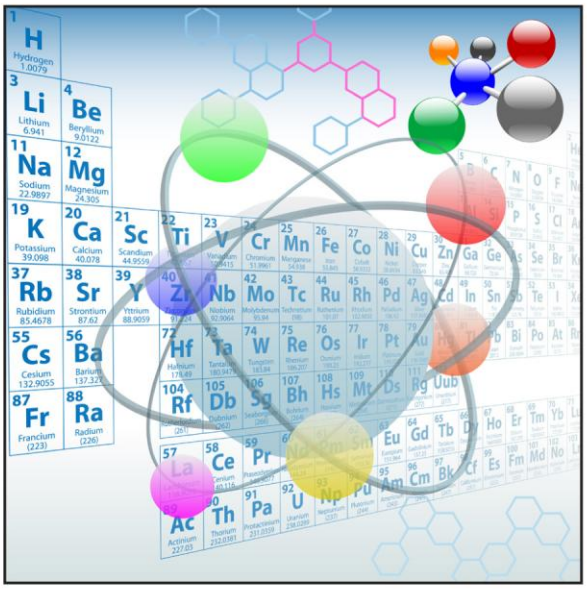

NBL–ME 2010 SMEP Annual Report

Safeguards Measurement Evaluation Program

CY 2009 Uranium Samples Exchange Annual Report

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PLEASE NOTE: Appendix A, SMES User Manual, has been removed from the public version of this report.

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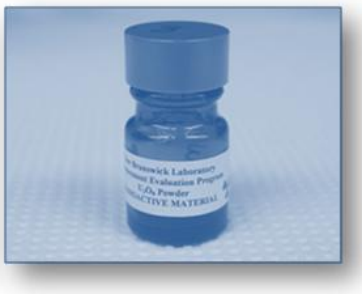
NBL: HISTORY AND MISSION

The New Brunswick Laboratory (NBL) is owned and operated by the United States Department of Energy Office of Science (SC). NBL was established in 1949 as an analytical chemistry laboratory to provide support to the United States Atomic Energy Commission. It was located in New Brunswick, New Jersey, at that time, and was staffed by scientists from the National Bureau of Standards who had contributed significantly to the Manhattan Project's nuclear material measurement programs. At NBL, they provided the technical expertise and skills to solve problems related to quantitative analyses of uranium-bearing materials. In 1977, the laboratory moved from New Jersey to its present location at Argonne National Laboratory site in Illinois.

Over the years, NBL scientists have expanded the capabilities of the laboratory to include chemical and mass spectrometric analyses of plutonium and other actinide elements, research and development activities in chemical analyses techniques, preparation of certified reference materials, and operation of the nuclear safeguards measurement evaluation program.

NBL's major mission is to provide technical assistance to the Department of Energy in the following areas: measurement evaluation program operation, certified (nuclear) reference materials preparation and measurement techniques development. 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.



ACKNOWLEDGEMENTS

The Safeguards Measurement Evaluation Program is administered by the United States Department of Energy, Office of Science (SC). The authors of this annual report thank the following ANL personnel for help with copy-editing and printing of the report: Linda Graf, Michelle Nelson, Kerri Schroeder, and Gary Weidner. Thanks are due to Mika Sumi of JAEA for requesting re-evaluation of U_3O_8 sample results; had it not been for this enquiry the error made in earlier U_3O_8 results evaluation would have gone unnoticed.

ABSTRACT

New Brunswick Laboratory (NBL) has been tasked by the United States Department of Energy, Office of Science (SC) with evaluating the quality of measurement results in nuclear materials accounting at the Department of Energy laboratories. The destructive analyses results are evaluated in the Safeguards Measurement Evaluation (SME) Program, and the non-destructive analyses results in the Calorimetric Exchange (CALEX) Program. This report describes the CY 2009 activities in the SME program.

The introductory sections A to C of this report state the objectives and goals of this program and contain an executive summary. The middle sections D to I constitute the main body of the report providing an account of the performance evaluation of measurement results of uranium test samples analyzed in safeguard laboratories in the DOE complex and elsewhere in laboratories outside the complex. The statistical methods used in the evaluation are also described in the middle section; tables and graphs display the accuracy and precision achieved in analyses. The concluding section J of the report contains a list of programmatic activities that are to be performed in 2011 and beyond.

ABBREVIATIONS AND ACRONYMNS

ABACC	Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials
ANL	Argonne National Laboratory
ARN	Autoridad Regulatoria Nuclear, Argentina
CALEX	Calorimetry Exchange Program
CNEA	Comisión Nacional De Energía Atómica
CONUAR	Combustibles Nucleares Argentinos
CY	Calendar year, January to December
DA	Destructive Analysis
D&G	Davies and Gray Titration
DOE	(United States) Department of Energy
DU	Depleted Uranium ($^{235}U < 0.3$ wt %)
GUM	Guide to the Expression of Uncertainty in Measurement
GSMS	Gas Source Mass Spectrometry
HEU	High-enriched uranium ($^{235}U \geq 20$ wt %)
IAEA	International Atomic Energy Agency
ICPMS	Inductively Coupled Plasma Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
INMM	Institute of Nuclear Materials Management
ISO	International Organization for Standardization
ITV	International Target Value
LEU	Low-enriched uranium (1wt % < ^{235}U < 20 wt %)
ME	Measurement Evaluation
NBL	New Brunswick Laboratory
NMCC	Nuclear Material Control Center
Q	Quarter (e.g., 1Q is first quarter of the year)
RD	Relative deviation (expressed in percent); also written as % RD
SC	Office of Science
SD	Standard deviation (expressed in percent)
SME	Safeguards Measurement Evaluation
SMES	Safeguards Measurement Evaluation System
TIMS	Thermal Ionization Mass Spectrometry
U	Uranium (0.3 wt % < ^{235}U < 1 wt %)
UF_6	Uranium hexafluoride
UNH	Uranyl nitrate hydrate (solution)
UO_2	Uranium dioxide

UO_3	Uranium trioxide
U_3O_8	Uranium (mixed) oxide
u(r)	Random component of uncertainty (indicative of precision) of ITV
u(s)	Systematic component of uncertainty (indicative of bias) of ITV
XRF	X-Ray Fluorescence

A. INTRODUCTION

The New Brunswick Laboratory (NBL) is a nuclear material measurement laboratory of the U.S. Department of Energy in the Office of Science. One of NBL's mission is to conduct the measurement evaluation program - a program designed to provide independent verification of internal analytical quality control in nuclear material accountability measurements done at DOE facilities. The program consists of two parts: the Safeguards Measurement Evaluation (SME) program for verification of measurement results from destructive methods of analyses of uranium and plutonium bearing materials (e.g., titration, mass spectrometry), and the Calorimetry Exchange (CALEX) program for verification of measurement results from non-destructive methods analyses of plutonium (e.g., calorimetry and gamma ray spectrometry). This annual report describes the CY 2009 SME program activities; a separate report has been prepared and issued for the CALEX program.

B. SAFEGUARDS MEASUREMENT EVALUATION PROGRAM

Good material control and accountability practices are essential elements of a successful nuclear safeguards program. The accountability measurements must yield quantitative information, within acceptable limits of accuracy and precision, on elemental as well as isotopic abundances so that nuclear material loss either by theft or by diversion or by other means can be detected. The SME program is designed to monitor performance in the measurements and ensure sufficient accuracy and precision are obtained in the analyses. These objectives are realized indirectly through a sample exchange program. Test samples of uranium and plutonium bearing materials, sent by NBL, are analyzed in safeguards laboratories by procedures routinely used in material accountability measurements. The results are evaluated by NBL for accuracy, precision, day-to-day variation, and compliance to method/material specific International Target Values (ITVs); a performance evaluation report is sent for every set of material/method specific results with recommendations (if any) for improving measurement quality.

C. CY 2009 SME PROGRAM: EXECUTIVE SUMMARY

The SME program activities in CY 2009 and extension of those activities into the following year are presented in this executive summary. An important reason for presenting this summary over this extended period is to provide an up-to-date account of the programmatic activities to the new Measurement Evaluation Program Coordinator who will take charge in January 2011.

Participants: DOE contractor laboratories, a NRC laboratory, and several international laboratories participated in the CY 2009-2010 SME program. Nine new participants were added during this period: the Institute for Trans-uranium Elements (ITU) in Germany, Paul Scherrer Institute in Switzerland, Preston National Nuclear Laboratory at the Springfield Site in the United Kingdom, South African Nuclear Energy Corporation (NECSA), and five Safeguards Measurement Laboratories in Japan. Safeguards measurement laboratories in Canada, China, Kazakhstan, Russia, and South Korea are expected to join the program in CY 2011.

Test samples: Uranium test samples (UNH solutions, UO_2 pellets, UO_3 powder, U_3O_8 powder and UF_6) only were used in the CY 2009-2010 program.

Test samples shipment: Test samples for analyses in CY 2009 were shipped during CY 2008-4Q and CY 2009-1Q. Similarly, samples for analyses in CY 2010 were shipped during CY 2009-4Q and CY 2010-1Q. The CY 2011 samples will be sent out in the first quarter of that year.

Analyses methods: SME program participants used a variety of analytical techniques to analyze the test samples. Elemental uranium measurements were made by Davies and Gray titration (D&G), Isotope Dilution Mass Spectrometry (IDMS), X-Ray Fluorescence (XRF), and Gravimetry methods; uranium isotopic compositions were determined by Thermal Ionization Mass Spectrometry (TIMS), Inductively Coupled Plasma Mass Spectrometry (ICPMS), and Gas Source Mass Spectrometry (GSMS).

Material/method specific measurement evaluations and reports: NBL evaluated the test samples measurement results for accuracy, precision, day-to-day variation, and compliance to ITVs. Participants received feedback for each set of material/ method specific results submitted by them.

Safeguards Measurement Evaluation System (SMES2): SMES2 is a “home-grown” web-based database and data analysis system for evaluating the measurement results. Data entry into SMES2 was done by SME program staff. Starting in CY 2011, DOE laboratory personnel will be

provided access to the system for entering their own results and retrieve performance evaluation reports. Training will be given by NBL staff. A "user-friendly" instruction manual was prepared to serve as an aid to this training and for ready reference; see Appendix A for a copy of the manual. Chickasaw National Industry – a computer service contractor – is assisting NBL in providing access to external users with full cognizance to cyber security measures. Laboratories outside the DOE complex will be given access later.

Annual report: The CY 2008 annual report of the SME program was prepared and distributed in CY 2009. The report is also available at http://www.nbl.doe.gov/.

Annual meetings: The CY 2009 Measurement Evaluation Program Annual Meeting was held in Tucson, Arizona on July 11, 2009. Forty two technical/scientific personnel attended the meeting and 17 papers were presented. The minutes of the meeting is available at http://www.nbl.doe.gov/.

The CY 2010 Measurement Evaluation Program Annual Meeting was held in Baltimore, Maryland on July 10, 2010. Thirty six technical/scientific personnel attended the meeting and 19 papers were presented. The minutes of the meeting is available at http://www.nbl.doe.gov/.

New test samples: Six new sets of test samples were made for distribution in CY 2011. Experimental characterization of these samples is expected to be done in CY 2011-1Q prior to shipment. The new test samples are:

a) Three sets of UNH solutions for elemental uranium measurements. Each set contains 100 or more ampoules, and each ampoule contains about 250 mg of uranium in 20 ml volume. A part of the preparative work related to flame sealing the samples was done at the Chemical Sciences and Engineering Division of the Argonne National Laboratory (ANL).

b) Three sets of uranium isotopic test samples. Each set contains 100 vials, and each vial contains about 5 mg of low enriched uranium (LEU); ^{235}U abundances are 1.5 or 3.0 or 4.9 wt %. The 1.5 and 4.9 wt % samples are dried residues of UNH solutions; the 3.0 wt % sample is U_3O_8 powder.

D&G titration training: Norman Johns (SRS), and Ralph Ilgner and Joe Giaquinto (both of ORNL) received D&G training at NBL in two separate sessions. Glennda Orlowicz was the trainer. The main objective of the training sessions is to improve accuracy and precision in elemental uranium measurements.

High precision titration training: NBL is providing help to LASAL (Laboratorio de Salvaguardas in Rio de Janeiro) to set up a Measurement Evaluation program for Brazilian laboratories. In that program, UO_2 pellets manufactured in Brazil will be used as test samples. NBL and LASAL tested a batch of pellets and found it to be unsuitable due to pellet-to-pellet variation (in elemental uranium content). LASAL has made another batch of pellets recently and expects that it will be compositionally uniform. It will characterize the new material using the NBL method of high precision titration and requested NBL to train LASAL chemists on this method of analysis. The high precision method is ideally suited to define the elemental uranium content with high accuracy and precision, and also to test for material homogeneity. Responding to LASAL's request, Anna Voeks of NBL offered a training session at LASAL for the benefit of scientists/technicians in that laboratory and a visiting scientist from a CNEA laboratory in Argentina.

ABACC/NBL collaboration: B. Srinivasan of NBL and William Guthrie of NIST visited a number of ABACC network laboratories in Buenos Aires, São Paulo, and Rio de Janeiro to (a) discuss network laboratories performance in uranium measurements, (b) identify training needs, (c) test samples and reference materials needs, (d) invite fuel fabrication facilities (CONUAR in Argentina and Resende Nuclear Fuel Factory in Brazil) to participate in the NBL-SME program, and (e) offer workshop/ training in uncertainty estimations in safeguards measurement results.

The U.S. team visited 11 laboratories and reviewed their performance in analyzing uranium samples. A majority of the network laboratories are able to obtain good quality results in elemental and isotopic analyses in conformity to the International Target Values. Several laboratories made requests for new working reference materials for impurity measurements and test samples to evaluate performance in these measurements.

The two fuel fabrication facilities, CONUAR in Argentina and Resende in Brazil, have decided to participate in the SME program starting in CY 2011.

The uncertainty workshop/training sessions were offered at central locations in the three cities visited. About 70 technical personnel attended the sessions. The trainees learned to use a new software tool developed by NIST - based on R-statistics application with Excel interface – for uncertainty estimations according to GUM (Guide to the Expression of Uncertainty in Measurement). All examples and exercises in the training sessions dealt with estimating uncertainties in elemental uranium and fissile isotope (^{235}U) measurements. The software is available free of cost to the users.

ABACC-HQ officials attached high importance to the following two items of collaborative work with NBL: (a) training in Thermal Ionization Mass Spectrometry, and (b) qualifying the "Cristallini" method of UF_6 sampling. The mass spectrometer training will be offered at NBL in CY 2011. Regarding the

Cristallini method of UF_6 sampling, NBL will assist ABACC in drawing up a sampling and analysis plan and in its execution. The results of analyses will be evaluated by NBL to demonstrate that the Cristallini samples indeed provide "true" and representative results for the (isotopic) composition.

Cristallini method: Osvaldo Cristallini, a CNEA scientist, now working as an official in the ABACC organization, has developed an elegant method to sample UF_6 for isotopic analysis. The method is based on absorption of UF_6 on alumina substrate and subsequent release of the hydrolyzed product (UO_2F_2) for analysis by TIMS and/or ICPMS methods. There are several advantages to the Cristallini method relative to sampling UF_6 in P-10 tubes; it is inexpensive, smaller quantity of material is sampled (about 0.2 g vs. 10 g in P-10), and ease of shipping (as solid vs. gas in P-10). The Cristallini method was tested in ABACC network laboratories with satisfactory results. The next step is to qualify the method as outlined in the paragraph above.

ARN/DOE collaboration: NBL personnel attended two meetings of the ARN/DOE Permanent Coordinating Group (PCG), one in July 2009 and the other in August 2010 to discuss progress made in DOE/ARN collaboration programs and to define new areas of work. Jon Neuhoff and B. Srinivasan attended the July 2009 meeting in Washington DC. At that meeting, NBL offered assistance to DOE to ship the Certified Reference Material 969 - a set of ^{235}U enrichment standards for gamma ray measurement – to ARN laboratories. The material was shipped to Argentina by the end of that year. In the second meeting held in Buenos Aires in August 2010, Srinivasan made a presentation identifying the following areas for new collaborative work with ARN/CNEA laboratories; development/training in destructive analysis (DA) and non-destructive analysis (NDA) methods, uncertainty estimation in NDA measurements, preparation of certified reference materials/working reference materials for DA and NDA programs, quality assurance/quality control in measurements, and establishing a measurement evaluation program in Argentina similar to the NBL program.

Measurement Evaluation program for Russian federation: NBL, in collaboration with ANL, hosted a two day meeting in July 2010 to present the highlights of the NBL-SME Program to a visiting delegation of scientists from Russia. Jon Neuhoff, Usha Narayanan, Colleen Gradle, B. Srinivasan, and Anna Voeks (all of NBL) made oral presentations at this meeting. The Russian group expressed their interest to collaborate with NBL in establishing a similar program for the Russian safeguards laboratories. Furthermore, they would encourage the Russian laboratories to take an active part in the NBL-SME program. Follow up meetings and tele-conferences were held with ANL and DOE-HQ personnel to give shape to this new collaboration effort.

NMCC round robin: NBL provided help to NMCC to conduct a Safeguards Measurement Evaluation program for laboratories in Japan. Four safeguards laboratories participated in a round-robin exercise and analyzed LEU-UO_2 test samples sent by NBL for elemental content and isotopic

composition. NBL evaluated the results and sent performance evaluation reports to the participants (through NMCC). NMCC is interested in continuing this collaboration effort; a new set of test materials will be provided by NBL for the next exercise.

Revision of International Target Values: The IAEA document on "International Target Values 2000 for Measurement Uncertainties in Safeguarding Nuclear Materials" is to be revised in 2010. NBL provided input to this revision by sharing the SME program measurement results in its database with IAEA; these records are likely to help in assessing the accuracy and precision routinely achieved in uranium and plutonium analyses. In addition, NBL submitted a paper in which the ITV 2000 and GUM methods for expressing measurement uncertainties were compared; the paper was prepared by B. Srinivasan (NBL), William Guthrie (NIST) and Charles Pietri (NBL consultant). It is certain that the target values to be specified in the ITV 2010 document will be along the same lines as in the ITV 2000 document, i.e., separately specifying the systematic and random components of material/method specific measurement uncertainties. In contrast, the GUM method will report a single value for the uncertainty by taking into account all known sources that contribute to the total. It is the preferred method to express measurement uncertainties in reference materials and calibration standards. Increasingly, nuclear measurement service laboratories, especially those with ISO accreditation, are using the GUM method. NBL recommends that future revision of ITVs must accommodate the GUM method. In order to provide additional information to the next revision, NBL plans to extend the comparative study given in the paper by evaluating a larger number of SME program measurement results using both methods for uncertainty estimation. The results of this study will be shared with IAEA.

Review of ISO standards on uranium/plutonium separation procedures: Usha Narayanan and B. Srinivasan (both of NBL), and Michael Holland (of SRS) reviewed two parts of an ISO standard (ISO/TC 85 Document N 1093) procedure on the separation of uranium and plutonium for isotopic measurements and provided critical comments for their revision.

INMM meeting: Fabio Dias of LASAL presented a talk in the 51st INMM Annual Meeting in Baltimore on the "The Role of Uncertainty Estimation on the Improvement of Measurement Processes and Comparison between Results". The paper was published in the proceedings of the meeting; B. Srinivasan of NBL is a co-author of this paper.

D. CY 2009-2010 SME PROGRAM PARTICIPANTS

DOE laboratories participated in the CY 2009-2010 Measurement Evaluation program in accordance to the mandate in Chapter II.4.e. (7) of DOE Manual 474.1-1 of November 2000: *"Each facility's measurement control program must include participation in appropriate inter-laboratory control programs to provide independent verification of internal analytical quality control."* In addition, a NRC laboratory and several international facilities participated. See Table D.1 for a list of the participants. The program is experiencing steady growth; nine new participants joined the program in CY 2009-2010.

Table D.1. CY 2009-2010 SME Program Participants

ABACC Laboratories
- CAE/CNEA, ARGENTINA
- COMPLEJO FABRIL CORDOBAL (CNEA) ARGENTINA
- LABORATORIO ICP-MS, DDCyE - DIOXITEK, S.A. ARGENTINA
- PFPU/CNEA, ARGENTINA
- UACN/CNEA, ARGENTINA
- CONUAR, ARGENTINA
- CDTN/CNEN, BRAZIL
- CTMSP II, SAO PAULO, BRAZIL
- IEN/CNEN- RJ, BRAZIL
- IPEN/CNEN, SAO PAULO, BRAZIL
- LASAL/CNEN, RJ, BRAZIL
- RESENDE FUEL FABRICATION PLANT, BRAZIL

IDAHO NATIONAL LABORATORY, USA
INSTITUTE of TRANSURANIUM ELEMENTS, GERMANY
INTERNATIONAL ATOMIC ENERGY AGENCY – SAL, AUSTRIA
JAPAN ATOMIC ENERGY AUTHORITY, JAPAN
LOS ALAMOS NATIONAL LABORATORY, USA
NEW BRUNSWICK LABORATORY, USA
NRC: NUCLEAR FUEL SERVICES, USA
NUCLEAR MATERIALS CONTROL CENTER, JAPAN
- GLOBAL NUCLEAR FUEL JAPAN, YOKOSUKA
- MITSUBISHI NUCLEAR FUEL, TOKAI
- NUCLEAR FUEL INDUSTRIES, KUMATORI
- NUCLEAR FUEL INDUSTRIES, TOKAI

OAK RIDGE NATIONAL LABORATORY, USA
PAUL SCHERRER INSTITUTE, SWITZERLAND
PRESTON NATIONAL NUCLEAR LABOARTORY, SPRINGFIELD SITE, UK
SAVANNAH RIVER NATIONAL LABORATORY, USA
SAVANNAH RIVER SITE, USA
SELLAFIELDS LTD., UK
SOUTH AFRICAN NUCLEAR ENERGY CORPORATION, SOUTH AFRICA
URANIUM ENRICHMENT COMPANY LTD. (URENCO) Laboratories
- URENCO (GERMANY)
- URENCO (NETHERLANDS)
- URENCO (UK)

Y-12 Plant - BWXT LLC, USA

E. TEST SAMPLES AND MEASUREMENT METHODS

E.1. Test samples

Uranium test samples were made from Certified Reference Materials (CRMs), Working Reference Materials (WRMs) or custom-made. The elemental content and/or isotopic abundances of the test samples were characterized at NBL. Test samples measurement results from participants were evaluated with respect to the characterized (reference) values. Uncertainties in characterized values were not propagated in these evaluations.

Uranyl nitrate solution (UNH): The uranyl nitrate solutions were made by dissolving uranium metal/uranium compounds in nitric acid. The solutions for elemental uranium analyses contained about 200 mg uranium in 20 ml volume. The solutions for isotopic analyses contained about 5 mg uranium in 5 ml volume. The ^{235}U enrichment of the isotopic samples covered a wide range, from less than 1 wt % to about 90 wt %.

UO_2 pellets: The UO_2 pellets were made in a single batch at the Westinghouse Commercial Nuclear Fuel Division (a NRC licensee) using a high temperature sintering process; ^{235}U enrichment is about 4 wt %. The pellets are known to be (compositionally) homogeneous, stable, suffer no change on exposure to air, and are resistant to moisture uptake. The pellets were used as test samples in elemental and isotopic measurements.

UO_3 powder: NBL packed the UO_3 test samples in crimp sealed glass vials under dry nitrogen atmosphere; ^{235}U enrichment of this material is about 0.9 wt %. One of the participants suspected that sample integrity might have been compromised due to moisture uptake in long-term storage. To clear the suspicion, NBL analyzed a number of samples with and without drying at 110°C; the results were inconclusive due to ill-defined experimental controls. NBL will analyze the material again in CY 2011, this time in accordance to a well designed sampling and analysis plan. The results of this new study will be taken together with other results in the SME program database to decide upon retaining the material for future use or discarding it.

