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EVALUATION OF AN INTERLABORATORY COMPARISON INVOLVING PYROCARBON- AND SILICON CARBIDE-COATED URANIUM–THORIUM CARBIDE BEADS: PHASE II

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and Elaine H. Jacob

Research and Development Report

NEW BRUNSWICK LABORATORY

Carleton D. Bingham, Director

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EVALUATION OF AN INTERLABORATORY COMPARISON INVOLVING PYROCARBON- AND SILICON CARBIDE-COATED URANIUM-THORIUM CARBIDE BEADS - PHASE II

ABSTRACT

Six chemistry laboratories and one nondestructive assay laboratory participated in the second phase of an interlaboratory comparison program, involving pyrocarbon- and silicon carbide-coated, uranium-thorium carbide beads. Accuracy and precision of measurements were estimated by supplying known quantities of mixtures of four different ratios of thorium oxide (ThO_2) to uranium oxide (UO_2). The ratios for the oxide mixtures were nominally 0, 10, 16, 25:1 of ThO_2 to UO_2.

The range of the chemical determination of uranium content in the oxide mixtures was less than 0.3% of the assigned value and the within-laboratory precision ranged from 0.07 to 0.33 (% standard deviation of relative difference). The determination of thorium in the mixtures exhibited a generally much lesser degree of refinement in the state-of-the art compared to the uranium analyses.

Chemical assay for the determination of the uranium (thorium) concentration in the BISO beads for all laboratories exhibited a range from -0.18 to +0.16 (-0.27 to +0.47) % relative to the assigned value. The within-laboratory standard deviation of relative difference ranged from 0.07 to 0.14 (0.11 to 0.57).

The chemical assay for the determination of uranium (thorium) concentration of the TRISO beads exhibited a range from -0.12 to 0.18 (-3.67 to +1.58) % relative to the assigned value. The within-laboratory standard deviation of the relative difference ranged from 0.07 to 0.45 (0.49 to 2.09).

NDA measurements for uranium on the mixed oxides showed a positive deviation ranging from 0.12 to 0.97%. The within-laboratory precision ranged from 0.57 to 1.61 standard deviation of the relative difference. The NDA measurements on BISO beads showed a value of -0.39 $\pm$ 0.83 (mean relative difference $\pm$ standard deviation of relative differences) relative to the assigned values for 178 samples. NDA measurements on TRISO beads showed a value of -0.42 $\pm$ 1.08 (mean relative difference $\pm$ standard deviation of relative differences) relative to the assigned value for 188 samples.

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Introduction

Phase II of the High Temperature Gas-Cooled Reactor (HTGR) Fuel Interlaboratory Comparison was undertaken following the completion of Phase I of the same program[1] for the following reasons: 1) to define more clearly the state-of-the-art of uranium and thorium analytical chemistry in HTGR fuel materials, 2) to look at variations in performance with time and with sample preparation, and 3) to reaffirm the Phase I results for accuracy and precision of the chemical measurements at each laboratory.

The following laboratories participated in Phase II:

Oak Ridge National Laboratory (ORNL)

General Atomic Company (GAC)
2 organizationally independent laboratories

Los Alamos Scientific Laboratory (LASL)

New Brunswick Laboratory (NBL)
2 organizationally independent laboratories

Conduct of the Experiment

As in Phase I, there was no standard material of uranium-thorium carbide available to test the accuracy of the measurements of the participating laboratories; therefore, a series of vials were prepared containing varying ratios of uranium dioxide and thorium dioxide in known quantities. With these known values, experimental measurement accuracies could be estimated from the performance of each of the participating laboratories. Uranium dioxide (93% enriched in ^{235}U) and thorium dioxide were obtained from GAC as materials typical of the starting ingredients in their production process. These materials were assayed at NBL for uranium and thorium, respectively, and for uranium isotopic content as described in Appendix 1. The sampling and randomization schemes are also outlined in Appendix 1.

A supply of BISO and TRISO beads was also obtained from GAC.

Both types of beads were successively split down to approximately 15-g samples which were then transferred to vials for distribution in the program.

After preparation, characterization and randomnization, the mixed oxides, BISO and TRISO samples were assayed by non-destructive analysis and then returned to NBL for distribution to the other participants of the program for destructive analysis. During the course of the experiment, 2 mixed oxides, 1 BISO and 1 TRISO sample were to be analyzed monthly and the results reported to NBL. The mixed oxide samples were to be totally dissolved and the resulting solutions were to be analyzed in quadruplicate for uranium and in duplicate for thorium. The vials of riffled beads were likewise to be totally dissolved and quadruplicate measurements made for uranium and duplicate measurements made for thorium. See Appendix 2 for more detailed instructions to the participants.

All data were forwarded to NBL for reduction and evaluation.

Statistical Treatment of the Data

The HTGR Interlaboratory Comparison Program is a many faceted experiment which looks at many questions in the preparation and analysis of uranium and thorium samples. This discussion attempts to explain the actions and philosophies adhered to throughout the experiment without overburdening the non-statistician with unnecessary matters of detail. Those readers with questions pertaining to the methodology and procedures used in this report, may contact the authors.

For the purpose of simplifying the analysis and providing results with a more useful intuitive meaning, the reported data were converted to relative differences (% differences) from the prepared /assigned values. The conversion was made in the following manner,

$$R.D. = \frac{\text{observed value - prepared/assigned value}}{\text{prepared/assigned value}} \times 100$$

Ideally, each laboratory would experience a relative difference distribution with a zero mean and a zero variance. Variance of the relative difference distribution [VAR(RD)] is calculated in the conventional manner from the individual RD values, i.e.,

$$VAR(RD) = \frac{\sum_i (RD_i - \overline{RD})^2}{n-1}$$

Tables and graphs have been provided which illustrate the relative performance of each laboratory. In some instances, laboratories are identified using several codes (e.g., A, A′,....) to indicate that certain data were excluded from the analysis as outliers. These outliers were identified using a statistical significance level of 10% (i.e. data within a 90% confidence limit were tentatively considered acceptable).

The discussion on the treatment of the data will be divided into two parts - UO_2/ThO_2 powder samples and fuel bead samples (both BISO and TRISO). Each of these parts of the report will be subdivided to consider individually the uranium analyses and the thorium analyses. Throughout this report a 5% statistical significance level was used except where otherwise noted. In some cases the results dictated the mode of analysis since the magnitude and variation of the observations invalidated some testing procedures.

UO_2/ThO_2 Powder Mixtures

The analyses of the UO_2/ThO_2 powder mixtures were examined for month-to-month variations within laboratories (variations with time or time effects), differences between the UO_2/ThO_2 ratio levels within laboratories and for relative differences between laboratories as illustrated by tables and graphs.

The time and ratio effects were examined simultaneously in each laboratory using a two-way analysis of variance for mixed effects (fixed and random).[2] The interaction of time and ratios was also considered. If the analysis of variance was somewhat indecisive regarding the effects of differing ratios, then regression techniques or nonparametric tests might be applied to provide answers to our questions.

The statistical model for the uranium (thorium) analyses could be written as,

$$Y_{ijk} = \mu + \alpha_i + \beta_j + \gamma_{ij} + \varepsilon_{ijk}$$

Where Y_{ijk} = observed value

μ = true bias

α_i = the month (time) effect

β_j = the ratio effect

γ_{ij} = the time/ratio interaction effect

ε_{ijk} = the random errors

$i = 1, \ldots, 10$ (No. of months)

$j = 1, \ldots, 4$ (No. of ratios)

and $k = 1, \ldots, 4(2)$ (No. of replicates)

The ε_{ijk} were assumed to be normally distributed with a mean equal to zero and a variance equal to σ^2. The α_i are also assumed to be normally distributed with a specified mean and variance. The only modification to the model for the thorium analyses is j=1,2, meaning two replicates per sample instead of four. The model was tested from the analysis of variance table to determine whether $\mu=0$ in order to reveal any statistically significant biases in the laboratories.

The oxide data contained a few outlying observations which were identified. These outliers were not excluded in any way since they were considered acceptable at the time of analysis by the submitting laboratory. Cochran's test for homogeneity of variance[3] was used to identify those groups of replicates in which poor precision was observed. This test also indicated the validity of the homogeneity of variance assumption.

The above model, leading to a two-way analysis of variance would have been preferable; however, in the presence of inhomogeneity of variance, interaction and an unbalanced design, this model lost its ability to provide more powerful tests. As a result the simple effect of time was considered separately.

BISO-TRISO Beads

The analyses of the BISO and TRISO beads were examined for month-to-month variations within laboratories (variations with time or time effects), method of sample preparation (TRISO only) and for relative differences between laboratories as illustrated by tables and graphs.

Perhaps the most critical place to begin is with the method used to determine the assigned value for the uranium and thorium content of the BISO and TRISO beads. Data from Laboratories B and G were not included in the calculation of the assigned value due to the lower degree of precision and accuracy of the measurement methods used in these laboratories. Laboratory H was not included in the calculation of the assigned value since it was inexperienced in the measurement procedure which is used.

Laboratories A, C, D, E, F were individually examined to determine if there were any statistical outliers (at the 10% statistical significance level) in the replicate determinations of the samples. In only a few cases could any of the replicate determinations be considered as outliers.

Next, the sample means were examined to see if any of the months could be considered as outliers. In this case, several outlying months were observed and "set aside" pending a diagnostic review of the data. A prime notation was used to indicate a laboratory with outliers excluded.

Lastly, all laboratory means (A, C, D, E, F, excluding outlying months) were tested to see if any laboratory was an outlier, overall. No laboratory could be rejected as being an outlier so that the interlaboratory mean was determined and used as an assigned value from which the conversion to R.D.'s was made.

Several outliers tests were used to screen the data for different types of anomalous observations. The tests[4] used for indicating possible outliers (in addition to subjective judgment) were:

1. range test
2. skewness test
3. kurtosis test
4. Dixon criterion
5. T_n test
6. high or low pair test

It must be stressed that outlier tests are only methods of identifying possible outliers and not an absolute means of labeling them.

The time effects (within laboratory) were examined using a one-way analysis of variance for a random effect. This analysis would not indicate whether or not there was a significant sample-to-sample variation which might cause the appearance of significant time effects.

The statistical model for the uranium (thorium) analyses on the beads could be written as,

$$Y_{ij} = \mu + \alpha_i + \varepsilon_{ij}$$

Where Y_{ij} = observed value

μ = the true bias

α_i = the time effect

ε_{ij} = the random errors

$i = 1, \ldots, 10$ (No. of months)

and $j = 1, \ldots, 4(2)$ (No. of replicates).

The ε_{ij} are assumed to be normally distributed with a mean equal to zero and a variance equal to σ^2. The only modification to the model for the thorium analyses is j=1,2, meaning two replicates instead of four.

The time effects were again considered as a pseudo-random factor and, in this case, treated as a random factor. The model was tested from the analysis of variance table to determine whether $\mu=0$ in order to reveal the occurrence of any significant bias in the laboratories. The Cochran's test was again used to determine if the homogeneity of variance assumption was valid.

Another factor of interest was the method of preparation of the TRISO bead samples prior to the uranium titrations. The effects of method of preparation were tested using a three-way analysis of variance with one nested factor (laboratories).[5] The time effect was the only factor treated as random. The statistical model could be written as,

$$Y_{ijkl} = \mu + \alpha_i + \beta_j + \gamma_{(i)jk} + \Delta_{ij} + \tau_{jk} + \varepsilon_{ijkl}$$

Where Y_{ijkl} = observed value

μ = the true bias

α_i = the method of preparation (grind-leach, high temperature chlorination or carbonate fusion),

β_j = the time effect

$\gamma_{(i)jk}$ = the laboratory factor nested under the method of preparation factor

Δ_{ij} = the interaction term for the time and method of preparation

T_{jk} = interaction term for time and laboratories

ε_{ijkl} = the random errors

$i = 1, \ldots, 3$ (No. of methods)

$j = 1, \ldots, 10$ (No. of months)

$k = 1, \ldots, 5$ (No. of laboratories)

and $l = 1, \ldots, 4(2)$ (No. of replicates).

The ε_{ijkl} are assumed to be normally distributed with a mean equal to zero and a variance equal to σ^2. Here the factor of primary interest is α_i.

Results

The graphs and tables provided in Figures 1-11 are for the purpose of indicating to each laboratory its performance relative to all other laboratories. The figures illustrate the relationships of the different sources of variation. The standard deviations were expressed as a percentage of the assigned/prepared value throughout these figures. The total height of the vertical lines indicates the magnitude of the variation in a particular measurement system (column 3, Figures 1-11). The total height of the vertical lines is the standard deviation (S.D.) of the relative differences of all determinations made in a laboratory irrespective of the month in which the measurements were made. The solid portion of the vertical lines, beginning at the base of the line, represents the pooled S.D. of the measurement/analytical (within month) error which is the square root of the mean square error term in a one-way analysis of variance with random effects, (column 5 in table). The solid portion of vertical lines indicates the degree of repeatability with which a laboratory can measure any given sample. Column 7 in the table is the square root of the variance component for month-to-month effects which is an estimate of the S.D. of the month-to-month (among months) variation. The horizontal position of the vertical lines designates the mean of the R.D.'s for a given laboratory (column 2 in table). Several laboratories are identified under two codes by using a prime (e.g. A, A′) to indicate that certain data were set aside from the analysis as outliers.

UO_2/ThO_2 Powder Mixtures

Uranium Analyses - In all laboratories there was a significant interaction between ratio and time effects meaning that no consistent time trends could be isolated over all ratios and likewise no consistent ratio effects could be isolated over all months involved. Further, non-parametric testing confirmed that the zero ratio oxide mixture results for the wet chemistry were significantly lower than for the other three ratios. This problem was thought to

be indicative of difficulties in quantitatively removing the pure UO_2 powder from the vials. There was much less difficulty in removing the powder from the vials containing uranium oxide mixed with high-fired thorium oxide using the suggested removal method. Quantitative extraction had not been a problem in the batch of UO_2 used in Phase I of this experiment (see Reference 1); therefore, the effect of variations in physical characteristics from batch-to-batch is apparent.

Figures 1-4 show the results for uranium determination on the oxide mixtures of all the participating laboratories. For the 0:1 (ThO_2:UO_2) ratio in Figure 1, it may be seen that the results of the more highly experienced wet chemistry laboratories (A, C, D, E, F) were quite tightly grouped together on the low side. The nondestructive analyses (NDA) of laboratory G were consistently the highest results for all ratios with statistically significant differences indicated on the 10, 16, 25:1 ratios. The mean of the relative differences for laboratory A was significantly different from zero on the 0:1 ratio (Figure 1) but was not statistically different from the other wet chemistry laboratories.

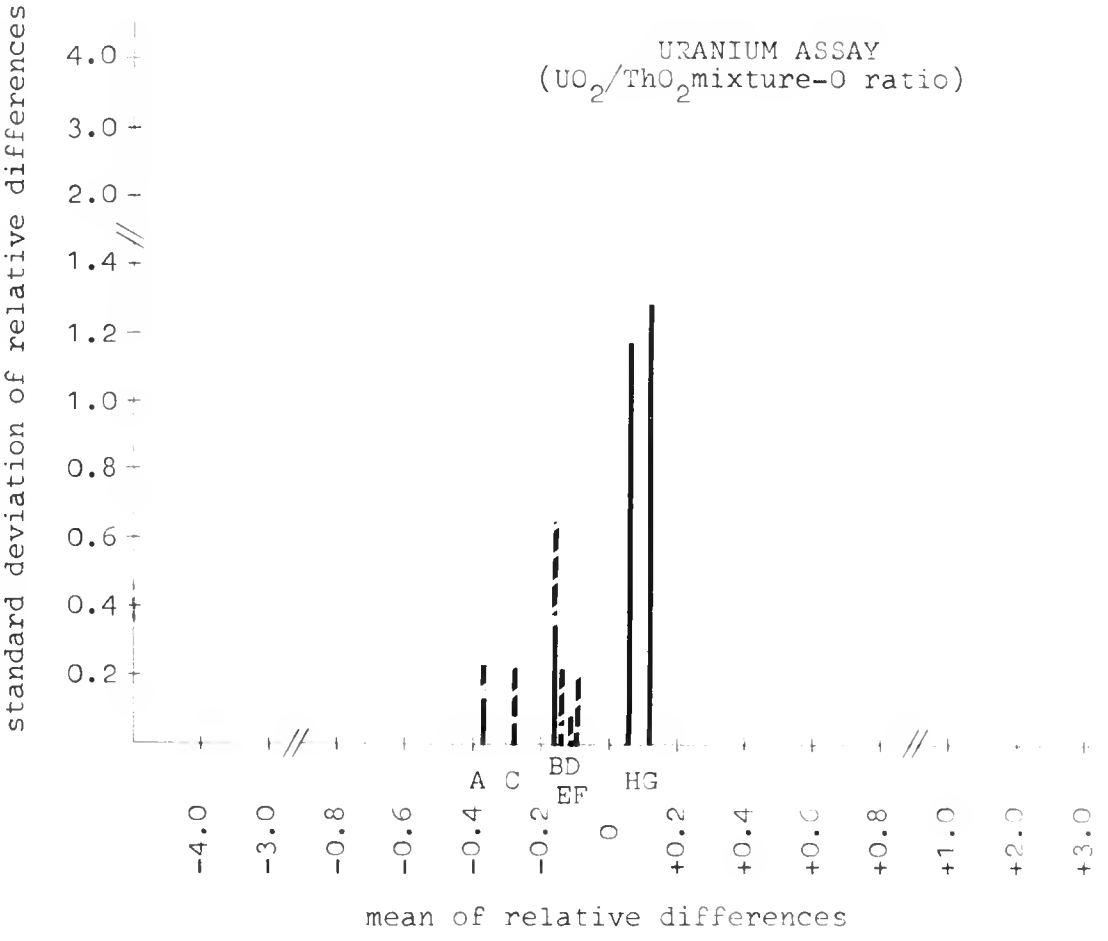
For the 10:1 ratio there was a statistically significant separation between laboratories A, C, F and laboratories D and E. This was the only ratio to indicate such a split. The analyses from Laboratory B were significantly high on the 25:1 ratio.

The degree of precision exhibited by the wet chemistry laboratories was consistent among laboratories with the exception of laboratory H due to its inexperience with the method utilized. Laboratories G and B, as would be expected from the method used, exhibited consistently less precise results (statistically significant) than laboratories A, C, D, E, F. Laboratory G reported consistently less precise results than did laboratory B.

Note that the range of relative differences for the more experienced wet chemistry laboratories averaged less than 0.2%, whereas overall the range averaged over 0.8% for the four different ratios. Laboratory G averaged over 0.4% (absolute) higher than all other laboratories.

