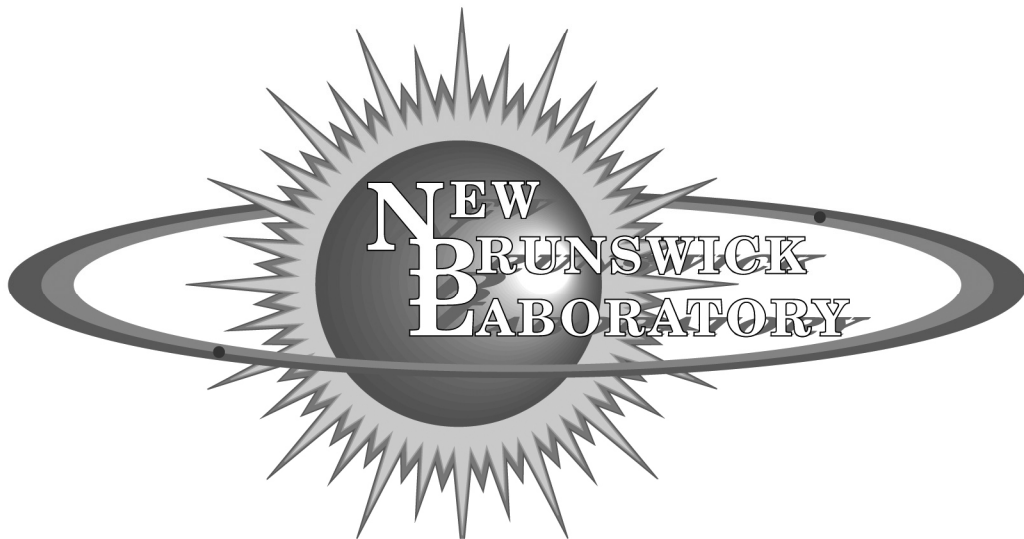


# SAFEGUARDS MEASUREMENT EVALUATION PROGRAM

## URANIUM AND PLUTONIUM SAMPLES EXCHANGE REPORT



**FISCAL YEAR 2004**

**B. Srinivasan, Willard C. Losinger,  
and Jon Neuhoff**





**U.S. DEPARTMENT OF ENERGY**

**SAFEGUARDS MEASUREMENT  
EVALUATION PROGRAM**

**URANIUM AND PLUTONIUM  
SAMPLES EXCHANGE REPORT  
FY 2004**

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## **NBL: HISTORY AND MISSION**

The New Brunswick Laboratory is owned and operated by the United States Department of Energy through the offices of Security and Safety Performance Assurance (SP-1) and Materials Control and Accountability (SP-70). 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 measurement services to domestic and international customers. 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 Materials Control and Accountability (SP-70) in the Office of Security and Safety Performance Assurance (SP-1). The authors of this report thank Nancy Hui of NBL for verifying database entries; Usha Narayanan of NBL for review and comments on an earlier draft of this report; and Michael Soriano of NBL for help in correcting coding errors in the database software. A part of the work in this report was performed under the leadership of Jay Thompson (SP-70) while he was an employee at NBL.



## **ABSTRACT**

The New Brunswick Laboratory has been tasked by the United States Department of Energy office of Materials Control and Accountability (SP-70) in the office of Security and Safety Performance Assurance (SP-1) to evaluate the quality of measurement techniques in nuclear materials accounting practices at the Department of Energy facilities. Both destructive and non-destructive methods of analyses come under this purview. The destructive methods are evaluated in the Safeguards Measurement Evaluation program. The non-destructive methods in the CALEX program. This report describes the activities in the FY 2004 Safeguards Measurement Evaluation program; a separate report will be issued on the CALEX program.

Several Department of Energy facilities participated in the FY 2004 Safeguards Measurement Evaluation program partly to satisfy a Department of Energy requirement on independent verification of internal analytical control of their measurements. A Nuclear Cycle Development Laboratory in Japan also participated, on a voluntary and cost-recovery basis.

At the beginning of the year, the New Brunswick Laboratory sent samples of uranium and plutonium bearing materials to the participating laboratories for elemental and isotopic abundance analyses. The participants analyzed the samples by methods used for accountability measurements at their facilities. The results of their analyses were evaluated by the New Brunswick Laboratory for bias and precision using statistical techniques. Performance evaluation reports were sent to the participants indicating adequacy or need for improvement.



## **A. INTRODUCTION**

The New Brunswick Laboratory (NBL) is a nuclear material measurement laboratory of the U.S. Department of Energy (DOE). NBL reports to the DOE office of Security and Safety Performance Assurance (SP-1) through the office of Materials Control and Accountability (SP-70). NBL provides the technical expertise to the department for the operation of the measurement evaluation program and for the preparation of certified reference materials.

In the measurement evaluation program, NBL evaluates the capabilities of DOE contractor-operated laboratories in nuclear materials accounting measurements. The program has two parts; the safeguards measurement evaluation (SME) program for destructive measurements of uranium and plutonium bearing materials (e.g. titration, isotope dilution mass spectrometry), and the calorimetric exchange (CALEX) program for non-destructive measurements. At present, in the latter program, calorimetric/gamma spectrometric measurements of plutonium oxide only are evaluated. In the future, evaluation of both uranium and plutonium measurements by neutron coincidence counting methods will be included.

## **B. SAFEGUARDS MEASUREMENT EVALUATION PROGRAM**

Materials control and accountability measurements form an essential part of the work in safeguarding nuclear materials. Destructive and non-destructive methods are used in accounting of nuclear materials in processing, in storage and in transit. These methods must be capable of providing results within acceptable limits of accuracy and precision. Large errors will compromise the ability to detect material loss should they occur either in processing or by theft or by diversion.

The SME program evaluates elemental and isotopic abundance measurement results from analyses of uranium and plutonium bearing materials. Well-characterized samples are sent to the participating laboratories for analyses by techniques that are routinely used in accountability measurements. The results are evaluated by NBL for accuracy and precision. Results falling within the accuracy and precision target values indicate satisfactory performance; those falling outside indicate the need for improvement. Note that international target values (ITVs) are used where available; if ITVs are not given for a particular method/material, then DOE target values are used instead (e.g., uranium by x-ray fluorescence).

The facilities analyze the SME program test samples using well documented experimental methods. Despite utmost care, occasionally, the laboratories fail to meet the target values. The SME program detects such instances of failures and informs the laboratories to take corrective action.

## **C. FY 2004 SAFEGUARDS MEASUREMENT EVALUATION PROGRAM**

NBL sent well characterized samples of uranium and plutonium bearing materials to the participants at the beginning of the year. The participants analyzed the samples several times during the year, usually at quarterly intervals, for elemental concentrations and isotopic abundance. The results of analyses were evaluated by NBL by statistical techniques. The participants received performance evaluation reports and statements regarding accuracy and precision achieved in the analyses and whether they were within the target values. In addition, the results were discussed at the measurement evaluation program annual meeting held in July 2005 in Phoenix, Arizona, with attendance of personnel from NBL, DOE-HQ, and international laboratories (ABACC, IAEA and Japan). The "minutes of the meeting" containing copies of slides used in the talks was prepared and sent to the participants.

Every year, the measurement evaluation program annual meeting is held in the same venue as the Institute of Nuclear Materials Management (INMM) annual meeting and at about the same time - usually the day before the start of the INMM meeting - to maximize participation.

### **C.1. FY 2004 SME Program Participants**

Several DOE laboratories participated in the FY 2004 SME program. Their participation is mandated by the requirement 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."* A laboratory in Japan participated on a voluntary and cost recovery basis, with prior approval from DOE.

The lists of participants in uranium samples analyses program are shown in Table 1, and plutonium samples analyses in Table 2.

**Table 1. FY 2004 SME program: Participants in uranium samples analyses**

ARGONNE NATIONAL LABORATORY-WEST (DOE contractor laboratory)
LOS ALAMOS NATIONAL LABORATORY (DOE contractor laboratory)
NEW BRUNSWICK LABORATORY (DOE laboratory)
SAVANNAH RIVER SITE (DOE contractor laboratory)
TOKAI SAFEGUARDS ANALYTICAL LABORATORY (Japan)
Y-12 NATIONAL SECURITY COMPLEX (DOE contractor laboratory)

**Table 2. FY 2004 program: Participants in plutonium samples analyses**

ARGONNE NATIONAL LABORATORY-WEST (DOE contractor laboratory)
LOS ALAMOS NATIONAL LABORATORY (DOE contractor laboratory)
NEW BRUNSWICK LABORATORY (DOE laboratory)
TOKAI SAFEGUARDS ANALYTICAL LABORATORY (Japan)

Nuclear Regulatory Commission (NRC) licensees were regular participants in the program prior to 2001. None of the licensees participated in FY 2004 possibly because of financial constraints. NBL would like NRC licensees to re-join the program for the following reason: nuclear materials transfers occur frequently between NRC and DOE facilities; sometimes shipper-receiver differences occur in these transfers. If both DOE and NRC facilities participate in a common evaluation program, such as the SME program, then shipper-receiver differences can be speedily resolved. NBL would like to increase the number of international participants also; it may prove beneficial to the conduct of the nuclear safeguards program on a global scale.

## **C.2. Materials and Measurement Methods**

The materials used and the measurement methods evaluated in the FY 2004 program are shown in Tables 3 and 4; uranium and plutonium assay in Table 3, and isotopic abundance measurements in Table 4. The participants are identified by code letters only to provide confidentiality.

**Table 3. FY 2004 SME program: Materials and methods used to evaluate uranium and plutonium assay. The participant laboratories are identified by code letters only. Numbers next to codes refer to number of times the laboratory participated in the program. For example, B3 means laboratory B participated in the program three times during the year.**

Method	UNH Solutions	UO <sub>2</sub> Pellets	UO <sub>3</sub> Powder	Pu Sulfate
Dichromate Titration (Davies-Gray)	B3 F2	F1 T2	F1	
Ceric Titration	G3			
High Precision Titration		F1		
IDMS	A4 B2 G1 J2		A4	F2 G1
XRF	A4		A8	

Notes: UNH, uranyl nitrate solutions. UO<sub>2</sub>, uranium dioxide pellets. UO<sub>3</sub>, uranium trioxide powder. Pu sulfate, dried material.

**Table 4. FY 2004 SME program: Materials and methods used to evaluate uranium and plutonium isotopic abundance measurements. The participant laboratories are identified by code letters only. Numbers next to codes refer to number of times the laboratory participated in the program. For example, A1 means laboratory A participated in the program once during the year.**

Method	LEU	HEU	Pu sulfate
TIMS	A1 B1 F2 T2	A3 B3 F2 G2 J2	F2 G1 J1 T2

Notes. LEU is low-enriched uranium containing <20 wt % <sup>235</sup>U. HEU is high-enriched uranium containing ≥20 wt % <sup>235</sup>U. Pu sulfate: dried material of either high burn-up or low burn-up composition.

### **C.3. Test Materials Characterization, Shipping and Analyses Schedule**

The FY 2004 SME program test materials were derived from certified reference materials (CRMs) or working reference materials (WRMs) or tailor-made materials. These materials were characterized at NBL for elemental concentrations and/or isotopic abundance.

The characterization measurements were performed according to statistical plans with full consideration to quality assurance and traceability of the measurements. A sufficiently large number of test samples were prepared from the characterized materials, and a sufficient number of



these were shipped to participants at the beginning of the year. Laboratories participating on a quarterly basis received more samples than those participating on semi-annual or annual basis.

Quarterly participants received eight samples of each type of test material. They analyzed two of the eight samples in duplicate, and on two different days in the same quarter. This analysis schedule generated at least eight results per quarter sufficient for a meaningful statistical evaluation of the results.

#### **C.4. SME Program Database**

The results submitted by the participants were entered into a FoxPro® database. The database program has been in continuous use since 1995. It was modified in 1999 to become Y2K compliant. The results were manually entered and manually verified for ensuring correctness. The entry and verification tasks are labor-intensive. Direct electronic transfer by participants is preferred – labor saving as well as it will be error-free. NBL intends to modify the database program to enable it to accept electronic transfer of data.

The results were evaluated using the FoxPro® software programs written several years ago with improvements continuously made to the programs. However, neither the original programs nor the improved versions were subjected to quality control tests. The need for quality control tests for these indigenously developed programs became evident while preparing the graphs and tables for the FY 2004 report. The tables and graphs (in the first draft of this report) contained a number of errors traceable to coding errors in the program. NBL statisticians, working closely with RSIS staff (DOE-CH contractor providing computer help), expended a lot of effort to eliminate these errors. It is essential that the programs be tested (for providing quality assurance) using expert help.

#### **C.5. Statistical Evaluation of Measurement Results**

The measurement results were evaluated using statistical techniques. First, the percent relative difference (% RD) of each experimental result was calculated with respect to the corresponding reference values, the latter obtained from characterization measurements. The % RD is defined as follows:

$$\% \text{ RD} = 100 \times \{(\text{observed value} - \text{reference value})/\text{reference value}\}.$$

Next, each set of % RDs was examined for outliers using a number of statistical tests. A result might be judged to be an outlying value if at least two of the tests found it as an outlier at  $\geq 99\%$  significance level. The data set, sans outliers, was then tested to identify significant sources of variations attributable to analyses protocols - day-to-day and/or analyst-to-analyst – using standard one-factor analysis of variance (ANOVA). If the ANOVA results indicated no significant variation, then the standard uncertainty in the mean %RD was calculated from the standard deviation of all results. It was the simple standard deviation of the results divided by the square root of  $n$ , where  $n$  was the number of measurements. The coverage factor used was the 95% Student's "t" factor with  $n-1$  degrees of freedom. For example, in a set of 8 results showing no day-to-day/analyst-to-analyst variation, the number of degrees of freedom is 7, and the coverage factor is 2.36.

If the ANOVA results indicated significant ( $\geq 95\%$ ) day-to-day and/or analyst-to-analyst variation, then the standard uncertainty in the mean % RD was estimated from the square root of the mean square for the "model" quantity obtained from ANOVA results. In this case, the coverage factor was the 95% Student's "t" factor with  $(k-1)$  degrees of freedom, where  $k$  was the number of days or the number of analysts. For example, in a set of 8 results obtained over a period of 2 days and showing day-to-day variation, the degree of freedom is 1 and the coverage factor is 12.71.

The uncertainties shown in the statistical reports were the 95% confidence limit (C.L.) of means. In the figures accompanying the reports, the 95% confidence interval (C.I.) of the mean was constructed from the C.L. (Note that 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 was considered to be bias-free if ( $\% \text{ RD} \pm \text{C.L. at } 95\%$ ) included zero. Otherwise, measurement bias was indicated. The standard deviation ( $\pm 1\sigma$ ) of the mean % RD was a measure of precision obtained in the analyses.

## **C.6. Examples of Statistical Evaluation Reports**

Two examples of the statistical analysis reports are shown in Figs.1 and 2, the former showing uranium assay results from Davies-Gray titration, and the latter from isotope dilution mass spectrometry (IDMS) measurements. There are 8 results in each set obtained from analyses of two samples in duplicate on two different days.

There are no outliers in Fig.1. There is no evidence for significant day-to-day variation. The statistical significance is 44.3%; variation will be considered significant only if it exceeds 95%. The mean % RD value is -0.154 and the uncertainty (95% C.L.) is 0.070. The uncertainty is calculated using a coverage factor of 2.36 corresponding to 7 degrees of freedom. The mean value extended by the confidence limit does not include zero, thereby indicating negative bias in the measurements. The standard deviation of the results is 0.083.

There are no outliers in Fig. 2 results also. However, there is evidence for significant day-to-day variation (statistical significance of 96.6%). The mean % RD value is 0.015 and the uncertainty at 95% C.L. is 1.319. The uncertainty is calculated using a coverage factor of 12.7 corresponding to 1 degree of freedom. The mean value extended by the confidence limit overlaps with zero indicating no bias. But, this conclusion is not meaningful since the uncertainty is very large. The standard deviation of the results is 0.149.

The bias and precision international target values (ITVs) are shown at the bottom of the statistical reports. In Fig.1, the mean % RD of -0.154 is beyond the bias ITV of 0.1%; the precision of 0.083 is within the precision ITV of 0.1%. The measurement suffers from negative bias. In Fig.2, the mean % RD of 0.015 is within the ITV of 0.1%, and the precision of 0.149 is also within the ITV of 0.15%. However, no conclusion is possible regarding bias because of the large uncertainty in % RD.

The bias and precision achieved in the measurements - negative bias in Fig.1 and the day-to-day variation in Fig.2 - are easily seen in the visual representations accompanying the statistical reports.

Statistical reports such as those shown in Fig.1 and Fig.2, are generated for each set of results submitted by the laboratories. The reports (including graphs) are sent to the laboratories along with a cover letter stating the conclusions of performance evaluation. Copies of the report and the letter are sent to the respective DOE site offices supervising the work done in the laboratories. The site offices are responsible for initiating action for improvements if bias and/or precision in the measurements failed to meet the target values. NBL can provide assistance in bringing improvement through critical review of measurement procedures and training in experimental techniques.

Figure 1

**SAMPLE DATA EVALUATION REPORT**

**No statistically significant difference due to analysis day**

U.S. Department of Energy  
New Brunswick Laboratory  
Safeguards Measurement Evaluation Program  
Data Evaluation Report

**Day to Day ANOVA analysis**

Report for Laboratory: XX

U02 Pellet – U Concentration

Davies-Gray Titration

Date of Report: July 30, 2003

<b>Sample Number</b>	<b>Aliquant Number</b>	<b>Analysis Date</b>	<b>Reported %U</b>	<b>% Relative Difference</b>	<b>Analyst Code</b>
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

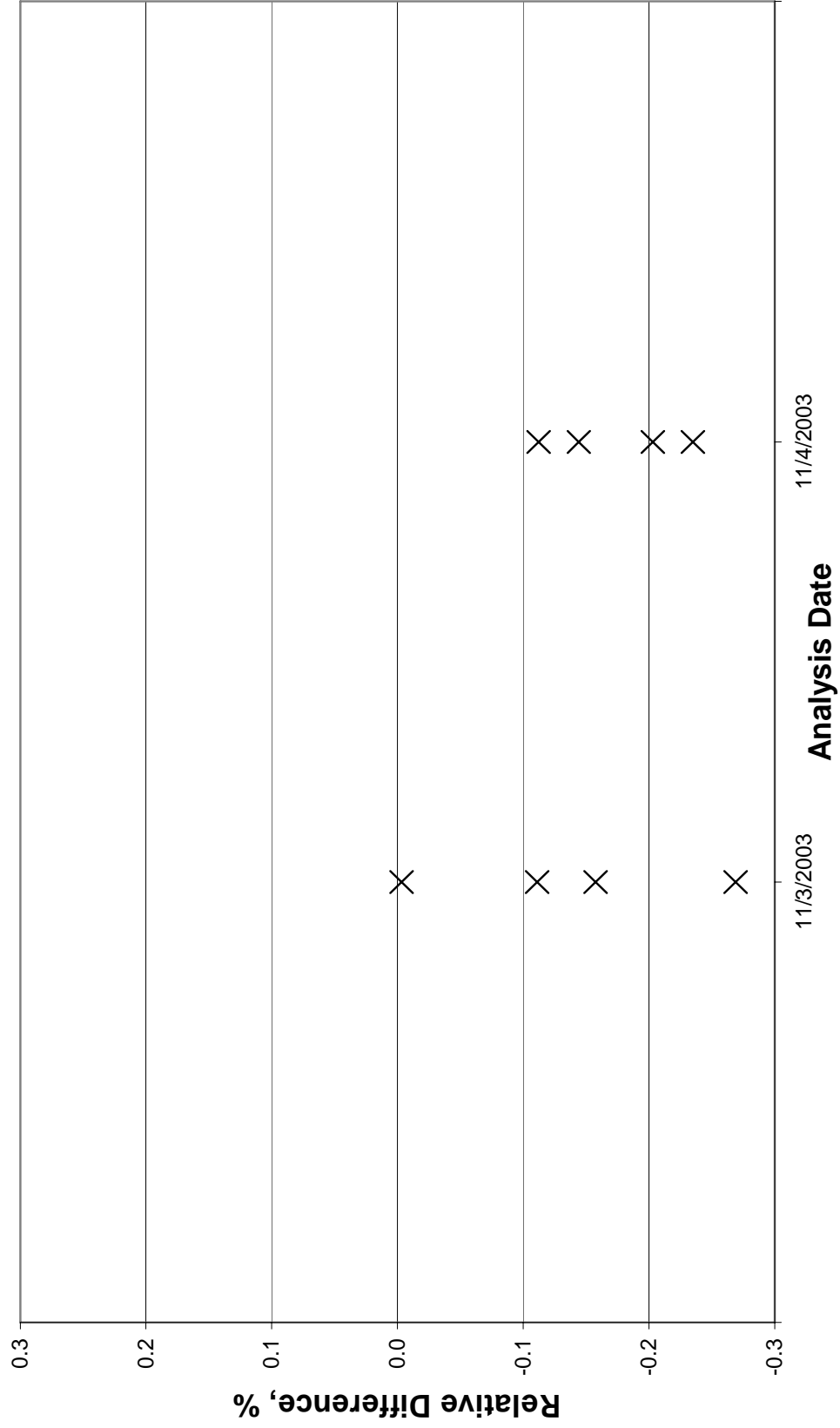
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<b>Number of Results Analyzed</b>	8
<b>Mean % Difference</b>	-0.154
<b>Mean Absolute % Difference</b>	0.154
<b>95% C.L. of Mean (df = 7)</b>	0.070
<b>Standard Deviation</b>	0.083
<b>Between-Day Standard Deviation (df = 1)</b>	0.054
<b>Within-Day Standard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

International target value for bias in Davies-Gray Titration is 0.1%.

International target value for precision in Davies-Gray Titration is 0.1%.

Laboratory XX  
UO2 Pellet -- Davies-Gray Titration



**Figure 2**

**SAMPLE DATA EVALUATION REPORT**

**Statistically significant difference due to analysis day**

U.S. Department of Energy  
New Brunswick Laboratory  
Safeguards Measurement Evaluation Program  
Data Evaluation Report

**Day to Day ANOVA analysis**

Report for Laboratory: XX

UNH Solution – U Concentration

IDMS

Date of Report: May 8, 2003

<b>Sample Number</b>	<b>Aliquant Number</b>	<b>Analysis Date</b>	<b>Reported %U</b>	<b>% Relative Difference</b>	<b>Analyst Code</b>
94NU0021-023	1	11/03/03	1.0000	-0.0590	XXX
94NU0021-023	2	11/03/03	1.0003	-0.0290	XXX
94NU0023-079	1	11/03/03	0.9991	-0.0080	XXX
94NU0023-079	2	11/03/03	0.9996	-0.2582	XXX
94NU0021-023	3	11/04/03	1.0022	0.1609	XXX
94NU0021-023	4	11/04/03	1.0004	-0.0190	XXX
94NU0023-079	3	11/04/03	1.0004	0.1221	XXX
94NU0023-079	4	11/04/03	1.0013	0.2122	XXX

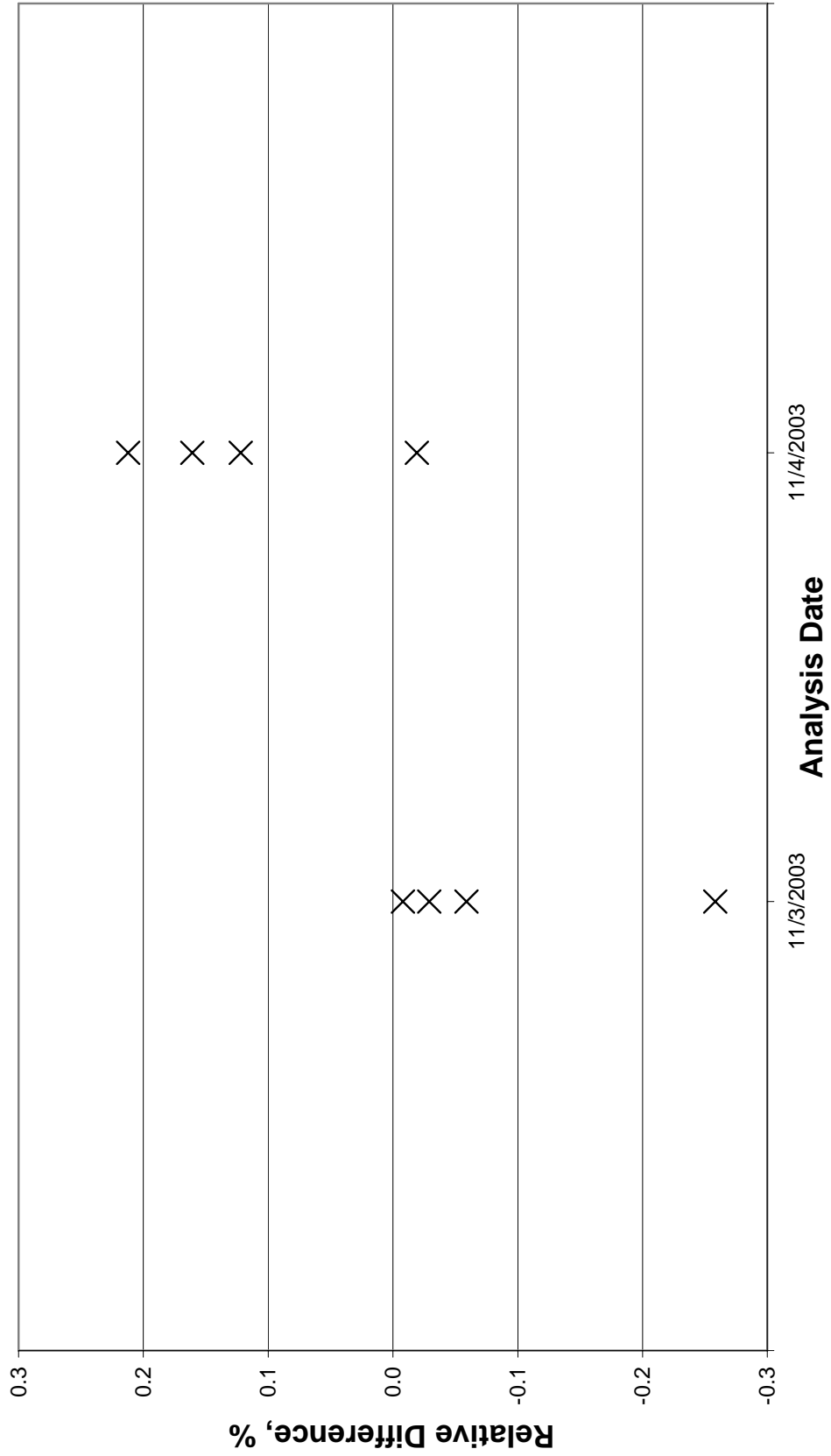
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<b>Number of Results Analyzed</b>	8
<b>Mean % Difference</b>	0.015
<b>Mean Absolute % Difference</b>	0.109
<b>95% C.L. of Mean (df = 1)</b>	1.319
<b>Standard Deviation</b>	0.149
<b>Between-Day Standard Deviation (df = 1)</b>	0.294
<b>Within-Day Standard Deviation (df = 6)</b>	0.107
<b>Statistical Significance of Between-Day Standard Deviation</b>	96.6%

International target value for bias in IDMS is 0.1%.

