

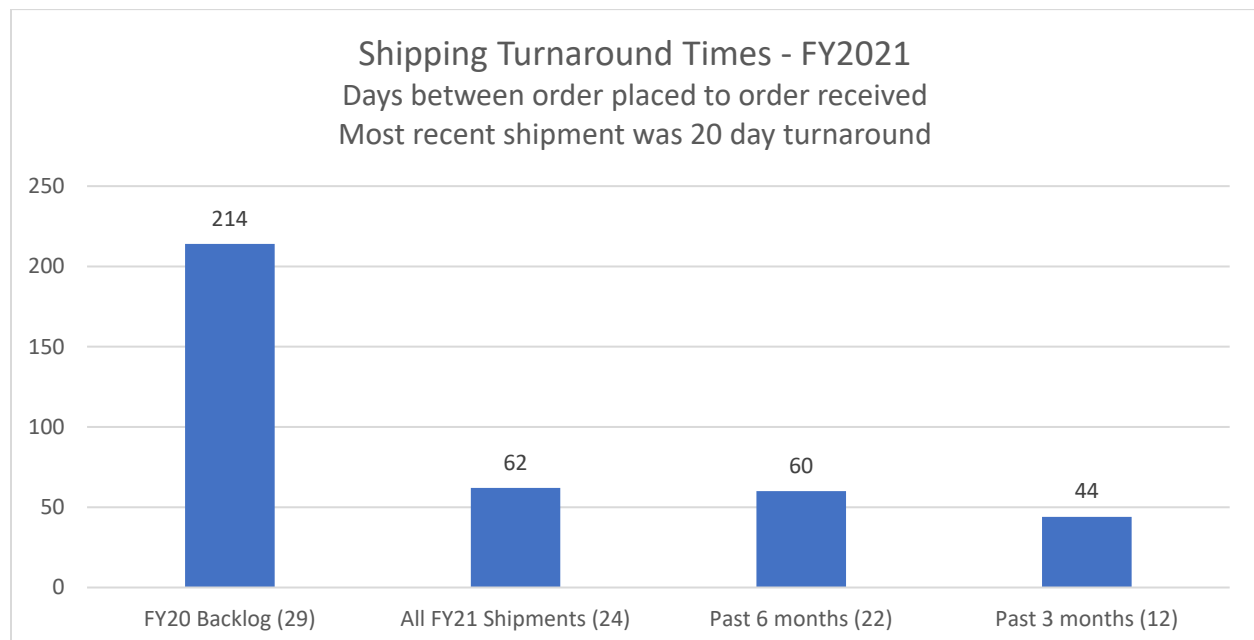
NNSA's NBL Program Office – Plans for Ensuring Uranium and Plutonium Measurement Quality

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The NBL Program Office (NBL PO) is the US certifying authority for special nuclear reference materials. The NBL PO mission transferred from the Department of Energy's Office of Science to the National Nuclear Security Administration beginning in FY2020. This talk will present the current status of the US nuclear reference material inventory, discuss shipping and storage capabilities and remaining challenges, review material needs collected from US and other customers, present the status of several CRM projects currently underway, and discuss the current plans for producing new uranium, plutonium and other CRM's. Additionally, the NBL PO's proficiency testing program (Safeguards Measurement Evaluation Program) will be reviewed with discussion on the status and plans to revitalize the program with new materials and operating approaches.

Sales, Shipping & Inventory Assessments:

A new staff member was added to the NBL Program Office in FY21, focused on sales, shipping, inventory and financial management and project planning/tracking. The backlog of CRM orders associated with transition from former NBL building 350 to Y12 has been completely processed. All NBL PO materials are now located in NBL PO's three locations at Y12 and at Los Alamos National Laboratory with the exception of UF6 materials currently at the Portsmouth site. With the completion of the material moves and clearing of backlogged orders, the turn-around time for shipments from Y12 has dramatically improved, even with COVID-19 impacts to site operations. Currently our metric for order turn-around time includes the time it takes for the receiving site to authorize the shipment, which has greatly distorted some of the data due to extended delays in receiving authorization to ship from several sites. The chart below shows the improvement in turnaround times, particularly in recent months.



The FY20 Backlog of orders with a 214-day turnaround that were shipped in FY21 include several Type B shipments that were greatly delayed, and six shipments of significant quantities of C116A HEU and C112A NU metal to JRC-Geel that were ordered and shipped under contract and met customer delivery deadlines. Shipping turnaround times between domestic and international orders are essentially the same (i.e. no significant difference).