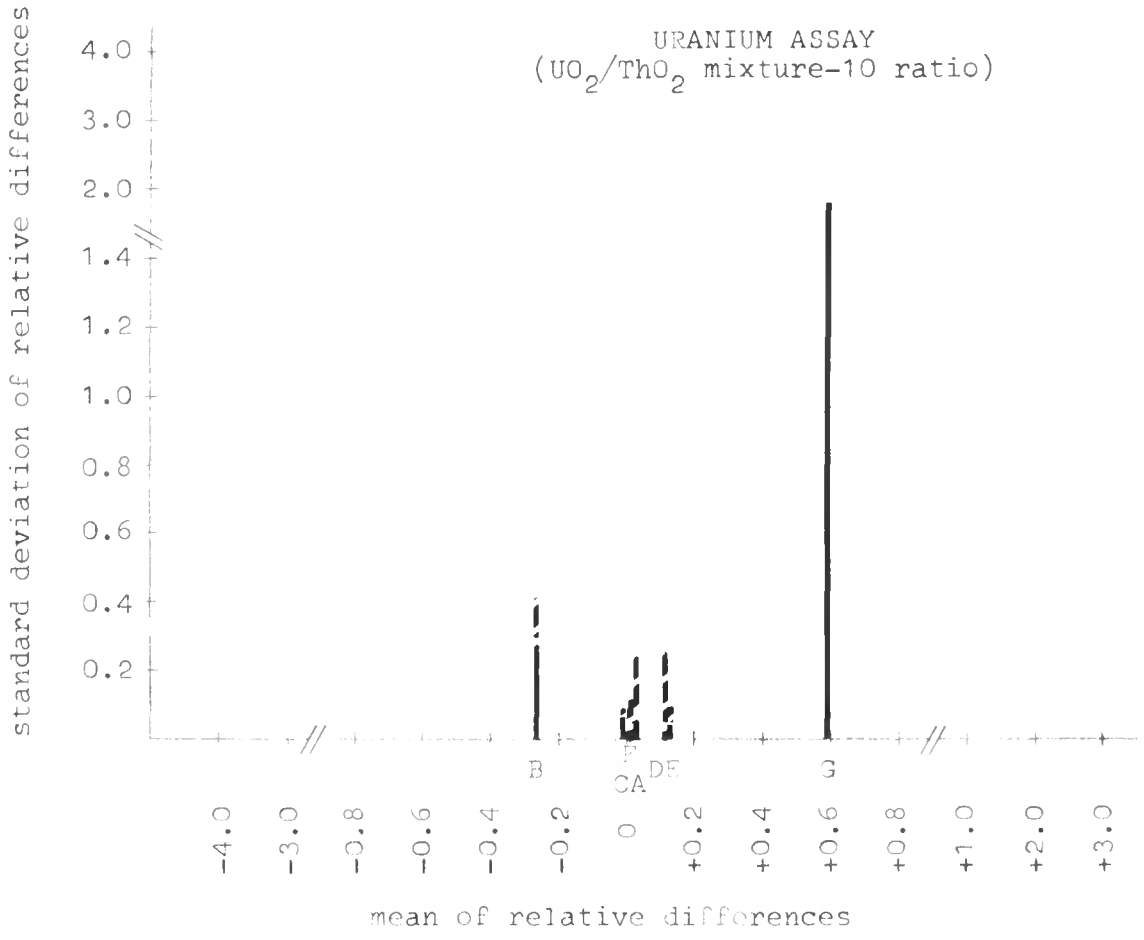
Thorium Analyses - In Figures 5-7 it is obvious that the present state-of-the art practiced for the determination of thorium content is not as highly refined as that for the determination of uranium content. Possible exceptions to this were laboratories E and F which exhibited levels of accuracy and precision comparable to those of the uranium determinations. Laboratory B was consistently low throughout, with a statistically significant difference found on the 25:1 ratio alone (Figure 7). The precision of laboratory B was as good for the thorium analyses as it was for the uranium analyses. The total precision of laboratory B compares quite well with the wet chemistry laboratories.

Laboratories A and C were consistently high with statistically significant differences from zero on the 10:1 ratio alone (Figure 6).



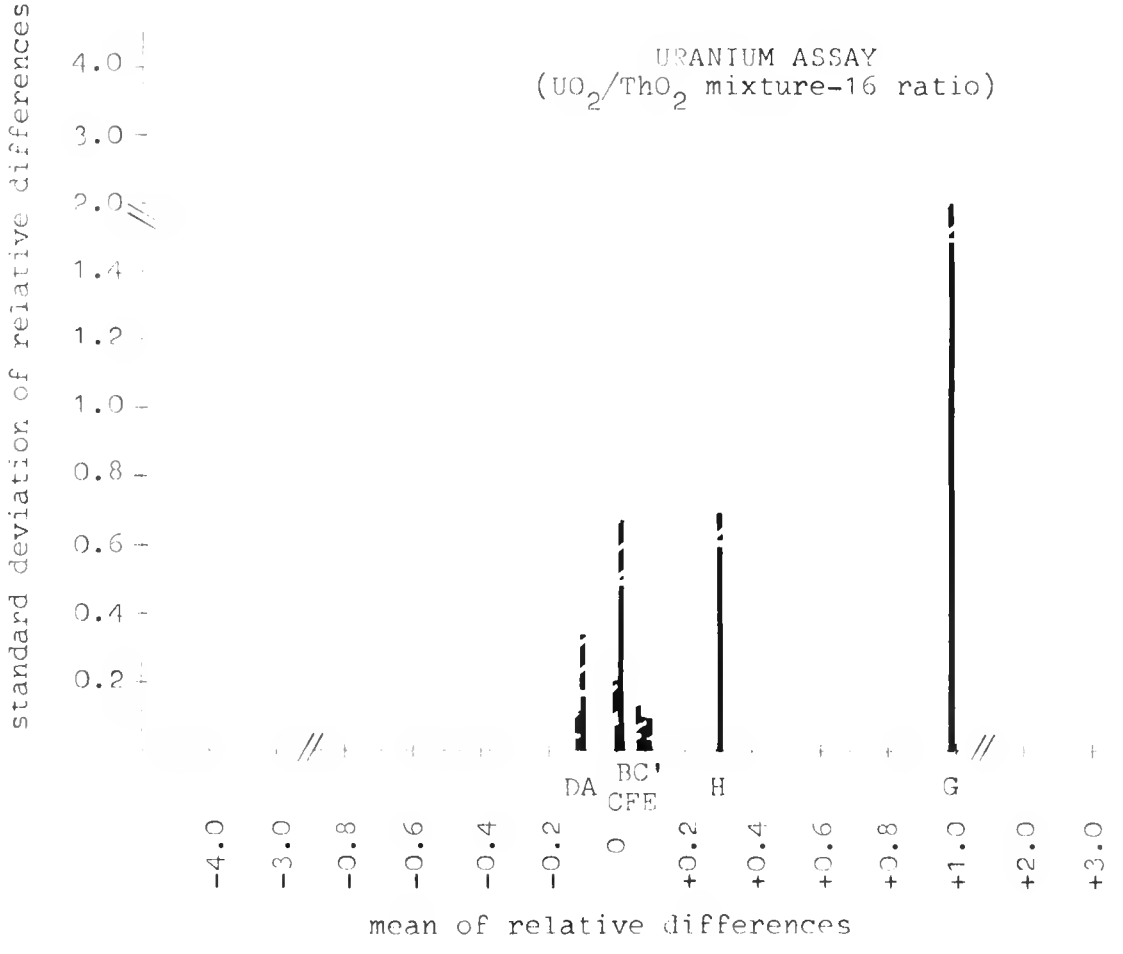
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	-0.37	0.23	15	0.11	12	0.22	3
B	-0.16	0.65	15	0.35	12	0.62	3
C	-0.28	0.22	15	0.06	12	0.23	3
D	-0.10	0.08	15	0.01	12	0.08	3
E	-0.14	0.22	15	0.03	12	0.25	3
F	-0.10	0.20	15	0.03	12	0.23	3
G	0.12	1.21	69	1.29	35	0.79	34
H	0.06	1.10	7	1.18	6	0.01	1

FIGURE 1



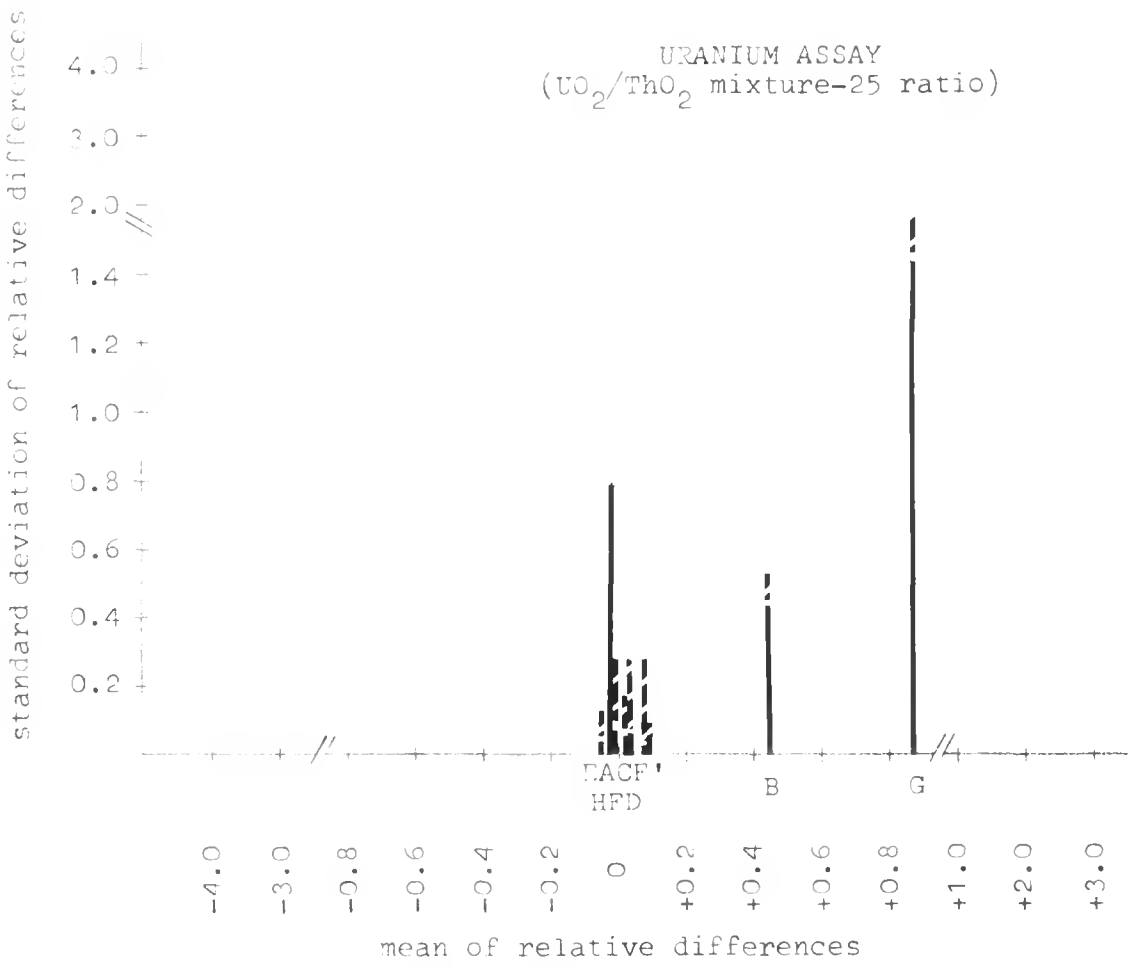
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.02	0.24	15	0.05	12	0.26	3
B	-0.27	0.41	15	0.27	12	0.35	3
C	-0.01	0.07	15	0.06	12	0.04	3
D	0.11	0.25	15	0.02	12	0.28	3
E	0.12	0.09	15	0.03	12	0.10	3
F	0.01	0.11	15	0.02	12	0.12	3
G	0.57	1.77	77	1.80	43	1.17	34
H	None reported						

FIGURE 2



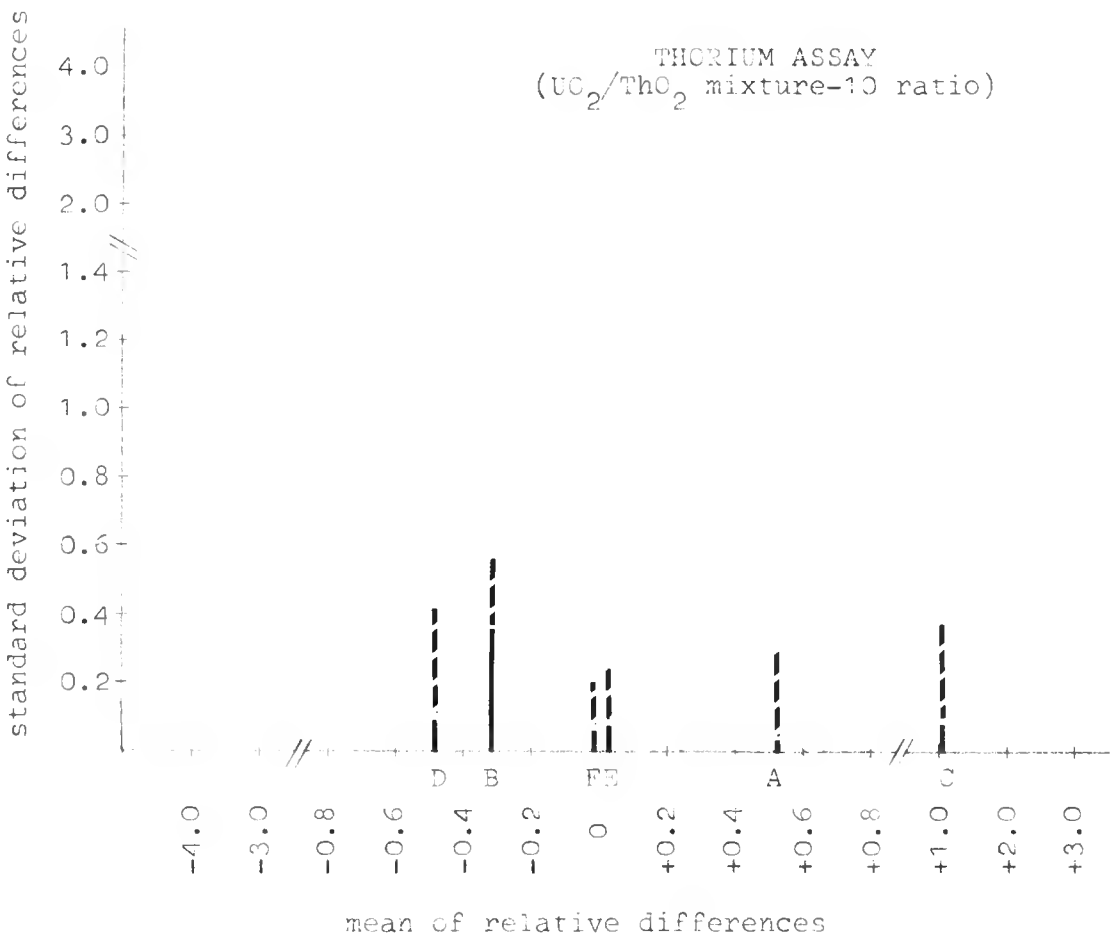
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	-0.10	0.33	23	0.15	18	0.31	5
B	0.01	0.67	23	0.48	18	0.50	5
C	0	0.20	23	0.07	18	0.20	5
C'	0.08	0.10	19	0.07	15	0.07	4
D	-0.11	0.11	23	0.03	18	0.11	5
E	0.09	0.09	23	0.03	18	0.09	5
F	0.07	0.13	23	0.03	18	0.14	5
G	0.97	1.61	113	1.49	61	1.19	52
H	0.30	0.69	7	0.59	6	0.56	1

FIGURE 3



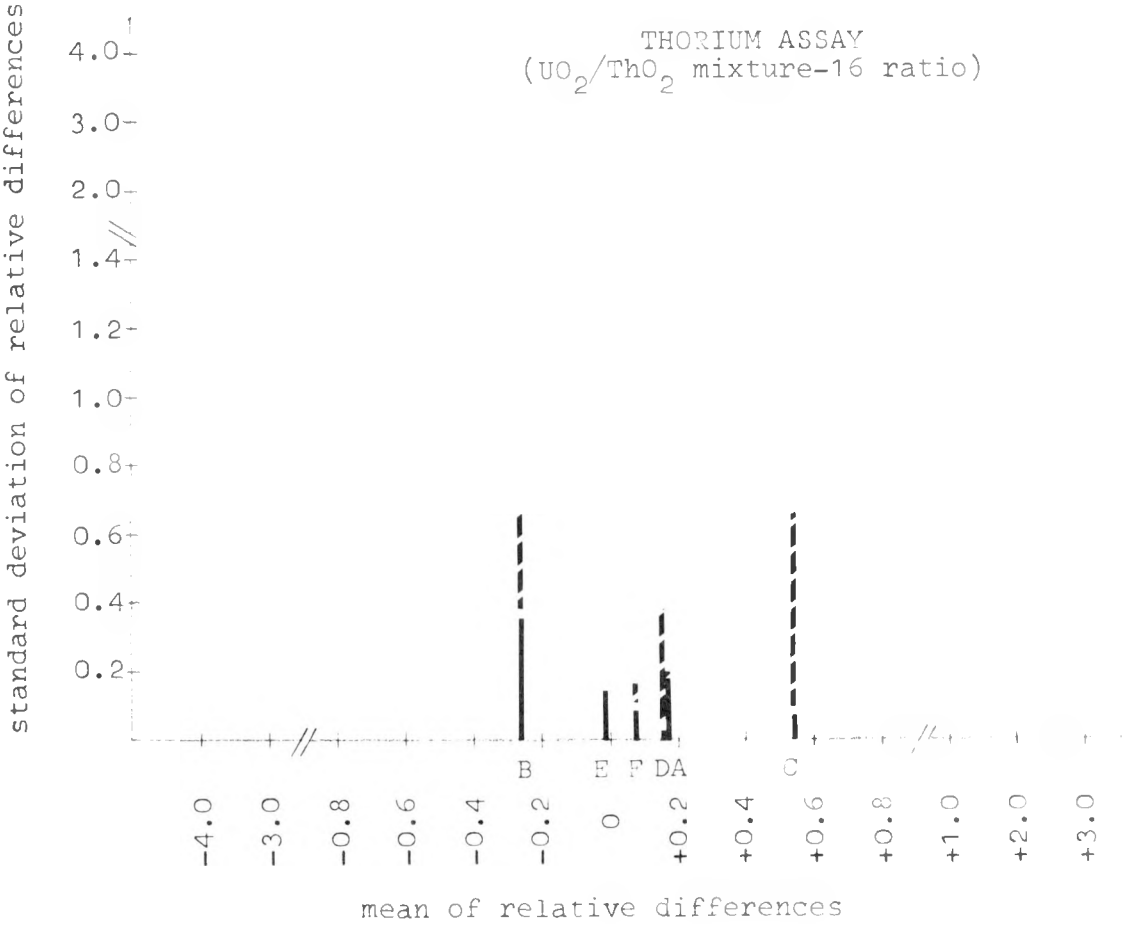
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	-0.01	0.28	23	0.07	18	0.30	5
B	0.44	0.53	23	0.43	18	0.32	5
C	0.03	0.28	23	0.06	18	0.30	5
D	0.08	0.28	23	0.02	18	0.30	5
E	-0.05	0.13	23	0.04	18	0.13	5
F	0.02	0.17	23	0.05	18	0.17	5
F'	0.08	0.09	19	0.06	15	0.08	4
G	0.87	1.57	136	1.43	72	1.19	64
H	-0.01	0.77	7	0.79	6	0.35	1