U_3O_8 powder: U_3O_8 powder is a suitable test material for both elemental and isotopic measurements; the ^{235}U abundance is about 0.7 wt %. The source material was prepared in 1984 by NLO Inc. starting from highly pure UO_2 pellets; the pellets were crushed, dissolved in nitric acid, and the uranium was precipitated with hydrogen peroxide, filtered, dried, calcined at 900 °C, milled, and screened. The elemental uranium content was characterized at NBL; samples were ignited at 800°C to drive away moisture and volatiles prior to characterization.

Several laboratories analyzed the samples as received, i.e., without ignition. These results were evaluated with reference to characterized value of the ignited samples causing an error of about 0.04 %. The error is now corrected in the annual evaluation of results given in Sections H.9 and H.10.

UF_6: The UF_6 test samples were made at the Portsmouth Gaseous Diffusion Plant for both elemental and isotopic measurements; ^{235}U abundances are in the range of 0.5 wt % to 4.8 wt %. Isotopic characterizations of the samples were done at NBL and also at the plant. The elemental uranium contents were not experimentally characterized, but calculated by assuming 100 % purity for the samples; uranium atomic weights needed for these calculations were obtained from isotopic abundance determinations.

E.2. Measurement methods

The SME program participants analyzed the test samples using a variety of methods. Elemental uranium contents were determined by D&G titration, IDMS, XRF, and Gravimetry methods. The isotopic analyses methods were TIMS, ICPMS, and GSMS. Note that UF_6 samples were analyzed either directly using GSMS or after hydrolysis using TIMS and ICPMS methods.

F. STATISTICAL METHODS IN PERFORMANCE EVALUATION

The material/method specific measurement results were evaluated using the built-in statistical analysis software in SMES2. Statistical reports generated by SMES2 were appended to performance evaluation letters. The letters were mailed to participants usually within 3 to 4 weeks after receiving the results.

Statistical evaluation: The percent relative difference (% RD) of each result in a set is calculated with respect to the characterized reference value according to the following equation:

% RD = 100 X {(Measurement result - reference value)/reference value}.

The results are examined for statistical outliers using a number of tests. If two of the tests show a particular result to be an outlier at ≥ 99% significance, then it may be removed following review either by the program coordinator or a statistician. With outliers removed, the mean % RD and the standard deviation (σ) are computed. Day-to-day variation is evaluated using standard one-factor analysis of variance (ANOVA). If the ANOVA results indicate no significant variation (i.e., < 95% significance), then the standard uncertainty is reported as the product of a "coverage factor" and the simple standard deviation (σ) of the results divided by the square root of n where n is the number of measurements; the coverage factor is the Student's 95% "t" factor with n-1 degrees of freedom (df). For example, the coverage factor is 2.36 for a set of 8 results showing no significant day-to-day variation (df = 7). On the other hand, if the ANOVA results indicate significant day-to-day variation (≥ 95% significance), then the standard uncertainty is estimated from a combination of the mean square for the "error" and the mean square for the "model" quantities from ANOVA, with degrees of freedom determined from Satterthwaite's approximation. For measurements done on two different days and exhibiting significant day-to-day variation, the formula for estimating the standard uncertainty in the mean % RD is reduced to the square root of the mean square for the "model" quantity obtained from ANOVA results; the Student's 95% "t" factor is 12.7 (df = 1) for this case.

The mean % RD is a measure of accuracy; it is compared against u(s), the systematic uncertainty component of the ITV. The standard deviation is a measure of precision; it is compared against u(r), the random uncertainty component of the ITV. Measurement bias is determined from the confidence interval (C.I.) of the mean constructed from the 95 % C.L. The C.I. represents the interval containing all values between the mean % RD minus the C.L. and the mean % RD plus the C.L. Thus, the 95 % C.L. of the mean are just the two end points of the C.I. A measurement is considered to be bias-free if the C.I. includes zero in the interval. Otherwise, (positive or negative) bias in measurements is indicated.

An example of the statistical report is shown in section F.1.

F.1. Example of material/method specific statistical evaluation report

Table F.1 shows eight measurement results for elemental uranium content in two UO_2 pellets. Each pellet was analyzed in duplicate on two different days by D&G titration. There are no statistical outliers in this set of results. The mean % RD is calculated to be -0.154%; it is outside the limit for u(s), the systematic uncertainty component of the ITV for D&G titration. The standard deviation is 0.083%; it is in compliance to u(r), the random uncertainty component of the ITV for D&G titration. Day-to-day variation is not significant. The 95 % C.L. of the mean is 0.070% (df = 7). The mean % RD extended by the 95% confidence limit (-0.154 $\pm$ 0.070) does not overlap with zero thereby indicating negative bias in the measurements.

Figure F.1 displays the eight % RD results from two days of measurements. All results plot below zero % RD and seven of the eight values are more negative than the lower bound of u(s).

Table F.1. Measurement Results Evaluation Report
U.S. Department of Energy
New Brunswick Laboratory
Safeguards Measurement Evaluation Program

Day to Day ANOVA analysis

Report for Laboratory: XX

$U0_2$ Pellet – U Concentration

Davies-Gray Titration

Date of Report: November 30, 2003

Sample Number	Aliquant Number	Analysis Date	Reported %U	% Relative Difference	Analyst Code
95EU0079-1	1	11/03/03	88.126	-0.0034	XXX
95EU0079-1	2	11/03/03	87.990	-0.1577	XXX
95EU0079-2	1	11/03/03	88.031	-0.1112	XXX
95EU0079-2	2	11/03/03	87.892	-0.2689	XXX
95EU0079-1	3	11/04/03	88.030	-0.1123	XXX
95EU0079-1	4	11/04/03	87.950	-0.2031	XXX
95EU0079-2	3	11/04/03	87.922	-0.2349	XXX
95EU0079-2	4	11/04/03	88.002	-0.1441	XXX

Number of Results Analyzed	8
Mean % Difference	-0.154
Mean Absolute % Difference	0.154
95% C.L. of Mean (df = 7)	0.070
Standard Deviation	0.083
Between-Day Standard Deviation (df = 1)	0.054
Within-Day Standard Deviation (df = 6)	0.087
Statistical Significance of Between-Day Standard Deviation	44.3 %

International target value for u(s) in Davies-Gray Titration is 0.1 %.

International target value for u(r) in Davies-Gray Titration is 0.1 %.

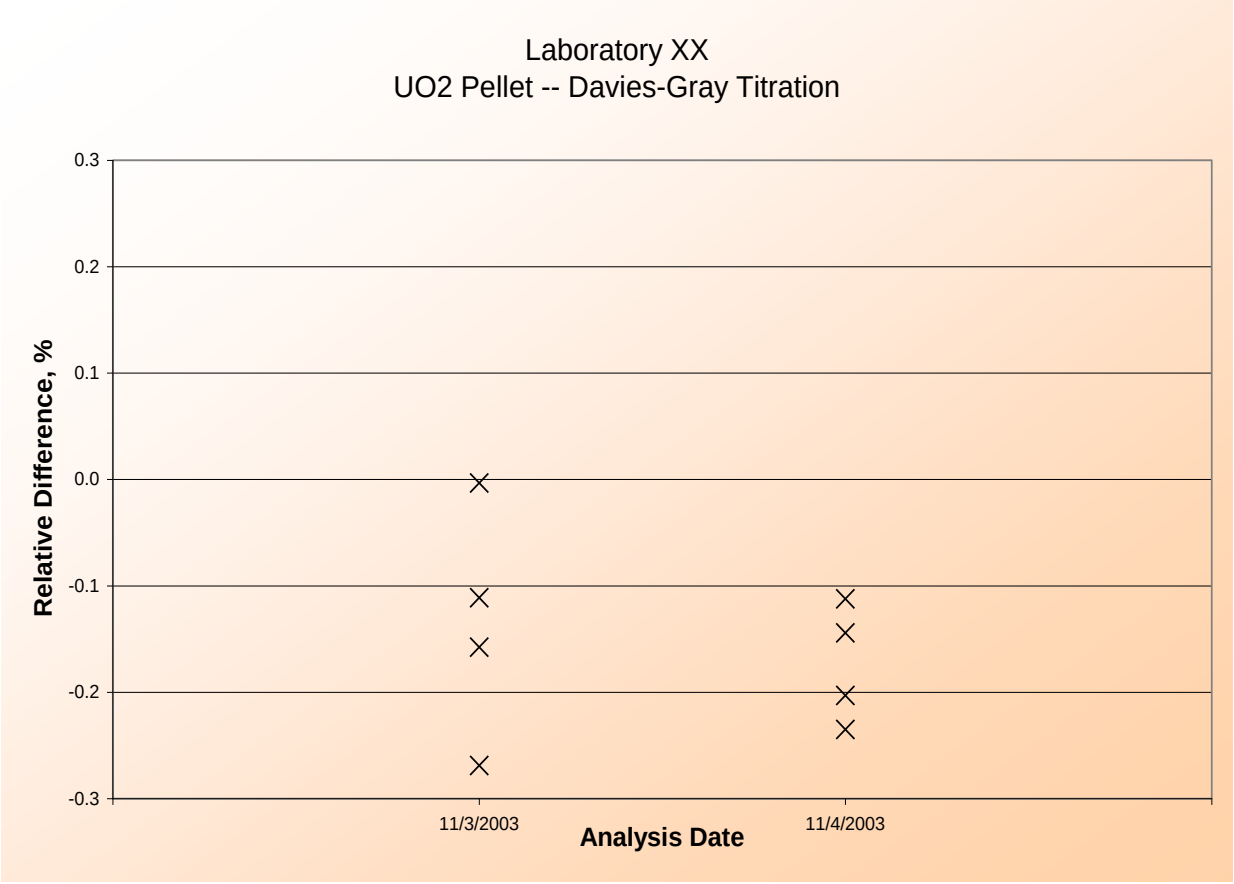


Figure F.1. Graphic representation of % RD of results from analyses of two UO_2 pellet samples. Each sample was analyzed in duplicate on two different days. All eight results for % RD are negative and seven of the eight results are below the lower bound for u(s).

G. ANNUAL EVALUATION OF CY 2009-2010 MEASUREMENT RESULTS

The annual evaluation of material/method/laboratory specific measurement results is an abbreviated version of the statistical evaluation shown in Section F. All (material/method specific) results obtained during the year are pooled together and the mean % RD and standard deviation are calculated for the entire set. The mean % RD is compared against u(s), the ITV for the systematic uncertainty component; the standard deviation is compared against u(r), the ITV for the random uncertainty component. The 95% C.L. and period-to-period variation are not evaluated.

The annual evaluations for elemental uranium measurements are presented in Section H and for uranium isotopes measurements in Section I. The tables in the two sections list the analyses methods, laboratory identification codes, number of measurements, mean % RDs, standard deviations, and compliance to the ITVs with a "yes/no" notation; yes indicates compliance to the ITV and no indicates the result is outside the ITV. Two graphs accompany each tabular data, one for mean % RDs and the other for standard deviations. In the relative deviation graphs, the positive and negative limits of u(s) are shown by two horizontal lines, equally spaced above and below zero % RD. In the standard deviations graphs, a single horizontal line above zero % SD represents u(r). Results falling within the uncertainty bounds are said to be in compliance to the respective ITVs. Note that in the RD graphs, the compliance to the ITV is based on the placement of the mean % RD value only without regard to its extension by the standard deviation.

H. ANNUAL EVALUATION OF ELEMENTAL URANIUM MEASUREMENTS

The tables and graphs of material/method specific annual evaluations of elemental uranium measurement results are presented in Sections H.1 to H.11.

Table H. Annual evaluation of elemental uranium measurement results.

Material	Method			
	D&G Titration	**IDMS**	**XRF**	**Gravimetry**
UNH solution	Section H.1	Section H.2	Section H.3	
UO_2 pellet	Section H.4			Section H.5
UO_3 powder	Section H.6	Section H.7	Section H.8	
U_3O_8 powder	Section H.9	Section H.10		
UF_6	Section H.11			

H.1. Uranyl nitrate solutions (UNH) by D&G titration

The D&G results from analyses of UNH solutions are shown in Table H.1, and Figures H.1a and H.1b.

Laboratories BC and U: Need to improve both accuracy and precision

Laboratory BA: Need improvement in accuracy

Laboratory F: Need improvement in precision.

Table H.1. Elemental uranium in UNH test samples by D&G titration.

UNH Solution by D&G Titration					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
BA	-0.130	0.085	10	No	Yes
BC	-0.360	0.114	16	No	No
U	-0.117	0.158	8	No	No
BF	0.064	0.044	8	Yes	Yes
F	-0.075	0.159	16	Yes	No
B	-0.018	0.224	8	Yes	No

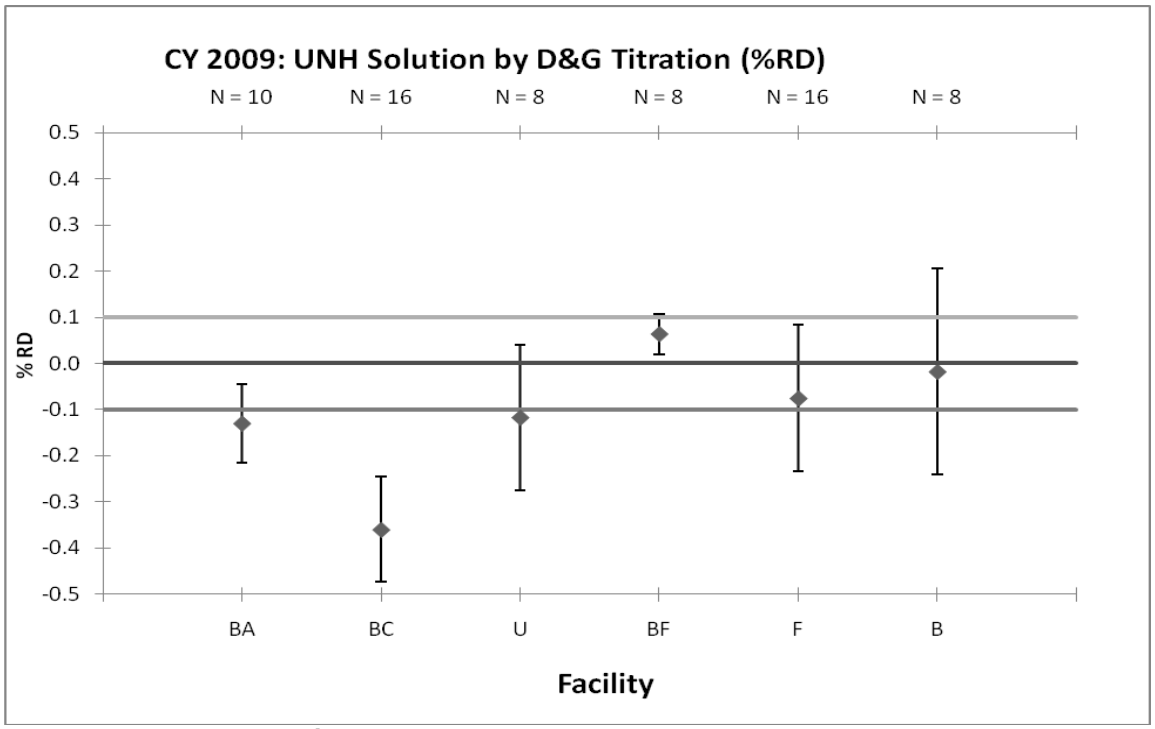


Figure H.1a. Mean % RD in elemental uranium determination in UNH test samples by D&G titration. Laboratories BF, F, and B are in compliance to u(s); laboratories BA, BC, and U are outside the ITV.

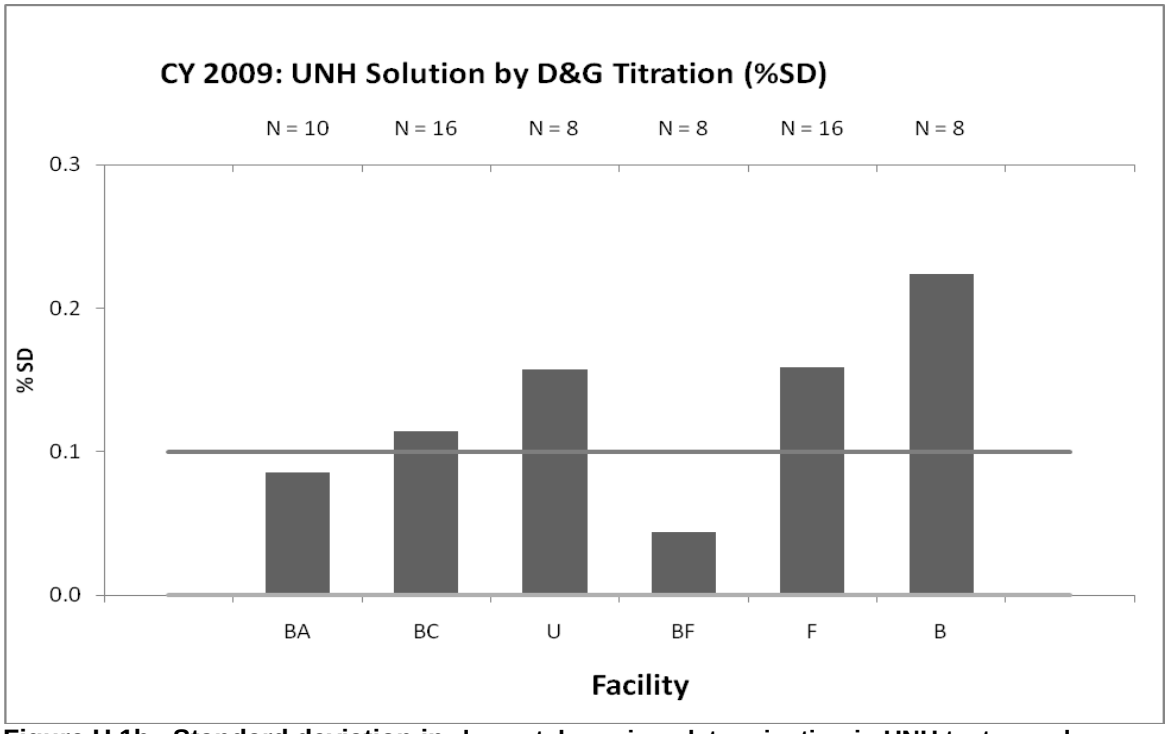


Figure H.1b. Standard deviation in elemental uranium determination in UNH test samples by D&G titration. Laboratories BA and BF are in compliance to u(r); laboratories BC, U, F, and B are outside the ITV.

H.2. Uranyl Nitrate Solutions (UNH) by IDMS

The IDMS results of analyses of UNH solutions are shown in Table H.2, and Figures H.2a and H.2b.

Laboratory A: Good accuracy, but poor precision

Laboratory B: Need improvement in both accuracy and precision.

Table H.2. Elemental uranium in UNH test samples by IDMS.

UNH Solution by IDMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.15
A	-0.051	0.308	24	Yes	No
B	-0.268	0.169	8	No	No

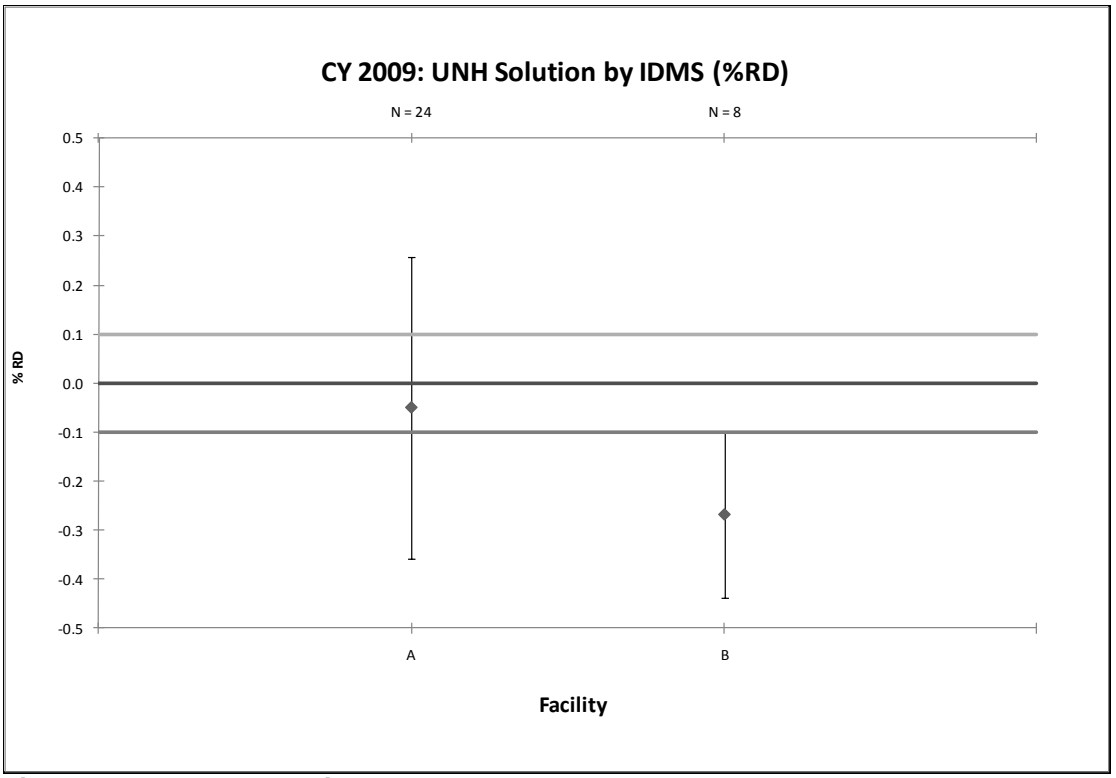


Figure H.2a. Mean % RD in elemental uranium determination in UNH test samples by IDMS. Laboratory A is in compliance to u(s); laboratory B is outside the ITV.

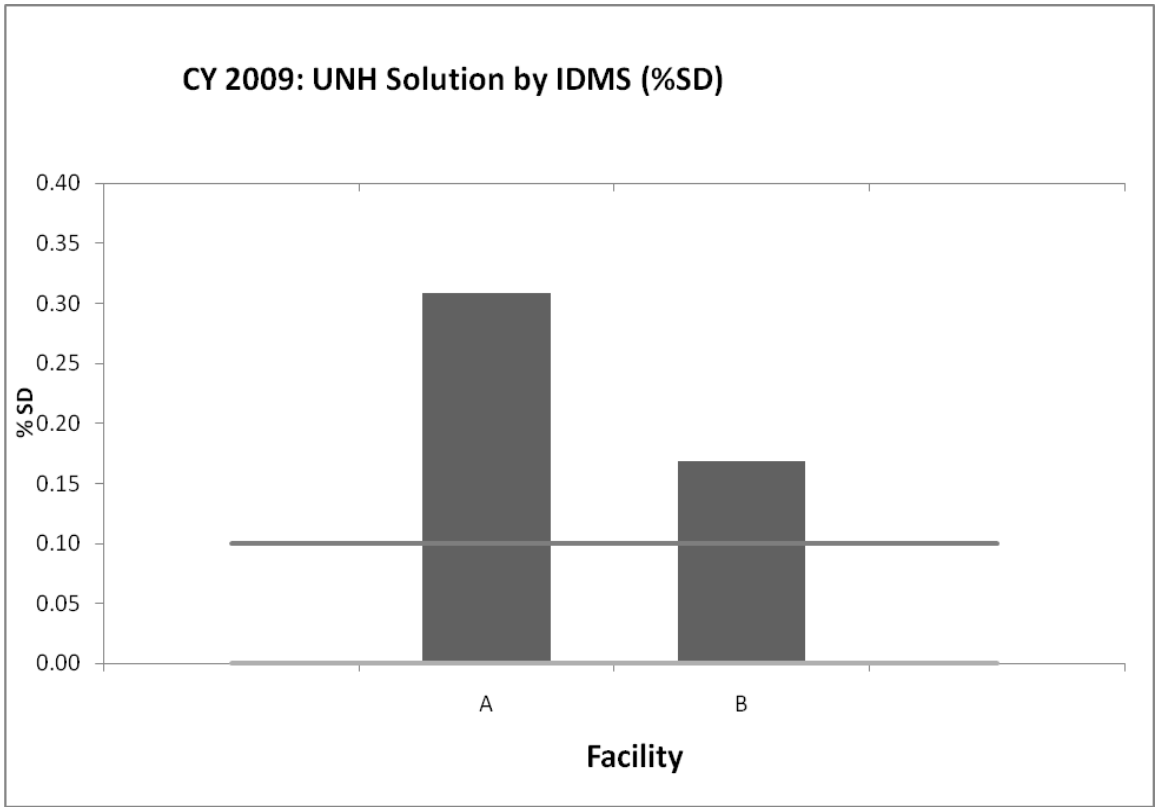


Figure H.2b. Standard deviation in elemental uranium determination in UNH test samples by IDMS. Both laboratories are outside u(r).

