# Report on the Workshop on UNCERTAINTIES IN NUCLEAR MEASUREMENTS

Organised by the

**ESARDA Working Group on Standards and Techniques for Destructive Analysis (WG DA)**

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## *Abstract*

The ESARDA Working Group on Standards and Techniques for Destructive Analysis (WG DA), in close collaboration with the International Atomic Energy Agency (IAEA), organised a dedicated workshop on 'Uncertainties in Nuclear Measurements'. The workshop was held in conjunction with the annual working group meeting at the IAEA Safeguards Analytical Services (IAEA-SGAS) Seibersdorf Laboratories (SAL), Austria, on 8-9 November 2011. The focus of the workshop was to exchange concepts and methods of measurement uncertainty estimation among reference measurement institutes, safeguards laboratories, nuclear and environmental material analysts and, in particular, operators on estimation of measurement uncertainty in nuclear measurements. Participation was open to ESARDA WG DA members and to a limited number of invited participants from expert and research institutes. Forty-eight representatives from the main European and international nuclear safeguards organisations, nuclear measurement laboratories, nuclear industry and experts from environmental sciences institutes, participated in this workshop. Fundamental metrological concepts for the estimation of uncertainty in nuclear measurements were presented by Roger Wellum, retired from Institute for Reference Materials and Measurements (IRMM) in the first plenary lecture. The second plenary lecture was given by Claude Norman from the IAEA on measurement uncertainty in material balance verification. The plenary lectures were followed by three sessions, the first on nuclear material analysis for accountancy purposes, the second on nuclear material analysis for non-accountancy purposes, and the third session was dedicated to Environmental Swipe Sample Analysis. The findings and points of discussion from these sessions were further discussed in a working group using the 'World-Café' approach around three selected topics, ensuring that all participants could benefit from the 'collective intelligence'. This report is a summary of the points of discussion raised during the sessions and in the working group, with main emphasis on the recommendations for the topics of approaches to uncertainty, sources of uncertainty, and knowledge of uncertainty. As in previous workshops organised by the ESARDA WGDA, all participants recognised the need and the benefit of intensifying cooperation between the nuclear safeguards and nuclear forensics communities, nuclear industry and environmental sciences institutes.

**Keywords:** measurement uncertainty, International Target Values (ITV2010), nuclear material analysis, material accountancy, nuclear safeguards, environmental sampling

## 1. Introduction

ESARDA support the international initiative on a holistic safety, security and safeguards ("3S") concept for nuclear energy, launched with the Nuclear Safety and Security Group (NSSG) at the G8 summit in 2008. The ESARDA WG DA seeks to emphasise the technical convergence of nuclear safeguards and nuclear security. One aspect to meet this objective is to strengthen the exchange beyond the safeguards community on dedicated technical topics relevant to all three fields using the WG DA as a platform. Previous workshops of the WG DA were dedicated to 'Measurements of minor isotopes in uranium in bulk and particle samples' [1], to 'Measurements of impurities in uranium' [2] and to the Direct Analysis of Solid Samples Using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) [3].

Verification of the non-diversion of fissile material from its intended and declared use is the primary goal of nuclear safeguards. A reliable nuclear material accountancy system has to be established by the plant operator, whilst a reliable and fully independent system of verification is the responsibility of the safeguards authority. Accountancy and control of nuclear material require analytical measurements that "shall either conform to the latest international standards or be equivalent in quality to such standards" according to IAEA INFCIRC/153. This means that operators and laboratories performing sample analysis for accountancy and verification purposes have to demonstrate that they deliver reliable and traceable measurement results that are "fit for intended purpose" and within the required measurement uncertainties of the International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials (ITV) [4]. The ESARDA WG DA is promoting use of the International Target Values, which are uncertainties to be considered in judging the reliability of analytical techniques applied to industrial nuclear and fissile material subject to safeguards verification. They are uncertainties considered to be achievable under routine measurement conditions. Analytical laboratories are increasingly adopting the Guide to the expression of Uncertainties in Measurements (GUM) and are striving for accreditation, mainly according to the ISO/IEC guide 17025 [5, 6]. Therefore it was a perfect timing to use the momentum of the release of the ITV2010, which includes now a chapter on GUM and the Use of ITV by Measurement Laboratories, to exchange views on estimation of measurement uncertainties. The announcement was distributed to all the WG DA members and posted on the ESARDA web-site. The interest in this workshop with about 50 participants was once more overwhelming and confirmed the importance of the topic being of great interest to a broad community. Particularly the participation from experts in the field of Non Destructive Analysis was highly appreciated and beneficial resulting in the recommendation to organise a joint DA/NDA/NA-NT follow-up workshop on the topic of uncertainty and reference materials in nuclear measurements for DA and NDA analysis.

The institutions that participated in the workshop are listed in Table 1.

Table 1: List of participating institutions

| **Institution** | **Country/organisation** |
|---|---|
| AREVA | France |
| Commissariat à l'Énergie Atomique - CEA / DAM Ile de France | France |
| Commissariat à l'Énergie Atomique – CEA / DEN Marcoule | France |
| Commissariat à l'Énergie Atomique – CEA / DEN Saclay | France |
| European Commission - Directorate-General for Energy | European Commission |
| European Commission - Joint Research Centre-Institute for Transuranium Elements - EC-JRC-ITU | European Commission |
| European Commission - Joint Research Centre-Institute for Reference Materials and Measurements - EC-JRC-IRMM | European Commission |
| Forschungszentrum Jülich GmbH | Germany |
| Hungarian Academy of Sciences - Institute of Isotopes | Hungary |
| Hungarian Atomic Energy Authority | Hungary |
| International Atomic Energy Agency - Office of Analytical Services, Dept. of Safeguards - SGAS | United Nations |
| International Atomic Energy Agency - Division of Information Management, Dept. of Safeguards- SGIM | United Nations |
| International Atomic Energy Agency - Division of Technical Services, Dept. of Safeguards- SGTS | United Nations |
| National Nuclear Laboratory | United Kingdom |
| Nuclear Material Control Center - NMCC | Japan |
| Paul Scherrer Institut - PSI | Switzerland |
| SCK - CEN | Belgium |
| Sellafield Site | United Kingdom |
| Swedish Defence Research Agency, FOI | Sweden |
| US - Department of Energy - Lawrence Livermore National Laboratory | United States of America |
| US - Department of Energy - Savannah River National Laboratory | United States of America |
| URENCO | Germany |
| URENCO Central Technology Group | The Netherlands |
| University of Natural Resources and Applied Life Sciences BOKU | Austria |

## 2. Objectives of the workshop

The focus of this workshop was on investigating major contributions to the final measurement uncertainties that depend upon the material and technique applied. Based on the discussions during the workshop, recommendations on the uncertainty estimations according to GUM should be given.