FIGURE 4



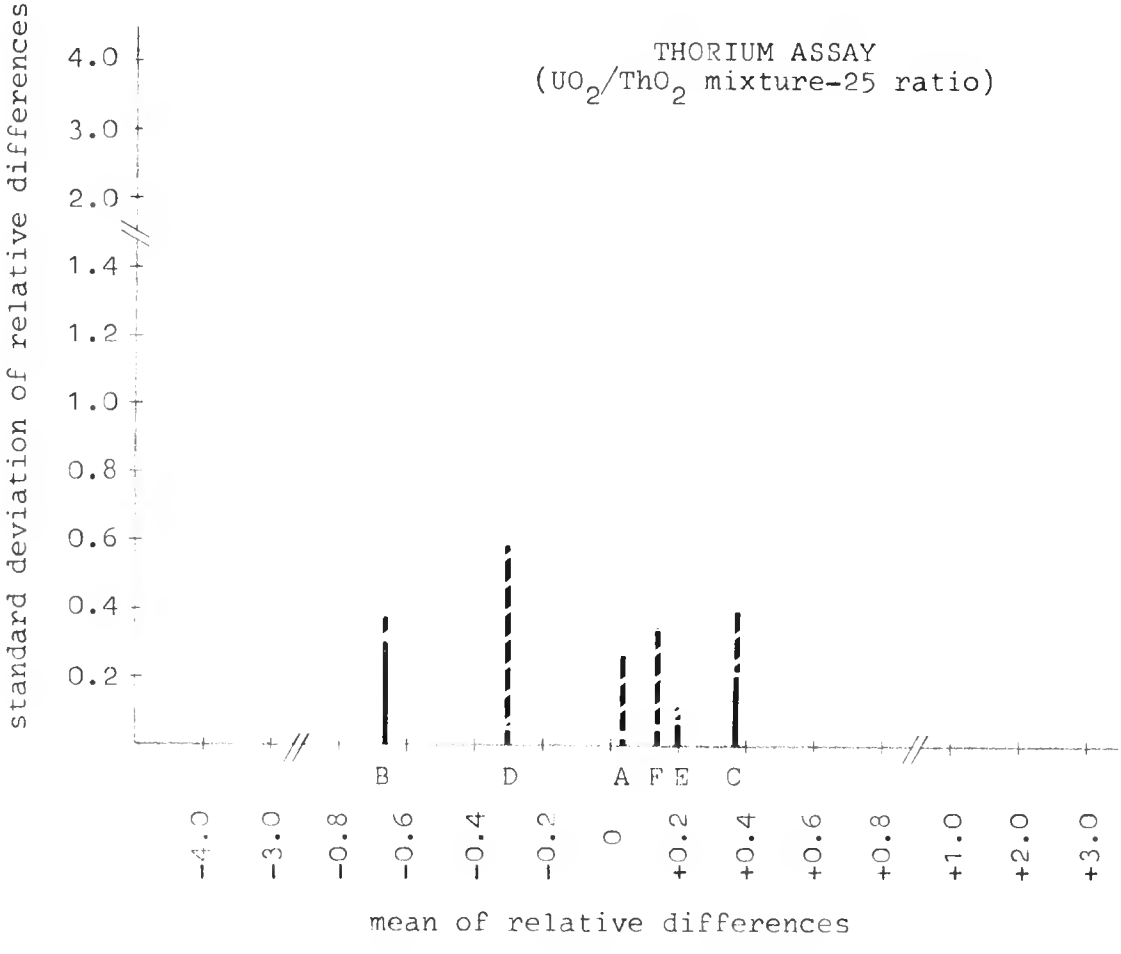
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.52	0.30	7	0.03	4	0.32	3
B	-0.31	0.56	15	0.35	12	0.48	3
C	1.06	0.37	7	0.08	4	0.39	3
D	-0.48	0.41	7	0.08	4	0.43	3
E	0.03	0.24	7	0.07	4	0.24	3
F	-0.01	0.20	7	0.05	4	0.21	3

FIGURE 5



Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.17	0.20	11	0.18	6	0.10	5
B	-0.26	0.67	23	0.35	18	0.61	5
C	0.55	0.66	11	0.07	6	0.69	5
D	0.16	0.38	11	0.02	6	0.40	5
E	-0.01	0.14	11	0.14	6	0	5
F	0.08	0.16	11	0.08	6	0.15	5

FIGURE 6



Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.04	0.26	11	0.02	6	0.27	5
B	-0.66	0.37	23	0.30	18	0.24	5
C	0.38	0.39	11	0.20	6	0.35	5
D	-0.30	0.58	11	0.04	6	0.61	5
E	0.20	0.11	11	0.06	6	0.09	5
F	0.14	0.34	11	0.04	6	0.35	5

FIGURE 7

BISO Bead Samples

Uranium Analyses (Figure 8) - The assigned uranium value for the BISO bead samples was 0.13388 ± 0.00001 (±0.007%) g U/g sample. This is the mean and standard error of 160 values reported by laboratories A′, C′, D, E′, and F. The results for these five laboratories (outliers set aside) were comparable to the results observed on the oxide mixtures.

The values reported by laboratory C in the first few months of the experiment were low due to a sample preparation problem later resolved; therefore, only the C′ results should be considered in evaluating the capability of that laboratory.

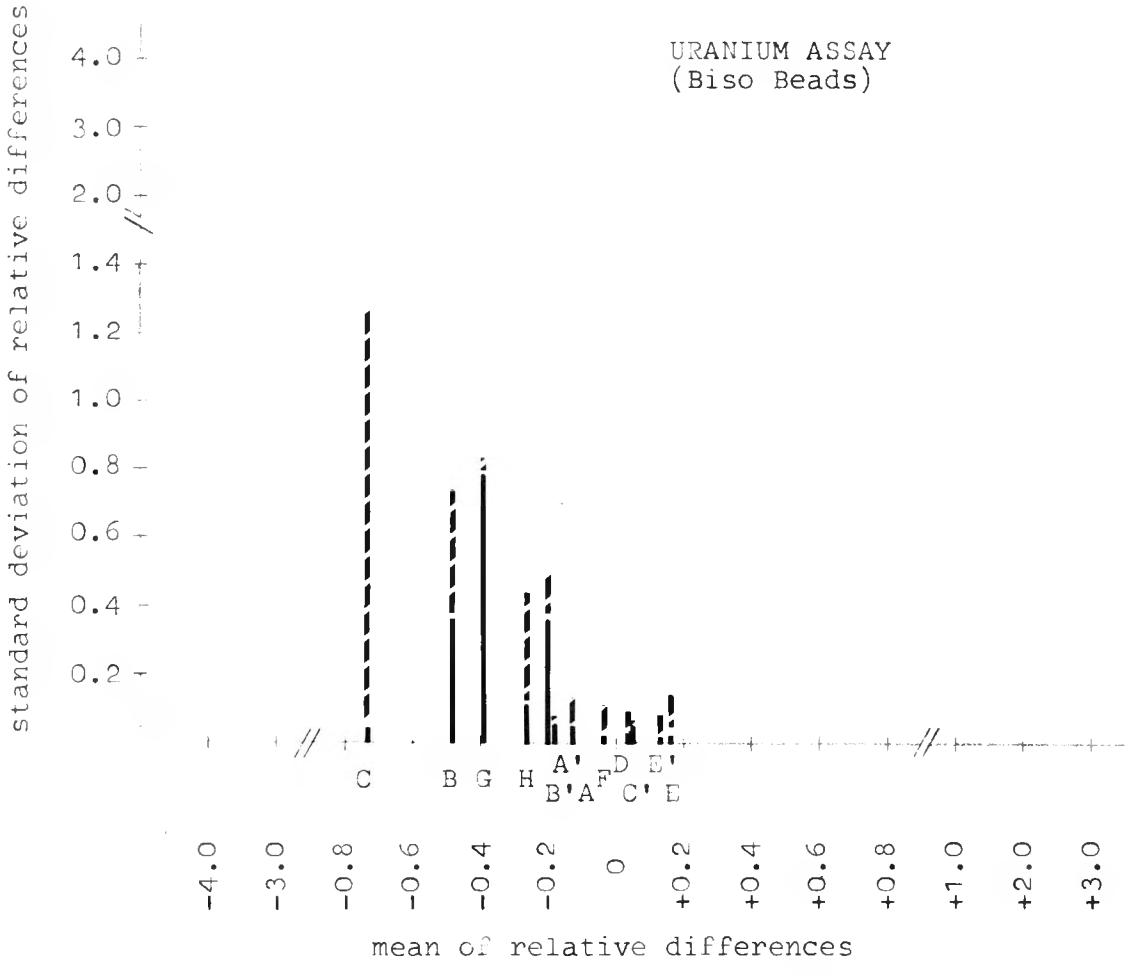
Note that laboratories B and G reported lower results than all the wet chemical laboratories.

Thorium Analyses (Figure 9) - The assigned thorium value for the BISO bead samples was 0.48324 ± 0.00004 (±0.008%) g Th/g sample. This is the mean and standard error of 82 values reported by laboratories A′, C′, D′, E and F. The range of the means for these five laboratories (excluding outliers) is approximately 0.7%. This range compares favorably with the results of the thorium analysis of the oxide mixtures.

TRISO Bead Samples

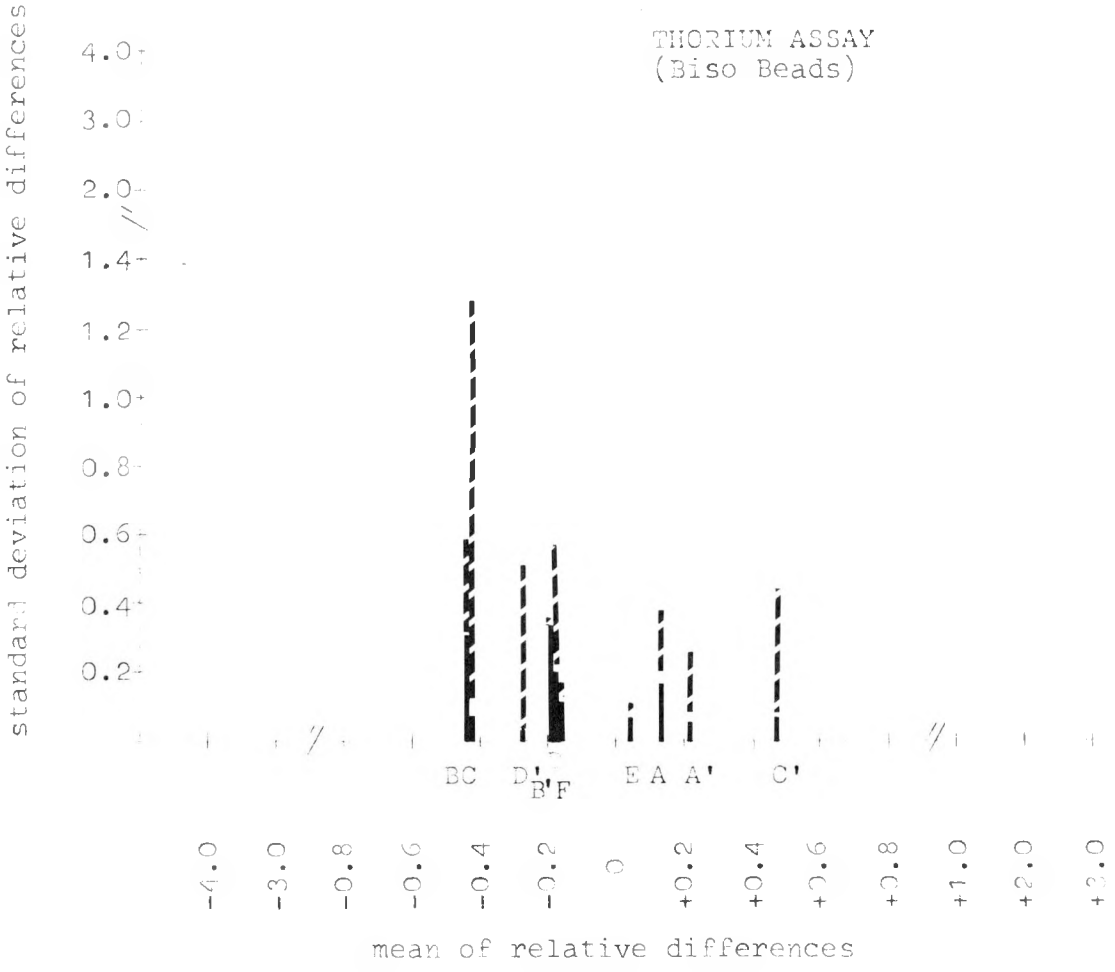
Uranium Analyses (Figure 10) - The assigned uranium value for the TRISO bead samples was 0.07128 ± 0.00002 (±0.028%) g U/g sample. This is the mean and standard error of 160 values reported by laboratories A′, C′, D′, E and F. A sample preparation problem was again observed in laboratory C in the first few months of the experiment for the uranium analyses of the TRISO beads; therefore, only the C′ results were considered in evaluating the capability of that laboratory. In general, a poorer degree of precision was noticed in the uranium analyses of TRISO beads than the BISO beads or oxides. This may be due to the increased difficulty in preparing the sample for analysis or maybe variation inherent in the manufacturing process. Again note that laboratory B and G are at the lowest extreme as was observed in the BISO beads. J and K are subsets of analyses from laboratory F such that J + K = F. The samples reported under designation J were prepared by a grind-leach procedure. The samples reported under designation K were prepared by a high temperature chlorination procedure. Refer to Appendix 3 for a more detailed explanation as to the method of analysis used in each laboratory.

With the exception of laboratory C, there was a very definite trend in the mean results for the uranium assay of the TRISO beads when compared according to the method of sample preparation. Observe the following laboratories which are ranked from low to high results in Table I:



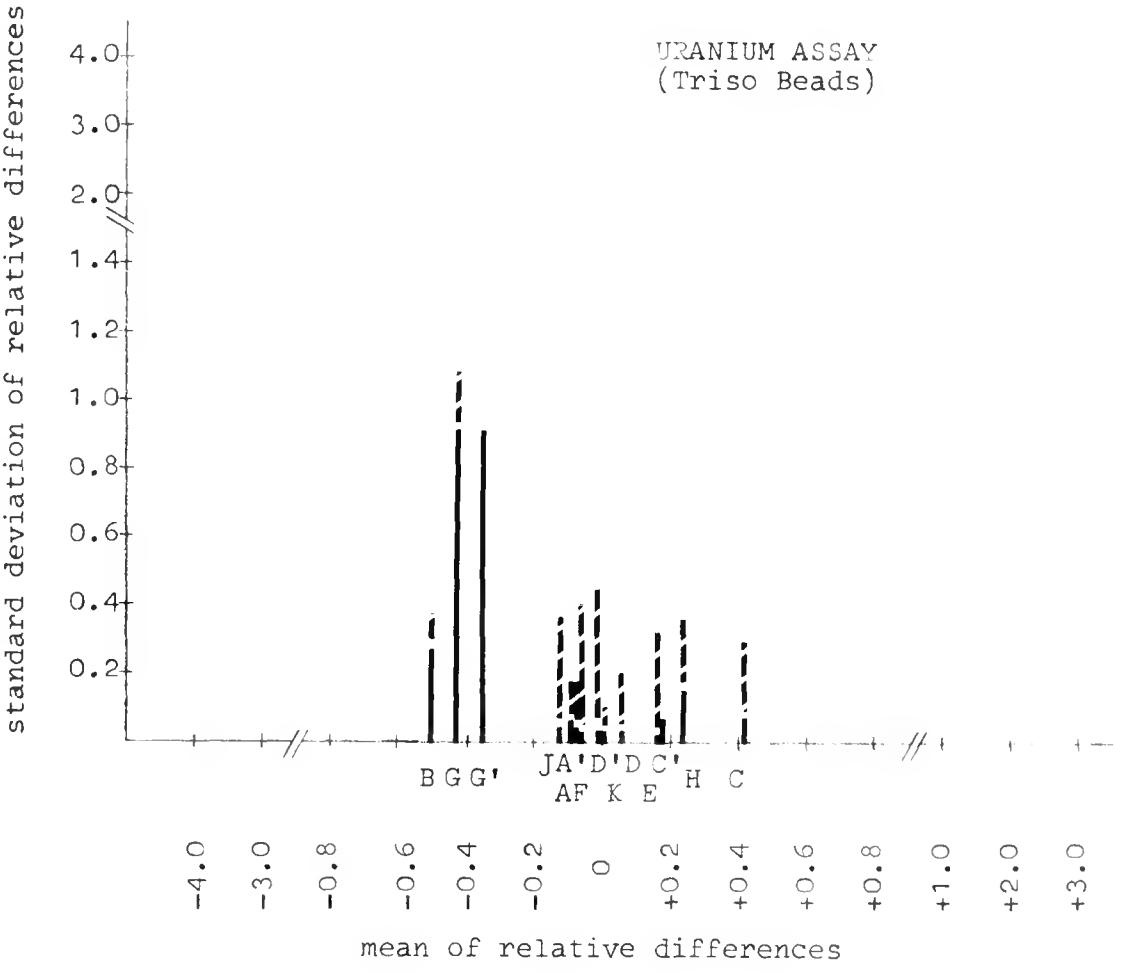
Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	-0.13	0.13	39	0.05	30	0.12	9
A'	-0.18	0.08	31	0.05	24	0.07	7
B	-0.48	0.74	39	0.36	30	0.70	9
B'	-0.20	0.50	31	0.36	24	0.40	7
C	-0.73	1.28	39	0.04	30	1.33	9
C'	0.05	0.07	15	0.05	12	0.06	3
D	0.04	0.09	39	0.02	30	0.10	9
E	0.16	0.14	39	0.02	30	0.15	9
E'	0.13	0.08	31	0.01	24	0.08	7
F	-0.03	0.11	39	0.02	30	0.11	9
G	-0.39	0.83	177	0.78	98	0.59	79
H	-0.26	0.44	31	0.11	24	0.45	7

