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Collaboration of the NBL Program Office and the National Institute of Standards and Technology on the Development of Reference Materials for the Nuclear Materials Measurement Community

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ABSTRACT

Since 2017, the NBL Program Office has continued its role as a reference material producer (RMP) in accordance with the requirements defined in the ISO standard 17034 *General requirements for the competence of reference materials producers* [1]. The NBL PO has developed and implemented a quality system based on the ISO standard, and it collaborates with DOE's national labs and other collaborators for the actual laboratory measurements, which meet the requirements of ISO/IEC 17025 *Testing and calibration laboratories* [2]. Recently, the NBL PO entered into an interagency agreement (IAA) with the National Institute of Standards and Technology (NIST). The collaboration with NIST is designed to bring the NBL PO's certified reference material (CRM) development into close alignment with NIST's CRM planning, experimental design, data evaluation and certification practices. Implementation of the IAA involves NIST consultation and review of the NBL PO's planning and design documents for each CRM project. In some cases, the NBL PO will employ NIST's Statistical Engineering Division for measurement data analysis and uncertainty estimation. Several CRM projects are proceeding within the NBL PO – NIST collaboration. They include Pu isotopic standard C137A, a Pu metal standard to be produced in FY23, an update to the uranium assay value in CRM C112A and several nuclear forensics materials. Details of how the NBL PO is interacting with select national labs for these projects, the interactions with NIST will be described in this presentation.

INTRODUCTION

In the mid-1980s, the National Bureau of Standards, now the National Institute of Standards and Technology (NIST), transferred all of its special nuclear certified reference materials (CRMs) to the New Brunswick Laboratory (NBL). Up until 2014 NBL maintained and distributed these materials and produced new materials using its own expertise, its specialized laboratory

facilities, and by joint efforts with one or more of DOE's national laboratories. Because of facility limitations, the NBL's actual laboratory effort was discontinued, and the new NBL Program Office (NBL PO) was established to continue the maintenance and development of special nuclear CRMs.

As the new Program Office, NBL PO set out to redefine its role as a reference material producer (RMP) to be in compliance with ISO 17034 and to closely align its approach to reference material production, characterization and certification designs with that of NIST.

CONFORMANCE WITH ISO 17034

As an RMP within the framework of ISO 17034, NBL PO is ultimately responsible for all aspects of reference material development, but can use the expertise and facilities of qualified collaborators (called "subcontractors" in the standard) to perform some specific functions. Overall steps in the RM development process include:

- Planning
- Material production
- Characterization
- Data analysis, value and uncertainty assignment
- Documentation

The details of these steps have been described previously [3]. Among these details, the ISO standard precludes an RMP from subcontracting the following:

- production planning
- selection of subcontractors
- assignment of property values and their uncertainties
- authorization of values and uncertainties, such as designating values as "certified"
- authorization of reference material documents, such as certificates or product information sheets.

PLANNING

The NBL PO addresses the planning phase of RM development by defining material production requirements and characterization requirements. Material production encompasses identifying the source material and how it is to be processed, packaged and stored. Most often assessing the homogeneity and stability of the material is part of the production phase, but it may also be incorporated in the material characterization effort. Characterization requirements define what measurands are to be determined, the target accuracy and precision of each, the specific analytical methods to be used, and the data reporting format. These requirements definitions, which may be separate documents or combined into a single document, are communicated to prospective collaborators to begin finalizing the plan.

SELECTION OF AND INTERACTION WITH COLLABORATORS

NBL PO chooses potential collaborators for performing the actual production process and characterization measurements based on the following:

- Known capabilities from past interactions
- Evidence of operating within a quality system in conformance with ISO/IEC 17025 with a scope that covers the required activities. Formal accreditation to the standard is especially desirable, and will be a requirement in the future.
- Results from proficiency testing exercises such as
    - NBL PO Safeguards Measurement Evaluation Programs
    - IAEA Nuclear Material Round Robins

Once identified for specific functions as defined in the requirements documentation, collaborators are invited to perform specific functions that address the defined requirements. Potential collaborator laboratories then respond by producing detailed material production and/or characterization plans as appropriate. The requirements documents and labs' documented plans form the basis of a dialog between the NBL PO and the labs to finalize how the RM will actually be developed.

ALIGNMENT WITH NIST

To begin the process of transitioning from a lab-centric RMP to a program office, the NBL PO first developed and implemented its quality system based on ISO 17034 and some specific criteria used by NIST in the certification of its Standard Reference Materials.

NIST's current approach to certification [4] and its relevance to the NBL PO are summarized in Table 1. Note that combinations of these modes are often used. The relevant NBL PO approach always relies on Mode 8, which involves the expertise and laboratory facilities of selected collaborators.

Table 1. NIST modes of certification and their relevance to the NBL PO

	NIST Mode	Notes	Relevance to NBL PO
1	Single higher order NIST method	A published reference measurement procedure of proven accuracy	NBL PO has identified select higher order methods that can be used for certification.
2	Two or more independent NIST methods	Differences in physical, spectroscopic, or chemical preparation	
3	One NIST method and others from collaborators	Method independence is still important.	
4	An interlaboratory study (ILS)	Suitably designed to allow a reliable	Could be used in cases where NBL PO designed the ILS and performed its own data analysis.

		assessment of the consensus value	
5	Value transfer from an existing SRM or primary standard	NIST is not specific here, but examples are value transfers performed by NIST on its own standards.	
6	Operationally defined measurands	The measurand is defined by the result of a well-documented procedure.	
7	Values defined by international convention	Examples are the stable isotope ratio materials developed with IAEA.	
8	Need driven exceptions to the above, involving expert collaborators outside of NIST	Occurs when NIST does not have the expertise or facilities to perform the required measurement(s)	This mode is the relevant foundation of NBL PO's approach.

INTERAGENCY AGREEMENT

To further align its approaches to development and certification of its CRMs with those of NIST, the NBL PO entered into an interagency agreement with NIST in November 2021. The terms of the agreement call for NIST to consult and review and consult on the NBL PO requirements documents, the resulting production and characterization plans, and the statistical data analysis performed by NBL PO for each of its CRM projects. Technical reviews and suggestions are provided by NIST's Chemical Sciences Division (CSD) and its Radiation Physics Division (RPD). Statistical design and analysis consultation is provided by NIST's Statistical Engineering Division (SED). The overall plan is to submit the requirements and plans documentation for NIST review at the start of each project. In some cases, SED may be consulted at an early stage regarding sampling plans for homogeneity assessment and characterization designs.

The NIST review process examines the selection of collaborators, the production of the candidate material, homogeneity and stability assessment, packaging, characterization methods, and the data analysis. Each of these steps in NBL PO's project is assessed relative to the requirements if ISO 17034, ISO/IEC 17025, and any other pertinent NIST practice.

PROJECTS COVERED BY THE IAA

A few NBL PO CRM projects were already underway at the time of the IAA signing, so these were reviewed by NIST after two of the projects were completed and one more is ready for NIST review. These are listed in Table 2.

Table 2. NIST independent review

Project	Intended use	Progress
^{231}Pa spike solution	Dopant for a ^{231}Pa/^{235}U chronometer	Reviewed and concurred by NIST
^{229}Th spike solution	Spike for Th assay by IDMS for a ^{230}Th/^{234}U chronometer	Reviewed and concurred by NIST
^{233}U spike nitrate salt	Spike for U assay and isotope ratios for the chronometers	All documents completed; to be submitted to NIST review

As reported in our companion paper [5], there are several other CRM development projects underway that will follow the development and NIST review process described here.

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