- To facilitate a technical exchange on approaches to the quantification of measurement uncertainty among reference material institutes, safeguards laboratories (nuclear material and environmental sample analysts), and operator laboratories
- To make recommendations on the approaches used in uncertainty estimation and the consistency with which the GUM is employed
- To make recommendations on uncertainty estimation for environmental sample analyses
- To make recommendations on the use of measurement uncertainties by customer

In particular the workshop addressed:

- GUM methodology for estimating measurement uncertainty
- Approaches in uncertainty estimations from state-of-the-art to state-of-the-practice
- Nuclear material analysis for accountancy purposes
- Nuclear material analysis for non-accountancy purposes
- Environmental swipe sample analysis
- Major uncertainty contributions depending on analytical techniques and sample nature
- Thermal Ionisation Mass Spectrometry (TIMS)
- Secondary Ionisation Mass Spectrometry (SIMS)
- Gas Mass Spectrometry for $UF_6$ analysis
- Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS)
- Hybrid K-Edge / K-XRF Densitometry
- Titration
- Coulometry
- Quality of measurement results (reliability, comparability, traceability)
- The safeguards point of view
- The operator point of view
- Revised International Target Values (ITV2010)

## 3. Workshop structure

Steve Balsley from the IAEA-SGAS welcomed the participants on behalf of the hosting organisation. Yetunde Aregbe, in her capacity as chair of the WG DA, opened the workshop with a short review on the conclusions from the Workshop on Uncertainties held in March 2006 at EC-JRC-IRMM, Geel. During that workshop, in 2006, the participants came to some of the following conclusions:

- Uncertainty estimation from state-of-the-art to state-of-the-practice measurements
- ISO Guide for Expression of Uncertainty in Measurement (GUM) is the basis for the evaluation of uncertainties in measurement of samples of nuclear material
- Knowledge of the measurement process
- Use of quality control data for estimation of measurement repeatability
- Uncertainty components that are due to sampling or to inhomogeneity
- Available software (GUM Workbench)
- ITV: comparing uncertainty estimates derived from interlaboratory exercises, from safeguards data and from a calculation according to the GUM

Subsequently, the workshop objective, structure and practicalities were outlined to the participants. During the first workshop day 17 presentations from workshop participants were given in one plenary and three topical sessions. The second day focused on the findings and points of discussion from these sessions and further discussions were held in a working group using the 'World-Café' approach around three selected topics. This approach ensures that all workshop participants contribute to the discussion and can benefit from the 'collective intelligence'. The outcome of the discussions around the three selected topics was presented to all workshop participants and first recommendations were drafted. This was followed by a presentation on the IAEA ECAS project and the new Nuclear Material Laboratory given by Steve Balsley with and optional laboratory tour of the IAEA Clean Laboratory Extension. The workshop was closed by Yetunde Aregbe and Steve Balsley acknowledging the IAEA for hosting the event and the contribution and good cooperation between the experts from nuclear safeguards, nuclear forensics, nuclear measurement laboratories, nuclear industry and environmental sciences institutes.

## 3.1. Plenary session

The plenary session was chaired by Klaus Mayer from the European Commission-Joint Research Centre-Institute for Transuranium Elements (EC-JRC-ITU), Karlsruhe, Germany. The two plenary lectures addressed the topic on uncertainties from the measurement and data evaluation point of views.

### 3.1.1. Uncertainty in nuclear measurements

The first plenary lecture, entitled 'Uncertainty in nuclear measurements', was given by Roger Wellum, individual ESARDA member (IRMM-retired). He recalled in his lecture that the improvements in measurement quality in the field of nuclear safeguards were driven by the comparisons operator – inspector, which led to the establishment and adoption of the ITV. The benefits of applying the GUM in measurement laboratories are unchallenged. Due to advances in instrumental techniques there has been a shift over time of distribution of uncertainty contributions from Type A to Type B.

Excerpt from the - Guide to the expression of uncertainty in measurement [5]

- **Type A evaluation** of standard uncertainty: the best available estimate of the expectation or expected value μ*q* of a quantity *q* that varies randomly, and for which ***n* independent observations** *qk* have been obtained under the same conditions of measurement is the **arithmetic mean** or **average** *q* of the *n* observations: This estimate of variance and its positive square root *s*(*qk*), termed the **experimental standard deviation** characterize the variability of the observed values *qk* , or more specifically, their dispersion about their mean *q*.

- **Type B evaluation** of standard uncertainty: For an estimate *xi* of an input quantity *Xi* that has **not been obtained from repeated observations**, the associated estimated variance *u*2(*xi*) or the **standard uncertainty** *u*(*xi*) is **evaluated by scientific judgement** based on all of the available information on the possible variability of *Xi* . (certificate of analysis, previous measurement data, manufacturer's specifications, experience, etc.)

- The **standard uncertainty of** *y*, where *y* is the estimate of the measurand *Y* and thus **the result of the measurement**, is obtained by appropriately **combining the standard uncertainties** of the input estimates *x*1, *x*2, ..., *xN* (see 4.1). This *combined standard uncertainty* of the estimate *y* is denoted by *u*c(*y*).

Subsequently R Wellum discussed the GUM “bottom up approach” for the estimation of measurement uncertainty using isotope dilution as example. This approach requires a thorough knowledge of the measurement process, its description in a mathematical model (expressed as equation) and the definition of all input parameters with their respective standard uncertainties. Correction factors should be included in the measurement equation and uncertainties propagated accordingly (Type A, Type B). R Wellum was referring to the revision of the ITV and expressed the opinion that defining target values for "random" and "systematic" components is not consistent with current terminology as used in the GUM. Particularly, nowadays when more nuclear laboratories are striving for accreditation in compliance with international guidelines, such as ISO/IEC 17025:2005 and ISO Guide 34:2009 [6, 7].

In this plenary and throughout the workshop discussions it was emphasised that the aim is not to reach the smallest possible uncertainty on a measurement result. The effort in establishing a full uncertainty budget has to balance the intent of the measurement using the principle of “fit for purpose”. A nuclear reference material laboratory for example has other requirements on the final uncertainty of the certified values compared to a routine measurement laboratory that has to demonstrate that products or materials meet legal or customer requirements. It was also stressed during this plenary that, from a metrological point of view, only measurement results with properly established combined uncertainties are truly comparable. It is, however, not always straightforward to take possible correlations properly into account when calculating the combined measurement uncertainty. As an example he mentioned the use of the same certified reference material (for different calibrations) within a set of measurements. The best approach would be if confirmation of results can be achieved by two laboratories using two different methods, (e.g. coulometry and IDMS) and different reference materials. Agreements in measurement results obtained under such conditions provide a strong basis for safeguards verification conclusions. In summary it can be said that confidence in measurement results (and the associated uncertainties) can best be achieved if the measurement method and its uncertainty components are really well understood.