International target value for precision in IDMS is 0.15%.

Laboratory XX  
UNH Solution -- IDMS



## D. FY 2004 ANALYSES RESULTS AND REPORTING FORMAT

The experimental results submitted by the participating laboratories during FY 2004 are shown in Appendices A to E. The results are arranged according to material type (uranium or plutonium), and also analysis type (elemental assay or isotopic abundance). Laboratories are identified by code letters only for maintaining confidentiality.

In Section E of this report, the results are discussed material-by-material. Uranium and plutonium assay results are discussed first (Sections E.1 to E.4) followed by uranium and plutonium isotopic abundance results (Sections E.5 and E.6). The measurement results are evaluated in terms of grand mean % RDs and their standard deviations. The grand means and standard deviations of are calculated for each material/method/laboratory from all results submitted during the year.

In section E, the grand mean % RDs and standard deviations are presented in Tables 5 to 12. The tables also contain the following information: code letter for the participant, the method of analysis, the number of good results (outliers removed), bias target values and precision target values.

The data presented in the tables are also shown graphically in Figs. 3 to 18. There are two types of figures: the material-measurement skeletal figures to evaluate bias, and the material-measurement line figures to evaluate precision. In the material-measurement skeletal figures (odd numbers figures between Fig.3 and Fig.18), the mean % RDs are shown as diamonds. The vertical line passing through each diamond represents the standard deviation for that set. The bias target values are shown as dotted horizontal lines. If the diamonds (extended by the respective standard deviation of the results) fall within the horizontal lines, then the measurements are said to satisfy the bias target values; those falling outside fail. The magnitude of bias (if any) can be estimated only with reference to the mean % RD and its uncertainty at 95% C.L. No bias is indicated if the mean % RD extended by the uncertainty includes zero. If it fails to include zero, bias is indicated; above zero indicates positive bias and below zero indicates negative bias.

The material-measurement line figures (even number figures between Fig.3 and Fig.18) show precisions achieved in the measurements. The vertical line represents the standard deviation for each mean % RD. If the top of the vertical line is below the corresponding precision target value - shown as a dotted horizontal line - then the laboratory has satisfied the precision target value. If the vertical line extends beyond the horizontal, then the laboratory has failed the precision criterion. In these figures, the diamonds represent the absolute values of the mean % RDs. The



measurements are assumed to be bias-free if the diamonds fall on the abscissa or very close to it. The magnitude of bias can be estimated only with reference to % RD taken in conjunction with 95% C.L. uncertainty.

In Section F, a long-term evaluation is shown in graphical form only for uranium assay measurements results submitted during FY 2002, FY 2003 and FY 2004. See Figs.19 to 35.

## **E. FY 2004 PERFORMANCE EVALUATION: MATERIAL BY MATERIAL**

The results for uranium assay are given in Sections E.1 to E.4; for uranium isotopic abundance in Section E.5; and for plutonium assay and isotope abundance in Section E.6.

### **E.1. Uranyl Nitrate Solutions**

Test samples of uranyl nitrate solutions were made from enriched uranium (> 0.7% in  $^{235}\text{U}$ ) as well as natural uranium materials. Three different types of uranyl nitrate solutions were made: one solution from 50% enriched material, three solutions from 90% enriched material, and three solutions from natural uranium material. The uranium concentrations of these solutions were in the range of 7 to 10 mg uranium per gram of solution. The uranium contents of the three natural uranium solutions differed from each other by no more than 0.2% of each other; it was so in the three solutions from 90% material. These solutions with such closely spaced values for concentrations are ideal samples to test the analytical skills of a laboratory.

Laboratories not permitted to work with enriched materials received test samples of natural uranium solutions only.

#### **E.1.1. Preparation and Packaging for Shipment**

The uranyl nitrate solutions were sent to participating laboratories in flame sealed glass ampoules with break-off tips. Each ampoule was packed in a plastic bag. The bag was wrapped in absorbent cushioning material and sealed in another large plastic bag. The large bag was then kept inside a screw-cap fiberboard can for shipping.

### E.1.2. Reference Value and Uncertainty

NBL characterized the test samples in the ampoules for uranium concentrations using the modified Davies and Gray titration procedure. The uncertainties (95% C.L.) in uranium concentrations are as follows:  $\pm 0.1\%$  for the 50% enriched uranium solution,  $\pm 0.02\%$  for the 90% enriched uranium solutions, and in the range of  $\pm 0.02$  to  $\pm 0.05\%$  for the natural uranium solutions.

In a separate experiment, it was shown that the solutions did not suffer concentration change as a result of flame sealing. Samples withdrawn from sealed ampoules of natural uranium solutions as well as from its original stock showed little or no difference in uranium concentrations; the results agreed within a few hundredths of one percent.

### E.1.3. Performance Evaluation

The participating laboratories determined the uranium concentrations of the test samples using a variety of methods: Davies-Gray titration, IDMS, and x-ray fluorescence (XRF). The results, in terms of mean % RDs, are shown in Table 5 along with the target values for each method. The % RDs along with standard deviations are shown in Fig.3 to evaluate bias and in Fig.4 to evaluate precision.

Laboratory B met neither bias nor precision target values for Davies-Gray titration. Laboratories B and G met neither bias nor precision target values for IDMS. All other laboratories satisfied both bias and precision target values.

**Table 5. Inter-laboratory performance summary for uranium assay in UNH solutions**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)	
					Bias	Precision
Davies-Gray Titration	B	0.230	0.287	33	0.1	0.1
	F	-0.011	0.052	30	0.1	0.1
	G	0.000	0.023	24	0.1	0.1
IDMS	A	0.033	0.100	31	0.1	0.15
	B*	0.116	1.321	16	0.1	0.15
	G*	0.755	0.196	6	0.1	0.15
	J	-0.048	0.116	28	0.1	0.15
X-Ray Fluorescence	A*	-0.235	0.276	29	0.5#	0.5#

# International Target Values are not available for XRF, and therefore DOE target values are shown.

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
UNH - Percent U

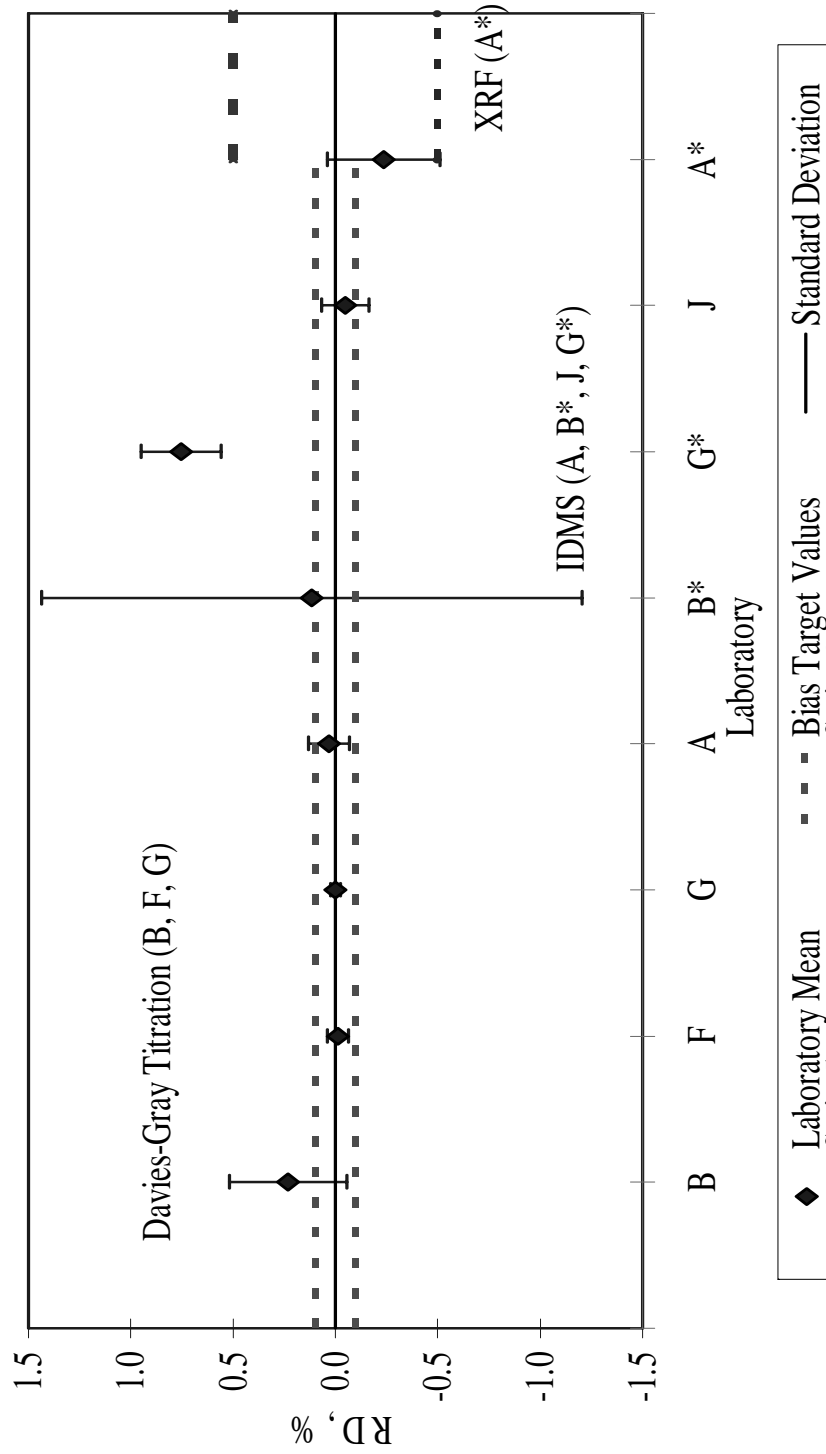
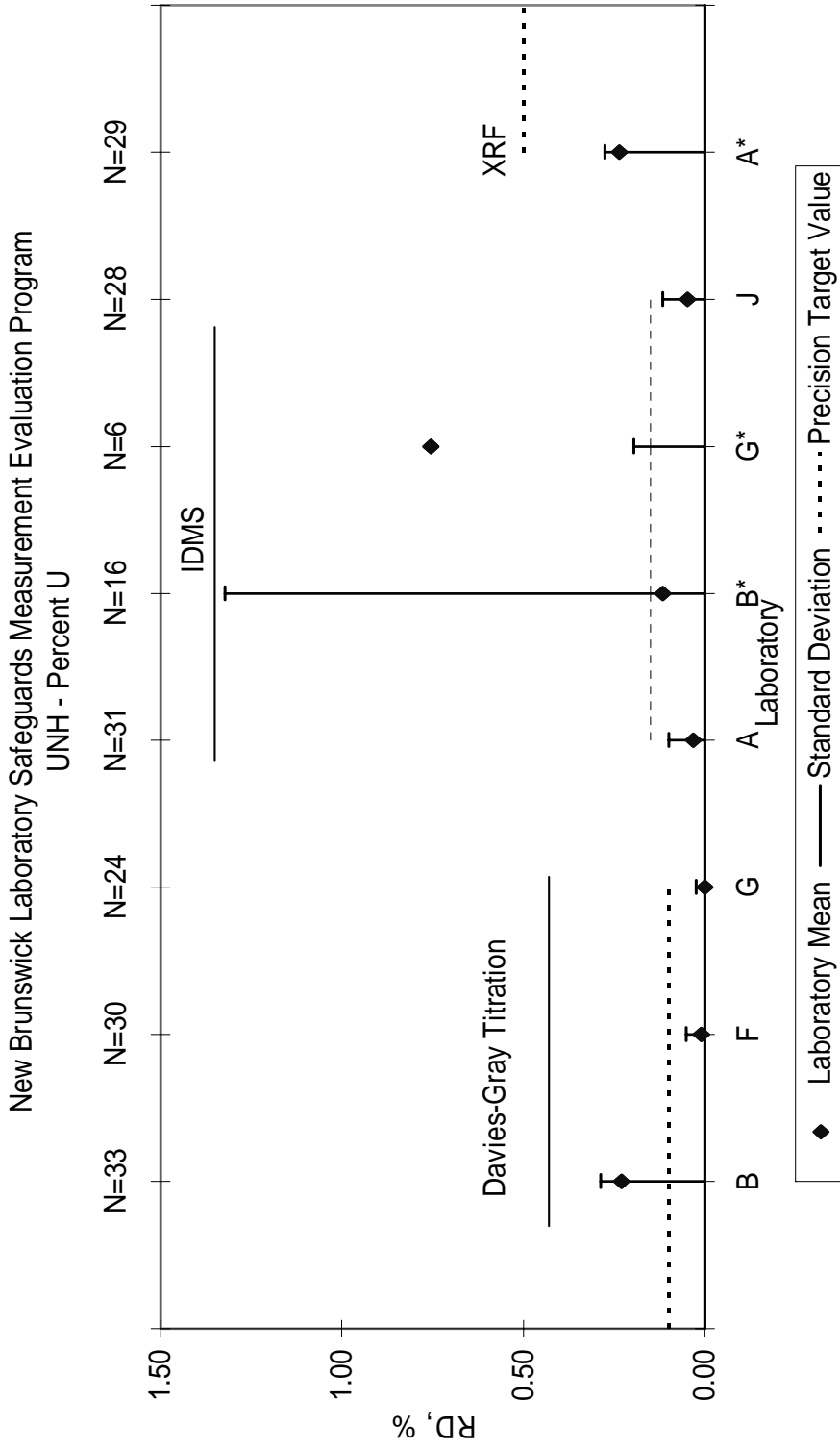


Figure 3

Figure 4



## E.2. Enriched Uranium Dioxide Pellet

The uranium dioxide pellet test sample is the same as the Certified Reference Material 125-A. The pellets were originally made in a single batch at the Westinghouse Commercial Nuclear Fuel Division (a NRC licensee), using a high temperature sintering process at 1700°C for 20 hours in a reducing atmosphere. The pellets are known to be stable; they suffer no compositional change on exposure to air and are resistant to moisture uptake. The pellets serve as a test material for both uranium assay and uranium isotopic abundance measurement. The <sup>235</sup>U content is about 4.5%.

### E.2.1. Preparation and Packaging for Shipment

The UO<sub>2</sub> pellets were wrapped in low-lint tissue to prevent chipping, placed in snap-cap glass bottles, and the bottles sealed in plastic bags. The bottles were shipped in cardboard tube containers.

### E.2.2. Reference Value and Uncertainty

The elemental uranium concentration of the pellets was determined by the NBL high-precision titration method. CRM 112-A, a uranium metal assay standard, was used for quality control and traceability. The uranium concentration was measured with an uncertainty of about ± 0.02% at 95% C.L.

### E.2.3. Performance Evaluation

Two laboratories analyzed the uranium oxide pellets for uranium concentration using Davies-Gray Titration. One of the two laboratories analyzed it by high-precision titration also. The mean of % RDs along with uncertainties are shown in Table 6 along with the target values for each method. The % RDs along with standard deviations are shown in Fig.5 to evaluate bias and in Fig.6 to evaluate precision. The two laboratories met the bias and precision criteria.

**Table 6. Inter-laboratory performance summary for uranium assay in UO<sub>2</sub> Pellets**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)	
					Bias	Precision
High Precision Titration	F	-0.019	0.006	6	0.05*	0.05*
Davies-Gray Titration	F*	-0.033	0.028	15	0.1	0.1
Davies-Gray Titration	T	-0.017	0.075	15	0.1	0.1

\* No ITVs were available, but were assumed to be the same as for the gravimetric method.

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
 UO2 Pellets - Percent U by Titration Method

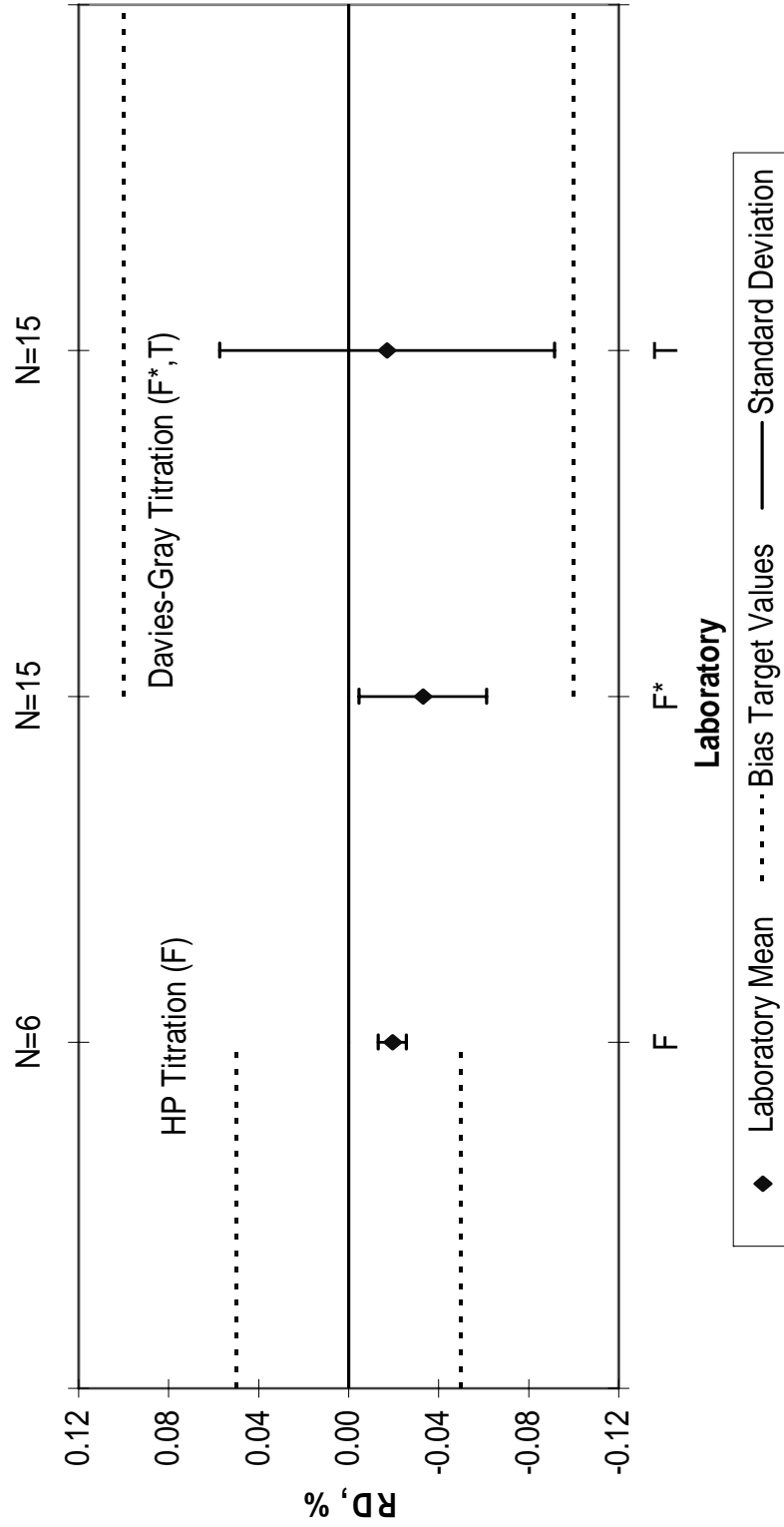
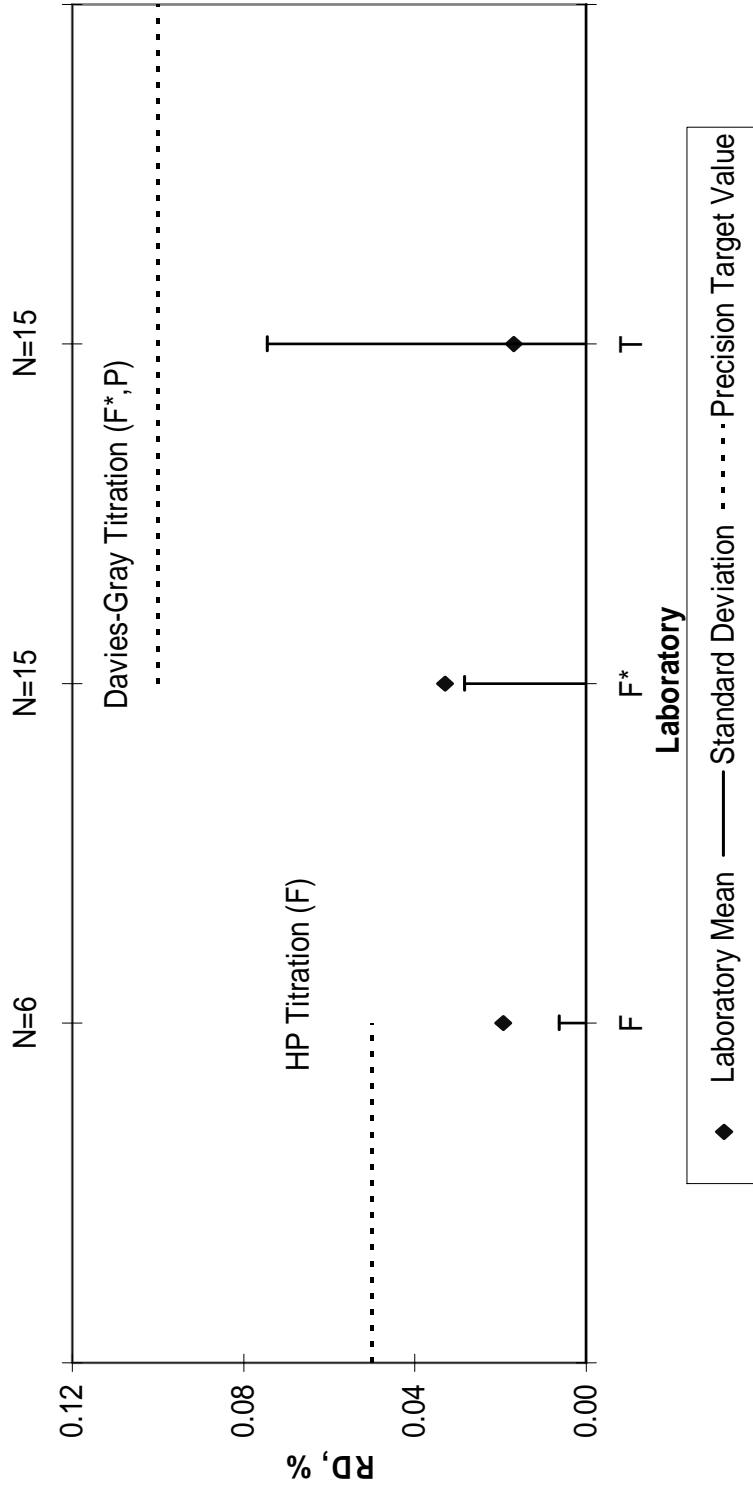


Figure 5

**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
UO2 Pellets - Percent U by Titration Method**



**Figure 6**

### **E.3. Uranium Hexafluoride**

In FY 1993, Portsmouth Gaseous Diffusion Plant donated two sampling manifolds to NBL for transferring UF<sub>6</sub> from 2S cylinders to P-10 tubes. One of the two manifolds was used to transfer natural UF<sub>6</sub>, and the other for enriched material. These manifolds have been taken out of service. Now, NBL is relying on Portsmouth Gaseous Diffusion facility for the preparation of SME test samples. Portsmouth will be using UF<sub>6</sub> material in its custody, but belonging to NBL, for making future SME test samples.

#### **E.3.1. Preparation and Packaging for Shipment**

The Portsmouth Gaseous Diffusion facility prepared and packaged UF<sub>6</sub> test samples in P-10 tubes. Each test sample contains 7 to 12 g of UF<sub>6</sub> and is about 4% enriched. These samples originate from the same stock from which CRM 113-B was made.

#### **E.3.2. Reference Value and Uncertainty**

The UF<sub>6</sub> test samples in P-10 tubes were characterized for uranium concentration using the NBL high-precision titration method. Quality assurance and traceability were provided through analyses of CRM 112-A (uranium metal assay standard), and UF<sub>6</sub> made from normal uranium. The uranium concentration of the test samples was defined with an uncertainty of  $\pm 0.033\%$  (95% C.L.). The isotopic abundance of the test samples (4% enriched) was also measured.

#### **E.3.3. Performance Evaluation**

In FY 2004, no laboratory participated in UF<sub>6</sub> analysis.