H.3. Uranyl Nitrate Solutions (UNH) by XRF

The XRF results of analyses of UNH solutions are shown in Table H.3, and Figures H.3a and H.3b.

Laboratory SA: Need improvement in both accuracy and precision.

Table H.3. Elemental uranium in UNH test samples by XRF.

CY 2009: UNH Solution by XRF					
Lab Code	Mean % RD	SD	N	Target Value Compliance*	
				u(s) = 0.5	u(r) =0.5
A	0.261	0.462	24	Yes	Yes
SA	1.976	1.019	12	No	No

*ITV is not specified for XRF; DOE target value is shown.

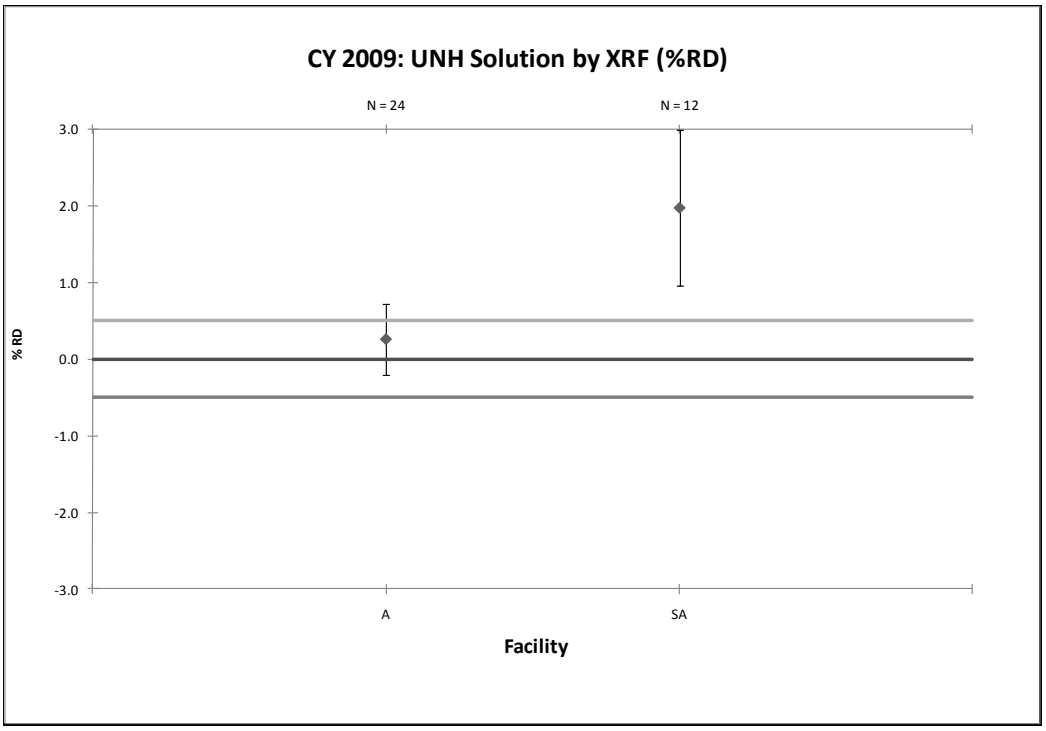


Figure H.3a. Mean % RD in elemental uranium determination in UNH test samples by XRF. Laboratory A is in compliance to u(s); laboratory SA is outside the target value.

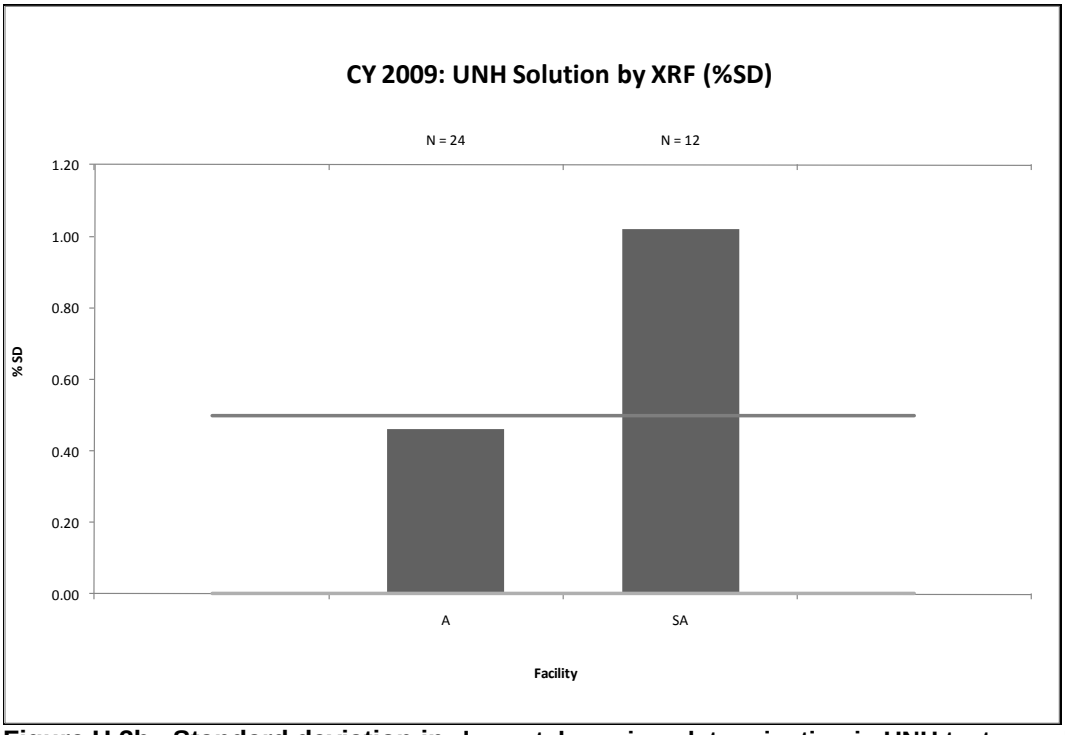


Figure H.3b. Standard deviation in elemental uranium determination in UNH test samples by XRF. Laboratory A is in compliance to u(r); laboratory SA is outside the target value.

H.4. LEU-UO_2 pellets by D&G titration

The D&G results from analyses of LEU-UO_2 pellets are shown in Table H.4, and Figures H.4a and H.4b.

Laboratory BC: Need improvement in both accuracy and precision.

Table H.4. Elemental uranium in LEU-UO_2 pellet test samples by D&G titration.

CY 2009: UO_2 Pellets by D&G Titration					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
BA	-0.101	0.068	21	Yes	Yes
BC	-0.125	0.134	36	No	No
ED	-0.013	0.067	16	Yes	Yes
T	0.009	0.070	28	Yes	Yes
V	0.019	0.016	8	Yes	Yes

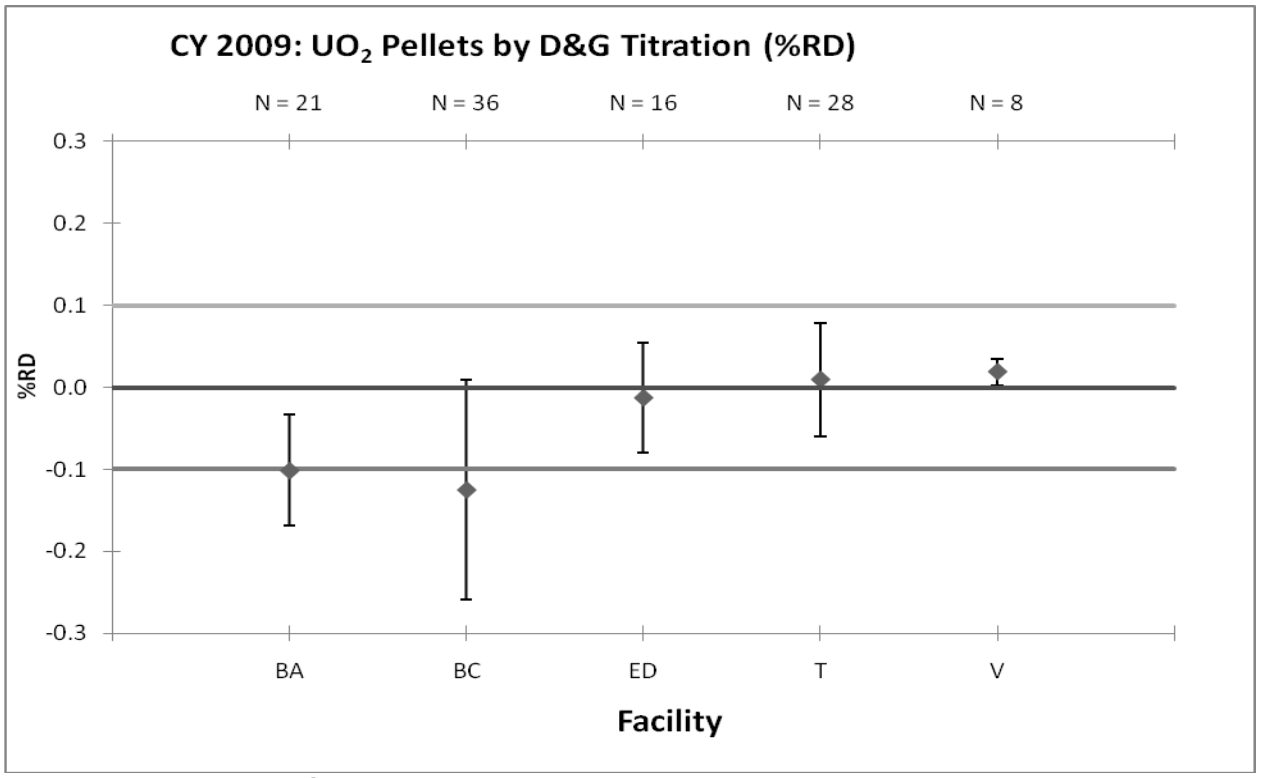


Figure H.4a. Mean % RD in elemental uranium determination in LEU-UO_2 test samples by D&G titration. Laboratories BA, ED, T and V are in compliance to u(s); laboratory BC is outside the ITV.

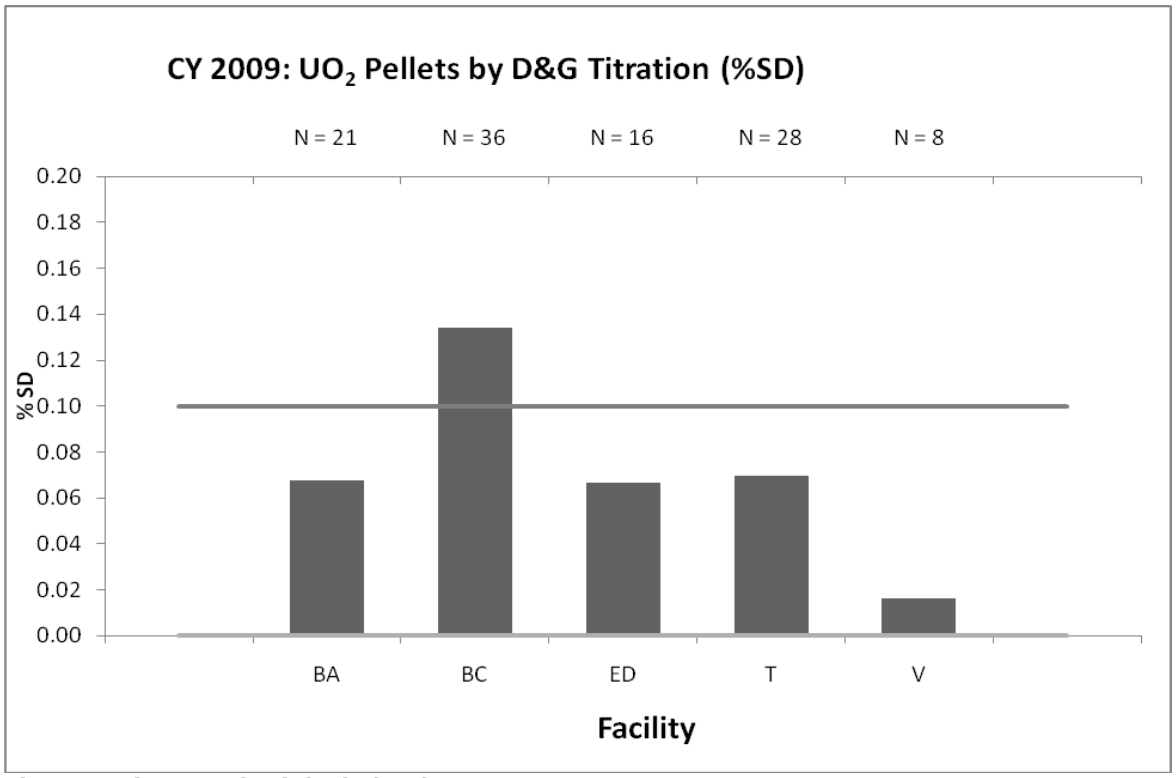


Figure H.4b. Standard deviation in elemental uranium determination in LEU-UO_2 test samples by D&G titration. Laboratories BA, ED, T, and V are in compliance to u(r); laboratory BC is outside the ITV.

H.5. LEU-UO_2 pellets by gravimetry

The gravimetric analyses results of LEU-UO_2 pellets are shown in Table H.5, and Figures H.5a and H.5b.

Table H.5. Elemental uranium in LEU-UO_2 pellet test samples by gravimetry.

CY 2009: UO_2 Pellets by Gravimetry					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.05	u(r) = 0.05
TO	0.0182	0.0083	16	Yes	Yes
TP	0.0153	0.0028	16	Yes	Yes
TH	0.0323	0.0066	16	Yes	Yes
TR	0.0189	0.0052	16	Yes	Yes

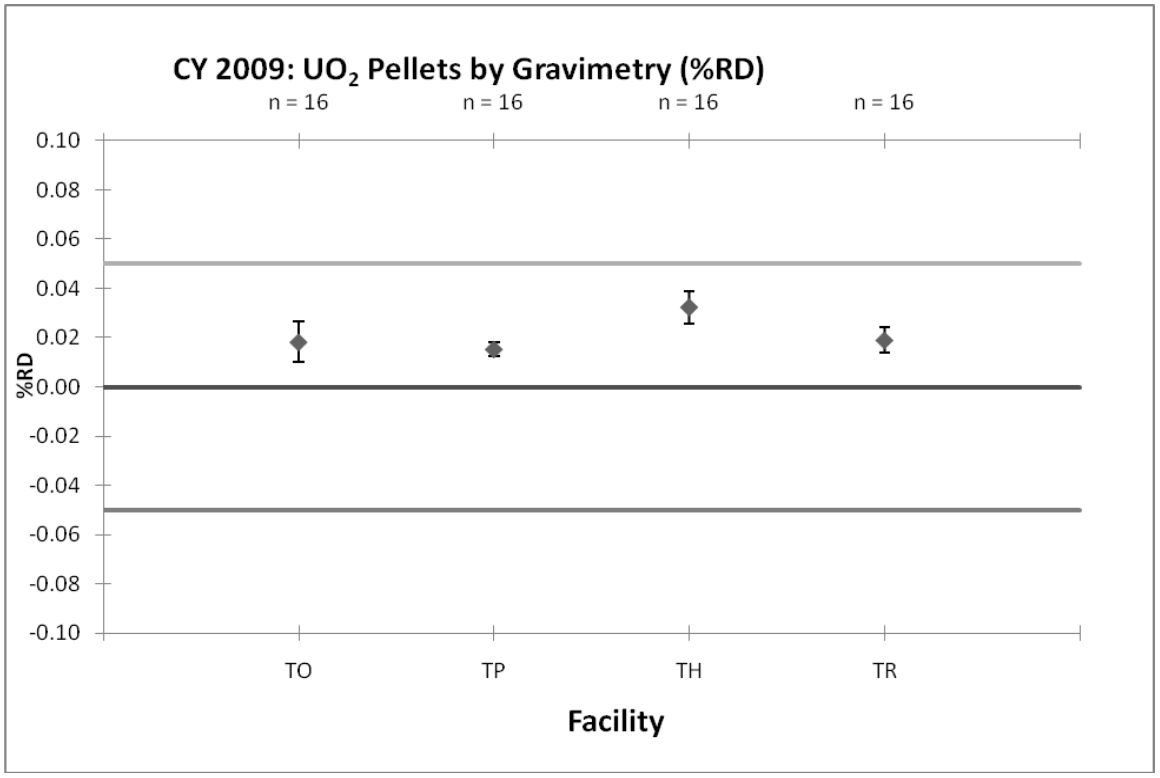


Figure H.5a. Mean % RD in elemental uranium determination in LEU-UO_2 test samples by gravimetry. All laboratories are in compliance to u(s).

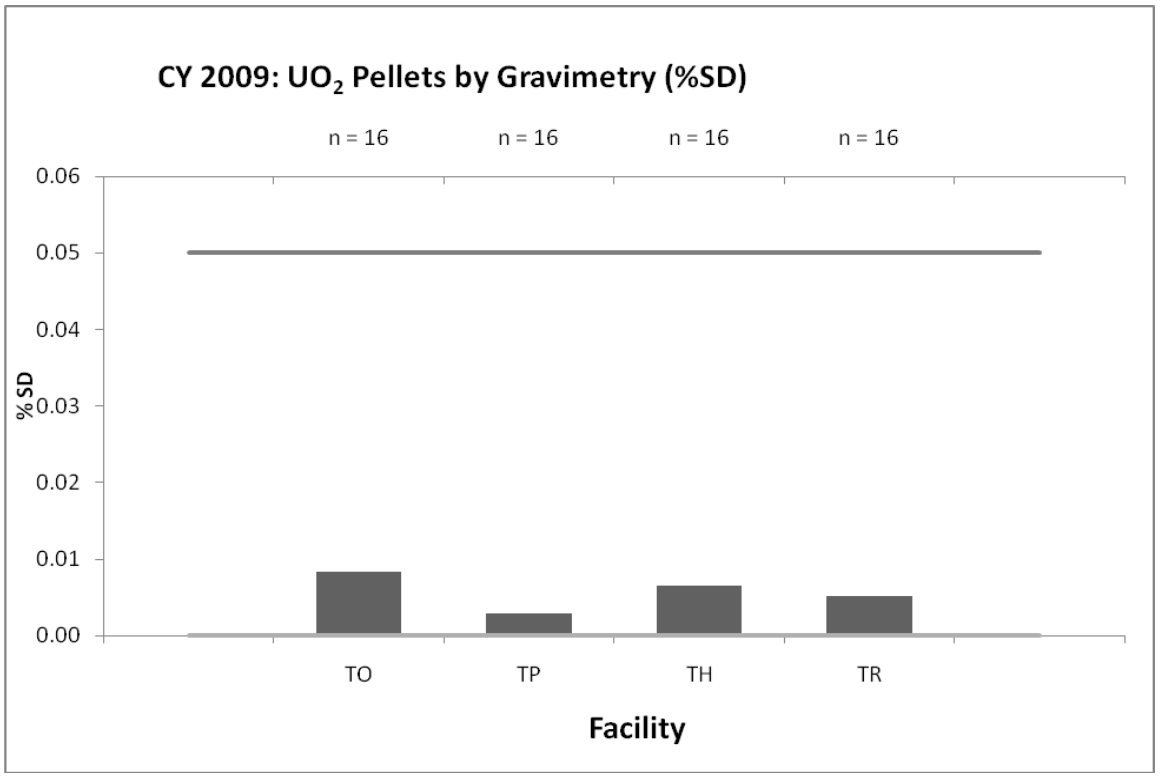


Figure H.5b. Standard deviation in elemental uranium determination in LEU-UO_2 test samples by gravimetry. All laboratories are in compliance to u(r).

H.6. UO_3 powder by D&G titration

The D&G results from analyses of UO_3 powders are shown in Table H.6, and Figures H.6a and H.6b. Mean % RDs from 4 out of 6 laboratories are within u(s); all standard deviations are within u(r).

NBL will re-characterize the test sample to ensure that the sample integrity has not been compromised due to long-term storage.

Laboratories BA and J: Need improvement in accuracy.

Table H.6. Elemental uranium in UO_3 powder test samples by D&G titration.

CY 2009: UO_3 Powder by D&G Titration					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AE	-0.081	0.035	8	Yes	Yes
BA	-0.198	0.093	28	No	Yes
BF	-0.033	0.101	8	Yes	Yes
F	-0.040	0.042	16	Yes	Yes
V	-0.081	0.037	8	Yes	Yes
J	0.145	0.062	8	No	Yes

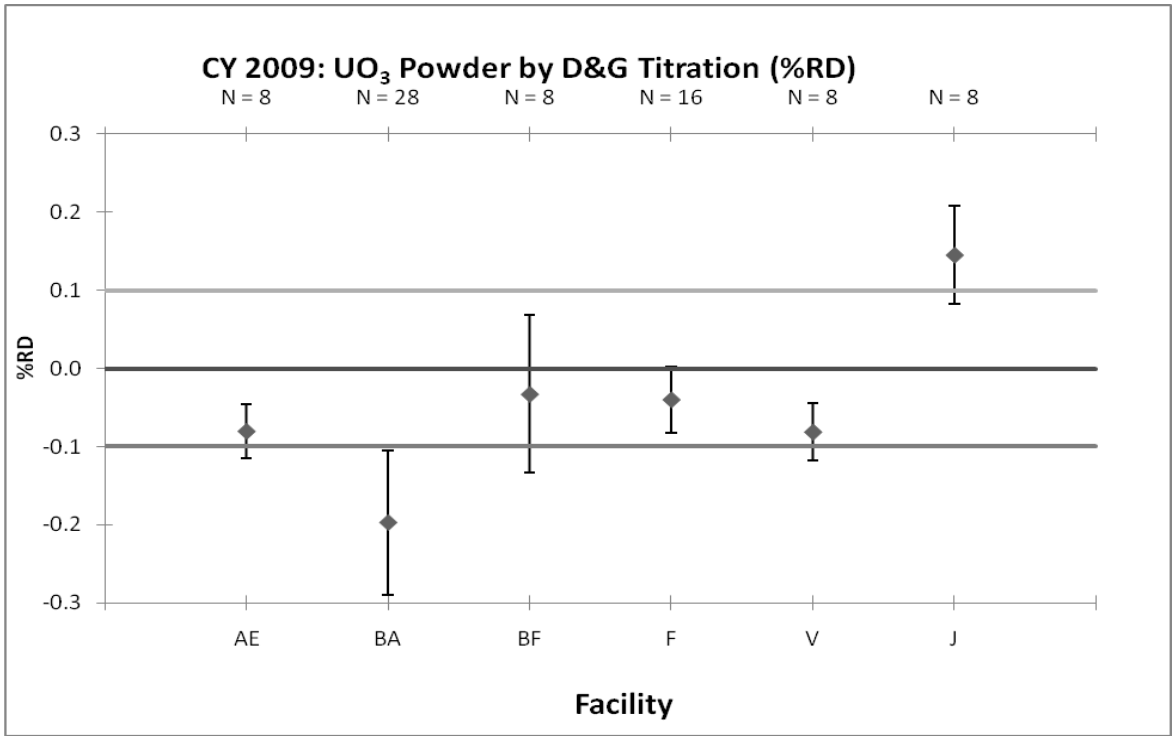


Figure H.6a. Mean % RD in elemental uranium determination in UO_3 powder test samples by D&G titration. Laboratories AE, BF, F, and V are in compliance to u(s); laboratories BA and J are outside the ITV.