FIGURE 8



Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.13	0.38	19	0.17	10	0.37	9
A'	0.22	0.26	15	0.06	8	0.27	7
B	-0.44	0.59	39	0.31	30	0.55	9
B'	-0.20	0.36	31	0.34	24	0.21	7
C	-0.43	1.28	19	0.07	10	1.31	9
C'	0.47	0.44	7	0.07	4	0.47	3
D	-0.18	0.57	19	0.20	10	0.57	9
D'	-0.27	0.51	17	0.03	9	0.52	8
E	0.04	0.11	19	0.07	10	0.10	9
F	-0.17	0.17	19	0.11	10	0.16	9

FIGURE 9



Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	-0.09	0.17	35	0.06	27	0.17	8
A'	-0.09	0.17	31	0.04	24	0.18	7
B	-0.50	0.37	39	0.27	30	0.30	9
C	0.42	0.29	39	0.07	30	0.28	9
C'	0.18	0.07	15	0.07	12	0.04	3
D	0.06	0.20	39	0.03	30	0.21	9
D'	0	0.10	35	0.03	27	0.10	8
E	0.17	0.32	39	0.02	30	0.33	9
F	-0.07	0.40	35	0.03	27	0.42	8
G	-0.42	1.08	187	0.91	108	0.88	79
G'	-0.35	0.90	185	0.91	107	0.57	78
H	0.24	0.36	31	0.15	24	0.35	7
J	-0.12	0.36	19	0.04	15	0.39	4
K	0	0.45	15	0.03	12	0.50	3

FIGURE 10

TABLE I

URANIUM ASSAY OF TRISO BEADS
SUMMARY OF METHODS USED
(Ranked by Means)

Laboratory	Mean Result	Method of Preparation	Method of Measurement
B	0.070921	GL	X-Ray
J	0.071190	GL	NBL
A	0.071214	GL	"
F	0.071230	GL/CL	"
K	0.071279	CL	"
D´	0.071280	CL	"
D	0.071321	CL	"
E	0.071399	CF	"
C´	0.071450	GL	"
H	0.071450	GL	"
C	0.071578	GL	"

Where: GL represents a grind-leach preparation

CL represents a high temperature chlorination

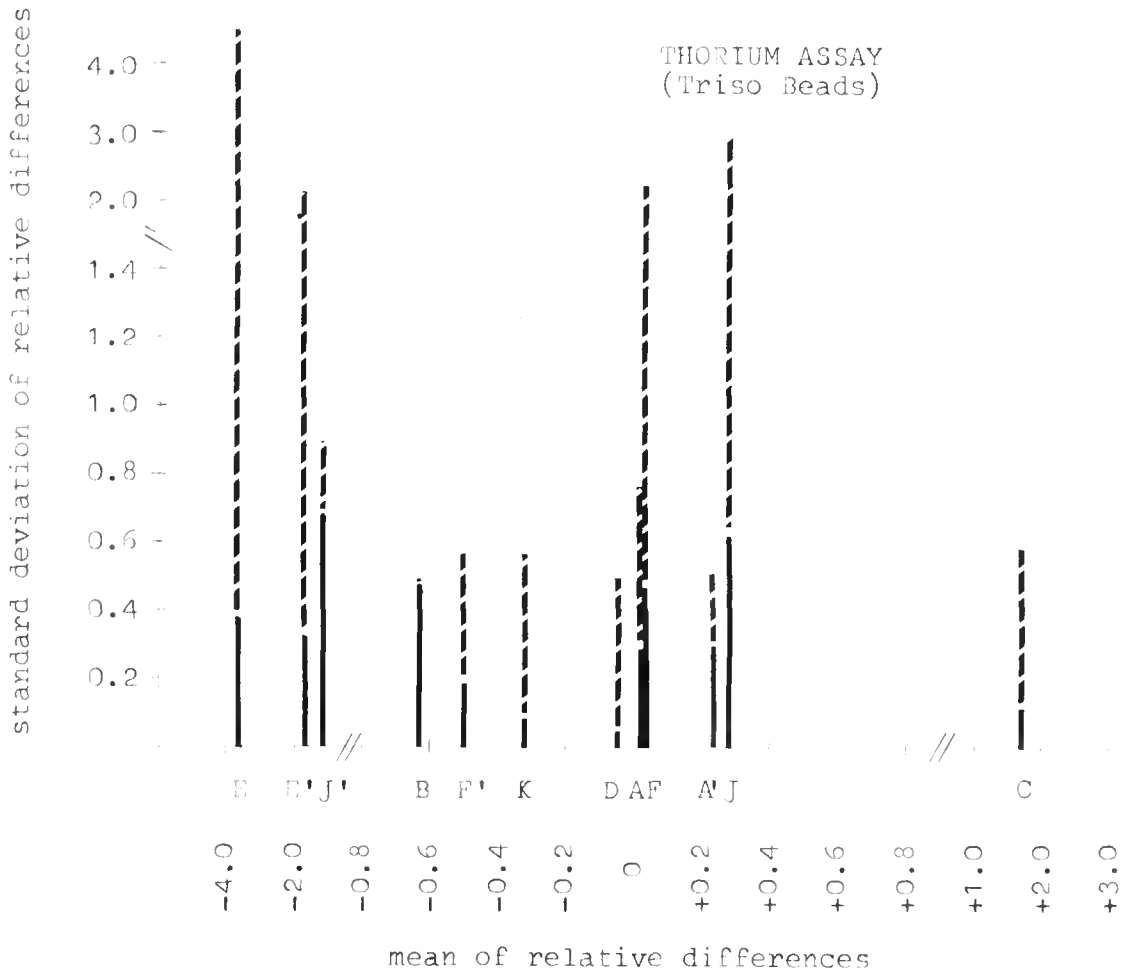
CF represents a carbonate fusion

For the uranium analyses of the TRISO beads, the method of preparation and the method of measurement were statistically significant contributions to the total variation. The method of preparation was the greater contribution to the total variation.

Thorium Analyses (Figure 11) - The assigned thorium value of the TRISO bead samples was 0.25754 ± 0.00034 (+0.133%) g Th/g samples. This is the mean and standard error of 44 values reported by laboratories A´, C´, D, E´ and K. The samples in laboratory J were not used in the calculations of the assigned value due to the extreme variation in the results.

Conclusions

Chemical Assay for Uranium - The 0:1 (ThO_2:UO_2) ratio reveals the need to adopt another method of quantitatively removing the powder mixture from the vials. It was somewhat disappointing that the sample transfer problem was not identified and reported earlier in the experiment since this was an obvious problem with visible traces of powder remaining in the vials. Even with the removal problem, the agreement between results obtained in this experiment improved by a factor of two over that observed in Phase I. Laboratories B and H were significantly less precise than the other laboratories. This was expected due to the inexperience of laboratory H with the measurement method utilized and due to the nature



Lab	Mean of R.D.	Total Variation S.D.	Total Variation df	Within Months Variation S.D.	Within Months Variation df	Among Months Variation S.D.	Among Months Variation df
A	0.02	0.76	17	0.28	9	0.75	8
A'	0.23	0.50	15	0.29	8	0.47	7
B	-0.63	0.48	39	0.47	30	0.24	9
C	1.68	0.58	19	0.11	10	0.59	9
D	-0.05	0.49	19	0.04	10	0.51	9
E	-3.67	4.47	19	0.37	10	4.58	9
E'	-1.69	2.09	15	0.32	8	2.15	7
F	0.02	2.20	17	0.46	9	2.24	8
F'	-0.50	0.56	13	0.18	7	0.56	6
J	0.28	2.94	9	0.61	5	3.09	4
J'	-1.06	0.89	7	0.68	4	0.79	3
K	-0.32	0.56	7	0.08	4	0.60	3

FIGURE 11

of x-ray fluorescence used in laboratory B. We might also note that laboratories B and H exhibited a lesser degree of accuracy than did the other laboratories.

The oxide results were quite impressive for the most part, with an average range over all ratios of less than 0.2% (excluding laboratories B and H). The improvement in the agreement between laboratory results for uranium in BISO and TRISO beads over that observed in Phase I is equally impressive, especially when different preparation methods were involved. It was consistently noted that the month-to-month (or sample-to-sample) variation contributed a significantly greater portion to the total variation than did the measurement errors. It remains to be resolved as to whether this large source of variation indicated sample preparation problems or time variation (random variation from month-to-month).

Chemical Analysis for Thorium - It is evident that the state-of-the-art practiced in the determination of thorium is not equivalent to that for the determination of uranium. This is due to the efforts made throughout the nuclear industry to upgrade the state-of-the-art in uranium chemistry for materials accountability and safeguards purposes.

Laboratories A, E, and F, all using similar methods of analysis (oxalate precipitation), exhibited consistent results on the oxides and BISO beads. Laboratories C and D using EDTA titration were not consistent with each other. Laboratory B (using x-ray) was consistently low. We might summarily conclude that oxalate precipitation yielded more consistent results in spite of laboratory differences than did EDTA titration. It should be noted that although the gravimetric (oxalate) procedure can be precisely performed, unbiased measurements are not guaranteed. The undetected presence of impurities is the most frequent cause of positive biases. Loss or incomplete precipitation is the most frequent cause of negative biases.

Nondestructive Analysis (NDA) for Uranium - The NDA work seemed to exhibit a strong degree of calibration dependence. A much lower level of precision than the wet chemistry was noted.

Recommendations for Further Study - A determination of Safeguards factors in the HTGR fuel cycle may require that additional questions be answered. If it is determined that a Phase III of the program is required, the experiment should be designed:

1. To determine whether the time of analysis introduced variations in observed results (month-to-month) variation or whether variations were due to sample-to-sample differences. Duplicate samples per month would be required.

2. To include carefully fabricated BISO beads → TRISO beads → fuel rods as necessary materials to adequately assess the contribution of the method of sample preparation.

Acknowledgment

The authors wish to acknowledge the efforts of the participating laboratories in providing timely responses. Thanks are due especially to the General Atomic Company for providing the materials necessary for the conduct of the experiment. Special gratitude is expressed to Agnes Bartley for typing and proofing the many drafts of this report.

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APPENDIX 1

CHARACTERIZATION OF URANIUM OXIDE AND THORIUM OXIDE

The materials received from GAC were acquired at nominal concentration values. It was necessary to assay them to assign prepared values to the individual vials. The contents of these vials were to serve as standards against which measurement accuracies were to be evaluated.

Uranium Dioxide

Samples of the UO_2 were weighed into each of 200 numbered vials in a random number sequence under constant humidity conditions. Twelve of these samples were dissolved and diluted to known volumes and the resulting solutions weighed. (The other 188 vials were set aside to be used in the experiment.) All subsequent aliquots were prepared on a weight basis.

The NBL titrimetric method was used by two different analysts to assay weighed aliquots for uranium. The manner in which the analysts prepared and analyzed these 12 samples is illustrated in Figure 12. The data from those analyses are presented in Tables II and III. A value of 0.87375 g U/g sample based upon the titrimetric assay was assigned to the UO_2 which had been weighed into the sample vials. Isotopic analysis by thermal-emission mass spectrometry was used to obtain a reference atomic weight. An enrichment of 93.276 wt % ^{235}U and an atomic weight of 235.21 were assigned to the uranium in the UO_2.

Thorium Dioxide

Samples of ThO_2 were weighed into each of 12 beakers under the same humidity conditions and at the same time as 153 other portions were added to the UO_2 in vials. The remaining 35 vials (188-153 = 35) were to be included in the experiment with no thorium oxide present to represent the 0:1 ratio of ThO_2 to UO_2. The samples were dissolved and diluted to known volumes and the resulting solutions weighed. All subsequent aliquots were taken on a weight basis.

Thorium was determined by precipitation of thorium as thorium oxalate and direct ignition to constant weight, weighed as thorium dioxide and corrected for spectrographically-determined impurities. Tables IV and V give comparative results. A value of 0.87825 g Th/g sample based on the oxalate precipitation was assigned to the ThO_2 which had been weighed into sample vials.

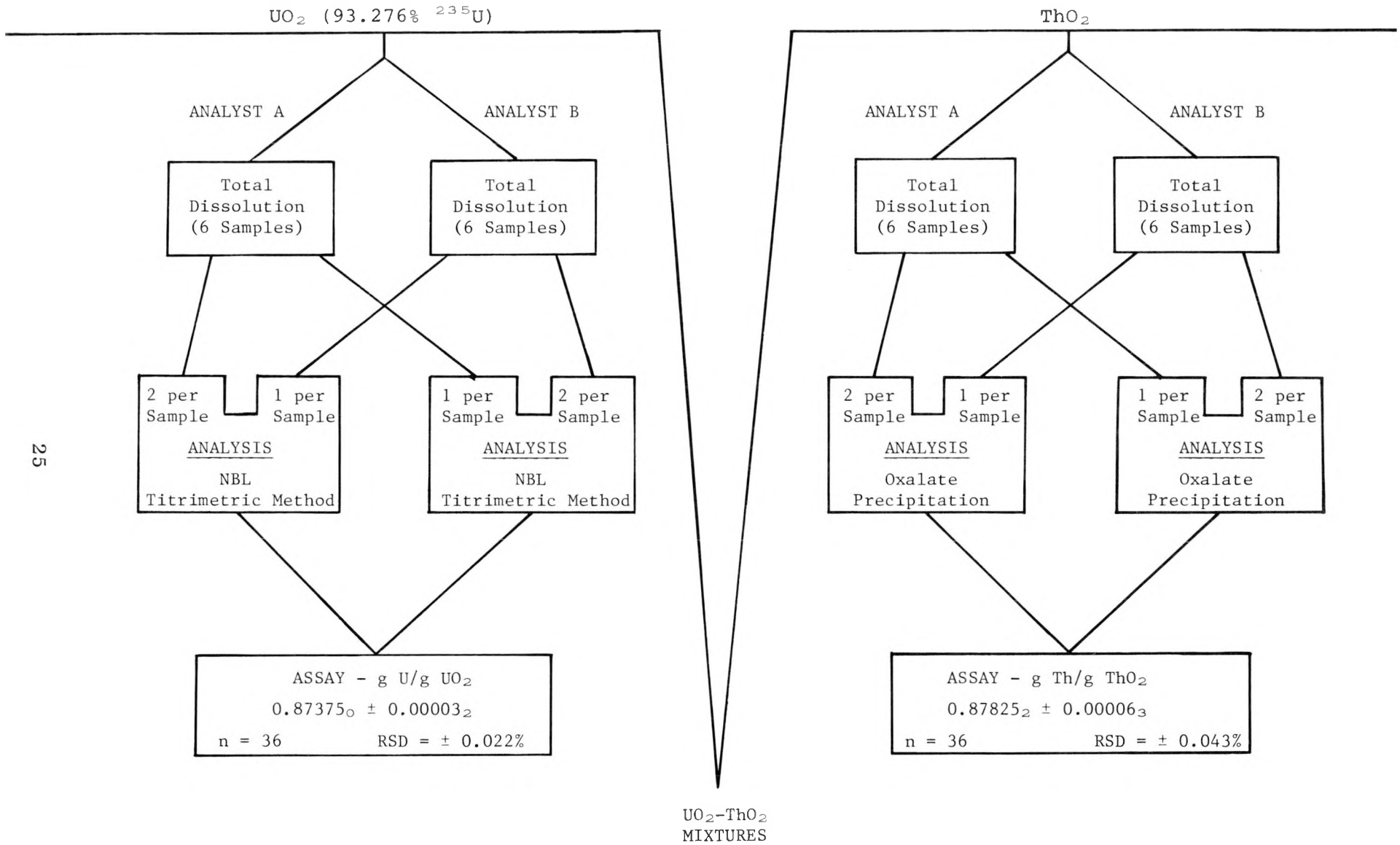

FIGURE 12. CHARACTERIZATION OF URANIUM AND THORIUM OXIDES.

TABLE II

ASSAY OF UO_2 FOR HTGR INTERLABORATORY COMPARISON PROGRAM - PHASE II

Sample	A/A[1]	A/B[1]	B/A[1]	B/B[1]
1	0.87389 0.87418	0.87395		
2			0.87384	0.87371 0.87364
3	0.87357 0.87372	0.87362		
4			0.87374	0.87337 0.87366
5	0.87379 0.87408	0.87376		
6			0.87372	0.87364 0.87352
7	0.87391 0.87400	0.87384		
8	0.87398 0.87361	0.87372		
9			0.87398	0.87341 0.87370
10			0.87387	0.87348 0.87357
11	0.87392 0.87377	0.87394		
12			0.87384	0.87340 0.87366

[1] A/B Represents solutions dissolved by Analyst A and analyzed by Analyst B.

TABLE III

(Summary of Table II)

ASSAY OF UO_2 FOR HTGR INTERLABORATORY COMPARISON PROGRAM - PHASE II

Analyzed by:	Prepared by: A	Prepared by: B	Prepared by: A and B
A	$\bar{x}$ = 0.873868 s = 0.000053 n = 12	$\bar{x}$ = 0.873832 s = 0.000038 n = 6	$\bar{x}$ = 0.873856 s = 0.000037 n = 18
B	$\bar{x}$ = 0.873805 s = 0.000053 n = 6	$\bar{x}$ = 0.873563 s = 0.000036 n = 12	$\bar{x}$ = 0.873644 s = 0.000040 n = 18
A and B	$\bar{x}$ = 0.873847 s = 0.000039 n = 18	$\bar{x}$ = 0.873653 s = 0.000040 n = 18	$\bar{x}$ = 0.873750 s = 0.000032 n = 36

s = Standard error of the mean.

TABLE IV

ASSAY OF ThO_2 FOR HTGR INTERLABORATORY COMPARISON PROGRAM - PHASE II

Sample	A/A[1]	A/B[1]	B/A[1]	B/B[1]
1	0.87801 0.87856		0.87840	
2		0.87848		0.87856 0.87831
3	0.87829 0.87847		0.87822	
4		0.87868		0.87829 0.87806
5	0.87846 0.87805		0.87827	
6		0.87869		0.87871 0.87853
7		0.87732		0.87765 0.87789
8	0.87812 0.87864		0.87821	
9	0.87890 0.87873		0.87854	
10		0.87762		0.87840 0.87813
11		0.87730		0.87790 0.87803
12	0.87804 0.87838		0.87822	

[1] A/B Represents solutions dissolved by Analyst A and analyzed by Analyst B.