During the discussion of the first plenary session, participants expressed their opinion on the revised ITV2010. On the one hand, the current ITV2010 revision still use the expression “random and systematic errors”, which is not fully consistent with the GUM but highly useful for safeguards evaluators. On the other hand participants very much appreciated that in the ITV2010 upon the recommendation of the ESARDA WG DA and NDA relative combined standard uncertainties together with a new chapter on *GUM and the Use of ITV by Measurement Laboratories* have been included. This new chapter in the ITV2010 document is an attempt to establish the link between the current approach of laboratories evaluating their measurement uncertainties according to the GUM and the safeguards evaluators needs to identify when an operator-inspector difference exceeds a certain limit.

### 3.1.2. Measurement uncertainty in material balance verifications

The second plenary lecture, entitled ‘Measurement uncertainty in material balance verifications’, was given by Claude Norman from IAEA-SGIM-IFC. She recalled in her lecture the objectives of safeguards:

- 1. Detection of undeclared material/activities
- 2. Detection of undeclared production or processing of nuclear material
- 3. Detection of diversion of declared material (INFCIRC/153)

The objective of material balance evaluation is to detect the following scenarios:

- Material is diverted without falsification of the records: the accounting records correspond to reality and reflect the diversion

- Material is diverted with falsification of the records: the accounting records do not correspond to reality and conceal the diversion

Particularly critical is the 'Bias Defect Diversion' where items or batches are slightly falsified so that only a small amount (difficult to detect) of material is missing. Discrepancies can arise from mistakes in the records or measurement errors. Therefore the concept of measurement uncertainty is very important for the evaluators to confirm the presence of discrepancies. From the point of view of the inspectorate "fitness for purpose" is the key! The evaluation of measurement values (including their respective uncertainties) and of information contained in state reports is performed using the ITV as an evaluation criterion. Based on this evaluation conclusions on the diversion are taken.
From the evaluator point of view a 'mistake' is defined as the difference between the recorded or reported value and the measured value, whereas and 'error' is defined as the difference between the measured value and the true value. C Norman empathised that contrary to the nuclear measurement laboratories the methodology and concepts of safeguards data evaluation need a term describing "random measurement error" and the "systematic measurement error" in the data to properly consider correlations in the safeguards data. Random errors and systematic errors are treated as being of the same nature but their mode of propagation is different. The effect of systematic errors is predominant (squares of sums as opposed to sums of squares for the random error). The IAEA Department of Safeguards uses an uncertainty model suited to the specific purpose of data evaluation for safeguards, which is different from the purpose of laboratory analyses.

The workshop started with a plenary session setting the scene and underlining the different points of view related to handling uncertainties. It was confirmed that analytical laboratories and nuclear inspectorates apply different approaches for uncertainty evaluation. It was clear that even during such an extensive workshop it would not be possible to find a final answer to all questions raised during the plenary session. There was, however, a common agreement already at that stage of the workshop that this topic requires continued exchange between the measurement and evaluator communities and will be further addressed by the ESARDA working groups and in follow-up workshops.

## 3.2. Session 1: Nuclear material analysis for accountancy purposes

The first session following the plenary was associated with measurements for nuclear material accountancy purposes, chaired by James Tushingham from the National Nuclear Laboratory, UK. In this session, approaches for uncertainty estimation were presented for the main destructive assay techniques applied to nuclear material accountancy, with contributions from facility operators and external measurement laboratories.

Four presentations dealt with isotopic measurements utilising mass spectrometry. The first, given by Stephan Richter, IRMM, Belgium, described state-of-the-art developments in determination of the isotopic composition of uranium using a new uranium hexafluoride ($UF_6$) gas source mass spectrometer (GSMS). The primary purpose of IRMM's measurements is the certification of reference materials, for use by safeguards authorities and facility operators, requiring not only high accuracy but a thorough understanding of the uncertainties associated with the measurements. GSMS offers unprecedented reproducibility for $^{235}U/^{238}U$ measurement, requiring reference materials certified with the lowest attainable total uncertainties in order to obtain maximum benefit from the analytical technique. Work at IRMM on the preparation of gravimetric mixtures of uranium isotopes was described and their application to both GSMS and thermal ionisation mass spectrometry (TIMS) was discussed. For, GSMS, the benefits of calibration using a

single standard and a double standard (DS) method were compared. Memory effects – essentially the carry over of material from one sample to the next within the mass spectrometer – were shown to be dependent upon instrument design and the relative isotopic difference between sample and standards. A 'memory-corrected double standard' procedure was described, to achieve the lowest uncertainty in $UF_6$ measurements, and results were presented for the URANUS GSMS. Method validation and performance of the mass spectrometer for minor isotope measurements (i.e. $^{234}U/^{238}U$ and $^{236}U/^{238}U$) were shown to be satisfactory, with selection of the standard dependent upon the $^{236}U$ content of the sample [8].

To contrast the work of IRMM, Andrew Dobney, SCK-CEN, Belgium, provided an operator's perspective on the application of TIMS for determination of uranium and plutonium isotopic composition and amount, the latter by isotope-dilution mass spectrometry (IDMS). For measurements supporting fuel qualification, total uncertainties of 0.25-0.3% were achieved on uranium and plutonium assay. Whilst application of control charts and external quality control exercises provided evidence of continuing performance, review and refinement of uncertainty budgets was found to be invaluable in assessing contributing factors to the overall uncertainty in measurement. Application of the GUM workbench enabled contributing factors to be assessed, and steps taken to reduce significant uncertainty components (e.g. spike concentration) where practical. Ultimately, the uncertainty on mass bias was found to dominate the overall uncertainty, contributing ca. 85% of the total uncertainty. However, in the overall fuel qualification, variations in sample moisture content were found to be critical, reminding the audience that the overall measurement performance depended upon more than those factors under the analysts immediate control. The question of 'how low can we go?' in terms of refining TIMS uncertainty budgets becomes 'how low do we need to go?' before other factors dominate the accuracy of analysis results in the industrial environment.

Hélène Isnard, CEA Saclay, France, presented the harmonised approach of CEA-AREVA in uncertainty estimation for uranium isotopic measurements in nuclear samples. CETAMA (Commission d'ETAblissement des Methodes d'Analyse), responsible for improving the quality of measurement and analysis results within the French nuclear industry, had instituted a 'network of expertise' based on the needs of laboratories, including to standardise methods, perform uncertainty evaluations and present innovative developments. The process was described whereby a guideline for uncertainty estimation in uranium measurements by TIMS was developed through consideration of sample types; pre-measurement sample treatments; filament type and method of loading; data acquisition (i.e. sequential measurements or total evaporation) and TIMS instruments employed across the group of laboratories. All sources of uncertainty were evaluated, and the dominant components identified in order to understand, manage and improve the measurement performance. The process enabled each laboratory to evaluate the major sources of uncertainty for their particular procedure and instrument combination.