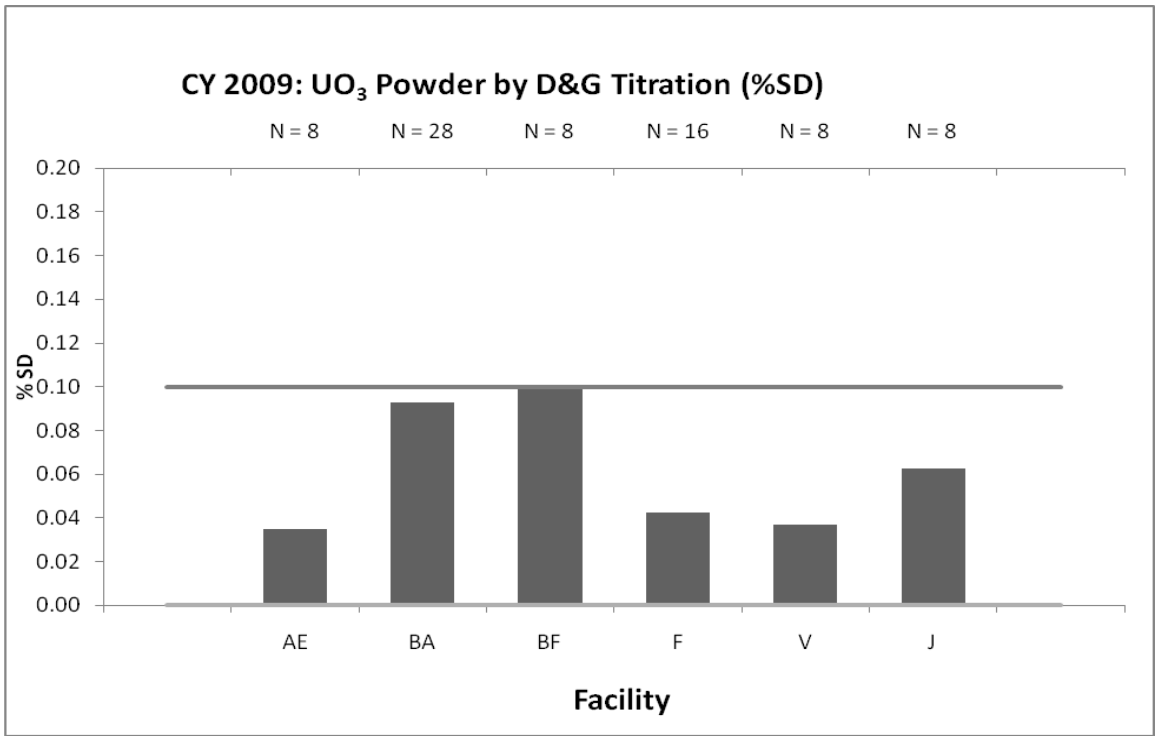


Figure H.6b. Standard deviation in elemental uranium determination in UO_3 powder test samples by D&G titration. All laboratories are in compliance to u(r).

H.7. UO_3 powder by IDMS

The IDMS results from analyses of UO_3 powders are shown in Table H.7, and Figures H.7a and H.7b. NBL will re-characterize the test sample to ensure that the sample integrity has not been compromised due to long-term storage.

Laboratory A: Need improvement in both accuracy and precision

Laboratory J: Need improvement in accuracy.

Table H.7. Elemental uranium in UO_3 powder test samples by IDMS.

CY 2009: UO_3 Powder by IDMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.15
J	0.083	0.088	8	Yes	Yes
A	-0.258	0.251	8	No	No

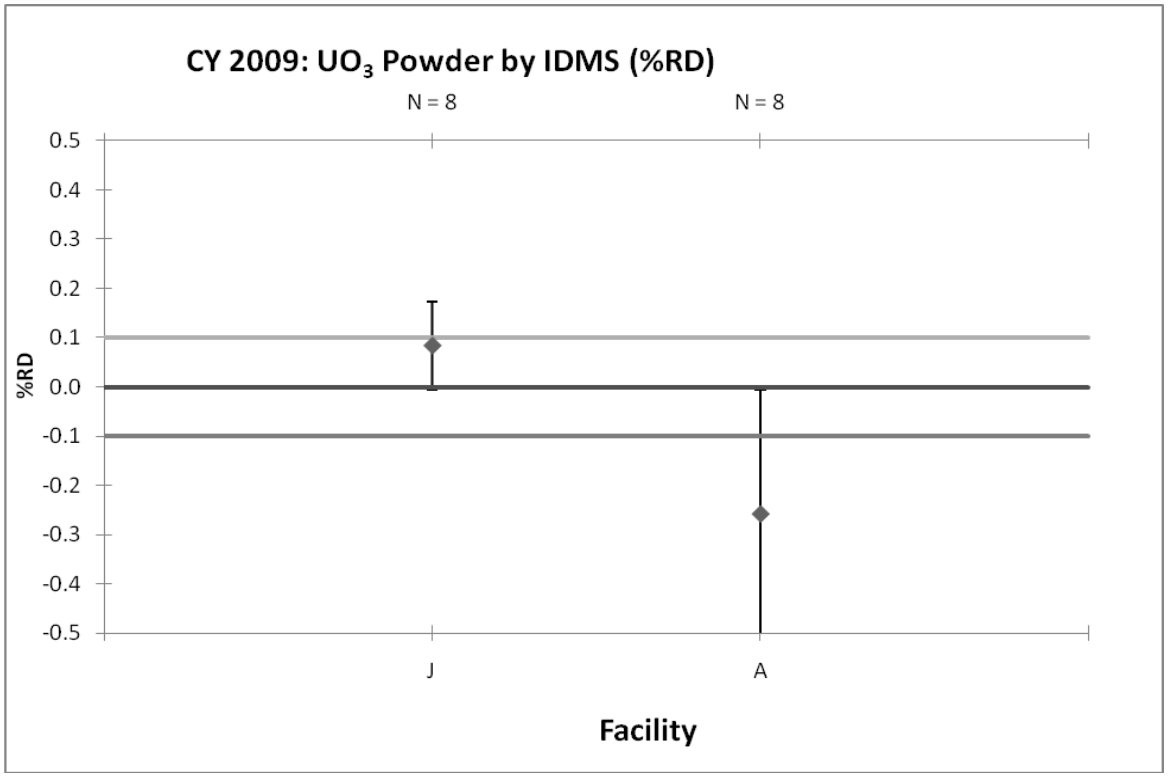


Figure H.7a. Mean % RD in elemental uranium determination in UO_3 powder test samples by IDMS. Laboratory J is in compliance to u(s); laboratory A is outside the ITV.

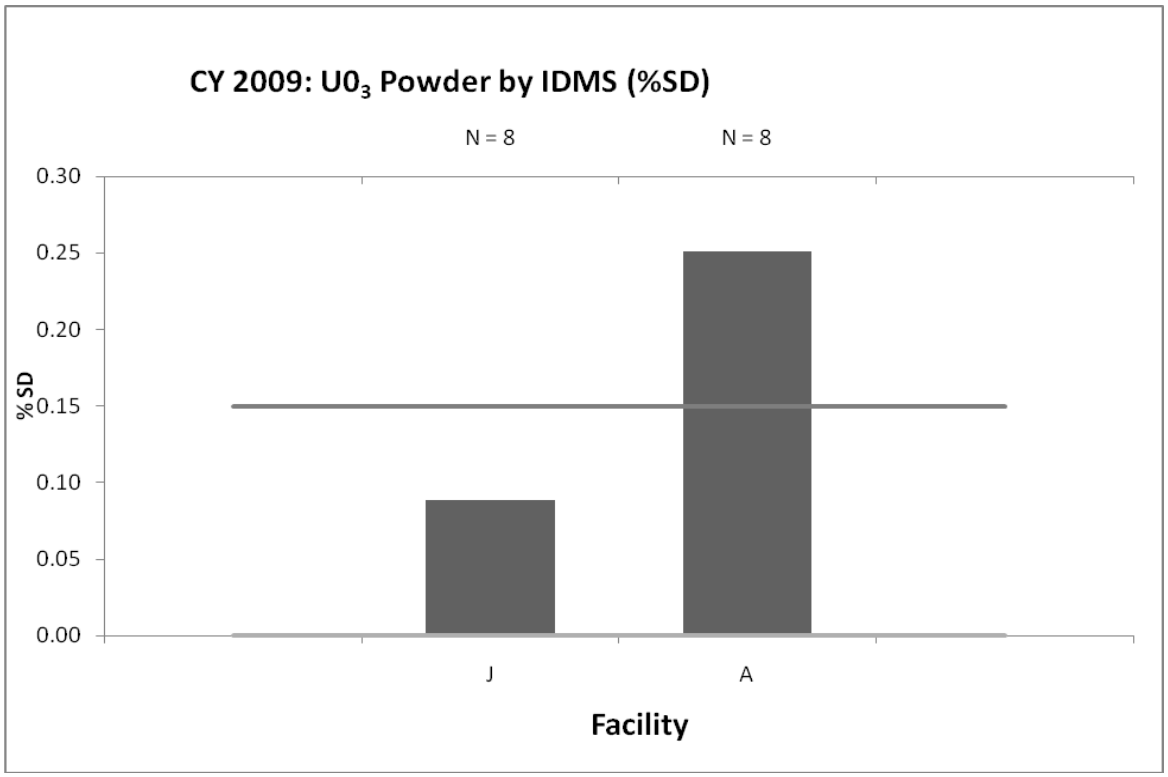


Figure H.7b. Standard deviation in elemental uranium determination in UO_3 powder test samples by IDMS. Laboratory J is in compliance to u(r); laboratory A is outside the ITV.

H.8. UO_3 powder by XRF

The XRF results from analyses of UO_3 powders are shown in Table H.8, and Figures H.8a and H.8b. NBL will re-characterize the test sample to ensure that the sample integrity has not been compromised due to long-term storage.

Table H.8. Elemental uranium in UO_3 powder test samples by XRF.

CY 2009: UO_3 Powder by XRF					
Lab Code	Mean % RD	SD	N	Target Value Compliance*	
				u(s) = 0.5	u(r) = 0.5
A	-0.428	0.312	16	Yes	Yes

*ITV is not specified for XRF; DOE target value is shown.

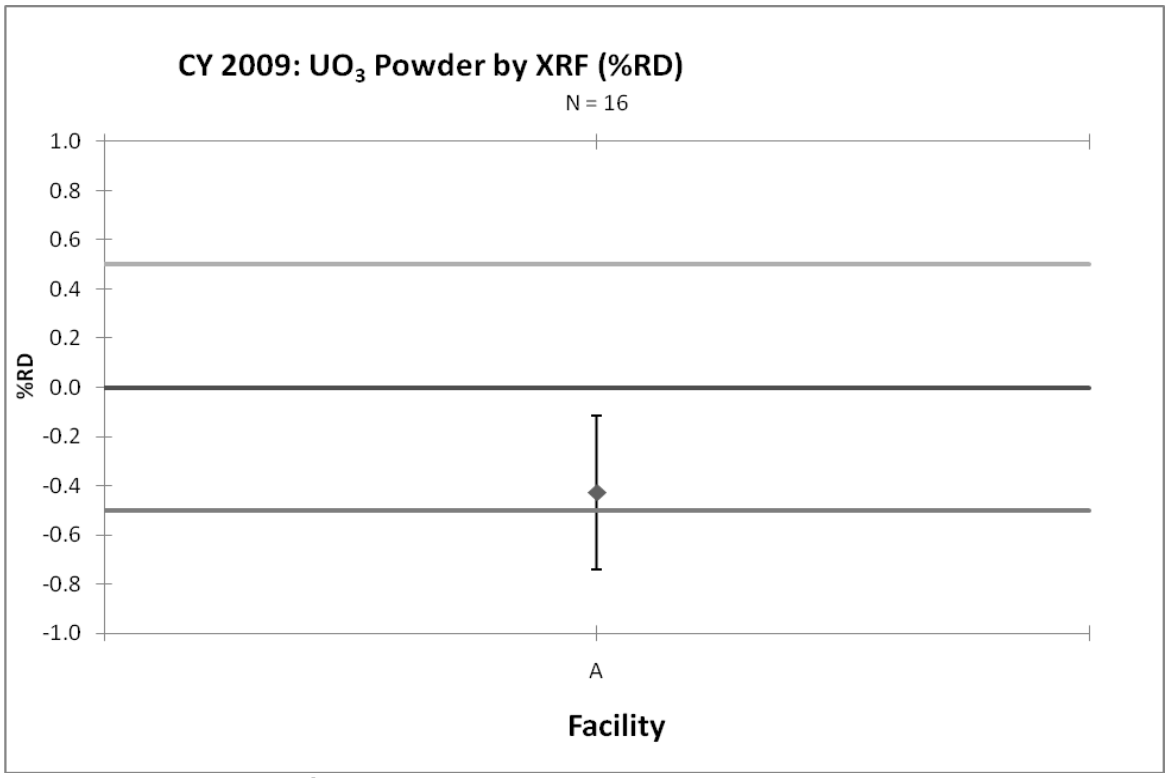


Figure H.8a. Mean % RD in elemental uranium content determination in UO_3 powder test samples by XRF. Laboratory A is in compliance with u(s).

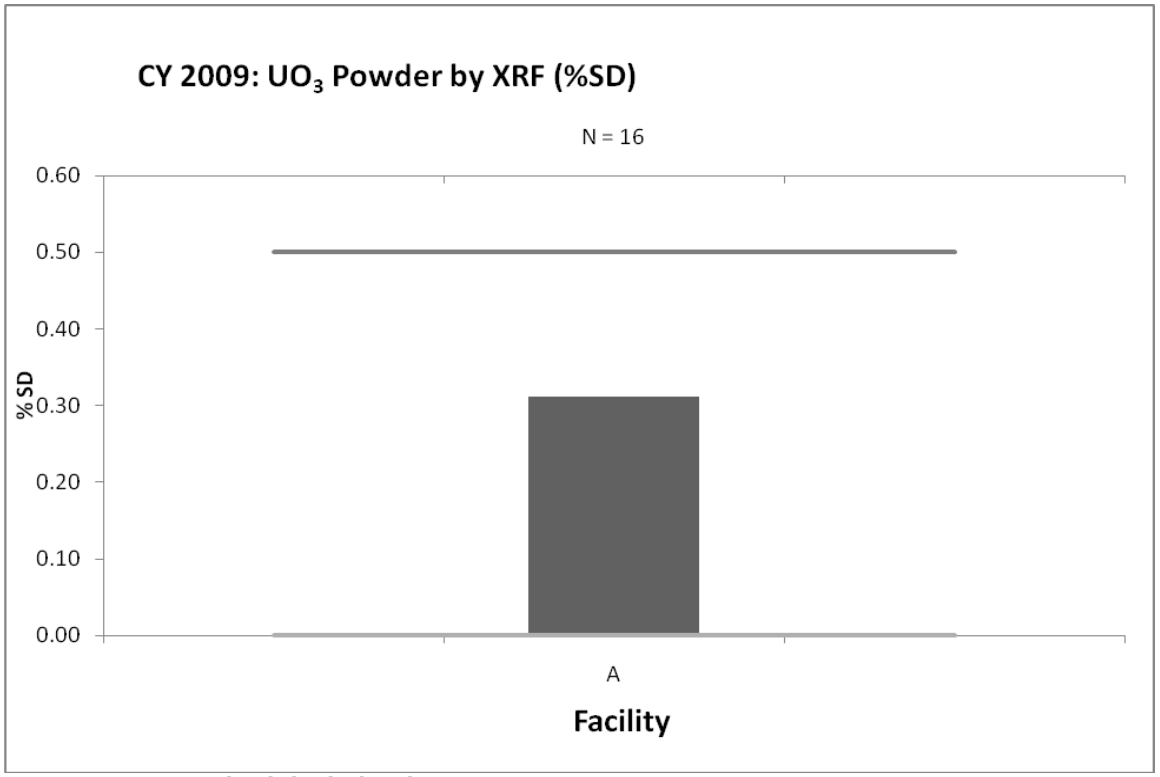


Figure H.8b. Standard deviation in elemental uranium content determination in UO_3 powder test samples by XRF. Laboratory A is in compliance with u(r).

H.9. U_3O_8 powder by D&G titration

The D&G results from analyses of U_3O_8 powders are shown in Table H.9, and Figures H.9a and H.9b. The mean % RD given in this table is about 0.04 % lower than the values reported earlier to the participants, except for laboratory BA. The error in the earlier evaluations occurred because of using a higher reference value corresponding to samples ignited at 800°C instead of using a lower value for samples analyzed as received (i.e., without ignition). Except for laboratory BA, all others analyzed the samples as received. The standard deviations (for all facilities) reported previously remain unchanged.

Laboratories BC and BE: Insufficient accuracy and precision in the determinations.

Laboratory AB and J: Insufficient accuracy; acceptable precision.

Table H.9. Elemental uranium in U_3O_8 powder test samples by D&G titration.

CY 2009: U3O8 Powder by D&G Titration					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AB	0.154	0.052	12	No	Yes
BA	-0.008	0.041	10	Yes	Yes
BC	-0.225	0.180	16	No	No
BE	-0.193	0.130	16	No	No
J	0.187	0.062	8	No	Yes

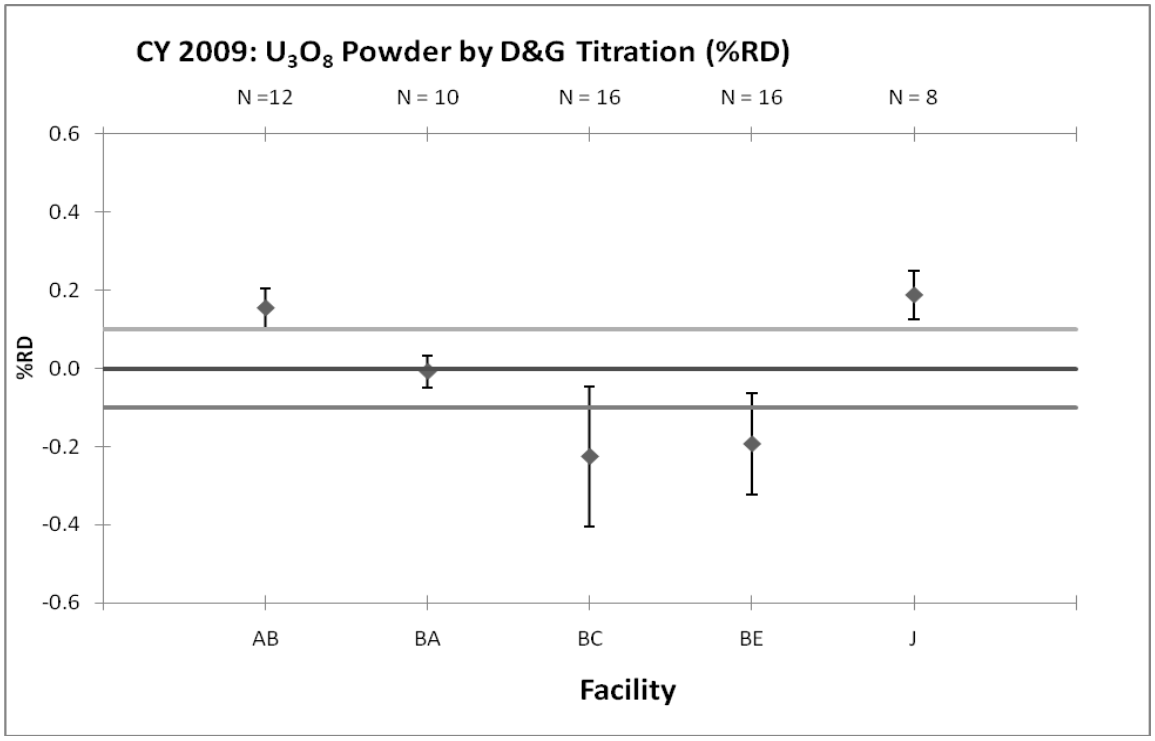


Figure H.9a. Mean % RD in elemental uranium determination in U_3O_8 powder test samples by D&G titration. Laboratory BA is in compliance to u(s); laboratories AB, BC, BE, and J are outside the ITV.

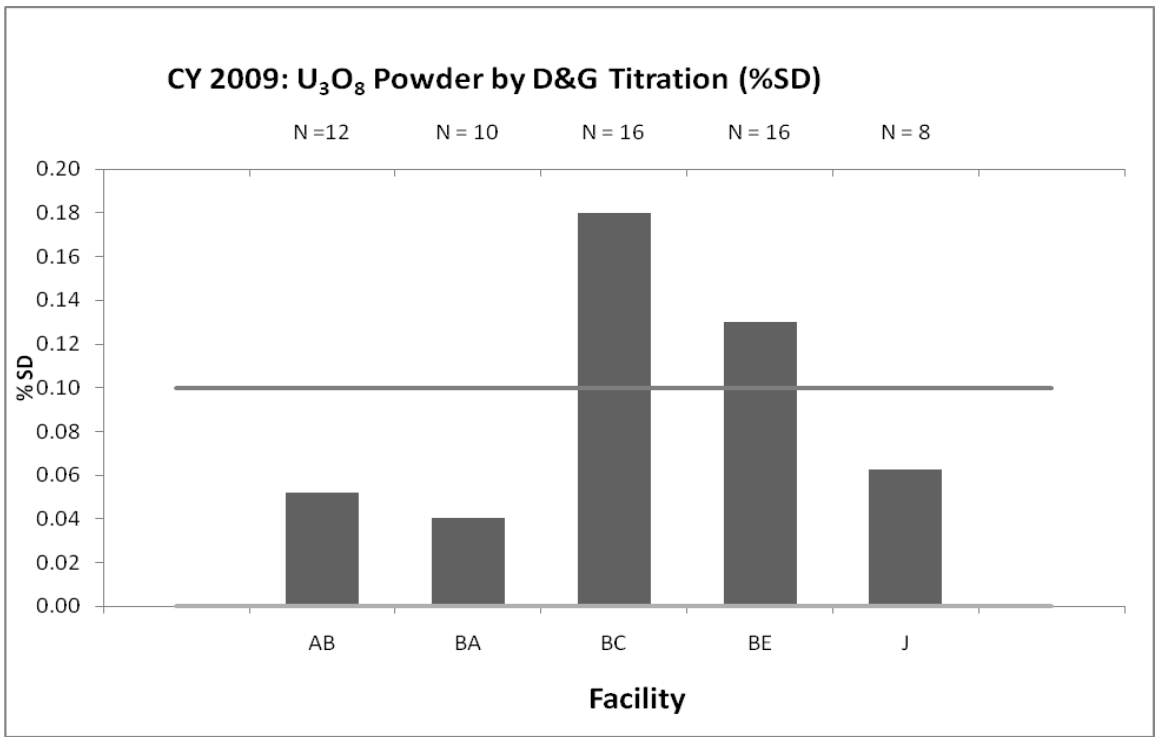


Figure H.9b. Standard deviation in elemental uranium determination in U_3O_8 powder test samples by D&G titration. Laboratories AB, BA, and J are in compliance to u(r); laboratories BC and BE are outside the ITV.

H.10. U_3O_8 powder by IDMS

The IDMS results from analyses of U_3O_8 powders are shown in Table H.10, and Figures H.10a and H.10b. The mean % RD given in this table is about 0.04 % lower than the values reported earlier to the participants. The error in the earlier evaluations occurred because of using a higher reference value corresponding to samples ignited at 800°C instead of using a lower value for samples analyzed as received (i.e., without ignition) in these two laboratories. The standard deviations previously reported remain unchanged.

Laboratories J and JA: Insufficient accuracy.

Table H.10. Elemental uranium in U_3O_8 powder test samples by IDMS.

