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A MEASUREMENT EVALUATION PROGRAM TO SUPPORT NUCLEAR MATERIAL CONTROL AND ACCOUNTABILITY MEASUREMENTS IN BRAZILIAN LABORATORIES

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ABSTRACT

A measurement evaluation program (MEP) is one of a number of valuable tools that analytical chemists can use to ensure that the data produced in the laboratory are fit for their intended purpose and consistent with expected performance values at a given time. As such, participation in a MEP is an important indicator of the quality of analytical data, and is recognized as such by independent regulatory and/or accreditation bodies. With the intent to implement such a program in Brazil, in November 2012 the Nuclear Energy Commission of Brazil (CNEN), with support from the Department of Energy of the United States' (US-DOE International Safeguards and Engagement Program), decided to initiate a technical cooperation project aiming at organizing a Safeguards Measurement Evaluation Program (SMEP) for Brazilian facilities. The project, entitled Action Sheet 23, was formalized under the terms of the *Agreement between the US-DOE and the CNEN Concerning Research and Development in Nuclear Material Control, Accountancy, Verification, Physical Protection, and Advanced Containment and Surveillance Technologies for International Safeguards Applications*. The work, jointly performed by the CNEN´s Safeguards Laboratory (LASAL) and the New Brunswick Laboratory (NBL), has the objective to strengthen the traceability of accountability measurements and ensure adequate quality of safeguards measurements for facilities within Brazil, utilizing test samples characterized and provided by NBL.

Recommendations to participants included measurement frequency, number of results per sample and format for reporting results using ISO methods for calculating and expressing measurement uncertainties. In this paper, we discuss the main steps taken by CNEN and NBL aiming at implementing such a program and the expected results, in particular the impact of uncertainty estimation on the evaluation of performance of each participant laboratory. The program is considered by Brazilian safeguards authorities as an important tool for ensuring adequate Brazilian facilities' measurement performance, identifying areas within each laboratory needing improvement, and improving the traceability and reliability of safeguards measurements performed by Brazilian laboratories.

1. INTRODUCTION

An effective system for accounting and control of nuclear materials requires reliable accountability measurements. Quantities of nuclear materials must be determined with appropriate quality levels, so that reliable conclusions about the disposition of those materials (material in stock, transferred, processed etc.) can be drawn. When a system of accounting and control of nuclear material is subject to verification, routine results obtained by facility operator and by external independent verification entities must be compared. This is the case

of Brazil since it is a State subject to external safeguards of regional and international inspectorates of the Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials (ABACC) and International Atomic Energy Agency (IAEA), as formalized in the relevant safeguards agreement [1].

National and international organizations formally recognized the importance of measurement evaluation programs (MEP) as a mechanism to provide independent verification of the quality control system in measurement laboratories. In this context, the Brazilian Nuclear Energy Commission (CNEN) decided to organize a national MEP to evaluate if relevant Brazilian nuclear facility laboratories are conducting accurate and traceable nuclear material measurements. In order to use appropriate tools to conduct the program, in particular using adequate test samples and accounting on recognized data evaluation expertize, CNEN decided to establish a technical cooperation agreement with the Department of Energy of the United States (DOE), through the New Brunswick Laboratory (NBL). The NBL is the U.S. Governments' certifying authority for nuclear reference materials and provides measurement and measurement quality assurance services to DOE, commercial and international customers under international agreements.

The objective of the cooperation is to strengthen the traceability of accountability measurements and ensure adequate quality of safeguards measurements by implementing a safeguards measurement exchange program for facilities within Brazil, utilizing test samples provided by NBL and jointly measured by CNEN´s Safeguards Laboratory (LASAL) and the selected participant laboratories.

2. PARTICIPANT LABORATORIES AND SELECTED TEST SAMPLES

Some Brazilian laboratories already participate in safeguards MEP organized by ABACC, in cooperation with NBL, since they are members of the network of analytical laboratories that support ABACC as a regional safeguards agency. In fact, the cooperation allows for Brazilian and Argentine laboratories to join the safeguards measurement evaluation program (SMEP) that is periodically organized by NBL and includes other international laboratories [2].