The final technical presentation related to mass spectrometry, given by Shin-ichi Inoue of the Nuclear Material Control Centre, Tokai Safeguards Centre (NMCC-TSC), Japan, compared different approaches to the monitoring of measurement performance. Within NMCC-TSR, control limits on analytical results are derived from results obtained, using Xbar-R control charts, rather than through error propagation on each measurement (i.e. the GUM approach). Using a TRITON-TI TIMS instrument with total evaporation, determination of plutonium concentration in a quality control sample by IDMS yielded an estimate of total uncertainty (approximately 1σ) of 0.24% using GUM methodology. This contrasted with 0.09% (1σ) using Xbar-R within a single laboratory. Across a three laboratory evaluation, the IAEA has calculated the total uncertainty in plutonium concentration to be 0.30% (input solution) and 0.28% (output solution). This evaluation is close to the GUM estimate of 0.24%. In contrast, for MOX samples, a three laboratory evaluation yields a total uncertainty in the plutonium content of MOX of 0.59%, compared with 0.24%**+α** by GUM, where α represents the condition of the MOX powder. For application of the GUM approach, it is necessary to consider how to estimate α, which will be dependent upon heterogeneity, moisture content, oxidation etc. Xbar-R is therefore applied in preference within NMCC-TSC, as a strong internal quality control tool.

The theme of quality control charts continued with a description by Thomas Morris, Sellafield Ltd, UK, of the quality system employed by the analytical laboratories at Sellafield. A combination of internal and external audits, validation and intercomparison exercises were described to satisfy the requirements of IS) 17025 accreditation. A case study was presented, involving comparison of results between TIMS and multi-collector inductively-coupled plasma mass spectrometer (MC-ICPMS) instruments with externally-produced (IRMM) and in-house large-size dried spikes. Multiple duplicate samples and standards were measured using multiple analysts over multiple days in a 'top down' approach to assess measurement performance.

From mass spectrometry, the session turned to consider some of the most common methods, other than IDMS, to determine elemental concentration in nuclear material samples. Rainer Ludwig of the IAEA On-Site Laboratory (OSL), Rokkasho Reprocessing Plant (RRP), Japan, provided an uncertainty estimation for nuclear material verification measurements using Hybrid K-Edge/K-XRF Densitometry (HKED). Application of the technique at RRP presented a number of challenges associated with the samples, including radiolysis, resulting in an increase in concentration, changes in sample matrix and bubble formation; moisture absorption in powder samples; and the presence of solid particles. These sources of uncertainty were considered alongside the more common uncertainties in HKED measurement, including those associated with calibration, sample positioning and instrument stability. Despite the challenges in implementation of HKED within the industrial reprocessing environment, uncertainty components were within the 95% confidence level defined by the ITV 2010 for all samples except low-burnup spent fuel, for which $u_r$ for plutonium was outside the 95% confidence level. Continuing improvements in hardware and software for the HKED systems were described, offering improved precision and reduced bias.

On behalf of Michael Soriano, New Brunswick Laboratory, USA, Rebecca Thomas of the IAEA Safeguards Office of Analytical Services presented an uncertainty analysis of Davies and Gray titration measurements, as performed at the IAEA Nuclear Material Laboratory (NML). The purpose of the analysis was to estimate the total uncertainty for a typical measurement result based upon duplicate measurements, to isolate sources of uncertainty specific to an individual sample and to provide a simple calculation that the analyst could perform to obtain an estimate of measurement uncertainty unique to the sample being measured. The major sources of uncertainty were found to be variation in the duplicate measurements, and variation in measurements of the calibration standard, with the uncertainty (95% CI) on the mean of two titration measurements ranging from 0.04% to 0.16%. The NML has a policy of rejecting any results where the relative standard deviation (RSD) of duplicate measurements is greater than 0.1%, or any set of calibration data has an RSD greater than 0.07%.

The session was completed with two presentations on controlled potential coulometry. The first, given by Michael Holland, Savannah River Nuclear Solutions, USA, described the development of coulometry as a primary measurement technique, traceable through electrical calibration to the SI via mass; current; temperature; faraday constant; and molar gas constant, independent of certified reference materials and with uncertainties consistent with International Target Values. The basis of the technique was described, and sources of uncertainty from sample preparation and measurement were assessed. In addition to those that could be quantified, such as sample weighing and electrical calibration, additional unquantifiable sources were identified. Many of these could be considered as 'human factors', such as the experience of the analyst and the demands of sample throughput: important considerations when assessing the 'true' uncertainty in any analytical technique.

The session concluded with a description of the coulometry method as applied in the LAMM (Laboratoire d'analyse et de Metrologie de Matiere), presented by Catherine Eysseric) CEA, France. There, coulometry is applied both to reference material certification and nuclear material accountancy, again without call upon chemical standards. Corrections for air buoyancy; radioactive decay; initial sample evaporation;

blank solution; residual intensity and non-electrolysed fraction are made, with ITV being achieved on samples as small as 3mg. For smaller samples whilst uncertainty can be improved by repeat measurement, the contribution to the overall uncertainty from the solution blank, residual current and non-electrolysed fraction suggest that ITV cannot currently be achieved on these small samples. However, the laboratory is engaged in a programme of technical improvements, and developments are awaited with interest.

## 3.3. Session 2: Nuclear material analysis for non-accountancy purposes

The second session was dedicated to nuclear material analysis for non-accountancy purposes, chaired by Guy Granier from CEA/CETAMA, Marcoule, France. In this session approaches for uncertainty estimation were presented for nuclear forensics by Zsolt Varga from the EC-JRC-ITU and special safeguards samples applications by Sergey Boulyga and Stefan Bürger† from the IAEA-SGAS.

If we consider measurement as capital in the field of nuclear material non proliferation, the determination of uncertainties is an essential element that validates measurement and the decision that follows. A growing area of importance in this regard is the determination of element traces and isotopic ratios.
In this context, 'dating' is an important tool to determine the date of the last chemical separation and to pursue investigations on the origin of nuclear materials. Radiometric dating techniques are based on the decay of isotopes with constant rates. If a chemical separation of a compound containing radioactive uranium and thorium occurs, a new equilibrium appears. Uranium-thorium dating is an absolute dating technique which uses the properties of the radio-active half-lives of $^{238}$U and $^{230}$Th. The half-life of $^{238}$U is 4 470 000 000 years, while that of $^{230}$Th is only 75 380 years [9]. When the amounts of uranium and thorium are compared, an accurate estimation of the age of a material can be obtained. This information is increasingly used for characterization purposes in order to determine the last separation date, to check declared nuclear material inventories, and to identify if necessary the origin of samples.