#### **E.4. Uranium Oxide (UO<sub>3</sub>) Powder**

UO<sub>3</sub> powder is an ideal test material to monitor the capability of a laboratory in analyzing hygroscopic materials. It was used as a test material several years ago, but was discontinued for sometime in between because of a perceived lack of interest in this material. A few years ago, it was re-introduced as a test material at the request of a participant laboratory. Two different laboratories analyzed it in FY 2004.

##### **E.4.1. Preparation and Packaging for Shipment**

The test samples come from preparations done several years ago. Originally, the samples were packaged into pharmaceutical vials with Teflon-lined stoppers, under dry nitrogen atmosphere. The vials were crimp sealed, then sealed in plastic, and packaged in cardboard tubes for shipping.

##### **E.4.2. Reference Value and Uncertainty**

The elemental concentration of uranium in UO<sub>3</sub> material was characterized through analysis of 8 different samples using the NBL-modified Davies and Gray titration method. Quality control and traceability were provided through analysis of CRM 112-A (a uranium metal assay standard). The uranium content of the test samples differed from the original value by about 0.064%, the new value being lower. The uncertainty (95% C.L.) in the new measurements was 0.012%. Apparently, the concentration of uranium in the UO<sub>3</sub> material was not altered to a significant extent. The newly determined uranium value was used as the characterized value in the FY 2004 program.

##### **E.4.3. Performance Evaluation**

One laboratory analyzed the UO<sub>3</sub> test samples for uranium concentration using three different methods: IDMS, XRF (liquid), and XRF (solid); and another laboratory by the Davies-Gray method only. The mean % RDs are shown in Table 7 along with the target values for each method. The % RDs along with the standard deviations are shown in Fig.7 to evaluate bias and in Fig.8 to evaluate precision. Laboratory A satisfactorily met the bias as well as precision target values for IDMS. The same laboratory missed the bias target value for x-ray fluorescence measurement, but met the precision target value. Laboratory F met both bias and precision target values.

**Table 7. Inter-laboratory performance summary of uranium assay in UO<sub>3</sub>**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)#	
					Bias	Precision
Davies-Gray Titration	F	-0.051	0.035	16	0.1	0.1
IDMS	A	-0.030	0.079	29	0.1	0.15
X-Ray Fluorescence, Liquid	A*	-0.624	0.272	32	0.5	0.5
X-Ray Fluorescence, Solid	A**	-0.805	0.387	31	0.5	0.5

# ITVs are not available for XRF methods; DOE values were used instead.

**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
UO3 Powder - Percent U**

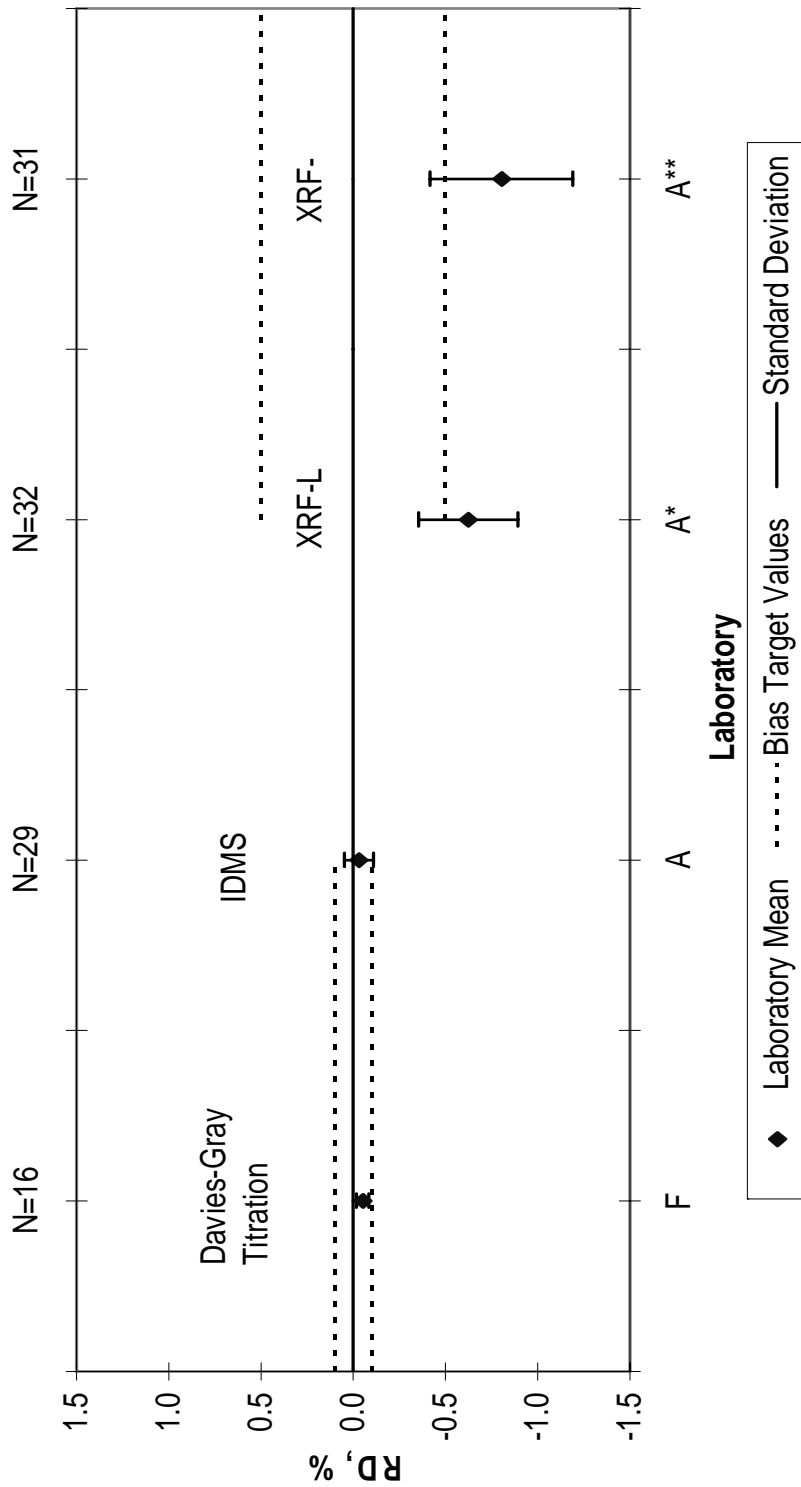
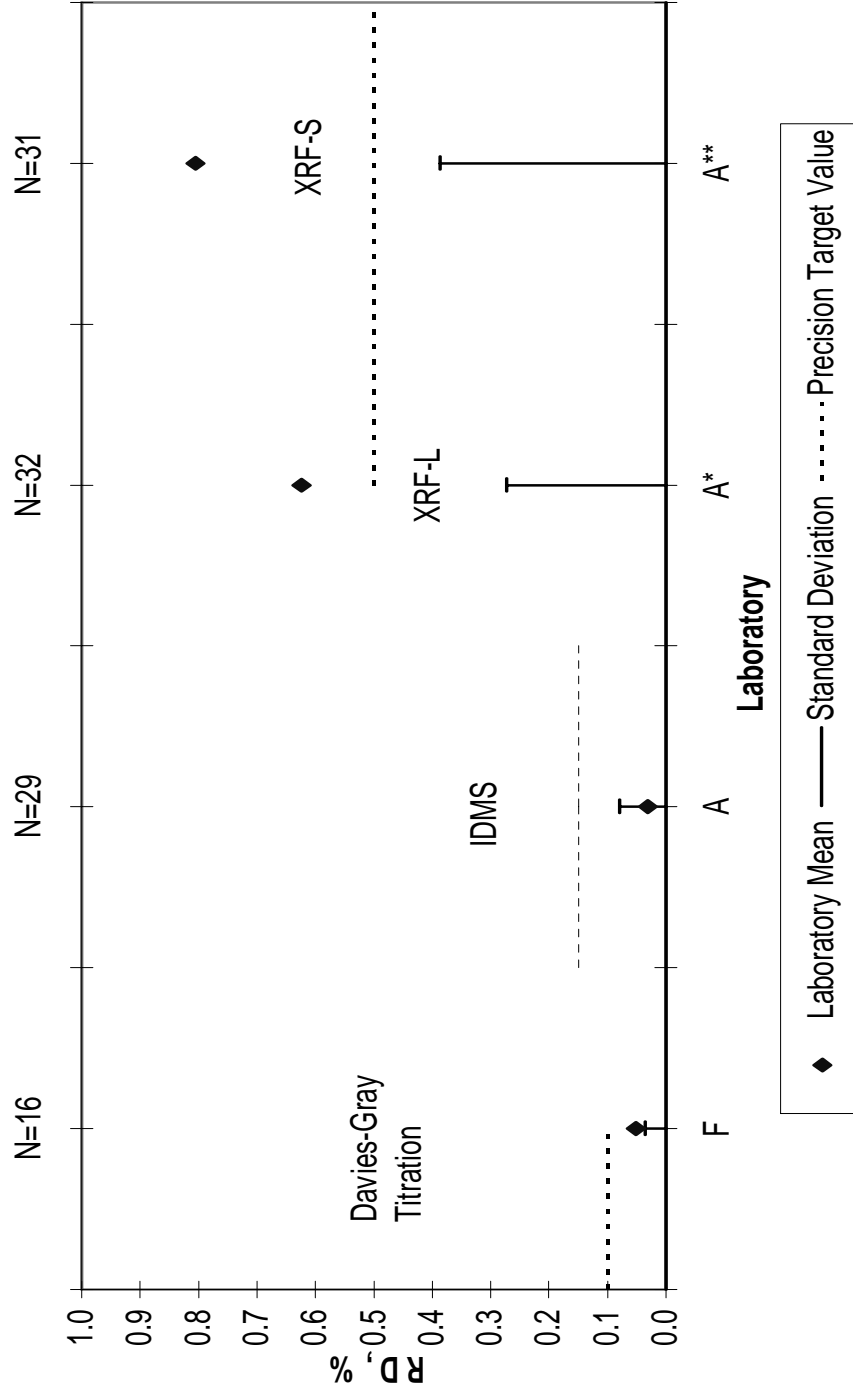


Figure 7

Figure 8

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
 UO3 Powder - Percent U



## **E.5. <sup>235</sup>U Enrichment**

A suite of enriched uranium test samples are available for evaluating isotopic abundance results: three uranyl nitrate solutions with 90% enrichment, one uranyl nitrate solution with 50% enrichment, one uranyl nitrate solution with 4% enrichment, solid UO<sub>2</sub> pellets of about 4% enrichment, and UF<sub>6</sub> solid of about 4.5% enrichment.

### **E.5.1. Preparation and Packaging for Shipment**

The uranyl nitrate solutions were packaged in flame-sealed glass ampoules with a break-off tip. The ampoules were sealed in plastic, wrapped in absorbent cushioning, sealed in plastic again, and packaged in cardboard tubes for shipping. Each solution contained 5-10 mg uranium/g solution.

The UO<sub>2</sub> pellets were packaged in a snap-cap glass bottle with a low-lint tissue for cushioning to prevent chipping. The glass bottles are sealed in plastic, and packaged in cardboard tubes for shipping.

The UF<sub>6</sub> test samples in P-10 tubes were packed in sealed plastic bags and shipped in cardboard containers with screw caps.

### **E.5.2. Reference Value and Uncertainty**

The uranium isotopic abundances in the test materials, except UF<sub>6</sub>, were characterized by thermal ionization mass spectrometry (TIMS). The experimental results were corrected for mass fractionation effects. The correction factors were determined through analyses of appropriate certified reference materials done under the same conditions as the test materials.

The UF<sub>6</sub> material was characterized by both TIMS and gas mass spectrometry. The TIMS measurements required hydrolyzed UF<sub>6</sub> samples; the entire sample in P-10 tube was hydrolyzed. On the other hand, gas spectrometry measurements were done directly utilizing a small amount of sample in the P-10 tube.

The uncertainties (95% C.L.) in <sup>235</sup>U abundance by TIMS were as follows: 0.02% for the 4% enriched uranyl nitrate solution; < 0.01% for the 50% and 90% enriched solutions; 0.07% for UO<sub>2</sub> pellets; and 0.053% for UF<sub>6</sub>. The uncertainties for the uranium nitrate solutions did not include the

uncertainties in determining the mass fractionation correction factors, whereas the uncertainties in  $UO_2$  and  $UF_6$  included mass fractionation correction factor uncertainties.

### E.5.3. Performance Evaluation

The participating laboratories analyzed the test samples using TIMS. The mean % RDs are shown in Table 8 for HEU materials ( $\geq 20\%$  enriched), and in Table 9 for LEU materials ( $<20\%$  enriched). Target values are also shown in the tables; the HEU target values are more stringent than those for LEU.

Five laboratories analyzed the HEU samples. The % RDs along with standard deviations are shown in Fig.9 to evaluate bias and in Fig.10 to evaluate precision. All five laboratories were able to measure  $^{235}U$  abundance within bias and precision target values.

Four laboratories analyzed the LEU samples. The % RDs along with standard deviations are shown in Fig.11 to evaluate bias and in Fig.12 to evaluate precision. All four laboratories were able to measure  $^{235}U$  abundance within bias and precision target values.

**Table 8. Inter-laboratory performance summary for  $^{235}U$  enrichment in HEU**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)	
					Bias	Precision
TIMS	A	0.007	0.019	24	0.05	0.05
TIMS	B	0.026	0.033	24	0.05	0.05
TIMS	F	0.001	0.001	12	0.05	0.05
TIMS	G	0.014	0.012	15	0.05	0.05
TIMS	J	0.000	0.014	28	0.05	0.05

**Table 9. Inter-laboratory performance summary for  $^{235}U$  enrichment in LEU**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)	
					Bias	Precision
TIMS	A	0.010	0.079	8	0.1	0.1
TIMS	B	0.047	0.055	8	0.1	0.1
TIMS	F	-0.026	0.028	17	0.1	0.1
TIMS	T	0.053	0.030	16	0.1	0.1

**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
U235 Enrichment - HEU**

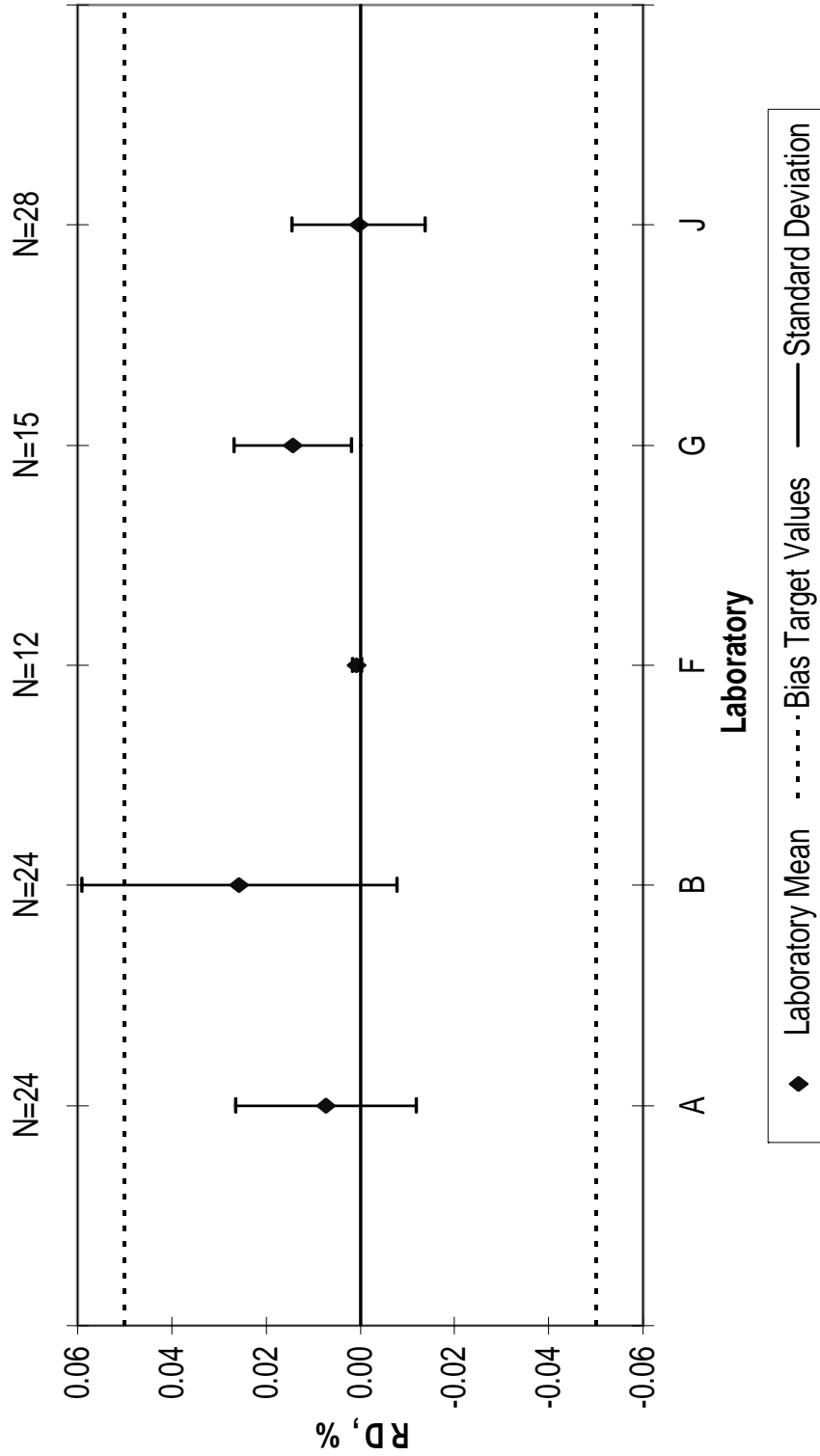


Figure 9

Figure 10

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
U235 Enrichment - HEU

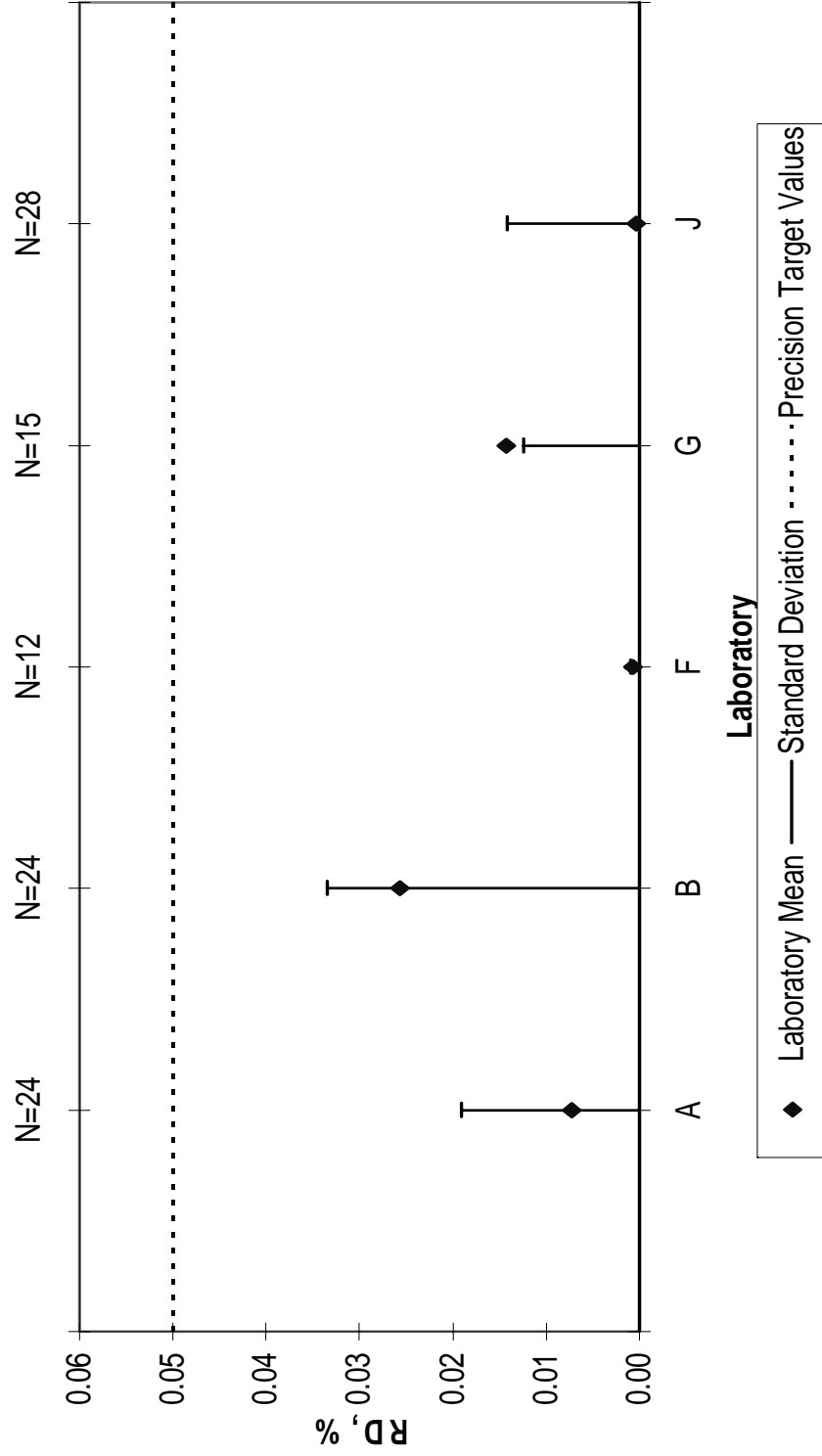
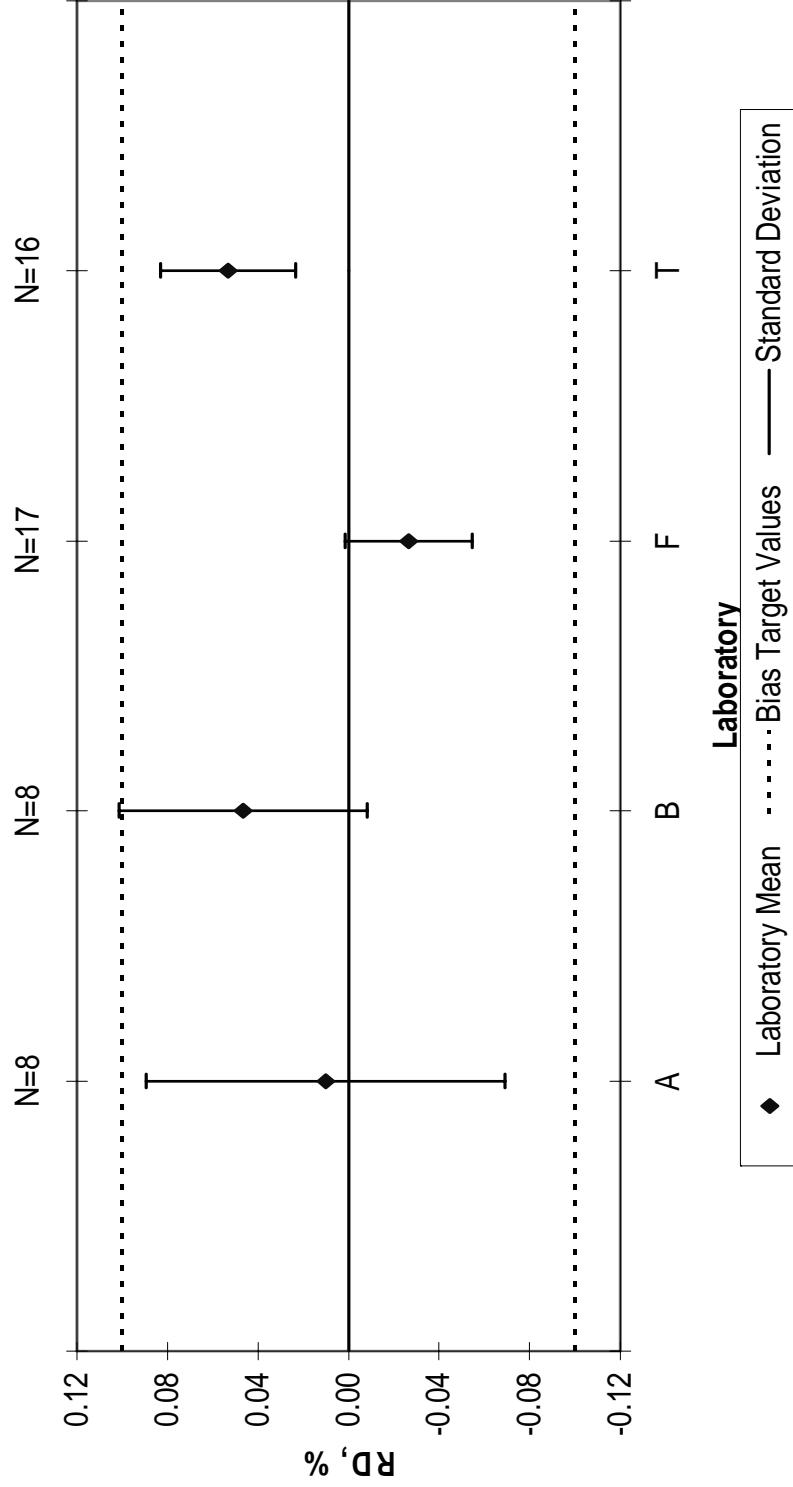




Figure 11

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
U235 Enrichment - LEU



**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
U235 Enrichment - LEU**

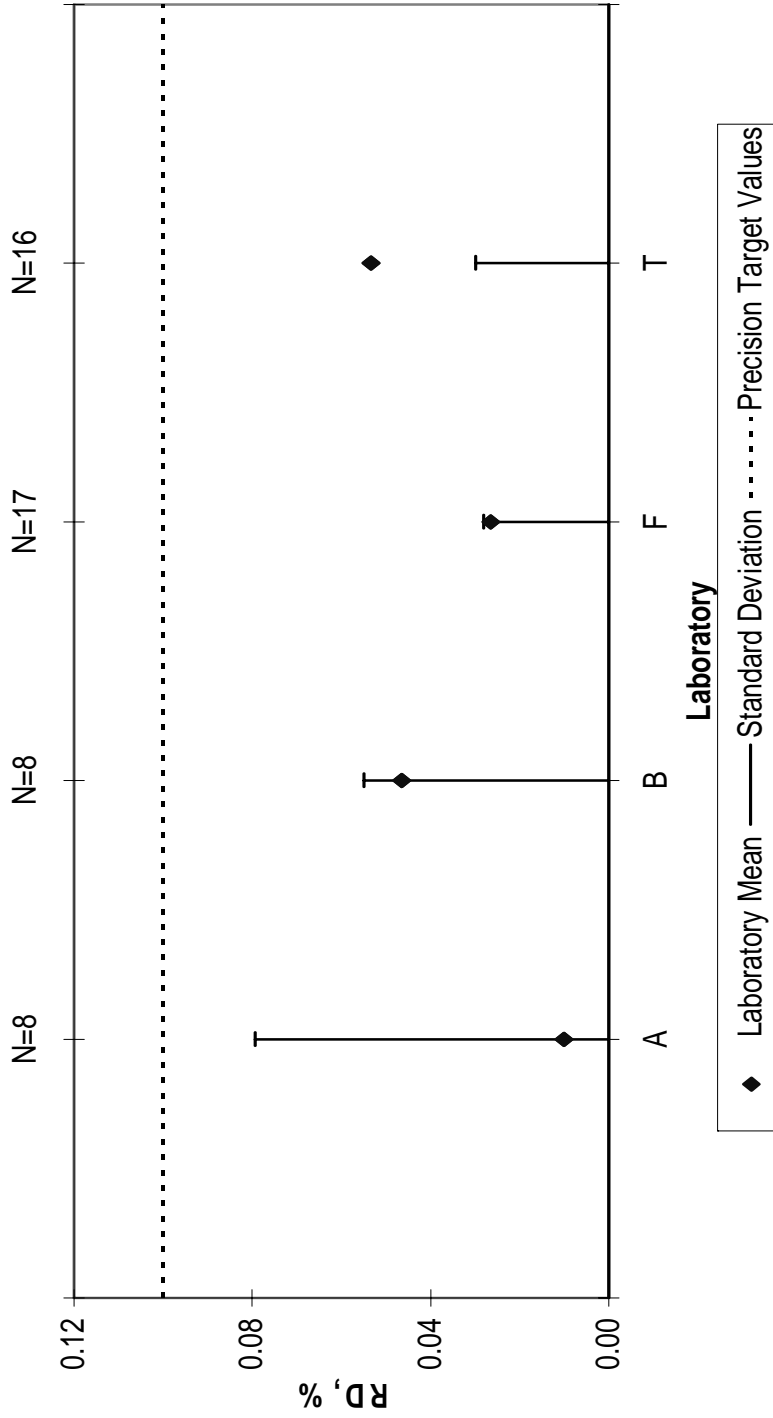


Figure 12

## **E.6. Plutonium Assay and Isotopic Abundance**

Test materials for plutonium assay came from two different sources: CRM 126, a plutonium metal standard, and CRM 122, a plutonium oxide standard. The CRMs were dissolved, and diluted to the required concentrations using 8M nitric acid. Aliquants containing approximately 20 and 40 micrograms of plutonium were placed in glass bottles and fumed to dryness in the presence of sulfuric acid.