The participants from Brazil in the NBL-SMEP program are laboratories that support regulatory, research and development activities rather than relevant fuel cycle facilities. Therefore, the focus of their participation in the NBL-SMEP is evaluation of performance in regards to analytical services provided to ABACC. The test samples they receive under the NBL-SMEP program are usually similar to the samples that are collected by ABACC inspectors during safeguards inspections in Brazil and Argentina.

In contrast, the intent of the MEP described in this paper is to cover Brazilian laboratories that provide analytical services to the nuclear industry. Therefore, they constitute critical elements in generating relevant nuclear material accountability data for existing and future nuclear facilities. The following laboratories decided to participate in the program:

- Laboratory for UF6 Isotopic Analysis at the Commercial Enrichment Plant
- Laboratory for UO2 Characterization at the Fuel Fabrication Plant
- Laboratory for UOC Analysis at the Uranium Concentration Plant

- Laboratory for Characterization of Nuclear Materials at the Experimental Center of the Navy

The Safeguards Laboratory of the Brazilian Nuclear Energy Commission is also participating in the program due to the work performed in support to nuclear regulatory activities in Brazil.

In regards to test samples used in the program, all of them were prepared and provided by NBL. LASAL received the samples, reported safeguards relevant accountability data and distributed the materials to the participants. The selection of the samples was done by each laboratory, based on the list of available samples provided by NBL, the type of measurement technique to be used and the types of materials they analyze in routine basis during normal production and process operations. The following list of samples was defined:

- Natural and Low enriched (3 and 4.7% U235) UF6, for isotopic determination
- Low enriched (4% U235) UO2 pellets, for enrichment and concentration determinations
- Natural U3O8 powder, for concentration and enrichment determination
- Natural U3O8 powder, for impurities determination
- Natural uranyl nitrate solution, for concentration determination

3. ANALYSIS INSTRUCTIONS AND REPORTING OF RESULTS

Prior to receiving test samples, all participant laboratories received a detailed set of analysis instructions. The laboratories were encouraged to handle the samples, measurement data and reporting in the same manner as “normal” samples. However, specific recommendations on results reporting were provided in order to allow the organizers to perform a comprehensive evaluation of the results.

Schedule: a maximum of 90 days was considered between the receipt of the test samples and the reporting of the results. Another 90 days were considered for data evaluation, preparation of the corresponding individual and general reports, as well as conduct of the final evaluation meeting.

Number of measurement runs: the laboratories were able to choose between one or two measurement runs within the period of analysis (up to 90 days). The measurement runs are usually conducted with 30 or more days in between. The intent is to evaluate the influence of different analysis periods. One test sample of each type has been sent for each measurement run. UF6 samples for enrichment determination constitute a particular case because a single ampoule can be analyzed several times by typical mass spectrometry techniques. Thus, several results can be reported for a single ampoule, covering two or more measurement runs. In general, samples for total uranium determination are totally consumed during a single measurement run.

Analysis scheme and reporting of results: two possible analysis schemes were considered and the participants were instructed to choose the preferred method, based upon their capability, reporting methods and schedule. Those labs that are ISO 17025 [3] accredited would typically choose the reporting system in place per 17025 requirements. In this case, the laboratory performs analyses and data evaluation based upon their own internal QA

manual/system, and submits a report including values, uncertainties and an uncertainty budget calculated from individual measurement results in accordance with ISO 17025 and/or JCGM 100:2008 (GUM Guide) [4] requirements. The laboratory is free to decide how many replicates for each sample, days of analysis and analysts are required. For those laboratory's that do not report uncertainty evaluation, it is suggest that each sample be analyzed a minimum of seven times, listing along with the result, the sample ID, aliquant number, date of analysis and analyst ID. If the laboratory desires an analysis of day-to-day variation in results, each sample should be analyzed on different days, with seven analyses on each day. A similar scheme should be considered for evaluation of analyst-to-analyst evaluation**.** This **s**cheme limits the evaluation to the sample preparation and measurement portion of the labs measurement system, and provides a limited, short-term examination of sources of variability.