Nuclear safeguards, nuclear forensics dating is similar to dating in geology but looks for time spans in a range of 50-60 years. Uranium age dating is conducted using radiochemical methodology. The methodology is based on uranium and thorium decay series, each including nuclides with different properties and with a wide range of decay half-lives. These nuclides can be separated, e.g. during reprocessing, by a chemical separation and the goal of analysis is to know the date of this separation as accurately as possible. Once separated, they tend to restore radioactive equilibria and determination of the ratio enables an estimation of this separation date. The so-called $^{234}$U/$^{230}$Th 'clock' is most commonly used for characterization purposes in order to determine the date of the last chemical separation of thorium from uranium. To calculate (estimate) the age of a material, the respective mother/daughter ratio is determined via IDMS (spiking, chemical separation) and isotope measurements are carried out using ICP SFMS. The uncertainties on this age come from the completeness of the chemical separation, the IDMS, and the half-lives used for the calculation of the last separation date. In this process applying the GUM is a useful tool for identifying major uncertainty contributions, and thus allows fine-tuning and optimization of the analytical methodology. Currently there are no reference materials available certified for the date of chemical separation. IRMM, ITU and NBL have recently engaged in the development of such reference materials [10, 11].

Determination of concentrations of inorganic impurities in uranium samples to assess the purity grade of nuclear materials and to allow an attribution of sample origin is another most vital analytical tasks entrusted to IAEA laboratories [12]. The determination of relative concentrations of trace elements expressed as µg/gU requires the investigation on procedural blanks to control memory effects (possible accidental contamination) during dissolution and measurement using ICP-MS, and the application of correction

factors for blanks to take into account matrix effects (different sensitivity with and without uranium matrix). However, the quality control of analytical data is difficult because the available certified reference materials (CRMs), which are used as QC samples for impurity analyses, are not certified for all required elements. Furthermore, the compositions (the range of trace element concentrations) in the available CRMs do not correspond to the compositions of inspection samples. For instance, inspection samples often contain relatively high concentration of Mo and Zr, which produce interferences on Cd, Te, some rear earth elements (REE). Such interferences are not significant in CRMs, which contain low concentrations of interfering elements, but they can be significant in the inspection samples. Uncertainty budgets are useful to improve the understanding of the analytical procedure through identifications of main factors that influence measurement accuracy (most important contributors to the uncertainty budgets), assessment of the effect of interferences, blanks etc. The evaluation of measurement uncertainties represents an important step in the quality control of impurity results allowing the identification of possible significant differences between results that are obtained in different laboratories.

Another focus during this session was on uncertainties in TIMS measurements of safeguards samples. Reference materials provider such as IRMM and NBL and metrology institutes apply almost exclusively TIMS for U and Pu analysis (IRMM and NBL) while other mass spectrometry techniques are only used to a lower extent. In the case of U and Pu isotope ratio & isotope dilution analyses, the common TIMS analytical procedures consider either multi-collector Faraday cup total evaporation analysis, possibly combined with single ion counter modified total evaporation analysis, or single ion counter (e.g. SEM) peak jumping. Sources of uncertainty associated with these procedures are estimated based on independent replicate sample analysis or on pooled estimates of standard deviations derived from adequately matched CRM replicate analyses. A GUM uncertainty evaluation is tailored to a specific analytical procedure. If the procedure is changed, the GUM uncertainty evaluation will need to be changed as well. In all cases, performing a GUM uncertainty evaluation requires a high degree of understanding of the analytical procedure. At the same time, implementation of the GUM boosts the analyst's understanding of the analytical method [13].

In either ICP MS or TIMS analyses, IAEA laboratories exclusively use the GUM to express the measurement uncertainty. This Guide has been adopted by most of the national metrology institutes in the world, and is regarded as a state-of-the-art approach to uncertainty in measurement.

## 3.4. Session 3: Environmental Swipe Sample Analysis

The third session was dedicated to environmental swipe sample analysis, chaired by Dave Donohue from the IAEA-SGAS. In this session approaches for uncertainty estimation were presented for bulk and particle analysis of environmental swipes. Examples were given by experts from CEA/DAM, BOKU-WIEN and the IAEA on Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) and Large-Geometry Secondary Ion Mass Spectrometry (LG-SIMS)

The CEA has two main missions the environmental survey of French nuclear sites, including the former French nuclear test sites (Mururoa, Fangataufa atolls) and the detection of nuclear proliferation; the laboratory is member of the IAEA's NWAL since 2001, both for bulk and particle analysis. Fabien Pointurier presented Pu bulk measurements at CEA carried out by ICP-MS. The samples are analysed after chemical purification, based on ion-exchange chromatography resins, and isotope dilution with a $^{242}$Pu tracer, the detection limit is the range of fg for Pu isotope analysis. The total combined uncertainties are calculated in compliance with recommendations from the GUM. Type A contributions originate from the counting statistics, measurements of instrumental blank, interferences and reference materials. Type B uncertainties are introduced to the overall combined uncertainty as stated on the certificates from the respective uranium and plutonium used, from corrections related to weighing, and partly from mass bias effects. Taking into account the low number of replicate measurements in

combination with the low count rates, particular care is required to avoid underestimation of uncertainties. The major uncertainties for low-level Pu measurements (fg-range) Pu measurement by SC/ICP-MS are resulting from Interferences corrections (low mass resolution mode) and are due to impurities in the tracer used for spiking the sample. The first can be addressed by improving the chemical purification, the latter by improving the accuracy of the impurity ratios.

Stefanie Kappel from the BOKU-Vienna presented uncertainty calculation for U isotope ratio analyses of single particles by LA-MC-ICP-MS [3]. The set-up of the instrumentation, data collection and data reduction fro a standard-sample-standard bracketing approach was outlined. An operator-independent threshold for the selection of intensities used for further evaluation is set as such that the isotope with the smallest number of single data points above twenty times the standard deviation of the determined blank signal defines the peak areas for all isotopes measured. Then the intensities of each isotope were integrated over the defined peak area, and the $^{234}U/^{238}U$, $^{235}U/^{238}U$ and $^{236}U/^{238}U$ isotope ratios were calculated by dividing the integrated signal intensities of the isotopes. Integrating over the defined peak area was performed because of counting statistics, thereby reducing the influence of small count rates on the isotope ratio. Uncertainties are calculated according to GUM where the main contribution is due to the $^{234}U/^{238}U$ and $^{235}U/^{238}U$ repeatability. Particularly the determination of $^{236}U/^{238}U$ is a challenge, main contributions due to peak-tailing resulting from $^{238}U^{+}$ and interference due to $^{235}U^{1}H^{+}$ ions, contributing significantly to the total combined uncertainty. Furthermore a new data reduction approach for evaluation of the entire data set (blank+data) was presented [14, 15].

The IAEA installed in September 2011 a new Large Geometry Secondary Ion Mass Spectrometer (LG-SIMS) in the Clean Laboratory Extension, which is part of the Environmental Sample Laboratory [16]. EC-JRC-ITU inaugurated its new LG-SIMS Laboratory on 1$^{st}$ of June 2012 [17].