Plutonium isotopic abundance test samples were prepared from three different certified reference materials: CRM 122 (plutonium oxide), CRM 136 (plutonium sulfate tetrahydrate), and CRM 137 (plutonium sulfate tetrahydrate). The CRMs were dissolved, and diluted to the required concentration using 8M nitric acid. Each aliquant of the test material containing about one milligram of plutonium was placed in a glass bottle and fumed to dryness in the presence of sulfuric acid. The dried test samples were sent to participants without further purification. Note that unpurified samples contain small amounts of isobaric nuclides ( $^{238}\text{U}$  and  $^{241}\text{Am}$ ) as impurities that may interfere in plutonium isotopic abundance determination.

### **E.6.1. Preparation and Packaging for Shipment**

The plutonium assay samples were contained in glass bottles (20 mL scintillation vials) to facilitate direct addition of IDMS spikes into the test materials. The isotopic test samples were also in the same type of glass bottles. The bottles were placed in a plastic bag, heat sealed; these were again sealed in another plastic bag. The samples were shipped in produce cans.

### **E.6.2. Reference Value and Uncertainty**

The characterized values for plutonium concentrations in the test samples were calculated from the certified values for plutonium assay and the masses of reference materials dissolved to make the test solutions. The uncertainties (95% C.L.) were about 0.02% for CRM 126, and about 0.04% for CRM 122.

The characterized values for plutonium isotopic abundance in the test samples were assumed to be the same as those in the certificates with appropriate corrections for radioactive decay. The uncertainties (95% C.L.) in the characterized values were assumed to be the same as those reported in the respective certificates. The ranges of isotopic abundance of plutonium nuclides in the three test materials were as follows:  $^{238}\text{Pu}$  from 0.05% to 0.25%;  $^{239}\text{Pu}$  from 78% to 88%;  $^{240}\text{Pu}$

from 12% to 19%;  $^{241}\text{Pu}$  from 0.05% to 1.3%; and  $^{242}\text{Pu}$  from 0.2% to 1.2%. Test samples with higher abundance of  $^{239}\text{Pu}$  (and lower  $^{240}\text{Pu}$ ) are characterized as low burn-up materials, whereas those with lower abundance of  $^{239}\text{Pu}$  (and higher  $^{240}\text{Pu}$ ) are characterized as high burn-up material.

### E.6.3. Performance Evaluation

The participating laboratories determined the plutonium concentrations in the test samples by IDMS, and the plutonium isotopic abundance using TIMS.

#### E.6.3.1. Plutonium Assay

Two laboratories participated in plutonium assay measurements. The mean % RDs are shown in Table 10 along with the target values. The % RDs and the standard deviations are shown in Fig.13 to evaluate bias and in Fig.14 to evaluate precision. Both laboratories failed to meet the bias target value. Laboratory G met the precision criterion, while laboratory F did not.

**Table 10. Inter-laboratory performance summary for plutonium assay in dried plutonium sulfate.**

Method	Lab code	Mean % RD	Standard deviation	N	ITV (%)	
					Bias	Precision
IDMS	F	-0.770	1.628	10	0.1	0.15
IDMS	G	-1.034	0.092	4	0.1	0.15

**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
Pu Sulfate - Percent Pu**

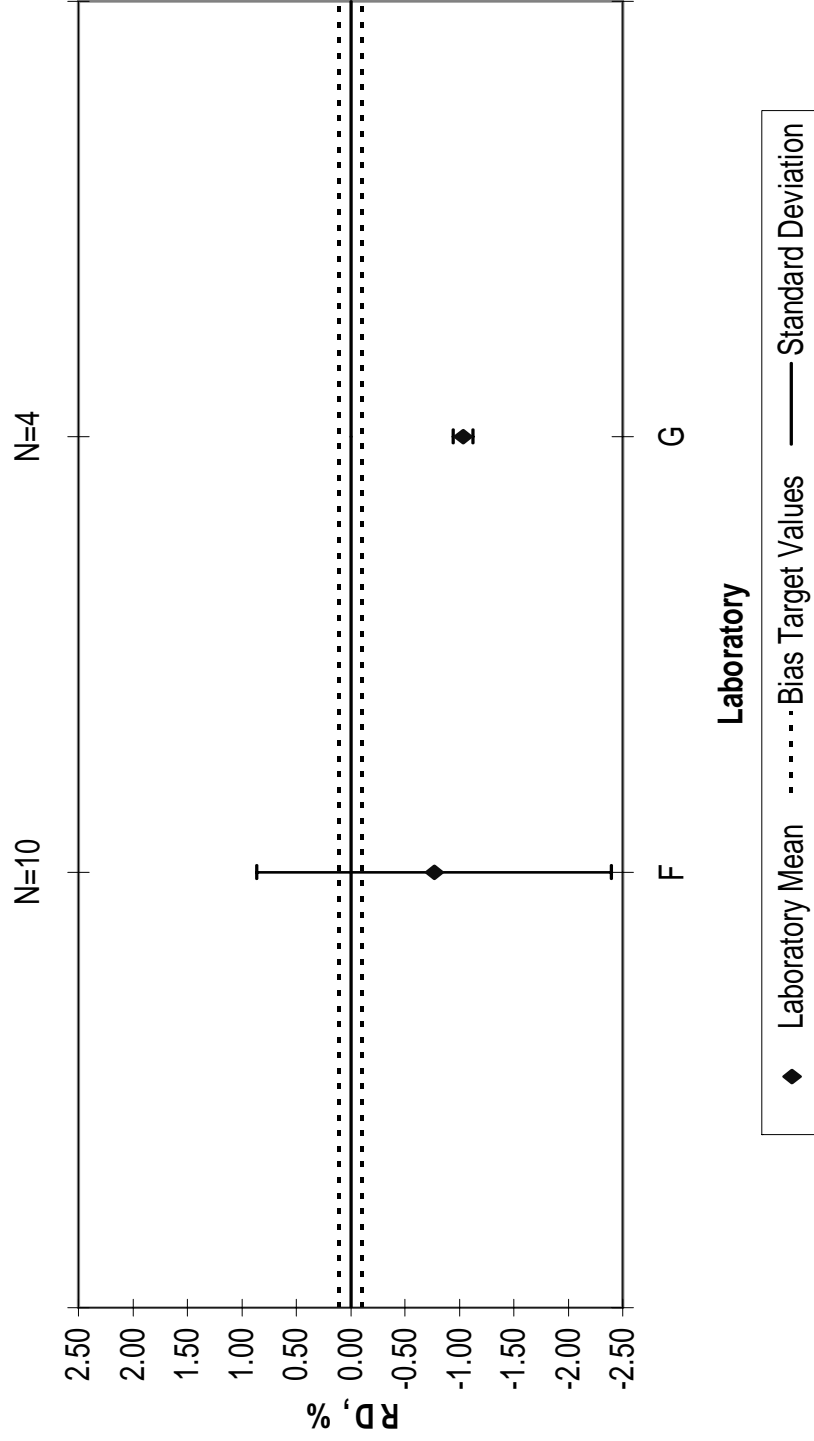


Figure 13

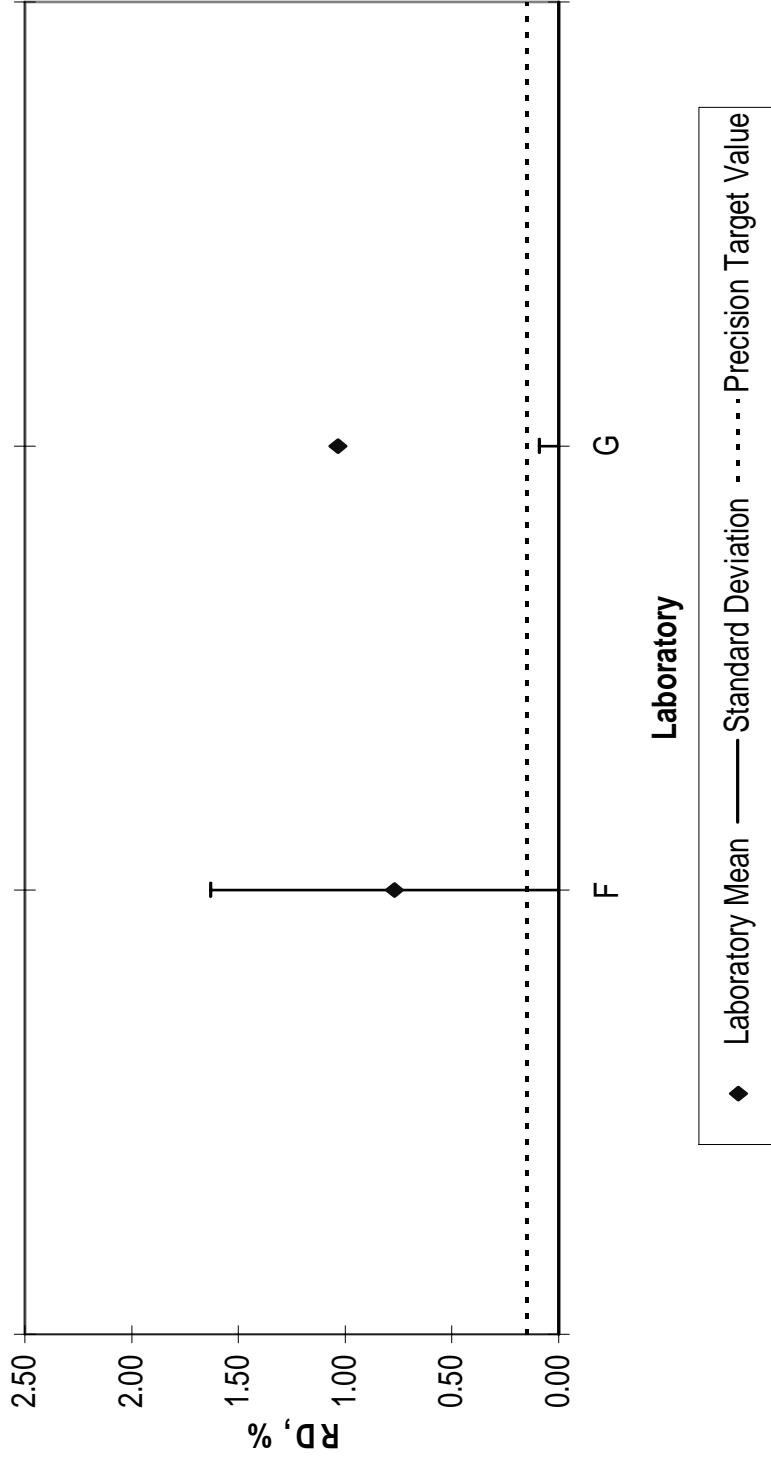
Figure 14

New Brunswick Laboratory Safeguards Measurement Evaluation Program

Pu Sulfate - Percent Pu

N=4

N=10



### E.6.3.2. <sup>239</sup>Pu Abundance

Four laboratories analyzed the test samples for isotopic abundance. Results for the two major isotopes, <sup>239</sup>Pu and <sup>240</sup>Pu, were evaluated; minor isotopes (<sup>238</sup>Pu, <sup>241</sup>Pu and <sup>242</sup>Pu) were not evaluated.

The mean % RDs for <sup>239</sup>Pu are shown in Table 11 along with the target values. The results from high burn-up and low burn-up plutonium samples are presented without making any distinction between them. The target values for low burn-up samples are more stringent than those for the high burn-up samples. All results are judged against the low burn-up target values.

The % RDs and the standard deviations for <sup>239</sup>Pu abundance measurements are shown in Fig.15 to evaluate bias, and again in Fig.16 to evaluate precision. In both figures, the target values corresponding to low burn-up material only are shown. Laboratories F, J and T satisfied both bias and precision target values, and laboratory G missed both.

**Table 11. Inter-laboratory performance summary for <sup>239</sup>Pu Abundance in dried plutonium sulfate**

Method	Lab code	Mean % RD	Standard deviation	N	Bias ITV (%)		Precision ITV (%)	
					High Burn-up	Low Burn-up	High Burn-up	Low Burn-up
TIMS	F	0.003	0.002	12	0.04	0.01	0.06	0.01
TIMS	G	-0.017	0.026	6	0.04	0.01	0.06	0.01
TIMS	J	-0.007	0.009	21	0.04	0.01	0.06	0.01
TIMS	T	0.001	0.005	16	0.04	0.01	0.06	0.01

# New Brunswick Laboratory Safeguards Measurement Evaluation Program

Pu239

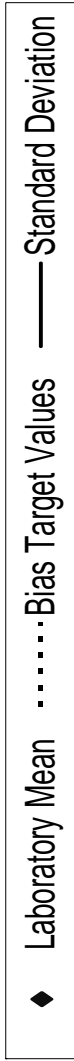
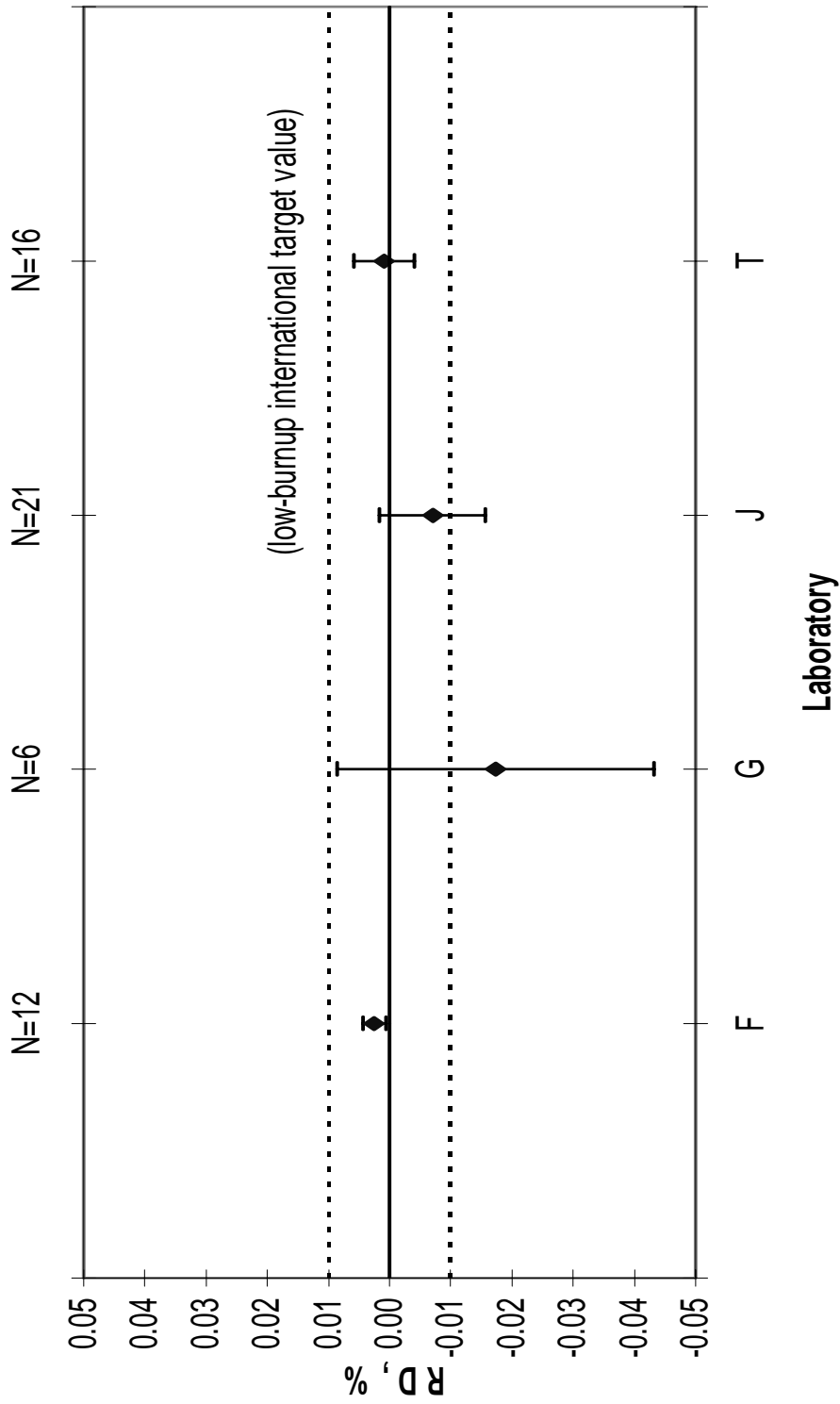


Figure 15



**New Brunswick Laboratory Safeguards Measurement Evaluation Program  
Pu239**

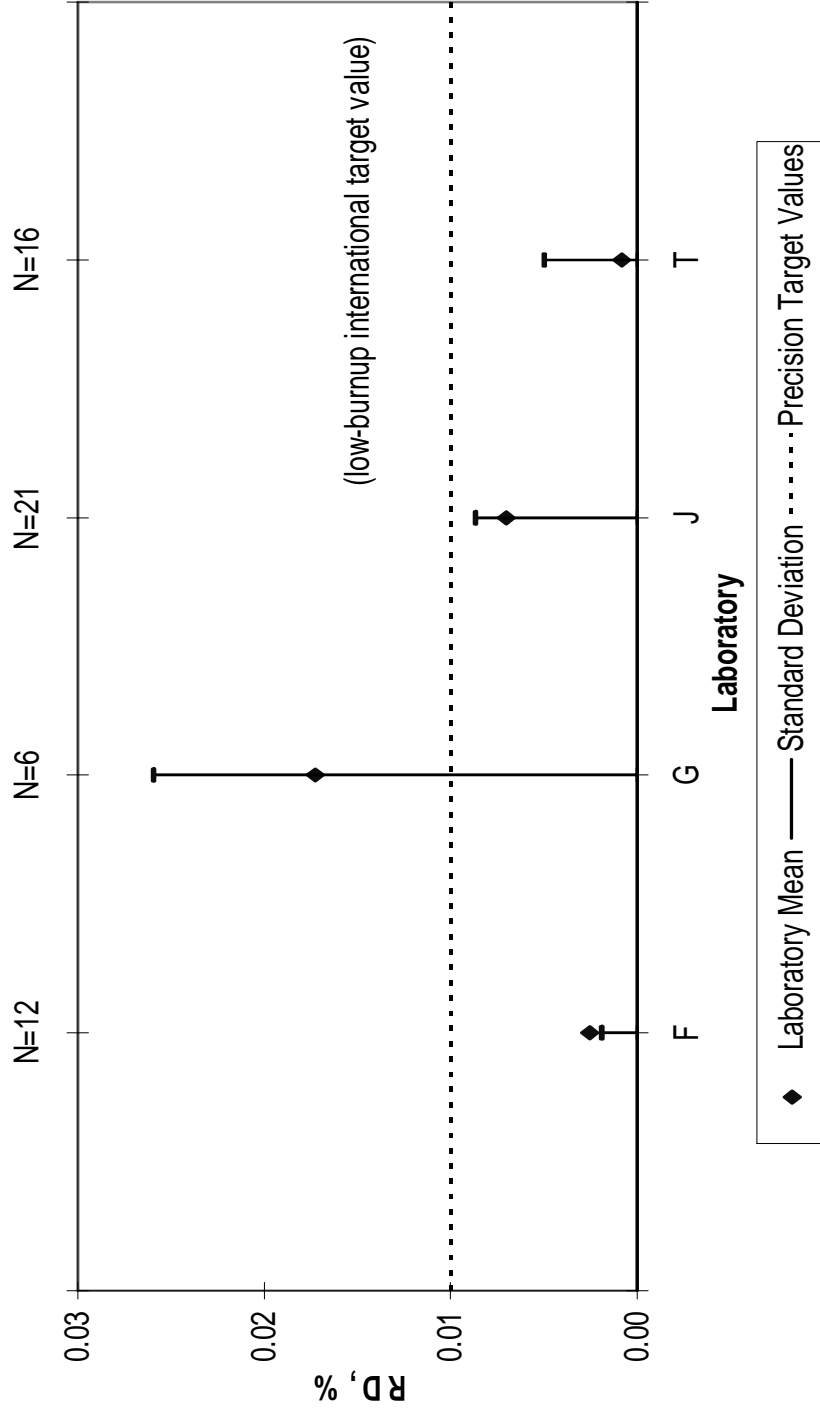


Figure 16

### E.6.3.3. <sup>240</sup>Pu Abundance

Four laboratories analyzed the test samples for isotopic abundance. Results for the two major isotopes, <sup>239</sup>Pu and <sup>240</sup>Pu, were evaluated; minor isotopes (<sup>238</sup>Pu, <sup>241</sup>Pu and <sup>242</sup>Pu) were not evaluated.

The mean % RDs for <sup>240</sup>Pu are shown in Table 12 along with the target values. The results from high burn-up and low burn-up plutonium samples are presented without making any distinction between them. The target values for high burn-up plutonium are more stringent than those for the low burn-up material. The results are judged against the high burn-up values.

The % RDs and the standard deviations for <sup>240</sup>Pu abundance measurements are shown in Fig.17 to evaluate bias, and again in Fig.18 to evaluate precision. In both figures, the target values corresponding to high burn-up material only are shown. Laboratories F, J and T satisfied both bias and precision ITVs. Laboratory G missed both bias and precision target values.

