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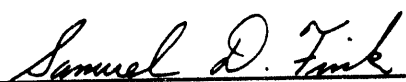
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**Solubility of Plutonium and Uranium
in Alkaline Salt Solutions (U)**


by

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Table of Contents

Section	Page
1.0 Summary	1
2.0 Introduction	1
3.0 Experimental	1
4.0 Results	5
5.0 Discussion	12
6.0 Quality Assurance	13
7.0 References	13
8.0 Appendices	14

1.0 SUMMARY

The solubility of plutonium and uranium in alkaline salt solutions, which will be processed in the In-Tank Precipitation (ITP) process, was investigated to screen for significant factors and interactions among the factors comprising the salt solutions. The factors included in the study were hydroxide, nitrate, nitrite, aluminate, sulfate, carbonate, and temperature. Over the range of factor concentrations studied, the level of hydroxide in the solution is not sufficient alone to predict the resulting concentration of plutonium and uranium in the solution. Other constituents of the salt solution play an important role in determining the amount of plutonium and uranium in solution.

Statistical models predicting the plutonium and uranium concentrations over the range of salt solutions investigated are provided. The results from these models are used to provide insight into other salt solutions (different concentrations of the constituents of the solution than those studied) that may provide higher concentrations of plutonium and/or uranium due to increased solubility.

For salt solutions within the range considered by this study, upper 95% tolerance limits for Pu and U solubilities, estimated from analytically measured specie concentrations, are 1.68 mg/L and 17.1 mg/L, respectively. The alkaline salt solutions considered in this study should bound those that will be treated in the ITP process.

2.0 INTRODUCTION

Monosodium titanate (MST) is used in the In-Tank Precipitation (ITP) process to remove radio-strontium from alkaline salt solutions. Recent work has shown that plutonium and uranium are also removed from solution [1]. As part of the nuclear safety assessment of the ITP process, the maximum amount of plutonium and uranium which can load onto the MST is needed.

The loading of plutonium and uranium on MST is dependent upon the chemical potential of the solution. Maximum loading will occur for solutions having the highest chemical potential or highest plutonium and uranium concentrations. Therefore, a test program was conducted to determine what combinations of the six major components (nitrate, nitrite, hydroxide, aluminate, carbonate, and sulfate) and temperature of the alkaline supernatant liquid would provide the maximum solubilities of plutonium and uranium. The range of factors were selected to bound the possible compositions of salt solutions which will be treated in the ITP process.

3.0 EXPERIMENTAL

This section provides details of the experimental method used for the study, an overview of the statistically-based design, and the data resulting from the tests. In addition to the samples analyzed as part of the main study, a number of special samples were also included to study other questions of interest. These samples are described in this section and the results of their analyses reported below. Special notation used to label the various data is described in this section as well.

3.1 Experimental Method

All salt solutions for the solubility tests were prepared in calibrated volumetric glassware using reagent grade chemicals of the same manufacturer and lot number (see Table 1 in Appendix 8.1). A single batch of deionized, distilled water was collected and stored in large polyethylene bottles for use in all solution preparations. All solutions were filtered through a 0.45 micron nitrocellulose filter to remove any undissolved solids prior to use.

For each test, a 500-mL polyethylene bottle was filled with 475 mL of salt solution. Each bottle was spiked with 0.25 mL of an enriched-uranium nitrate solution (200 g U/liter, ca. 90% U-235) and 0.025 mL of a weapons-grade plutonium nitrate solution (30.7 g Pu/L, 94% Pu-239, 6% Pu-240). The bottles were stoppered and placed within a two-tier rack equipped with an air-actuated shaker. The rack was then placed into a water bath such that the bottom tier of bottles was immersed in water and the top tier was above the water. A layer of insulation was placed beneath the top tier of bottles to minimize heat transfer from the heated water.

During the course of the solubility testing, the position of the bottles was rotated to reduce the possibility of uncontrolled variations in the temperature within the shaker/water-bath. The order of sampling for each set of 10 bottles on each tier was also varied. Prior to sampling, the air supply to the shaker was shut off overnight to allow any solids to settle.

The sampling procedure consisted of filtering a small sample of the test solution into clean vials, and then acidifying a known quantity of the filtered solution with nitric acid. Approximately, six milliliters of the test solution were drawn into a disposable plastic syringe, and then passed through a Acrodisc[®] syringe filter (0.45 μ m pore size) into a clean bottle. Five milliliters of the filtered solution were pipetted into a glass vial containing sufficient amount of a 1:1 mixture (by weight) of concentrated nitric acid and deionized, distilled water to neutralize the sample and provide a final pH of ≤ 1 .

Bottle 21 was prepared using deionized, distilled water and placed inside the glovebox in which the test matrix bottles were placed to provide a check for a possible cross-contamination problem among the samples over the course of the experiment. These data are presented in Table 2 in Appendix 8.1 and analyzed in Exhibit 1 of Appendix 8.2. The uranium concentration measurements by the Chemchek method were always below the detection limit of 0.01 mg/L. This exhibit shows that the average values for Pu and U for samples taken for this bottle were near zero indicating that no cross-contamination of the samples was likely during the course of the experiment.

Two additional solubility tests were conducted to determine the effects of coprecipitation of the Pu and U with ferric hydroxide and lower free hydroxide concentration on the solubility. The coprecipitated test slurry was prepared by neutralizing a solution of ferric nitrate spiked with the plutonium and uranium stock solutions [mole rate of Fe:U:Pu = 3100:6.6:1.0] with 5M sodium hydroxide solution to produce a final free hydroxide concentration of about 0.1M. The precipitated solids were separated from the solution, washed with a small portion of the concentrated salt solution, and added to the test bottle containing a concentrated salt solution 1.0M in

nitrate, 0.1M in nitrite, 0.5M in free hydroxide, 0.33M in aluminate, 0.30M in carbonate, and 0.40M in sulfate. In the lower hydroxide test, a salt solution 1.0M in nitrate, 0.1M in nitrite, 0.051M in free hydroxide, 0.33M in aluminate, 0.30M in carbonate, and 0.40M in sulfate, was spiked with uranium and plutonium in the same manner as described previously. Both of these test solutions were mixed at room temperature using a magnetic stirrer.

Each sample was analyzed by the Analytical Development Section of the Savannah River Technology Center for uranium and plutonium content. The uranium content was determined by two different instrumental methods: (1) kinetic phosphorescence analyzer denoted by Ck and (2) inductively coupled plasma/mass spectrometry denoted by ICP/MS. The plutonium content was also determined by two separate methods: (1) alpha scintillation counting denoted by C and (2) inductively coupled plasma/mass spectrometry (ICP/MS).

The solubility tests were concluded after six months. In addition to the determination of plutonium and uranium content at the conclusion of the tests, each of the test solutions were also analyzed for chemical composition. Samples of the unspiked solutions were also submitted for chemical analysis at the conclusion of the test so that changes in the chemical composition of the test solutions over the six-month test period could be assessed. The concentrations of the free hydroxide and carbonate were determined by titration. Nitrate, nitrite, and sulfate concentrations were determined by ion chromatography. Aluminate and sodium concentrations were determined by inductively coupled plasma emission spectroscopy.

3.2 Experimental Design

The experimental design used for this study was comprised of 20 treatment combinations and is presented in [2]. One of the 20 treatment combinations, all factors at their high level, could not be prepared as not all of the salts would dissolve into solution. It was found that all salts would dissolve at 70% of the high levels. This combination was substituted for the "all high" treatment in the original design. Table 3 of Appendix 8.1 shows the factors and their concentration levels as prepared for the 20 treatment combinations. The factors investigated were: nitrate (NO₃), nitrite (NO₂), hydroxide (OH), aluminate (Al(OH)₄), carbonate (CO₃), sulfate (SO₄), and temperature. The levels of each factor are given in the table with the unit of measurement being moles/liter (M) for each factor except temperature which is in degrees Celsius (°C). The temperature levels are designated as High (H) and Low (L) where H is 60 °C (± 2 °C) and L is 25 °C (± 2 °C). The prepared levels of the factors were based on the weights of salts dissolved in each solution.

Each row of Table 3 corresponds to a sample or bottle. The 20 bottles were numbered 1 through 20. The levels of the factors as well as the Pu and U concentrations were investigated periodically for each bottle. Tables 4 and 5 in Appendix 8.1 provide the results for the unspiked initial and 6-months sample analyses, respectively. The Pu and U concentrations were determined more frequently during this 6-month period, and the frequency of their analyses is discussed below.

Over the course of the six-month test, the nitrate, nitrite, and sulfate concentrations did not change significantly. Since there was no systematic increase in the concentrations

of these species, it is concluded that there was no significant evaporation of water from the solutions during the test. Compared to the as-prepared compositions, the initial and 6-month solutions are almost always slightly lower in the concentrations of nitrite and sulfate. This may indicate that a small fraction of these species were removed from solution or did not dissolve completely during solution preparation. In the case of nitrite, some of the nitrite may have been oxidized to nitrate. Because of the high nitrate concentration and the relatively small amount of nitrite involved, no change in the nitrate concentration was apparent.

There is considerable scatter in the carbonate results for both the initial and 6-month samples. However, all of the low-carbonate solutions (0.02M) of the 6-month samples showed a significant increase in the carbonate concentration (average relative increase of 90%) over the course of the experiment. The higher carbonate concentration is due to the absorption of atmospheric carbon dioxide. This result is not unexpected since there was no attempt to exclude carbon dioxide from the test bottles during the experiment. The average increase in the carbonate concentration for the initial samples was only 33%. These samples were stored in unopened bottles for six months. The greater increase in the solubility test bottles reflects the more frequent opening of these bottles to sample for plutonium and uranium content.

Six of the solubility test solutions also showed significant decreases in the aluminate concentration and increases in the hydroxide concentration after six months. Five of the six solutions were prepared having high aluminate (0.33M) and low hydroxide (0.5M) concentrations. The sixth solution was one of the four center point solutions which had intermediate aluminate (0.19M) and hydroxide (1.25M) concentrations. Apparently, the aluminum crystallized from solution during the experiment, probably as a hydrated aluminum oxide. No attempt was made to isolate and analyze the solids from any of the bottles.

The crystallization of the unknown aluminum compound(s) during the experiment is not due to the higher temperature since three of the solutions were kept at ambient laboratory temperature. The unspiked solution samples stored for six months did not show significant decreases in the aluminate concentrations. It is possible that the presence of the plutonium and uranium solids provided nucleation sites for crystallization of the aluminum compound(s) from the high aluminum and low hydroxide solutions.

3.3 Experimental Data

The responses of interest for each bottle were the plutonium and uranium concentrations. A measurement of the plutonium concentration was made for each bottle over time using two methods: counting (C) and inductively coupled plasma/mass spectrometry (ICP/MS). The measurements of plutonium concentration in milligrams/liter (mg/L) using counting and inductively coupled plasma methods over time are designated by Pu-C_n and Pu-ICP/MS_n, respectively, where n represents the number of days into the study at which the analysis was made. The measurements over time of the uranium concentration in mg/L were made using inductively coupled plasma/mass spectrometry (ICP/MS) and a kinetic phosphorescence technique (Chemchek or Ck) and are represented by U-ICP/MS_n and U-Ck_n, respectively, where once again n represents the number of days into the study. These data by bottle

number are presented in Tables 6-9 in Appendix 8.1. Some analytical results are missing as a result of lost samples and each of these is indicated by a dot in the tables.

4.0 RESULTS

The data from the plutonium (Pu) and uranium (U) solubility experiment as outlined in memo SCS-ASG-92037 [2] have been statistically analyzed. The objectives of this analysis were to:

- estimate the effects of the main factors and interactions (as outlined in [2]),
- determine prediction equations and prediction limits with a 95% confidence level, and
- investigate the analytical methods used to determine Pu and U solubility.

In this section, the data are investigated using various statistical methods. The factors are screened for importance. Prediction equations for Pu and U concentrations are developed, and salt solutions that may lead to higher Pu and U solubilities are identified. Other special bottles that were prepared during the experimentation are also investigated.

4.1 Time Effect for Solubilities

Plots of the various treatment solubilities over time were prepared (Exhibits 2-17 in Appendix 8.2). Although general patterns are present in some of these plots; little evidence of a systematic time effect in the concentrations for either U or Pu for either of the analytical methods is seen. The behavior revealed in the plots could be common analytical effects such as day-to-day variation. This variation is further discussed as part of the analysis of special bottle # 23.

4.2 Main Effects and Interactions for the Full Model

In addition to the main effect for each factor, the experiment was designed to investigate the two-way interactions of OH with each of the other factors, the two-way interaction of NO₂*NO₃, and the three-way interaction, OH*NO₂*NO₃. The average Pu and U concentrations in mg/L were computed for each of the analytical methods, and these values were used as the response variables. These responses in relation to the 6-month as well as prepared concentrations of the factors were of interest. A regression analysis was completed for each of the following situations (see Appendix 8.2 for exhibits):

	<u>Level of Factors</u>	
	Prepared Levels	6-month Levels
Average Pu solubility		
using Counting	- Exhibit 18	Exhibit 22
using ICP/MS	- Exhibit 19	Exhibit 23

	<u>Level of Factors</u>	
	Prepared Levels	6-month Levels
Average U solubility		
using ICP/MS	- Exhibit 20	Exhibit 24
using Ck	- Exhibit 21	Exhibit 25

In these exhibits, the **Prob> |t|** values provide the observed significance probability for the test statistic for the hypothesis that the corresponding model parameter is zero. Those parameter estimates (main effects and interactions) with a **Prob> |t|** value near 0.05 or smaller would be considered significant. Highlights from these exhibits are:

Exhibit	Specie	Analytical Method	Significant Main Effects	Significant Interactions
18	Pu	Counting	None	OH*SO4
19	Pu	ICP/MS	SO4	OH*SO4, OH*NO2*NO3
20	U	ICP/MS	None	OH*NO3
21	U	Ck	None	None
22	Pu	C	None	None
23	Pu	ICP/MS	None	None
24	U	ICP/MS	None	OH*NO3
25	U	Ck	None	None

Some of these models are not significant at the 0.05 level (**Prob>F** in the Analysis of Variance section is greater than 0.05). The parameter estimates for the significant main effects and interactions for each of the species, Pu and U, appear to be consistent across the analytical methods and the two data sets for the factor levels.

A Lack of Fit section is available for the analyses of the solubilities for the prepared concentrations (see Exhibits 18-21). The experiment was designed with two pairs of exact replicates which are used to provide a check for the adequacy of the model tested. This check is based on the comparison of the estimate of pure error as measured for the replicates and the residual error from the model. The difference between these estimates of the error is called the lack of fit error, and if it is significantly larger than the pure error, there is an indication of an inadequate functional form for the model. For the U solubility results, there is an indication of a lack of fit in the model. Thus, a functional form involving quadratic terms for one or more of the factors might be more appropriate to model U solubility over this range of salt solutions.

4.3 Main Effects and Interactions for Reduced Models

Exhibits 26-33 in Appendix 8.2 provide the results of the statistical investigation of each of the situations outlined in exhibits 18-25 with the more insignificant factors and interactions of each of the full models used to help estimate the error variance. Thus, the factors and interactions of the more insignificant parameter estimates were eliminated from each of the full models to develop a reduced model. The significant and marginally significant terms (both main factors and interactions) for these reduced models are shown in these exhibits. The main effects for factors involved in interactions are retained in the model regardless of their significance. When the three-way interaction OH*NO2*NO3 is significant, all two-way interactions involving OH, NO2, and NO3 are also included in the model regardless of their significance.