NBL PO, as part of the needs assessment discussed below, completed a formal inventory assessment of all materials at our DOE sites and identified specific actions for those materials requiring additional effort or evaluation. This included identifying those needing updated packaging/labeling, estimated remaining inventory lifetime based on previous 10 years sales volume, reviewing the status of long-term stability evaluations of several materials (e.g. dry nitrates) requiring further verification, evaluation of items for replacement or replenishment, and identifying those materials that may be candidates for discard. This assessment also included historical Safeguards Measurement Evaluation Program samples. Finally, the NBL PO evaluated the inventory to ensure that a sub-set of the most important CRMs and SMEs are established at a second storage location, independent from the Y12 site. This will ensure that if there is an event that prevents Y12 from distributing NBL PO materials for an extended period of time the second site will be able to meet urgent needs in a timely manner.

The results of the inventory assessment were incorporated into NBL PO program planning, along with compiling needs assessments conducted in FY19 and FY20 for select customers. Currently underway activities include repackaging of C123 and C116A HEU units, retrieving bulk C116A materials from a production area and packaging for safekeeping for future use and production of new 2 gram units of C112A NU metal.

There still remain a number of UF6 cylinders containing CRM and SME materials at the Portsmouth site. Portsmouth, in cooperation with Y12 and ORNL, is preparing to transfer the UF6 into new cylinders due to hydrostatic testing issues and to ship the materials to the UF6 storage and handling lab being established at ORNL. The NBL PO expects the transfer of these materials to occur in early FY22.

The NBL PO, Y12 and JRC-Geel have identified excess US CRM and base materials to be returned to NBL PO possession. We hope to execute the first of several shipments within the calendar year, and planning is underway. These materials include a variety of U-series isotopic standards, some plutonium materials, and base material that was used to produce the CRM 969/EC171 gamma standards.

Programmatic Activities

In general, for the near future the NBL PO focus will be to work with DOE laboratory's to establish the infrastructure and methods needed for the mission, to update our aging inventory of reference materials (low inventory, outdated certified values), add key materials that are clearly lacking a "primary" CRM, or to address a clear measurement need in support of DOE and other Agencies. The NBL PO continues to actively participate in a variety of international standards bodies including ASTM, ANSI and ISO committees. The NBL PO recently conducted the first

of a recurring series of 'Brown Bag Lectures' on metrological traceability, and will continue with topics of interest to our customers and collaborators. The NBL PO will also continue to support individual laboratories in their efforts to develop working reference materials and quality control materials (QCMs) to address site-specific needs.

The NBL PO is completing a survey of key DOE laboratory collaborators related to ISO 17025 accreditation and particularly what methods are currently accredited. The NBL PO will compare key methods listed in our modes of certification to those at our key DOE collaborators and will work to ensure that methods utilized for future NBL PO certification efforts meet the requirements of our Quality Management System and ISO 17034 and 17043.

As part of the effort to relocate UF₆ materials from Portsmouth to ORNL to enable NBL PO to resume UF₆-related CRM and proficiency testing activities, the NBL PO has funded ORNL for refurbishment of a dedicated lab space for UF₆ sampling and handling. This includes the purchase and installation of a three sampling manifolds. The NBL PO plans to return UF₆ to the proficiency testing program in late FY22 and resume sales of select CRMs in this chemical form.

The uranium high precision titration (HPT) laboratory at ORNL continues to operate and provide highly accurate and precise uranium assay measurements. A second chemical laboratory is nearing completion of a floor to ceiling renovation. This should be complete in FY21 and equipment purchases and installation will commence in FY22. The NBL PO and ORNL are also funding additional laboratory space to serve as storage, plutonium and uranium handling, and possible additional instrumentation related to the NBL PO mission. These labs are expected to be used for preparation of uranium calibration mixes for use in producing new, highly accurate uranium isotopic materials as CRM's and a new suite of proficiency testing materials. Additionally, the NBL PO is collaborating with ORNL chemists to evaluate the traditional Davies and Gray titrimetric method using direct potassium dichromate calibration in order to improve the traceability of the method to make it suitable for CRM characterization efforts.

