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The U.S. Support Program to IAEA Safeguards – Destructive Analysis Priority

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Abstract
The U.S. Support Program (USSP) to IAEA Safeguards priority of destructive analysis is aimed at strengthening the IAEA's ability to use destructive analysis as a safeguards tool. IAEA inspectors bring back nuclear and environmental samples from inspections, which are first cataloged by the IAEA and then analyzed by a network of laboratories located in many Member States and the IAEA's own Safeguards Analytical Laboratory in Seibersdorf, Austria. Historically, the USSP was instrumental in introducing environmental sampling techniques to the IAEA in order to enhance its understanding of material processing activities conducted at nuclear facilities. The USSP has also worked with the IAEA to improve understanding of measurement uncertainty and measurement quality, incorporate new and improved analytical methods, and purchase analytical and computer equipment. Recent activities include a temporary increase in analysis of environmental samples using secondary ion mass spectrometry and provision of a cost-free expert to restore secondary ion mass spectroscopy laboratory functionality and to modernize the IAEA's Safeguards Analytical Laboratory Information System.

Note: The above abstract was submitted just prior to a revision of this priority area by the USSP. The following discussion will address the revised priority now renamed "Analysis and Evaluation of Inspection Samples." The revised priority views destructive analysis (DA) and other analytical methods in the context of the entire end-to-end process from sample collection planning to evaluation of analytical results. This expanded view of the entire process yields a greater understanding of it so that the USSP can make better decisions for supporting the IAEA.

Analysis and Evaluation of Inspection Samples - Background and History

The IAEA Department of Safeguards relies on the IAEA's Safeguards Analytical Laboratory (SAL) at its Seibersdorf Laboratory and the associated Network of Analytical Laboratories (NWAL), located in many Member States, to analyze IAEA inspection samples in order to verify the correctness and completeness of a State's declaration. Each sample may be analyzed using one or more methods of destructive analysis and/or non-destructive analysis or assay (NDA) in an attempt to gain knowledge about the sample's composition, quantity, age, processing history, and other unique attributes. Safeguards inspectors collect samples, and Safeguards support divisions distribute them to SAL and the NWAL for analysis, afterwards evaluating the analysis results for input to Safeguards' verification reports. Keys to maintaining the effectiveness of inspection sample analysis and evaluation include judicious planning for sample collection (types and number of samples collected), improving measurement sensitivity and quality control, selecting and maintaining appropriate tools, controlling costs, and maintaining a highly qualified and efficient workforce.

Nuclear Material Sample Analysis

Analysis of nuclear material samples from declared nuclear facilities, in conjunction with traditional safeguards inspections, yields data essential to evaluating the correctness of a State's declaration in the operation of its nuclear facilities and nuclear material storage areas. Nuclear material samples are taken from process solutions and stored materials in facilities under IAEA safeguards. Measurement of these nuclear materials from declared facilities can provide precise information for bias defect assessments (calculations based on measurements of sufficient accuracy and precision to verify the total material balance in some bulk handling facilities).

Analysis techniques for nuclear materials are typically destructive in that the sample is not replaced once measured, and the samples themselves may be physically and/or chemically altered in the course of either preparing for the measurement or conducting it. The main techniques used by the IAEA for measurement of nuclear material samples include titration, coulometry, gravimetric analysis, mass spectrometry, and densitometry. Associated functions include using calibrated standards to verify NDA equipment, verification of operators' measurement equipment, overseeing quality assurance for on-site laboratory measurements, and preparation and validation of large-sized dried spikes for assay determination of liquid waste samples from reprocessing plants.

The USSP has funded many projects designed to improve the sensitivity and overall value of analytical methods, including training for IAEA staff and new equipment procurements. Past projects include automating a titration system, developing new methods to increase the detection level for radionuclides and reduce the associated waste, and provision of and training on a coulometry system with improved sensitivity. Current tasks include providing reference materials, improving hybrid K-edge densitometry methods and supporting IAEA participation in New Brunswick Laboratory's (NBL) program of sharing quality control samples for evaluation of the SAL's analytical methods.

SAL currently analyzes 95% of nuclear material inspection samples taken during IAEA inspections (about 800 per year), with little backup in the NWAL, resulting in an unacceptable level of risk. The IAEA is working with two laboratories to qualify them for nuclear material analyses, but currently there is no backup plan or other qualified laboratory in the event of a catastrophic shutdown of SAL or a sudden surge in the number of nuclear materials samples. The USSP, along with other Member States, elevated the IAEA's awareness of this potential single-point-of-failure problem during a workshop requested by the IAEA to address a proposed laboratory upgrade and held in November 2006. Consequently, the Department of Safeguards is undertaking a study to analyze how best to expand the NWAL in order to lower risk, maintain or improve quality and throughput, and do so at a manageable cost.