Let Pu and U represent the plutonium and uranium concentrations, respectively, in mg/L and NO3, NO2, etc. the molar concentrations of the respective species and Temp represent the temperature in degrees Celsius, then the models displayed in these exhibits can be summarized as follows:

Pu-Counting [Prepared Factor Levels]:

$$\begin{aligned} \text{Pu} = & -0.03 + 0.01 \text{ NO}_3 - 0.05 \text{ NO}_2 - 0.02 \text{ OH} + 0.58 \text{ CO}_3 - 0.35 \text{ SO}_4 \\ & + 0.70 (\text{OH} \cdot \text{SO}_4) + 0.03 (\text{OH} \cdot \text{NO}_3) + 0.09 (\text{OH} \cdot \text{NO}_2) + 0.06 (\text{NO}_2 \cdot \text{NO}_3) \\ & - 0.10 (\text{OH} \cdot \text{NO}_2 \cdot \text{NO}_3) \end{aligned}$$

Pu-ICP/MS [Prepared Factor Levels]:

$$\begin{aligned} \text{Pu} = & -0.01 + 0.02 \text{ NO}_3 - 0.01 \text{ NO}_2 - 0.09 \text{ OH} + 0.59 \text{ CO}_3 - 0.44 \text{ SO}_4 \\ & - 0.19 \text{ Al}(\text{OH})_4 + 0.31 (\text{OH} \cdot \text{Al}(\text{OH})_4) + 0.61 (\text{OH} \cdot \text{SO}_4) + 0.01 (\text{OH} \cdot \text{NO}_3) \\ & + 0.05 (\text{OH} \cdot \text{NO}_2) + 0.04 (\text{NO}_2 \cdot \text{NO}_3) - 0.06 (\text{OH} \cdot \text{NO}_2 \cdot \text{NO}_3) \end{aligned}$$

U-ICP/MS [Prepared Factor Levels]:

$$\text{U} = 3.40 + 0.51 \text{ NO}_3 - 1.00 \text{ NO}_2 + 4.28 \text{ OH} - 5.36 \text{ CO}_3 - 1.02 (\text{OH} \cdot \text{NO}_3)$$

U-Ck [Prepared Factor Levels]:

$$\text{U} = 2.63 + 0.44 \text{ NO}_3 - 0.80 \text{ NO}_2 + 3.53 \text{ OH} - 4.25 \text{ CO}_3 - 0.82 (\text{OH} \cdot \text{NO}_3)$$

Pu-Counting [6 month Factor Levels]:

$$\begin{aligned} \text{Pu} = & 0.02 + 0.01 \text{ NO}_3 - 0.01 \text{ NO}_2 - 0.08 \text{ OH} - 0.31 \text{ CO}_3 - 0.19 \text{ SO}_4 \\ & + 0.61 (\text{OH} \cdot \text{CO}_3) + 0.64 (\text{OH} \cdot \text{SO}_4) + 0.03 (\text{OH} \cdot \text{NO}_3) \\ & + 0.04 (\text{OH} \cdot \text{NO}_2) + 0.06 (\text{NO}_2 \cdot \text{NO}_3) - 0.08 (\text{NO}_3 \cdot \text{NO}_2 \cdot \text{OH}) \end{aligned}$$

Pu-ICP [6 month Factor Levels]:

$$Pu = 0.06 + 0.03 NO_3 - 0.09 OH + 0.53 CO_3 - 0.44 SO_4 - 0.92 Al(OH)_4 + 0.57 (OH \cdot Al(OH)_4) + 0.54 (OH \cdot SO_4)$$

U-ICP [6 month Factor Levels]:

$$U = 2.95 + 0.56 NO_3 - 1.06 NO_2 + 5.30 OH + 1.27 CO_3 - 0.02 Temp - 6.15 (OH \cdot CO_3) - 0.95 (OH \cdot NO_3)$$

U-Ck [6 month Factor Levels]:

$$U = 2.44 + 0.44 NO_3 - 0.78 NO_2 + 3.38 OH - 5.11 CO_3 - 0.70 (OH \cdot NO_3)$$

4.4 Prediction Intervals for Pu and U Solubilities

The models developed in Exhibits 26-33 can be used to predict Pu and U solubilities and to determine various 95% confidence limits. Once such confidence interval is computed for the expected Pu and U concentrations for each of the salt solutions (bottles) of this study. A second interval, a 95% prediction interval, is determined for an individual (the next) Pu or U concentration measurement for a given salt solution. Finally, for each bottle, a simultaneous, upper 95% tolerance limit is determined at a confidence level of 95%. An explanation of these tolerance limits is provided in Appendix 8.3 [3]. These upper tolerance limits determine a confidence band which provides simultaneous coverage for 95% of the specie concentrations over all salt solutions within the range of this study with 95% confidence. These results are provided in Appendix 8.1 in Tables 10-13 for the prepared factor levels and Tables 14-17 for the 6-month factor levels.

Since each of the fitted models is linear, the upper tolerance limit of the salt solution with the largest upper tolerance limit in each table provides an upper tolerance limit for at least 95% of the specie solubilities over the entire range of salt solutions included in this study with 95% confidence. These upper limits are:

	Prepared	6-month
Pu (mg/L)		
Counting	1.54	1.68
ICP/MS	0.95	0.81
U (mg/L)		
ICP/MS	16.4	17.1
Ck	13.3	13.0

Thus, selecting the largest value from this table for each specie provides upper 95% tolerance limits for Pu and U solubilities of 1.68 mg/L and 17.1 mg/L, respectively, as estimated from these analytically measured specie concentrations, for salt solutions within the range considered by this study.

A review of these tables identifies that the predicted Pu concentration is highest for Bottle # 20 for both Counting and ICP/MS and that predicted U concentration is highest for Bottle #7 for both ICP/MS and Chemchek. This is true for both the prepared factor levels and the 6-month levels. From Table 3, the following conditions (levels of the factors) correspond to these bottles:

	<u>Pu (Bottle # 20)</u>	<u>U (Bottle # 7)</u>
NO3	High	Low
NO2	Low	Low
OH	High	High
Al(OH)4	Low	High
CO3	High	Low
SO4	High	High
Temp	Low	Low

Even though the predictive models of section 4.3 are somewhat inconsistent in their parameters (some factors and interactions appear in one model but not in another), the models are consistent in the combinations of levels of the factors that lead to the higher Pu and U solubilities. The inconsistency in the models may be the result of the compositional changes in several of the solutions over the course of the 6-month test (see the discussion at the end of Section 3.2).

If additional experimental treatments were to be planned, these combinations leading to higher solubilities would help determine factor levels that should be checked. The range of levels for those factors that are at the high level should be increased slightly, and the range of levels for those factors at low levels should be decreased slightly as new treatment combinations are considered. The results of the lack of fit checks, as indicated in the exhibits, for the reduced models for U concentration indicate the need for a quadratic term in the model for one or more of the factors. However, note that the plots of actual versus predicted U concentrations for Exhibits 28 and 29 reveal that the curvature appears to be downward indicating that the maximum solubility of U occurs at the boundary of the design space.

4.5 Comparison of Analytical Methods

No rigorous statistical study was conducted to compare the various analytical methods used here. Some special bottles were prepared, however, to provide an opportunity for some investigation into the accuracy and precision of the analytical methods.

Bottle 23 was comprised of an alkaline salt solution which had been acidified with concentrated nitric acid to a pH of 3.04 and doped with 2.8 mg/L plutonium and 18 mg/L uranium. Analysis of samples from this bottle indicated concentrations lower than these values, which suggested that the plutonium and uranium were undergoing hydrolysis and precipitating from solution. Therefore, this bottle was not suitable for use as an absolute standard but it could be used to investigate the sampling and instrumental variability over the course of the experiment.

Three aliquots were prepared from bottle # 23 over time and each of the aliquots were analyzed for Pu and U by each method over time. Table 18 in Appendix 8.1 provides the data from this study. Having three aliquots from the same bottle analyzed over time provides an opportunity to investigate the variance contributions from various sources. The variability within the group of three aliquots includes sources of variability such as sampling and instrumental including analytical method preparations. The variability between the groups of three aliquots also includes those sources of variability related to day-to-day changes such as different analysts, day effects, and re-calibration of instruments. Thus, this relationship may be modelled as follows:

$$y_{ij} = \mu + T_i + \epsilon_{ij}$$

where y_{ij} represents the Pu or U concentration as measured by one of the analytical methods for i^{th} time period for the j^{th} aliquot,
 μ represents the average Pu or U concentration,
 T_i represents the time effect where $E(T_i) = 0$ by assumption and the variance of T_i is to be estimated, and
 ϵ_{ij} represents the error where $E(\epsilon_{ij}) = 0$ by assumption and the variance of ϵ_{ij} is to be estimated.

Exhibits 34-37 in Appendix 8.2 provide the details of a study of these components of variance. The variance component for the time random effect represents the variability between the groups of three aliquots, and the component for the error (labelled residual) variable the within group variability. These exhibits provide an analysis which tests for the significance of the time random effect and also provides an estimate of these within (residual) and between (time) variances for each of the analytical methods used to measure Pu and U concentrations.

The time effect for Pu concentration measurements using the Counting method is investigated in Exhibit 34. The time effect is significant at the 0.058 level, but the estimate of its variance (0.033 mg/L) is less than the within variance (0.079 mg/L). For the ICP/MS measurements of Pu concentration, Exhibit 35 shows that the Time effect is not significant. The within (residual) variance for these measurements is 0.39 mg/L, a larger variance than the 0.079 mg/L value for Counting. Thus, there is some evidence that the Counting method is more precise than the ICP/MS analytical method. More will be said on the precision of the analytical methods below.

Exhibit 36 provides the analysis of the time random effect for U concentrations measured using the ICP/MS method. The time effect is significant, and the estimates of the variances for time and residual components are 1.18 mg/L and 0.35 mg/L, respectively. Thus, the between (time) group variance is larger than the within (residual) group variance. Similar results for U concentrations measured using the Chemchek method are provided in Exhibit 37. Once again, the time effect is statistically significant. The estimated variances are 2.57 and 0.89 mg/L for the time and residual components, respectively. Thus, the between group (time) variance is larger than the within group (residual) variance both analytical methods used to measure U concentration. This indicates that day-to-day effects contribute

substantially to the variability in the U concentration measurements. Comparing the within (residual) variances for the two analytical methods (0.35 mg/L for ICP/MS and 0.89 mg/L for Chemchek) indicates that the ICP/MS analysis may be more precise than the Chemchek method. More comparisons related to analytical precision are made below.

Two bottles were prepared with a known (only negligible error) concentration of U and Pu in 0.1M nitric acid. These were bottles 24 and 26 for Pu and U, respectively. A statistical analysis was conducted to investigate the biases of each pair of analytical methods using the solubility results from the analyses of these two bottles over time.

The nominal standard concentration of Pu in bottle 24 was 3.07 mg/L. Table 19 in Appendix 8.1 provides the data from the Counting and ICP/MS analyses, denoted by C and ICP/MS, respectively, over time. One value for ICP/MS (0.191 for day 40) appears to be an outlier, but it was included in the analysis. Exhibit 38 in Appendix 8.2 presents a comparison of the two analytical methods. There is no indication of a difference in the mean estimates of Pu solubility for the two methods (3.20 mg/L for Counting and 2.86 mg/L for ICP/MS). Also, there is no evidence that either method is biased from the 3.07 mg/L true value. The standard deviations of the data from C and ICP/MS are 0.49 and 1.27 mg/L, respectively. The statistical result (Levene's test) indicates that the variances are not statistically different. Thus, there is no indication of a difference in analytical precision over time (the standard deviations) of the two methods used to determine Pu concentrations.

The U nominal standard concentration in bottle 26 is 3.98 mg/L. The data from the analyses of this bottle are presented in Table 20 in Appendix 8.1. Exhibit 39 in Appendix 8.2 provides statistical details from the analyses of this bottle by the two methods: ICP/MS and Chemchek, denoted by ICP/MS and Ck, respectively. Both methods provide averages near the nominal 3.98 mg/L (3.88 mg/L for ICP/MS and 4.28 mg/L for Ck), and there is no evidence of a statistically significant difference between the mean values for the two methods, nor between each of these means and the true value of 3.98 mg/L. The precision is better for the Ck method than the ICP/MS method for these small samples. The standard deviation for ICP/MS is 0.81 mg/L while that for Ck is only 0.31 mg/L. However, this difference is not large enough to declare that the precision of the two analytical methods over time is statistically different due to the small sample sizes involved.

4.6 Effects of Coprecipitation of Pu and U with Ferric Hydroxide

Bottle 22 was prepared so that it may be compared to bottle 19 to determine the effects of coprecipitation of Pu and U with ferric hydroxide. Table 21 in Appendix 8.1 provides these data. The statistical analysis presented in Exhibit 40 shows that the least squares means for the Pu solubilities of bottles 19 and 22 are 0.11 and 0.02 mg/L, respectively. Thus, the Pu solubility for bottle 19 is statistically significantly higher than that of bottle 22. Exhibit 41 shows a similar analysis for the U solubility. Bottle 22 shows a significantly higher U solubility, 10.7 mg/L, versus bottle 19 at 4.6 mg/L.

4.7 Effects of Lower Free Hydroxide Concentration

Bottle 28A and 28B were prepared with factor concentrations similar to bottle 19 except for the free hydroxide concentrations. For bottles 28A and 28B, the salt solution contained only 0.051M in free hydroxide as compared to the 0.5M concentration for bottle 19. Table 22 in Appendix 8.1 provides the Pu and U concentrations in mg/L for these bottles.

Exhibits 42 and 43 in Appendix 8.2 provide a comparison for these bottles for Pu and U, respectively. These results indicate that higher Pu and U solubilities were obtained for bottles 28A and 28B than for bottle 19. For Pu, the least squares means were 0.1, 0.2, and 0.4 mg/L for bottles 19, 28A, and 28B, respectively. For U, the least squares means were 4.6, 40.7, and 29.9 mg/L for bottles 19, 28A, and 28B, respectively. Thus, higher solubilities than those from our 20 bottles may also be possible over a range of OH values lower than that covered by the 20 bottles of primary interest in this study.

5.0 DISCUSSION

Over the range of factors studied, the level of hydroxide is not sufficient alone to predict the resulting concentrations of plutonium and uranium in solution. Other major constituents of the salt solution play an important role in determining the amount of plutonium and uranium in solution. Neither temperature or time exhibited a significant effect on the solubility of plutonium or uranium.

The solubilities of plutonium and uranium are affected by the individual components to varying degrees. Thus, conditions which increase the plutonium solubility do not necessarily increase the uranium solubility. Maximum plutonium solubility is predicted at high nitrate, high hydroxide, high carbonate, and high sulfate concentrations and low nitrite and low aluminate concentrations. Maximum uranium solubility is predicted at high hydroxide, high aluminate and high sulfate concentrations and low nitrate, low nitrite, and low carbonate concentrations.

The conditions predicted to maximize the plutonium solubility agree fairly well with those reported by Delegard [4]. Delegard reports that the solubility of plutonium increases with increasing concentrations of hydroxide, and in alkaline solutions containing increasing concentrations of nitrate and nitrite, aluminate, and carbonate. With the exception of the effect of aluminate, the same trends were observed in this study. The decreasing effect of aluminate in the present study may reflect the more complex chemistry occurring in salt solutions comprised of six different anionic components as compared to the two and three-component mixtures in the Delegard studies.

The conditions which maximize the uranium solubility are slightly different than those for plutonium. Unexpectedly, uranium solubility decreases with an increase in the carbonate concentration. Carbonate is known to form stable complexes with uranium. At a hydroxide concentration of 0.051M and a carbonate concentration of 0.30M, the uranium solubility is observed to be about an order of magnitude higher than that at a hydroxide concentration of 0.50M. Apparently, in the six-component matrix of this study, the concentration of carbonate is not sufficiently high except at low hydroxide concentrations to affect the uranium solubility.

For salt solutions within the range considered by this study, upper 95% tolerance limits for Pu and U solubilities, estimated from the analytically measured specie concentrations, are 1.68 mg/L and 17.1 mg/L, respectively. The alkaline salt solutions considered in this study should bound those that will be treated in the ITP process.

The solubilities reported in this document may be higher than those which will be observed in actual waste due to coprecipitation of the plutonium and uranium with iron, manganese, and aluminum during neutralization of fresh waste. In a single experiment in which the plutonium and uranium were coprecipitated with iron, the solubility of plutonium was decreased by a factor of about five, and that of uranium increased by about a factor of two. It is recommended that additional experiments be conducted to assess the impact of coprecipitation on the solubilities of plutonium and uranium in alkaline salt solutions.

6.0 QUALITY ASSURANCE

The experimental work was conducted in accordance with Technical Task Plan WSRC-RP-92-574 and Quality Assurance Plan WSRC-RP-92-575.

The statistical analyses were completed using JMP[®] Version 2 by SAS Institute, Inc., a commercial software package for the Apple[®] Macintosh[®] computer. Please see reference [5] for more information on this package.

7.0 REFERENCES

- [1] D. T. Hobbs and D. D. Walker, "Plutonium and Uranium Adsorption on Monosodium Titanate," WSRC-RP-92-93, August 13, 1992.
- [2] J. H. Weber, Memorandum to D. T. Hobbs, "Fractional Factorial Design," SCS-ASG-92037, May 6, 1992.
- [3] Miller, R. G., Jr. **Simultaneous Statistical Inference**, McGraw-Hill, New York, 1966.
- [4] Calvin H. Delegard, "Solubility of PuO₂ · H₂O in Alkaline Hanford High-Level Waste Solution," **Radiochim. Acta.**, 41, 11-21(1987).
- [5] **JMP[®] User's Guide: Version 2 of JMP**, SAS Institute Inc., SAS Campus Drive, Cary, NC, 1989.