TABLE V

(Summary of Table IV)

ASSAY OF ThO_2 FOR HTGR INTERLABORATORY COMPARISON PROGRAM - PHASE II

Analyzed by:	Prepared by: A	Prepared by: B	Prepared by: A and B
A	$\bar{x}$ = 0.878388 s = 0.000085 n = 12	$\bar{x}$ = 0.878310 s = 0.000054 n = 6	$\bar{x}$ = 0.878362 s = 0.000059 n = 18
B	$\bar{x}$ = 0.878015 s = 0.000275 n = 6	$\bar{x}$ = 0.878205 s = 0.000091 n = 12	$\bar{x}$ = 0.878142 s = 0.000107 n = 18
A and B	$\bar{x}$ = 0.878263 s = 0.000111 n = 18	$\bar{x}$ = 0.878240 s = 0.000063 n = 18	$\bar{x}$ = 0.878252 s = 0.000063 n = 36

s = Standard error of the mean.

APPENDIX 2

INSTRUCTIONS FOR PARTICIPANTS IN THE WET CHEMICAL ANALYSIS OF UO_2/ThO_2 MIXTURES AND $(U\text{-}Th)C_2$ BEADS

Ten packages and ten sets of data sheets, similarly lettered to facilitate identification, were provided. Each month of the program any one package was selected for analyses and the following instructions were completed:

Unpackaging - Remove the 4 vials from the package, carefully wipe each vial and weigh to the nearest 0.1 mg to obtain the gross weight as received. REPORT the GROSS WEIGHT of each vial on its corresponding data sheet. In case of a major discrepancy between your observed gross weight and the value provided by NBL, contact NBL (Nancy Trahey) for instructions or guidance.

Sampling and Analysis

UO_2/ThO_2 Samples

1. Unscrew the cap and empty the entire contents of each vial into a beaker of desired size.

2. Dislodge into the beaker as much powder as possible that is adhering to the vial and cap by rapping them smartly (careful, do not chip or break) with a wooden handle of a spatula or similar tool, reweigh the empty vial and cap and REPORT the NET WEIGHT of sample obtained.

3. Fill the vial about 1/3 full with distilled water, cap the vial, shake the vial vigorously, and empty the water into the beaker; repeat this treatment once more.

4. Consider the material in the beaker to be the analytical sample and dissolve the entire sample by your normal procedure.

5. Transfer the sample solution to a tared container and obtain the net weight of the solution.

6. Make 4 determinations of uranium and 2 of thorium on aliquots taken by weight and REPORT the results as GRAMS ELEMENT/VIAL.

7. Use an atomic weight of 235.21 for the uranium and 232.04 for the thorium.

BISO and TRISO (U-Th)C_2 Beads

1. Unscrew the cap and empty the entire contents of each vial into a beaker or dish of desired size.

2. Dislodge into the beaker or dish any remaining material using the technique employed on the oxides; reweigh the empty vial and cap and REPORT the NET WEIGHT of sample obtained.

3. Consider the material in the beaker or dish to be the analytical sample and dissolve the entire sample by your normal procedure.

4. Transfer the sample solution to a tared container and obtain the net weight of the solution.

5. Make 4 determinations of uranium and 2 of thorium on aliquots taken by weight and REPORT the results as GRAMS ELEMENT/GRAM sample (use the net weight observed in 2 above as the weight of the analyzed sample).

6. Use an atomic weight of 235.21 for the uranium and 232.04 for the thorium.

Data Reporting

Complete all information requested on each data sheet and RETURN the set WITHIN ONE MONTH OF UNPACKAGING to permit rapid turn around of monthly status report data.

NOTES: For the UO_2/ThO_2 mixtures, each vial contains about 0.35 g of UO_2 and from 4 to 9 g of ThO_2. The approximate uranium and thorium concentration in the BISO beads are 13% and >25%, respectively; in the TRISO beads, 6% and >25%, respectively.

APPENDIX 3

SUMMARY OF DISSOLUTION AND ANALYTICAL METHODS USED BY PARTICIPANTS

Laboratory	Dissolution Method[a] (1) UO_2-ThO_2, (2) BISO, (3) TRISO	Uranium Analytical Method[a] UO_2-ThO_2, BISO, TRISO	Thorium Analytical Method[a] UO_2-ThO_2, BISO, TRISO
A	1. AD 2. I; AD 3. Crush-burn-leach	T, NBL	G, oxalate precipitation
B	1. AD 2. I; AD 3. Crush-burn-leach	XRF, internal standard	XRF, internal standard
C	1. AD 2. I; crush; AD 3. Burn-crush-leach	T, NBL	T, EDTA-xylenol orange
D	1. AD 2. I; AD 3. High temperature chlorination	T, NBL	T, EDTA-xylenol orange SP, Arsenazo(III)
E	1. AD 2. I; AD 3. Carbonate fusion	T, NBL	G, oxalate precipitation
F(J&K)	1. AD 2. I; AD 3-J High temperature chlorination 3-K Crush-burn-leach	T, NBL	G, oxalate precipitation
G	--	NDA	--

a. AD = Acid Dissolution, HNO_3-HF
I = Ignition
T = Titrimetry
XRF = X-ray Fluorescence
NDA = Delayed Fusion Neutron Counting
G = Gravimetry
SP = Spectrophotometry

Laboratory A

Mixed oxide samples were dissolved in thorex solution (16M HNO_3 - 0.05M HF) and the resulting solution weighed.

BISO bead samples were weighed and burned at 900°C, then dissolved in thorex solution, diluted to about 300 ml and the solution weighed. TRISO beads were weighed into a tungsten carbide vial, crushed under alcohol in a mixer/mill, transferred to a 150-ml quartz beaker and fired overnight at 750-800°C. The material was leached for 4 hr with 25 ml thorex solution heated to near boiling then stirred at low temperature overnight. The beaker was again heated for 4 hr to near boiling, cooled and the solution decanted through No. 50 filter paper. Insolubles (SiC shards) remaining in the beaker were leached an additional 1-2 hr at near boiling with 15 ml thorex solution then filtered through the paper previously used and rinsed well with H_2O. The filtrates were combined, diluted to about 300 ml and weighed. The filter paper was charred and fired and the shards gamma counted to determine uranium holdup. Any uranium found was added to the final result.

Uranium was determined using the NBL titrimetric method on weighed aliquants of the sample solutions.

Thorium was determined on weighed solution aliquants by oxalate gravimetry.

Laboratory B

Mixed oxide samples were dissolved in 16M HNO_3 with F^- added, made to volume and weighed.

BISO bead samples were burned then dissolved in HNO_3-HF, made to volume and weighed. The TRISO beads were weighed into a tungsten carbide vial, crushed 10-15 min in a mixer/mill then transferred to a silica crucible and burned 4 hr (minimum) in air at 900°C. The oxidized material was leached at least 20 min at ~90°C with 45 ml HNO_3-HF. The cooled solution was filtered through No. 541 filter paper and the residue rinsed thoroughly with deionized H_2O. The filtrate and rinsings were set aside and the filter paper was burned off in a platinum crucible. The remaining residue was re-leached ~20 min with 15 ml HNO_3-HF and refiltered into the main solution which was then weighed. The filter paper was later checked for uranium content.

Uranium and thorium were determined using x-ray fluorescence by ratioing U and Th L_α lines to the Sr K_α line and comparing to a curve prepared from several standards bracketing the unknown concentration.

Laboratory C

Mixed oxide samples were dissolved in 13M HNO_3-0.05M HF, diluted to 100 ml and weighed.

Initally, the BISO and TRISO bead samples were weighed and placed in a 400°C furnace. After 15 min, the temperature was raised to 900°C and air (10-20 ml/min) was introduced and maintained overnight.

Evidence of sample losses during ignition caused the procedure to be modified. Bead samples were placed in a 100°C furnace and CO_2 (10-20 ml/min) was introduced. The temperature was raised 200° every 30 min until 900°C was reached and maintained overnight.

BISO beads were crushed in a mixer/mill, dissolved in 13M HNO_3-0.05M HF, diluted to 200 ml and weighed. TRISO beads were crushed in a mixer/mill then reignited at 900°C. The material remaining was leached with 50 ml of 13M HNO_3-0.05M HF at 80-90°C for 7 hr. The solution was filtered and the residue fused with sodium carbonate. The melt was dissolved in water, filtered, diluted to 100 ml and analyzed fluorometrically for uranium content. The filter paper was digested with HNO_3 and the resulting solution fumed to near dryness with dilute HF. The solution was combined with original filtrate, diluted to 200 ml and weighed.

Uranium was determined using the NBL titrimetric method on weighed sample solution aliquants. The equivalence point of each titration was reached by adding most of the dichromate from a weighed buret and the remainder by micrometric syringe.

Thorium was determined on weighed solution aliquants by titrating with 0.025N EDTA at pH 3 to a xylenol orange end point.

Laboratory D

Mixed oxides were dissolved in 8M HNO_3-0.05M HF, fumed, redissolved in 6M HCl and weighed.

BISO bead samples were weighed into platinum dishes, ignited at 700°C overnight then dissolved in 8M HNO_3-0.05M HF, fumed and redissolved in 6M HCl.

TRISO beads were weighed into a QFC combustion tube equipped with a quartz frit at the exit end. The tube was mounted vertically into the furnace and attached to a trap system consisting of one dry trap and four water traps. The furnace temperature was set at 950°C and oxygen (500 ml/min) was passed through the tube for 30 min. The oxygen was turned off and argon (200 ml/min) flowed through while the furnace temperature was raised to 1100-1200°C.

At temperature, chlorine (500 ml/min) was passed through for 1.5 hr. Argon was readmitted while the furnace cooled to 950°C and then oxygen passed through for another 30 min. The tube was removed from the furnace, cooled and the cap rinsed with 15 ml 8M HNO_3-0.05M HF. The rinsings were placed in an Erlenmeyer flask and heated to dissolve the sample residue in the tube by reflux action. The solution obtained was filtered through No. 42 filter paper as were the contents of the water traps. Each filter was ignited, the residues treated with HF and evaporated to dryness then redissolved in a small volume of 8M HNO_3-0.05M HF. The solutions were combined with the tube and trap filtrates (except trap No. 4) and evaporated to dryness. The residue was dissolved in HCl, evaporated to dryness again, redissolved in 6M HCl and weighed.

Uranium and thorium in weighed aliquants of the sample solutions were separated by anion exchange on AG 1-2X resin. Uranium was then determined by the NBL titrimetric method.

Thorium was determined by titrating with 0.1N EDTA at pH2 to a xylenol orange end point. The contents of trap No. 4 were analyzed for thorium by an Arsenazo(III) colorimetric method.

Laboratory E

Mixed oxide samples were dissolved in HNO_3-HF solution and weighed.

BISO bead samples were weighed into platinum dishes, ignited at 700°C overnight then dissolved in HNO_3-HF. TRISO beads were weighed into platinum dishes, ignited overnight at 700°C then fused with 35 g sodium carbonate for 6 hr. The fusion cake was transferred to a covered 1-l beaker and the dish rinsed with water containing a few drops H_2SO_4. The rinsings and 75 ml HNO_3 and 100 ml H_2SO_4 were carefully added to the beaker. After the reaction subsided, 75 ml HF was added and the solution heated overnight on a steam bath. Any carbon remaining was then removed by slowly fuming the solution while adding HNO_3 dropwise. The beaker was cooled, rinsed with water and the solution evaporated to strong fumes. The salts were dissolved with stirring in 700 ml water and filtered through No. 42 filter paper. The paper was rinsed with 1% H_2SO_4 then ignited in a vycor crucible at 700°C for 5 hr and the residue fused with 1-10 g sodium bisulfate. The fusion cake was dissolved in 300 ml water, filtered through No. 42 filter paper and combined with the original filtrate in a tared glass bottle and weighed.

Uranium was determined using the NBL titrimetric method on weighed aliquants of the sample solutions.

Thorium was determined on solution aliquants by oxalate gravimetry.

Laboratory F

Mixed oxide samples were dissolved in HNO_3-HF solution and weighed.

BISO bead samples were weighed into platinum dishes, ignited at 700°C overnight and dissolved in HNO_3-HF. TRISO bead samples were dissolved by two methods: crush-burn-leach; and high temperature chlorination. In the first method, beads were weighed into a motor-driven porcelain mortar equipped with a stainless steel pestle, slurried with kerosene and ground. The ground material was filtered on No. 42 filter paper to remove the kerosene, rinsed and the paper ignited at 800°C for 2 hr. The residue was then leached with HNO_3-HF solution at 80°-90°C overnight. The solution was cooled, diluted to 250 ml with water and filtered. The filter paper was burned off and the residue fused with 10 g sodium carbonate. Dissolution of the fusion cake was done with HNO_3, H_2SO_4 and HF. The solution was fumed to volatilize the silica. The solution and the original filtrate were combined and weighed.

In the second method, TRISO beads were weighed and loaded into a quartz tube fitted with quartz wool plugs. The tube was placed in a horizontal tube furnace at 900°-1000°C, and a stream of air (300 ml/min) was passed through for 2 hr. The furnace tempera-

ture was then dropped to 600°C and the tube removed and cooled to room temperature. The exit end was packed with a 15-cm length of activated charcoal separated from the beads by a quartz wool plug and the tube was reinserted into the furnace. As the temperature was being raised to 1000°C, chlorine (300-500 ml/min) was passed through the tube by means of a fresh piece of tubing fitted over the entrance end. Chlorination conditions were maintained for 45 min after 1000°C was reached; the furnace and chlorine were then turned off and the tube removed and cooled. From the exit end the plugs and charcoal were removed and placed in a 125-ml platinum dish. Small amounts of methanol were then used to wet the sample charge in tube and transfer the residue to the dish. The tube was dried before 25 ml 1:1 HNO_3 was added and sucked through to dissolve the U/Th chlorides. After the methanol was evaporated, the platinum dish was put into a furnace at 700°C and ignited overnight. When cool, the residue was dissolved in HNO_3-HF solution and filtered. The filtrate was added to the acid from the tube wash and the combined solution was weighed.

Uranium was determined by the NBL titrimetric method on weighed aliquants of the sample solutions.

Thorium was determined on weighed aliquants by oxalate gravimetry.

Laboratory G

All mixed oxide, BISO, and TRISO samples prepared for the evaluation program were analyzed for uranium by delayed fission neutron counting. Using a LASL Van de Graff small-sample assay system, the ^{235}U content of each vial was assayed twice over a three-week period and compared to uranium-thorium standards of prepared oxide and graphite mixtures. Total uranium in each sample was calculated using the isotopic weight provided by NBL.

APPENDIX 4

PHASE II - HTGR INTERLABORATORY COMPARISON PROGRAM

REPORT OF ANALYSES

Sample Number_____

Gross Wt._____(as prepared at NBL)

Gross Wt._____(observed in reporting lab)

Net Sample Wt. _____(observed in reporting lab)

URANIUM (use atomic weight - 235.21) Date of Analysis_____

g U/vial (oxide and beads)	g U/g sample (beads only - use your net wt.)
1._____	1._____
2._____	2._____
3._____	3._____
4._____	4._____

THORIUM (use atomic weight - 232.04) Date of Analysis_____

g Th/vial (oxide and beads)	g Th/g sample (beads only-use your net wt.)
1._____	1._____
2._____	2._____

Participating Laboratory_____

Responsible Individual _____

APPENDIX 5

TABULATION OF DATA

The following tables contain all of the raw data submitted in the HTGR Interlaboratory Comparison Program. In addition, per cent differences from prepared or assigned values are included. The codes in column 3 (Ratio) represent the ratio of thorium oxide to uranium oxide, i.e., 16 means 16:1. The B and T designators represent BISO or TRISO bead samples, respectively.

Note the large per cent errors in all laboratories on the observed net sample weight (column 5) for the 0:1 ratio oxide mixtures. These observed values in column 5 were calculated on the basis of the known per cent uranium and the reporting laboratories results for grams of uranium in a given vial.

