

**U.S. DEPARTMENT OF ENERGY
MEASUREMENT EVALUATION
PROGRAM MEETING MINUTES**

JW Marriott Starr Pass Resort, Tucson, Arizona.

JULY 7, 2007

Organized by

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TABLE OF CONTENTS

NBL: HISTORY AND MISSION..... 1

INTRODUCTION 2

LIST OF ATTENDEES..... 3

AGENDA 5

SUMMARY 6

ACKNOWLEDGEMENTS 9

ABSTRACTS AND GRAPHICS 10

NBL: HISTORY AND MISSION

The New Brunswick Laboratory (NBL) is owned and operated by the United States Department of Energy through the Office of Science, Chicago Office. The laboratory was established in 1949 as an analytical chemistry laboratory in New Brunswick, New Jersey to provide support to the United States Atomic Energy Commission. At that time, it was staffed by scientists from the National Bureau of Standards who had contributed significantly to nuclear material measurement programs in the Manhattan Project. At the New Brunswick Laboratory, they provided the technical expertise and skills to solve problems related to quantitative analyses of uranium-bearing materials. Over the years, these scientists and others following them have expanded the capabilities of the laboratory to include chemical and mass spectrometric analyses of plutonium and other trans-uranium elements, research and development activities in chemical analyses techniques, preparation of certified reference materials, and operation of the nuclear safeguards measurement evaluation program. In 1977, the laboratory moved from New Jersey to its present location at the Argonne National Laboratory site in Illinois.

The major mission of the New Brunswick Laboratory is to provide technical assistance to the Department of Energy in the following areas: measurement evaluation program operation, certified (nuclear) reference materials preparation, measurement techniques development, and actual measurements of special nuclear materials. In addition to fulfilling these tasks, the laboratory helps the Department in three other areas: conducting technical audits, resolving shipper/receiver differences in material transfers, and assisting in nuclear nonproliferation programs within the United States and internationally.

INTRODUCTION

The New Brunswick Laboratory (NBL) Measurement Evaluation (ME) Program was initiated in 1985 to assess and evaluate the adequacy of measurement technology as practiced in Department of Energy (DOE) facilities for nuclear materials accounting. Later on, laboratories outside the DOE complex were allowed to join the program on a cost-recovery basis. The current program serves the DOE facilities as well as international laboratories, and it consists of two parts: the Safeguards Measurement Evaluation (SME) program for the evaluation of destructive analyses results of uranium and plutonium, and the Calorimetric Exchange (CALEX) program for the evaluation of non-destructive analyses results of plutonium.

In the SME program, participants analyze uranium and plutonium test samples for elemental content and isotopic abundance. The test samples are made from certified reference materials and/or other well characterized materials (e.g., working reference materials). Participants analyze the test samples and submit the results to NBL for evaluation of bias and precision. Participants receive feed back on performance through evaluation reports.

In the CALEX program, participants analyze plutonium oxide contained in two different working reference material standards (known as Calex I and Calex II) for heat output, and plutonium and ²⁴¹Am isotopes abundances. NBL evaluates the measurement results for bias and precision as in the SME program. In addition, two other quantities (effective specific power and plutonium mass) calculated from the measurement results are also evaluated. Note that the main goal of this program is to assess performance in plutonium mass determinations. Participants receive feed back on performance through annual reports.

NBL conducts a meeting once a year to discuss the progress made in the measurement evaluation program and to hold discussions on topics of interest to the participants; new and/or modifications to existing measurement techniques, new initiatives such as environmental/forensic analyses, performance evaluation methods including uncertainty estimation, need for new test material etc. The annual meeting is usually held a day prior to the start of the International Nuclear Material Management (INMM) annual meeting and at the same venue. This year the measurement evaluation program annual meeting was held on July 7th at the JW Marriott Starr Pass Resort in Tucson, Arizona.