8.0 APPENDICES

8.1 Tables

- | | |
|-----------|--|
| Table 1. | Manufacturer and Lot # of Chemicals Used in Solubility Tests |
| Table 2. | Plutonium and Uranium Concentrations for Special Bottle # 21 |
| Table 3. | Prepared Levels for Experimental Factors |
| Table 4. | Initial Levels for Experimental Factors |
| Table 5. | Levels for Experimental Factors at 6-months |
| Table 6. | Plutonium Concentration Measurements Over Time Using Counting Analytical Method |
| Table 7. | Plutonium Concentration Measurements Over Time Using ICP/MS Analytical Method |
| Table 8. | Uranium Concentration Measurements Over Time Using ICP/MS Analytical Method |
| Table 9. | Uranium Concentration Measurements Over Time Using Kinetic Phosphorescence Technique Analytical Method |
| Table 10. | Predictions for Expected Plutonium Concentrations Using Model Developed from Counting Analytical Data and Prepared Levels of the Factors |
| Table 11. | Predictions for Expected Plutonium Concentrations Using Model Developed from ICP/MS Analytical Data and Prepared Levels of the Factors |
| Table 12. | Predictions for Expected Uranium Concentrations Using Model Developed from ICP/MS Analytical Data and Prepared Levels of the Factors |
| Table 13. | Predictions for Expected Uranium Concentrations Using Model Developed from Chemchek Analytical Data and Prepared Levels of the Factors |
| Table 14. | Predictions for Expected Plutonium Concentrations Using Model Developed from Counting Analytical Data and 6-month Levels of the Factors |
| Table 15. | Predictions for Expected Plutonium Concentrations Using Model Developed from ICP/MS Analytical Data and 6-month Levels of the Factors |

- Table 16. Predictions for Expected Uranium Concentrations Using Model Developed from Chemchek Analytical Data and 6-month Levels of the Factors
- Table 17. Predictions for Expected Uranium Concentrations Using Model Developed from ICP/MS Analytical Data and 6-month Levels of the Factors
- Table 18. Plutonium and Uranium Concentrations for Special Bottle # 23
- Table 19. Plutonium Concentrations for Special Bottle # 24
- Table 20. Uranium Concentrations for Special Bottle # 26
- Table 21. Plutonium and Uranium Concentrations for Bottles 19 and 22
- Table 22. Plutonium and Uranium Concentrations for Bottles 19 and 28

Table 1. Manufacturer and Lot # of Chemicals Used in Solubility Tests

<u>Chemical</u>	<u>Manufacturer</u>	<u>Lot #</u>
NaNO ₃	Fisher	912637
NaOH	EM	32015207
Na ₂ SO ₄	Fisher	895669
Na ₂ CO ₃ H ₃ O	Baker	B45165
NaNO	Fisher	916644
Al(NO ₃) ₃ · 12H ₂ O	Fisher	920145

Table 2. Plutonium and Uranium Concentrations for Special Bottle # 21

Rows	Species	Analytical	Time (days)	Conc (mg/L)
1	Pu	C	3	0.0034
2	Pu	C	7	0.0024
3	Pu	C	14	0.0172
4	Pu	C	25	0.0011
5	Pu	C	31	0.0016
6	Pu	ICP/MS	31	0.0000
7	Pu	C	40	0.0023
8	Pu	ICP/MS	40	0.0054
9	Pu	C	56	0.0081
10	Pu	ICP/MS	56	0.0002
11	Pu	C	74	0.0009
12	Pu	ICP/MS	74	0.0000
13	Pu	C	81	0.0024
14	Pu	ICP/MS	81	0.0000
15	Pu	C	84	0.0358
16	Pu	ICP/MS	84	0.0000
17	Pu	C	122	0.0219
18	Pu	ICP/MS	122	0.0000
19	Pu	C	153	0.0059
20	Pu	ICP/MS	153	0.0010
21	Pu	C	185	0.0010
22	U	ICP/MS	31	.
23	U	Ck	31	.
24	U	ICP/MS	40	0.0068
25	U	Ck	40	.
26	U	ICP/MS	56	0.0022
27	U	Ck	56	.
28	U	ICP/MS	74	0.0000
29	U	Ck	74	.
30	U	ICP/MS	122	0.0020
31	U	Ck	122	.
32	U	ICP/MS	153	0.0010
33	U	Ck	153	.
34	U	Ck	185	.

Table 3. Prepared Levels for Experimental Factors

Values are concentrations in moles/liter (M) except for temperature for which H = 60 °C and L = 25 °C.

Bottle #	NO3	NO2	OH	Al(OH)4	CO3	SO4	Temp
1	1.00	2.00	2.00	0.05	0.02	0.40	H
2	2.50	1.05	1.25	0.19	0.16	0.21	H
3	2.50	1.05	1.25	0.19	0.16	0.21	H
4	2.50	1.05	1.25	0.19	0.16	0.21	L
5	4.00	0.10	0.50	0.33	0.30	0.02	L
6	4.96	2.00	0.75	0.36	0.02	0.02	L
7	1.00	0.10	2.00	0.33	0.02	0.40	L
8	1.00	2.00	2.00	0.33	0.30	0.02	L
9	1.00	0.10	2.00	0.05	0.30	0.02	H
10	4.00	2.00	0.50	0.33	0.02	0.40	L
11	4.00	0.10	2.00	0.33	0.02	0.02	H
12	1.00	0.10	0.50	0.05	0.02	0.02	L
13	2.50	1.05	1.25	0.19	0.16	0.21	L
14	2.80	1.40	1.40	0.23	0.21	0.28	H
15	4.00	2.00	0.50	0.05	0.30	0.02	H
16	4.00	0.10	0.50	0.05	0.02	0.40	H
17	1.00	2.00	0.50	0.33	0.02	0.02	H
18	1.00	2.00	0.50	0.05	0.30	0.40	L
19	1.00	0.10	0.50	0.33	0.30	0.40	H
20	4.00	0.10	2.00	0.05	0.30	0.40	L

Table 4. Initial Levels for Experimental Factors

Values are concentrations in moles/liter (M) except for temperature for which H \approx 60 °C and L \approx 25 °C.

Bottle #	NO3	NO2	OH	Al(OH) ₃	CO ₃	SO ₄	Temp
1	9.16E-01	1.87E+00	1.97E+00	5.25E-02	1.10E-02	4.07E-01	H
2	2.51E+00	9.80E-01	1.30E+00	1.94E-01	1.31E-01	2.07E-01	H
3	2.51E+00	9.80E-01	1.30E+00	1.94E-01	1.31E-01	2.07E-01	H
4	2.51E+00	9.80E-01	1.30E+00	1.94E-01	1.31E-01	2.07E-01	L
5	4.14E+00	1.03E-01	5.57E-01	3.31E-01	2.23E-01	1.80E-02	L
6	4.81E+00	1.88E+00	8.12E-01	3.61E-01	3.20E-02	1.82E-02	L
7	9.16E-01	1.02E-01	1.97E+00	3.38E-01	1.50E-02	3.92E-01	L
8	9.41E-01	1.91E+00	2.07E+00	3.31E-01	2.22E-01	1.89E-02	L
9	9.40E-01	1.02E-01	2.04E+00	5.32E-02	1.98E-01	2.16E-02	H
10	4.14E+00	1.91E+00	5.40E-01	3.29E-01	4.30E-02	3.94E-01	L
11	4.17E+00	9.85E-02	2.08E+00	3.34E-01	2.80E-02	1.79E-02	H
12	9.81E-01	9.66E-02	4.23E-01	7.53E-02	1.60E-02	2.50E-02	L
13	2.51E+00	9.80E-01	1.30E+00	1.94E-01	1.31E-01	2.07E-01	L
14	2.78E+00	1.30E+00	1.44E+00	2.29E-01	1.62E-01	2.72E-01	H
15	4.05E+00	1.89E+00	4.97E-01	5.06E-02	2.78E-01	2.12E-02	H
16	4.11E+00	9.00E-02	3.35E-01	5.17E-02	3.90E-02	3.95E-01	H
17	9.53E-01	1.92E+00	5.82E-01	3.34E-01	2.90E-02	2.05E-02	H
18	9.45E-01	1.95E+00	4.95E-01	5.07E-02	2.13E-01	3.96E-01	L
19	9.49E-01	9.60E-02	4.85E-01	3.37E-01	2.54E-01	3.97E-01	H
20	4.09E+00	9.95E-02	1.95E+00	5.20E-02	2.63E-01	4.12E-01	L

Table 5. Levels for Experimental Factors at 6-months

Values are concentrations in moles/liter (M) except for temperature for which H \approx 60 °C and L \approx 25 °C.

Bottle #	NO3	NO2	OH	Al(OH)4	CO3	SO4	Temp
1	9.88E-01	2.04E+00	2.16E+00	4.43E-02	5.60E-02	4.00E-01	H
2	2.63E+00	1.04E+00	1.34E+00	1.57E-01	2.00E-01	2.15E-01	H
3	2.67E+00	1.04E+00	1.38E+00	6.95E-02	2.02E-01	2.12E-01	H
4	2.74E+00	1.06E+00	1.39E+00	1.89E-01	1.60E-01	2.50E-01	L
5	4.40E+00	9.69E-02	9.23E-01	1.23E-01	3.06E-01	2.17E-01	L
6	4.83E+00	1.91E+00	1.01E+00	2.60E-01	4.50E-02	1.75E-01	L
7	1.00E+00	1.03E-01	2.21E+00	2.76E-01	5.40E-02	3.71E-01	L
8	9.62E-01	1.95E+00	2.27E+00	2.91E-01	3.58E-01	2.10E-02	L
9	9.44E-01	9.61E-02	2.17E+00	3.98E-02	2.90E-01	2.00E-02	H
10	4.06E+00	1.94E+00	7.50E-01	1.69E-01	3.40E-02	3.82E-01	L
11	4.25E+00	1.05E-01	2.18E+00	3.03E-01	3.90E-02	2.31E-02	H
12	1.00E+00	9.41E-02	4.42E-01	6.62E-02	2.30E-02	2.01E-02	L
13	2.51E+00	9.98E-01	1.38E+00	1.87E-01	1.70E-01	2.20E-01	L
14	2.73E+00	1.30E+00	1.50E+00	2.25E-01	2.24E-01	2.74E-01	H
15	4.12E+00	1.96E+00	5.24E-01	5.16E-02	3.11E-01	1.99E-02	H
16	3.97E+00	9.57E-02	3.56E-01	4.82E-02	2.40E-02	3.98E-01	H
17	9.36E-01	1.91E+00	7.22E-01	2.05E-01	2.90E-02	2.08E-02	H
18	9.32E-01	1.92E+00	5.74E-01	4.96E-02	3.01E-01	3.94E-01	L
19	9.88E-01	9.58E-02	7.54E-01	2.04E-01	2.87E-01	4.02E-01	H
20	4.02E+00	9.89E-02	2.28E+00	4.90E-02	2.87E-01	3.66E-01	L

Table 6. Plutonium Concentration Measurements Over Time Using Counting Analytical Method

Values are in milligrams/liter (mg/L).

Pu-Cn: n represents the number of days into the experiment at which the analysis was conducted.

Bottle #	Pu-C3	Pu-C7	Pu-C14	Pu-C25	Pu-C31	Pu-C40	Pu-C56
1	3.91E-01	2.88E-01	3.12E-01	2.97E-01	3.30E-01	2.82E-01	3.77E-01
2	2.70E-01	8.68E-02	7.97E-02	7.43E-02	8.91E-02	5.74E-02	6.51E-02
3	2.02E-01	1.17E-01	3.31E-01	8.83E-02	1.08E-01	2.73E-01	6.86E-02
4	1.28E-01	1.70E-01	1.52E-01	1.16E-01	9.93E-02	1.16E-01	1.19E-01
5	1.51E-01	2.12E-01	2.31E-01	5.25E-02	2.70E-01	2.78E-01	2.92E-01
6	1.68E-01	8.44E-02	1.41E-01	1.56E-01	1.41E-01	1.13E-01	8.44E-02
7	5.62E-01	4.13E-01	1.34E-01	5.29E-01	4.94E-01	5.11E-01	7.33E-01
8	1.99E-01	2.47E-01	4.93E-01	3.13E-01	2.89E-01	3.32E-01	3.11E-01
9	2.67E-01	2.33E-01	3.04E-01	1.31E-01	1.31E-01	1.24E-01	1.27E-01
10	5.23E-02	1.13E-01	1.33E-01	8.83E-02	8.13E-02	6.75E-01	6.14E-02
11	2.23E-01	1.86E-01	7.14E-02	1.99E-01	1.92E-01	1.61E-01	1.61E-01
12	2.45E-02	2.51E-02	1.52E-01	3.52E-02	6.36E-02	1.37E-02	1.72E-02
13	1.63E-01	1.89E-01	1.41E-02	1.41E-01	1.27E-01	1.34E-01	1.04E-01
14	3.39E-01	1.43E-01	2.00E-01	1.68E-01	1.70E-01	1.64E-01	1.59E-01
15	3.28E-01	2.50E-01	2.40E-01	2.06E-01	1.93E-01	1.62E-01	1.47E-01
16	3.43E-01	6.14E-02	2.24E-01	1.92E-02	2.45E-02	1.31E-02	1.90E-02
17	4.56E-02	1.26E-02	3.07E-02	1.33E-02	8.83E-03	1.44E-02	1.63E-02
18	3.47E-01	4.79E-01	1.70E-02	2.88E-01	9.30E-02	2.04E-01	4.19E-02
19	2.23E-01	1.53E-01	3.08E-01	1.35E-01	1.14E-01	1.32E-01	1.27E-01
20	3.66E-01	6.54E-01	1.40E-01	1.23E+00	1.00E+00	1.81E+00	9.77E-01

Bottle #	Pu-C84	Pu-C122	Pu-C153	Pu-C185	Pu-C
1	2.43E-01	2.57E-01	7.82E-01	1.40E-01	0.336
2	1.11E-01	9.93E-02	4.47E-02	1.01E-01	0.098
3	3.92E-01	3.99E-01	7.36E-02	5.95E-02	0.192
4	1.05E-01	8.91E-02	1.01E-01	2.74E-01	0.134
5	2.67E-01	1.76E-01	1.53E-01	1.45E-01	0.203
6	8.21E-02	1.13E+00	5.23E-02	1.06E-01	0.205
7	4.07E-01	3.81E-01	.	6.51E-01	0.482
8	3.12E-01	2.58E-01	2.88E-01	2.92E-01	0.303
9	1.30E-01	7.32E-02	1.55E-01	8.44E-02	0.160
10	3.53E-02	4.69E-02	5.04E-02	3.12E-01	0.150
11	1.52E-01	1.06E-01	1.52E-01	9.44E-02	0.154
12	7.68E-03	1.32E-02	1.16E-02	6.89E-03	0.034
13	1.02E-01	9.30E-02	1.08E-02	1.20E-01	0.109
14	3.64E-01	1.17E-01	1.57E-01	2.92E-01	0.207
15	1.36E-01	5.31E-01	7.58E-01	6.67E-03	0.269
16	5.57E-02	4.07E-02	2.38E-01	3.00E-01	0.122
17	1.10E-02	2.29E-02	1.95E-02	8.60E-03	0.019
18	1.56E-01	1.42E-01	2.98E-01	2.85E-01	0.214
19	9.77E-02	7.11E-02	8.44E-02	7.53E-02	0.138
20	6.55E-01	6.03E-01	6.14E-01	1.41E+00	0.860

Table 7. Plutonium Concentration Measurements Over Time Using ICP/MS Analytical Method

Values are in milligrams/liter (mg/L).

Pu-ICP/MSn: n represents the number of days into the experiment at which the analysis was conducted.

Bottle #	Pu-ICP/MS31	Pu-ICP/MS40	Pu-ICP/MS56	Pu-ICP/MS84	Pu-ICP/MS122	Pu-ICP/MS153	Pu-ICP/MS
1	2.38E-01	1.40E-01	1.81E-01	1.75E-01	1.58E-01	1.57E-01	0.175
2	7.76E-02	3.52E-02	4.23E-02	9.26E-02	7.89E-02	4.41E-02	0.062
3	9.12E-02	1.34E-01	4.80E-02	3.19E-01	2.30E-01	4.40E-02	0.144
4	8.51E-02	6.57E-02	7.26E-02	9.25E-02	8.18E-02	8.66E-02	0.081
5	2.83E-01	1.91E-01	2.15E-01	2.40E-01	1.61E-01	1.37E-01	0.205
6	1.22E-01	6.89E-02	4.70E-02	7.09E-02	7.92E-02	6.93E-02	0.076
7	4.62E-01	2.96E-01	4.10E-01	3.66E-01	2.06E-01	2.65E-01	0.334
8	2.84E-01	1.58E-01	2.25E-01	2.66E-01	2.51E-01	2.42E-01	0.238
9	1.07E-01	6.68E-02	8.75E-02	9.97E-02	6.64E-02	7.08E-02	0.083
10	6.53E-02	3.74E-02	4.22E-02	4.45E-02	3.66E-02	2.54E-02	0.042
11	1.65E-01	9.30E-02	1.13E-01	1.44E-01	7.79E-02	7.33E-02	0.111
12	1.24E-02	3.70E-03	6.01E-03	4.52E-03	3.39E-03	5.65E-03	0.006
13	1.00E-01	6.36E-02	8.01E-02	7.57E-02	7.19E-02	8.05E-02	0.079
14	1.49E-01	7.73E-02	1.15E-01	2.93E-01	1.01E-01	1.01E-01	0.139
15	1.40E-01	9.09E-02	1.29E-01	1.16E-01	3.58E-01	5.93E-01	0.238
16	1.13E-02	4.50E-03	7.56E-03	3.50E-02	2.48E-02	1.14E-01	0.033
17	9.60E-03	5.60E-03	8.08E-03	7.95E-03	1.75E-02	1.27E-02	0.010
18	1.73E-01	1.02E-01	1.46E-01	1.31E-01	1.17E-01	1.70E-01	0.140
19	1.01E-01	6.09E-02	9.52E-02	8.86E-02	3.05E-02	6.93E-02	0.074
20	7.31E-01	5.08E-01	5.72E-01	5.08E-01	4.05E-01	4.60E-01	0.531

Table 8. Uranium Concentration Measurements Over Time Using ICP/MS Analytical Method

Values are in milligrams/liter (mg/L).