TABLE VI

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
1	26	0	0.35081	0.3480	-0.80	0.30652	0.30658	0.02	0		
							0.30653	0			
							0.30579	-0.24			
							0.30532	-0.39			
	125	16	5.96677	5.9634	-0.06	0.31080	0.31042	-0.12	4.92791	4.9226	-0.11
							0.30885	-0.63		4.9316	0.07
							0.30849	-0.74			
							0.31059	-0.07			
	219	B	14.95446	14.9535	-0.01	0.13388	0.1337	-0.14	0.48298	0.4800	-0.62
							0.1334	-0.36		0.4794	-0.74
							0.1336	-0.21			
							0.1336	-0.21			
	288	T	15.07701	15.0726	-0.03	0.07128	0.07102	-0.36	0.25734	0.2536	-1.45
							0.07092	-0.50		0.2528	-1.76
							0.07101	-0.38			
							0.07102	-0.36			
2	51	25	9.10351	9.1010	-0.03	0.30499	0.30574	0.25	7.68860	7.6901	0.02
							0.30537	0.12		7.6868	-0.02
							0.30537	0.12			
							0.30587	0.29			
	149	10	3.86835	3.8669	-0.04	0.31007	0.31132	0.40	3.08571	3.0951	0.30
							0.31108	0.33		3.0932	0.24
							0.31120	0.36			
							0.31130	0.40			

TABLE VI (Continued)

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	237	B	15.01003	15.0092	-0.01	0.13388	0.1336	-0.21	0.48298	0.4836	0.13
							0.1336	-0.21		0.4841	0.23
							0.1335	-0.29			
							0.1336	-0.21			
	357	T	15.05113	15.0467	-0.03	0.07128	0.07135	0.10	0.25734	0.2583	0.37
							0.07129	0.02		0.2582	0.33
							0.07136	0.11			
							0.07136	0.11			
3	18	25	9.11595	9.1215	0.06	0.30758	0.30712	-0.15	7.69692	7.6646	-0.42
							0.30686	-0.23		7.6628	-0.44
							0.30695	-0.20			
							0.30709	-0.16			
	160	16	5.95035	5.9578	0.13	0.30334	0.30291	-0.14	4.92099	4.9302	0.19
							0.30299	-0.12		4.9217	0.01
							0.30304	-0.10			
							0.30290	-0.15			
	256	B	14.99808	14.9966	-0.01	0.13388	0.13368	-0.15	0.48298	0.4853	0.48
							0.13370	-0.14		0.4847	0.35
							0.13370	-0.14			
							0.13370	-0.14			
	313	T	15.23813	15.2334	-0.03	0.07128	0.07118	-0.14	0.25734	0.2602	1.11
							0.07117	-0.15		0.2597	0.92
							0.07116	-0.17			
							0.07116	-0.17			
4	31	25	9.10761	9.1078	0	0.30768	0.30614	-0.50	7.68949	7.7091	0.26
							0.30638	-0.42		7.7119	0.29
							0.30657	-0.36			
							0.30656	-0.36			

TABLE VI (Continued)

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
4	107	10	3.85502	3.8550	0	0.30132	0.30078	-0.18	3.08280	3.1011	0.59
							0.30122	-0.03		3.1007	0.58
							0.30061	-0.24			
							0.30075	-0.19			
	266	B	15.04588	15.0450	-0.01	0.13388	0.13380	-0.06	0.48298	0.4854	0.50
							0.13372	-0.12		0.4853	0.48
							0.13377	-0.08			
							0.13379	-0.07			
	292	T	15.10652	15.1021	-0.03	0.07128	0.07135	0.10	0.25734	0.2590	0.64
							0.07117	-0.15		0.2592	0.72
							0.07114	-0.19			
							0.07114	-0.19			
5	79	25	9.11639	9.1128	-0.04	0.31221	0.31318	0.31	7.69265	7.7178	0.33
							0.31350	0.41		7.7198	0.35
							0.31320	0.32			
							0.31340	0.38			
	103	16	5.96281	5.9658	0.05	0.30316	0.30493	0.58	4.93212	4.9222	-0.20
							0.30488	0.57		4.9494	0.35
							0.30465	0.49			
							0.30433	0.39			
	213	B	14.98290	14.9828	0	0.13388	0.1340	0.09	0.48298	0.4843	0.27
							0.1340	0.09		0.4846	0.33
							0.1339	0.01			
							0.1340	0.09			
	309	T	Bumped on the hotplate								

TABLE VI (Continued)

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
6	67	25	9.10649	9.1035	-0.03	0.31117	0.31206	0.29	7.68500	7.6900	0.07
							0.31144	0.09		7.6920	0.09
							0.31192	0.24			
							0.31164	0.15			
	73	10	3.85757	3.8577	0	0.30473	0.30412	-0.20	3.08159	3.0911	0.31
							0.30409	-0.21		3.0893	0.25
							0.30424	-0.16			
							0.30419	-0.18			
	206	B	14.76961	14.7689	0	0.13388	0.1340	0.09	0.48298	0.4828	-0.04
							0.1340	0.09		0.4831	0.02
							0.1340	0.09			
							0.1340	0.09			
	314	T	14.76004	14.7558	-0.03	0.07128	0.07125	-0.04	0.25734	0.2579	0.22
							0.07121	-0.10		0.2577	0.14
							0.07124	-0.05			
							0.07129	0.02			
7	56	16	5.95737	5.9553	-0.03	0.30969	0.30960	-0.03	4.92077	4.9321	0.23
							0.30947	-0.07		4.9339	0.27
							0.30931	-0.12			
							0.30934	-0.11			
	148	0	0.35095	0.3475	-0.98	0.30664	0.30606	-0.19	0		
							0.30567	-0.32			
							0.30589	-0.24			
							0.30616	-0.16			
	201	B	14.83363	14.8324	-0.01	0.13388	0.1337	-0.14	0.48298	0.4824	-0.12
							0.1336	-0.21		0.4816	-0.29
							0.1336	-0.21			
							0.1337	-0.14			

TABLE VI (Continued)

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
7	297	T	14.71434	14.7100	-0.03	0.07128	0.07112	-0.22	0.25734	0.2569	-0.17
							0.07110	-0.25		0.2581	0.30
							0.07110	-0.25			
							0.07109	-0.26			
8	36	16	5.95450	5.9628	0.14	0.30593	0.30538	-0.18	4.92203	4.9418	0.40
							0.30542	-0.17		4.9447	0.46
							0.30535	-0.19			
							0.30544	-0.16			
	90	0	0.34772	0.3428	-1.41	0.30382	0.30153	-0.75	0		
							0.30205	-0.58			
							0.30177	-0.67			
							0.30202	-0.59			
	259	B	15.05757	15.0573	0	0.13388	0.1337	-0.14	0.48298	0.4857	0.56
							0.1338	-0.06		0.4855	0.52
							0.1336	-0.21			
							0.1338	-0.06			
	335	T	14.56989	14.5651	-0.03	0.07128	0.07132	0.06	0.25734	0.2562	-0.44
							0.07135	0.10		0.2586	0.49
							0.07136	0.11			
							0.07135	0.10			
9	25	10	3.84880	3.8491	0.01	0.31296	0.31312	0.05	3.06564	3.0945	0.94
							0.31325	0.09		3.0951	0.96
							0.31290	-0.02			
							0.31301	0.02			
	117	25	9.11855	9.1219	0.04	0.30449	0.30406	-0.14	7.70231	7.7004	-0.02
							0.30392	-0.19		7.6992	-0.04
							0.30387	-0.20			
							0.30363	-0.28			

TABLE VI (Continued)

LABORATORY A

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
9	247	B	14.60995	14.6089	-0.01	0.13388	0.1336	-0.21	0.48298	0.4858	0.58
							0.1337	-0.14		0.4823	-0.14
							0.1336	-0.21			
							0.1337	-0.14			
	328	T	14.34517	14.3400	-0.04	0.07128	0.07139	0.16	0.25734	0.2569	-0.17
							0.07133	0.07		0.2579	0.22
							0.07134	0.09			
							0.07131	0.04			
10	82	16	5.96098	5.9658	0.08	0.30508	0.30413	-0.31	4.92858	4.9376	0.18
							0.30396	-0.37		4.9395	0.22
							0.30406	-0.33			
							0.30415	-0.30			
	106	0	0.35754	0.3533	-1.19	0.31240	0.31086	-0.49	0		
							0.31104	-0.44			
							0.31102	-0.44			
							0.31118	-0.39			
	242	B	14.91904	14.9186	0	0.13388	0.1336	-0.21	0.48298	0.4832	0.04
							0.1335	-0.29		0.4830	0
							0.1334	-0.36			
							0.1335	-0.29			
	347	T	14.89431	14.8892	-0.03	0.07128	0.07120	-0.11	0.25734	0.2562	-0.44
							0.07120	-0.11		0.2557	-0.64
							0.07118	-0.14			
							0.07119	-0.12			

TABLE VII

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
1	65	16	5.96747	5.9699	0.04	0.30918	0.3109	0.56	4.93016	4.9841	1.09
							0.3111	0.62		4.9732	0.87
							0.3107	0.49		4.9569	0.54
							0.3121	0.94		4.9747	0.90
	129	10	3.85803	3.8575	-0.01	0.31098	0.3073	-1.18	3.07574	3.0890	0.43
							0.3100	-0.32		3.1016	0.84
							0.3087	-0.73		3.0809	0.17
							0.3087	-0.73		3.0751	-0.02
	211	B	15.29519	15.2944	-0.01	0.13388	0.1330	-0.66	0.48298	0.4792	-0.78
							0.1344	0.39		0.4841	0.23
							0.1334	-0.36		0.4815	-0.31
							0.1343	0.31		0.4825	-0.10
	282	T	15.05774	15.0529	-0.03	0.07128	0.0707	-0.81	0.25734	0.2542	-1.22
							0.0706	-0.95		0.2558	-0.60
							0.0710	-0.39		0.2564	-0.36
							0.0705	-1.09		0.2536	-1.45
2	41	25	9.10910	9.1108	0.02	0.30607	0.3100	1.28	7.69242	7.6759	-0.21
							0.3088	0.89		7.6475	-0.58
							0.3064	0.11		7.6604	-0.42
							0.3093	1.06		7.6845	-0.10
	47	16	5.96261	5.9608	-0.03	0.30713	0.3051	-0.66	4.92795	4.9155	-0.25
							0.3058	-0.43		4.9382	0.21
							0.3031	-1.31		4.8950	-0.67
							0.3081	0.32		4.9524	0.50

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	249	B	14.77152	14.7703	-0.01	0.13388	0.1328	-0.81	0.48298	0.4816	-0.29
							0.1313	-1.93		0.4800	-0.62
							0.1331	-0.58		0.4831	0.02
							0.1331	-0.58		0.4837	0.15
	334	T	14.72686	14.7218	-0.03	0.07128	0.07104	-0.33	0.25734	0.2549	-0.95
							0.07142	0.20		0.2590	0.64
							0.07091	-0.52		0.2550	-0.91
							0.07140	0.17		0.2577	0.14
3	137	0	0.35222	0.3530	0.22	0.30775	0.3076	-0.05	0	0.000163	
							0.3089	0.37		0.000124	
							0.3117	1.28			
							0.3092	0.47			
	154	16	5.95444	5.9565	0.03	0.30522	0.3012	-1.32	4.92270	4.8575	-1.32
							0.3052	-0.01		4.8909	-0.65
							0.3043	-0.30		4.8519	-1.44
							0.3029	-0.76		4.8853	-0.76
	269	B	14.81832	14.8173	-0.01	0.13388	0.1334	-0.36	0.48298	0.4819	-0.23
							0.1337	-0.14		0.4831	0.02
							0.1337	-0.14		0.4864	0.70
							0.1338	-0.06		0.4850	0.42
	291	T	14.67919	14.6746	-0.03	0.07128	0.07075	-0.74	0.25734	0.2541	-1.26
							0.07083	-0.63		0.2566	-0.29
							0.07112	-0.22		0.2590	0.64
							0.07063	-0.91		0.2573	-0.02

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
4	39	0	0.34678	0.3468	0.01	0.30300	0.2995	-1.16	0	0.000033	
							0.3000	-0.99			
							0.2994	-1.19			
							0.3007	-0.76			
	109	25	9.11645	9.1191	0.03	0.30922	0.3116	0.77	7.69571	7.6665	-0.38
							0.3109	0.54		7.6514	-0.58
							0.3080	-0.39		7.6379	-0.75
							0.3104	0.38		7.6514	-0.58
	234	B	14.76561	14.7646	-0.01	0.13388	0.1316	-1.71	0.48298	0.4766	-1.32
							0.1327	-0.88		0.4770	-1.24
							0.1316	-1.71		0.4763	-1.39
							0.1320	-1.41		0.4767	-1.30
	296	T	14.57124	14.5662	-0.03	0.07128	0.07075	-0.74	0.25734	0.2570	-0.13
							0.07089	-0.54		0.2556	-0.68
							0.07051	-1.08		0.2557	-0.64
							0.07100	-0.39		0.2555	-0.71
5	98	0	0.34939	0.3470	-0.68	0.30528	0.3056	0.10	0		
							0.3035	-0.58			
							0.3056	0.10			
							0.3047	-0.19			
	172	16	5.96112	5.9605	-0.01	0.30690	0.3081	0.39	4.92688	4.8862	-0.83
							0.3097	0.91		4.9103	-0.34
							0.3073	0.13		4.9097	-0.35
							0.3065	-0.13		4.8912	-0.72

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
5	244	B	15.18401	15.1828	-0.01	0.13388	0.1332	-0.51	0.48298	0.4780	-1.03
							0.1335	-0.29		0.4808	-0.45
							0.1343	0.31		0.4830	0
							0.1337	-0.14		0.4818	-0.25
	308	T	15.27556	15.2705	-0.03	0.07128	0.07120	-0.11	0.25734	0.2570	-0.13
							0.07099	-0.40		0.2551	-0.87
							0.07080	-0.67		0.2556	-0.68
							0.07119	-0.12		0.2558	-0.60
6	114	16	5.95372	5.9588	0.09	0.30272	0.3010	-0.57	4.92458	4.8805	-0.90
							0.3033	0.19		4.9014	-0.47
							0.3015	-0.40		4.9214	-0.06
							0.3012	-0.50		4.9097	-0.30
	169	10	3.85680	3.8610	0.11	0.30808	0.3057	-0.77	3.07757	3.0632	-0.47
							0.3079	-0.06		3.0758	-0.06
							0.3070	-0.35		3.0816	0.13
							0.3069	-0.38		3.0687	-0.29
	279	B	14.92191	14.9216	0	0.13388	0.1334	-0.36	0.48298	0.4821	-0.18
							0.1330	-0.66		0.4800	-0.62
							0.1327	-0.88		0.4790	-0.83
							0.1332	-0.51		0.4828	-0.04
	337	T	15.04215	15.0372	-0.03	0.07128	0.07085	-0.60	0.25734	0.2543	-1.18
							0.07089	-0.54		0.2548	-0.99
							0.07113	-0.21		0.2570	-0.13
							0.07085	-0.60		0.2571	-0.09

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
7	86	16	5.95798	5.9614	0.06	0.30063	0.3010	0.12	4.93039	4.8985	-0.65
							0.3010	0.12		4.9119	-0.38
							0.3034	0.92		4.9205	-0.20
							0.3036	0.99		4.9298	-0.01
	173	0	0.35258	0.3551	0.71	0.30807	0.3087	0.20	0		
							0.3075	-0.19			
							0.3080	-0.02			
							0.3082	0.04			
	216	B	15.18432	15.1837	0	0.13388	0.1338	-0.06	0.48298	0.4809	-0.43
							0.1339	0.01		0.4827	-0.06
							0.1344	0.39		0.4819	-0.23
							0.1340	0.09		0.4832	0.04
	333	T	15.12205	15.1174	-0.03	0.07128	0.07119	-0.12	0.25734	0.2556	-0.68
							0.07128	0		0.2554	-0.75
							0.07105	-0.32		0.2558	-0.60
							0.07086	-0.59		0.2542	-1.22
8	22	25	9.08911	9.1160	0.30	0.30560	0.3068	0.39	7.67533	7.6334	-0.55
							0.3051	-0.16		7.6215	-0.70
							0.3077	0.69		7.6289	-0.60
							0.3079	0.75		7.6441	-0.41
	60	25	9.22645	9.2341	0.08	0.30534	0.3080	0.87	7.79622	7.7479	-0.62
							0.3105	1.69		7.7670	-0.37
							0.3066	0.41		7.7414	-0.70
							0.3081	0.90		7.7496	-0.60

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
8	203	B	14.90720	14.9199	0.09	0.13388	0.1316	-1.71	0.48298	0.4753	-1.59
							0.1321	-1.33		0.4766	-1.32
							0.1311	-2.08		0.4756	-1.53
							0.1312	-2.00		0.4752	-1.61
	289	T	14.79141	14.8013	0.07	0.07128	0.07061	-0.94	0.25734	0.2543	-1.18
							0.07041	-1.22		0.2549	-0.95
							0.07038	-1.26		0.2545	-1.10
							0.07067	-0.85		0.2559	-0.56
9	70	25	9.11087	9.1309	0.22	0.30564	0.3061	0.15	7.69441	7.6199	-0.97
							0.3059	0.09		7.6494	-0.58
							0.3056	-0.01		7.5639	-1.70
							0.3050	-0.21		7.5878	-1.39
	89	10	3.86124	3.8778	0.43	0.30884	0.3087	-0.05	3.08070	3.0361	-1.45
							0.3091	0.08		3.0645	-0.53
							0.3093	0.15		3.0618	-0.61
							0.3083	-0.17		3.0736	-0.23
	241	B	15.26412	15.2782	0.09	0.13388	0.1338	-0.06	0.48298	0.4816	-0.29
							0.1341	0.16		0.4830	0
							0.1339	0.01		0.4841	0.23
							0.1342	0.24		0.4815	-0.31
	305	T	14.47629	14.4865	0.07	0.07128	0.0709	-0.53	0.25734	0.2552	-0.83
							0.0713	0.03		0.2559	-0.56
							0.0711	-0.25		0.2548	-0.99
							0.0712	-0.11		0.2557	-0.64

TABLE VII (Continued)

LABORATORY B

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
10	38	10	3.85738	3.8770	0.51	0.30687	0.3064	-0.15	3.07929	3.0570	-0.72
							0.3074	0.17		3.0560	-0.76
							0.3065	-0.12		3.0581	-0.69
							0.3079	0.34		3.0558	-0.76
	124	25	9.10524	9.1340	0.32	0.30749	0.3087	0.39	7.68760	7.5790	-1.41
							0.3064	-0.35		7.6369	-0.66
							0.3073	-0.06		7.6288	-0.76
							0.3087	0.39		7.6625	-0.33
	225	B	14.72815	14.7384	0.07	0.13388	0.1345	0.46	0.48298	0.4815	-0.31
							0.1346	0.54		0.4823	-0.14
							0.1339	0.01		0.4817	-0.27
							0.1337	-0.14		0.4815	-0.31
	326	T	15.12792	15.1333	0.04	0.07128	0.07090	-0.53	0.25734	0.2553	-0.79
							0.07080	-0.67		0.2551	-0.88
							0.07105	-0.32		0.2562	-0.44
							0.07119	-0.12		0.2562	-0.44

