa.eu	DG ENER
VACCARO , Stephano	stefano.vaccaro@ec.europa.eu	JRC/ Karlsruhe
ZSIGRAI , Jozsef	jozsef.zsigrai@ec.europa.eu	JRC/ Karlsruhe
ZULEGER , Evelyn	evelyn.zuleger@ec.europa.eu	JRC/Karlsruhe
FRANCE		
BARON , David	david.baron@orano.group	Orano-MELOX
BRUEL , Vincent	vincent.brueel@orano.group	Orano
CROZET , Marielle	marielle.crozet@cea.fr	French Atomic Energy Commission (CEA)
ESBELIN , Eric	eric.esbelin@cea.fr	French Atomic Energy Commission (CEA)
FUNK , Pierre	pierre.funk@irsn.fr	Institute for Radiological Protection and Nuclear Safety (IRSN)
ISNARD , H�el�ene	helene.isnard@cea.fr	CEA Lanie
JANIN , Vincent	vincent.janin@orano.group	Orano
MIALLE , S�ebastien	sebastien.mialle@cea.fr	CEA Lanie
NARDOUX , Pascal	pascal.nardoux@orano.group	Orano Cycle
PICART , Sebastien	sebastien.picart@cea.fr	CEA Marcoule
RIVIER , Cedric	cedric.rivier@cea.fr	French Atomic Energy Commission (CEA)
ROUDIL , Danielle	danielle.roudil@cea.fr	French Atomic Energy Commission (CEA)
TISON , Eric	eric.tison@orano.group	Orano
WEBER , Anne-Laure	anne-laure.weber@irsn.fr	Institute for Radiological Protection and Nuclear Safety (IRSN)
GERMANY		
KRIEGER , Thomas	krieger@fz-juelich.de	FZ J�ulich
NEUMEIER , Stefan	s.neumeier@fz-juelich.de	FZ J�ulich
NIEMEYER , Irmgard	i.niemeyer@fz-juelich.de	FZ J�ulich
ROTH , Markus	markus.roth@physik.tu-darmstadt.de	TU Darmstadt
WEINBERG , Dagmar	dagmar.weinberg@bmwi.bund.de	Bundesministeriums f�ur Wirtschaft und Energie
JAPAN		
HARA , Shinji	shinji.hara@jmcc.or.jp	Nuclear Material Control Center (NMCC)
KABUKI , Toshihide	toshihide_kabuki@nsr.go.jp	Japan Safeguards Office (JSGO)
KUMAKURA , Shinichi,	kumakura@jmcc.or.jp	Nuclear Material Control Center (NMCC)
SUZUKI , Minoru	minoru_suzuki@nsr.go.jp	Japan Safeguards Office (JSGO)
WATANABE , Satoshi	swatanabe@jmcc.or.jp	Nuclear Material Control Center (NMCC)
NETHERLANDS		
KAMP , Bertwin	bertwin.kamp@ureenco.com	Ureenco
KLAASSEN , Frodo	klaassen@nrg.eu	Nuclear Research and Consultancy Group (NRG)
VAN, WIJNKOOP , Maarten	maarten.vanwijkoop@ureenco.com	Ureenco
VELDHOF , Roy	roy.veldhof@ureenco.com	Ureenco

ROK		
KIM, Hyun, Young	hykim33@kinac.re.kr	Korea Institute of Nuclear Non-proliferation and Control (KINAC)
LEE, Haneol	haneol@kinac.re.kr	Korea Institute of Nuclear Non-proliferation and Control (KINAC)
RUSSIA		
DAVYDOV, Ivan, L.	ildavydov@rosatom.ru	Rosatom
USA		
BRISSON, Mike	mike.brisson@srnl.doe.gov	Savannah River National Laboratory (SRNL)
CARBONARO, Joe	carbonar@bnl.gov	Brookhaven National Laboratory (BNL)
DOUGAN, Arden	arden.dougan@nnsa.doe.gov	NNSA/DOE
GARNER, James	garnerjr@ornl.gov	Oak Ridge National Laboratory (ORNL)
HOLLAND, Mike	michael.holland@nnsa.doe.gov ; hollandmk@icloud.com	NNSA/DOE
KOSKELO, Markku	mkoskelo@aquilagroup.com	Aquila Group
MASON, Peter	peter.mason@nnsa.doe.gov	NNSA/NBL
MCELROY, Robert	mcelroyrd@ornl.gov	Oak Ridge National Laboratory (ORNL)
MCGINNIS, Brent	brentmcginnis@insolves.com	Insolves
MORALES-ARTEAGA, Maria	maria.morales-arteaga@srnl.doe.gov	Savannah River National Laboratory (SRNL)
SANDERS, Jeff	jeff.sanders@inl.gov	Idaho National Laboratory (INL)
SWINHOE, Martyn	swinhoe@lanl.gov	Los Alamos National Laboratory (LANL)
TACKENTIEN, Josh	tackentien@bnl.gov	Brookhaven National Laboratory (BNL)
TICKNOR, Brian	ticknorbw@ornl.gov	Oak Ridge National Laboratory (ORNL)
VENKATARAMAN, Ram	venkataramar@ornl.gov	Oak Ridge National Laboratory (ORNL)
VO, Duc	ducvo@lanl.gov	Los Alamos National Laboratory (LANL)
WATTERS, Bob	robert.watters@nnsa.doe.gov	NNSA/NBL
XU, Ning	ning.xu@nnsa.doe.gov	NNSA/SGtech
IAEA		
AMARAGGI, David	d.amaraggi@iaea.org	KRZYSZTOSZEK, Kamil k.krzyzstoszek@iaea.org
ANICHENKO, Alexey	a.anichenko@iaea.org	LEBRUN Alain a.lebrun@iaea.org
BEAUMONT, Jonathan	j.beaumont@iaea.org	MAYOROV, Mikhail m.mayorov@iaea.org
BERLIZOV, Andriy	a.berlizov@iaea.org	MICHALAK, Sarah s.michalak@iaea.org
BEVILL, Aaron	a.bevill@iaea.org	NAKAZAWA, Dante d.nakazawa@iaea.org
BINNER, Robert	r.binner@iaea.org	NIZHNIK, Vladimir, v.nizhnik@iaea.org
BONNER, Elisa	e.bonner@iaea.org	NORMAN, Claude c.norman@iaea.org
BOSKO, Andrey	a.bosko@iaea.org	PENKIN, Maxim m.penkin@iaea.org
BOURVA, Ludovic	l.c.bourva@iaea.org	PORTAIX, Chris, c.portaix@iaea.org
BULYHA, Serguei	s.bulyha@iaea.org	REPINC, Urska u.repinc@iaea.org
CHEN, Suzhen	s.chen@iaea.org	RICHET, Sebastien s.richet@iaea.org
DEJEANT, Adrien	a.dejeant@iaea.org	SANTI, Peter p.santi@iaea.org
DUHAMEL, George	g.duhamel@iaea.org	SCHACHINGER, Andreas, a.schachinger@iaea.org
FRANCIS, Stephen	s.francis@iaea.org	SCHOENFELD, Julia j.schoenfeld@iaea.org
HAMABE, Kenichi	k.hamabe@iaea.org	SUMI, Mika m.sumi@iaea.org
HENDERSON, Kevin	k.henderson@iaea.org	WUESTER, Jan j.wuester@iaea.org
KIM, Gwang Sub	g.kim@iaea.org	ZHAO, Ke k.zhao@iaea.org
KOHL, Markus	m.kohl@iaea.org	