The uncertainty budget for uranium particle analysis by LG-SIMS was presented by Alexander Schwanhäußer from the IAEA-SGAS. After a brief introduction to the SIMS instrumental technique and the swipe sample preparation steps prior to particle analysis, he gave a detailed outline of the individual components of the fairly complex uncertainty budget for particle analysis by LG-SIMS covering components from reproducibility, biases, mass bias calibration, and individual particle uncertainties. To estimate the contribution from mass bias correction reference particles (NBS010, 030a, 200, 500, 930) were measured.. The final mass bias uncertainty encompasses the uncertainty for the mass bias correction plus a factor for the demonstrated accuracy over a long period. For instance, the total mass bias uncertainty for $^{235}U/^{238}U$ consists of a global component assessed via the average variability of CRMs calibrated against a day-to-day curve established via frequent measurements of NBS010, including a component for any remaining significant bias, and of a (day-to-day) component depending on the relative standard deviation from the measurement of the CRM used to correct for the mass bias and the uncertainty of the certified values. The approach for the minor uranium isotope ratios is similar trying to measure the variability and mass bias by means of CRMs. Variability can be averaged and is quite low, but biases can become quite significant in the low (6ppm) range, and cannot been covered for the whole range of abundances in a satisfactory manner, resulting in an abundance depended uncertainty component for the minor uranium isotope ratios. The reported total combined uncertainty for $^{234}U/^{238}U$ and $^{236}U/^{238}U$ consists of a component described as “daily” mass bias error and the uncertainty assessed by using CRMs and the uncertainty from the analysis of the individual field particle (counting statistics, interference, dead time etc…). Room for improvement should be given towards development of new reference particles and in a better understanding of $^{236}U$ measurement and bias.

## 3.5. Working Group

After the three sessions with presentations from invited speakers, the second part of the workshop was dedicated to discussing the findings from those presentations in a working group and to draft a set of recommendations on approaches in uncertainty estimations in nuclear measurements for accountancy, non-accountancy and environmental sampling applications. Due to the number of workshop participants, the ESARDA WG DA returned to the 'World-Café' approach already successfully applied in the previous WG DA workshop on 'Direct Analysis of Solid Samples Using LA-ICP-MS'[3]. The 'World-Café' is a workshop method based on the assumption of a collective knowledge. The participants are guided to interact in a constructive way in their discussions, where each participant can express his/her point of view. They are spread within different topics, where they deal with a specific question. To each of the topics a facilitator is assigned. After a set time, the participants change within the topics, get a résumé by the facilitator of the topic and restart the discussion with the next question related to this topic. The 'World-Café' was chaired by T Prohaska from BOKU-WIEN with assistance from Y Aregbe from IRMM and S Balsley and S Vogt from the IAEA-SGAS.
Within the ESARDA workshop, the participants were divided in three groups, of about eleven participants per group, to discuss the three topics in line with the objective of the workshop. Around these three topics, questions were raised in three rounds and discussed by the three working groups in a rotational sequence:

*Topic 1 – Approaches to uncertainty*

What type of uncertainty calculations are you currently applying?
What kind of improvements would you require/request?
What is required in order to achieve those improvements?

*Topic 2 – Sources of uncertainty*

What sources of uncertainty are you currently observing?
Which sources of uncertainties would need more research?
What research is required in order to improve the knowledge about these sources of uncertainties?
What reference materials are requested?

*Topic 3 – Knowledge of uncertainty*

What is the current knowledge of uncertainty?
Where do you see a need for improvement?
What measures should be taken in order to improve the knowledge about uncertainties of measurements?

Discussions between experts from the various fields of application proved to be highly beneficial. Each workshop participant contributed to each of the three topics, therefore all participants could benefit from the 'collective intelligence' in the room. This approach enabled a first set of recommendations per topic to be drafted and presented to all participants at the end of the workshop day. Those draft recommendations were immediately after the workshop circulated for comments to all participants and subsequently finalised. The following three paragraphs list the complete set of recommendations, identified by the workshop participants.

### 3.5.1. Recommendations for approaches to uncertainty

**Topic 1 – approaches to uncertainty** dealt with the different approaches applying algorithms, software, quality control tools to calculate uncertainties of measurement results. A reoccurring point was that the uncertainties reported should be "fit for purpose" and not aiming at the smallest achievable uncertainty. The workshop participants discussed the following questions in detail to finally come up with a list of recommendations:

- **T1: What type of uncertainty calculations are you currently applying?**
    - How do you currently calculate/estimate uncertainties?
    - What methods do you apply? What standards do you apply?
    - Which software are you using?
    - Who is doing the calculations?
    - How are these calculations implemented in your current work (routine, on demand)?
    - What criteria are applied (ITV...)? Measurement quality goals?
- **T1: What kind of improvements would you require/request?**
    - Implementation?
    - Training?
    - Software?
    - Fit for purpose – Do we always need the smallest uncertainties? How do we get ‚applicable' approaches? What do evaluators need (e.g. heterogeneity)?
    - Define target values (e.g. environmental analysis); measurement quality goals?
- **T1: What is required in order to achieve those improvements?**

**Recommendations to Topic 1 – approaches to uncertainty**

- Industrialize approaches
- Improve applicability of GUM
- Find easier approaches which are applicable and still compliant with GUM
- Harmonize approaches
- Improve level of knowledge
- Initialization of workshops dedicated to uncertainties (e.g. ESARDA) (enhance exchange of knowledge)
- More publications (quantity; quality)
- Implementation of uncertainty calculation as part of QA/QC programs or instrumental software
- Increase number of trained staff
- Interlaboratory comparisons/round robins using well characterised test samples with reference values and stated uncertainties (CRMs if possible) and ask participants to report their measurement uncertainty
- Include detailed uncertainty protocols when participating in round robins
- Technical sheet examples (e.g. on ESARDA website)
- Close collaboration between GUM experts and MBA SG evaluators

- Uncertainty estimates as driving force for ITV revisions/measurements targets and technology

As can be seen from the recommendation list not all topics for improvement are equally easy to implement on either a laboratory or institute level. To highlight the main issues, it was clear that the GUM is sometimes seen as too complicated to be implemented easily in a routine analytical laboratory environment. More training and exchange between experts on uncertainty approaches and industry, but also instrument developers would be appreciated. Particularly collaboration between 'metrology institutes' and safeguards evaluators is recommended to be strengthened in the future. It was also argued whether the revision of the ITV every 10 years is sufficient or whether it would be more beneficial to revise the ITV in a 3 years reoccurring cycle. There was an overall agreement that the ESARDA WGs via technical sheets on analytical techniques, reference materials, quality control tools and uncertainty estimation plays an important role in disseminating knowledge to the community. The ESARDA technical sheets of the working groups should be more 'advertised'.