U-ICP/MSn: n represents the number of days into the experiment at which the analysis was conducted.

Bottle #	U-ICP/MS31	U-ICP/MS40	U-ICP/MS56	U-ICP/MS84	U-ICP/MS122	U-ICP/MS153	U-ICP/MS
1	7.60E+00	8.54E+00	7.90E+00	7.59E+00	7.71E+00	1.04E+01	8.29
2	3.82E+00	4.34E+00	2.95E+00	3.79E+00	3.87E+00	4.33E+00	3.85
3	3.83E+00	4.55E+00	3.25E+00	3.80E+00	4.07E+00	4.51E+00	4.00
4	4.53E+00	4.89E+00	3.36E+00	4.44E+00	4.28E+00	4.68E+00	4.36
5	4.71E+00	5.03E+00	3.68E+00	4.42E+00	4.68E+00	5.57E+00	4.68
6	5.02E+00	5.98E+00	4.09E+00	4.65E+00	4.84E+00	4.92E+00	4.92
7	1.31E+01	1.35E+01	9.71E+00	1.31E+01	1.16E+01	1.47E+01	12.62
8	6.29E+00	6.76E+00	4.94E+00	6.11E+00	6.13E+00	6.97E+00	6.20
9	7.67E+00	7.84E+00	6.50E+00	8.09E+00	8.21E+00	1.02E+01	8.09
10	3.66E+00	4.06E+00	3.23E+00	3.89E+00	3.39E+00	3.46E+00	3.62
11	5.51E+00	6.07E+00	4.52E+00	5.94E+00	5.18E+00	6.83E+00	5.68
12	4.64E+00	5.04E 00	4.19E+00	4.53E+00	4.26E+00	4.30E+00	4.49
13	4.10E+00	4.26E+00	3.81E+00	3.64E+00	3.77E+00	4.34E+00	3.99
14	3.93E+00	4.34E+00	3.65E+00	4.09E+00	3.80E+00	4.94E+00	4.13
15	1.51E+00	1.70E+00	1.63E+00	1.44E+00	1.73E+00	2.23E+00	1.71
16	4.75E+00	4.50E+00	4.08E+00	3.25E+00	2.62E+00	3.04E+00	3.71
17	3.04E+00	3.58E+00	3.12E+00	4.18E+00	3.76E+00	4.13E+00	3.64
18	2.71E+00	2.85E+00	2.71E+00	2.68E+00	2.49E+00	3.33E+00	2.80
19	4.44E+00	4.79E+00	5.01E+00	5.48E+00	3.32E+00	7.04E+00	5.01
20	4.49E+00	5.63E+00	2.85E+00	4.91E+00	4.73E+00	6.07E+00	4.78

Table 9. Uranium Concentration Measurements Over Time Using Kinetic Phosphorescence Technique Analytical Method

Values are in milligrams/liter (mg/L).

U-Ckn: n represents the number of days into the experiment at which the analysis was conducted.

Bottle #	U-Ck31	U-Ck40	U-Ck56	U-Ck84	U-Ck122	U-Ck153	U-Ck185	U-Ck
1	7.1E+00	6.7E+00	6.9E+00	7.9E+00	7.7E+00	7.1E+00	6.4E+00	7.11
2	3.0E+00	3.0E+00	3.0E+00	3.5E+00	3.3E+00	2.9E+00	2.6E+00	3.05
3	3.0E+00	6.2E+00	3.2E+00	3.5E+00	3.5E+00	3.0E+00	2.3E+00	3.53
4	3.6E+00	3.6E+00	.	.	.	3.2E+00	2.9E+00	3.34
5	3.6E+00	3.6E+00	3.6E+00	4.0E+00	3.9E+00	3.7E+00	3.7E+00	3.73
6	8.3E+00	3.9E+00	4.0E+00	4.6E+00	4.0E+00	3.3E+00	2.6E+00	4.38
7	9.9E+00	9.5E+00	9.8E+00	1.1E+01	1.0E+01	9.2E+00	9.9E+00	9.89
8	5.0E+00	5.1E+00	5.1E+00	5.3E+00	5.1E+00	4.6E+00	4.0E+00	4.88
9	7.1E+00	6.3E+00	6.8E+00	7.7E+00	7.4E+00	6.9E+00	6.6E+00	6.97
10	2.7E+00	3.0E+00	3.2E+00	3.2E+00	2.9E+00	2.9E+00	2.2E+00	2.87
11	4.6E+00	4.4E+00	4.6E+00	5.2E+00	4.6E+00	4.3E+00	4.1E+00	4.54
12	4.0E+00	4.0E+00	4.0E+00	4.1E+00	3.7E+00	3.3E+00	2.8E+00	3.69
13	3.2E+00	3.5E+00	3.3E+00	3.8E+00	3.2E+00	3.0E+00	2.9E+00	3.27
14	3.1E+00	3.3E+00	3.3E+00	3.4E+00	3.3E+00	3.3E+00	2.6E+00	3.19
15	1.5E+00	1.3E+00	1.4E+00	1.4E+00	1.5E+00	1.8E+00	7.0E-01	1.37
16	4.6E+00	4.0E+00	3.4E+00	3.0E+00	2.5E+00	2.1E+00	1.7E+00	3.05
17	2.6E+00	2.7E+00	2.9E+00	3.7E+00	3.0E+00	2.9E+00	2.6E+00	2.91
18	2.1E+00	2.4E+00	2.4E+00	2.6E+00	2.1E+00	2.3E+00	1.7E+00	2.23
19	3.5E+00	3.5E+00	3.7E+00	4.7E+00	4.6E+00	4.3E+00	4.3E+00	4.08
20	3.8E+00	4.4E+00	4.4E+00	4.9E+00	4.3E+00	3.9E+00	4.3E+00	4.28

Table 10. Predictions for Expected Plutonium Concentrations Using Model Developed from Counting Analytical Data and Prepared Levels of the Factors

Values are in milligrams/liter (mg/L) for plutonium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	Pu-C	Predicted Pu	Lower 95% CI for Expected Pu	Upper 95% CI for Expected Pu	Lower 95% PI for Indiv Pu	Upper 95% PI for Indiv Pu	Simultaneous Upper 95% Tolerance Limit with 95% Confidence
1	0.336	0.423	0.246	0.600	0.146	0.700	1.163
2	0.098	0.182	0.112	0.252	0.000	0.406	0.645
3	0.192	0.182	0.112	0.252	0.000	0.406	0.645
4	0.134	0.182	0.112	0.252	0.000	0.406	0.645
5	0.203	0.230	0.053	0.408	0.000	0.507	0.970
6	0.205	0.086	0.000	0.243	0.000	0.350	0.773
7	0.482	0.428	0.250	0.605	0.151	0.704	1.168
8	0.303	0.184	0.008	0.361	0.000	0.461	0.922
9	0.160	0.189	0.012	0.366	0.000	0.465	0.927
10	0.150	0.182	0.016	0.348	0.000	0.451	0.893
11	0.154	0.214	0.026	0.402	0.000	0.498	0.983
12	0.034	0.000	0.000	0.169	0.000	0.269	0.728
13	0.109	0.182	0.112	0.252	0.000	0.406	0.645
14	0.207	0.215	0.075	0.354	0.000	0.469	0.858
15	0.269	0.343	0.179	0.507	0.074	0.612	1.049
16	0.122	0.069	0.000	0.246	0.000	0.345	0.807
17	0.019	0.007	0.000	0.184	0.000	0.284	0.746
18	0.214	0.168	0.001	0.335	0.000	0.439	0.882
19	0.138	0.154	0.000	0.327	0.000	0.428	0.884
20	0.860	0.775	0.588	0.962	0.492	1.058	1.539

Table 11. Predictions for Expected Plutonium Concentrations Using Model Developed from ICP/MS Analytical Data and Prepared Levels of the Factors

Values are in milligrams/liter (mg/L) for plutonium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	Pu-ICP/MS	Predicted Pu	Lower 95% CI for Expected Pu	Upper 95% CI for Expected Pu	Lower 95% PI for Individ Pu	Upper 95% PI for Individ Pu	Simultaneous Upper 95% Tolerance Limit with 95% Confidence
1	0.175	0.204	0.097	0.310	0.046	0.361	0.673
2	0.062	0.110	0.071	0.149	0.000	0.233	0.388
3	0.144	0.110	0.071	0.149	0.000	0.233	0.388
4	0.081	0.110	0.071	0.149	0.000	0.233	0.388
5	0.205	0.219	0.113	0.325	0.061	0.377	0.689
6	0.076	0.027	0.000	0.115	0.000	0.173	0.445
7	0.334	0.329	0.213	0.445	0.164	0.494	0.828
8	0.238	0.196	0.090	0.302	0.039	0.353	0.664
9	0.083	0.078	0.000	0.194	0.000	0.242	0.575
10	0.042	0.048	0.000	0.145	0.000	0.200	0.492
11	0.111	0.146	0.040	0.252	0.000	0.303	0.615
12	0.006	0.000	0.000	0.088	0.000	0.140	0.451
13	0.079	0.110	0.071	0.149	0.000	0.233	0.388
14	0.139	0.145	0.067	0.222	0.005	0.285	0.533
15	0.238	0.276	0.173	0.379	0.120	0.431	0.737
16	0.033	0.009	0.000	0.113	0.000	0.165	0.475
17	0.010	0.002	0.000	0.108	0.000	0.160	0.471
18	0.140	0.124	0.027	0.222	0.000	0.277	0.570
19	0.074	0.087	0.000	0.193	0.000	0.245	0.556
20	0.531	0.486	0.381	0.591	0.329	0.643	0.952

Table 12. Predictions for Expected Uranium Concentrations Using Model Developed from ICP/MS Analytical Data and Prepared Levels of the Factors

Values are in milligrams/liter (mg/L) for uranium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	U-ICP/MS	Predicted U	Lower 95% CI for Expected U	Upper 95% CI for Expected U	Lower 95% PI for Individ U	Upper 95% PI for Individ U	Simultaneous Upper 95% Tolerance Limit with 95% Confidence
1	8.29	8.34	6.81	9.87	5.39	11.29	14.34
2	3.85	4.95	4.36	5.54	2.36	7.54	9.13
3	4.00	4.95	4.36	5.54	2.36	7.54	9.13
4	4.36	4.95	4.36	5.54	2.36	7.54	9.13
5	4.68	3.84	2.26	5.43	0.87	6.82	9.95
6	4.92	3.26	1.81	4.72	0.35	6.17	9.12
7	12.62	10.24	8.65	11.83	7.26	13.22	16.35
8	6.20	6.84	5.31	8.38	3.89	9.79	12.85
9	8.09	8.74	7.20	10.28	5.78	11.69	14.76
10	3.62	3.45	2.15	4.74	0.61	6.28	9.00
11	5.68	5.67	3.89	7.46	2.58	8.76	12.17
12	4.49	5.34	3.77	6.91	2.37	8.31	11.42
13	3.99	4.95	4.36	5.54	2.36	7.54	9.13
14	4.13	4.32	3.51	5.13	1.67	6.97	8.94
15	1.71	1.95	0.51	3.38	0.00	4.85	7.76
16	3.71	5.34	3.83	6.86	2.40	8.29	11.32
17	3.64	3.44	1.89	4.99	0.48	6.40	9.48
18	2.80	1.94	0.41	3.48	0.00	4.90	7.96
19	5.01	3.84	2.33	5.35	0.90	6.78	9.80
20	4.78	4.17	2.39	5.96	1.09	7.26	10.66

Table 13. Predictions for Expected Uranium Concentrations Using Model Developed from Chemchek Analytical Data and Prepared Levels of the Factors

Values are in milligrams/liter (mg/L) for uranium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	U-Ck	Predicted U	Lower 95% CI for Expected U	Upper 95% CI for Expected U	Lower 95% PI for Indiv U	Upper 95% PI for Indiv U	Simultaneous Upper 95% Tolerance Limit with 95% Confidence
1	7.11	6.81	5.56	8.07	4.40	9.23	11.73
2	3.05	4.07	3.58	4.55	1.95	6.19	7.49
3	3.53	4.07	3.58	4.55	1.95	6.19	7.49
4	3.34	4.07	3.58	4.55	1.95	6.19	7.49
5	3.73	3.16	1.86	4.45	0.72	5.59	8.15
6	4.38	2.73	1.54	3.92	0.34	5.11	7.52
7	9.89	8.33	7.03	9.63	5.89	10.77	13.33
8	4.88	5.63	4.37	6.88	3.21	8.04	10.55
9	6.97	7.14	5.88	8.40	4.72	9.56	12.07
10	2.87	2.83	1.77	3.89	0.51	5.15	7.37
11	4.54	4.73	3.27	6.19	2.20	7.26	10.05
12	3.69	4.26	2.97	5.55	1.83	6.70	9.24
13	3.27	4.07	3.58	4.55	1.95	6.19	7.49
14	3.19	3.58	2.92	4.25	1.42	5.75	7.36
15	1.37	1.64	0.47	2.82	0.00	4.02	6.40
16	3.05	4.35	3.10	5.59	1.94	6.75	9.23
17	2.91	2.75	1.48	4.02	0.32	5.17	7.69
18	2.23	1.56	0.30	2.82	0.00	3.98	6.48
19	4.08	3.07	1.84	4.31	0.67	5.48	7.95
20	4.28	3.54	2.08	5.00	1.01	6.07	8.85

Table 14. Predictions for Expected Plutonium Concentrations Using Model Developed from Counting Analytical Data and 6-month Levels of the Factors

Values are in milligrams/liter (mg/L) for plutonium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	Pu-C	Predicted Pu	Lower 95% CI for Expected Pu	Upper 95% CI for Expected Pu	Lower 95% PI for Individ Pu	Upper 95% PI for indiv Pu	Simultaneous Upper 95% Tolerance Limits with 95% Confidence
1	0.336	0.368	0.185	0.551	0.091	0.645	1.151
2	0.098	0.186	0.117	0.254	0.000	0.405	0.656
3	0.192	0.187	0.114	0.260	0.000	0.407	0.669
4	0.134	0.189	0.117	0.261	0.000	0.409	0.670
5	0.203	0.214	0.049	0.379	0.000	0.480	0.948
6	0.205	0.078	0.000	0.234	0.000	0.338	0.785
7	0.482	0.404	0.229	0.579	0.132	0.676	1.165
8	0.303	0.237	0.052	0.422	0.000	0.515	1.025
9	0.160	0.211	0.038	0.383	0.000	0.481	0.964
10	0.150	0.245	0.080	0.411	0.000	0.511	0.980
11	0.155	0.146	0.000	0.353	0.000	0.439	0.992
12	0.034	0.006	0.000	0.191	0.000	0.284	0.793
13	0.109	0.180	0.117	0.243	0.000	0.397	0.637
14	0.207	0.224	0.116	0.332	0.000	0.458	0.802
15	0.269	0.266	0.073	0.459	0.000	0.550	1.076
16	0.122	0.086	0.000	0.269	0.000	0.363	0.868
17	0.019	0.046	0.000	0.221	0.000	0.318	0.807
18	0.214	0.135	0.000	0.312	0.000	0.408	0.900
19	0.138	0.143	0.000	0.316	0.000	0.414	0.897
20	0.860	0.836	0.632	1.041	0.545	1.128	1.678

Table 15. Predictions for Expected Plutonium Concentrations Using Model Developed from ICP/MS Analytical Data and 6-month Levels of the Factors

Values are in milligrams/liter (mg/L) for plutonium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	Pu-ICP/MS	Predicted Pu	Lower 95% CI for Expected Pu	Upper 95% CI for Expected Pu	Lower 95% PI for Indiv Pu	Upper 95% PI for Indiv Pu	Simultaneous Upper 95% Tolerance Limits with 95% Confidence
1	0.175	0.231	0.135	0.327	0.063	0.398	0.612
2	0.062	0.153	0.119	0.186	0.012	0.294	0.397
3	0.144	0.170	0.126	0.214	0.026	0.314	0.437
4	0.081	0.147	0.111	0.184	0.005	0.289	0.398
5	0.205	0.207	0.138	0.276	0.054	0.361	0.529
6	0.076	0.049	0.000	0.134	0.000	0.210	0.408
7	0.334	0.296	0.188	0.404	0.122	0.470	0.703
8	0.238	0.202	0.100	0.305	0.031	0.373	0.597
9	0.083	0.075	0.000	0.189	0.000	0.254	0.497
10	0.042	0.020	0.000	0.095	0.000	0.176	0.354
11	0.111	0.115	0.010	0.220	0.000	0.287	0.515
12	0.006	0.011	0.000	0.111	0.000	0.181	0.401
13	0.079	0.136	0.101	0.172	0.000	0.278	0.385
14	0.139	0.205	0.153	0.256	0.058	0.351	0.488
15	0.238	0.247	0.142	0.353	0.075	0.420	0.649
16	0.033	0.010	0.000	0.117	0.000	0.184	0.414
17	0.010	0.000	0.000	0.028	0.000	0.100	0.314
18	0.140	0.112	0.021	0.204	0.000	0.277	0.485
19	0.074	0.059	0.000	0.155	0.000	0.226	0.440
20	0.531	0.422	0.323	0.522	0.253	0.592	0.811