TABLE VIII

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
1	29	25	9.08787	9.0840	-0.04	0.30801	0.30662	-0.45	7.67182	7.6860	0.18
							0.30723	-0.25		7.6973	0.33
							0.30693	-0.35			
							0.30661	-0.45			
	157	16	5.95929	5.9570	-0.04	0.30767	0.30761	-0.02	4.92449	4.9468	0.45
							0.30767	0		4.9567	0.65
							0.30815	0.16			
							0.30768	0			
	254	B	15.19053	15.1898	0	0.13388	0.13348	-0.30	0.48298	0.47951	-0.72
							0.13346	-0.32		0.47937	-0.75
							0.13345	-0.32			
							0.13341	-0.35			
	315	T	15.42544	15.4197	-0.04	0.07128	0.071785	0.71	0.25734	0.26097	1.41
							0.071778	0.70		0.26053	1.24
							0.071681	0.56			
							0.071817	0.75			
2	14	16	5.95808	5.9541	-0.07	0.31035	0.31056	0.07	4.92074	4.9475	0.54
							0.31059	0.08		4.9525	0.65
							0.31039	0.01			
							0.31057	0.07			
	142	0	0.35050	0.3441	-1.83	0.30625	0.30643	0.06	0		
							0.30637	0.04			
							0.30593	-0.10			
							0.30579	-0.15			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	250	B	14.58871	14.5876	-0.01	0.13388	0.13413	0.18	0.48298	0.48436	0.28
							0.13423	0.26		0.48510	0.44
							0.13414	0.19			
							0.13416	0.21			
	331	T	15.02032	15.0158	-0.03	0.07128	0.071485	0.29	0.25734	0.26296	2.18
							0.071471	0.27		0.26290	2.16
							0.071505	0.32			
							0.071505	0.32			
3	21	10	3.85466	3.8500	-0.12	0.30680	0.30666	-0.05	3.07698	3.1122	1.14
							0.30667	-0.04		3.1081	1.01
							0.30683	0.01			
							0.30706	0.08			
	115	25	9.11367	9.1091	-0.05	0.30866	0.30906	0.13	7.69383	7.7319	0.49
							0.30908	0.14		7.7117	0.23
							0.30894	0.09			
							0.30901	0.11			
	277	B	14.93480	14.9338	-0.01	0.13388	0.13358	-0.23	0.48298	0.48232	-0.14
							0.13353	-0.26		0.48161	-0.29
							0.13356	-0.24			
							0.13362	-0.20			
	320	T	15.03515	15.0304	-0.03	0.07128	0.071628	0.49	0.25734	0.26090	1.38
							0.071595	0.44		0.26101	1.43
							0.071608	0.46			
							0.071635	0.50			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
4	62	25	9.11151	9.1183	0.07	0.30993	0.30999	0.02	7.69066	7.7453	0.71
							0.30956	-0.12		7.7442	0.70
							0.31022	0.09			
							0.31004	0.04			
	143	10	3.86252	3.8645	0.05	0.31013	0.30988	-0.08	3.08053	3.1193	1.26
							0.31051	0.12		3.1186	1.24
							0.31024	0.04			
							0.31042	0.09			
	245	B	15.01989	15.0187	-0.01	0.13388	0.13239	-1.12	0.48298	0.48181	-0.24
							0.13230	-1.18		0.48187	-0.23
							0.13249	-1.04			
							0.13233	-1.16			
	384	T	15.38446	15.3790	-0.04	0.07128	0.071500	0.31	0.25734	0.26265	2.06
							0.071669	0.55		0.26285	2.14
							0.071533	0.36			
							0.071507	0.32			
5	13	0	0.35408	0.3321	-6.21	0.30938	0.30755	-0.59	0		
							0.30762	-0.57			
							0.30756	-0.59			
							0.30752	-0.60			
	120	16	5.96062	5.9587	-0.03	0.31060	0.30917	-0.46	4.92271	4.9559	0.67
							0.30949	-0.36		4.9550	0.66
							0.30933	-0.41			
							0.30957	-0.33			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
5	274	B	15.00043	14.9993	-0.01	0.13388	0.12873	-3.85	0.48298	0.46660	-3.39
							0.12871	-3.86		0.46686	-3.34
							0.12862	-3.93			
							0.12870	-3.87			
	343	T	14.65812	14.6538	-0.03	0.07128	0.071940	0.93	0.25734	0.26423	2.68
							0.072002	1.01		0.26481	2.90
							0.071940	0.93			
							0.072036	1.06			
6	92	16	5.96941	5.9654	-0.07	0.31078	0.31157	0.25	4.93026	5.0145	1.71
							0.31134	0.18		5.0152	1.72
							0.31134	0.18			
							0.31147	0.22			
	101	0	0.35255	0.3292	-6.62	0.30804	0.30736	-0.22	0		
							0.30757	-0.15			
							0.30749	-0.18			
							0.30749	-0.18			
	230	B	14.85571	14.8550	0	0.13388	0.13097	-2.18	0.48298	0.47377	-1.91
							0.13096	-2.18		0.47363	-1.94
							0.13095	-2.19			
							0.13092	-2.21			
	306	T	15.18100	15.1767	-0.03	0.07128	0.071768	0.69	0.25734	0.26250	2.00
							0.071794	0.72		0.26259	2.04
							0.071761	0.68			
							0.071748	0.66			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
7	16	25	9.10221	9.1102	0.09	0.30373	0.30552	0.59	7.68872	7.6832	-0.07
							0.30543	0.56		7.6641	-0.32
							0.30509	0.45			
							0.30528	0.51			
	77	16	5.95629	5.9550	-0.02	0.30243	0.30237	-0.02	4.92712	4.9083	-0.38
							0.30302	0.20		4.9110	-0.33
							0.30242	0			
							0.30286	0.14			
	208	B	14.82855	14.8277	-0.01	0.13388	0.13387	-0.01	0.48298	0.48270	-0.06
							0.13390	0.01		0.48302	0.01
							0.13365	-0.17			
							0.13390	0.01			
	304	T	14.75850	14.7542	-0.03	0.07128	0.071437	0.22	0.25734	0.26165	1.67
							0.071437	0.22		0.26119	1.50
							0.071444	0.23			
							0.071424	0.20			
8	45	25	9.10791	9.1065	-0.02	0.30839	0.30843	0.01	7.68904	7.7390	0.65
							0.30856	0.06		7.7755	1.12
							0.30840	0			
							0.30836	-0.01			
	100	10	3.83619	3.8346	-0.04	0.30449	0.30429	-0.07	3.06307	3.1091	1.50
							0.30450	0		3.1037	1.33
							0.30450	0			
							0.30436	-0.04			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
8	248	B	15.37284	15.3723	0	0.13388	0.13404	0.12	0.48298	0.48778	0.99
							0.13401	0.09		0.48793	1.03
							0.13401	0.09			
							0.13401	0.09			
	356	T	14.83568	14.8314	-0.03	0.07128	0.071295	0.02	0.25734	0.26122	1.51
							0.071409	0.18		0.26107	1.45
							0.071328	0.07			
							0.071436	0.22			
9	52	16	5.94380	5.9417	-0.04	0.30490	0.30485	-0.02	4.91368	4.9216	0.16
							0.30520	0.10		4.9184	0.10
							0.30502	0.04			
							0.30461	-0.10			
	74	25	9.10907	9.1132	0.05	0.30165	0.30122	-0.14	7.69683	7.7018	0.06
							0.30115	-0.17		7.7288	0.42
							0.30138	-0.09			
							0.30138	-0.09			
	252	B	14.91289	14.9121	-0.01	0.13388	0.13394	0.04	0.48298	0.48351	0.11
							0.13403	0.11		0.48441	0.29
							0.13403	0.11			
							0.13399	0.08			
	327	T	14.60689	14.6021	-0.03	0.07128	0.071332	0.07	0.25734	0.26002	1.04
							0.071476	0.28		0.25963	0.89
							0.071325	0.06			
							0.071408	0.18			

TABLE VIII (Continued)

LABORATORY C

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
10	49	0	0.34943	0.3373	-3.47	0.30531	0.30421	-0.36	0		
							0.30436	-0.31			
							0.30427	-0.34			
							0.30441	-0.29			
	64	10	3.86512	3.8628	-0.06	0.30785	0.30753	-0.10	3.08511	3.0996	0.47
							0.30767	-0.06		3.1018	0.54
							0.30772	-0.04			
							0.30756	-0.09			
	229	B	14.63977	14.6388	-0.01	0.13388	0.13389	0.01	0.48298	0.48629	0.68
							0.13399	0.08		0.48629	0.68
							0.13400	0.09			
							0.13401	0.09			
	351	T	14.86472	14.8601	-0.03	0.07128	0.071440	0.23	0.25734	0.26015	1.09
							0.071426	0.21		0.25928	0.75
							0.071399	0.17			
							0.071433	0.22			

TABLE IX

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
1	57	16	5.95575	5.95233	-0.06	0.30885	0.30866	-0.06	4.92019	4.943	0.46
							0.30855	-0.10		4.944	0.48
							0.30880	-0.02			
							0.30851	-0.11			
	95	0	0.34564	0.33844	-2.08	0.30200	0.30154	-0.15	0	0.000234	
							0.30165	-0.12		0.000240	
							0.30157	-0.14			
							0.30165	-0.12			
	278	B	14.82216	14.82167	0	0.13388	0.13392	0.03	0.48298	0.4839	0.19
							0.13397	0.06		0.4882	1.08
							0.13392	0.03			
							0.13393	0.04			
	342	T	14.75212	14.74969	-0.02	0.07128	0.07139	0.16	0.25734	0.2588	0.57
							0.07139	0.16		0.2587	0.53
							0.07145	0.24			
							0.07145	0.24			
2	19	25	9.09675	9.09470	-0.02	0.30802	0.30765	-0.12	7.67961	7.720	0.53
							0.30767	-0.11		7.714	0.45
							0.30759	-0.14			
							0.30784	-0.06			
	145	16	5.97108	5.96756	-0.06	0.30843	0.30859	0.05	4.93408	4.953	0.38
							0.30857	0.05		4.953	0.38
							0.30841	-0.01			
							0.30837	-0.02			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	202	B	15.01839	15.01850	0	0.13388	0.13393	0.04	0.48298	0.4840	0.21
							0.13384	-0.03		0.4843	0.27
							0.13387	-0.01			
							0.13389	0.01			
	319	T	15.11698	15.11357	-0.02	0.07128	0.07131	0.04	0.25734	0.2582	0.33
							0.07130	0.03		0.2582	0.33
							0.07132	0.06			
							0.07135	0.10			
3	80	25	9.10034	9.09880	-0.02	0.30777	0.30858	0.26	7.68302	7.697	0.18
							0.30842	0.21		7.699	0.21
							0.30838	0.20			
							0.30853	0.25			
	84	10	3.85260	3.85406	0.04	0.30857	0.30923	0.21	3.07338	3.077	0.12
							0.30913	0.18		3.077	0.12
							0.30922	0.21			
							0.30936	0.26			
	214	B	14.99360	14.99547	0.01	0.13388	0.13400	0.09	0.48298	0.4841	0.23
							0.13407	0.14		0.4842	0.25
							0.13406	0.13			
							0.13399	0.08			
	301	T	15.05505	15.05158	-0.02	0.07128	0.07124	-0.05	0.25734	0.2579	0.22
							0.07124	-0.05		0.2578	0.18
							0.07123	-0.07			
							0.07123	-0.07			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
4	83	16	5.96293	5.96102	-0.03	0.30134	0.30086	-0.16	4.93405	4.930	-0.08
							0.30095	-0.13		4.931	-0.06
							0.30093	-0.14			
							0.30090	-0.15			
	158	0	0.35096	0.33533	-4.45	0.30665	0.30650	-0.05	0	0.000336	
							0.30644	-0.07		0.000426	
							0.30654	-0.04			
							0.30649	-0.05			
	233	B	14.73027	14.72996	0	0.13388	0.13406	0.13	0.48298	0.4832	0.04
							0.13404	0.12		0.4837	0.15
							0.13404	0.12			
							0.13406	0.13			
	336	T	14.84492	14.84122	-0.02	0.07128	0.07128	0	0.25734	0.2574	0.02
							0.07129	0.02		0.2575	0.06
							0.07127	-0.01			
							0.07129	0.02			
5	32	25	9.10691	9.10870	0.02	0.30767	0.30726	-0.13	7.68889	7.685	-0.05
							0.30724	-0.14		7.689	0
							0.30727	-0.13			
							0.30733	-0.11			
	161	16	5.95058	5.95070	0	0.31106	0.31089	-0.05	4.91343	4.906	-0.15
							0.31102	-0.01		4.909	-0.09
							0.31108	0.01			
							0.31087	-0.06			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
5	255	B	14.73452	14.73473	0	0.13388	0.13384	-0.03	0.48298	0.4839	0.19
							0.13384	-0.03		0.4841	0.23
							0.13385	-0.02			
							0.13384	-0.03			
	285	T	14.77998	14.77700	-0.02	0.07128	0.07126	-0.03	0.25734	0.2574	0.02
							0.07122	-0.08		0.2575	0.06
							0.07123	-0.07			
							0.07126	-0.03			
6	104	16	5.96672	5.96782	0.02	0.30545	0.30447	-0.32	4.93325	4.966	0.66
							0.30455	-0.29		4.966	0.66
							0.30447	-0.32			
							0.30448	-0.32			
	111	0	0.35152	0.34341	-2.31	0.30714	0.30656	-0.19	0	0.000178	
							0.30652	-0.20		0.000245	
							0.30654	-0.20			
							0.30655	-0.19			
	257	B	15.14716	15.14842	0.01	0.13388	0.13397	0.06	0.48298	0.4824	-0.12
							0.13403	0.11		0.4822	-0.16
							0.13394	0.04			
							0.13396	0.06			
	340	T	14.98137	14.97829	-0.02	0.07128	0.07132	0.06	0.25734	0.2592	0.72
							0.07135	0.10		0.2589	0.61
							0.07136	0.11			
							0.07133	0.07			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
7	28	0	0.35537	0.30740	-13.50	0.31050	0.31050	0	0	0.000218	
							0.31042	-0.03		0.000190	
							0.31047	-0.01			
							0.31053	0.01			
	118	25	9.09321	9.09317	0	0.30344	0.30349	0.02	7.68111	7.599	-1.07
							0.30355	0.04		7.606	-0.98
							0.30346	0.01			
							0.30360	0.05			
	276	B	14.98060	14.98073	0	0.13388	0.13371	-0.13	0.48298	0.4789	-0.85
							0.13379	-0.07		0.4789	-0.85
							0.13371	-0.13			
							0.13371	-0.13			
	312	T	15.39418	15.39118	-0.02	0.07128	0.07125	-0.04	0.25734	0.2557	-0.64
							0.07128	0		0.2559	-0.56
							0.07127	-0.01			
							0.07123	-0.07			
8	27	10	3.87854	3.88024	0.04	0.30858	0.30829	-0.09	3.09616	3.068	-0.91
							0.30824	-0.11		3.068	-0.91
							0.30812	-0.15			
							0.30809	-0.16			
	68	25	9.10541	9.11299	0.08	0.30617	0.30565	-0.17	7.68908	7.622	-0.87
							0.30558	-0.19		7.620	-0.90
							0.30563	-0.18			
							0.30561	-0.18			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
8	271	B	14.71118	14.71129	0	0.13388	0.13380	-0.06	0.48298	0.4798	-0.66
							0.13378	-0.08		0.4797	-0.68
							0.13382	-0.05			
							0.13376	-0.09			
	329	T	14.63442	14.63204	-0.02	0.07128	0.07115	-0.18	0.25734	0.2559	-0.56
							0.07115	-0.18		0.2560	-0.52
							0.07117	-0.15			
							0.07115	-0.18			
9	53	25	9.09963	9.10627	0.07	0.30675	0.30860	0.60	7.68342	7.641	-0.55
							0.30867	0.63		7.642	-0.54
							0.30865	0.62			
							0.30858	0.60			
	108	10	3.85639	3.85488	-0.04	0.31199	0.31339	0.45	3.07328	3.055	-0.59
							0.31332	0.43		3.051	-0.72
							0.31345	0.47			
							0.31338	0.45			
	222	B	14.97900	14.97893	0	0.13388	0.13411	0.17	0.48298	0.4774	-1.16
							0.13414	0.19		0.4774	-1.16
							0.13409	0.15			
							0.13413	0.18			
	286	T	14.73287	14.72880	-0.03	0.07128	0.07168	0.56	0.25734	0.2561	-0.48
							0.07169	0.58		0.2562	-0.44
							0.07169	0.58			
							0.07172	0.62			

TABLE IX (Continued)

LABORATORY D

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
10	40	16	5.97526	5.97809	0.05	0.30638	0.30605	-0.11	4.93981	4.924	-0.32
							0.30610	-0.09		4.922	-0.36
							0.30599	-0.13			
							0.30602	-0.12			
	150	10	3.85580	3.85646	0.02	0.30860	0.30863	0.01	3.07617	3.065	-0.36
							0.30860	0		3.059	-0.56
							0.30875	0.05			
							0.30853	-0.02			
	268	B	15.10412	15.10432	0	0.13388	0.13407	0.14	0.48298	0.4812	-0.37
							0.13407	0.14		0.4811	-0.39
							0.13406	0.13			
							0.13407	0.14			
	324	T	14.80724	14.80514	-0.01	0.07128	0.07127	-0.01	0.25734	0.2554	-0.75
							0.07129	0.02		0.2554	-0.75
							0.07129	0.02			
							0.07121	-0.10			