Environmental Sample Analysis

The environmental sampling program is a vital component of the IAEA's strengthened safeguards system. Analysis of environmental samples has been instrumental in detecting and identifying undeclared nuclear activities in countries such as Iran and North Korea. The United States and the USSP helped fund a number of activities and instruments central to the successful launch of this

program. The U.S. provided funds to build a clean laboratory at SAL where a variety of analytical techniques must be conducted.

Environmental samples are collected in and around nuclear facilities and other sites of interest. Inspectors have recently been returning between 600 and 800 samples a year to the IAEA. Samples are categorized as "bulk" or "particle." Environmental bulk samples are taken from process liquids or solids but are not part of the traditional nuclear material accountancy program at a given facility. They are measured as a whole sample. Particle samples, a majority of the environmental samples, are collected by swiping equipment, containers, floors, walls and other surfaces with a pre-packaged cloth. The cloth is then re-sealed to prevent cross contamination and sent to IAEA Safeguards for distribution and analysis. The cloth picks up residues of nuclear processes or other materials that have been handled in the sampling area. Environmental samples are first screened using high resolution gamma spectroscopy or x-ray fluorescence. Bulk sample analysis may also be conducted at higher sensitivity by thermal ionization mass spectrometry (TIMS) or inductively-coupled plasma mass spectrometry (ICP-MS). Particle analysis techniques employed by NWAL laboratories include scanning electron microscopy X-ray spectrometry (SEM-XRS), fission-track TIMS (FT-TIMS), or SIMS, in which individual particles are selected for elemental and isotopic determination.

The USSP has supported environmental sample analysis at the IAEA by providing equipment and training. This support includes training for clean laboratory procedures and provision of a SIMS instrument for SAL, which has been used successfully to analyze up to 120 samples per year. In 2007, the USSP also provided a cost-free expert (CFE) to restore the SIMS at SAL following the loss of two key SIMS personnel and to train new SAL staff charged with SIMS responsibilities. Currently, the USSP is funding a project to improve the sensitivity of sample screening using high-resolution gamma spectroscopy.

SAL is in the process of replacing its obsolete laboratory information management system (LIMS), which is used to record and maintain all sample information and analysis data. The USSP contributed to this effort by placing a CFE at SAL to manage the project. In addition, it has provided consulting support for the preparation and review of project planning and implementation documents.

Location-specific environmental sampling can identify activities at a particular facility, site, or other location, and is permitted under comprehensive safeguards agreements and at a wider range of locations under the Additional Protocol. Further, the number of complementary access (CA) inspections is increasing under the Additional Protocol, and inspectors are sending back new types of environmental samples (soil, vegetation, water, metal, air filters, etc.) that might require new analysis protocols and methods to detect elements of interest, in particular uranium and plutonium. The IAEA is increasingly interested in obtaining new or more sensitive instruments and techniques for measuring and quantifying the composition and concentration of minor isotopes and daughter isotopes of uranium and plutonium; and determining physical properties including color, grain size distribution, and microstructure. Recent requests to the USSP and other Member State Support Programs (MSSP) reflect these new needs.

In contrast to SAL's nuclear material sample throughput, SAL itself only performs analysis on 20% – 30% of the annual environmental samples provided by IAEA inspectors, as the NWAL has the capacity to perform most types of analyses for environmental samples. Nevertheless, the IAEA is still seeking to expand the number of laboratories and improve the overall quality and effectiveness of environmental sample analysis capabilities.

General USSP Priority Objectives

The U.S. Government has been offering extrabudgetary support to the IAEA for over 30 years. This support is in addition to dues levied on the United States to support the IAEA. Even so, these funds are limited and must be applied judiciously. USSP priorities are developed in line with both expressed IAEA needs as well as U.S. policy objectives. Strategies and tactical goals are established for each priority as a way to monitor and measure progress. The USSP reviews its priorities yearly to assess performance and updates them to keep them relevant. Updated priorities are shared with the IAEA and posted on the International Safeguards Project Office website at www.bnl.gov/ispo.

2008 USSP Analysis and Evaluation of Inspection Samples Priority

The USSP has traditionally funded inspection sample analysis, related training, the development and improvement of analytical methods, and the provision of instrumentation and expertise. The USSP has emphasized DA in referring to this priority when, in fact, it has been supporting other elements of inspection sample analysis processes. This revised USSP priority, now called "Analysis and Evaluation of Inspection Samples," takes a wider view than that of the previous priority called, "Destructive Analysis." The new priority includes DA, as well as other analytical methods used for inspection samples, many of which have been introduced or improved through USSP tasks. The priority includes pre-analytical processes, such as sample planning, preparation, and handling; post-analytical processes of evaluating sample analysis data; and cross-cutting areas, such as data maintenance and security, and quality management. All elements of the IAEA's inspection sampling program must be held to comparably high standards of quality, effectiveness, and efficiency in order to ensure its overall success. This expanded view should assist the USSP in viewing the entire program in context and aid it in prioritizing areas of support within the IAEA.