4. DATA EVALUATION

Data evaluation in safeguards measurement evaluation programs aims usually at verifying the consistence of relevant statistical parameters, i.e. accuracy and precision, against well-established reference values. These values are currently published by the International Atomic Energy Agency (IAEA) as *International Target Values 2010 for Measurement Uncertainties in Safeguarding Nuclear Materials* [5, 6]. This latest version is commonly referred to as "ITV-2010". This document is extensively used by international and regional safeguards inspectorates and by analytical laboratories, in particular those labs that generate accountancy data subjected to external safeguards verification. The ITV´s-2010 are expressed as a two component system – designated as random and systematic – that result in a single uncertainty estimate (ITV) for each material (U and Pu) in different forms, concentrations and isotopic compositions and methods of analyses. The publication presents standard uncertainty values in the form of tables, grouped as follows:

- Bulk and Density Measurements
- Uranium Element Concentration Measurements (by Destructive Assay)
- Plutonium Element Concentration Measurements (by Destructive Assay)
- ^{235}U Abundance Measurements (by Destructive Assay)
- ^{235}U Abundance Measurements (by Non-Destructive Assay)
- Plutonium Isotope Assay of Pu and U/Pu materials
- Total Mass of ^{235}U (by Non-Destructive Assay)
- Total Mass of Pu (by Non-Destructive Assay)

For labs that are ISO 17025 accredited or are going through this process, the provision of results including a comprehensive uncertainty statement based on the GUM guide is strongly recommended. The GUM approach yields a single value for the uncertainty, and recommends the preparation of a "budget" table that describes the relative contributions of all known sources that make up the total reported uncertainty. It also establishes standard statistical methods for estimation and expression of uncertainties, conducting to the estimation of uncertainties from the traditional random and systematic components, as well as uncertainties from all other known sources (e.g., reference materials used for calibrations, contributions associated with temperature, day-to-day and analyst-to-analyst variations etc). The method allows a robust analysis of the reported result and appropriate pair comparison.

The ideal situation appears when a laboratory reports a reasonable number of replicates (seven or more in this MEP) per measurement condition, all of them being GUM compliant. In this case, it should be also possible to check the consistency between relevant uncertainty components as detailed in the reported budgets and variations calculated based on the sets of replicate results. If only a single result plus the corresponding uncertainty statement is reported, it may be difficult to do such consistency verification.

5. CONCLUSIONS

This safeguards MEP will support relevant fuel cycle Brazilian laboratories in the evaluation of their performance in regards to nuclear material accountability measurements. In their routine work, all laboratories produce measurement data that may be used to generate relevant safeguards declarations that may be subject to independent verification by national, regional and international organizations. Thus, the use of the ITV's as "state-of-the-practice" reference performance values is essential to link the program to the same standards that are used by the international safeguards community. In addition, at the end of the program the participants will have available important information to support the identification of areas where improvements are necessary.

From the point of view of the Brazilian nuclear regulatory authority, the program is a valuable opportunity to observe the consistency between the actual and the expected performance for each participant. The intent is to have a clear picture of the current status for each laboratory and make recommendations aiming at continuous improvement in the measurement performance, as necessary. The participation of a laboratory of the state regulatory authority in the program increases the confidence on the objectives of the MEP and creates a valuable technical exchange channel that benefits all involved organizations.

One focal point for the program is to explore the importance of adequate uncertainty estimation on the evaluation of the quality of measurement results. Analysis techniques have improved steadily due to advances in data analysis methods and through the use of "state-of-the-art" instruments. One possible consequence of these improvements is changes in the relative contributions of the various uncertainty sources that impact a specific measurement process. For example, in mass spectrometry measurements of fissile isotope abundances, the uncertainty contribution from reference materials (used to establish the traceability chain) is becoming an important contributor comparable to measurement uncertainties themselves. In consequence, the contribution of the reference material to the final uncertainty can no longer be ignored. The appropriate identification of the used reference material is also relevant due to possible correlation effects if different results are compared.

The procedures and methods used to calculate results and estimate uncertainties must be "transparent". This is facilitated if a laboratory uses standard methods and terminologies to calculate results and estimate uncertainties. It is clear that the GUM method helps ensure this. Additional training efforts may be necessary to help some laboratories in understanding and using GUM principles. The MEP must be capable of providing data evaluation outputs in compliance with GUM and support laboratories and analysts in case of non-compliance.

At the moment this paper was concluded, the MEP was in the phase of joint data evaluation by NBL and LASAL. As the program is intended to be completed by the end of 2013, in 2014

NBL and LASAL will jointly prepare an overview report evaluating all of the performance data for publication.

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