**Table 12. Inter-laboratory performance summary for <sup>240</sup>Pu abundance in dried plutonium sulfate**

Method	Lab code	Mean % RD	Standard deviation	N	Bias ITV (%)		Precision ITV (%)	
					High Burn-up	Low Burn-up	High Burn-up	Low Burn-up
TIMS	F	-0.055	0.069	12	0.12	0.15	0.07	0.10
TIMS	G	0.076	0.167	6	0.12	0.15	0.07	0.10
TIMS	J	0.021	0.038	21	0.12	0.15	0.07	0.10
TIMS	T	-0.017	0.027	16	0.12	0.15	0.07	0.10

Figure 17

### New Brunswick Laboratory Safeguards Measurement Evaluation Program

Pu240

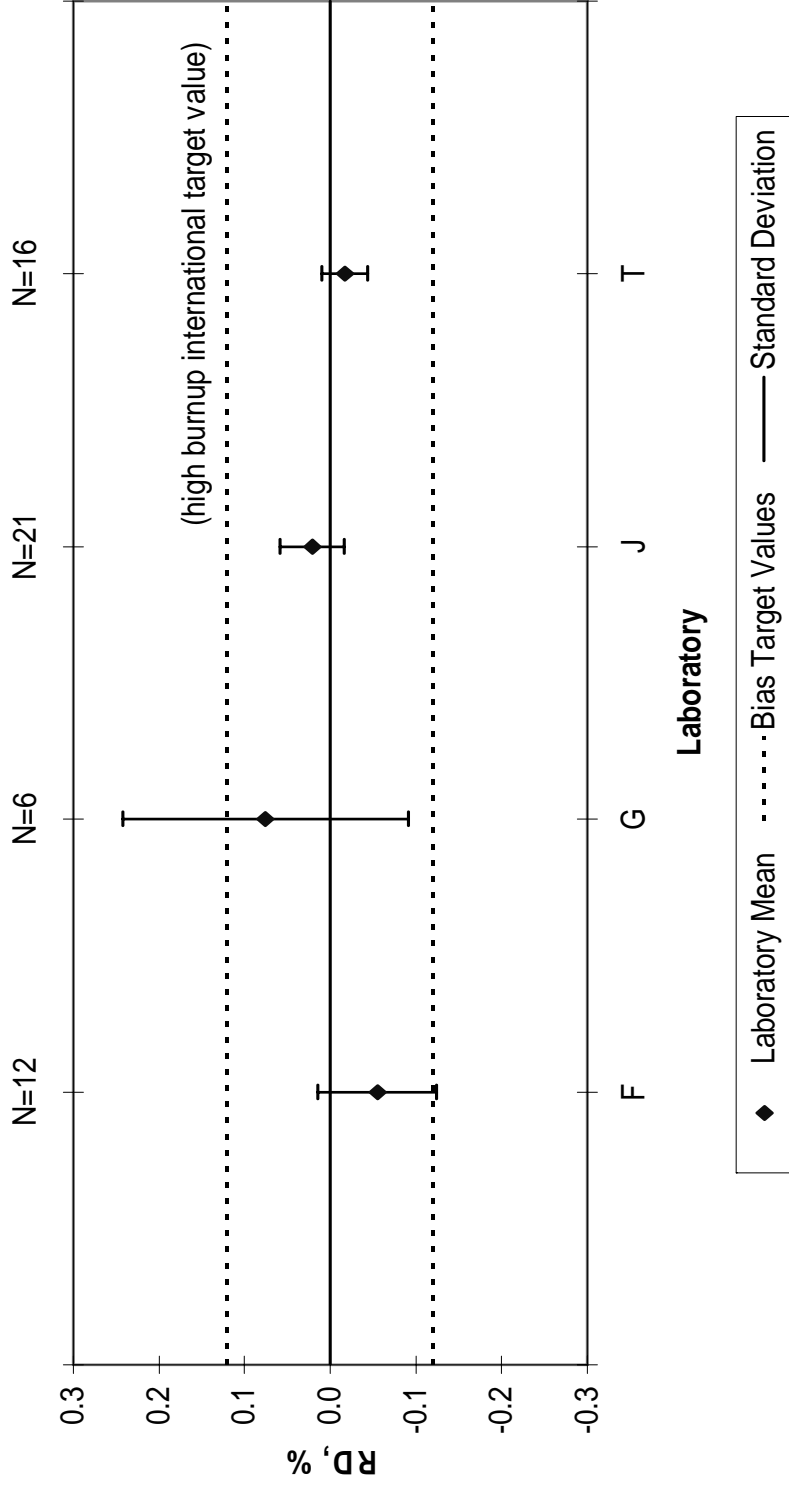
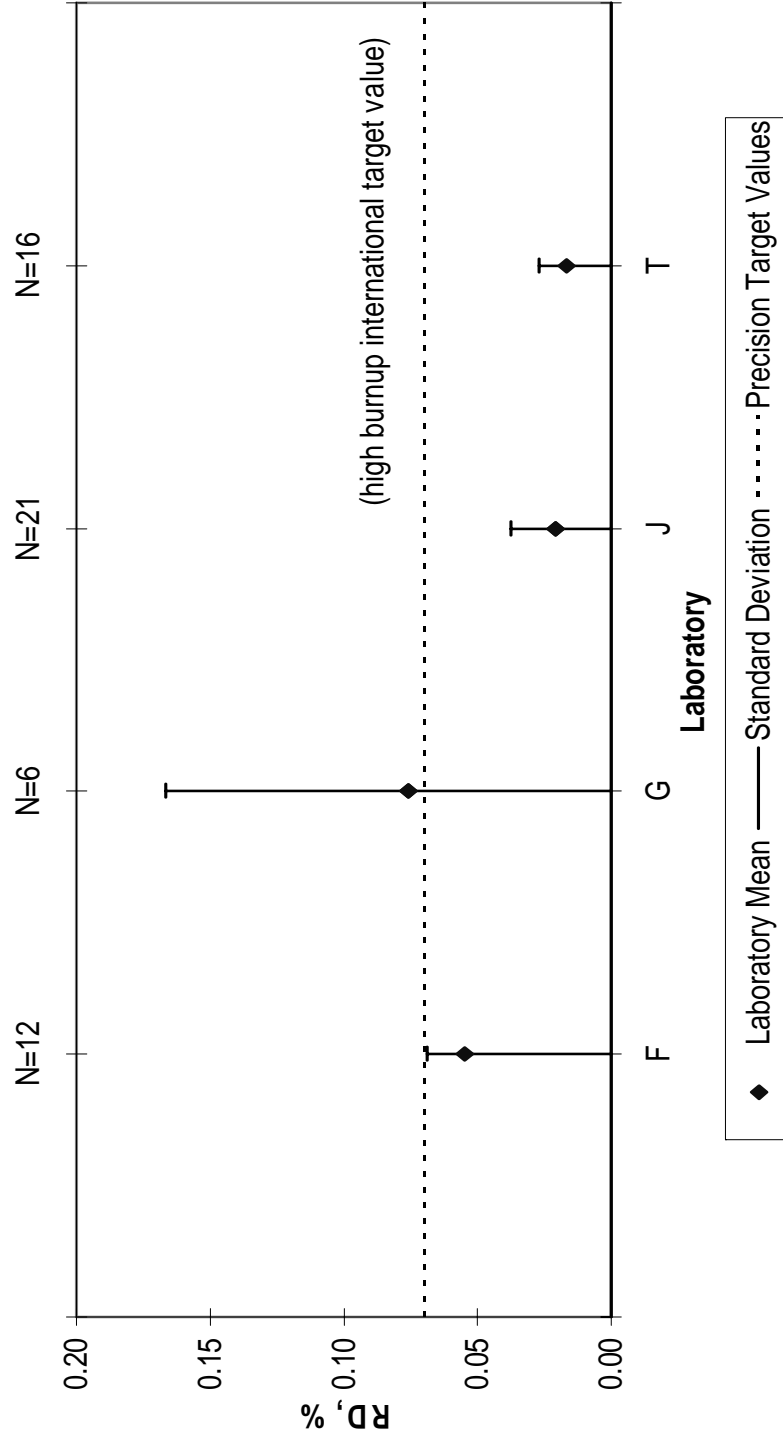


Figure 18

### New Brunswick Laboratory Safeguards Measurement Evaluation Program

Pu240



## **F. LONG TERM EVALUTION OF URANIUM MEASUREMENTS: FY 2002-2004**

The uranium assay results submitted by the participating laboratories during FY 2002 to FY 2004 are evaluated in this section. The % RDs calculated from the submitted results are shown in Figs.19 to 35. Each figure contains results from one laboratory for a material/method combination. For example, Fig. 19 shows results from laboratory A for uranyl nitrate solution analyzed by IDMS, whereas Fig. 20 shows results from the same laboratory for the analysis of the same solution by a different method (XRF). The figures provide a visual display of long-term performance.

Figure 19

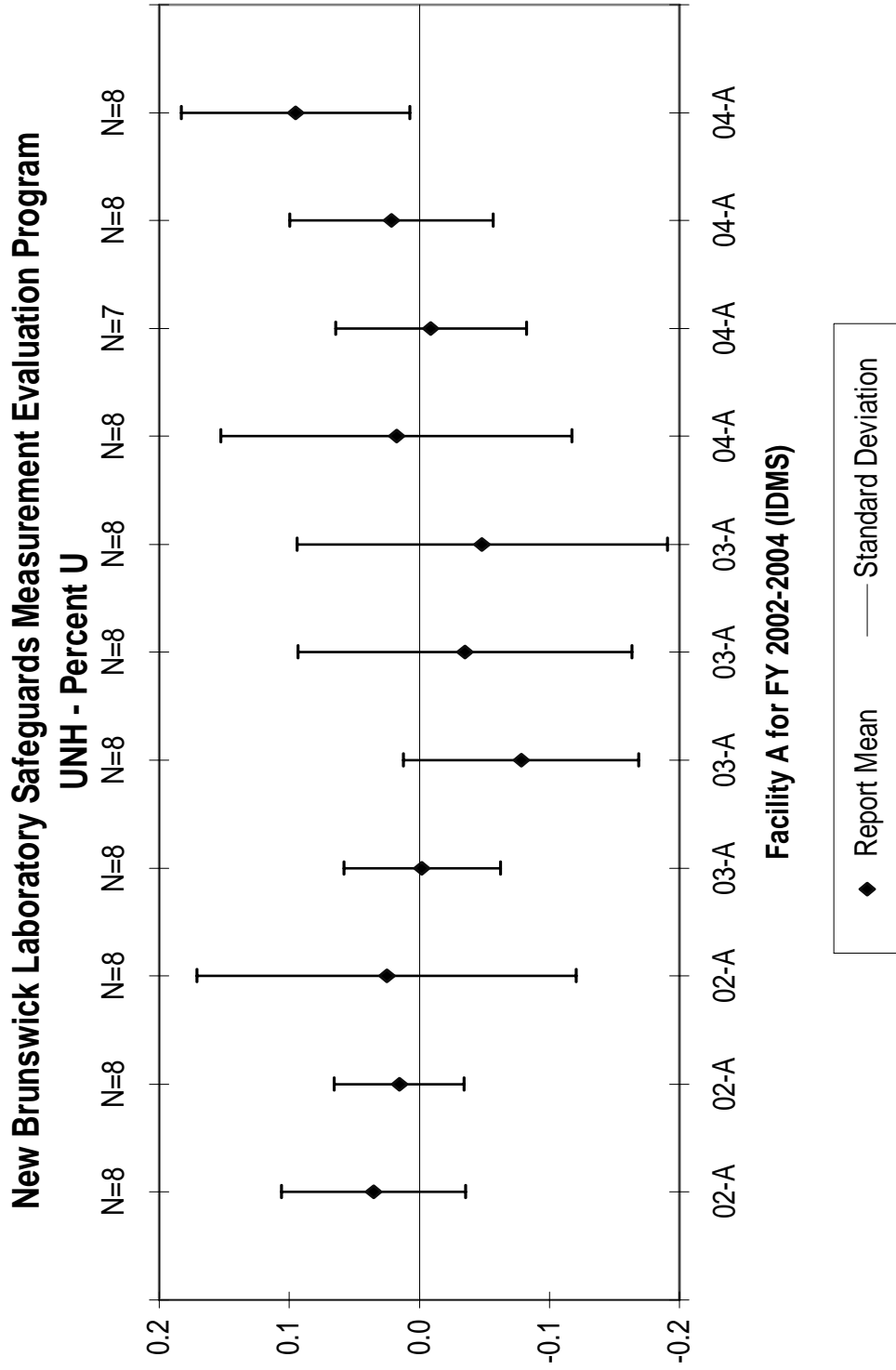




Figure 21

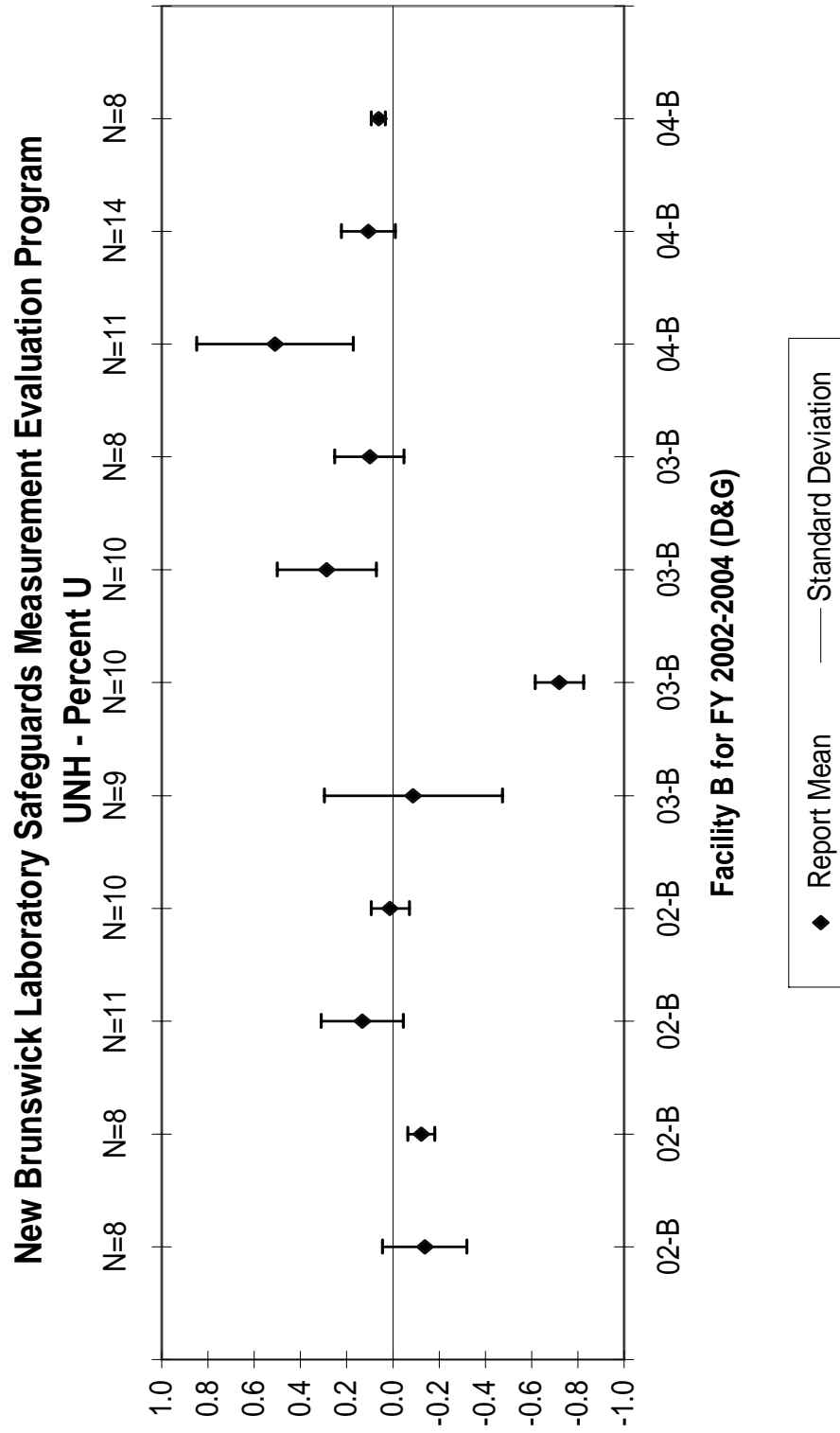
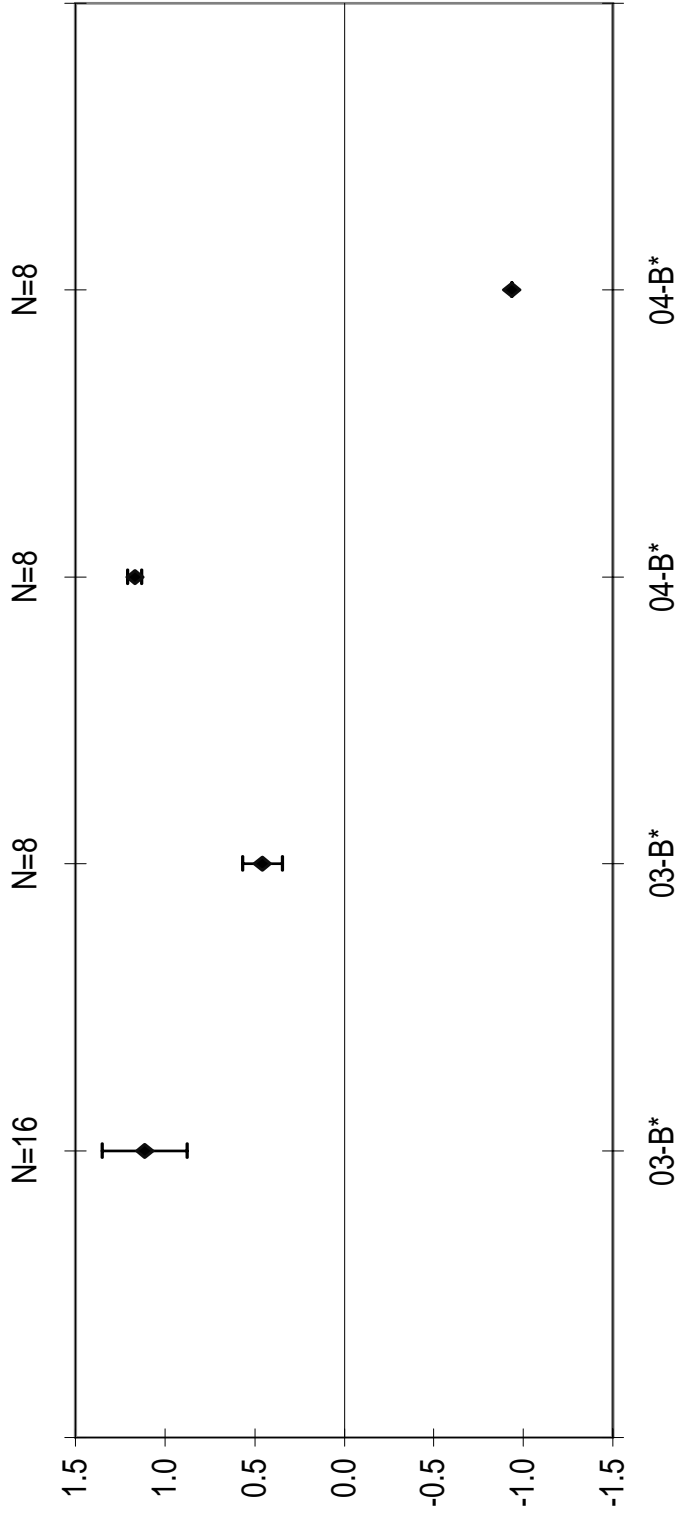




Figure 22

New Brunswick Laboratory Safeguards Measurement Evaluation Program

UNH - Percent U



Facility B for FY 2002-2004 (IDMS)