CY 2009: U_3O_8 Powder by IDMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.15
J	0.125	0.088	8	No	Yes
JA	-0.138	0.074	8	No	Yes

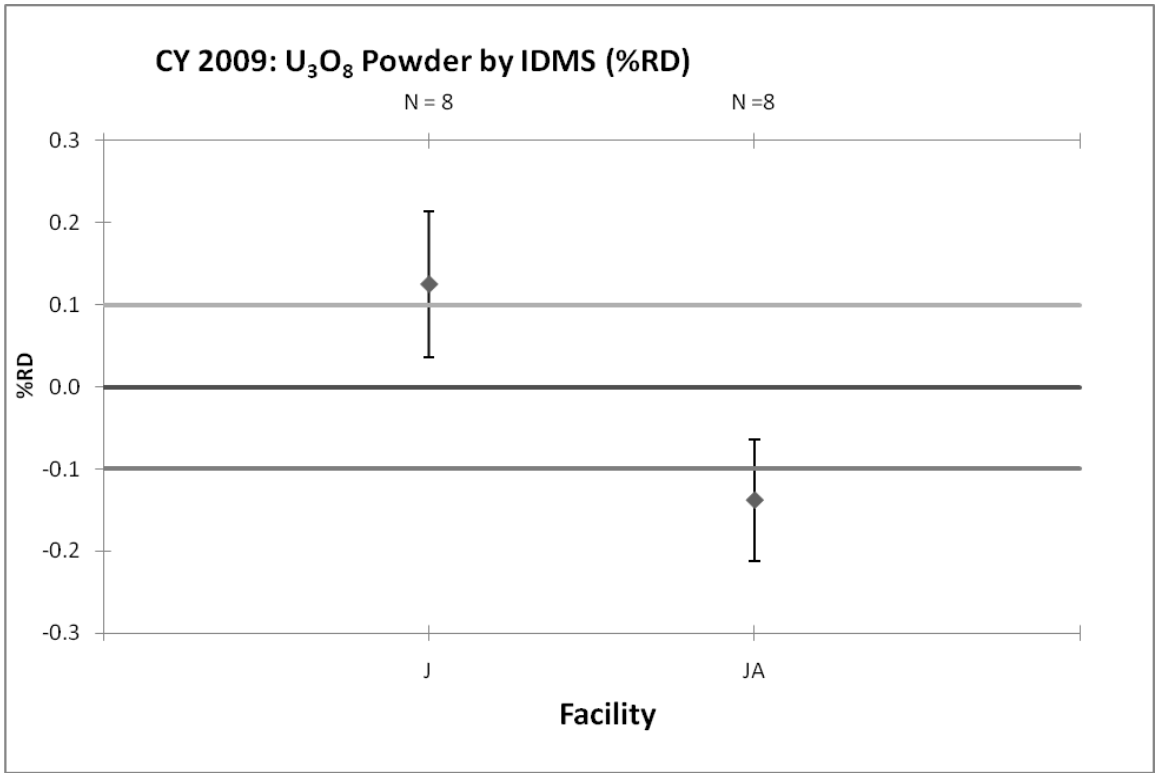


Figure H.10a. Mean % RD in elemental uranium determination in U_3O_8 powder test samples by IDMS. Both laboratories J and JA are outside the ITV.

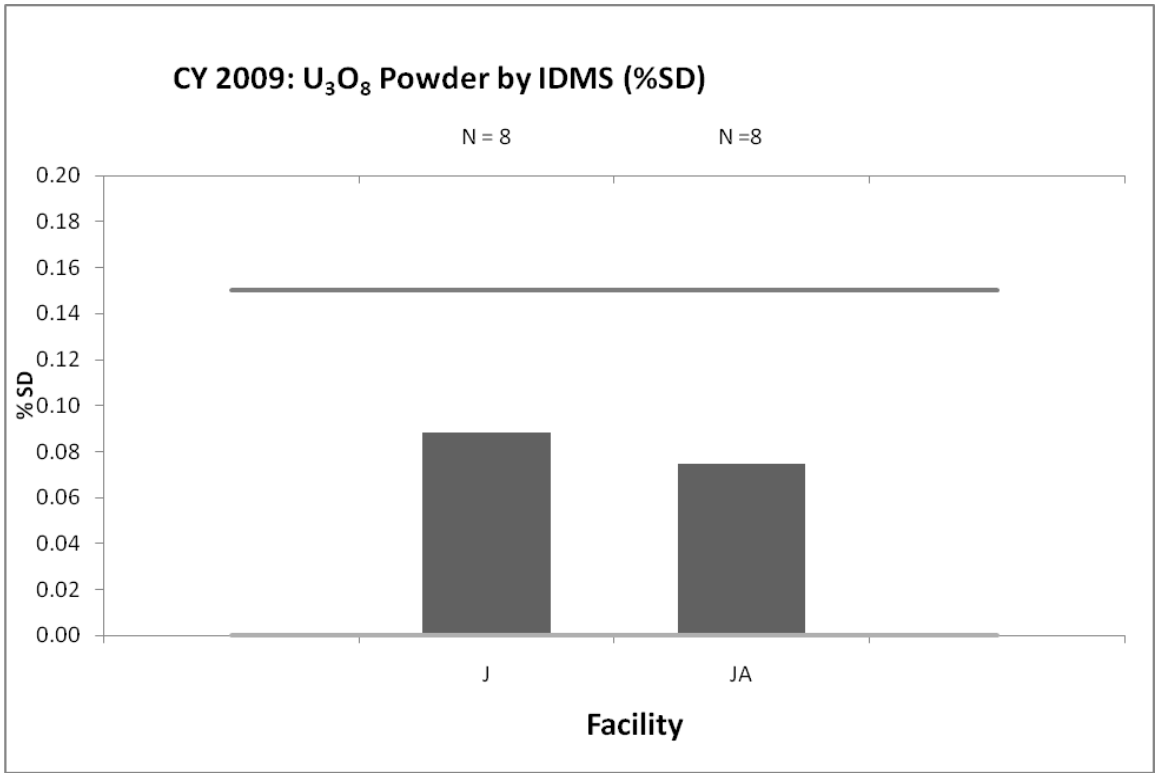


Figure H.10b. Standard deviation in elemental uranium determination in U_3O_8 powder test samples by IDMS. Both laboratories J and JA are in compliance to u(r).

H.11. UF_6 by D&G titration

The D&G results from analyses of hydrolyzed solutions of UF_6 test samples are shown in Table H.11, and Figures H.11a and H.11b.

Laboratory EA: Need improvement in both accuracy and precision

Laboratory BF and EC: Need improvement in accuracy.

Table H.11. Elemental uranium in UF_6 test samples by D&G titration.

CY 2009: UF_6 by D&G Titration					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AE	-0.050	0.022	8	Yes	Yes
BF	-0.513	0.021	19	No	Yes
EA	-0.216	0.169	16	No	No
EC	0.147	0.025	8	No	Yes

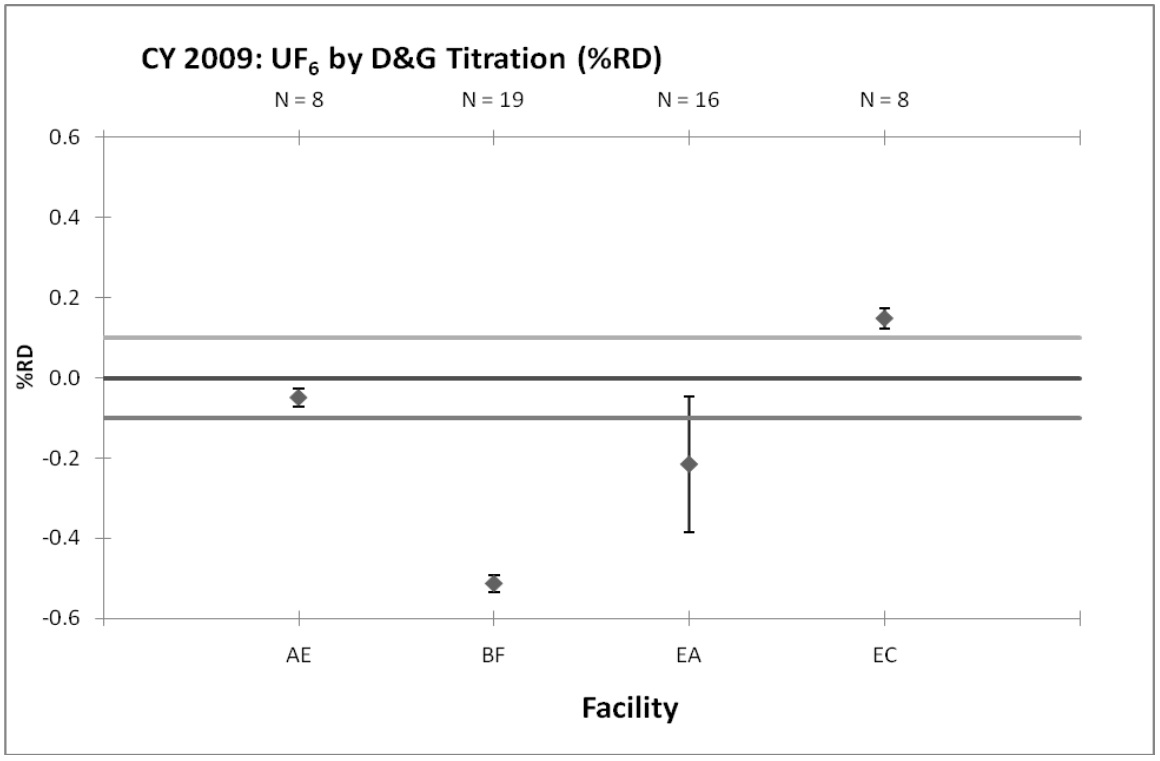


Figure H.11a. Mean % RD in elemental uranium determination in UF_6 test samples by D&G titration. Laboratory AE is in compliance to u(s); laboratories BF, EA, and EC are outside the ITV.

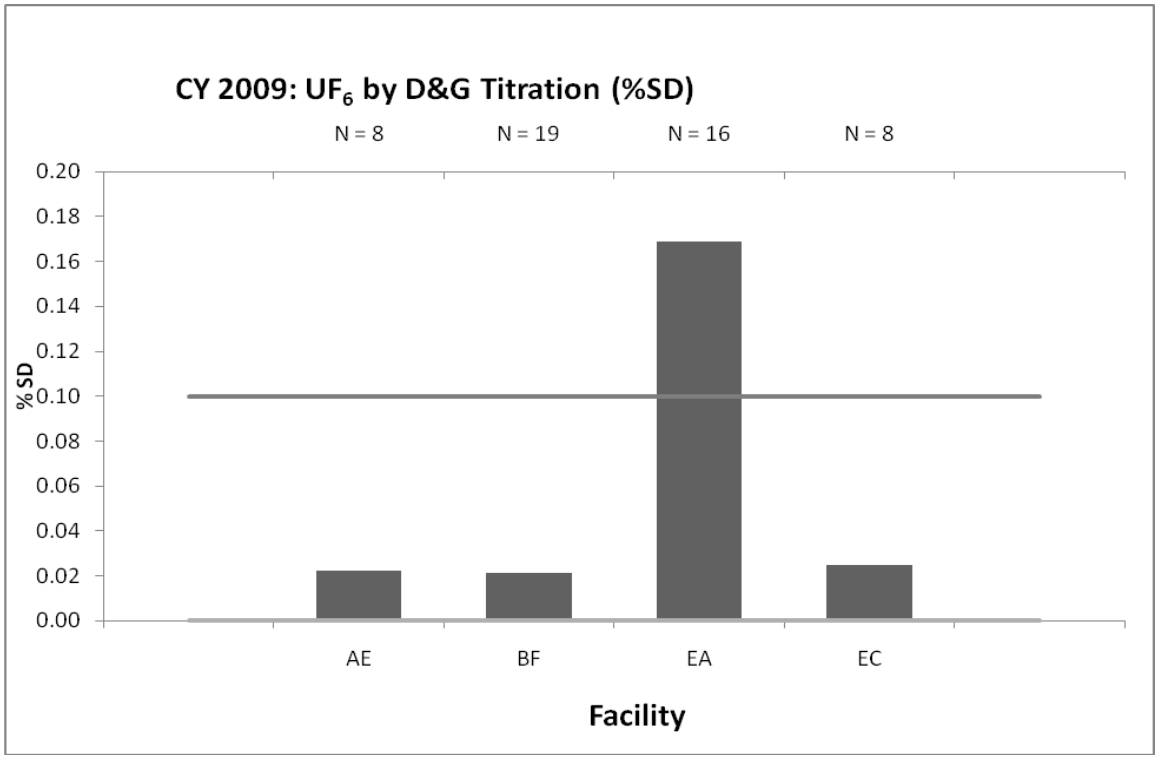


Figure H.11b. Standard deviation in elemental uranium determination in UF_6 test samples by D&G titration. Laboratories AE, BF, and EC are in compliance to u(r); laboratory EA is outside the ITV.

I. ANNUAL EVALUATION OF ^{235}U ISOTOPE ABUNDANCE MEASUREMENTS

The tables and graphs of material/method/laboratory/enrichment specific annual evaluations of ^{235}U isotope measurement results are presented in sections I.1 to I.12. The ^{235}U enrichment levels are divided into four groups in accordance to the scheme in ITV 2000:

DU	^{235}U < 0.3 wt %
U:	0.3 wt % < ^{235}U < 1 wt %
LEU:	1 wt % < ^{235}U < 20 wt %
HEU	^{235}U > 20 wt %.

Table I. Annual evaluation of ^{235}U isotope measurement results.

Material	**Method**		
	TIMS	**ICPMS**	**GSMS**
U (0.3 wt % < ^{235}U < 1 wt %)			
UNH solution	Section I.1	Section I.2	
UO_3 powder	Section I.3		
U_3O_8 powder	Section I.4		
LEU (1 wt % < ^{235}U < 20 wt %)			
UNH solution	Section I.5	Section I.6	
UO_2 pellet	Section I.7		
UF_6	Section I.8	Section I.9	Section I.10
HEU (^{235}U > 20 wt %)			
UNH solution	Section I.11	Section I.12	

I.1. U-UNH (Uranyl nitrate solutions) by TIMS

The TIMS results from analyses of U-UNH (0.3 wt % < ^{235}U < 1 wt %) solutions are shown in Table I.1, and Figures I.1a and I.1b.

Table I.1. ^{235}U abundance in U-UNH test samples by TIMS.

CY 2009: U-UNH Solution by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.2	u(r) = 0.2
J	-0.046	0.058	8	Yes	Yes

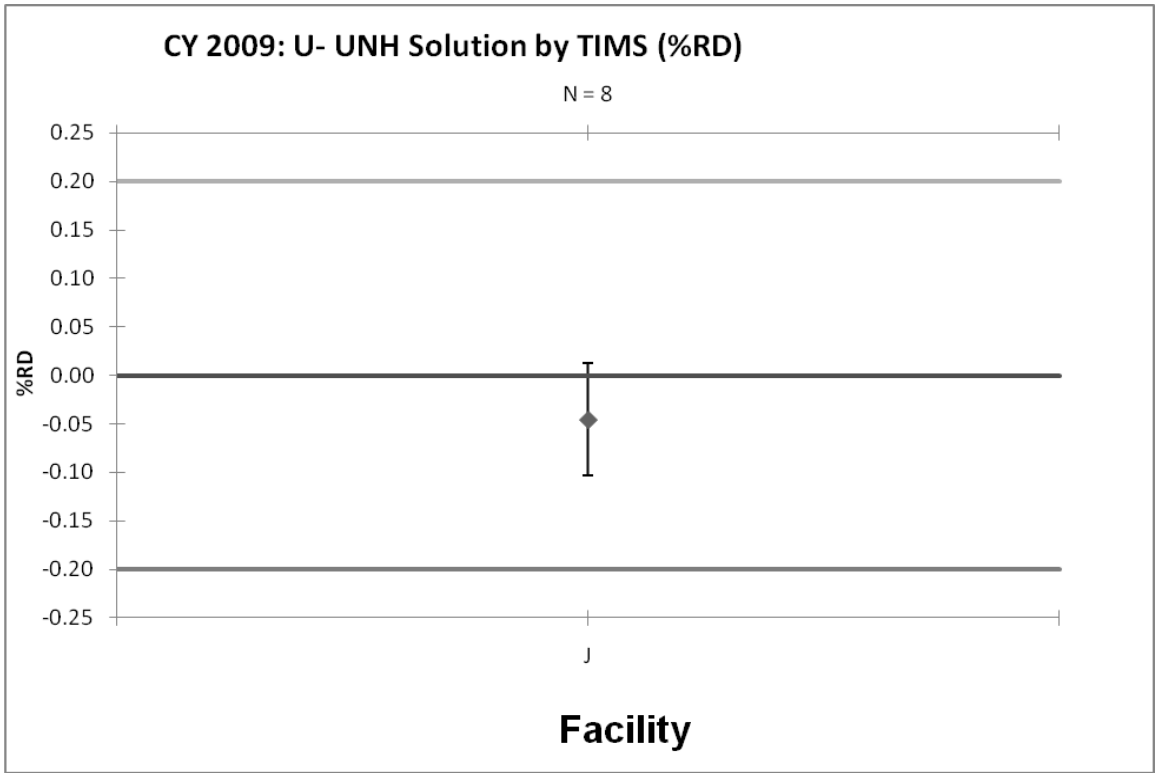


Figure I.1a. Mean % RD in ^{235}U abundance determination in U-UNH test samples by TIMS. Laboratory J is in compliance to u(s).

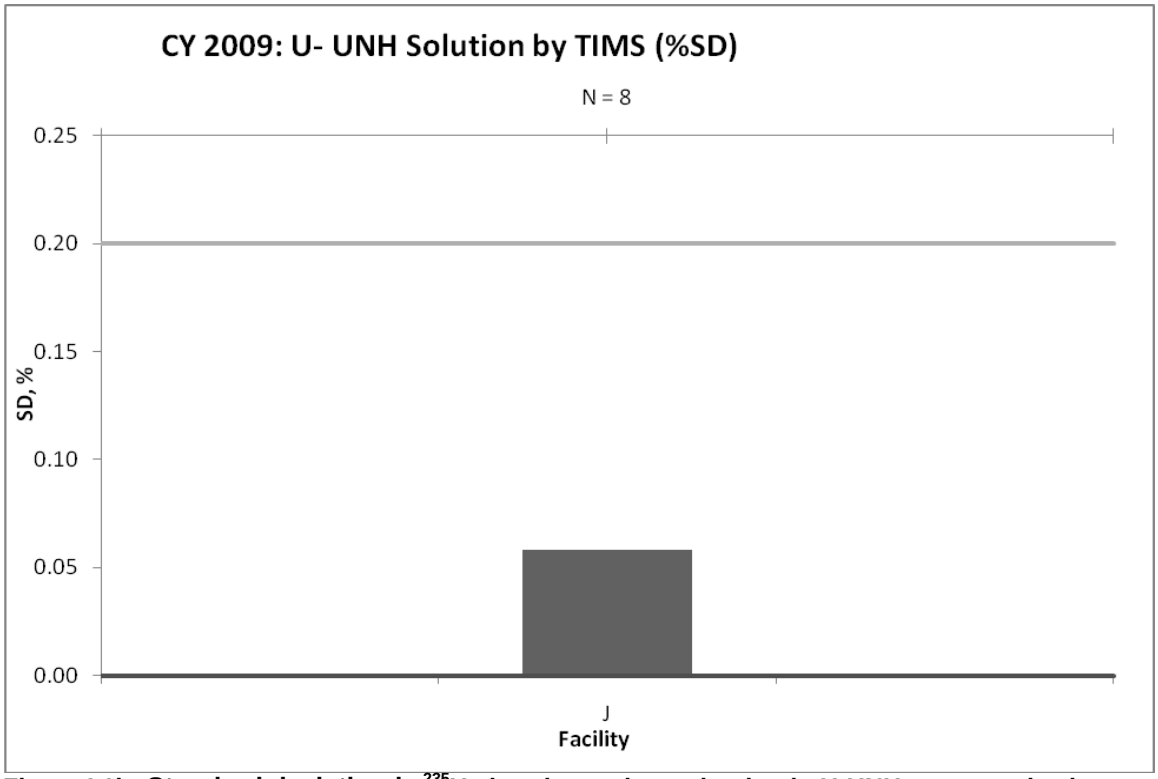


Figure I.1b. Standard deviation in ^{235}U abundance determination in U-UNH test samples by TIMS. Laboratory J is in compliance to u(r).

I.2. U-UNH (Uranyl nitrate solutions) by ICPMS

The ICPMS results from analyses of U-UNH (0.3 wt % < ^{235}U < 1 wt %) solutions are shown in Table I.2, and Figures I.2a and I.2b.

Laboratory D: Need to improve both accuracy and precision.

Table I.2. ^{235}U abundance in U-UNH test samples by ICPMS.

CY 2009: U-UNH Solution by ICP-MS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.2	u(r) = 0.2
D	-0.615	0.253	8	No	No
EA	-0.025	0.063	32	Yes	Yes

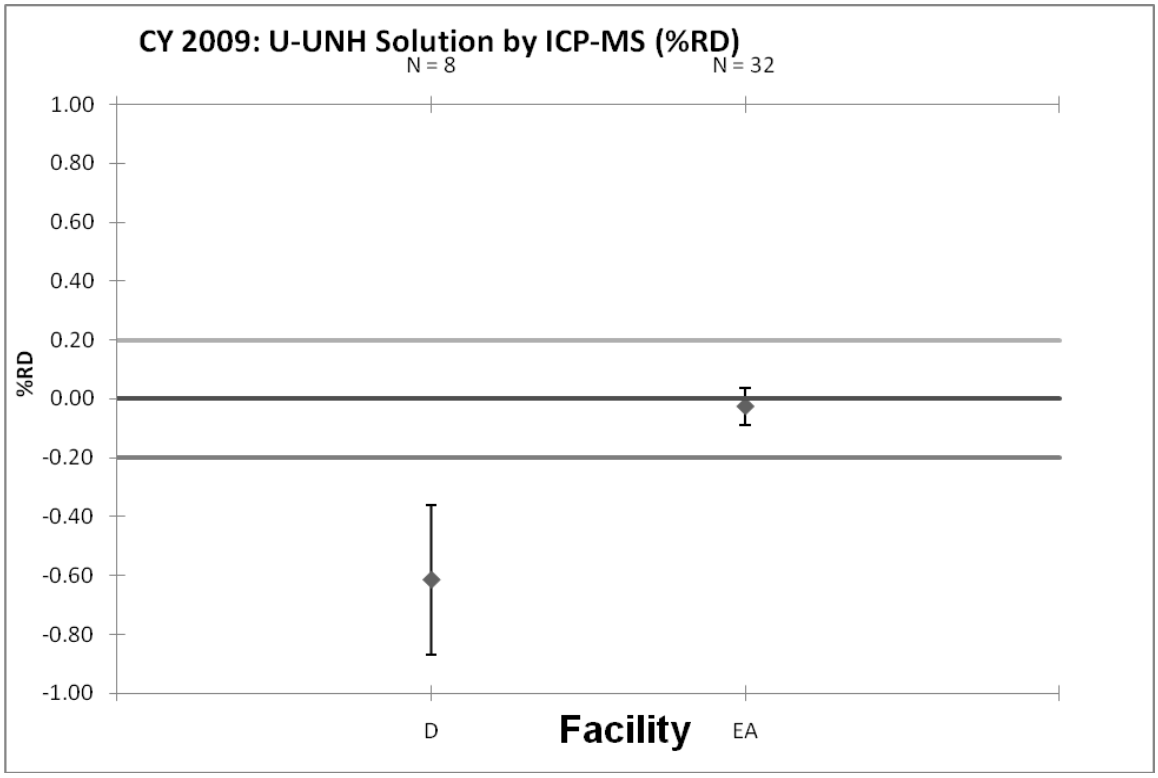


Figure I.2a. Mean % RD in ^{235}U abundance determination in U-UNH test samples by ICPMS. Laboratory EA is in compliance to u(s); laboratory D is outside the ITV.