Table 16. Predictions for Expected Uranium Concentrations Using Model Developed from ICP/MS Analytical Data and 6-month Levels of the Factors

Values are in milligrams/liter (mg/L) for uranium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	U-ICP/MS	Predicted U	Lower 95% CI for Expected U	Upper 95% CI for Expected U	Lower 95% PI for Indiv U	Upper 95% PI for Indiv U	Simultaneous Upper 95% Tolerance Limits with 95% Confidence
1	8.29	8.60	7.37	9.83	6.45	10.75	13.49
2	3.85	4.20	3.61	4.78	2.33	6.06	7.68
3	4.00	4.21	3.62	4.81	2.35	6.08	7.71
4	4.36	5.39	4.81	5.97	3.53	7.25	8.87
5	4.68	4.53	3.33	5.72	2.39	6.66	9.34
6	4.92	3.58	2.55	4.61	1.53	5.62	8.03
7	12.62	11.79	10.38	13.20	9.53	14.05	17.08
8	6.20	6.28	4.90	7.66	4.04	8.52	11.50
9	8.09	7.94	6.77	9.11	5.82	10.06	12.70
10	3.62	3.59	2.60	4.58	1.56	5.62	7.97
11	5.68	6.00	4.62	7.38	3.76	8.24	11.22
12	4.49	4.75	3.47	6.04	2.57	6.94	9.77
13	3.99	5.54	4.97	6.12	3.68	7.40	9.00
14	4.13	3.89	3.22	4.56	2.00	5.78	7.56
15	1.71	1.82	0.60	3.04	0.00	3.97	6.70
16	3.71	4.12	2.77	5.47	1.89	6.34	9.28
17	3.64	3.06	1.90	4.23	0.95	5.18	7.81
18	2.80	2.75	1.47	4.02	0.57	4.92	7.75
19	5.01	4.24	3.14	5.34	2.16	6.32	8.86
20	4.78	4.25	2.90	5.60	2.03	6.47	9.41

Table 17. Predictions for Expected Uranium Concentrations Using Model Developed from Chemchek Analytical Data and 6-month Levels of the Factors

Values are in milligrams/liter (mg/L) for uranium and moles/liter for other factors.

CI - Confidence Interval for Expected Concentration Measurement

PI - Prediction Interval for Individual Concentration Measurement

Bottle #	U-Ck	Predicted U	Lower 95% CI for Expected U	Upper 95% CI for Expected U	Lower 95% PI for Indiv U	Upper 95% PI for Indiv U	Simultaneous Upper 95% Tolerance Limit with 95% Confidence
1	7.11	6.80	5.64	7.96	4.58	9.02	11.32
2	3.05	3.81	3.36	4.26	1.87	5.75	6.96
3	3.53	3.84	3.38	4.29	1.89	5.78	7.00
4	3.34	4.02	3.57	4.46	2.07	5.96	7.17
5	3.73	2.96	1.83	4.09	0.76	5.16	7.42
6	4.38	2.82	1.78	3.85	0.66	4.97	7.09
7	9.89	8.44	7.24	9.84	6.20	10.68	13.03
8	4.88	5.65	4.45	6.85	3.41	7.89	10.25
9	6.97	7.19	6.08	8.30	5.00	9.39	11.62
10	2.87	2.92	1.96	3.88	0.80	5.04	7.05
11	4.54	4.88	3.55	6.20	2.57	7.19	9.72
12	3.69	3.87	2.64	5.10	1.62	6.12	8.52
13	3.27	4.12	3.69	4.55	2.18	6.06	7.24
14	3.19	3.67	3.12	4.21	1.70	5.64	7.00
15	1.37	1.37	0.24	2.51	0.00	3.58	5.85
16	3.05	4.19	2.91	5.46	1.91	6.47	8.93
17	2.91	3.18	2.07	4.29	0.99	5.37	7.60
18	2.23	1.38	0.18	2.57	0.00	3.61	5.96
19	4.08	3.36	2.29	4.43	1.18	5.53	7.71
20	4.28	3.92	2.62	5.22	1.63	6.22	8.71

Table 18. Plutonium and Uranium Concentrations for Special Bottle # 23

Rows	Species	Analytical	Time	Aliquot	Conc (mg/L)
1	Pu	C	14	1	1.77
2	Pu	C	14	2	2.01
3	Pu	C	14	3	1.81
4	Pu	C	25	1	1.86
5	Pu	C	25	2	1.91
6	Pu	C	25	3	1.77
7	Pu	C	31	1	1.96
8	Pu	C	31	2	2.49
9	Pu	C	31	3	2.56
10	Pu	C	40	1	1.99
11	Pu	C	40	2	1.86
12	Pu	C	40	3	2.41
13	Pu	C	56	1	1.96
14	Pu	C	56	2	1.29
15	Pu	C	56	3	2.16
16	Pu	C	74	1	1.79
17	Pu	C	74	2	1.63
18	Pu	C	74	3	1.92
19	Pu	C	81	1	1.92
20	Pu	C	81	2	1.63
21	Pu	C	81	3	1.78
22	Pu	C	84	1	1.87
23	Pu	C	84	2	1.65
24	Pu	C	84	3	1.78
25	Pu	C	122	1	1.74
26	Pu	C	122	2	1.37
27	Pu	C	122	3	2.17
28	Pu	C	153	1	2.17
29	Pu	C	153	2	1.86
30	Pu	C	153	3	-
31	Pu	C	185	1	2.21
32	Pu	C	185	2	2.21
33	Pu	C	185	3	2.92
34	Pu	ICP/MS	31	1	1.55
35	Pu	ICP/MS	31	2	1.58
36	Pu	ICP/MS	31	3	1.58
37	Pu	ICP/MS	40	1	1.71
38	Pu	ICP/MS	40	2	1.72
39	Pu	ICP/MS	40	3	1.72
40	Pu	ICP/MS	56	1	1.44
41	Pu	ICP/MS	56	2	1.74
42	Pu	ICP/MS	56	3	1.34
43	Pu	ICP/MS	74	1	1.74
44	Pu	ICP/MS	74	2	1.88
45	Pu	ICP/MS	74	3	1.68
46	Pu	ICP/MS	81	1	1.64
47	Pu	ICP/MS	81	2	1.92
48	Pu	ICP/MS	81	3	1.55
49	Pu	ICP/MS	84	1	1.29
50	Pu	ICP/MS	84	2	1.93
51	Pu	ICP/MS	84	3	1.64
52	Pu	ICP/MS	122	1	1.65
53	Pu	ICP/MS	122	2	4.08
54	Pu	ICP/MS	122	3	1.63
55	Pu	ICP/MS	153	1	1.87
56	Pu	ICP/MS	153	2	3.56
57	Pu	ICP/MS	153	3	-
58	U	Cx	31	1	9.1
59	U	Cx	31	2	11.5
60	U	Cx	31	3	12.1
61	U	Cx	40	1	11.8
62	U	Cx	40	2	11.8
63	U	Cx	40	3	12.5
64	U	Cx	56	1	12.3
65	U	Cx	56	2	12.4
66	U	Cx	56	3	12.2
67	U	Cx	74	1	14.1
68	U	Cx	74	2	13.9
69	U	Cx	74	3	14
70	U	Cx	81	1	13.7
71	U	Cx	81	2	13.9
72	U	Cx	81	3	14.1
73	U	Cx	84	1	13.7
74	U	Cx	84	2	13.3
75	U	Cx	84	3	13.7
76	U	Cx	122	1	14.1
77	U	Cx	122	2	13.4

Rows	Species	Analytical	Time	Aliquot	Conc (mg/L)
78	U	Cx	122	3	13.7
79	U	Cx	153	1	11.3
80	U	Cx	153	2	11.5
81	U	Cx	185	1	13.1
82	U	Cx	185	2	13.4
83	U	Cx	185	3	13
84	U	ICP/MS	31	1	12.8
85	U	ICP/MS	31	2	13.1
86	U	ICP/MS	31	3	13
87	U	ICP/MS	40	1	15.2
88	U	ICP/MS	40	2	15.2
89	U	ICP/MS	40	3	15.1
90	U	ICP/MS	56	1	12.4
91	U	ICP/MS	56	2	11.2
92	U	ICP/MS	56	3	11.6
93	U	ICP/MS	74	1	14.9
94	U	ICP/MS	74	2	14.2
95	U	ICP/MS	74	3	14.6
96	U	ICP/MS	81	1	14.3
97	U	ICP/MS	81	2	14
98	U	ICP/MS	81	3	13.6
99	U	ICP/MS	84	1	11.2
100	U	ICP/MS	84	2	14.4
101	U	ICP/MS	84	3	14.3
102	U	ICP/MS	122	1	15.3
103	U	ICP/MS	122	2	12.3
104	U	ICP/MS	122	3	14.8
105	U	ICP/MS	153	1	18.4
106	U	ICP/MS	153	2	17.7

Table 19. Plutonium Concentrations for Special Bottle # 24

Rows	Time (Days)	Bottle 24 - Pu-C	Bottle 24 - Pu-ICP/MS
1	25	3.54	.
2	31	3.6	3.17
3	40	3.5	0.191
4	56	2.68	2.52
5	74	2.24	3.25
6	81	3.78	3.32
7	84	2.73	3.45
8	122	3.24	3.36
9	153	3.27	3.63
10	185	3.38	.

Table 20. Uranium Concentrations for Special Bottle # 26

Rows	Time (Days)	Bottle 26 - U - ICP/MS	Bottle 26 - U - Ck
1	56	3.08	4.1
2	74	4.33	4.2
3	84	4.35	3.9
4	122	4.29	4.1
5	153	5.37	3.5
6	185	.	3.5

Table 21. Plutonium and Uranium Concentrations for Bottles 19 and 22

Rows	Bottle	Species	Analytical	Time (days)	Conc (mg/L)
1	19	Pu	C	3	0.223
2	19	Pu	C	7	0.153
3	19	Pu	C	14	0.308
4	19	Pu	C	25	0.135
5	19	Pu	C	31	0.114
6	19	Pu	ICP/MS	31	0.101
7	19	Pu	C	40	0.132
8	19	Pu	ICP/MS	40	0.0609
9	19	Pu	C	56	0.127
10	19	Pu	ICP/MS	56	0.0952
11	19	Pu	C	84	0.0977
12	19	Pu	ICP/MS	84	0.0886
13	19	Pu	C	122	0.0711
14	19	Pu	ICP/MS	122	0.0305
15	19	Pu	C	153	0.0844
16	19	Pu	ICP/MS	153	0.0693
17	19	Pu	C	185	0.0753
18	22	Pu	C	6	0.0206
19	22	Pu	ICP/MS	6	0.00444
20	22	Pu	C	12	0.0245
21	22	Pu	ICP/MS	12	0.0111
22	22	Pu	C	21	0.0257
23	22	Pu	ICP/MS	21	0.0156
24	22	Pu	C	37	0.0249
25	22	Pu	ICP/MS	37	0.0078
26	22	Pu	C	55	0.0291
27	22	Pu	ICP/MS	55	0.0125
28	22	Pu	C	62	0.0237
29	22	Pu	ICP/MS	62	0.00973
30	22	Pu	C	103	0.0141
31	22	Pu	ICP/MS	122	0.00831
32	22	Pu	C	153	0.0288
33	22	Pu	ICP/MS	153	0.0153
34	22	Pu	C	185	0.0385
35	19	U	ICP/MS	31	4.44
36	19	U	Ck	31	3.5
37	19	U	ICP/MS	40	4.79
38	19	U	Ck	40	3.5
39	19	U	ICP/MS	56	5.01
40	19	U	Ck	56	3.7
41	19	U	ICP/MS	84	5.48
42	19	U	Ck	84	4.7
43	19	U	ICP/MS	122	3.32
44	19	U	Ck	122	4.6
45	19	U	ICP/MS	153	7.04
46	19	U	Ck	153	4.3
47	19	U	Ck	185	4.3
48	22	U	ICP/MS	12	10.7
49	22	U	Ck	12	8.3
50	22	U	ICP/MS	21	12.2
51	22	U	Ck	21	9
52	22	U	ICP/MS	37	10.2
53	22	U	Ck	37	9.6
54	22	U	ICP/MS	55	11.7
55	22	U	Ck	55	10.5
56	22	U	ICP/MS	62	11.7
57	22	U	Ck	62	10.6
58	22	U	ICP/MS	103	10
59	22	U	Ck	103	10.7
60	22	U	ICP/MS	153	14.6
61	22	U	Ck	153	9
62	22	U	Ck	185	10.4

Table 22. Plutonium and Uranium Concentrations for Bottles 19 and 28

Rows	Bottle	Species	Analytical	Time (days)	Conc (mg/L)
1	19	Pu	C	3	0.223
2	19	Pu	C	7	0.153
3	19	Pu	C	14	0.308
4	19	Pu	C	25	0.135
5	19	Pu	C	31	0.114
6	19	Pu	ICP/MS	31	0.101
7	19	Pu	C	40	0.132
8	19	Pu	ICP/MS	40	0.0609
9	19	Pu	C	56	0.127
10	19	Pu	ICP/MS	56	0.0952
11	19	Pu	C	84	0.0977
12	19	Pu	ICP/MS	84	0.0886
13	19	Pu	C	122	0.0711
14	19	Pu	ICP/MS	122	0.0305
15	19	Pu	C	153	0.0844
16	19	Pu	ICP/MS	153	0.0693
17	19	Pu	C	185	0.0753
18	28A	Pu	C	4	0.303
19	28A	Pu	ICP/MS	4	0.277
20	28A	Pu	C	14	0.287
21	28A	Pu	ICP/MS	14	0.0168
22	28B	Pu	C	14	0.389
23	28B	Pu	ICP/MS	14	0.289
24	28B	Pu	C	21	0.407
25	28B	Pu	ICP/MS	21	0.335
26	28A	U	Ck	4	51.7
27	28A	U	ICP/MS	4	47.6
28	28A	U	Ck	11	36.4
29	28A	U	ICP/MS	14	33.5
30	28A	U	Ck	14	33.7
31	28B	U	ICP/MS	14	34.2
32	28B	U	Ck	14	29.4
33	28B	U	ICP/MS	21	28.8
34	28B	U	Ck	21	27
35	19	U	ICP/MS	31	4.44
36	19	U	Ck	31	3.5
37	19	U	ICP/MS	40	4.79
38	19	U	Ck	40	3.5
39	19	U	ICP/MS	56	5.01
40	19	U	Ck	56	3.7
41	19	U	ICP/MS	84	5.48
42	19	U	Ck	84	4.7
43	19	U	ICP/MS	122	3.32
44	19	U	Ck	122	4.6
45	19	U	ICP/MS	153	7.04
46	19	U	Ck	153	4.3
47	19	U	Ck	185	4.3

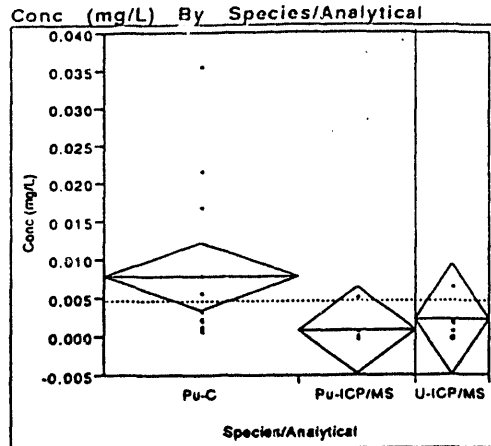
8.2 Exhibits

- Exhibit 1. Statistical Analysis of Plutonium and Uranium Concentrations for Special Bottle # 21
- Exhibit 2. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 1-5
- Exhibit 3. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 6-10
- Exhibit 4. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 11-15
- Exhibit 5. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 16-20
- Exhibit 6. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 1-5
- Exhibit 7. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 6-10
- Exhibit 8. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 11-15
- Exhibit 9. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 16-20
- Exhibit 10. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 1-5
- Exhibit 11. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 6-10
- Exhibit 12. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 11-15
- Exhibit 13. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 16-20
- Exhibit 14. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 1-5
- Exhibit 15. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 6-10
- Exhibit 16. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 11-15