◆ Report Mean    — Standard Deviation

Figure 23

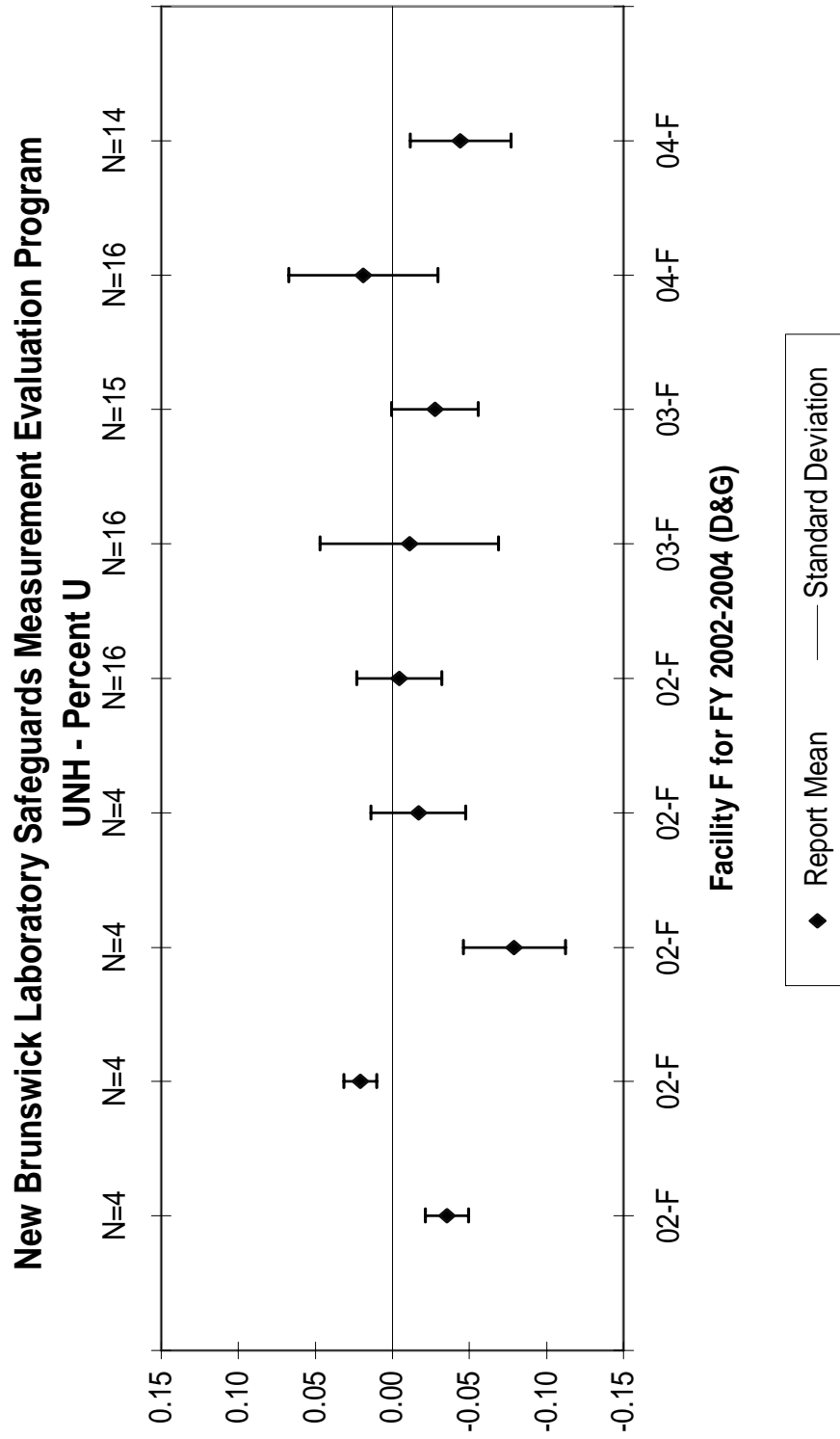


Figure 24

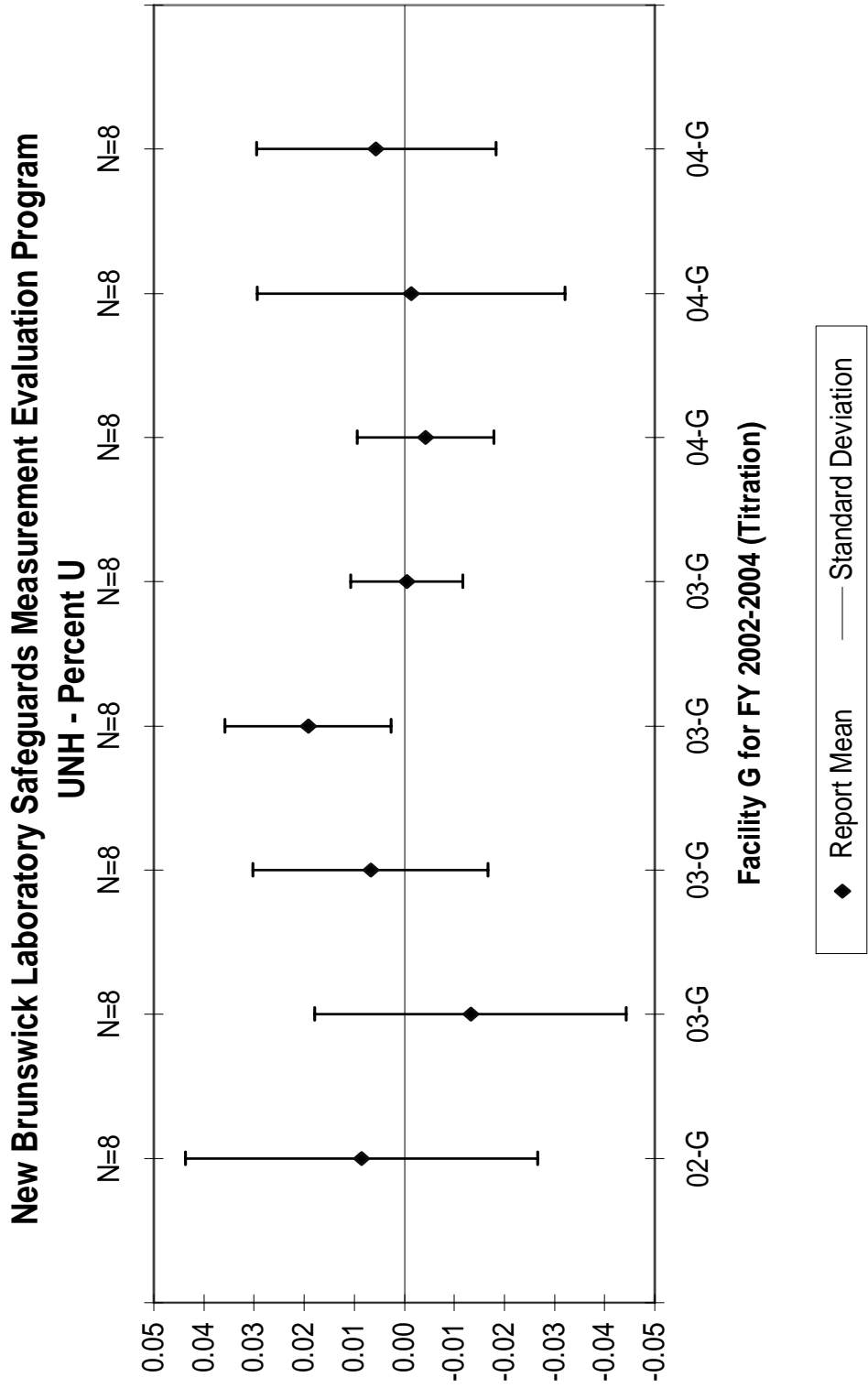


Figure 25

New Brunswick Laboratory Safeguards Measurement Evaluation Program

UNH - Percent U

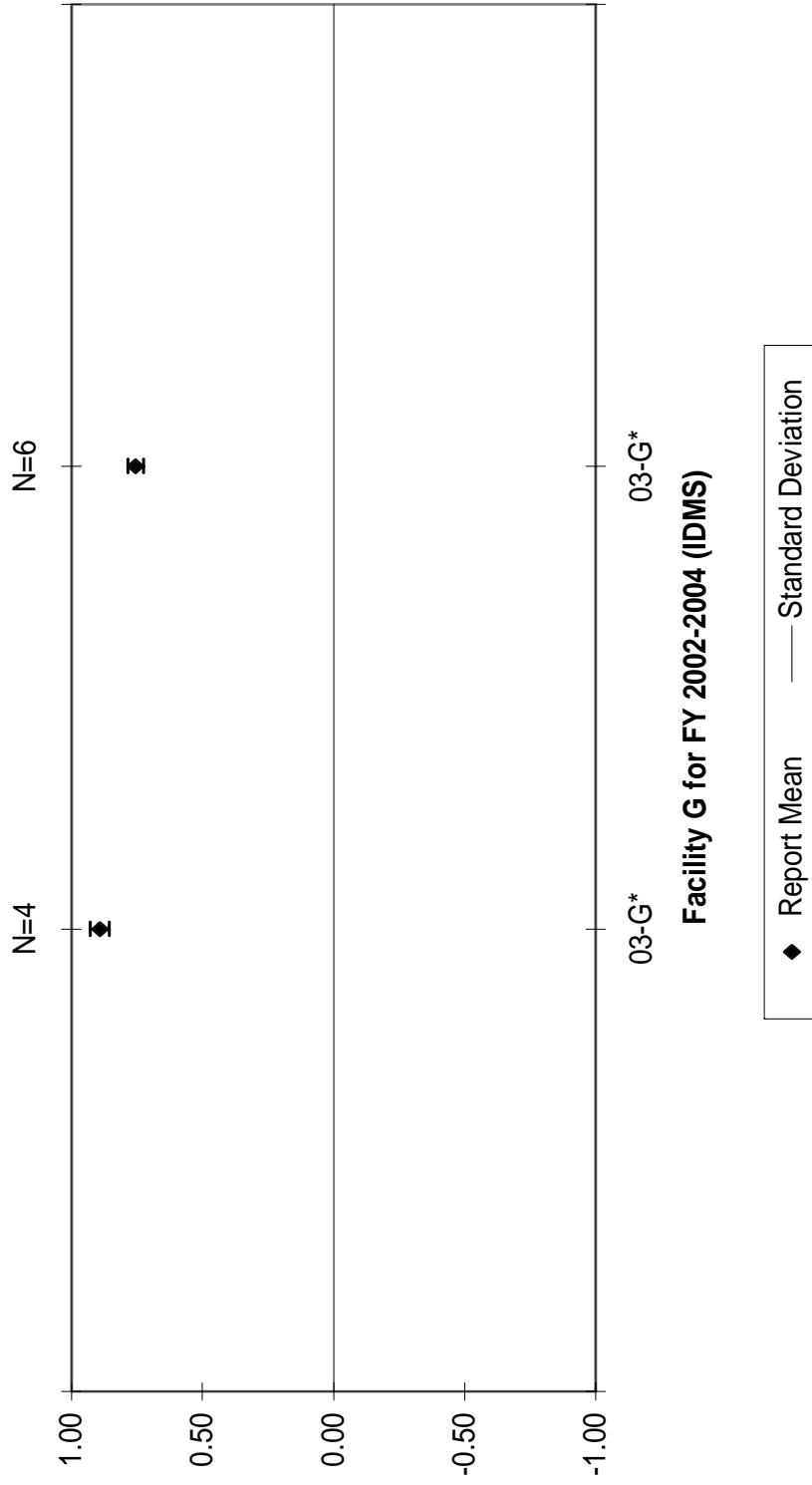


Figure 26

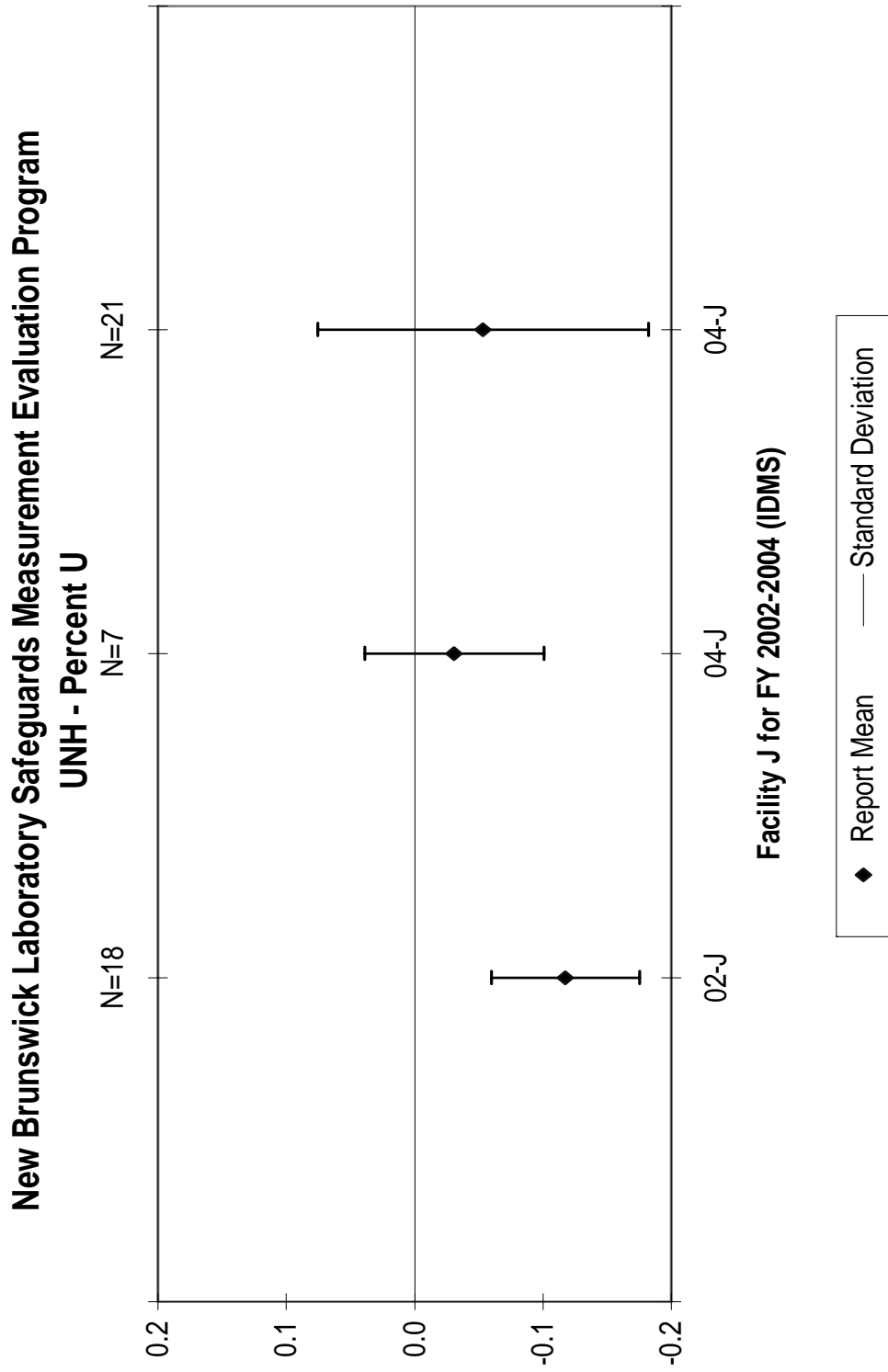
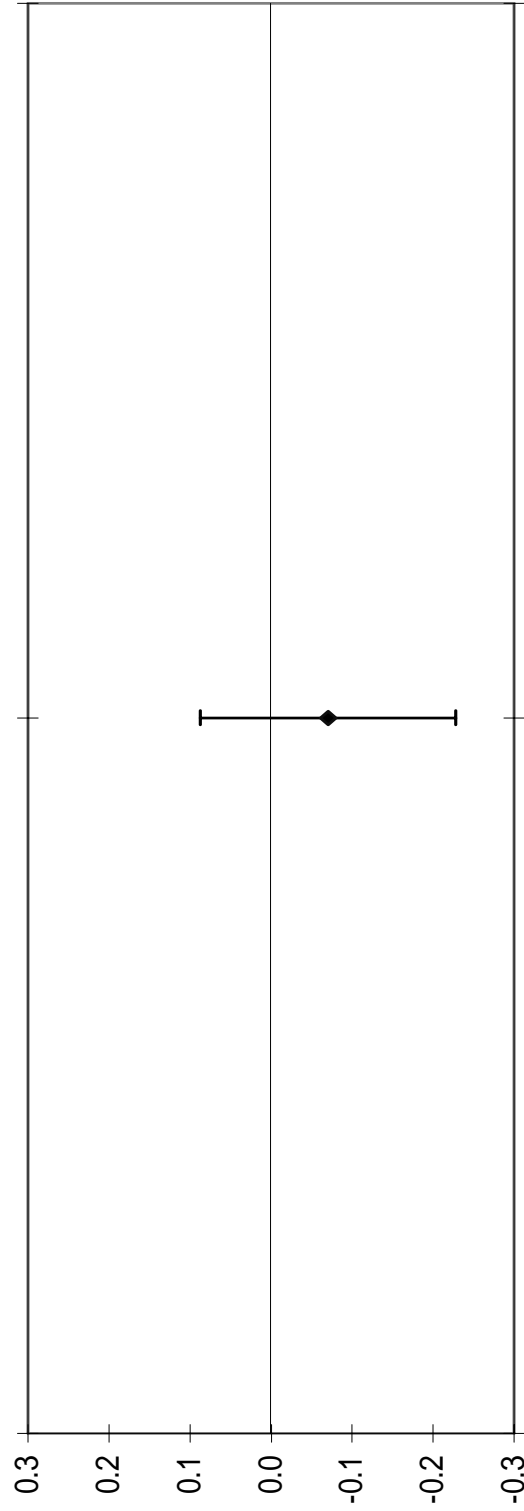


Figure 27

**New Brunswick Laboratory Safeguards Measurement Evaluation Program**

**UNH - Percent U**

N=12



03-U

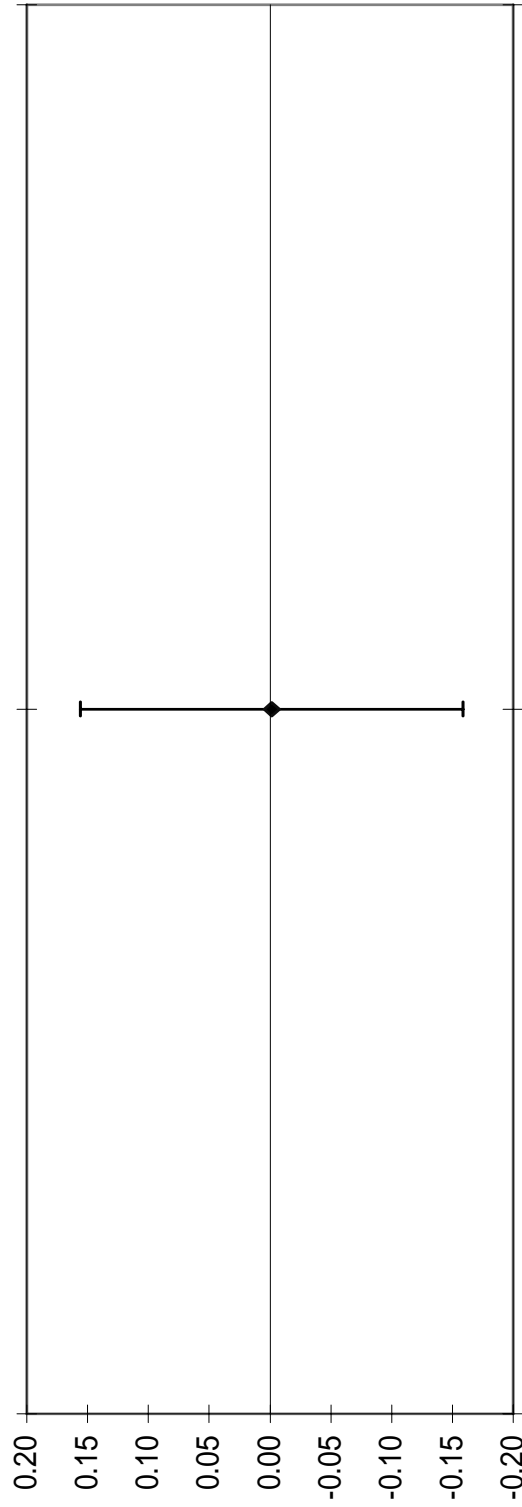
**Facility U for FY 2002-2004 (D&G)**



Figure 28

**New Brunswick Laboratory Safeguards Measurement Evaluation Program**

**UO2 - Percent U**  
N=16



03-BE

**Facility BE for FY 2002-2004 (ICP-MS)**



Figure 29

New Brunswick Laboratory Safeguards Measurement Evaluation Program

UO2 - Percent U - Davies-Gray Titration

N=29

N=30

N=15

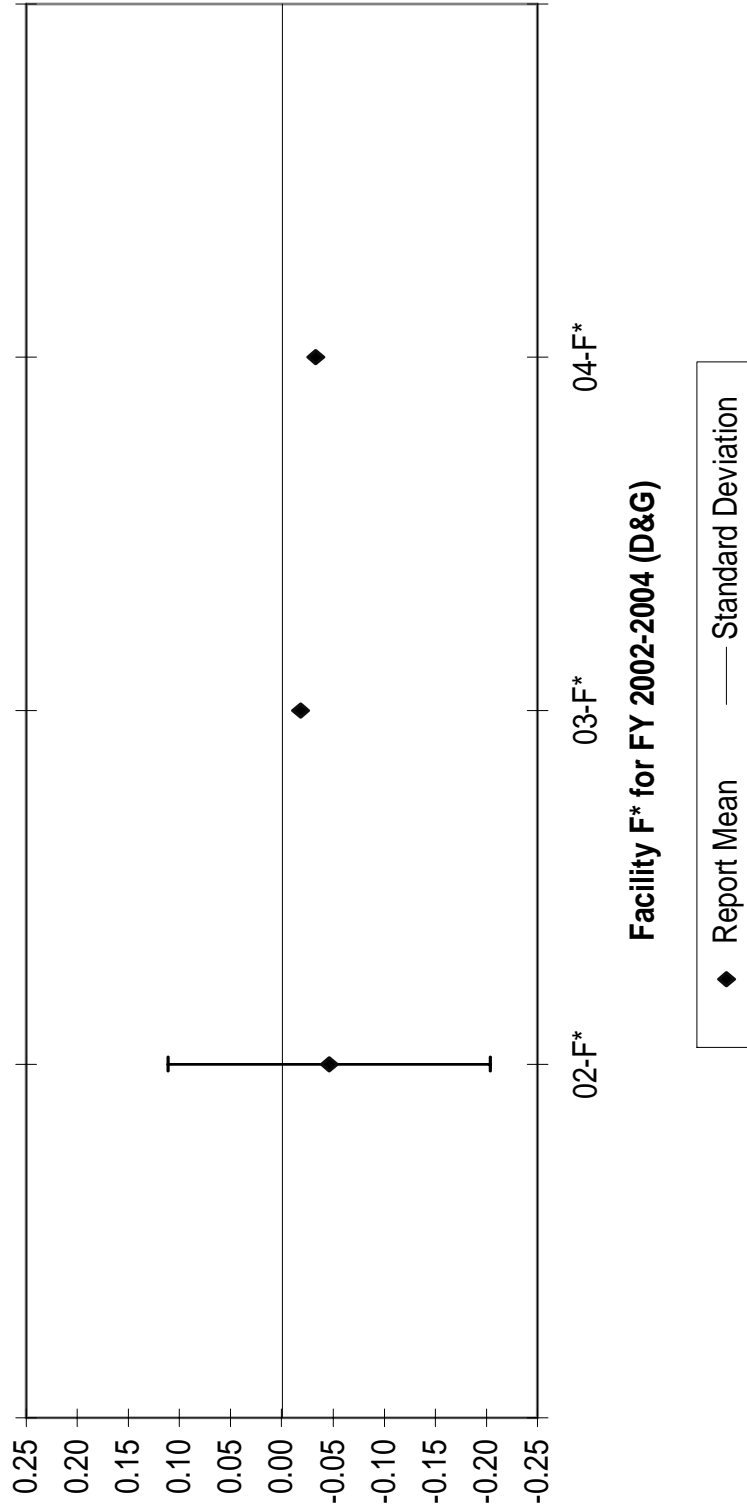






Figure 31

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
UO2 Pellet - Percent U by Davies-Gray Titration

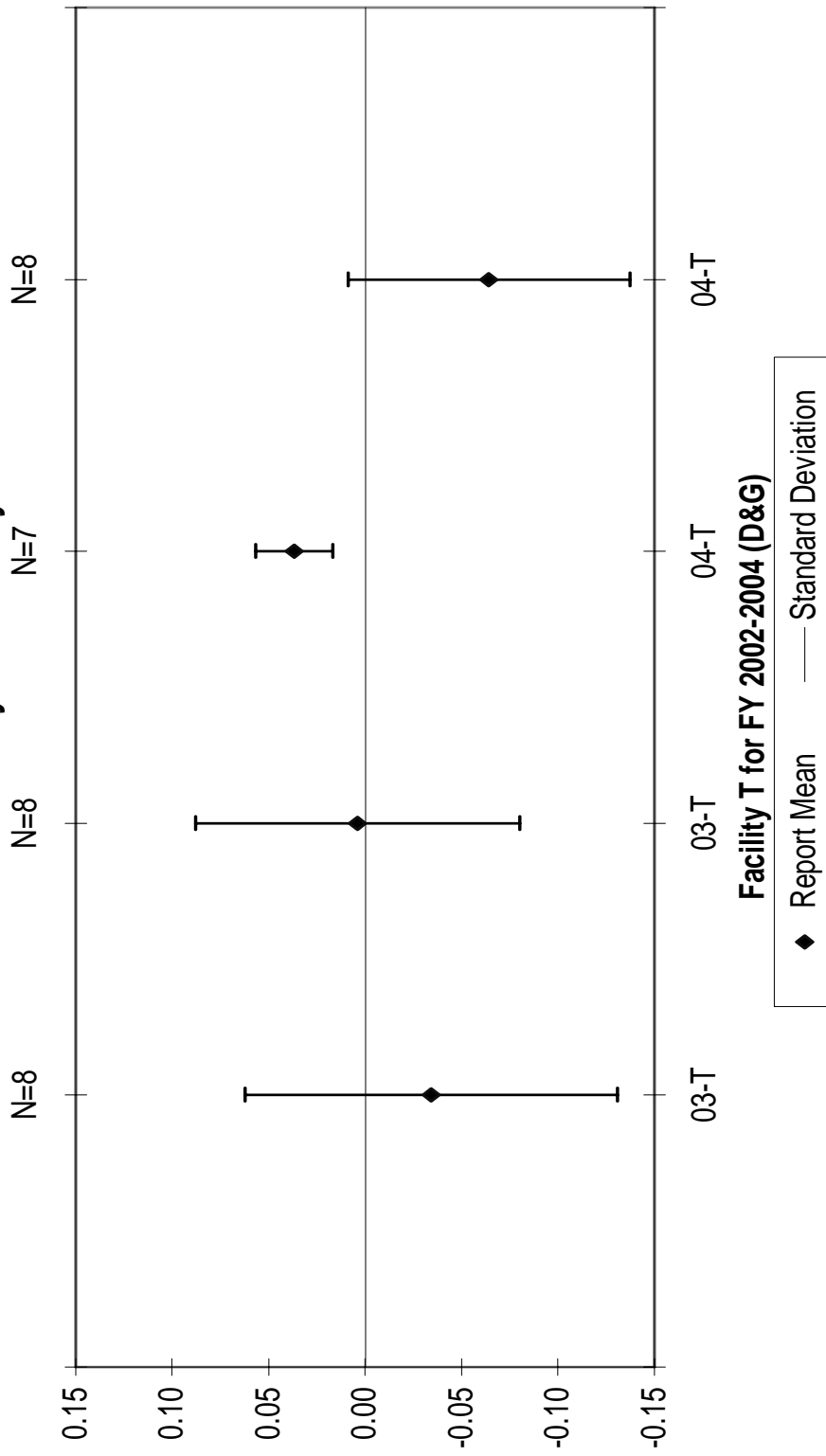


Figure 32

New Brunswick Laboratory Safeguards Measurement Evaluation Program

UO3 Pellet - Percent U

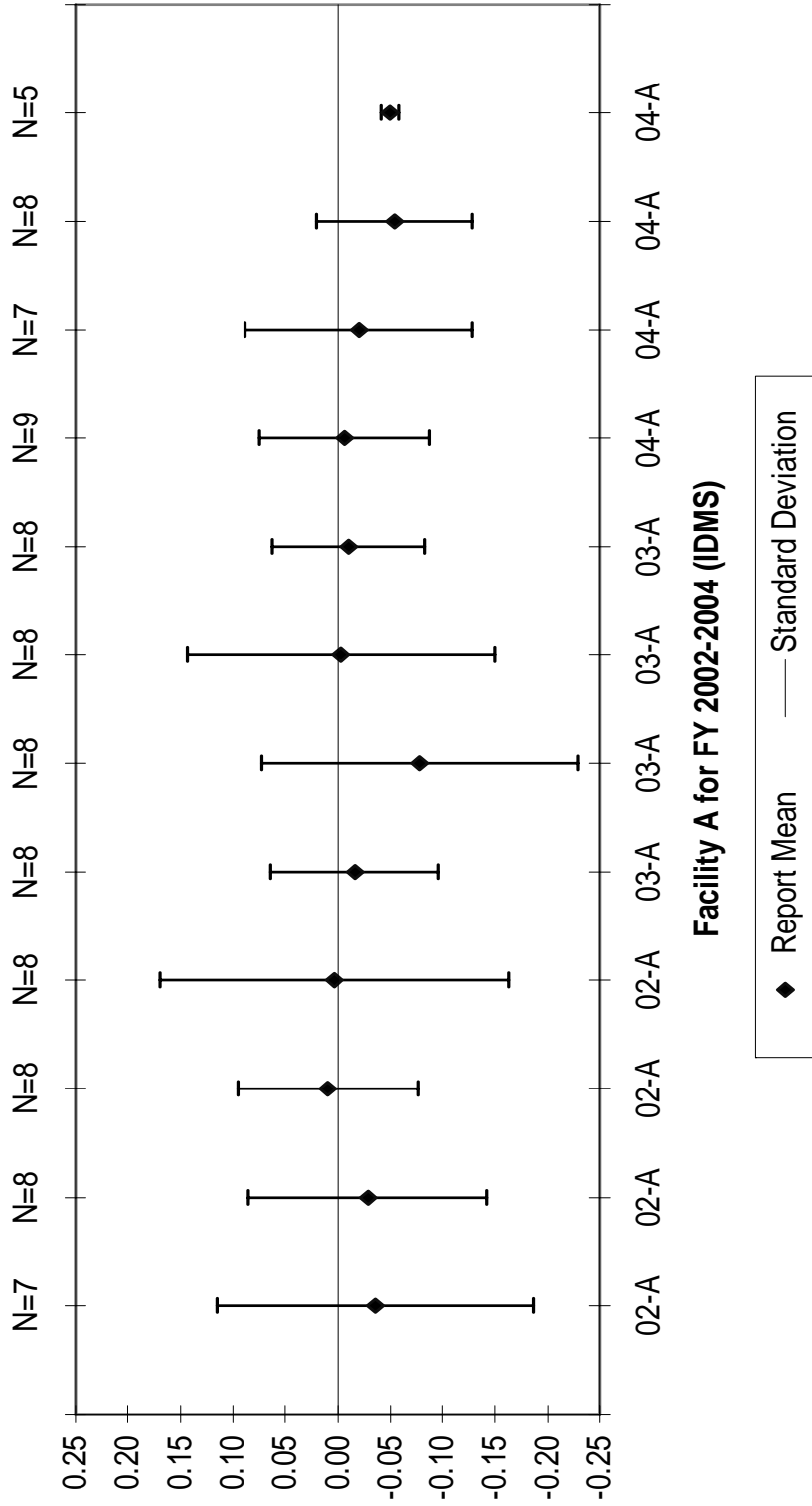
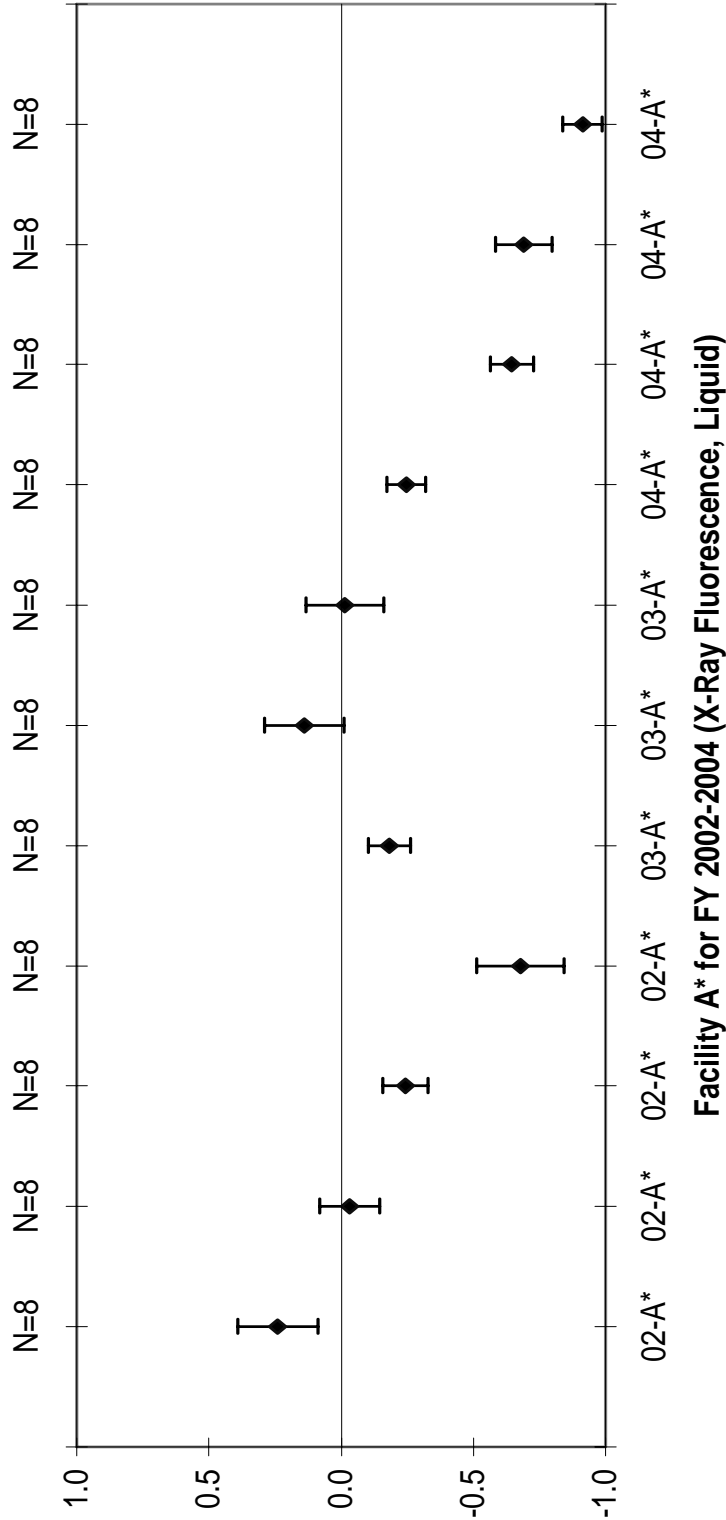