The USSP priority relates to the following IAEA projects, newly updated and revised for their publication "Research and Development Programme for Nuclear Verification 2008-2009." [1]

- SGTS-06: Destructive Analysis of Nuclear Materials for Safeguards
- SGIM-07: Environmental Sampling for Safeguards
- SGIM-08: R&D for Statistical Analysis

2008 Strategies for Analysis and Evaluation of Inspection Samples

Each year the USSP reviews and, if necessary, revises its priorities areas to reflect current U.S. policy and objectives for IAEA Safeguards. The USSP established the following strategy statements for 2008 this priority area of Analysis and Evaluation of Inspection Samples:

- Strengthen quality assurance measures in the collection and analysis of inspection samples through the provision of high quality reference materials and standards, participation in related measurement programs, and training to ensure competency in calculating and reporting measurement uncertainty.

- Support development or implementation of measurement techniques that reduce consumption of expensive and rare reference materials and reduce waste and contamination.
- Determine the requirements of SAL and the NWAL to maintain a viable and sustainable network of laboratories identify needs and gaps, and provide technical support as necessary.
- Support training for IAEA Safeguards and SAL staff and other technical assistance to IAEA and SAL to strengthen sample analysis and evaluation processes and help alleviate the affects of attrition due to age and term limits.

2008 Tactical Goals

Note: The following tactical goals are in no particular order or priority. Associated with each goal are performance measures that help the USSP track and monitor progress for each of the goals. Many of the tactical goals are ongoing activities and do not have a particular deadline associated with them. For most goals, a yearly review is sufficient to monitor progress and any particular difficulties that might be associated with them, if not already addressed.

A. Support the IAEA's quality assurance and sample screening missions. Provide U.S.-sourced certified reference materials for analysis from existing U.S. supplies and cooperative efforts to produce additional supplies or reduce the need thereof.

Performance Measures:

1) Facilitate provision of requested plutonium reference materials.
2) Facilitate cooperation in negotiations between the IAEA and Russia for the contract for separation of the plutonium reference materials.
3) Continue support of Task A.218, "Controlled Potential Coulometry of 3 – 5 mg Pu with SRL Coulometer" (a technique for determining concentration of a substance in solution). Consider the use of coulometry as a standard practice because it looks promising for significantly reducing the use of rare and expensive plutonium (Pu) reference materials, e.g., the IAEA's On-Site Laboratory in Japan.
4) Provide training in estimating uncertainty in measurements at the IAEA and SAL.
5) Support analytical quality control. For example, USSP-supported Task A.264, "Analytical Quality Control – Participation of SAL in NBL SME Program" at NBL distributes quality control samples to participating laboratories to analyze and compare results.
6) Complete Task A.268, "Improvement of NWAL Capability in Gamma Spectrometer Analysis for U/Pu and Fission and Activation Products."

B. Consider appropriate support for IAEA requests resulting from recommendations of the December 2006 SAL Infrastructure Upgrade Workshop and the Director General's SAL Study Group of 2007, among others.[2]

Performance Measures:

1) Continue periodic interagency meetings and meet with other Member States' representatives as necessary to discuss IAEA requests for assistance at SAL.

2) Assist the IAEA in strengthening the NWAL by supporting NWAL role evaluation and risk studies.
3) Study the feasibility of maintaining SAL's SIMS instrumentation and assist the IAEA in developing a reasonable life-extension plan to ensure SIMS capability until a new instrument is purchased.

C. Provide financial and human resources support to assist in the sample analysis and evaluation-related areas of Safeguards and SAL.

Performance Measures:

1) Consider placing a Junior Professional Officer (JPO) and a CFE in SGTS to support expansion of nuclear materials analysis at the NWAL and the risk analysis evaluation study now underway in the Department of Safeguards.
2) Continue support for the SAL LIMS project currently managed by a U.S. CFE. Other support provided under USSP task "Software Development Support: LIMS for the SAL."
3) Continue support for the U.S. CFE to train new environmental sample analysis evaluators and consider requests for updates of evaluation tools.
4) Provide clean laboratory training for new SAL staff.
5) Provide access to U.S. sources of training that can help existing SAL staff expand and improve their abilities.

Conclusions

The practice of establishing priorities has been useful in helping the USSP focus its attention on areas where U.S. support will have the greatest benefit to the IAEA. The focus of this priority was recently expanded from highlighting destructive analysis to include all analytical methods and incorporating a view of the entire end-to-end process from sample collection planning to the evaluation of analytical results. The USSP has traditionally supported the IAEA in most of these areas and continues to do so. This area is predicted to remain a high priority and the USSP will continue to support innovation and improvements to efficiency and effectiveness. The expanded view should offer a more balanced perspective and understanding of the inspection sample analysis and evaluation processes, which play an essential role in Safeguards assessments of States' adherence to their safeguards obligations.

References

[1] "Research and Development Programme for Nuclear Verification 2008-2009," International Atomic Energy Agency, 2008.

[2] "The Safeguards Analytical Laboratory: to 2010 and Beyond," results of the 2006 SAL Feasibility Workshop held at the IAEA, February 19, 2007.