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AGENDA

Safeguards Measurements	9:00 AM	Opening remarks	Jon Neuhoff, NBL
		SME Program	B. Srinivasan, NBL
		New database development	B. Srinivasan, Michael Soriano, Joe Waggoner* and Ron Kuziel, NBL
	9:30 AM	Validation of the Automated Davies Gray Procedure Results and Conclusions	Helmuth Aigner, Steven Balsley, Josef Berger, Norbert Doubek* and Alfred Zoigner, IAEA
	9:55 AM	ABACC activities in the field of DA measurements	Erwin G. Galdoz, ABACC
	10:20 AM	TIMS vs. ICP-MS, an update on work performed at INL-MFC	Andrew Maddison, INL
10:45 AM - 11:00 AM Break			
Low-Level Measurements	11:00 AM	NBL's environmental initiative: Tracing nuclear activities and signatures	Stephan Vogt, NBL
Standards	11:25 AM	News on uranium isotope reference materials and measurements at IRMM	Stephan Richter*, A. Alonzo-Munoz, R. Eykens, U. Jacobsson, H. Kuehn, A. Verbruggen, R. Wellum, IRMM
	11:50 AM	Including Particles as a form of standard materials in the NUSIMEP campaigns: Some reflections	R. Wellum* and R.Kips, EC-JRC-IRMM
Laboratory Management	12:15 PM	SRS automated QC charts for accountability methods	Clint Gregory* and Hasan Syed, SRS
12:45 - 2:30 PM Lunch			
CALEX	2:30 PM	Calex 1 and Calex 2 Materials Characterization	B. Srinivasan*, Usha Narayanan and Kattathu Mathew, NBL
	2:55 PM	Dissolution concerns for Pu assay	Laurie Walker, LANL
Calorimetry	3:20 PM	An examination of the benefits of final power prediction algorithms employed in current AWE calorimeter systems and a possible approach to the production of new heat standards	Steve Holloway, AWE
	3:45PM	Error analysis of FRAM gamma-ray isotopic analysis data	Tom Sampson*, T.L.Burr and D.T. Vo, LANL
4:10 PM - 4:25 PM Break			
Calorimetry	4:25 PM	Calorimetry at LANL	Peter Santi, LANL
Measurement Uncertainty	4:50 PM	Measurement Assurance Program (MAP) from the view point of the GUM	Raghu Kacker, NIST
	5:15 PM	Bayesian uncertainty analysis for radiometric applications using WinBUGS	William Guthrie, NIST
	5:40 PM	The role of correlation in uncertainty evaluation	Fabio Diaz, LASAL

*Speaker

SUMMARY

Session 1

Jon Neuhoff of NBL welcomed the attendees. Chino Srinivasan of NBL provided an overview of the SME program. Joe Waggoner of NBL described the new Safeguards Measurement Evaluation System (SMES) that will replace the present FoxPro system for evaluating SME and CALEX program measurements results. The new system is web based; it will permit direct input of measurement results by the participants, and will also allow direct retrieval of evaluation reports.

Norbert Doubek of IAEA presented a report on uranium elemental determination using an automated Davies-Gray titration procedure – an auto titrator coupled to a “robotic” arm. The new procedure was qualified using test samples supplied by NBL. Apparently, the procedure yields results with acceptable levels of bias and precision, but suffers from an unacceptably large number of failures. The development work on the new procedure continues.

Erwin Galdoz of ABACC delivered a talk on destructive analysis and measurement evaluation programs in the ABACC network laboratories. His talk included a description of a simple and elegant sampling method of UF_6 - through adsorption on alumina pellets - for isotopic abundance analysis.

Andrew Maddison of INL compared the TIMS and ICP-MS results of uranium and plutonium isotope abundance determinations. This comparative study has enabled INL to shift the work load from TIMS to the faster ICP-MS method for many different types of samples.

Session 2

Stephan Vogt of NBL provided an account of the development work in progress for a new initiative on environmental and forensic analyses. NBL will be able to provide new types of certified reference materials for ultra-low level analyses and conduct a measurement evaluation program to support the new initiative.

Stephan Richter of IRMM presented a talk on the production of a suite of uranium isotope reference materials for (mass spectrometer) instrument calibration. They were made by mixing

materials of known purities and isotopic compositions, the latter verified experimentally using “high precision” thermal ionization mass spectrometry.

Roger Wellum of IRMM spoke on the need to produce uranium oxide particles of well defined density and known isotopic abundance for quality control in the analyses of environmental and forensic samples. IRMM is developing techniques to produce these standards and distribute them in the next NUSIMEP campaign.

Clint Gregory of SRS delivered a talk on the development of a new software system to monitor, review and communicate quality control results in nuclear material accountability measurements. SRS personnel find the new system to be quiet efficient for information exchange and timely communication among laboratory personnel (bench chemists, laboratory management, contractor staff and DOE management).