### 3.5.2. Recommendations for sources of uncertainty

**Topic 2 – sources of uncertainty** focused mainly on the identification of major contributions to the combined uncertainty and what factors can be neglected under given circumstances and depending on the analytical techniques applied. This question was looked at from the perspective of sample preparation, instrumentation, analytical protocols and use of calibrants and matrix matched reference materials, particularly on improvements towards new referene materials with considerably smaller combined uncertainties of the certified values.

- **T2: What sources of uncertainty are you currently observing?**
    - Which parameters are crucial?
    - What are the parameters, which you assess on a routine basis? (What are the major contributors?
    - What are the parameters, you are 'neglecting' (what are the minor contributors)?
    - What reference materials are used?
    - How are you dealing with homogeneity?
- **T2: Which sources of uncertainties would need more research?**
    - Which parameters are not approached so far?
    - Which parameters need more investigations?
    - What are the major unknowns in your uncertainty budget?
    - Which uncertainties should be reduced (reference materials...)?
    - Repeatability < Reproducibility < Standard uncertainty – What if not?
- **T2: What research is required in order to improve the knowledge about these sources of uncertainties? What reference materials are requested?**

**Recommendations to Topic 2 – sources of uncertainty**

- Sampling, sample preparation
    - Contamination/cross contamination
    - Storage of samples/reference materials
    - Dissolution
    - Chemical separation
    - Sample containers (fit for purpose?)

        - Sampling devices
        - Complete sampling procedure review
    - Instrumental
        - Interferences; Isobars – improving the chemical procedures; hardware
        - Improve chemical separation
        - Matrix effects; impurity effects
        - Detectors - cross calibration, yield, linearity, stability, dead time (for LA-ICP-MS if short duration large spikes in signal at < integration time; for SIMS if particle size is < raster area by a significant amount – missed by the manufacturer, and hardcoded into the output data
        - Peak tailing
        - Fractionation and mass bias
        - Memory effects, blank correction
        - Instrumental stability
    - Analyst effects
    - Calibration and internal standards
    - Reference materials
        - Matrix matched RM
        - High purity solution CRM
        - RM with selected impurities
        - CRM of target interferences
        - Rethink the fit for the indented use CRM (are the RM appropriately used)
        - Comparison of different CRM (e.g. U-PU spikes (IRMM, IAEA, Japan))
        - Improve speed of production
        - RM stability
        - Faster feedback loop and realization process for RM
        - ...more CRM (age determination, particles, K-edge XRF)
        - Use of NWAL in certifying new working RM
    - Timing of uncertainty estimation and measurements
    - Systematic studies (sampling uncertainty)
    - Interlaboratory comparison results can enhance the level of knowledge about uncertainties

It was clear from the discussions that a thorough knowledge of the entire analytical process, from sample collection, to preparation to measurement, is required to identify the major contributors to the overall uncertainty. This is of a major concern for environmental swipe sample analysis where traces of nuclear materials need to be measured accurately on micrometer sized particles. For some techniques with transient signals, such as LA-ICPMS, some of the relevant contributors are still either not understood or neglected [3]. Cross contamination effects are often analyst-dependent and are often a source of bias of the measurement result. There was a common agreement on the usefulness of CRMs for the estimation of measurement uncertainty. In the nuclear field a number of isotopic reference materials are on the market for decades. With the improved instrumental techniques there is a need for new reference materials with considerably smaller uncertainties. This is especially true for Pu CRMs. Application in nuclear forensics and nuclear security require new types of reference materials such as U, Pu material certified for the last separation date, U, Pu reference particles and reference solution for calibration of K-edge XRF. Although it was acknowledged that nuclear reference material providers are currently developing new materials and issuing new certificates for old materials, the time needed for certification of a

material was considered to be too long to serve the community in an optimal way. A suggestion was made to involve the Network of Analytical Laboratories in so-called 'certification campaigns' to speed up the process and to have results from different laboratories applying different techniques as external verification. Another major issue was the time to be invested in identifying all relevant contributors to the uncertainty in relation to the time required for the measurement itself. Again it was stressed that this time effort has to be 'fit for intended purpose'. Interlaboratory Comparisons have been carried out for decades in the nuclear field and are important external quality control mechanisms for laboratories to improve their level of knowledge about uncertainties.

### 3.5.3. Recommendations for knowledge of uncertainty

**Topic 3 – knowledge of uncertainty** discussed the important issue on the current status of knowledge of uncertainty and training provided in this field. The participants investigated this topic from the point of view of in-house on the job learning/training but also from available guidelines and related training courses.

- **T3: What is the current knowledge of uncertainty?**
  - What is your knowledge?
  - What is the knowledge of your co-workers/employees?
  - What is the knowledge in the scientific community, colleagues....?
  - Where did you/they get the knowledge/training?
- **T3: Where do you see a need for improvement? (in Training)**
  - What contents need more information/training?
  - Who should be trained?
  - Where training should be provided?
  - How should the training be provided?
  - Who should provide the training?
- **T3: What measures should be taken in order to improve the knowledge about uncertainties of measurements?**

**Recommendations to Topic 3 – knowledge of uncertainty**

- Creating awareness => beneficiary to customers/operators
- Educate the customers(cost benefit)
- Improve feedback - Platform for feedback (ESARDA can only initiate)
- Extend existing platforms (e.g. TRAINMIC)
- IAEA/EURATOM should continue to work with the measurement community to explore the potential for the Agency's evaluation requirements to be accommodated by laboratories reporting results in accordance with current measurement standards, including the GUM; (include guidelines; measurement goals – particle, bulk, impurities)
- Increase the exchange nuclear/non nuclear fields (extend ITV to non nuclear)
- More university courses (start at the basic level)
- More trainings at professional levels
- => close the gap!
- Tailor made training
- Improve mentoring
- Improve quality of training material, trainers

- Harmonized training
- Promote use of GUM and metrological concepts to NDA => how can it be applied (workshop?)

=> promote! YOU

Summarising the recommendation on this topic was to create awareness that reporting of results with expanded combined uncertainties is beneficial for the laboratory but also for the customer from industry or from an inspection body. Initiatives like this workshop cannot address all issues and cannot replace training and knowledge transfer done by experts or via training courses. ESARDA can serve as a platform of exchange/feedback and initiate workshop, courses or activities taken up by others. During the discussion the participants noticed that guides, courses and training on uncertainty matters also applicable to nuclear measurements are available in different communities, organised in several countries but not known outside the targeted community (see paragraph 5. on web-links). Concluding it was recalled that metrological concepts, including evaluation of measurement uncertainty, apply to all measurements, regardless of destructive or non-destructive techniques are applied. Therefore a major benefit of this workshop was the participation of NDA experts, although this workshop was mainly organised for destructive analytical techniques. The recommendation to promote use of GUM and metrological concepts to NDA has to be understood in that sense that the present approach not be interpreted as non-compliance with the state-of-the-art but is different from the purpose of laboratory analyses. The question was raised how can it be applied to NDA? As a result of the discussions on this topic the idea of organising a joint ESARDA workshop with experts from destructive, non-destructive analysis, industry and Euratom and IAEA safeguards evaluators was enthusiastically embraced.