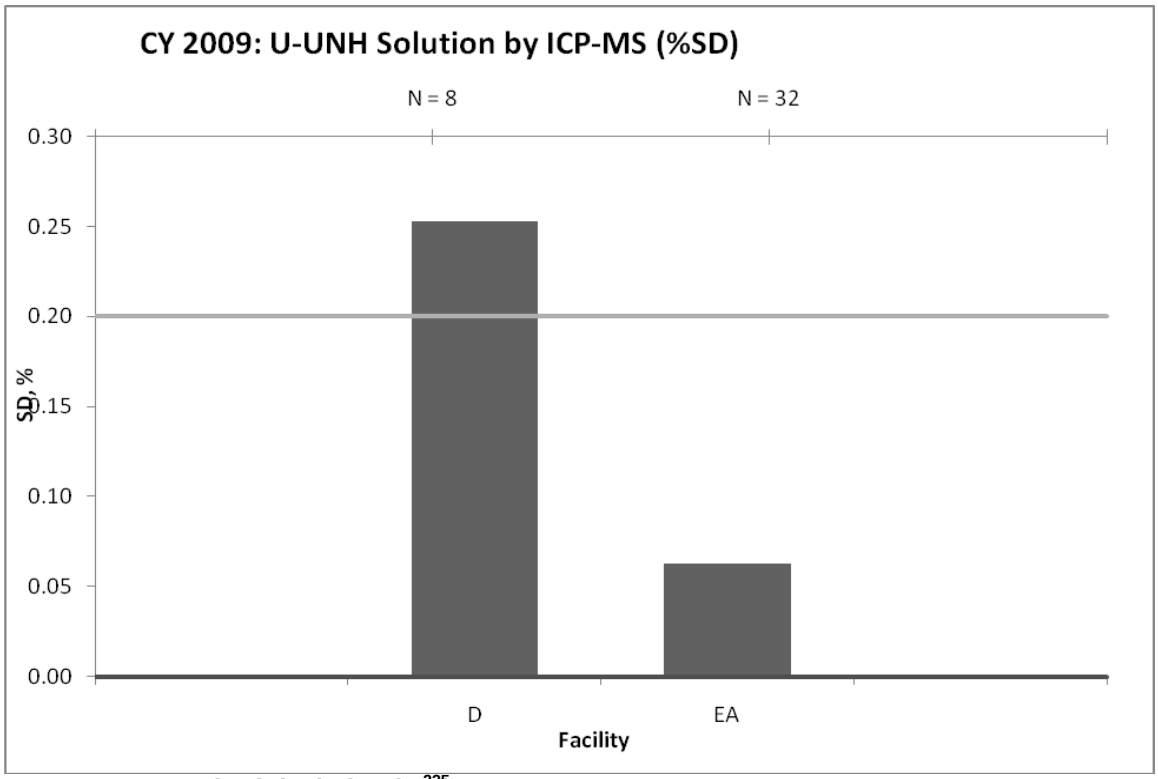


Figure I.2b. Standard deviation in ^{235}U abundance determination in U-UNH test samples by ICPMS. Laboratory EA is in compliance to u(r); laboratory D is outside the ITV.

I.3. $U\text{-}UO_3$ powder by TIMS

The TIMS results from analyses of $U\text{-}UO_3$ (0.3 wt % < ^{235}U < 1 wt %) powder are shown in Table I.3, and Figures I.3a and I.3b.

Table I.3. ^{235}U abundance in $U\text{-}UO_3$ powder test samples by TIMS.

CY 2009: $U\text{-}UO_3$ Powder by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.2	u(r) = 0.2
J	-0.031	0.041	8	Yes	Yes

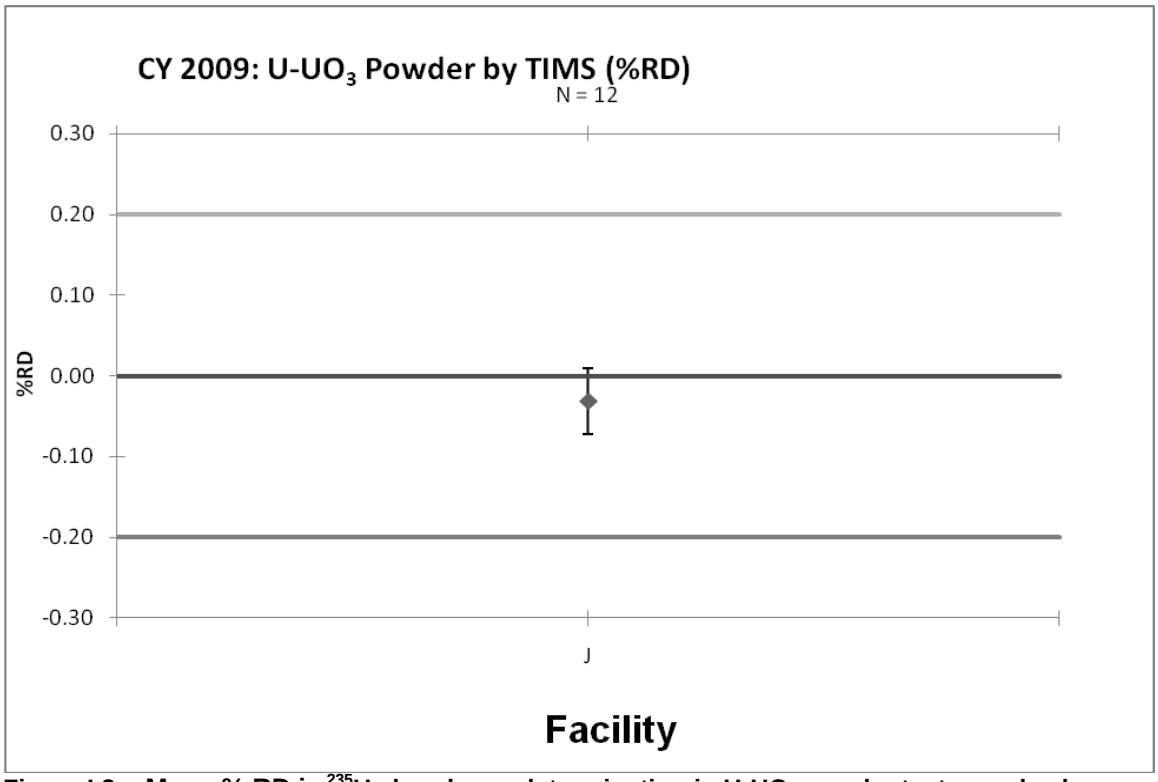


Figure I.3a. Mean % RD in ^{235}U abundance determination in U-UO_3 powder test samples by TIMS. Laboratory J is in compliance to u(s).

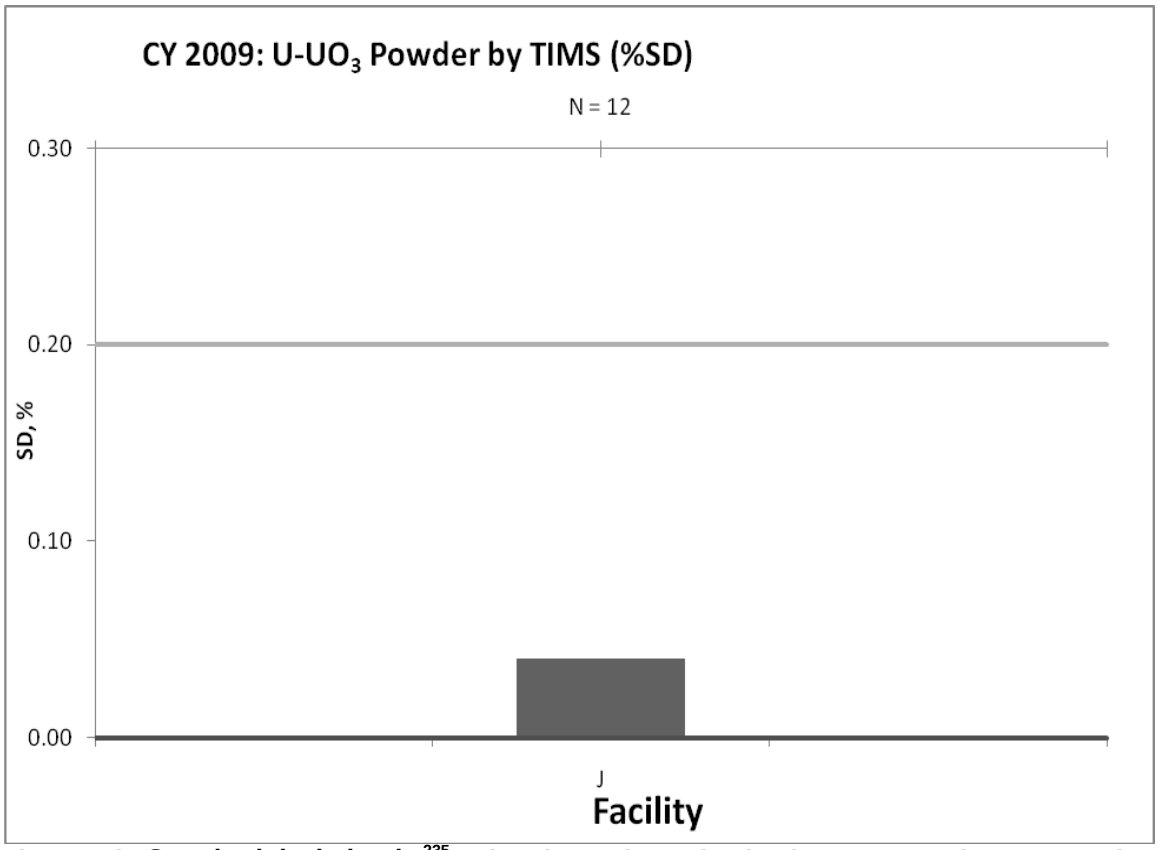


Figure I.3b. Standard deviation in ^{235}U abundance determination in U-UO_3 powder test samples by TIMS. Laboratory J is in compliance to u(r).

I.4. $U\text{-}U_3O_8$ powder by TIMS

The TIMS results from analyses of $U\text{-}U_3O_8$ (0.3 wt % < ^{235}U < 1 wt %) powder are shown in Table I.4, and Figures I.4a and I.4b.

Table I.4. ^{235}U abundance in $U\text{-}U_3O_8$ powder test samples by TIMS.

CY 2009: $U\text{-}U_3O_8$ Powder by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.2	u(r) = 0.2
BC	-0.108	0.055	12	Yes	Yes

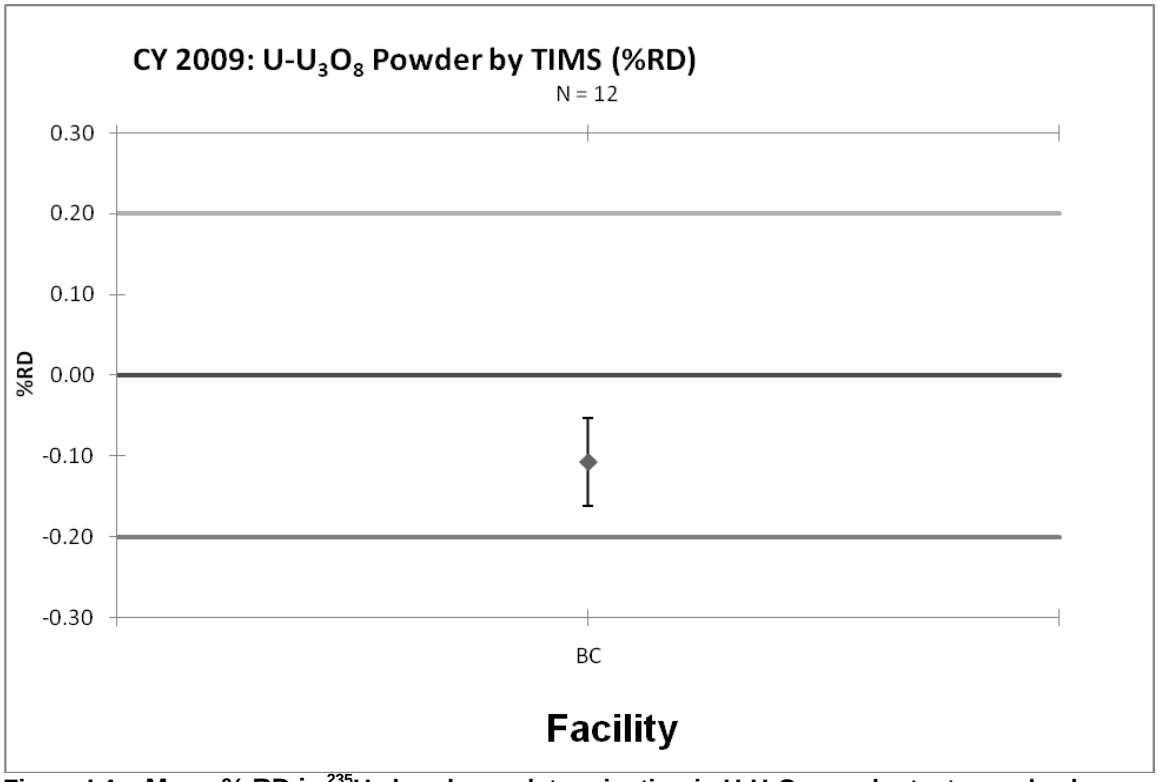


Figure I.4a. Mean % RD in ^{235}U abundance determination in U-U_3O_8 powder test samples by TIMS. Laboratory BC is in compliance to u(s).

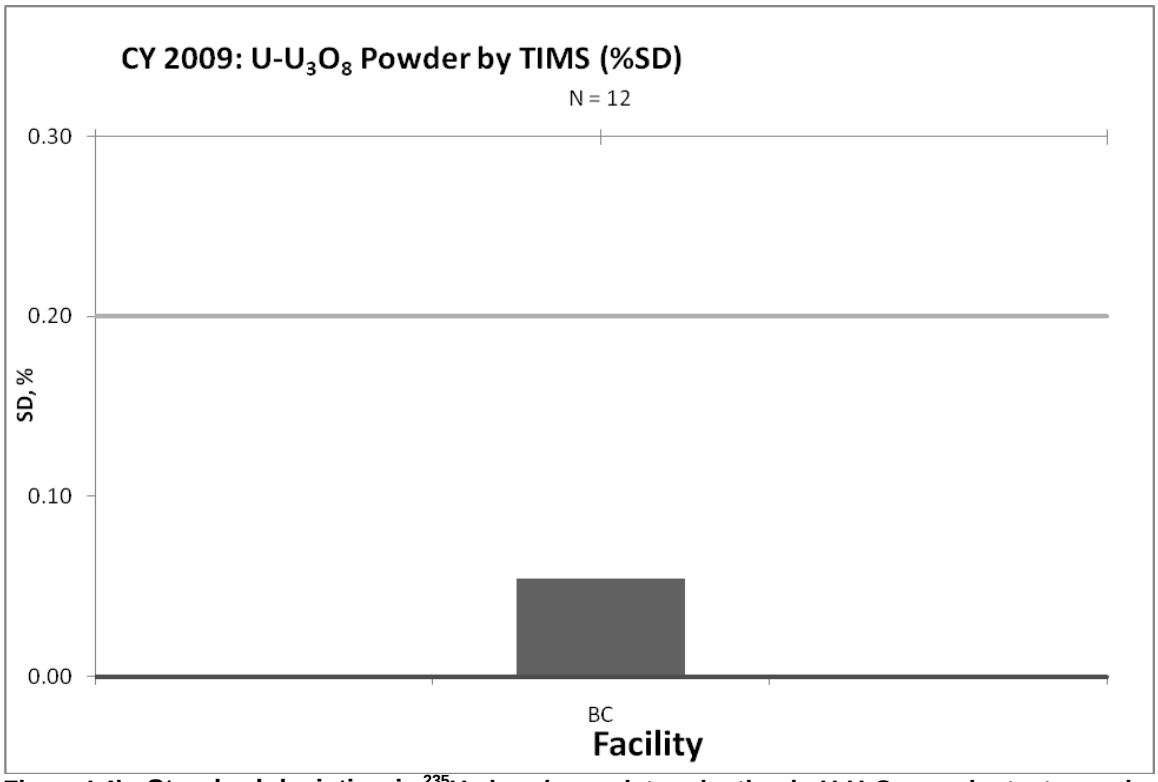


Figure I.4b. Standard deviation in ^{235}U abundance determination in U-U_3O_8 powder test samples by TIMS. Laboratory BC is in compliance to u(r).

I.5. LEU-UNH (Uranyl nitrate solutions) by TIMS

The TIMS results from analyses of LEU-UNH (1 wt % < ^{235}U < 20 wt %) solutions are shown in Table I.5, and Figures I.5a and I.5b.

Laboratory B: Need improvement in accuracy

Laboratory SA: Need improvement in precision.

Table I.5. ^{235}U abundance in LEU-UNH test samples by TIMS.

CY 2009: LEU-UNH Solution by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AA	-0.023	0.099	16	Yes	Yes
B	0.242	0.035	8	No	Yes
BC	0.010	0.089	8	Yes	Yes
F	0.002	0.017	8	Yes	Yes
J	-0.004	0.020	8	Yes	Yes
SA	-0.059	0.215	8	Yes	No

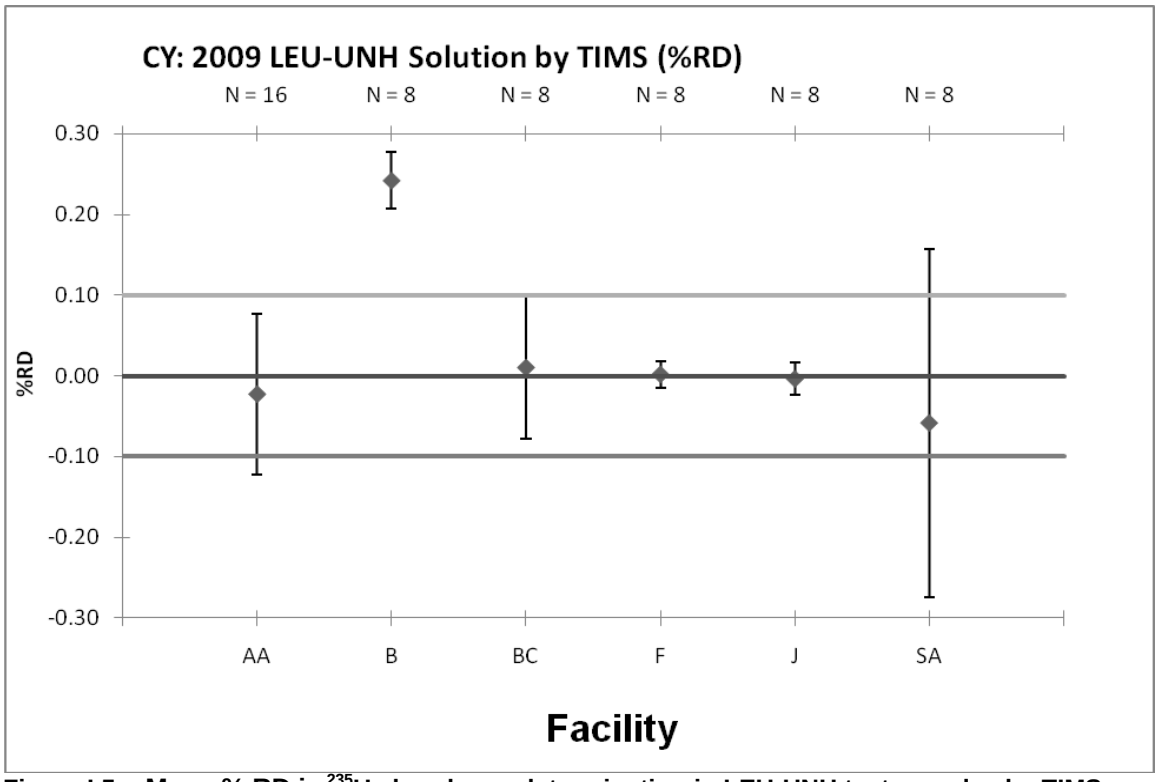


Figure I.5a. Mean % RD in ^{235}U abundance determination in LEU-UNH test samples by TIMS. Laboratories AA, BC, F, J, and SA are in compliance to u(s); laboratory B is outside the ITV.

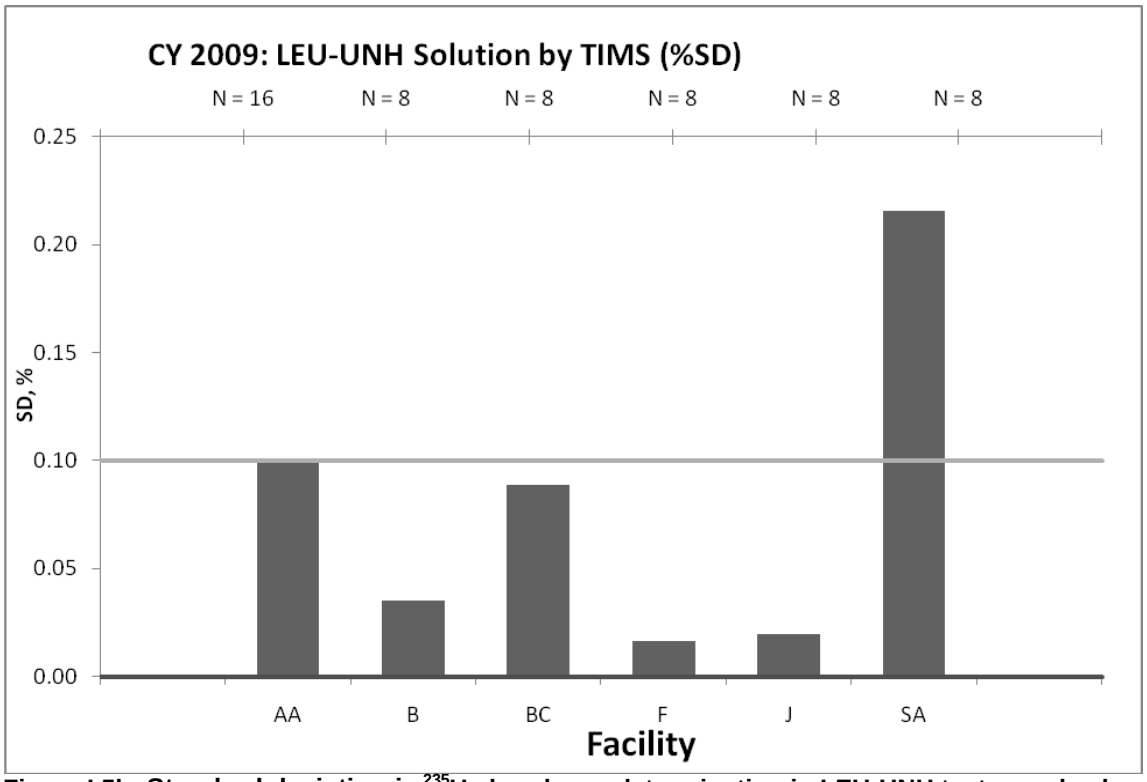


Figure I.5b. Standard deviation in ^{235}U abundance determination in LEU-UNH test samples by TIMS. Laboratories AA, B, BC, F, and J are in compliance to u(s); laboratory SA is outside the ITV.

I.6. LEU-UNH (Uranyl nitrate solutions) by ICPMS

The ICPMS results from analyses of LEU-UNH (1 wt % < ^{235}U < 20 wt %) solutions are shown in Table I.6, and Figures I.6a and I.6b.

Laboratories AD, AF, and D: Need to improve both accuracy and precision.

Table I.6. ^{235}U abundance in LEU-UNH test samples by ICPMS.