Session 3

Chino Srinivasan of NBL presented a review of destructive analyses results of plutonium oxide in the Calex I and II standards. These results were obtained several years ago, but formal reports of analyses have not been issued. This deficiency is being corrected. A formal report was issued earlier this year for Calex II, and the results in that report for plutonium assay, plutonium isotopes and ^{241}Am abundances were presented in this talk. A similar report is under preparation for Calex I.

Laurie Walker of LANL discussed the importance of achieving complete dissolution of materials in the quantitative analysis of uranium and plutonium. She described several techniques available for dissolution of different types of samples.

Steve Holloway of AWE provided an account of the final power prediction algorithms in use at AWE to reduce calorimetry assay time. He also described AWE plans to produce new heat standards utilizing well-characterized plutonium metal.

Tom Sampson of LANL reviewed 15 years of FRAM gamma-ray isotopic analysis data (for plutonium and ^{241}Am isotopes) obtained from a combination of a variety of germanium detectors and data acquisition systems. The gamma ray results were compared with corresponding results from mass spectrometer measurements. The information presented in this talk is pertinent to uncertainty estimation in isotopic abundances in calorimetry.

Session 4

Peter Santi of LANL gave a review talk on LANL contributions to calorimetry; a) maintaining a Pu-238 heat standards laboratory, b) new modeling capability to simulate the performance of a calorimeter, and c) development of LVC (Large Volume Calorimeter) for measuring plutonium in 55 gallon drums.

Raghu Kacker of NIST spoke on the “measurement assurance program from the viewpoint of GUM”. He pointed out that the uncertainty estimation based on the “Guide to the Expression of Uncertainty in Measurements” is different from traditional methods, and it is essential to introduce the GUM method for calculating uncertainties in measurement assurance program results.

William Guthrie of NIST compared uncertainty estimations of radiometric measurement results from Bayesian analysis with those of the GUM method. His study showed essentially identical results from the two different evaluations.

Fabio Diaz of LASAL delivered a talk on the importance of recognizing correlations while estimating the combined uncertainties in measurements. Ignoring correlations leads to incorrect estimation of uncertainties.

ACKNOWLEDGEMENTS

The organizers of the annual meeting thank the speakers for the talks, and the attendees for active participation during discussion and question/answer sessions. The meeting lasted a full hour beyond the scheduled closing time; the courtesy and patience shown by the attendees during the extended hour are very much appreciated.

The organizers thank Ms. Leah McCrackin of INMM for excellent arrangements of the meeting room, Nancy Hui of NBL with help in coordinating the printing of the minutes, and Louise Kickels, Michele Nelson, and Gary Weidner (from Argonne) for printing and producing electronic copies of the minutes.

ABSTRACTS AND GRAPHICS

The abstracts and graphics (slides, pictures etc.) used in the presentation of talks at the 2007 ME Program Meeting are included in the following pages. They are given in the same order as in the agenda. Note that no graphics were used in the introductory remarks by Jon Neuhoff.

Abstracts and Graphics are linked from the Agenda Bookmark

**Measurement Evaluation Program Meeting
July 7, 2007
JW Marriott Starr Pass Resort, Tucson, Arizona**

ABSTRACTS

9:05 AM SME Program, B.Srinivasan, NBL

The routine tasks in the Safeguards Measurement Evaluation (SME) Program are a) preparation and characterization of test samples, b) distribution of test samples to participants for analyses, c) preparation and distribution of the statistical evaluation reports of the test samples analyses results, d) preparation and distribution of annual evaluation reports, e) hosting the annual meeting, and f) preparation and distribution of the minutes of the annual meeting. This year, three other tasks were added: i) develop a new database, ii) expand the program to include new participants, and iii) administer the safeguards assistance and non-proliferation program. The progress made in performing these tasks will be discussed with a critical assessment on timely performance.



Measurement Evaluation Program Annual Meeting

SME Program Highlights
July 7, 2007
Tucson, AZ.