Figure 33

New Brunswick Laboratory Safeguards Measurement Evaluation Program

UO3 Pellet - Percent U

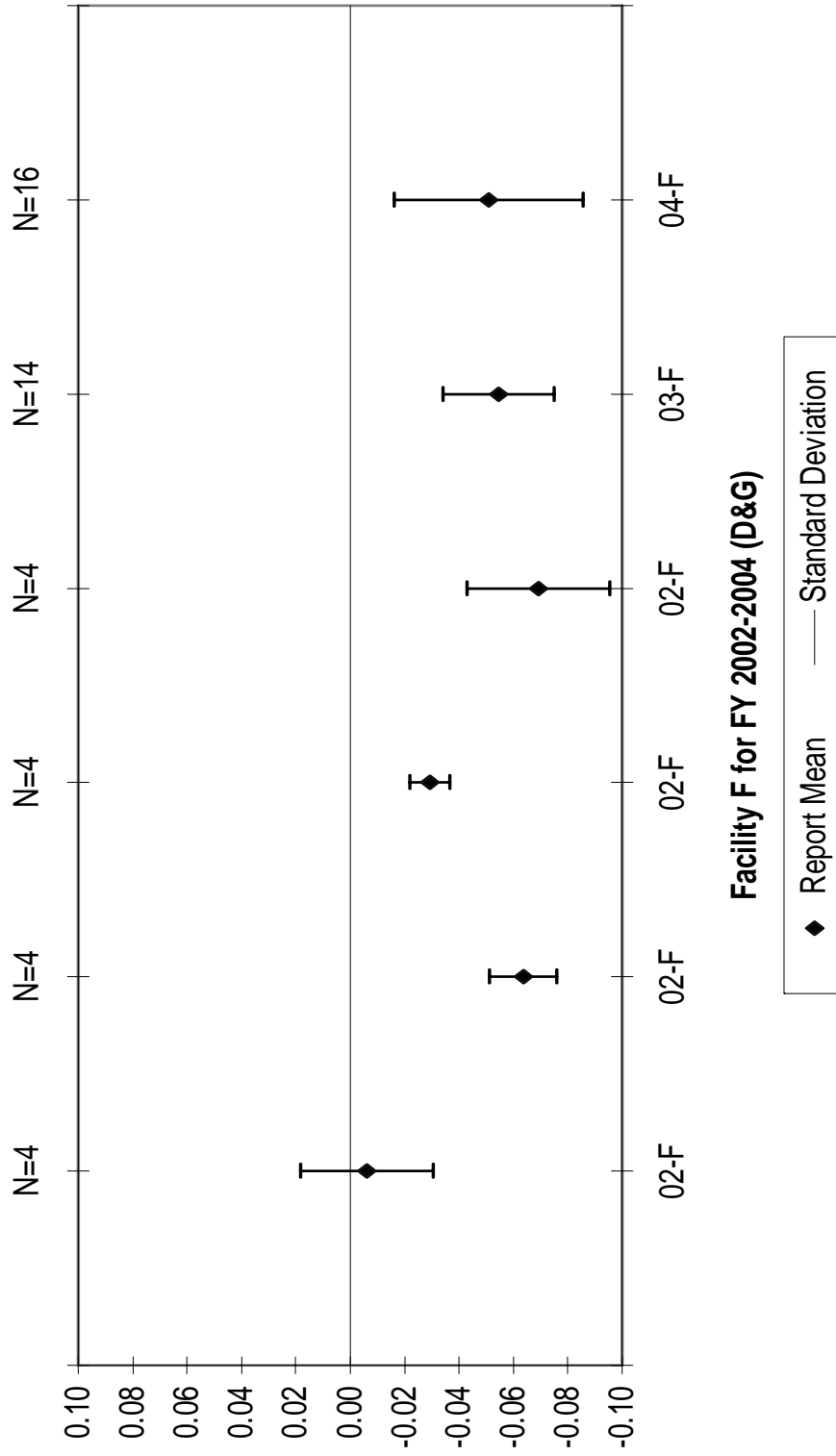


◆ Report Mean  
 — Standard Deviation



Figure 35

New Brunswick Laboratory Safeguards Measurement Evaluation Program  
UO3 Pellet - Percent U by Davies-Gray Titration



## APPENDICES

Appendix A: Uranium Assay Results

Appendix B: Uranium Isotopic Results

Appendix C: Plutonium Assay Results

Appendix D: <sup>239</sup>Pu Isotopic Results

Appendix E: <sup>240</sup>Pu Isotopic Results

### Key to symbols in the tables in the appendices

#### Material Type Symbols

UNH	Uranyl Nitrate Solution
UO <sub>2</sub>	Uranium Dioxide Pellet
HEU	Uranium Enrichment (High)
LEU	Uranium Enrichment (Low)
PU	Dried Plutonium Sulfate
PUXXX	Plutonium Isotope
UO <sub>3</sub>	Uranium Trioxide
UF <sub>6</sub>	Uranium Hexafluoride

#### Method Type Symbols

IDMS	Isotope Dilution Mass Spectrometry
XRFL	X-Ray Fluorescence - Liquid
XRFS	X-Ray Fluorescence - Solid
DG	Davies-Gray Titration
Ceric	Ceric Titration
TIMS	Thermal Ionization Mass Spectrometry
HPT	High Precision Titration
ICP-MS	ICP Mass Spectrometry





## Appendix A: Uranium Assay Results

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UNH	DG	B	1/9/04	1.0057	0.515	4749
UNH	DG	B	1/9/04	1.0069	0.635	4749
UNH	DG	B	1/10/04	1.0024	0.185	4515
UNH	DG	B	1/10/04	1.0033	0.275	4515
UNH	DG	B	1/13/04	1.0136	1.304	849
UNH	DG	B	1/13/04	1.0065	0.595	849
UNH	DG	B	1/9/04	1.0104	0.635	4749
UNH	DG	B	1/10/04	1.0048	0.078	4515
UNH	DG	B	1/10/04	1.0059	0.187	4515
UNH	DG	B	1/13/04	1.0092	0.516	848
UNH	DG	B	1/13/04	1.0109	0.685	849
UNH	DG	B	3/20/04	1.0049	0.088	6219
UNH	DG	B	3/20/04	1.0062	0.217	6219
UNH	DG	B	3/21/04	1.0043	0.028	6219
UNH	DG	B	3/21/04	1.0041	0.008	6219
UNH	DG	B	3/23/04	1.0052	0.118	6861
UNH	DG	B	3/23/04	1.0079	0.386	6861
UNH	DG	B	3/24/04	1.0059	0.187	6861
UNH	DG	B	3/24/04	1.006	0.197	6861
UNH	DG	B	3/21/04	1.0028	0.050	6219
UNH	DG	B	3/21/04	1.0021	-0.020	6219
UNH	DG	B	3/23/04	1.0022	-0.010	6219
UNH	DG	B	3/23/04	1.0021	-0.020	6219
UNH	DG	B	3/23/04	1.0042	0.190	6861
UNH	DG	B	3/23/04	1.003	0.070	6861
UNH	DG	B	7/10/04	1.003	0.070	6219
UNH	DG	B	7/10/04	1.0032	0.090	6219
UNH	DG	B	7/11/04	1.0026	0.030	6219
UNH	DG	B	7/11/04	1.0033	0.100	6219
UNH	DG	B	7/10/04	1.00097	0.042	6219
UNH	DG	B	7/10/04	1.00104	0.049	6219
UNH	DG	B	7/11/04	1.0008	0.025	6219
UNH	DG	B	7/11/04	1.0015	0.095	6219
UNH	DG	F	4/14/04	1.004	-0.002	164
UNH	DG	F	4/14/04	1.0041	0.008	164
UNH	DG	F	4/16/04	1.0035	-0.052	164
UNH	DG	F	4/16/04	1.0036	-0.042	164
UNH	DG	F	4/15/04	1.0046	0.058	197
UNH	DG	F	4/15/04	1.0044	0.038	197
UNH	DG	F	4/22/04	1.0046	0.058	197
UNH	DG	F	4/22/04	1.0047	0.068	197
UNH	DG	F	4/14/04	1.001	0.045	164
UNH	DG	F	4/14/04	1.0013	0.075	164
UNH	DG	F	4/16/04	1.0002	-0.035	164
UNH	DG	F	4/16/04	0.9997	-0.085	164
UNH	DG	F	4/15/04	1.0009	0.035	197
UNH	DG	F	4/15/04	1.0009	0.035	197
UNH	DG	F	4/22/04	1.0011	0.055	197
UNH	DG	F	4/22/04	1.001	0.045	197

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UNH	DG	F	8/19/04	1.0018	-0.050	164
UNH	DG	F	8/19/04	1.0016	-0.070	164
UNH	DG	F	8/20/04	1.0025	0.020	164
UNH	DG	F	9/8/04	1.0014	-0.090	231
UNH	DG	F	9/8/04	1.0016	-0.070	231
UNH	DG	F	9/9/04	1.0018	-0.050	231
UNH	DG	F	8/19/04	1.0004	-0.015	164
UNH	DG	F	8/19/04	1.0004	-0.015	164
UNH	DG	F	8/20/04	1.0004	-0.015	164
UNH	DG	F	8/20/04	1.0004	-0.015	164
UNH	DG	F	9/8/04	1	-0.055	231
UNH	DG	F	9/8/04	0.9997	-0.085	231
UNH	DG	F	9/9/04	1.0002	-0.035	231
UNH	DG	F	9/9/04	0.9998	-0.075	231
UNH	DG	G	12/15/03	1.00391	-0.011	
UNH	DG	G	12/15/03	1.00395	-0.007	
UNH	DG	G	12/16/03	1.00415	0.013	
UNH	DG	G	12/16/03	1.00399	-0.003	
UNH	DG	G	12/15/03	1.00238	0.008	
UNH	DG	G	12/15/03	1.00204	-0.026	
UNH	DG	G	12/16/03	1.00239	0.009	
UNH	DG	G	12/16/03	1.00213	-0.017	
UNH	DG	G	6/15/04	1.00079	0.024	
UNH	DG	G	6/15/04	1.0005	-0.005	
UNH	DG	G	6/16/04	1.00017	-0.038	
UNH	DG	G	6/16/04	1.00015	-0.040	
UNH	DG	G	6/15/04	1.00246	0.016	
UNH	DG	G	6/15/04	1.00212	-0.018	
UNH	DG	G	6/16/04	1.00231	0.001	
UNH	DG	G	6/16/04	1.00279	0.049	
UNH	DG	G	3/22/04	1.00392	-0.010	
UNH	DG	G	3/22/04	1.00366	-0.036	
UNH	DG	G	3/23/04	1.00438	0.036	
UNH	DG	G	3/23/04	1.00413	0.011	
UNH	DG	G	3/22/04	1.00246	0.016	
UNH	DG	G	3/22/04	1.00216	-0.014	
UNH	DG	G	3/23/04	1.00259	0.029	
UNH	DG	G	3/23/04	1.00243	0.013	

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UNH	IDMS	A	10/30/03	1.0049	0.088	DLB/JM
UNH	IDMS	A	10/30/03	1.0069	0.287	DLB/JM
UNH	IDMS	A	10/31/03	1.0029	-0.112	WJS/JM
UNH	IDMS	A	10/31/03	1.0029	-0.112	WJS/JM
UNH	IDMS	A	10/30/03	1.0033	0.100	DLB/JM
UNH	IDMS	A	10/30/03	1.0017	-0.060	DLB/JM
UNH	IDMS	A	10/31/03	1.0021	-0.020	RAG/JM
UNH	IDMS	A	10/31/03	1.002	-0.030	RAG/JM
UNH	IDMS	A	2/27/04	1.0003	-0.025	DLB/JM
UNH	IDMS	A	3/3/04	1.0006	0.005	WJS/JM
UNH	IDMS	A	3/3/04	1.0001	-0.045	WJS/JM
UNH	IDMS	A	2/27/04	1.003	-0.102	DLB/JM
UNH	IDMS	A	2/27/04	1.0035	-0.052	DLB/JM
UNH	IDMS	A	3/3/04	1.0053	0.127	WJS/JM
UNH	IDMS	A	3/3/04	1.0043	0.028	WJS/JM
UNH	IDMS	A	8/26/04	1.0032	-0.082	WJS/JM
UNH	IDMS	A	8/26/04	1.0044	0.038	WJS/JM
UNH	IDMS	A	8/27/04	1.0056	0.157	DLB/JM
UNH	IDMS	A	8/27/04	1.0042	0.018	DLB/JM
UNH	IDMS	A	8/26/04	1.001	0.045	WJS/JM
UNH	IDMS	A	8/26/04	1.0001	-0.045	WJS/JM
UNH	IDMS	A	8/27/04	1.0014	0.085	DLB/JM
UNH	IDMS	A	8/27/04	1.0001	-0.045	DLB/JM
UNH	IDMS	A	4/29/04	1.0023	0.000	DLB/JM
UNH	IDMS	A	4/29/04	1.0034	0.110	DLB/JM
UNH	IDMS	A	4/30/04	1.002	-0.030	WJS/JM
UNH	IDMS	A	4/30/04	1.0045	0.219	WJS/JM
UNH	IDMS	A	4/30/04	1.0026	0.205	DLB/JM
UNH	IDMS	A	4/30/04	1.0017	0.115	DLB/JM
UNH	IDMS	A	5/3/04	1.0013	0.075	WJS/JM
UNH	IDMS	A	5/3/04	1.0012	0.065	WJS/JM
UNH	IDMS	B	1/24/04	0.4673	0.423	NSH
UNH	IDMS	B	1/24/04	0.4672	0.402	NSH
UNH	IDMS	B	1/25/04	0.4689	0.767	CPT
UNH	IDMS	B	1/25/04	0.4692	0.832	CPT
UNH	IDMS	B	1/24/04	0.4744	2.471	NSH
UNH	IDMS	B	1/24/04	0.4743	2.449	NSH
UNH	IDMS	B	1/25/04	0.4677	1.024	CPT
UNH	IDMS	B	1/25/04	0.4675	0.981	CPT
UNH	IDMS	B	7/2/04	0.4674	-0.579	HCH
UNH	IDMS	B	7/2/04	0.4704	0.060	HCH
UNH	IDMS	B	7/5/04	0.4686	-0.323	MDM
UNH	IDMS	B	7/5/04	0.4687	-0.302	MDM
UNH	IDMS	B	7/2/04	0.4633	-1.451	HCH
UNH	IDMS	B	7/2/04	0.4625	-1.621	HCH
UNH	IDMS	B	7/5/04	0.4621	-1.706	MDM
UNH	IDMS	B	7/5/04	0.4627	-1.578	MDM
UNH	IDMS	G	6/29/04	0.47321	0.657	
UNH	IDMS	G	6/29/04	0.47473	0.981	
UNH	IDMS	G	6/29/04	0.68747	0.845	
UNH	IDMS	G	6/29/04	0.68483	0.458	
UNH	IDMS	G	6/29/04	0.68799	0.921	
UNH	IDMS	G	6/29/04	0.68626	0.667	

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UNH	IDMS	J	10/23/03	0.46987	-0.053	U115
UNH	IDMS	J	10/23/03	0.4702	0.017	U114
UNH	IDMS	J	10/17/03	0.46486	-0.101	U118
UNH	IDMS	J	10/23/03	0.46548	0.032	U118
UNH	IDMS	J	1/14/04	0.68227	0.082	U116
UNH	IDMS	J	1/14/04	0.68089	-0.120	U113
UNH	IDMS	J	1/19/04	0.68184	0.019	U112
UNH	IDMS	J	1/19/04	0.46967	-0.096	U114
UNH	IDMS	J	1/19/04	0.4685	-0.345	U115
UNH	IDMS	J	2/6/04	0.68254	0.122	U117
UNH	IDMS	J	2/6/04	0.68294	0.180	U116
UNH	IDMS	J	2/13/04	0.68239	0.100	U116
UNH	IDMS	J	2/13/04	0.68214	0.063	U117
UNH	IDMS	J	2/6/04	0.68226	0.081	U112
UNH	IDMS	J	2/6/04	0.68199	0.041	U113
UNH	IDMS	J	2/13/04	0.68129	-0.062	U113
UNH	IDMS	J	2/13/04	0.68092	-0.116	U112
UNH	IDMS	J	2/6/04	0.46926	-0.183	U115
UNH	IDMS	J	2/6/04	0.46952	-0.128	U114
UNH	IDMS	J	2/13/04	0.46977	-0.074	U114
UNH	IDMS	J	2/13/04	0.4697	-0.089	U115
UNH	IDMS	J	2/6/04	0.46454	-0.170	U119
UNH	IDMS	J	2/6/04	0.46447	-0.185	U118
UNH	IDMS	J	2/13/04	0.46465	-0.146	U118
UNH	IDMS	J	2/13/04	0.46489	-0.095	U119

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UNH	XRFL	A	11/10/03	1.002	-0.030	MER/RBD
UNH	XRFL	A	11/10/03	1.003	0.070	MER/RBD
UNH	XRFL	A	12/4/03	1	-0.229	MER/RBD
UNH	XRFL	A	12/4/03	1.005	0.269	MER/RBD
UNH	XRFL	A	11/10/03	1.001	0.045	MER/RBD
UNH	XRFL	A	11/10/03	1.003	0.245	MER/RBD
UNH	XRFL	A	12/4/03	1.001	0.045	MER/RBD
UNH	XRFL	A	12/4/03	1.001	0.045	MER/RBD
UNH	XRFL	A	3/1/04	1.001	-0.301	MER/RDB
UNH	XRFL	A	3/1/04	1.002	-0.201	MER/RDB
UNH	XRFL	A	3/4/04	1.004	-0.002	MER/RDB
UNH	XRFL	A	3/4/04	1.005	0.098	MER/RDB
UNH	XRFL	A	3/1/04	1.001	-0.301	MER/RDB
UNH	XRFL	A	3/1/04	1	-0.400	MER/RDB
UNH	XRFL	A	3/4/04	1.001	-0.301	MER/RDB
UNH	XRFL	A	3/4/04	1.005	0.098	MER/RDB
UNH	XRFL	A	7/22/04	1.001	-0.301	MER/RBD
UNH	XRFL	A	8/5/04	1.001	-0.301	MER/RBD
UNH	XRFL	A	8/5/04	1.001	-0.301	MER/RBD
UNH	XRFL	A	8/5/04	0.9991	-0.319	MER/RBD
UNH	XRFL	A	8/5/04	0.9989	-0.339	MER/RBD
UNH	XRFL	A	5/13/04	0.9961	-0.619	MER/RBD
UNH	XRFL	A	5/13/04	0.9979	-0.439	MER/RBD
UNH	XRFL	A	6/1/04	0.9946	-0.768	MER/RBD
UNH	XRFL	A	6/1/04	0.997	-0.529	MER/RBD
UNH	XRFL	A	5/13/04	0.9957	-0.485	MER/RBD
UNH	XRFL	A	5/13/04	0.997	-0.355	MER/RBD
UNH	XRFL	A	6/1/04	0.9935	-0.705	MER/RBD
UNH	XRFL	A	6/1/04	0.9955	-0.505	MER/RBD
UO2	DG	F	6/1/04	88.09	-0.041	164
UO2	DG	F	6/1/04	88.07	-0.070	164
UO2	DG	F	6/3/04	88.15	0.028	164
UO2	DG	F	6/3/04	88.11	-0.017	164
UO2	DG	F	6/1/04	88.08	-0.052	164
UO2	DG	F	6/1/04	88.07	-0.062	164
UO2	DG	F	6/3/04	88.13	0.004	164
UO2	DG	F	6/3/04	88.12	-0.008	164
UO2	DG	F	6/1/04	88.10	-0.037	164
UO2	DG	F	6/1/04	88.10	-0.039	164
UO2	DG	F	6/3/04	88.10	-0.030	164
UO2	DG	F	6/3/04	88.06	-0.077	164
UO2	DG	F	6/1/04	88.09	-0.050	164
UO2	DG	F	6/3/04	88.10	-0.032	164
UO2	DG	F	6/3/04	88.12	-0.014	164

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UO2	DG	T	12/24/03	88.13	0.001	
UO2	DG	T	12/24/03	88.16	0.035	
UO2	DG	T	12/24/03	88.16	0.035	
UO2	DG	T	3/17/04	88.17	0.047	
UO2	DG	T	3/17/04	88.15	0.024	
UO2	DG	T	3/17/04	88.18	0.058	
UO2	DG	T	3/17/04	88.18	0.058	
UO2	DG	T	6/15/04	87.94	-0.214	
UO2	DG	T	6/15/04	88.11	-0.022	
UO2	DG	T	6/15/04	88.14	0.012	
UO2	DG	T	6/15/04	88.08	-0.056	
UO2	DG	T	10/6/04	88.06	-0.078	
UO2	DG	T	10/6/04	88.14	0.012	
UO2	DG	T	10/6/04	88.06	-0.078	
UO2	DG	T	10/6/04	88.05	-0.090	
UO2	HPT	F	8/11/04	88.11	-0.022	025
UO2	HPT	F	8/9/04	88.10	-0.028	025
UO2	HPT	F	8/11/04	88.12	-0.014	025
UO2	HPT	F	8/9/04	88.11	-0.024	025
UO2	HPT	F	8/11/04	88.12	-0.013	025
UO2	HPT	F	8/11/04	88.12	-0.015	025
UO3	DG	F	3/30/04	82.59	-0.096	164
UO3	DG	F	3/30/04	82.57	-0.122	164
UO3	DG	F	3/31/04	82.63	-0.049	164
UO3	DG	F	3/31/04	82.61	-0.077	164
UO3	DG	F	7/1/04	82.64	-0.034	197
UO3	DG	F	7/1/04	82.63	-0.045	197
UO3	DG	F	7/2/04	82.64	-0.037	197
UO3	DG	F	7/2/04	82.66	-0.017	197
UO3	DG	F	3/30/04	82.61	-0.078	164
UO3	DG	F	3/30/04	82.61	-0.070	164
UO3	DG	F	3/31/04	82.61	-0.070	164
UO3	DG	F	3/31/04	82.63	-0.045	164
UO3	DG	F	7/1/04	82.62	-0.061	197
UO3	DG	F	7/1/04	82.66	-0.013	197
UO3	DG	F	7/2/04	82.65	-0.020	197
UO3	DG	F	7/2/04	82.69	0.018	197