## 4. Summary and Outlook

The ESARDA WGDA adopted in 2010 the objective t*o emphasise the technical convergence of nuclear safeguards, nuclear forensics and nuclear security by looking at available and new methodologies that serve all three purposes* in its Action Plan [18]. Part of meeting this objective is the organisation of dedicated workshops since they are not only a means of bringing together experts from safeguards, nuclear forensics, environmental sciences, but also allow WGDA members from industry to get authorisation for participation from their hierarchy, which is often not the case for the regular annual WG meetings.
This was the second WGDA workshop dedicated to uncertainties in nuclear measurements. The workshop focused on investigating the major contributions to the final measurement uncertainty. Important contributors to the material and technique applied but also on the differences in approaches, and very important, the knowledge transfer and training. Applying the GUM enables consistency of measurements carried out by nuclear laboratories and by operators, where the aim is not to achieve the lowest possible uncertainty but to provide a sound and transparent estimation under routine analytical conditions, and thus being 'fit for intended purpose'. The inclusion of a chapter on '*GUM and the Use of ITV by Measurement Laboratories*' in the revised ITV2010 is a step forward in establishing the link between laboratories and evaluator needs. The discussions held in the working groups and the sessions resulted in broad recommendations, which cannot be all followed up by the ESARDA WGDA, but were seen by many of the participants as an incentive to review approaches to measurement uncertainties in their laboratories and to promote training of staff. There was an agreement among the participants that ESARDA workshops dedicated on measurement uncertainty should be held in regular intervals.

Summarising a list of major issues that should be addressed in follow up workshops is given:

- Difficulties in defining sources of uncertainty for environmental swipe sample analysis
- Strengthen the link between operator and safeguards laboratories and evaluator needs
- Fit-for-purpose criteria - revision of the ITV document on as-needed basis
- Availability of matrix-matched 'real-life' bulk and particle reference materials

- Availability of reference materials for age-determinations
- GUM and GUM compliant approaches to estimate measurement uncertainty (time vs. effort)
- Training in metrological concepts
- Bridge the gap - joint workshop DA and NDA

Once more, the outcome of the workshop exceeded the expectations of the organisers with respect to participation, discussions and to meeting the workshop objectives. The present report is a further attempt of the WGDA to share the outcome of technical discussions and findings with a broader community using ESARDA as platform.
A first success of this workshop is that the next dedicated workshop will be a joint ESARDA DA/NDA/NA-NT workshop on '*reference materials needs and evaluation of measurement uncertainty in DA and NDA'* hosted by the European Commission Directorate General for Energy – Euratom Safeguards in Spring 2013 in Luxembourg. The focus of this workshop is on the exchange between reference measurement institutes, safeguards laboratories, operators, safeguards inspectors and evaluators defining the needs for standards/reference materials supporting DA and NDA instrument metrology and conformity assessment and their application in estimation of measurement uncertainty, including uncertainty in nuclear data in view of new approaches in safeguards [19, 20]. The workshop will be open to ESARDA WG DA, NDA and NA/NT members and a limited number of participants from expert and research institutes [21]. As a result of the workshop, we expect to increase the understanding based on metrological principles and the GUM between different approaches in uncertainty estimation for DA, NDA and in the evaluation of reported results [4, 5]. One aim of this workshop is to establish a priority ranking on required standards/reference materials and quality control tools. Emphasis will be given to the exchange between the inspectors in the field carrying out technical operations and the institutes/laboratories ensuring calibration of those instrumental systems. Therefore the ESARDA working groups appreciate very much that Euratom Safeguards (DGENER) is hosting the next dedicated workshop.

## Important web links:

This paragraph gives a list of relevant web-links to relevant international guides and training courses organised on metrology in chemistry and evaluation of measurement uncertainty

A) Guides:

- ITV2010:http://esarda2.jrc.it/internal_activities/WG-DA/STR_368_--_International_Target_Values_2010_for_Measurement_Uncertainties_in_Safeguarding_Nuclear_Materials.pdf

- JCGM 200: 2012 International vocabulary of metrology – Basic and general concepts and associated terms (VIM): http://www.bipm.org/en/publications/guides/vim.html

- The new EURATOM/CITAC guide: http://eurachem.org/index.php/publications/guides/quam

- JCGM 100: 2008 Guide to the expression of uncertainty in measurement - GUM: http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf

  http://www.bipm.org/en/publications/guides/gum.html
- JCGM 104:2009 Evaluation of measurement data — An introduction to the "Guide to the expression of uncertainty in measurement" and related documents

- JCGM 101:2008 Evaluation of measurement data — Guide to the expression of uncertainty in measurement Evaluation of measurement data — Supplement 1 to the "Guide to the expression of uncertainty in measurement" — Propagation of distributions using a Monte Carlo method

- JCGM 102:2011 Evaluation of measurement data – Supplement 2 to the "Guide to the expression of uncertainty in measurement" – Extension to any number of output quantities

- JCGM 106:2012 Evaluation of measurement data – The role of measurement uncertainty in conformity assessment

B) Training courses:

- TRAINMIC: http://irmm.jrc.ec.europa.eu/TRAINING/TRAINMIC/Pages/index.aspx

- Europe and Metrology in Turkey (EMIT) - Improving chemical and ionising radiation metrology in Turkey: http://irmm.jrc.ec.europa.eu/Turkey

- Use of reference materials and the estimation of measurement uncertainty: http://irmm.jrc.ec.europa.eu/events/Pages/121010_ref_mat_and_measure_uncertainty.aspx

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## 5. List of Acronyms

- **CRM** – Certified Reference Material
- **ES** - Environmental Sampling
- **GSMS** – Gas Source Mass Spectrometry
- **GUM** - •Guide to the expression of uncertainty in measurement
- **HKED** - Hybrid K-Edge / K-XRF Densitometry
- **ICP-(SF)MS** - Inductively Coupled Plasma - (Sector Field) Mass Spectrometry
- **IDMS** – Isotope Dilution Mass Spectrometry
- **ILC** – Interlaboratory Comparison
- **LA-MC-ICP-MS** - Laser Ablation Multi Collector Inductively Coupled Plasma Mass Spectrometry
- **(LG)-SIMS** – (Large Geometry) Secondary Ion Mass Spectrometry
- **NWA**L - Network of Analytical Laboratories
- **RM** - Reference Material
- **TIMS** - Thermal Ionisation Mass Spectrometry
- **WG NA/NT** - Working Group on Novel Approaches and Novel Technologies
- **WG DA** - Working Group on Standards and Techniques for Destructive Analysis
- **WG NDA** - Working Group on Standards and Techniques for Non Destructive Analysis
- **ITV** - International Target Values

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