B. Srinivasan

New Brunswick Laboratory/Office of Science/U. S. Department of Energy



CY 2007 Participants

DOE	NRC	International
INL	*USEC , Paducah	ABACC (Argentina and Brazil)
LANL	*USEC, Portsmouth	IAEA, Austria
NBL	NFS, Erwin	IRMM, Belgium
ORNL		NMCC, Japan
SRS		*Plutonium Fuel fabrication Facility, Japan
Y-12		*Tokai Reprocessing Plant, Japan
		URENCO, England

* These facilities are expected to participate in the second half of CY 2007
or in CY 2008

New Brunswick Laboratory/Office of Science/U. S. Department of Energy



Available Uranium Test Samples

UNH solution (assay and isotopes)

- natural uranium, LEU and HEU

UO₂ pellet (assay and isotopes)

- about 4% enriched

UO₃ powder (assay and isotopes)

- about 0.8% enriched
- re-characterization in progress; moisture absorption

UF₆ (assay and isotopes)

- range of enrichment - depleted to 5%
- characterization work not yet completed; awaits safety reviews and approval to restart work with UF₆ laboratory work will begin after completing safety reviews of experimental procedures



Plutonium Test Samples

Dried plutonium sulfate

- Very few available for IDMS and isotopics
- Need to replenish supplies
- Pu laboratory work expected to start in 2008
- DSA needed for restart and under preparation



New test samples

UNH solution for assay and isotopes (natural uranium)

- 20 g samples with 10 mg uranium/gram
- Characterization completed in CY 2007- 2Q

Uranium isotopic test samples

- Mix end members with different levels of enrichment
- In planning stage

Dried plutonium sulfate for assay and isotopics

- Will be made after Pu laboratory work resumes

Coulometry test samples

- Sufficient interest; coordinate work with SRS and LANL

Chronometry (e.g., $^{232}\text{U}/^{228}\text{Th}$)

- Test samples under preparation



Test samples shipment

CY 2007: test samples shipped during 1Q and 2Q

- LANL
- ORNL
- SRS
- Y-12
- ABACC
- IAEA
- Tokai
- URENCO

CY 2008 test samples

- Ship during the last quarter of CY 2007



Reports

Statistical evaluation reports

- Performance metrics: Evaluation report to be sent within three weeks of receiving test sample results
- Performance metrics missed in several instances in CY 2006 and CY 2007 1Q
- CY 2007 2Q performance metrics improved; able to send reports on time

Annual reports

- Performance metrics: Annual reports to be distributed at this meeting
- Reports will be distributed by middle of August; one month delay

Minutes of the meeting

- Minutes will be distributed by middle of August; on time



Safeguards Assistance

- Consolidation of Measurement Evaluation and Safeguards Assistance programs
- Assistance provided by NBL in facility audits
 - Paducah gaseous diffusion plant
 - Portsmouth gaseous diffusion plant
 - ORNL
 - SRS
 - ANL



NBL Participation in other Programs

- ABACC: UF_6 analysis (to be done in collaboration with USEC)
NBL/ABACC meeting August 2006
ABACC hands-on workshop (uranium assay and uncertainty evaluation) in August 2007
- LASAL: UO_2 pellet analyses (in progress)
- AWE: Uranium exchange (in planning stage)
- IAEA: Qualification of Robotics system



New Database Development

- A new database system replacing the FoxPro
 - More reliable
 - Developed by CNI (DOE-CH contractor)
 - Quality assurance
- Phase I work completed in April 2007
- Phase II: In progress



Morning Session

SME	9:00 AM	Opening remarks SME Program New database development	Jon Neuhoff, NBL B. Srinivasan, NBL B. Srinivasan, Michael Soriano, Joe Waggoner* and Ron Kuziel, NBL
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	11:50 AM	Including Particles as a form of standard materials in the NUSIMEP campaigns: Some reflections	R. Wellum* and R.Kips, EC-JRC-IRMM
	12:15 PM	SRS automated QC charts for accountability methods	Clint Gregory* and Hasan Syed, SRS



Acknowledgements

- Test materials characterization: Richard Essex, Nancy Hui, Maria Morales, Michael Soriano, Alma Stiffin
- Shipping: Maria Morales, Frank Orlowicz, Glenda Orlowicz, Alma Stiffin, Al Thomas and Heidi Williams
- Test samples results evaluation: Michael Soriano, Rebecca Thomas and Joseph Waggoner
- Annual reports: Kattathu Mathew, Michael Soriano, Joe Waggoner
- Annual meeting: Nancy Hui, Kattathu Mathew, Mary Pyrzynski, Alma Stiffin and Judy Zero
- New database: Kevin Atto, Joel Catausan, Miguelito Domingo, Mark Jilek, Ron Kuziel, Michael Soriano and Joe Waggoner
- Management: Jon Neuhoff, Steve Goldberg and Usha Narayanan