- Exhibit 17. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 16-20
- Exhibit 18. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Full Model for Prepared Levels of Factors
- Exhibit 19. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Full Model for Prepared Levels of Factors
- Exhibit 20. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Full Model for Prepared Levels of Factors
- Exhibit 21. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Full Model for Prepared Levels of Factors
- Exhibit 22. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Full Model for 6-month Levels of Factors
- Exhibit 23. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Full Model for 6-month Levels of Factors
- Exhibit 24. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Full Model for 6-month Levels of Factors
- Exhibit 25. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Full Model for 6-month Levels of Factors
- Exhibit 26. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Reduced Model for Prepared Levels of Factors
- Exhibit 27. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Reduced Model for Prepared Levels of Factors
- Exhibit 28. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Reduced Model for Prepared Levels of Factors
- Exhibit 29. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Reduced Model for Prepared Levels of Factors
- Exhibit 30. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Reduced Model for 6-month Levels of Factors

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- Exhibit 31. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Reduced Model for 6-month Levels of Factors
 - Exhibit 32. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Reduced Model for 6-month Levels of Factors
 - Exhibit 33. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Reduced Model for 6-month Levels of Factors
 - Exhibit 34. Statistical Analysis of Plutonium Concentration Measurements by Counting Method for Special Bottle # 23
 - Exhibit 35. Statistical Analysis of Plutonium Concentration Measurements by ICP/MS Method for Special Bottle # 23
 - Exhibit 36. Statistical Analysis of Uranium Concentration Measurements by ICP/MS Method for Special Bottle # 23
 - Exhibit 37. Statistical Analysis of Uranium Concentration Measurements by Chemchek Method for Special Bottle # 23
 - Exhibit 38. Statistical Comparison of Analytical Methods for Measuring Plutonium Concentration for Special Bottle # 24
 - Exhibit 39. Statistical Comparison of Analytical Methods for Measuring Uranium Concentration for Special Bottle # 26
 - Exhibit 40. Statistical Analysis of Plutonium Concentrations by Analytical Method and Bottle for Bottles # 19 and 22
 - Exhibit 41. Statistical Analysis of Uranium Concentrations by Analytical Method and Bottle for Bottles # 19 and 22
 - Exhibit 42. Statistical Analysis of Plutonium Concentrations by Analytical Method and Bottle for Bottles # 19 and 28
 - Exhibit 43. Statistical Analysis of Uranium Concentrations by Analytical Method and Bottle for Bottles # 19 and 28

Exhibit 1. Statistical Analysis of Plutonium and Uranium Concentrations for Special Bottle # 21



Means with confid. interval

Means

Summary of Fit

Rsquare 0.169704
 Root Mean Square Error 0.007831
 Mean of Response 0.004716
 Observations (or Sum Wgts) 26

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.00028828	0.000144	2.3505
Error	23	0.00141044	0.000061	Prob>F
C Total	25	0.00169872		0.1178

Mean Estimates

Level	number	Mean	Std Error
Pu-C	13	0.008002	0.00217
Pu-ICP/MS	8	0.000827	0.00277
U-Ck	0	-	-
U-ICP/MS	5	0.002396	0.00350

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDII to Mean	MeanAbsDII to Median
Pu-C	13	0.0106396	0.0078373	0.0063812
Pu-ICP/MS	8	0.0018798	0.0011866	0.0008269
U-Ck	0	-	0.0000000	0.0000000
U-ICP/MS	5	0.0026119	0.0017616	0.0016320

Test	F Ratio	DF Num	DF Den	Prob>F
O'Brien[.5]	1.2778	2	22	0.2985
Brown-Forsythe	1.5667	2	22	0.2311
Levene	5.0703	2	22	0.0155
Bartlett	10.2055	3	-	0.0000

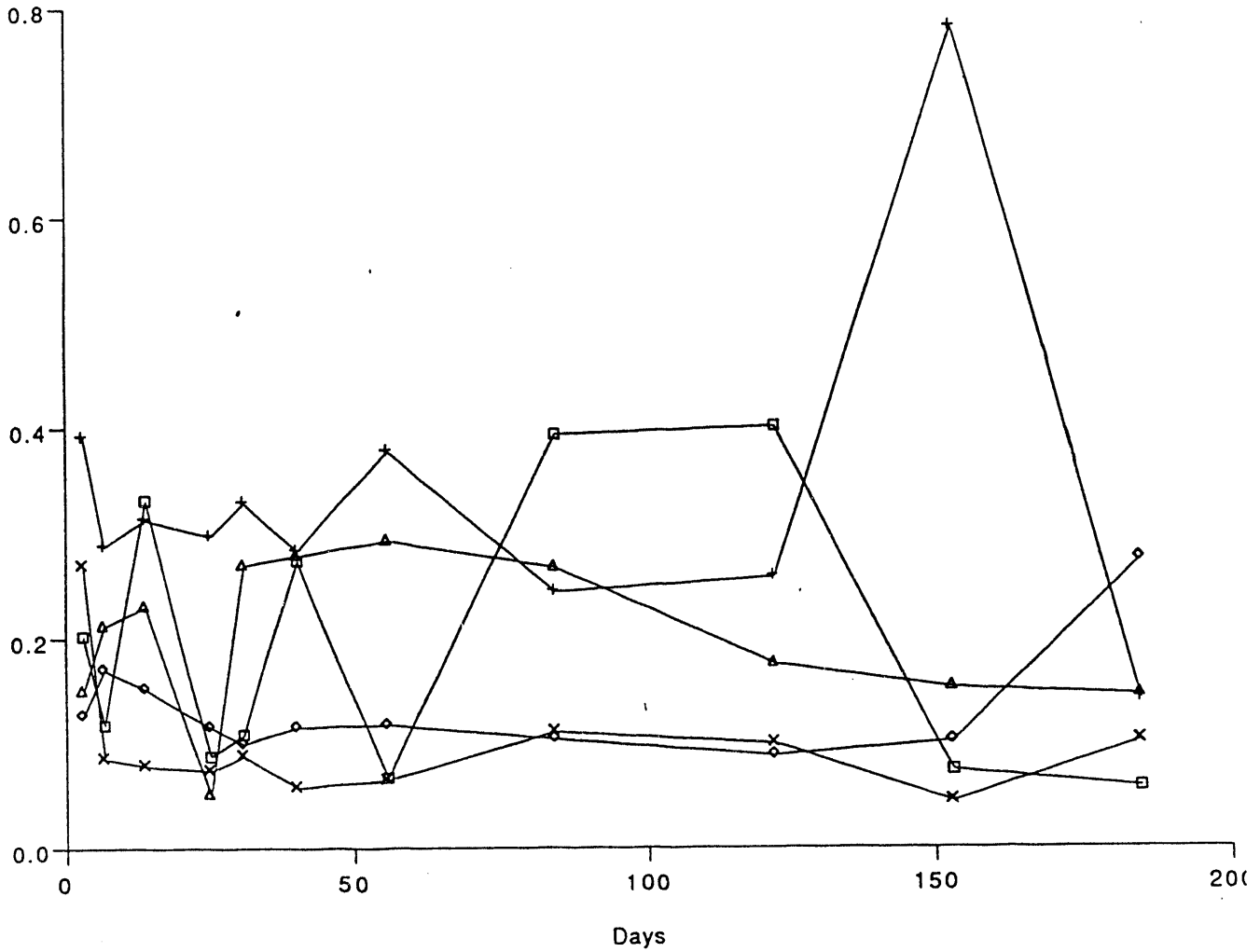
Warning: Small sample sizes. Use Caution.

Welch Anova testing Means Equal, allowing Std's Not Equal

F Ratio	DF Num	DF Den	Prob>F
-	2	11.396	-1

Exhibit 2. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 1-5

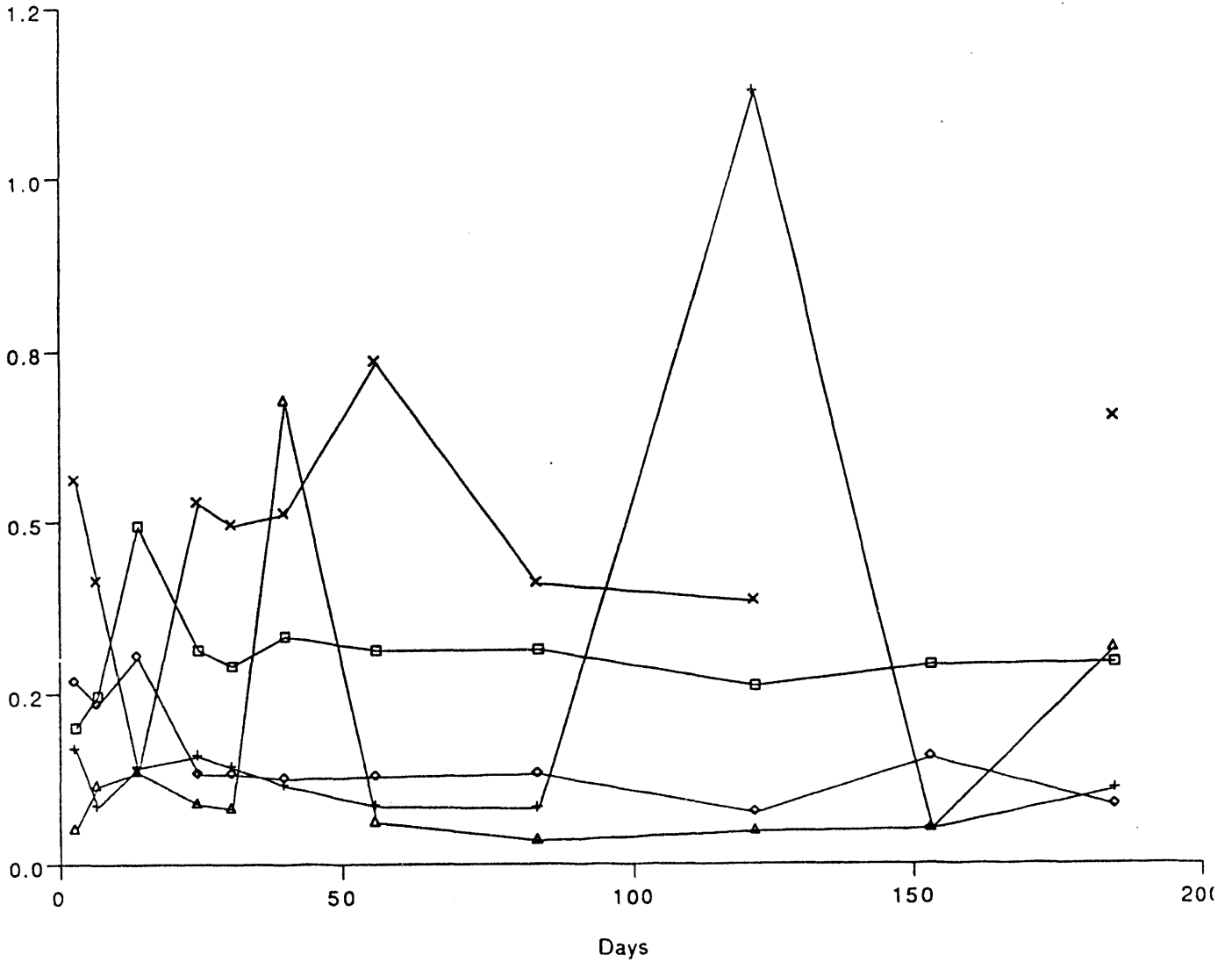
Plutonium concentrations are in milligrams/liter.



- + — Bottle 1
- x — Bottle 2
- — Bottle 3
- ◇ — Bottle 4
- △ — Bottle 5

Exhibit 3. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 6-10

Plutonium concentrations are in milligrams/liter.



- + — Bottle 6
- x — Bottle 7
- — Bottle 8
- ◇ — Bottle 9
- Δ — Bottle 10

Exhibit 4. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 11-15

Plutonium concentrations are in milligrams/liter.

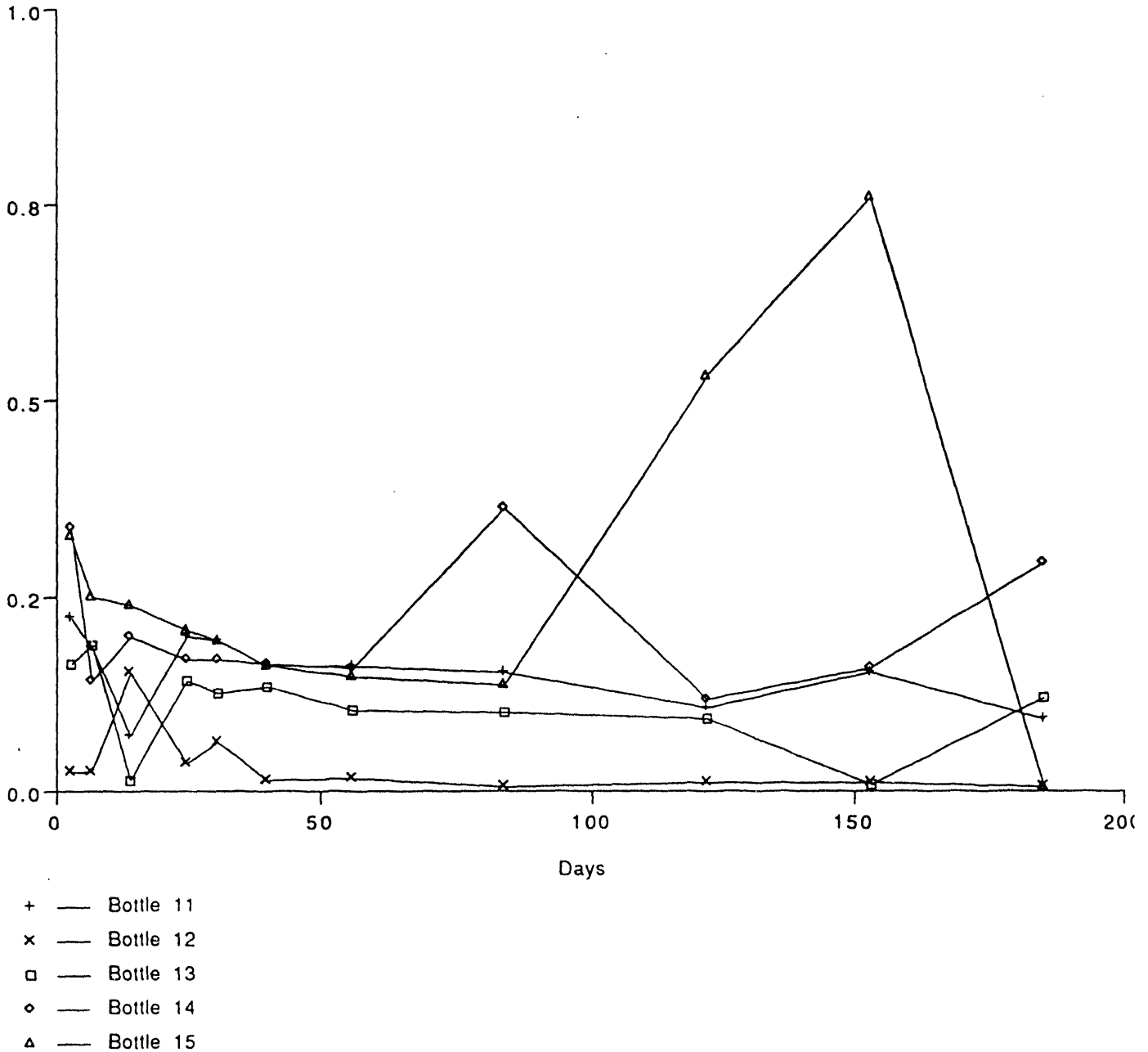


Exhibit 5. Time Plot of Plutonium Concentrations as Measured by Counting Method for Bottles 16-20

Plutonium concentrations are in milligrams/liter.