CY 2009: LEU-UNH Solution by ICP-MS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AD	-0.552	0.466	16	No	No
AF	-2.318	0.176	32	No	No
D	0.295	0.417	8	No	No
EA	-0.011	0.050	32	Yes	Yes
SF	0.019	0.035	16	Yes	Yes

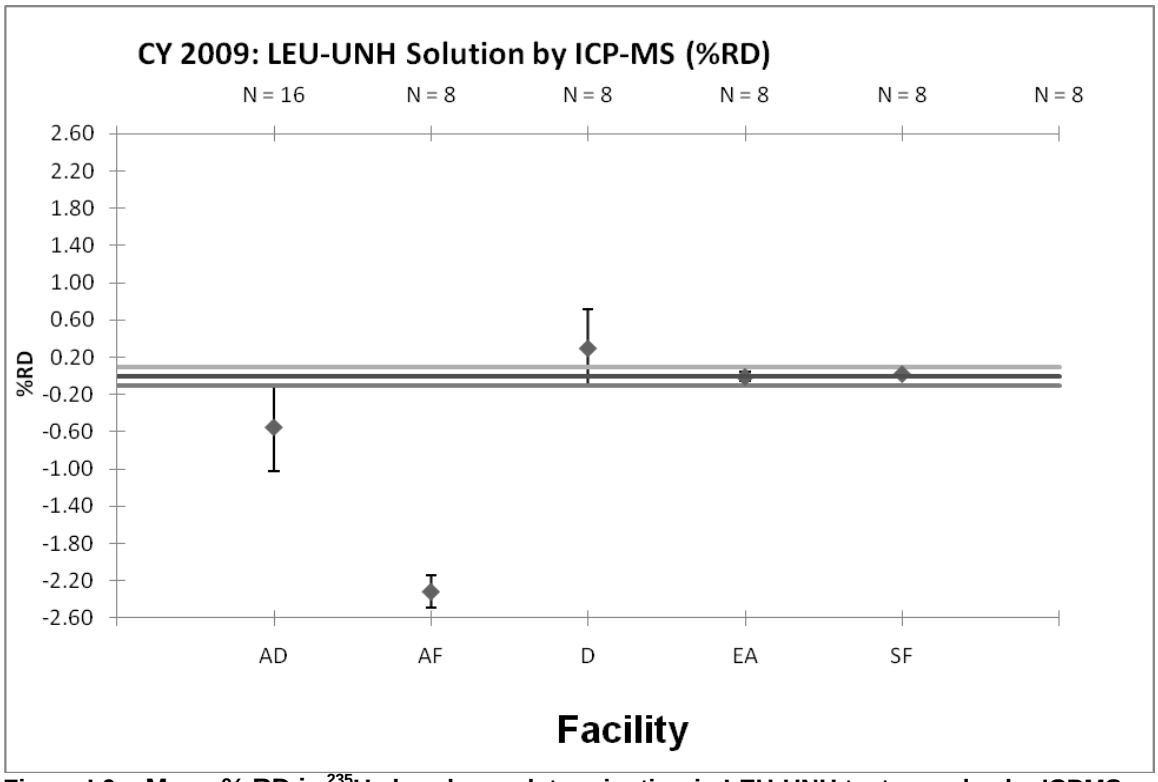


Figure I.6a. Mean % RD in ^{235}U abundance determination in LEU-UNH test samples by ICPMS. Laboratories EA and SF are in compliance to u(s); laboratories AD, AF, and D are outside the ITV.

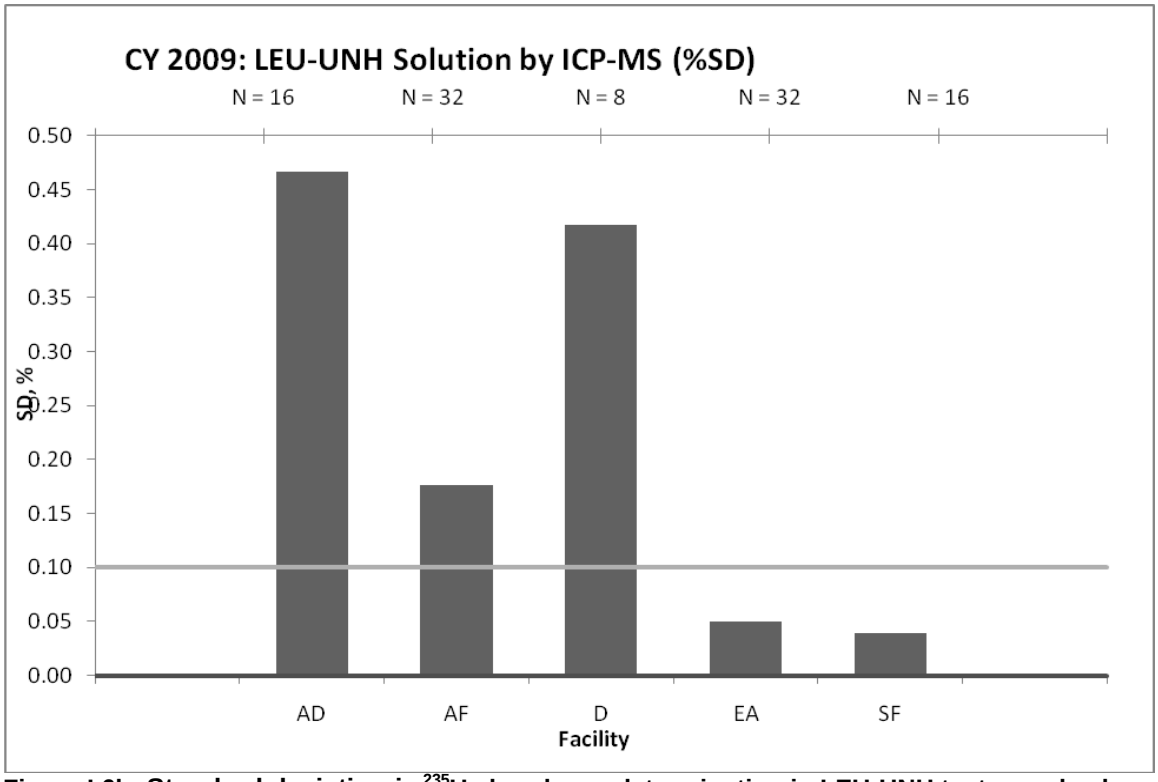


Figure I.6b. Standard deviation in ^{235}U abundance determination in LEU-UNH test samples by ICPMS. Laboratories EA and SF are in compliance to u(r); laboratories AD, AF, and D are outside the ITV .

I.7. LEU-UO_2 pellets by TIMS

The TIMS results from analyses of LEU-UO_2 (1 wt % < ^{235}U < 20 wt %) pellets are shown in Table I.7, and Figures I.7a and I.7b.

Table I.7. ^{235}U abundance in LEU-UO_2 pellet test samples by TIMS.

CY 2009: LEU-UO_2 Pellets by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
BC	0.020	0.055	12	Yes	Yes
J	0.008	0.009	8	Yes	Yes
T	0.068	0.020	40	Yes	Yes
TH	0.105	0.045	16	Yes	Yes
TO	0.029	0.045	16	Yes	Yes
TP	0.065	0.046	16	Yes	Yes
TR	-0.058	0.046	16	Yes	Yes

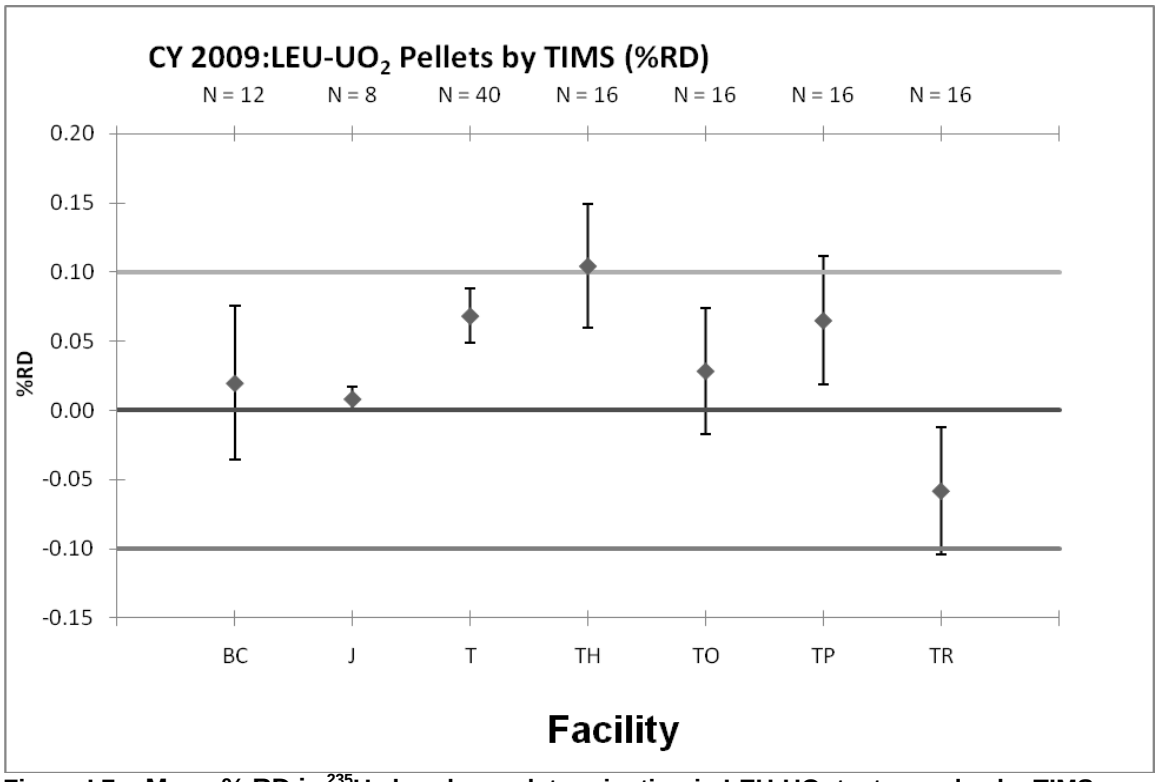


Figure I.7a. Mean % RD in ^{235}U abundance determination in LEU-UO_2 test samples by TIMS. All laboratories are in compliance to u(s).

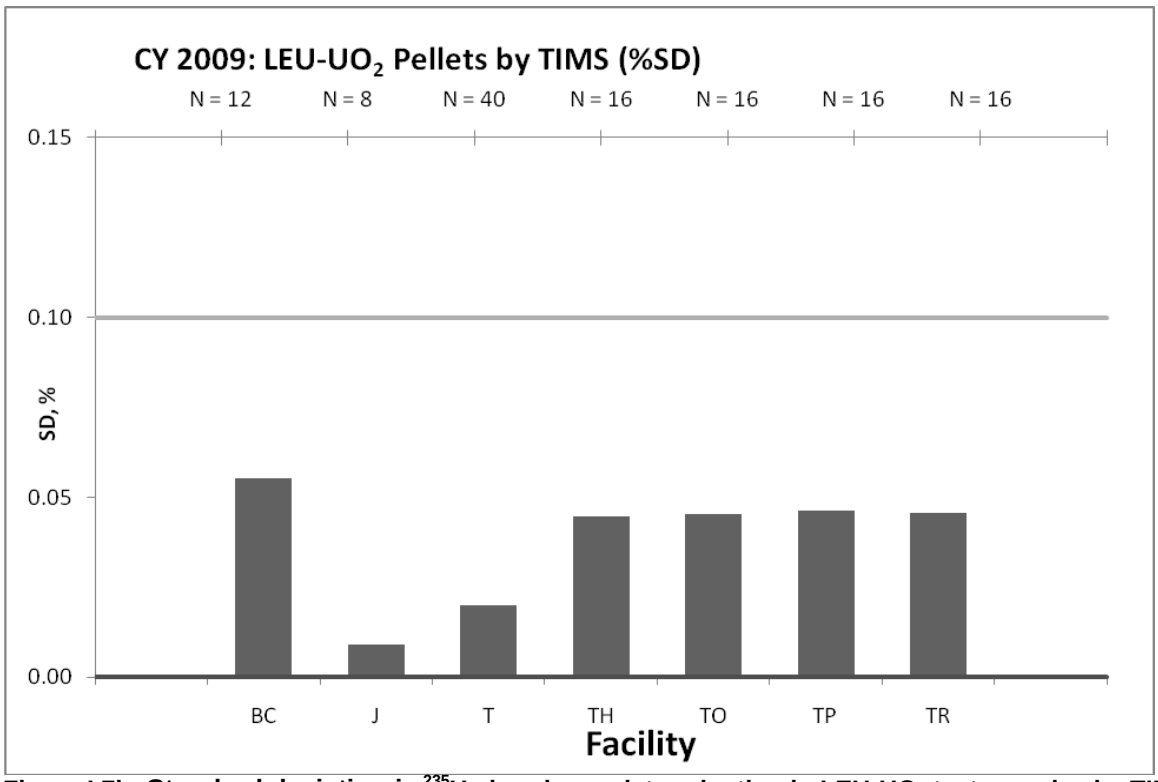


Figure I.7b. Standard deviation in ^{235}U abundance determination in LEU-UO_2 test samples by TIMS. All laboratories are in compliance to u(r).

I.8. LEU-UF_6 by TIMS

The TIMS results from analyses of LEU-UF_6 (1 wt % < ^{235}U < 20 wt %) are shown in Table I.8, and Figures I.8a and I.8b.

Table I.8. ^{235}U abundance in LEU-UF_6 test samples by TIMS.

CY 2009: LEU-UF_6 Solution by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.1	u(r) = 0.1
AA	0.062	0.027	8	Yes	Yes

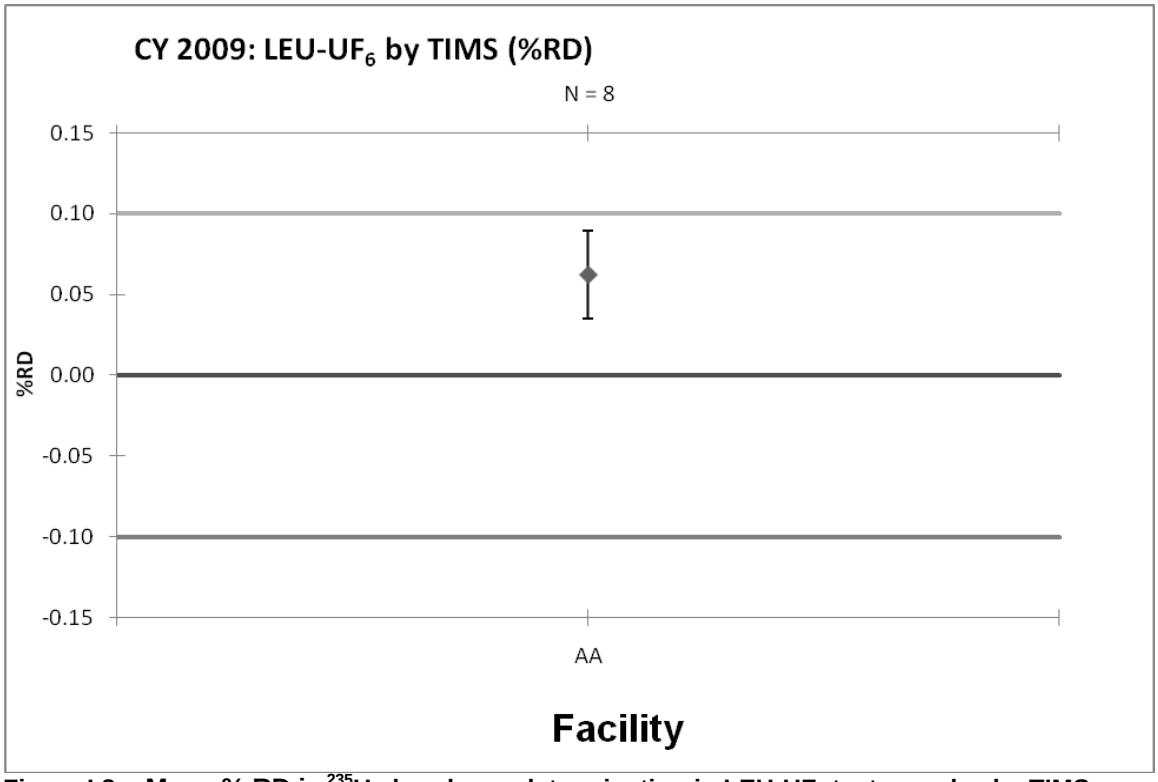


Figure I.8a. Mean % RD in ^{235}U abundance determination in LEU-UF_6 test samples by TIMS. Laboratory AA is in compliance to u(s).

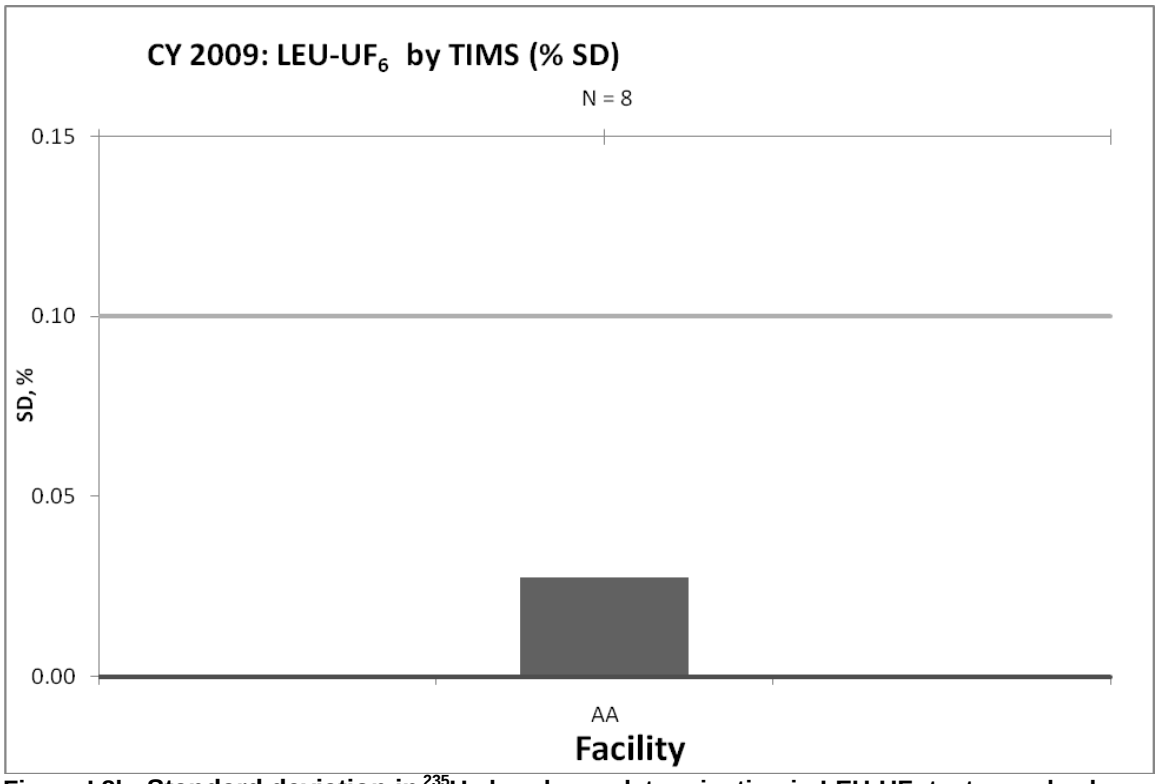


Figure I.8b. Standard deviation in ^{235}U abundance determination in LEU-UF_6 test samples by TIMS. Laboratory AA is in compliance to u(r).

I.9. LEU-UF_6 by ICPMS

The ICPMS results from analyses of LEU-UF_6 (1 wt % < ^{235}U < 20 wt %) are shown in Table I.9, and Figures I.9a and I.9b.

Laboratory SF: Need improvement in precision.

Table I.9. ^{235}U abundance in LEU-UF_6 test samples by ICPMS.

CY 2009: LEU-UF_6 Solution by ICPMS					
Lab Code	Mean % RD	SD	N	ITV Compliance*	
				u(s) = 0.1	u(r) = 0.1
EA	0.063	0.014	16	Yes	Yes
SF	0.008	0.118	15	Yes	No

*ITVs for ICPMS are assumed to be the same as TIMS.

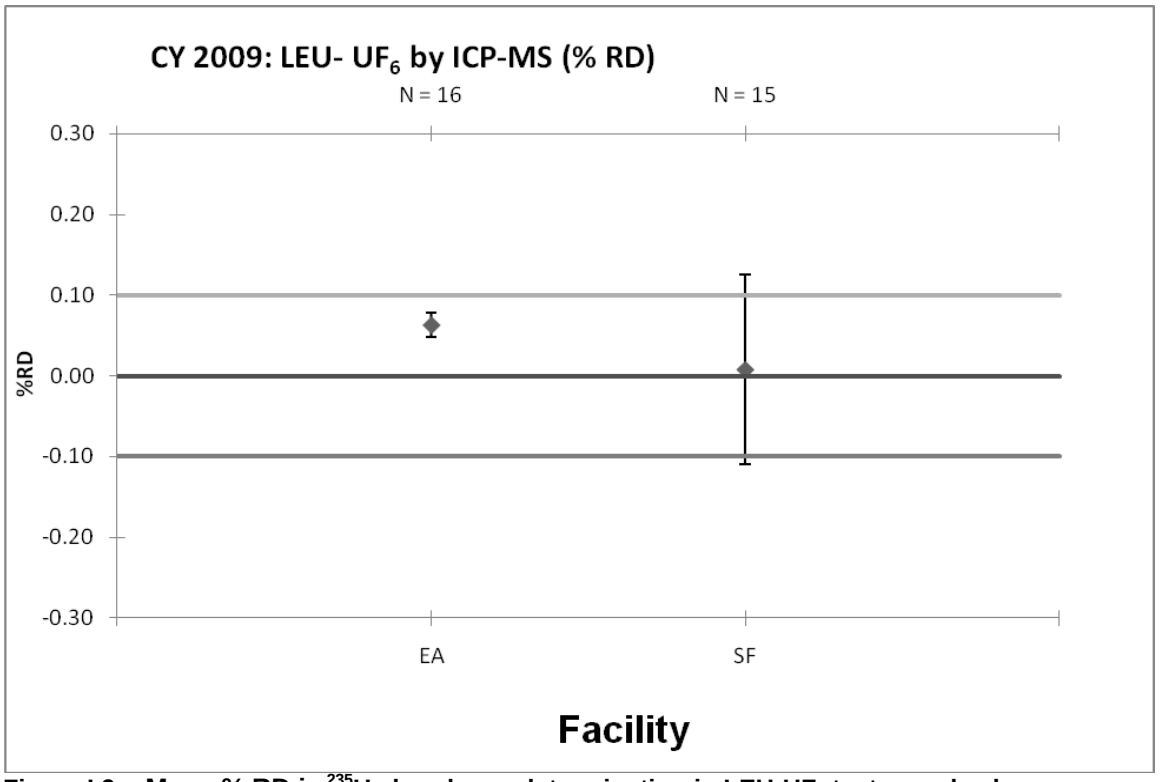


Figure I.9a. Mean % RD in ^{235}U abundance determination in LEU-UF_6 test samples by ICPMS. Laboratory EA is in compliance to u(s); laboratory SF is outside the ITV.

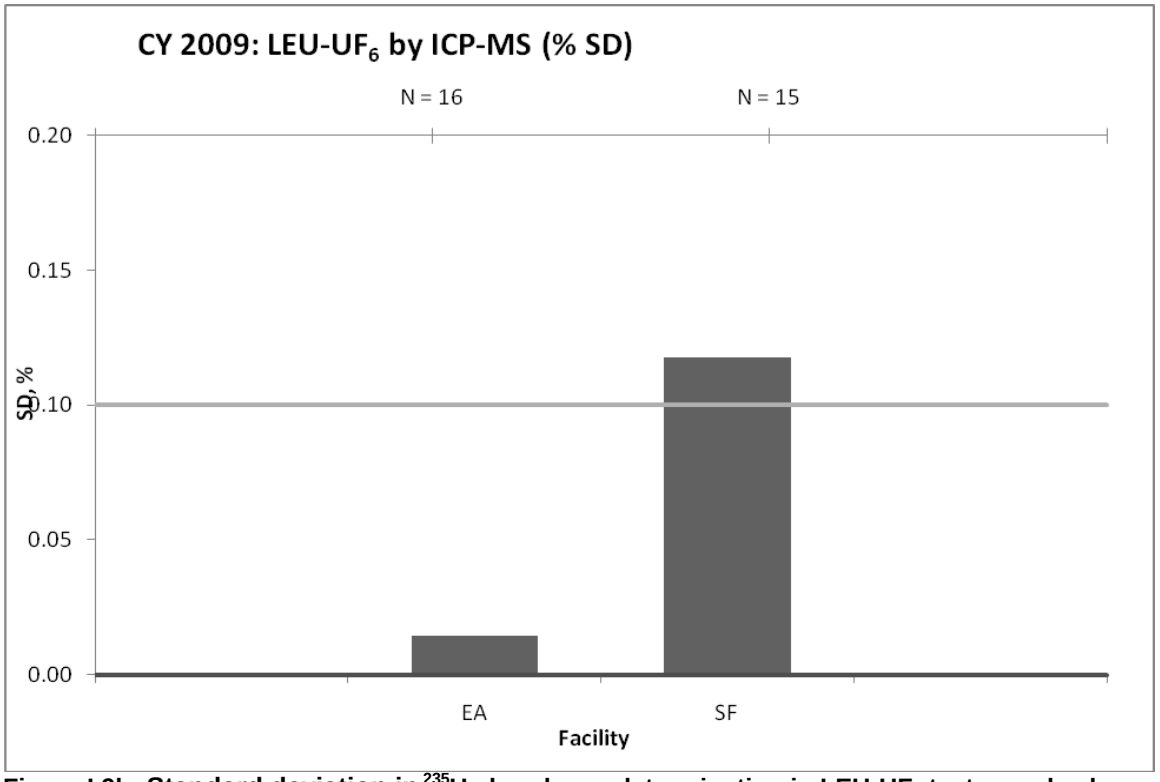


Figure I.9b. Standard deviation in ^{235}U abundance determination in LEU-UF_6 test samples by ICPMS. Laboratory EA is in compliance to u(r); laboratory SF is outside the ITV.