TABLE X

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
1	15	0	0.34995	0.3322	-5.07	0.30577	0.30420	-0.51	0		
							0.30415	-0.53			
							0.30432	-0.47			
							0.30419	-0.52			
	116	25	9.09532	9.0905	-0.05	0.30206	0.30145	-0.20	7.68435	7.69680	0.16
							0.30171	-0.12		7.69900	0.19
							0.30161	-0.15			
							0.30145	-0.20			
	267	B	15.25067	15.2486	-0.01	0.13388	0.13389	0.01	0.48298	0.48404	0.22
							0.13391	0.02		0.48396	0.20
							0.13392	0.03			
							0.13389	0.01			
	350	T	15.23803	15.2327	-0.03	0.07128	0.07102	-0.36	0.25734	0.26060	1.27
							0.07102	-0.36		0.25959	0.87
							0.07097	-0.43			
							0.07100	-0.39			
2	78	16	5.96868	5.9664	-0.04	0.30393	0.30441	0.16	4.93649	4.93779	0.03
							0.30450	0.19		4.93157	-0.10
							0.30445	0.17			
							0.30454	0.20			
	146	10	3.86116	3.8611	0	0.30706	0.30774	0.22	3.08242	3.06947	-0.42
							0.30769	0.21		3.07452	-0.26
							0.30789	0.27			
							0.30786	0.26			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	275	B	14.93573	14.9370	0.01	0.13388	0.13462	0.55	0.48298	0.483569	0.12
							0.13457	0.51		0.483611	0.13
							0.13455	0.50			
							0.13457	0.51			
	284	T	14.94265	14.9395	-0.02	0.07128	0.07162	0.48	0.25734	0.245683	-4.53
							0.07160	0.45		0.246843	-4.08
							0.07165	0.52			
							0.07164	0.51			
3	50	25	9.10456	9.1103	0.06	0.30563	0.30573	0.03	7.68888	7.717256	0.37
							0.30576	0.04		7.701540	0.16
							0.30600	0.12			
							0.30586	0.08			
	105	10	3.86366	3.8636	0	0.30633	0.30699	0.22	3.08535	3.087135	0.06
							0.30675	0.14		3.088139	0.09
							0.30672	0.13			
							0.30679	0.15			
	226	B	15.00047	15.0015	0.01	0.13388	0.13394	0.04	0.48298	0.482825	-0.03
							0.13394	0.04		0.482402	-0.12
							0.13394	0.04			
							0.13393	0.04			
	287	T	14.80226	14.7996	-0.02	0.07128	0.07127	-0.01	0.25734	0.247500	-3.82
							0.07128	0		0.247661	-3.76
							0.07124	-0.05			
							0.07127	-0.01			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
4	93	16	5.97628	5.9774	0.02	0.30391	0.30383	-0.03	4.94319	4.956461	0.27
							0.30392	0		4.951291	0.16
							0.30369	-0.07			
							0.30377	-0.05			
	144	0	0.34753	0.3461	-0.41	0.30365	0.30341	-0.08	0		
							0.30356	-0.03			
							0.30356	-0.03			
							0.30343	-0.07			
	207	B	15.10212	15.1034	0.01	0.13388	0.13407	0.14	0.48298	0.482450	-0.11
							0.13409	0.15		0.482397	-0.12
							0.13408	0.15			
							0.13408	0.15			
	322	T	14.99117	14.9893	-0.01	0.07128	0.07099	-0.40	0.25734	0.229478	-10.82
							0.07098	-0.42		0.227083	-11.75
							0.07099	-0.40			
							0.07102	-0.36			
5	63	25	9.10961	9.1125	0.03	0.30469	0.30420	-0.16	7.69425	7.711414	0.22
							0.30429	-0.13		7.7115505	0.22
							0.30444	-0.08			
							0.30423	-0.15			
	71	10	3.84126	3.8438	0.07	0.30319	0.30317	-0.01	3.06883	3.074450	0.18
							0.30329	0.03		3.074031	0.17
							0.30321	0.01			
							0.30319	0			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
5	218	B	15.04723	15.0501	0.02	0.13388	0.13392	0.03	0.48298	0.482828	-0.03
							0.13394	0.04		0.483629	0.13
							0.13403	0.11			
							0.13402	0.10			
	344	T	14.73556	14.7354	0	0.07128	0.07157	0.41	0.25734	0.259069	0.67
							0.07157	0.41		0.261295	1.54
							0.07154	0.37			
							0.07154	0.37			
6	102	0	0.34829	0.3237	-7.06	0.30432	0.30426	-0.02	0		
							0.30414	-0.06			
							0.30408	-0.08			
							0.30413	-0.06			
	122	16	5.96697	5.9664	-0.01	0.30850	0.30865	0.05	4.93040	4.939743	0.19
							0.30881	0.10		4.933952	0.07
							0.30870	0.06			
							0.30863	0.04			
	205	B	14.77171	14.7729	0.01	0.13388	0.13405	0.12	0.48298	0.482854	-0.03
							0.13407	0.14		0.483551	0.12
							0.13404	0.12			
							0.13407	0.14			
	358	T	15.15557	15.1533	-0.01	0.07128	0.07162	0.48	0.25734	0.226084	-12.14
							0.07162	0.48		0.227448	-11.61
							0.07164	0.51			
							0.07161	0.46			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
7	30	25	9.10182	9.1071	0.06	0.30771	0.30750	-0.07	7.68438	7.687741	0.04
							0.30738	-0.11		7.683024	-0.02
							0.30731	-0.13			
							0.30742	-0.09			
	54	16	5.94655	5.9553	0.15	0.30379	0.30395	0.05	4.91720	4.908093	-0.19
							0.30397	0.06		4.911609	-0.11
							0.30388	0.03			
							0.30388	0.03			
	262	B	14.80889	14.8091	0	0.13388	0.13410	0.16	0.48298	0.483462	0.10
							0.13410	0.16		0.483789	0.16
							0.13410	0.16			
							0.13409	0.15			
	299	T	14.71417	14.7110	-0.02	0.07128	0.07140	0.17	0.25734	0.251531	-2.26
							0.07138	0.14		0.251856	-2.13
							0.07140	0.17			
							0.07141	0.18			
8	17	25	9.08898	9.1078	0.21	0.30396	0.30430	0.11	7.67687	7.698309	0.28
							0.30449	0.17		7.701087	0.32
							0.30448	0.17			
							0.30452	0.18			
	23	10	3.85245	3.8642	0.31	0.31078	0.31106	0.09	3.07104	3.075251	0.14
							0.31102	0.08		3.078791	0.25
							0.31112	0.11			
							0.31108	0.10			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
8	217	B	15.03243	15.0335	0.01	0.13388	0.13407	0.14	0.48298	0.483462	0.10
							0.13405	0.12		0.483245	0.05
							0.13406	0.13			
							0.13407	0.14			
	311	T	14.84463	14.8413	-0.02	0.07128	0.07146	0.25	0.25734	0.252623	-1.83
							0.07148	0.28		0.251364	-2.32
							0.07150	0.31			
							0.07151	0.32			
9	35	16	5.95640	5.9653	0.15	0.30469	0.30518	0.16	4.92495	4.923409	-0.03
							0.30546	0.25		4.921815	-0.06
							0.30539	0.23			
							0.30546	0.25			
	55	0	0.34455	0.3522	2.22	0.30105	0.30101	-0.01	0		
							0.30118	0.04			
							0.30133	0.09			
							0.30115	0.03			
	239	B	14.78188	14.7809	-0.01	0.13388	0.13427	0.29	0.48298	0.483261	0.06
							0.13429	0.30		0.482421	-0.11
							0.13429	0.30			
							0.13426	0.28			
	302	T	15.30747	15.3027	-0.03	0.07128	0.07155	0.38	0.25734	0.249455	-3.06
							0.07154	0.37		0.248096	-3.59
							0.07153	0.35			
							0.07152	0.34			

TABLE X (Continued)

LABORATORY E

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
10	76	25	9.11501	9.1341	0.21	0.30791	0.30734	-0.19	7.69576	7.712334	0.22
							0.30757	-0.11		7.709929	0.18
							0.30750	-0.13			
							0.30757	-0.11			
	159	16	5.95653	5.9754	0.32	0.30979	0.30987	0.03	4.91994	4.921617	0.03
							0.31001	0.07		4.919696	0
							0.30999	0.06			
							0.31008	0.09			
	228	B	14.60783	14.6051	-0.02	0.13388	0.13405	0.12	0.48298	0.482860	-0.03
							0.13405	0.12		0.482915	-0.02
							0.13405	0.12			
							0.13408	0.15			
	341	T	15.29330	15.2870	-0.04	0.07128	0.07148	0.28	0.25734	0.257193	-0.06
							0.07151	0.32		0.257220	-0.05
							0.07150	0.31			
							0.07150	0.31			

TABLE XI

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
1	33	25	9.10458	9.1093	0.05	0.30640	0.30524	-0.38	7.68812	7.69687	0.11	
							0.30548	-0.30		7.69374	0.07	
							0.30552	-0.29				
							0.30556	-0.27				
	96	0	0.34854	0.3347	-3.97	0.30454	0.30517	0.21	0			
							0.30517	0.21				
							0.30534	0.26				
							0.30517	0.21				
	224	B	14.96129	14.9595	-0.01	0.13388	0.13368	-0.15	0.48298	0.48246	-0.11	
							0.13367	-0.16		0.48270	-0.06	
							0.13363	-0.19				
							0.13365	-0.17				
	321	T	14.83232	14.8278	-0.03	0.07128	0.07127	-0.01	0.25734	0.249705	-2.96	GL
							0.07134	0.09		0.254361	-1.16	
							0.07136	0.11				
							0.07135	0.10				
2	69	25	9.10602	9.1002	-0.06	0.30549	0.30488	-0.20	7.69030	7.696596	0.08	
							0.30527	-0.07		7.69532	0.07	
							0.30547	-0.01				
							0.30567	0.06				
	88	10	3.85633	3.8557	-0.02	0.30621	0.30638	0.06	3.07903	3.07690	-0.07	
							0.30645	0.08		3.07573	-0.11	
							0.30653	0.10				
							0.30639	0.06				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
2	236	B	15.13437	15.1335	-0.01	0.13388	0.13364	-0.18	0.48298	0.480734	-0.47	
							0.13369	-0.14		0.481287	-0.35	
							0.13366	-0.17				
							0.13370	-0.14				
	325	T	14.91813	14.9139	-0.03	0.07128	0.07167	0.55	0.25734	0.257884	0.21	CL
							0.07166	0.53		0.257846	0.20	
							0.07169	0.58				
							0.07167	0.55				
3	34	0	0.35323	0.3160	-10.54	0.30863	0.30773	-0.29	0			
							0.30761	-0.33				
							0.30770	-0.30				
							0.30774	-0.29				
	147	16	5.96123	5.9641	0.05	0.30478	0.30430	-0.16	4.92910	4.923331	-0.12	
							0.30429	-0.16		4.924575	-0.09	
							0.30419	-0.19				
							0.30425	-0.17				
	209	B	14.92914	14.9288	0	0.13388	0.13390	0.01	0.48298	0.481566	-0.30	
							0.13388	0		0.482849	-0.03	
							0.13389	0.01				
							0.13387	-0.01				
	323	T	15.04839	15.0440	-0.03	0.07128	0.07141	0.18	0.25734	0.256994	-0.13	GL
							0.07140	0.17		0.256934	-0.16	
							0.07140	0.17				
							0.07140	0.17				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
4	46	16	5.96018	5.9582	-0.03	0.30434	0.30439	0.02	4.92862	4.925786	-0.06	
							0.30449	0.05		4.932367	0.08	
							0.30447	0.04				
							0.30445	0.04				
	171	16	5.96301	5.9649	0.03	0.30293	0.30291	-0.01	4.93252	4.934477	0.04	
							0.30292	0		4.944408	0.24	
							0.30313	0.07				
							0.30301	0.03				
	215	B	14.51313	14.5129	0	0.13388	0.13383	-0.04	0.48298	0.481815	-0.24	
							0.13382	-0.05		0.482590	-0.08	
							0.13383	-0.04				
							0.13383	-0.04				
	307	T	15.08555	15.0808	-0.03	0.07128	0.07106	-0.31	0.25734	0.256202	-0.44	CL
							0.07105	-0.32		0.256316	-0.40	
							0.07106	-0.31				
							0.07113	-0.21				
5	20	25	9.11769	9.1220	0.05	0.30794	0.30831	0.12	7.69808	7.670574	-0.36	
							0.30832	0.12		7.662396	-0.46	
							0.30843	0.16				
							0.30826	0.10				
	81	25	9.11572	9.1135	-0.02	0.30994	0.31028	0.11	7.69435	7.743289	0.64	
							0.31014	0.06		7.749664	0.72	
							0.31007	0.04				
							0.31020	0.08				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
5	264	B	15.05385	15.0527	-0.01	0.13388	0.13393	0.04	0.48298	0.481130	-0.39	
							0.13396	0.06		0.482204	-0.16	
							0.13396	0.06				
							0.13397	0.06				
	316	T	Spilled									
6	37	10	3.83295	3.8278	-0.13	0.30690	0.30649	-0.13	3.05780	3.058042	0.01	
							0.30653	-0.12		3.058127	0.01	
							0.30668	-0.07				
							0.30667	-0.07				
	121	25	9.11698	9.1121	-0.05	0.30628	0.30671	0.14	7.69913	7.707870	0.11	
							0.30687	0.19		7.703374	0.06	
							0.30674	0.15				
							0.30694	0.22				
	204	B	14.84898	14.8486	0	0.13388	0.13390	0.01	0.48298	0.481233	-0.36	
							0.13389	0.01		0.482670	-0.07	
							0.13387	-0.01				
							0.13393	0.04				
	283	T	15.03938	15.0345	-0.03	0.07128	0.07080	-0.67	0.25734	0.255323	-0.78	GL
							0.07086	-0.59		0.254567	-1.08	
							0.07084	-0.61				
							0.07082	-0.64				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
7	59	16	5.95346	5.9496	-0.06	0.30521	0.30552	0.10	4.92184	4.918466	-0.07	
							0.30556	0.11		4.921907	0	
							0.30559	0.12				
							0.30563	0.14				
	163	0	0.35089	0.3392	-3.33	0.30659	0.30594	-0.21	0			
							0.30604	-0.18				
							0.30585	-0.24				
							0.30612	-0.15				
	210	B	14.89524	14.8944	-0.01	0.13388	0.13361	-0.20	0.48298	0.481742	-0.26	
							0.13360	-0.21		0.481840	-0.24	
							0.13364	-0.18				
							0.13363	-0.19				
	290	T	15.72173	14.7172	-6.39	0.07128	0.07089	-0.54	0.25734	0.254209	-1.22	CL
							0.07090	-0.53		0.254671	-1.04	
							0.07090	-0.53				
							0.07090	-0.53				
8	85	16	5.97810	5.9723	-0.10	0.31002	0.31064	0.20	4.93865	4.953944	0.31	
							0.31077	0.24		4.957660	0.38	
							0.31070	0.22				
							0.31068	0.21				
	162	10	3.84668	3.8452	-0.04	0.30545	0.30592	0.15	3.07132	3.065714	-0.18	
							0.30580	0.11		3.061829	-0.31	
							0.30595	0.16				
							0.30593	0.16				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium			
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff	
8	246	B	14.74649	14.7458	0	0.13388	0.13402	0.10	0.48298	0.483678	0.14	
							0.13404	0.12		0.483786	0.16	
							0.13403	0.11				
							0.13405	0.12				
	310	T	14.90128	14.8966	-0.03	0.07128	0.07142	0.20	0.25734	0.255197	-0.83	GL
							0.07146	0.25		0.253737	-1.40	
							0.07144	0.23				
							0.07143	0.21				
9	112	16	5.95611	6.0557	1.67	0.30573	0.30616	0.14	4.92365	4.929168	0.11	
							0.30640	0.22		4.930305	0.14	
							0.30623	0.16				
							0.30645	0.24				
	133	0	0.34778	0.3359	-3.42	0.30387	0.30342	-0.15	0			
							0.30342	-0.15				
							0.30356	-0.10				
							0.30356	-0.10				
	258	B	14.87483	14.8740	-0.01	0.13388	0.13393	0.04	0.48298	0.481684	-0.27	
							0.13399	0.08		0.481899	-0.23	
							0.13391	0.02				
							0.13398	0.07				
	300	T	15.35825	15.3536	-0.03	0.07128	0.07146	0.25	0.25734	0.257631	0.11	CL
							0.07146	0.25		0.257338	0	
							0.07148	0.28				
							0.07149	0.30				

TABLE XI (Continued)

LABORATORY F

Mo.	Vial	Ratio	Net Sample Weight, g			Uranium			Thorium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff	Prepared	Observed	% Diff
10	58	25	9.10384	9.0996	-0.05	0.30347	0.30369	0.07	7.69041	7.713912	0.31
							0.30382	0.12		7.712518	0.29
							0.30370	0.08			
							0.30367	0.07			
	113	10	3.86236	3.8365	-0.67	0.30999	0.30969	-0.10	3.08053	3.088451	0.26
							0.30975	-0.08		3.089161	0.28
							0.30982	-0.05			
							0.30980	-0.06			
	251	B	14.75708	14.7563	-0.01	0.13388	0.13395	0.05	0.48298	0.482930	-0.01
							0.13395	0.05		0.482979	0
							0.13395	0.05			
							0.13395	0.05			
	298	T	15.14450	15.1408	-0.02	0.07128	0.07094	-0.47	0.25734	0.272257	5.80
							0.07099	-0.40		0.271635	5.55
							0.07094	-0.47			
							0.07093	-0.49			

TABLE XII

LABORATORY H

Mo.	Vial	Ratio	Net Sample Wt.			Uranium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff
2	41	25	9.10910	9.1108	0.02	0.30607	0.3040	-0.68
							0.3113	1.71
							0.3075	0.47
							0.3044	-0.55
	47	16	5.96261	5.9608	-0.03	0.30713	0.3065	-0.21
							0.3073	0.06
							0.3084	0.41
							0.3051	-0.66
	249	B	14.77152	14.7703	-0.01	0.13388	0.1332	-0.51
							0.1331	-0.58
							0.1332	-0.51
							0.1335	-0.29
	334	T	14.72686	14.7218	-0.03	0.07128	0.07096	-0.45
							0.07115	-0.18
							0.07131	0.04
							0.07107	-0.29
3	137	0	0.35222	0.3530	0.22	0.30775	0.3145	2.19
							0.3058	-0.63
							0.3091	0.44
							0.3024	-1.74
	154	16	5.95444	5.9565	0.03	0.30522	0.3063	0.35
							0.3071	0.62
							0.3104	1.70
							0.3056	0.12

TABLE XII (Continued)

LABORATORY H

Mo.	Vial	Ratio	Net Sample Wt.			Uranium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff
3	269	B	14.81832	14.8173	-0.01	0.13388	0.1335	-0.29
							0.1336	-0.21
							0.1335	-0.29
							0.1336	-0.21
	291	T	14.67919	14.6746	-0.03	0.07128	0.07135	0.10
							0.07162	0.48
							0.07121	-0.10
							0.07142	0.20
4	39	0	0.34678	0.3468	0.01	0.30300	0.3032	0.07
							0.3029	-0.03
							0.3031	0.03
							0.3034	0.13
	109	25	9.11645	9.1191	0.03	0.30922	0.3081	-0.36
							0.3089	-0.10
							0.3082	-0.33
							0.3085	-0.23
	234	B	14.76561	14.7646	-0.01	0.13388	0.1329	-0.73
							0.1327	-0.88
							0.1325	-1.03
							0.1326	-0.96
	296	T	14.57124	14.5662	-0.03	0.07128	0.07103	-0.35
							0.07103	-0.35
							0.07121	-0.10
							0.07123	-0.07

TABLE XII (Continued)

LABORATORY H

Mo.	Vial	Ratio	Net Sample Wt.			Uranium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff
12	221	B	15.01666	15.0155	-0.01	0.13388	0.1329	-0.73
							0.1332	-0.51
							0.1330	-0.66
							0.1330	-0.66
	294	T	14.97638	14.9699	-0.04	0.07128	0.07194	0.93
							0.07160	0.45
							0.07173	0.63
							0.07172	0.62
	235	B	14.75553	14.7547	-0.01	0.13388	0.1337	-0.14
							0.1333	-0.44
							0.1336	-0.21
							0.1338	-0.06
	330	T	14.74033	14.7335	-0.05	0.07128	0.07170	0.59
							0.07176	0.67
							0.07187	0.83
							0.07185	0.80
	260	B	14.75985	14.7585	-0.01	0.13388	0.1346	0.54
							0.1348	0.68
							0.1346	0.54
							0.1347	0.61
	338	T	15.13523	15.1279	-0.05	0.07128	0.07150	0.31
							0.07150	0.31
							0.07155	0.38
							0.07152	0.34

TABLE XII (Continued)

LABORATORY H

Mo.	Vial	Ratio	Net Sample Wt.			Uranium		
			Prepared	Observed	% Diff	Prepared	Observed	% Diff
12	270	B	14.93862	14.9372	-0.01	0.13388	0.1340	0.09
							0.1340	0.09
							0.1338	-0.06
							0.1336	-0.21
	345	T	14.73692	14.7297	-0.05	0.07128	0.07150	0.31
							0.07157	0.41
							0.07142	0.20
							0.07151	0.32
	280	B	14.76415	14.7629	-0.01	0.13388	0.1336	-0.21
							0.1338	-0.06
							0.1337	-0.14
							0.1337	-0.14
	360	T	14.64151	14.6336	-0.05	0.07128	0.07141	0.18
							0.07138	0.14
							0.07135	0.10
							0.07142	0.20