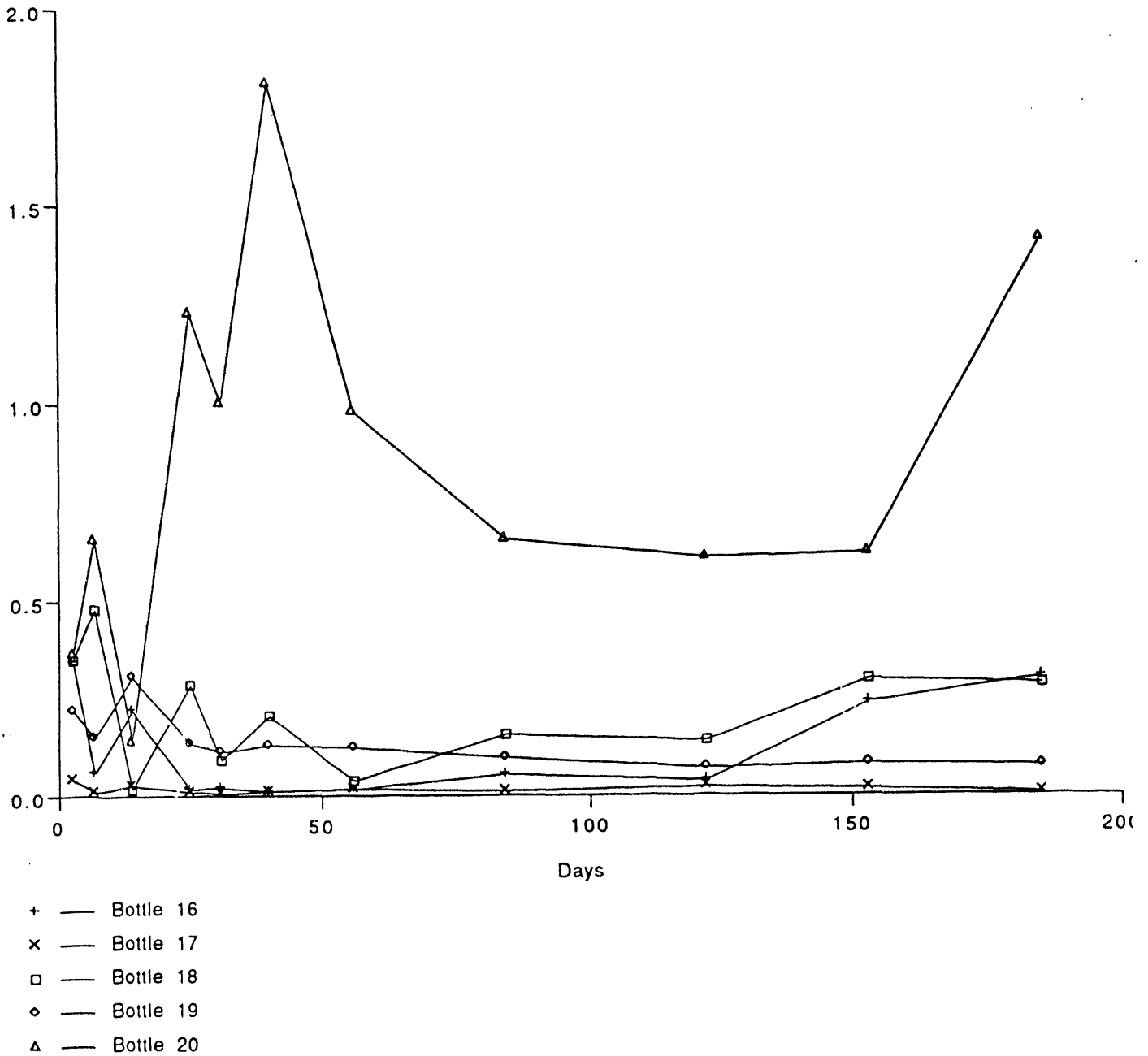
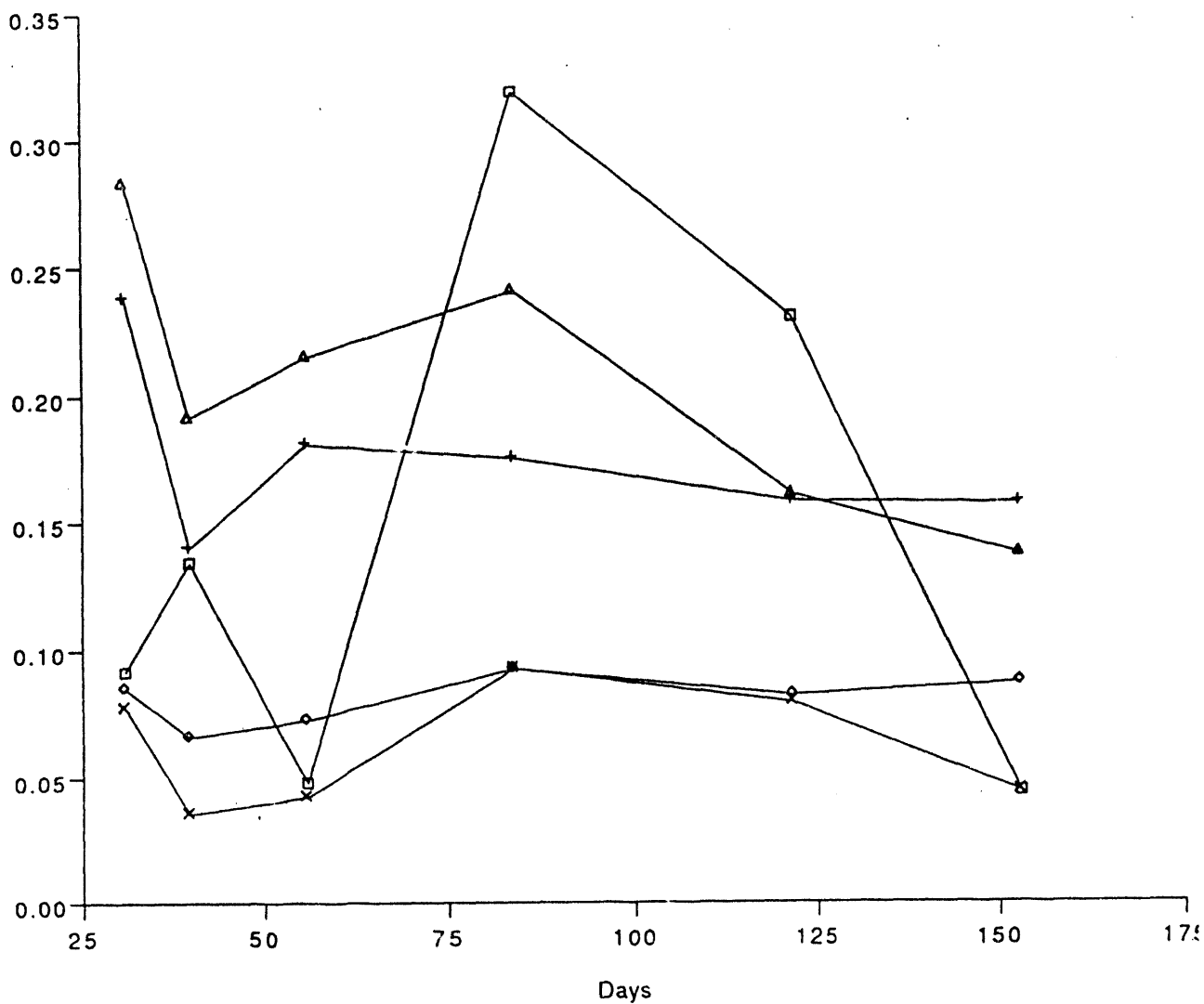


Exhibit 6. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 1-5

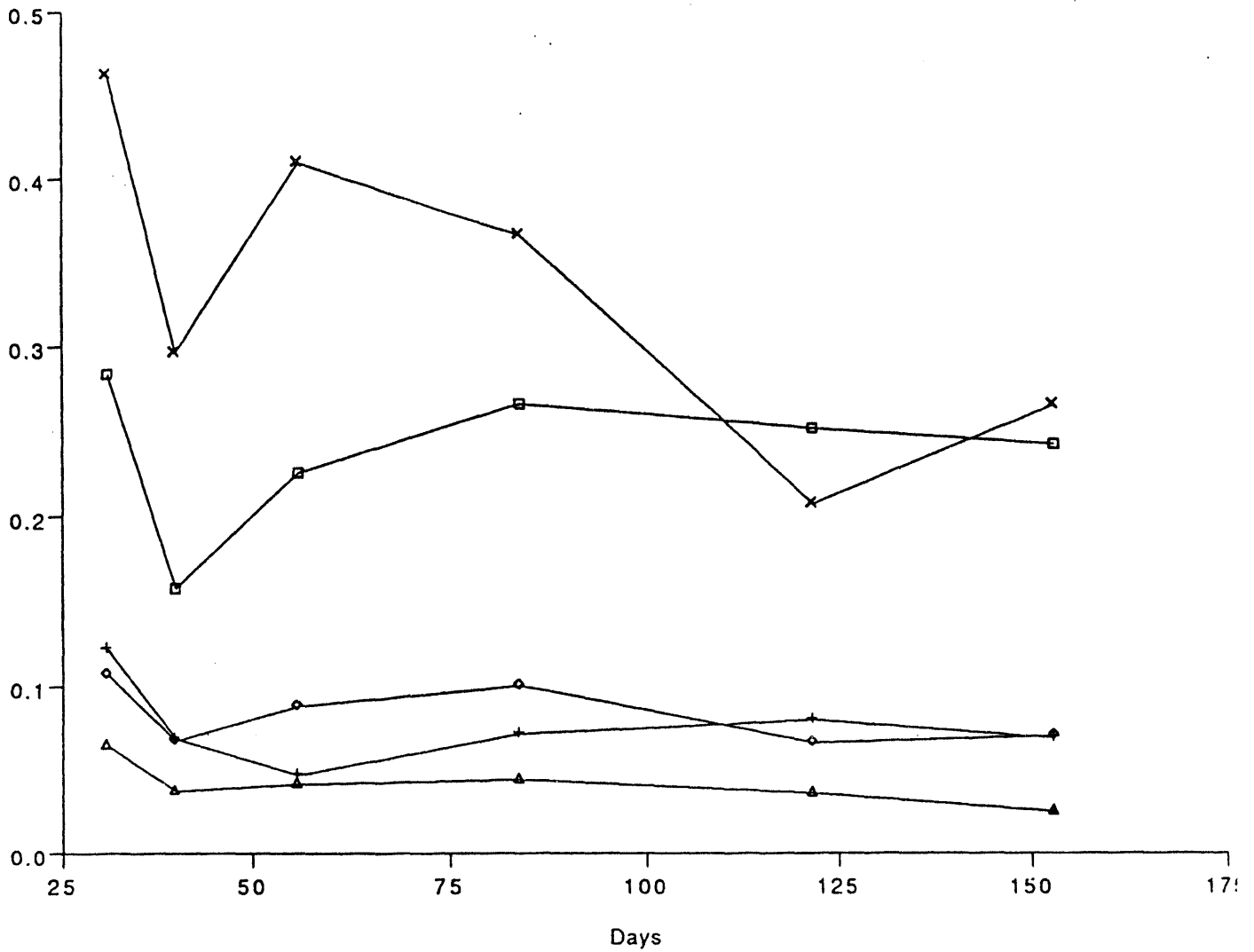
Plutonium concentrations are in milligrams/liter.



- + — Bottle 1
- x — Bottle 2
- — Bottle 3
- ◇ — Bottle 4
- △ — Bottle 5

Exhibit 7. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 6-10

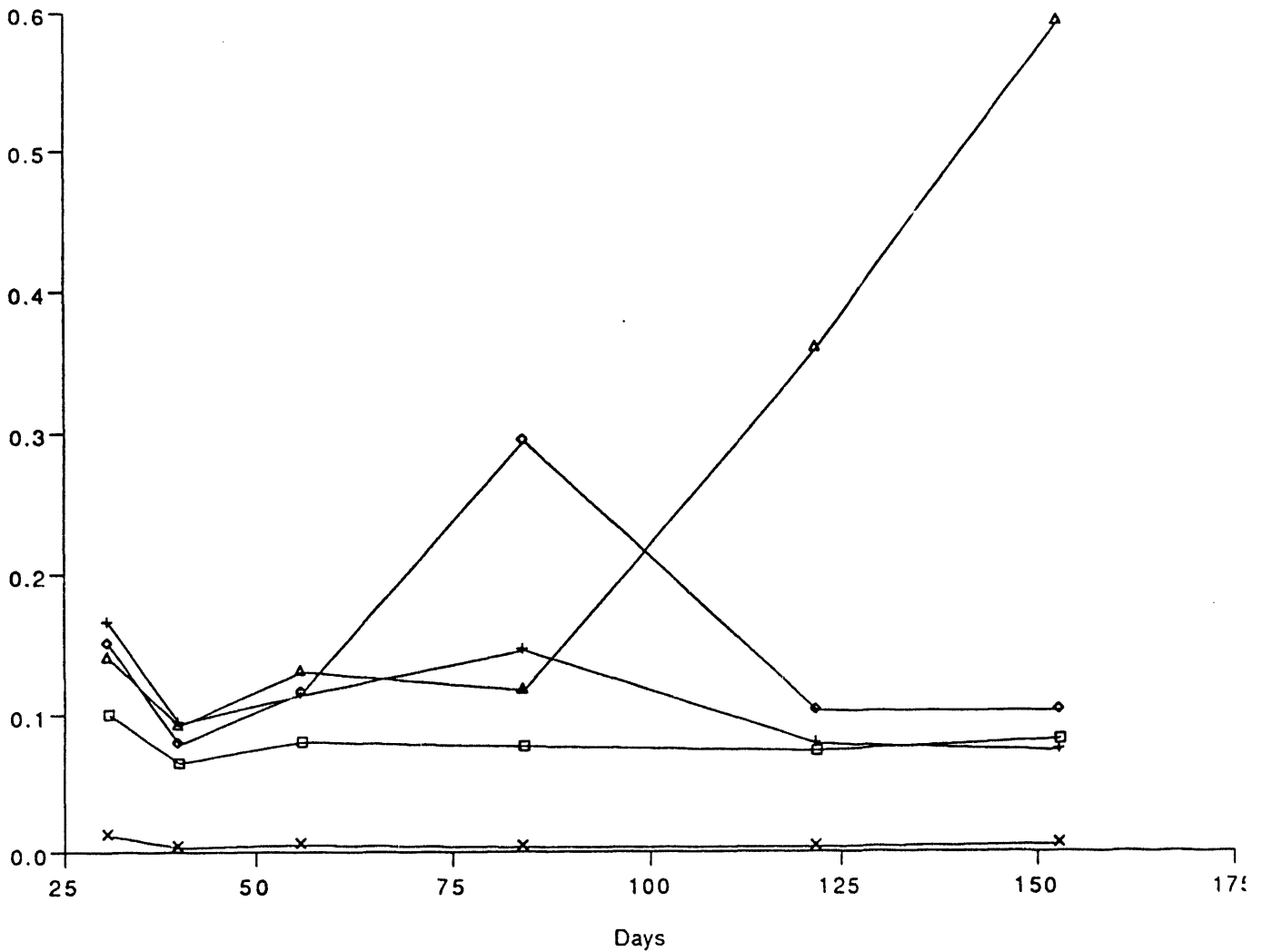
Plutonium concentrations are in milligrams/liter.



- + — Bottle 6
- x — Bottle 7
- — Bottle 8
- ◇ — Bottle 9
- △ — Bottle 10

Exhibit 8. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 11-15

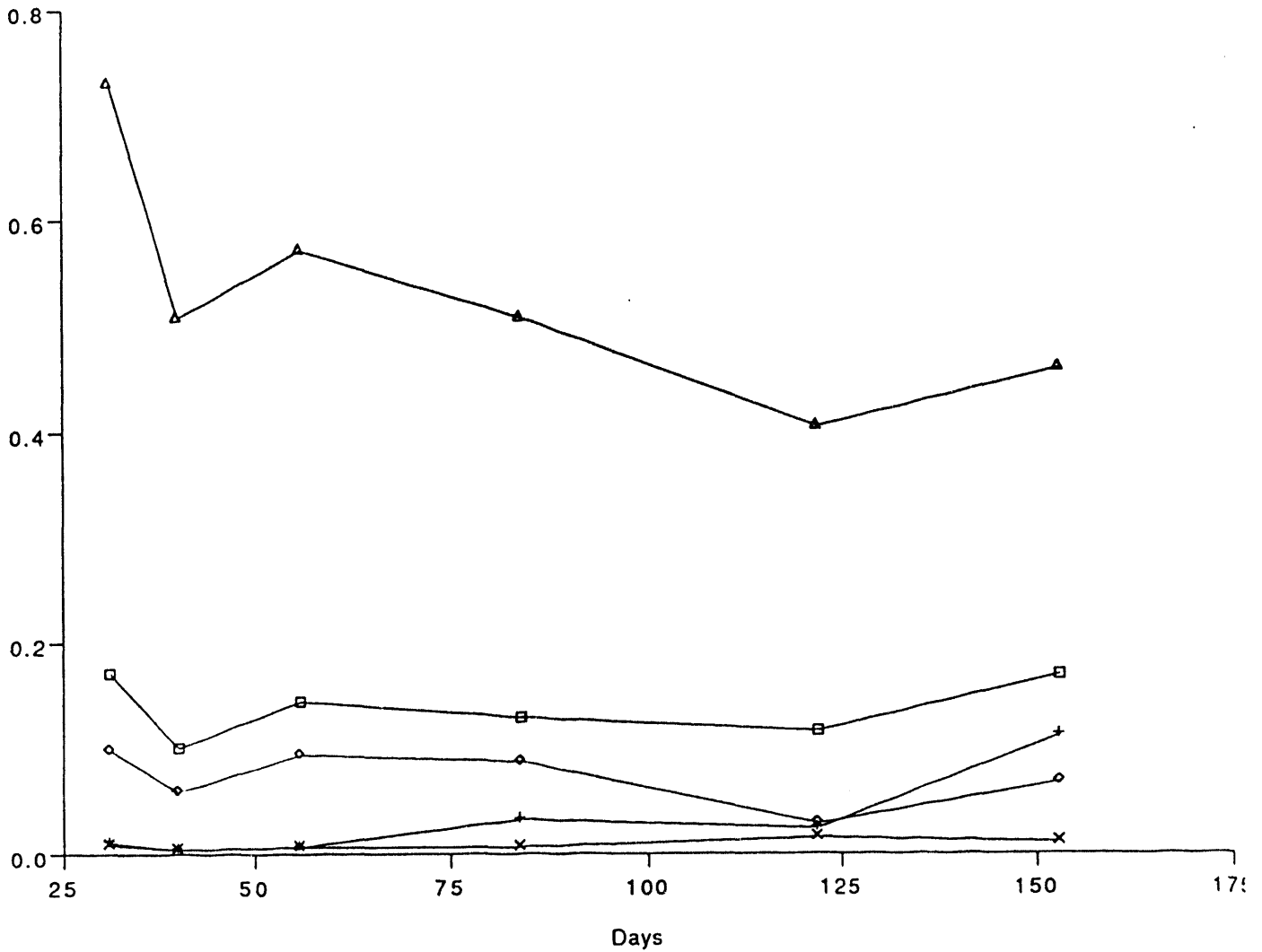
Plutonium concentrations are in milligrams/liter.