The SRNL has been funded to establish HPT capability in FY22. Having two sites capable of performing this key method will ensure continuity in primary uranium assay measurements.

The NBL PO and NIST are currently processing an Inter Agency Agreement (IAA) related to technical support and review and assessment of NBL PO operations and projects. The IAA is currently being reviewed by Department of Commerce attorneys and is expected to be implemented in late FY21/early FY22. This agreement includes support from NIST's Chemical Sciences Division and Statistical Evaluation Division, in addition to the Radiation Physics Division. The IAA will provide NBL PO an independent expert evaluation of future NBL CRM projects to ensure compliance with the fundamental principles of CRM production, provide statistical expertise to NBL, and ensure our CRM's and proficiency testing programs meet the high standards of a national metrology institute. As part of the IAA, the NIST will assess NBL PO operations for compliance with ISO 17034, and the NBL PO plans to seek commercial accreditation to the 17034 and 17043 standards in the near future.

CRM Projects

The recent validation of SRM 136f dichromate standard for use in NBL PO reference material production efforts will allow the NBL PO to re-issue the CRM 112A natural metal Certificate of Analysis. The uranium assay value, originally certified by NBS as SRM 960 by uranium coulometry, will not change as the two methods are in nearly perfect agreement. However, the NBL PO will update the uncertainty of the assay value to comply with JCGM 100 (“GUM” guide), and ensure the Certificate meets the updated requirements of ISO Guide 31 “Contents of Certificates”.

Other CRM projects currently underway or planned include the recertification of CRMs 136, 137 and 138. The LLNL will commence unit production of C137A materials in September, and a number of DOE laboratories are working with NBL PO collaboratively to develop measurement plans leading to the recertified plutonium isotopic values. In FY22, the remaining C136 and C138 materials will be produced, and recertified in an identical manner.

The NBL PO is currently planning and has initially funded the production of a neptunium assay CRM, a new DU metal CRM to replace low stocks of CRM 115 DU metal, and is working collaboratively with a DOE laboratory to develop uranium radiochronometry standards. Additionally, the NBL PO is working with Los Alamos National Laboratory on a replacement for plutonium metal standard C126A, to be produced in FY22-FY23.

Additional materials that have long been identified as an important need due to a lack of any CRM include thorium and americium. The NBL PO will have more to communicate regarding these materials next year. As stated above, an update to the uranium isotopic series (eg NBS U-series standards) is long overdue, and NBL PO and ORNL are performing the necessary development and infrastructure upgrades to allow this effort to begin. The production and certification of uranium isotopic standards will be a multi-laboratory effort that relies on both chemistry and mass spectrometry expertise from a number of DOE labs. Initially, the NBL PO focus is on establishing a few key chemistry capabilities and ensuring the necessary infrastructure is in place to allow for the handling of end-members and production of calibration mixes. These materials will also be utilized to update the NBL PO proficiency testing program.

Proficiency Testing Program

The Safeguards Measurement Evaluation Program (SME) suffered serious delays and lack of attention due to the relocation and reorganization of the NBL Program Office in 2019 and 2020. The NBL PO has initially modified the SME program to comply with many of the requirements of ISO 17043, including a significant change to how NBL PO instructs labs to analyze material and the data evaluation processing by NBL PO upon submission. In FY21 some low inventory materials have been replenished and the NBL PO has begun shipping samples to customers. The NBL PO funded production of custom size C115 DU metal samples for proficiency testing and method validation at a European laboratory, and replenishment of historical NBL SME samples needed to maintain program operations until we are able to produce entirely new materials. In addition, a new 2% enriched material, ME013, has been produced for isotopic proficiency testing and will be included next year, along with the return of UF₆ materials. The NBL PO has also begun discussions with several NNSA program offices regarding the importance of proficiency testing and plans to include more formality in participation in the future. In FY22, the NBL PO will develop a program plan for proficiency testing and begin identifying key materials needed to

update the current inventory of materials and to provide more value to customers and address specific measurement challenges.

Conclusion

The transformation of the former NBL to the NBL Program Office continues. We have made significant strides in transferring key methods and developing dedicated infrastructure. We will continue to work closely with the DOE national laboratories as we shift our focus from improving storage, shipping and related inventory management to updating our current CRM materials and developing new materials to meet the needs of the many DOE, other US government programs and international collaborators who regularly utilize nuclear reference materials for method calibration, development and quality control.