I.10. LEU-UF_6 by GSMS

The GSMS results from analyses of LEU-UF_6 (1 wt % < ^{235}U < 20 wt %) are shown in Table I.10, and Figures I.10a and I.10b.

Laboratory EB — Need improvement in accuracy.

Laboratory BC — Need improvement in precision.

Table I.10. ^{235}U abundance in LEU-UF_6 test samples by GSMS.

CY 2009: LEU-UF_6 Solution by GSMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.05	u(r) = 0.05
BC	0.039	0.094	16	Yes	No
EB	0.119	0.010	7	No	Yes
EC	0.000	0.013	18	Yes	Yes

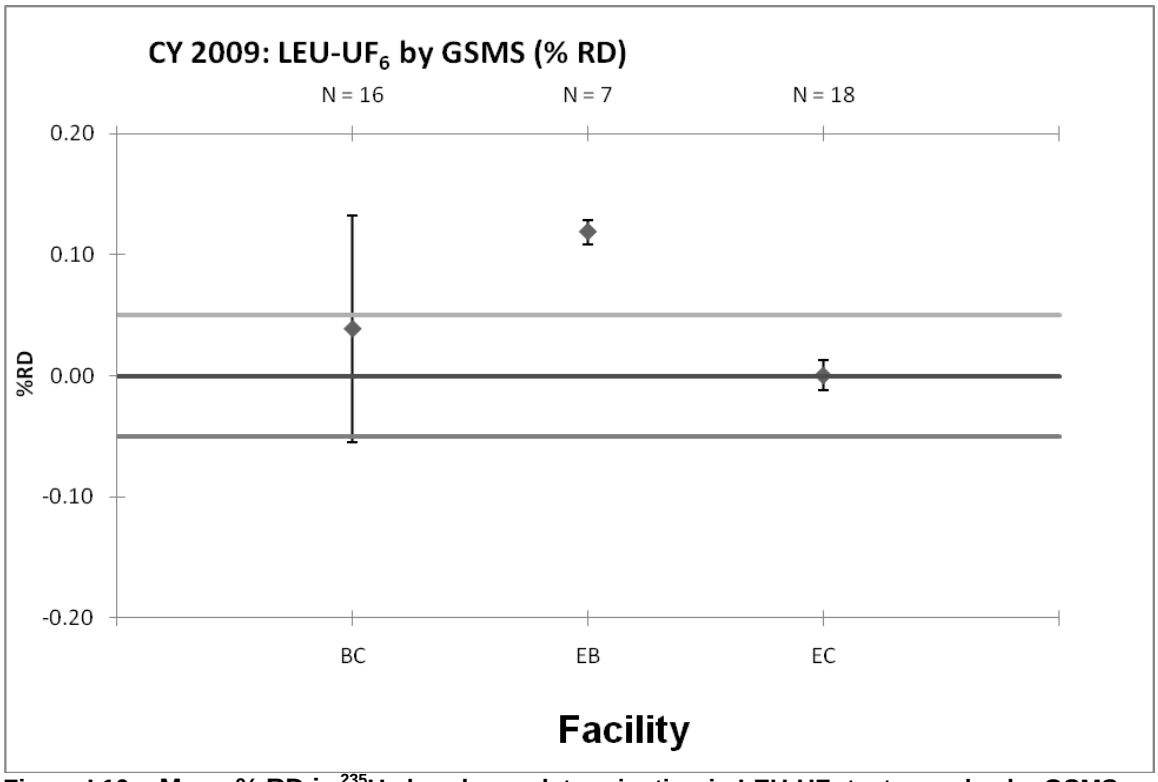


Figure I.10a. Mean % RD in ^{235}U abundance determination in LEU-UF_6 test samples by GSMS. Laboratories BC and EC are in compliance to u(s); laboratory EB is outside the ITV.

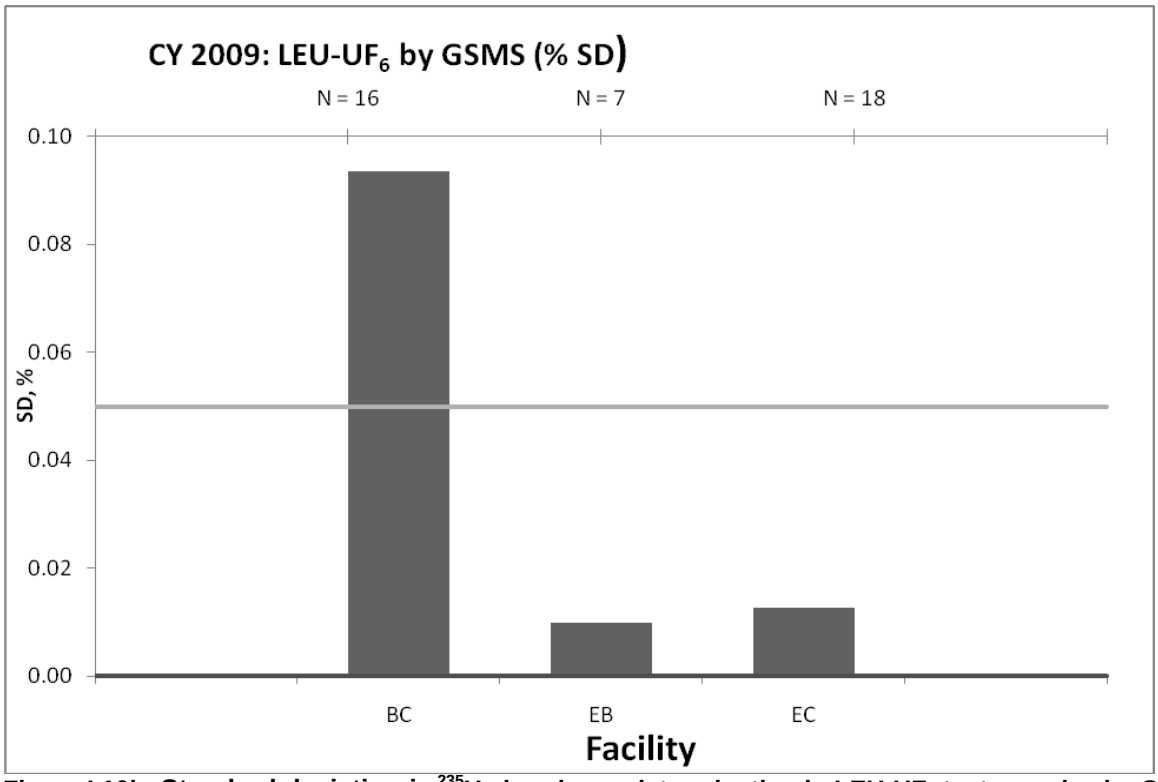


Figure I.10b. Standard deviation in ^{235}U abundance determination in LEU-UF_6 test samples by GSMS. Laboratories EB and EC are in compliance to u(r); laboratory BC is outside the ITV.

I.11. HEU-UNH by TIMS

The TIMS results from analyses of HEU-UNH (^{235}U > 20 wt %) solutions are shown in Table I.11, and Figures I.11a and I.11b.

Table I.11. ^{235}U abundance in HEU-UNH test samples by TIMS.

CY 2009: HEU-UNH Solution by TIMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.05	u(r) = 0.05
A	-0.026	0.038	16	Yes	Yes
F	0.001	0.004	8	Yes	Yes
SA	0.019	0.019	8	Yes	Yes
U	0.010	0.003	8	Yes	Yes

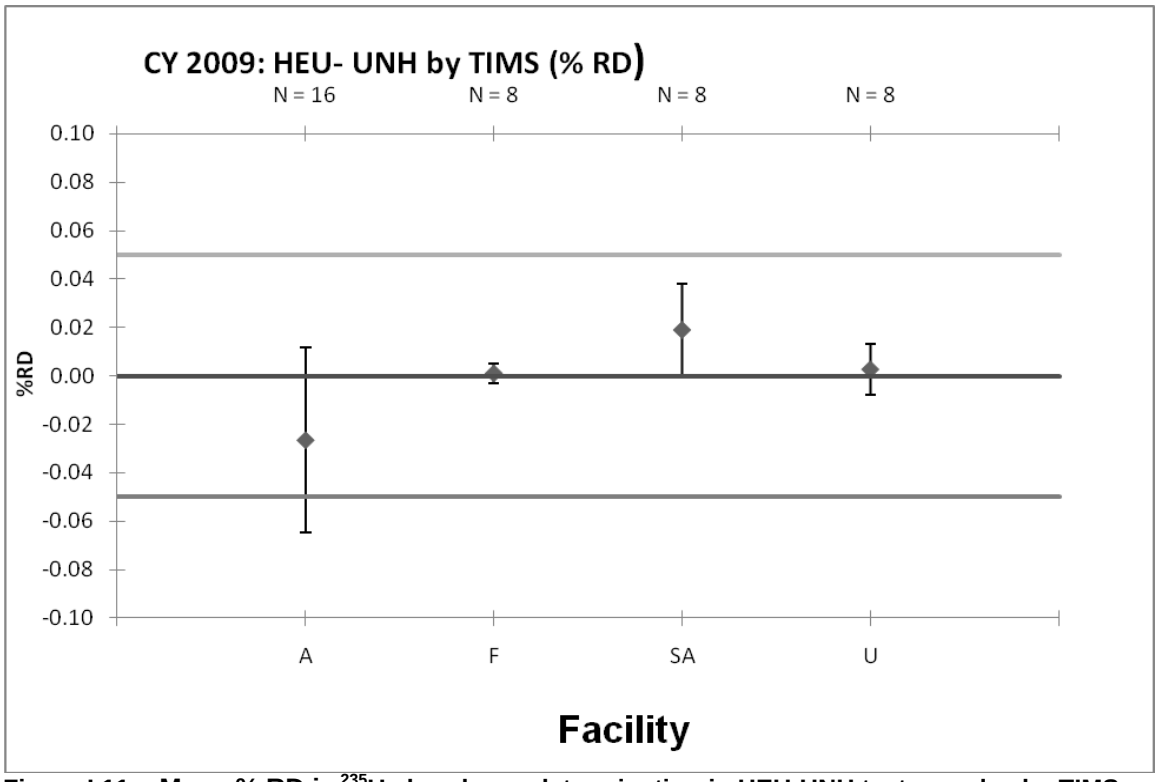


Figure I.11a. Mean % RD in ^{235}U abundance determination in HEU-UNH test samples by TIMS. All laboratories are in compliance to u(s).

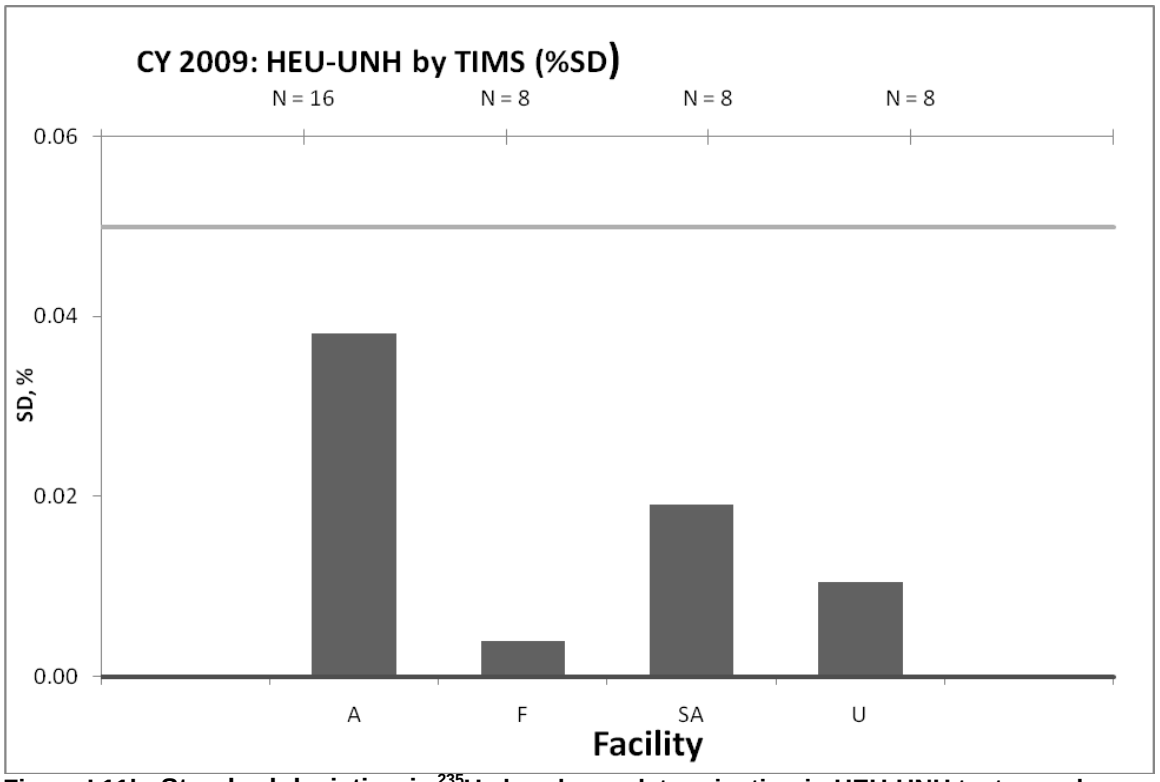


Figure I.11b. Standard deviation in ^{235}U abundance determination in HEU-UNH test samples by TIMS. All laboratories are in compliance to u(r).

I.12. HEU-UNH by ICPMS

The ICPMS results from analyses of HEU-UNH (^{235}U > 20 wt %) solutions are shown in Table I.12, and Figures I.12a and I.12b.

Laboratory D; Need improvement in both accuracy and precision.

Table I.12. ^{235}U abundance in HEU-UNH test samples by ICPMS.

CY 2009: HEU-UNH Solution by ICPMS					
Lab Code	Mean % RD	SD	N	ITV Compliance	
				u(s) = 0.05	u(r) = 0.05
D	0.632	0.467	16	No	No

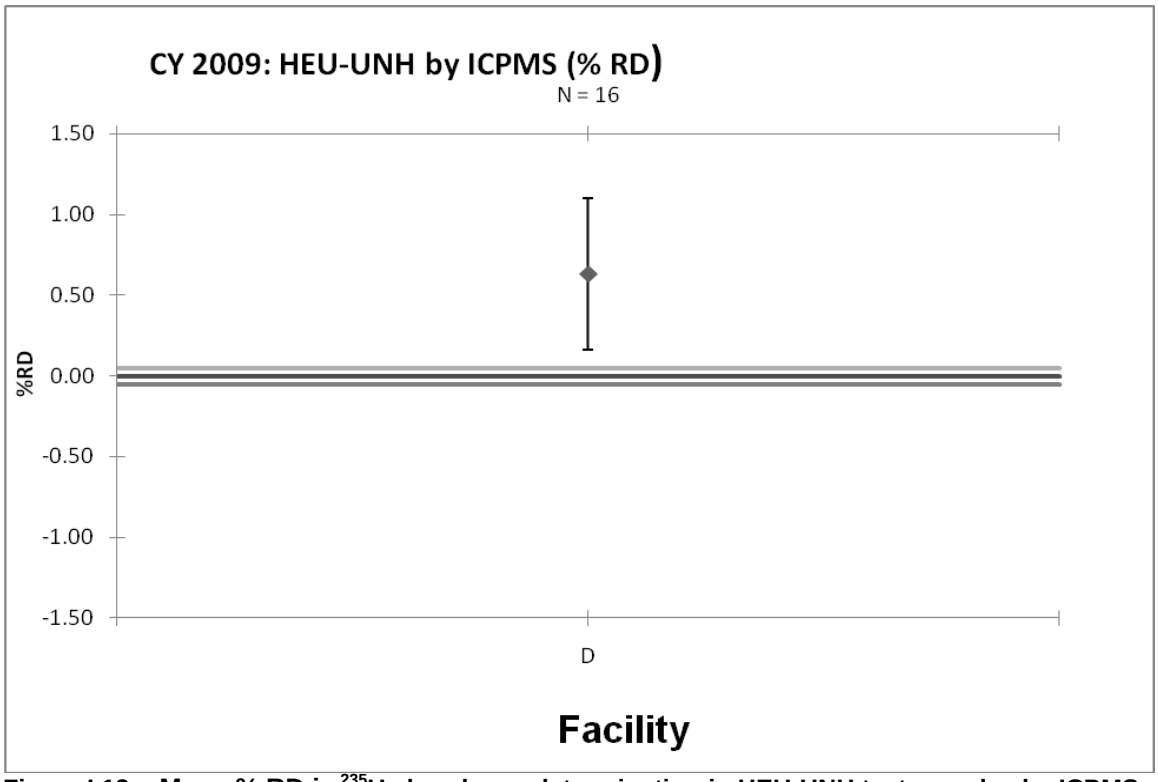


Figure I.12a. Mean % RD in ^{235}U abundance determination in HEU-UNH test samples by ICPMS. Laboratory D is outside the ITV for u(s).

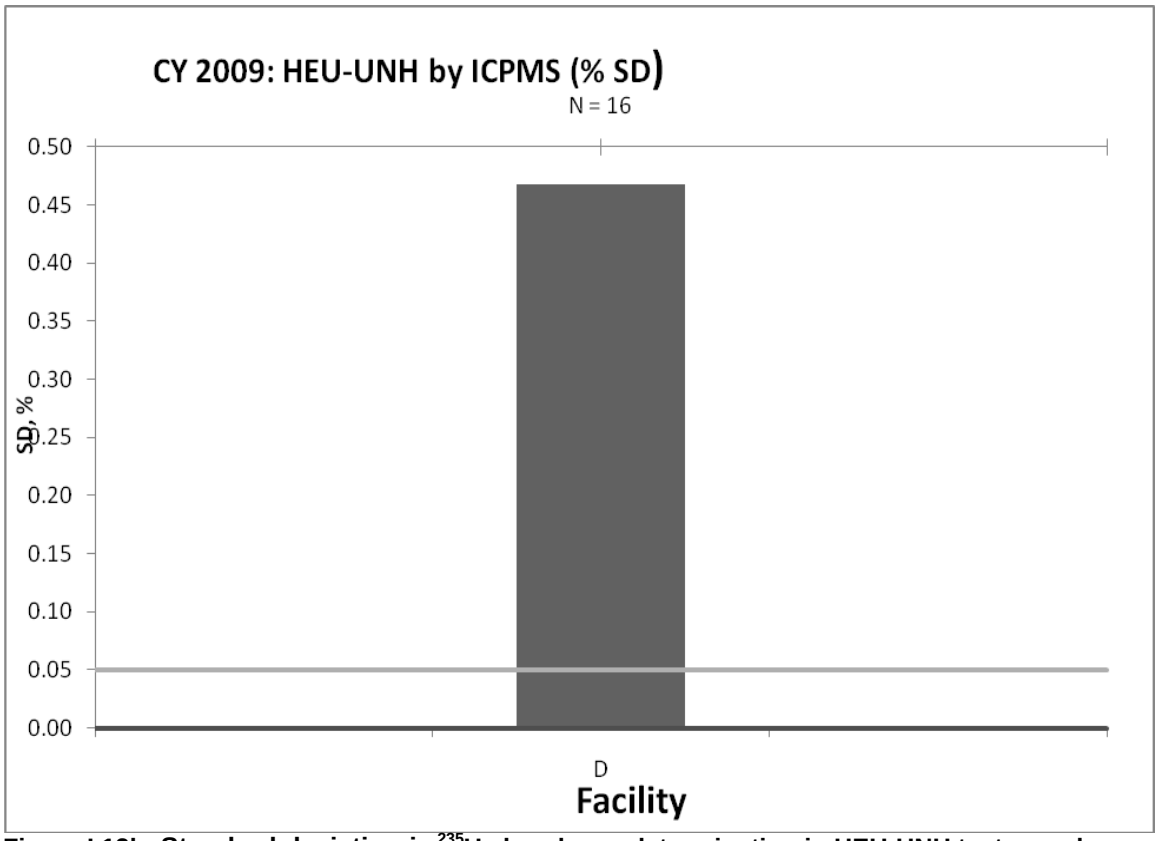


Figure I.12b. Standard deviation in ^{235}U abundance determination in HEU-UNH test samples by ICPMS. Laboratory D is outside the ITV for u(r).

J. CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The NBL-SME program has been providing useful service to nuclear safeguard laboratories in the DOE complex through monitoring their internal control practices so that fissile nuclide measurements made in these facilities remain accurate and precise. This service is now offered to several laboratories outside the DOE complex also; both government and commercial facilities in the U.S. and abroad are active participants in the program. In addition to performance testing, NBL has been providing assistance towards improving measurement quality and conducting hand-on training sessions. These efforts by NBL, taken together, are making significant contributions to the advancement of Material Control and Accountability and Nuclear Non-proliferation programs.

SME programmatic activities in CY 2011 and beyond

Programmatic activities that are to be tackled in CY 2011 and beyond are listed below. Priorities are to be set by NBL management and the newly appointed program coordinator.

New test samples: NBL made six new sets of test samples in CY 2010, three for elemental uranium analysis and three for uranium isotopic composition analysis. These test samples are to be characterized prior to shipping the samples to the program participants.

New test samples for uranium as well as plutonium measurements are needed for distribution in CY 2012 and beyond.

Shipping of CY 2011 test samples: CY 2011 test samples are to be shipped to participants in the first quarter of the year.

Working reference material for impurity analyses: Several facilities would like to acquire working reference material of impurity elements in uranium matrix and participate in an evaluation program for impurity elements analysis.

CY 2011 Measurement Evaluation Program Annual Meeting: Conduct the CY 2011 Measurement Evaluation program annual meeting on July 2011. The meeting will be held on July 16, 2011 in Palm Desert, California, a day before the start of the INMM 52nd Annual Meeting.

CY 2010 Annual report: Release the CY 2010 annual report at the time of holding the CY 2011 ME program annual meeting. The evaluation of 2010 samples results will constitute the main body of this report.

ABACC collaboration: (a) Qualify Cristallini method of UF_6 sampling, and (b) organize TIMS training session at NBL for ABACC network laboratory personnel.

CNEN collaboration: Assist LASAL in establishing the Brazilian ME program.

ARN/CNEA collaboration: Work with NNSA to begin this new collaboration with ARN/CNEA laboratories in Argentina.

NMCC collaboration: Provide support to NMCC in conducting the round robin exercise for safeguard laboratories in Japan.

Program expansion: Offer the program to (a) domestic laboratories (DOE, NRC, and enrichment facilities) not currently participating in the program, and (b) International laboratories (in Canada, China, Kazakhstan, Russia, and South Korea).

Accreditation of SME program: SME program functions are to be included as a part of the general laboratory accreditation.