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UO3	IDMS	A	11/4/03	82.7	0.035	DLB/JM
UO3	IDMS	A	11/4/03	82.71	0.047	DLB/JM
UO3	IDMS	A	11/3/03	82.6	-0.086	WJS/JM
UO3	IDMS	A	11/3/03	82.68	0.011	WJS/JM
UO3	IDMS	A	11/4/03	82.72	0.059	DLB/JM
UO3	IDMS	A	11/4/03	82.58	-0.110	DLB/JM
UO3	IDMS	A	11/3/03	82.56	-0.134	WJS/JM
UO3	IDMS	A	11/3/03	82.74	0.083	WJS/JM
UO3	IDMS	A	2/27/04	82.7	0.035	DLB/JM
UO3	IDMS	A	2/27/04	82.64	-0.037	DLB/JM
UO3	IDMS	A	3/3/04	82.77	0.120	WJS/JM
UO3	IDMS	A	3/3/04	82.58	-0.110	WJS/JM
UO3	IDMS	A	2/27/04	82.77	0.120	DLB/JM
UO3	IDMS	A	2/27/04	82.53	-0.171	DLB/JM
UO3	IDMS	A	3/3/04	82.65	-0.025	WLS/JM
UO3	IDMS	A	3/3/04	82.64	-0.037	WLS/JM
UO3	IDMS	A	8/26/04	82.6	-0.086	WJS/JM
UO3	IDMS	A	8/26/04	82.55	-0.146	WJS/JM
UO3	IDMS	A	8/30/04	82.69	0.023	BLM/JM
UO3	IDMS	A	8/30/04	82.71	0.047	BLM/JM
UO3	IDMS	A	8/26/04	82.64	-0.037	WJS/JM
UO3	IDMS	A	8/26/04	82.62	-0.062	WJS/JM
UO3	IDMS	A	8/30/04	82.54	-0.158	BLM/JM
UO3	IDMS	A	8/30/04	82.66	-0.013	BLM/JM
UO3	IDMS	A	4/29/04	82.63	-0.050	DLB/JM
UO3	IDMS	A	5/3/04	82.64	-0.037	WJS/JM
UO3	IDMS	A	4/29/04	82.63	-0.050	DLB/JM
UO3	IDMS	A	5/3/04	82.62	-0.062	WJS/JM
UO3	IDMS	A	5/3/04	82.63	-0.050	WJS/JM

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UO3	XRFL	A	11/10/03	82.47	-0.243	MER/RBD
UO3	XRFL	A	11/10/03	82.56	-0.134	MER/RBD
UO3	XRFL	A	12/4/03	82.43	-0.292	MER/RBD
UO3	XRFL	A	12/4/03	82.53	-0.171	MER/RBD
UO3	XRFL	A	11/10/03	82.43	-0.292	MER/RBD
UO3	XRFL	A	11/10/03	82.39	-0.340	MER/RBD
UO3	XRFL	A	12/4/03	82.44	-0.279	MER/RBD
UO3	XRFL	A	12/4/03	82.49	-0.219	MER/RBD
UO3	XRFL	A	3/1/04	81.97	-0.848	MER/RBD
UO3	XRFL	A	3/1/04	82.07	-0.727	MER/RBD
UO3	XRFL	A	3/4/04	81.99	-0.824	MER/RBD
UO3	XRFL	A	3/4/04	82.26	-0.497	MER/RBD
UO3	XRFL	A	3/1/04	82.04	-0.763	MER/RBD
UO3	XRFL	A	3/1/04	82.17	-0.606	MER/RBD
UO3	XRFL	A	3/4/04	82.3	-0.449	MER/RBD
UO3	XRFL	A	3/4/04	82.3	-0.449	MER/RBD
UO3	XRFL	A	7/22/04	81.9	-0.933	MER/RBD
UO3	XRFL	A	7/22/04	82.08	-0.715	MER/RBD
UO3	XRFL	A	8/5/04	82.2	-0.570	MER/RBD
UO3	XRFL	A	8/5/04	82.02	-0.787	MER/RBD
UO3	XRFL	A	7/22/04	82.1	-0.691	MER/RBD
UO3	XRFL	A	7/22/04	82.16	-0.618	MER/RBD
UO3	XRFL	A	8/5/04	82.2	-0.570	MER/RBD
UO3	XRFL	A	8/5/04	82.14	-0.642	MER/RBD
UO3	XRFL	A	5/13/04	81.98	-0.836	MER/RBD
UO3	XRFL	A	5/13/04	82.03	-0.775	MER/RBD
UO3	XRFL	A	6/1/04	81.95	-0.872	MER/RBD
UO3	XRFL	A	6/1/04	81.89	-0.945	MER/RBD
UO3	XRFL	A	5/13/04	81.84	-1.005	MER/RBD
UO3	XRFL	A	5/13/04	82.04	-0.763	MER/RBD
UO3	XRFL	A	6/1/04	81.85	-0.993	MER/RBD
UO3	XRFL	A	6/1/04	81.75	-1.114	MER/RBD



<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
UO3	XRFS	A	11/21/03	82.08	-0.715	MER/RBD
UO3	XRFS	A	11/21/03	82.01	-0.800	MER/RBD
UO3	XRFS	A	12/2/03	82.09	-0.703	MER/RBD
UO3	XRFS	A	12/2/03	82.55	-0.146	MER/RBD
UO3	XRFS	A	11/21/03	82.28	-0.473	MER/RBD
UO3	XRFS	A	11/21/03	82.51	-0.195	MER/RBD
UO3	XRFS	A	12/2/03	82.07	-0.727	MER/RBD
UO3	XRFS	A	12/2/03	81.98	-0.836	MER/RBD
UO3	XRFS	A	2/27/04	82.45	-0.267	MER/RBD
UO3	XRFS	A	2/27/04	82.21	-0.558	MER/RBD
UO3	XRFS	A	3/10/04	82.35	-0.388	MER/RBD
UO3	XRFS	A	2/27/04	82.24	-0.521	MER/RBD
UO3	XRFS	A	2/27/04	82.14	-0.642	MER/RBD
UO3	XRFS	A	3/10/04	82.32	-0.425	MER/RBD
UO3	XRFS	A	3/10/04	82.32	-0.425	MER/RBD
UO3	XRFS	A	7/20/04	81.94	-0.884	MER/RBD
UO3	XRFS	A	7/20/04	81.41	-1.525	MER/RBD
UO3	XRFS	A	7/27/04	81.4	-1.537	MER/RBD
UO3	XRFS	A	7/27/04	81.82	-1.029	MER/RBD
UO3	XRFS	A	7/20/04	81.39	-1.550	MER/RBD
UO3	XRFS	A	7/20/04	81.5	-1.416	MER/RBD
UO3	XRFS	A	7/27/04	81.52	-1.392	MER/RBD
UO3	XRFS	A	7/27/04	81.65	-1.235	MER/RBD
UO3	XRFS	A	5/7/04	81.93	-0.896	MER/RBD
UO3	XRFS	A	5/7/04	81.96	-0.860	MER/RBD
UO3	XRFS	A	5/21/04	82.1	-0.691	MER/RBD
UO3	XRFS	A	5/21/04	82.07	-0.727	MER/RBD
UO3	XRFS	A	5/7/04	82.02	-0.787	MER/RBD
UO3	XRFS	A	5/7/04	81.88	-0.957	MER/RBD
UO3	XRFS	A	5/21/04	81.94	-0.884	MER/RBD
UO3	XRFS	A	5/21/04	82.05	-0.751	MER/RBD



## Appendix B: Uranium Isotopic Results

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis</u>	<u>Reported</u>	<u>% RD</u>	<u>Analyst</u>
			<u>Date</u>	<u>Result</u>		
HEU	TIMS	A	10/30/03	89.671	-0.009	LHC/JM
HEU	TIMS	A	10/30/03	89.683	0.005	LHC/JM
HEU	TIMS	A	11/7/03	89.674	-0.005	LHC/JM
HEU	TIMS	A	11/7/03	89.674	-0.005	LHC/JM
HEU	TIMS	A	10/30/03	90.338	0.001	LHC/JM
HEU	TIMS	A	10/30/03	90.343	0.006	LHC/JM
HEU	TIMS	A	11/7/03	90.339	0.002	LHC/JM
HEU	TIMS	A	11/7/03	90.334	-0.004	LHC/JM
HEU	TIMS	A	2/27/04	51.356	0.061	LCH/JM
HEU	TIMS	A	2/27/04	51.336	0.022	LCH/JM
HEU	TIMS	A	3/2/04	51.332	0.015	LCH/JM
HEU	TIMS	A	3/2/04	51.326	0.003	LCH/JM
HEU	TIMS	A	2/27/04	51.349	0.048	LCH/JM
HEU	TIMS	A	2/27/04	51.347	0.044	LCH/JM
HEU	TIMS	A	3/2/04	51.317	-0.015	LCH/JM
HEU	TIMS	A	3/2/04	51.318	-0.013	LCH/JM
HEU	TIMS	A	4/29/04	89.677	-0.002	LHC/JM
HEU	TIMS	A	4/29/04	89.675	-0.004	LHC/JM
HEU	TIMS	A	4/30/04	89.685	0.007	LHC/JM
HEU	TIMS	A	4/30/04	89.682	0.004	LHC/JM
HEU	TIMS	A	4/29/04	90.333	-0.005	LHC/JM
HEU	TIMS	A	4/29/04	90.344	0.008	LHC/JM
HEU	TIMS	A	4/30/04	90.350	0.014	LHC/JM
HEU	TIMS	A	4/30/04	90.334	-0.004	LHC/JM
HEU	TIMS	B	1/24/04	90.3214	-0.017	NSH
HEU	TIMS	B	1/24/04	90.3225	-0.016	NSH
HEU	TIMS	B	1/25/04	90.3235	-0.015	CPT
HEU	TIMS	B	1/25/04	90.3216	-0.017	CPT
HEU	TIMS	B	1/24/04	89.8931	0.002	NSH
HEU	TIMS	B	1/24/04	89.8863	-0.005	NSH
HEU	TIMS	B	1/25/04	89.8884	-0.003	CPT
HEU	TIMS	B	1/25/04	89.8940	0.003	CPT
HEU	TIMS	B	7/2/04	89.6888	0.011	HCH
HEU	TIMS	B	7/2/04	89.6821	0.004	HCH
HEU	TIMS	B	7/5/04	89.7067	0.031	MDM
HEU	TIMS	B	7/5/04	89.6947	0.018	MDM
HEU	TIMS	B	7/2/04	89.9132	0.025	HCH
HEU	TIMS	B	7/2/04	89.9151	0.027	HCH
HEU	TIMS	B	7/5/04	89.9159	0.028	MDM
HEU	TIMS	B	7/5/04	89.9101	0.021	MDM
HEU	TIMS	B	10/5/04	51.3604	0.070	CPT
HEU	TIMS	B	10/5/04	51.3590	0.067	CPT
HEU	TIMS	B	9/29/04	51.3444	0.039	JLB
HEU	TIMS	B	9/29/04	51.3657	0.080	JLB
HEU	TIMS	B	10/5/04	51.3463	0.042	CPT
HEU	TIMS	B	10/5/04	51.3518	0.053	CPT
HEU	TIMS	B	9/29/04	51.3675	0.084	JLB
HEU	TIMS	B	9/29/04	51.3691	0.087	JLB

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
HEU	TIMS	F	12/1/03	89.6802	0.002	247
HEU	TIMS	F	12/1/03	89.6806	0.002	247
HEU	TIMS	F	12/1/03	89.6805	0.002	247
HEU	TIMS	F	12/1/03	89.8927	0.002	247
HEU	TIMS	F	12/1/03	89.8922	0.001	247
HEU	TIMS	F	12/1/03	89.8920	0.001	247
HEU	TIMS	F	8/30/04	89.6788	0.000	247
HEU	TIMS	F	8/30/04	89.6790	0.000	247
HEU	TIMS	F	8/30/04	89.6781	-0.001	247
HEU	TIMS	F	8/30/04	90.3375	0.000	247
HEU	TIMS	F	8/30/04	90.3371	0.000	247
HEU	TIMS	F	8/30/04	90.3369	0.000	247
HEU	TIMS	G	4/27/04	89.6831	0.005	
HEU	TIMS	G	4/27/04	89.6832	0.005	
HEU	TIMS	G	4/27/04	89.6788	0.000	
HEU	TIMS	G	4/27/04	89.6796	0.001	
HEU	TIMS	G	4/27/04	90.3408	0.004	
HEU	TIMS	G	4/27/04	90.3418	0.005	
HEU	TIMS	G	4/27/04	90.3400	0.003	
HEU	TIMS	G	4/27/04	90.3400	0.003	
HEU	TIMS	G	4/27/04	51.3398	0.030	
HEU	TIMS	G	4/27/04	51.3391	0.028	
HEU	TIMS	G	4/27/04	51.3379	0.026	
HEU	TIMS	G	4/27/04	51.3361	0.023	
HEU	TIMS	G	4/27/04	51.3390	0.028	
HEU	TIMS	G	4/27/04	51.3382	0.027	
HEU	TIMS	G	4/27/04	51.3385	0.027	
HEU	TIMS	J	1/14/04	51.3268	0.004	U116
HEU	TIMS	J	2/6/04	51.3225	-0.004	U117
HEU	TIMS	J	2/6/04	51.3125	-0.023	U116
HEU	TIMS	J	2/13/04	51.3243	0.000	U116
HEU	TIMS	J	2/13/04	51.3306	0.012	U117
HEU	TIMS	J	10/23/03	51.3127	-0.023	U112
HEU	TIMS	J	1/14/04	51.3386	0.027	U113
HEU	TIMS	J	1/19/04	51.3211	-0.007	U112
HEU	TIMS	J	2/6/04	51.3140	-0.020	U112
HEU	TIMS	J	2/6/04	51.3167	-0.015	U113
HEU	TIMS	J	2/13/04	51.3321	0.015	U113
HEU	TIMS	J	2/13/04	51.3423	0.035	U112
HEU	TIMS	J	10/17/03	89.6737	-0.006	U114
HEU	TIMS	J	10/17/03	89.6742	-0.005	U115
HEU	TIMS	J	10/23/03	89.6717	-0.008	U115
HEU	TIMS	J	10/23/03	89.6655	-0.015	U114
HEU	TIMS	J	1/19/04	89.6798	0.001	U114
HEU	TIMS	J	1/19/04	89.6807	0.002	U115
HEU	TIMS	J	2/6/04	89.6900	0.012	U115
HEU	TIMS	J	2/6/04	89.6842	0.006	U114
HEU	TIMS	J	2/13/04	89.6773	-0.002	U114
HEU	TIMS	J	2/13/04	89.6805	0.002	U115
HEU	TIMS	J	10/17/03	90.3384	0.001	U118
HEU	TIMS	J	10/23/03	90.3200	-0.019	U118
HEU	TIMS	J	2/6/04	90.3501	0.014	U119
HEU	TIMS	J	2/6/04	90.3478	0.012	U118
HEU	TIMS	J	2/13/04	90.3438	0.007	U118
HEU	TIMS	J	2/13/04	90.3421	0.005	U119

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
LEU	TIMS	A	8/25/04	4.392	0.010	WJS/JM
LEU	TIMS	A	8/25/04	4.387	-0.103	WJS/JM
LEU	TIMS	A	8/26/04	4.394	0.056	WJS/JM
LEU	TIMS	A	8/26/04	4.391	-0.012	WJS/JM
LEU	TIMS	A	8/25/04	4.457	-0.063	WJS/JM
LEU	TIMS	A	8/25/04	4.461	0.027	WJS/JM
LEU	TIMS	A	8/26/04	4.467	0.161	WJS/JM
LEU	TIMS	A	8/26/04	4.460	0.004	WJS/JM
LEU	TIMS	B	4/10/04	4.4604	0.000	CDN
LEU	TIMS	B	4/10/04	4.4605	0.002	CDN
LEU	TIMS	B	4/11/04	4.4598	-0.013	NSH
LEU	TIMS	B	4/11/04	4.4601	-0.007	NSH
LEU	TIMS	B	4/10/04	4.3959	0.099	CDN
LEU	TIMS	B	4/10/04	4.3958	0.097	CDN
LEU	TIMS	B	4/11/04	4.3955	0.090	NSH
LEU	TIMS	B	4/11/04	4.3961	0.104	NSH
LEU	TIMS	F	12/3/03	4.0079	-0.007	247
LEU	TIMS	F	12/3/03	4.0076	-0.015	247
LEU	TIMS	F	12/3/03	4.0077	-0.012	247
LEU	TIMS	F	12/3/03	4.0073	-0.022	247
LEU	TIMS	F	12/3/03	4.0077	-0.012	247
LEU	TIMS	F	12/3/03	4.0076	-0.015	247
LEU	TIMS	F	9/7/04	4.0049	-0.083	247
LEU	TIMS	F	9/7/04	4.0078	-0.011	247
LEU	TIMS	F	9/7/04	4.0061	-0.053	247
LEU	TIMS	F	9/7/04	4.0079	-0.008	247
LEU	TIMS	F	9/7/04	4.0043	-0.098	247
LEU	TIMS	F	9/7/04	4.0067	-0.038	247
LEU	TIMS	F	9/7/04	4.0071	-0.028	247
LEU	TIMS	F	9/7/04	4.0070	-0.031	247
LEU	TIMS	F	9/7/04	4.0079	-0.008	247
LEU	TIMS	F	9/7/04	4.0075	-0.018	247
LEU	TIMS	F	9/7/04	4.0087	0.012	247
LEU	TIMS	T	3/16/04	4.0098	0.040	
LEU	TIMS	T	3/16/04	4.0118	0.090	
LEU	TIMS	T	6/21/04	4.01076	0.063	
LEU	TIMS	T	6/21/04	4.01076	0.063	
LEU	TIMS	T	6/21/04	4.010801	0.064	
LEU	TIMS	T	6/21/04	4.011749	0.088	
LEU	TIMS	T	10/8/04	4.009851	0.040	
LEU	TIMS	T	10/8/04	4.01076	0.063	
LEU	TIMS	T	10/8/04	4.010800	0.064	
LEU	TIMS	T	10/8/04	4.010760	0.063	



## Appendix C: Plutonium Assay Results

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis Date</u>	<u>Reported Result</u>	<u>% RD</u>	<u>Analyst</u>
Pu sulfate	IDMS	F	12/13/03	43	-0.002	201
Pu sulfate	IDMS	F	12/5/03	42.998	-0.007	201
Pu sulfate	IDMS	F	12/13/03	43.507	-0.053	201
Pu sulfate	IDMS	F	12/5/03	43.5	-0.069	201
Pu sulfate	IDMS	F	12/12/03	43.511	-0.044	201
Pu sulfate	IDMS	F	12/13/03	43.271	0.060	201
Pu sulfate	IDMS	F	12/12/03	43.278	0.076	201
Pu sulfate	IDMS	F	12/4/03	43.269	0.055	201
Pu sulfate	IDMS	F	12/12/03	41.881	-3.863	201
Pu sulfate	IDMS	F	12/4/03	41.875	-3.849	201
Pu sulfate	IDMS	G	5/6/04	45.534	-1.135	
Pu sulfate	IDMS	G	5/6/04	42.632	-0.986	
Pu sulfate	IDMS	G	5/6/04	45.558	-1.083	
Pu sulfate	IDMS	G	5/6/04	42.655	-0.933	





Appendix D: <sup>239</sup>Pu Isotopic Results

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis</u>		<u>Analyst</u>
			<u>Date</u>	<u>% RD</u>	
Pu-239	TIMS	F	12/12/03	0.003	201
Pu-239	TIMS	F	12/13/03	0.002	201
Pu-239	TIMS	F	12/12/03	0.004	201
Pu-239	TIMS	F	12/13/03	0.004	201
Pu-239	TIMS	F	12/12/03	-0.001	201
Pu-239	TIMS	F	12/13/03	0.000	201
Pu-239	TIMS	F	12/12/03	0.003	201
Pu-239	TIMS	F	12/13/03	0.002	201
Pu-239	TIMS	F	12/12/03	0.005	201
Pu-239	TIMS	F	12/13/03	0.005	201
Pu-239	TIMS	F	12/12/03	0.002	201
Pu-239	TIMS	F	12/13/03	0.001	201
Pu-239	TIMS	G	5/5/04	-0.049	
Pu-239	TIMS	G	5/5/04	-0.051	
Pu-239	TIMS	G	5/5/04	0.000	
Pu-239	TIMS	G	5/5/04	-0.007	
Pu-239	TIMS	G	5/5/04	-0.003	
Pu-239	TIMS	G	5/5/04	0.007	
Pu-239	TIMS	J	1/27/04	-0.008	
Pu-239	TIMS	J	1/30/04	-0.014	
Pu-239	TIMS	J	2/26/04	-0.002	
Pu-239	TIMS	J	1/27/04	-0.007	
Pu-239	TIMS	J	2/26/04	-0.003	
Pu-239	TIMS	J	1/27/04	-0.003	
Pu-239	TIMS	J	1/30/04	0.000	
Pu-239	TIMS	J	2/26/04	0.012	
Pu-239	TIMS	J	1/27/04	-0.002	
Pu-239	TIMS	J	1/27/04	0.002	
Pu-239	TIMS	J	1/30/04	-0.001	
Pu-239	TIMS	J	2/26/04	0.008	
Pu-239	TIMS	J	12/10/03	-0.018	
Pu-239	TIMS	J	12/10/03	-0.014	
Pu-239	TIMS	J	12/10/03	-0.019	
Pu-239	TIMS	J	12/10/03	-0.012	
Pu-239	TIMS	J	12/10/03	-0.012	
Pu-239	TIMS	J	12/10/03	-0.018	
Pu-239	TIMS	J	12/10/03	-0.013	
Pu-239	TIMS	J	12/10/03	-0.012	
Pu-239	TIMS	J	12/10/03	-0.014	

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis</u> <u>Date</u>	<u>% RD</u>	<u>Analyst</u>
Pu-239	TIMS	T	1/8/04	0.008	
Pu-239	TIMS	T	1/8/04	0.004	
Pu-239	TIMS	T	1/8/04	-0.001	
Pu-239	TIMS	T	1/8/04	-0.002	
Pu-239	TIMS	T	3/25/04	-0.008	
Pu-239	TIMS	T	3/25/04	-0.002	
Pu-239	TIMS	T	3/23/04	-0.007	
Pu-239	TIMS	T	3/23/04	-0.005	
Pu-239	TIMS	T	6/25/04	0.000	
Pu-239	TIMS	T	6/25/04	0.001	
Pu-239	TIMS	T	6/25/04	-0.001	
Pu-239	TIMS	T	6/25/04	0.002	
Pu-239	TIMS	T	10/5/04	0.008	
Pu-239	TIMS	T	10/5/04	0.008	
Pu-239	TIMS	T	10/5/04	0.004	
Pu-239	TIMS	T	10/5/04	0.004	

Appendix E: <sup>240</sup>Pu Isotopic Results

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis</u>		<u>Analyst</u>
			<u>Date</u>	<u>% RD</u>	
Pu-240	TIMS	F	12/12/03	-0.039	201
Pu-240	TIMS	F	12/13/03	-0.040	201
Pu-240	TIMS	F	12/12/03	-0.019	201
Pu-240	TIMS	F	12/13/03	-0.022	201
Pu-240	TIMS	F	12/12/03	-0.007	201
Pu-240	TIMS	F	12/13/03	-0.004	201
Pu-240	TIMS	F	12/12/03	-0.039	201
Pu-240	TIMS	F	12/13/03	-0.038	201
Pu-240	TIMS	F	12/12/03	-0.023	201
Pu-240	TIMS	F	12/13/03	-0.024	201
Pu-240	TIMS	F	12/12/03	-0.202	201
Pu-240	TIMS	F	12/13/03	-0.197	201
Pu-240	TIMS	G	5/5/04	0.297	
Pu-240	TIMS	G	5/5/04	0.282	
Pu-240	TIMS	G	5/5/04	-0.036	
Pu-240	TIMS	G	5/5/04	-0.058	
Pu-240	TIMS	G	5/5/04	0.004	
Pu-240	TIMS	G	5/5/04	-0.034	
Pu-240	TIMS	J	1/27/04	0.048	
Pu-240	TIMS	J	1/30/04	0.060	
Pu-240	TIMS	J	2/26/04	-0.004	
Pu-240	TIMS	J	1/27/04	0.015	
Pu-240	TIMS	J	2/26/04	-0.012	
Pu-240	TIMS	J	1/27/04	-0.002	
Pu-240	TIMS	J	1/30/04	-0.004	
Pu-240	TIMS	J	2/26/04	-0.045	
Pu-240	TIMS	J	1/27/04	-0.006	
Pu-240	TIMS	J	1/27/04	-0.017	
Pu-240	TIMS	J	1/30/04	-0.008	
Pu-240	TIMS	J	2/26/04	-0.040	
Pu-240	TIMS	J	12/10/03	0.097	
Pu-240	TIMS	J	12/10/03	0.063	
Pu-240	TIMS	J	12/10/03	0.070	
Pu-240	TIMS	J	12/10/03	0.035	
Pu-240	TIMS	J	12/10/03	0.037	
Pu-240	TIMS	J	12/10/03	0.049	
Pu-240	TIMS	J	12/10/03	0.038	
Pu-240	TIMS	J	12/10/03	0.029	
Pu-240	TIMS	J	12/10/03	0.037	

<u>Material</u>	<u>Method Type</u>	<u>Facility</u>	<u>Analysis</u> <u>Date</u>	<u>% RD</u>	<u>Analyst</u>
Pu-240	TIMS	T	1/8/04	-0.060	
Pu-240	TIMS	T	1/8/04	-0.076	
Pu-240	TIMS	T	1/8/04	0.001	
Pu-240	TIMS	T	1/8/04	-0.007	
Pu-240	TIMS	T	3/25/04	0.013	
Pu-240	TIMS	T	3/25/04	-0.011	
Pu-240	TIMS	T	3/23/04	0.024	
Pu-240	TIMS	T	3/23/04	0.024	
Pu-240	TIMS	T	6/25/04	-0.013	
Pu-240	TIMS	T	6/25/04	-0.012	
Pu-240	TIMS	T	6/25/04	-0.013	
Pu-240	TIMS	T	6/25/04	-0.018	
Pu-240	TIMS	T	10/5/04	-0.035	
Pu-240	TIMS	T	10/5/04	-0.036	
Pu-240	TIMS	T	10/5/04	-0.019	
Pu-240	TIMS	T	10/5/04	-0.031	