- + — Bottle 11
- x — Bottle 12
- — Bottle 13
- ◇ — Bottle 14
- △ — Bottle 15

Exhibit 9. Time Plot of Plutonium Concentrations as Measured by ICP/MS Method for Bottles 16-20

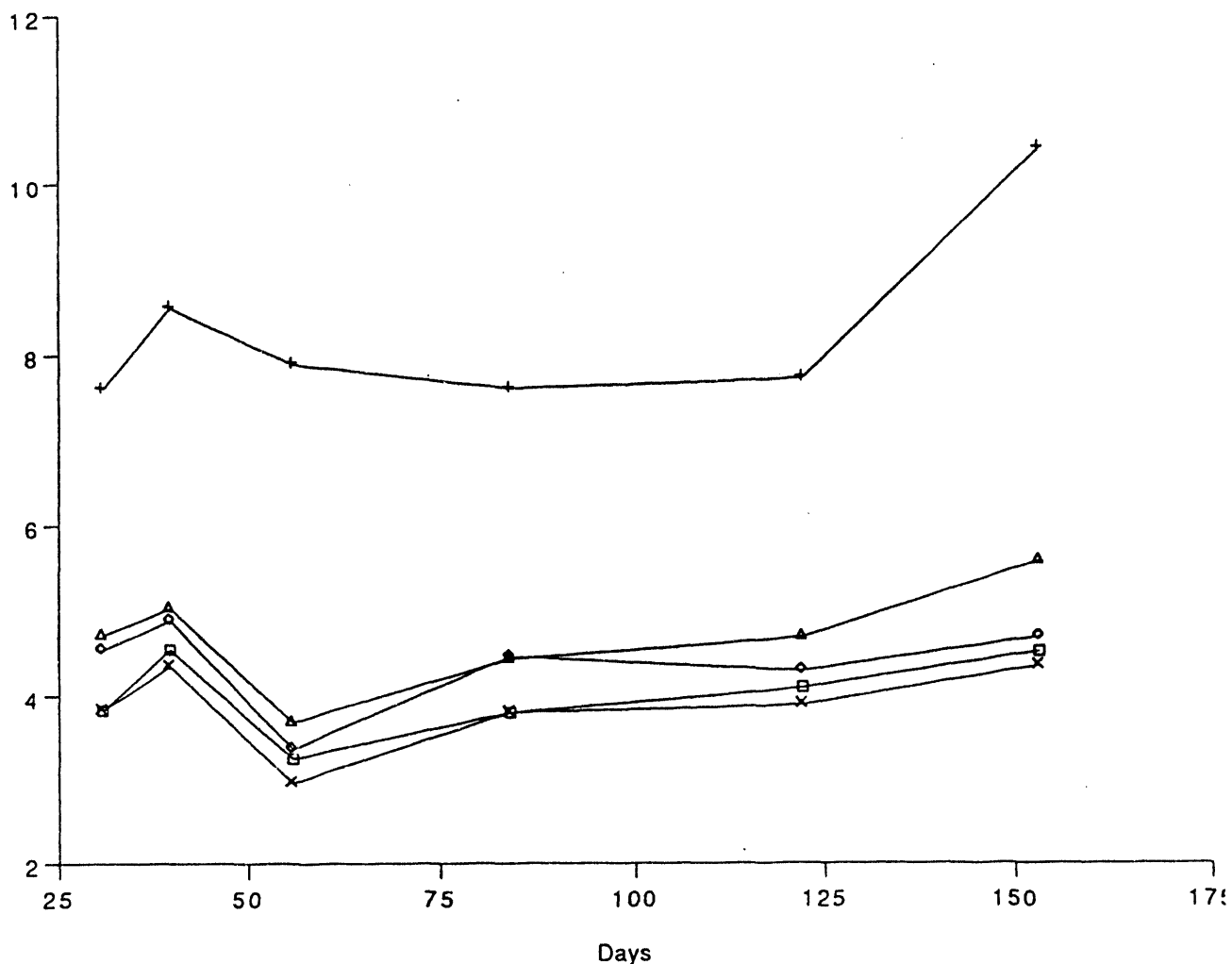
Plutonium concentrations are in milligrams/liter.



- + — Bottle 16
- x — Bottle 17
- — Bottle 18
- ◊ — Bottle 19
- Δ — Bottle 20

Exhibit 10. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 1-5

Uranium concentrations are in milligrams/liter.



- + — Bottle 1
- x — Bottle 2
- — Bottle 3
- ◇ — Bottle 4
- △ — Bottle 5

Exhibit 11. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 6-10

Uranium concentrations are in milligrams/liter.

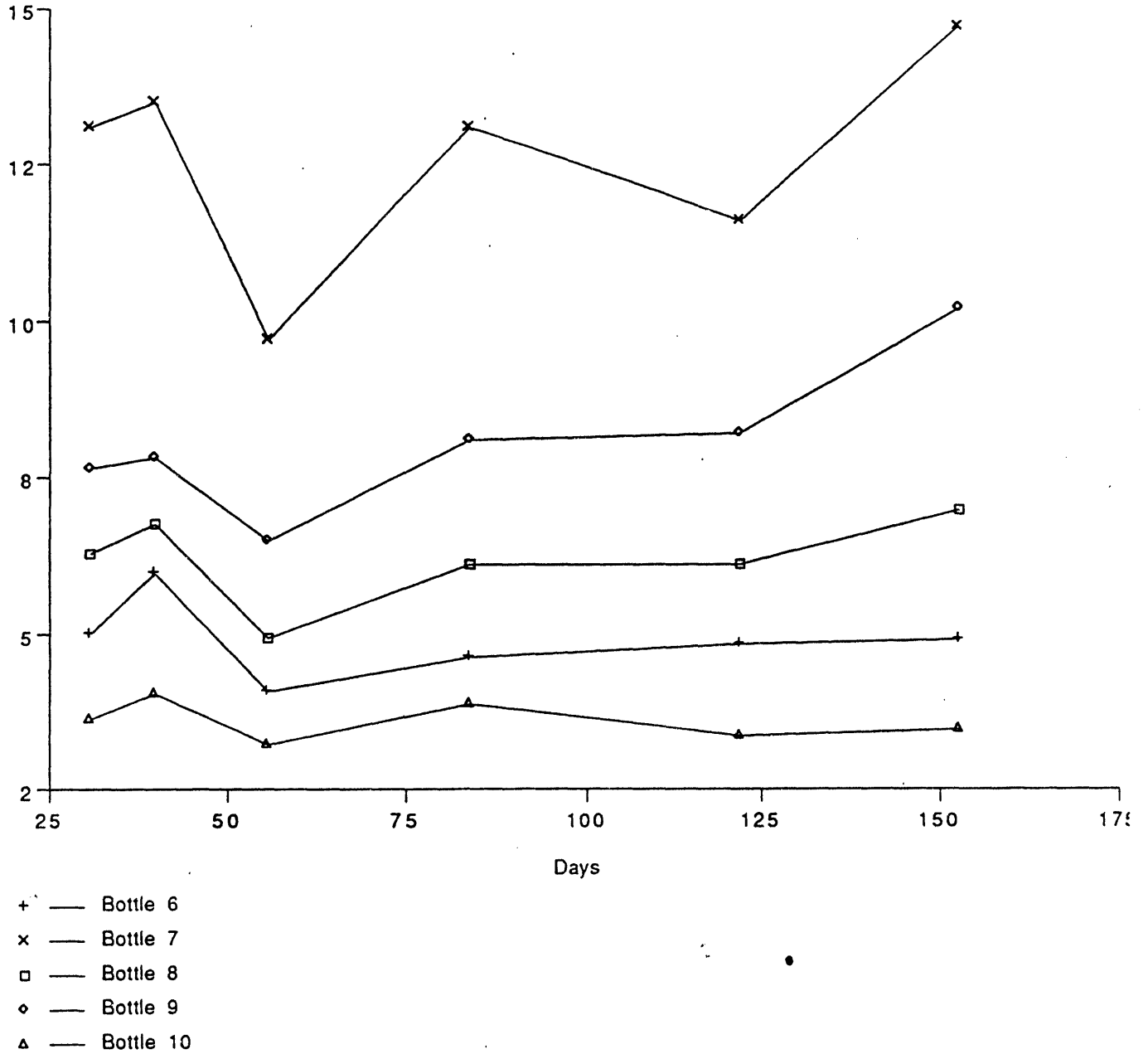
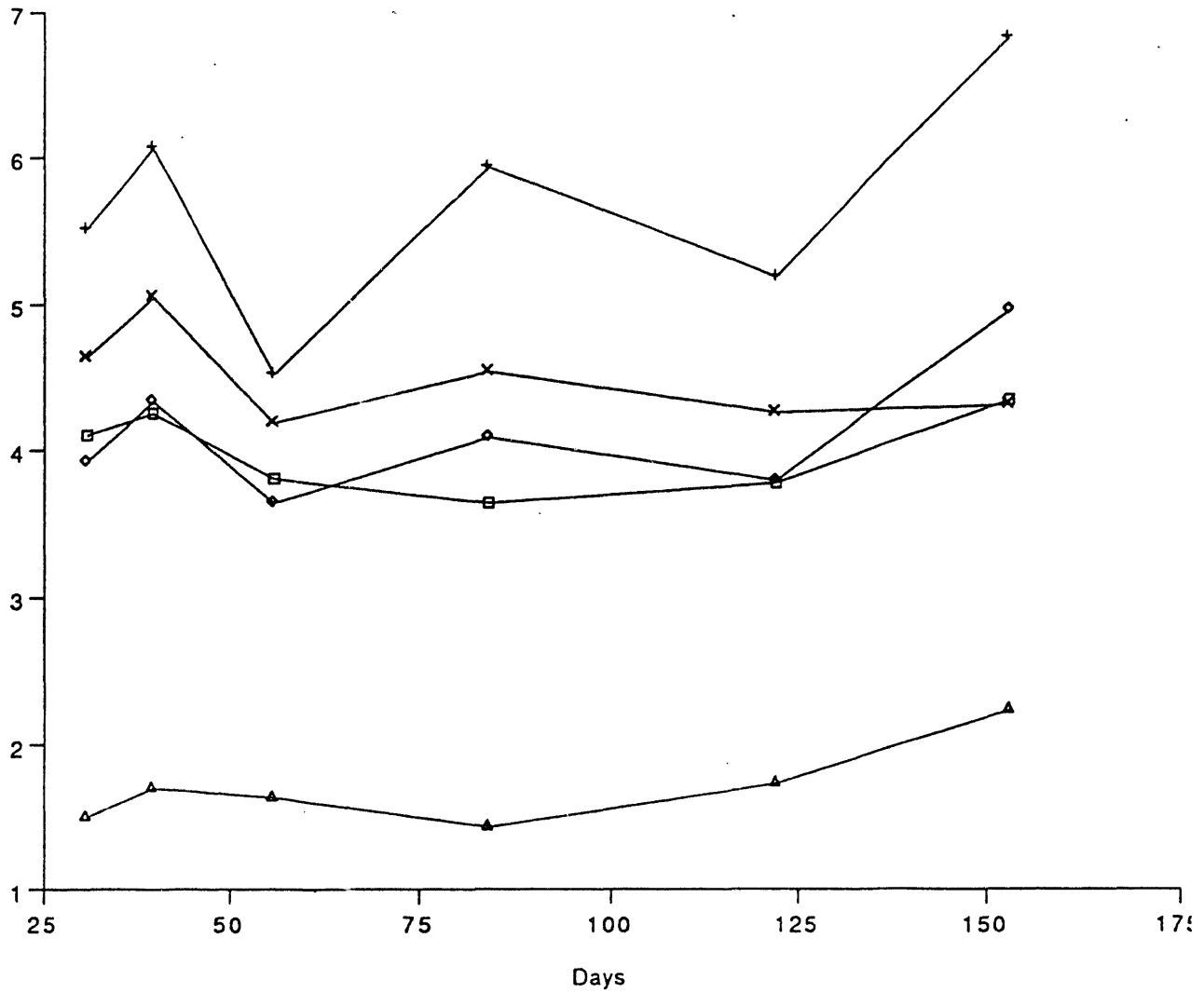


Exhibit 12. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 11-15

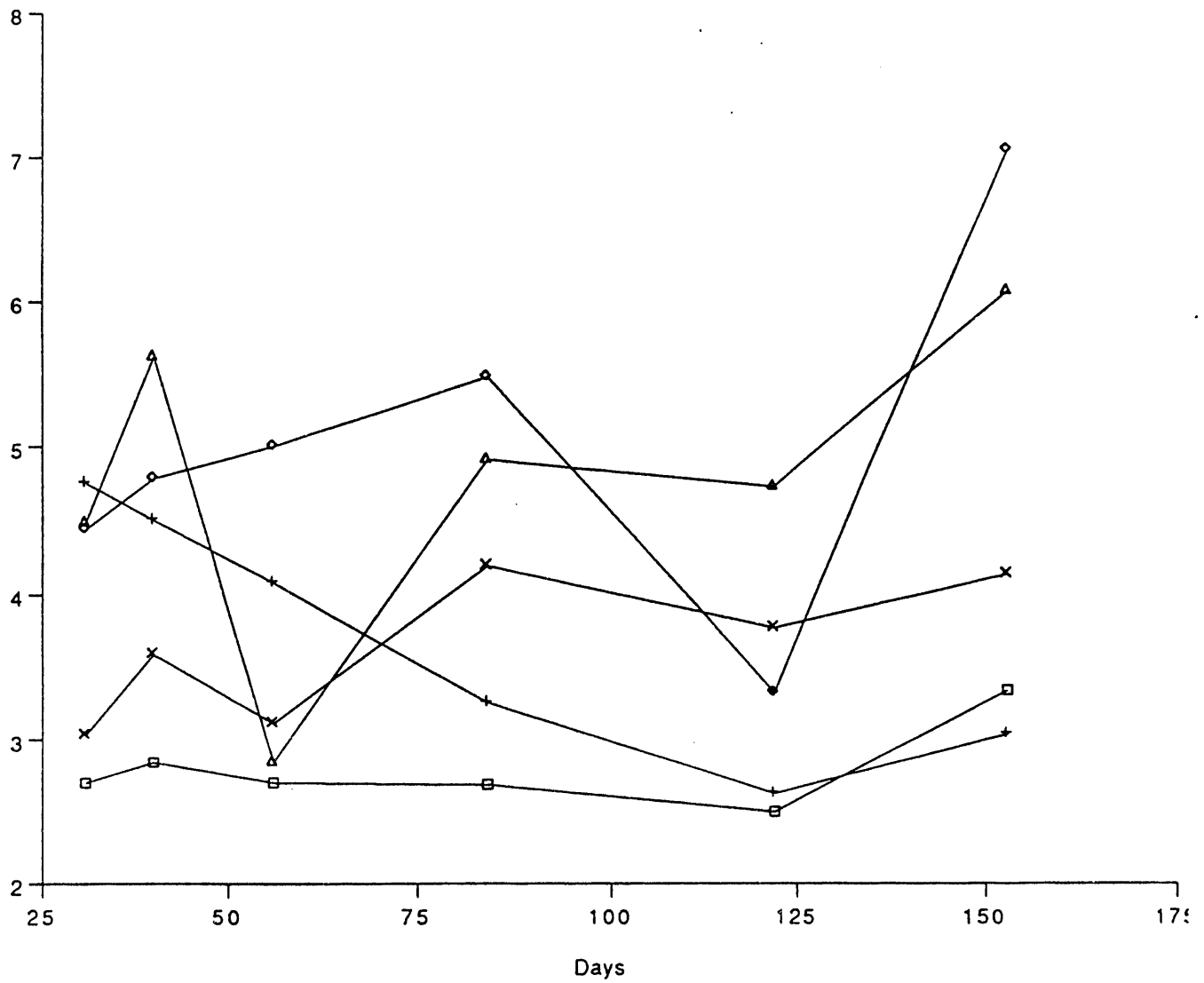
Uranium concentrations are in milligrams/liter.



- + — Bottle 11
- x — Bottle 12
- — Bottle 13
- ◇ — Bottle 14
- △ — Bottle 15

Exhibit 13. Time Plot of Uranium Concentrations as Measured by ICP/MS Method for Bottles 16-20

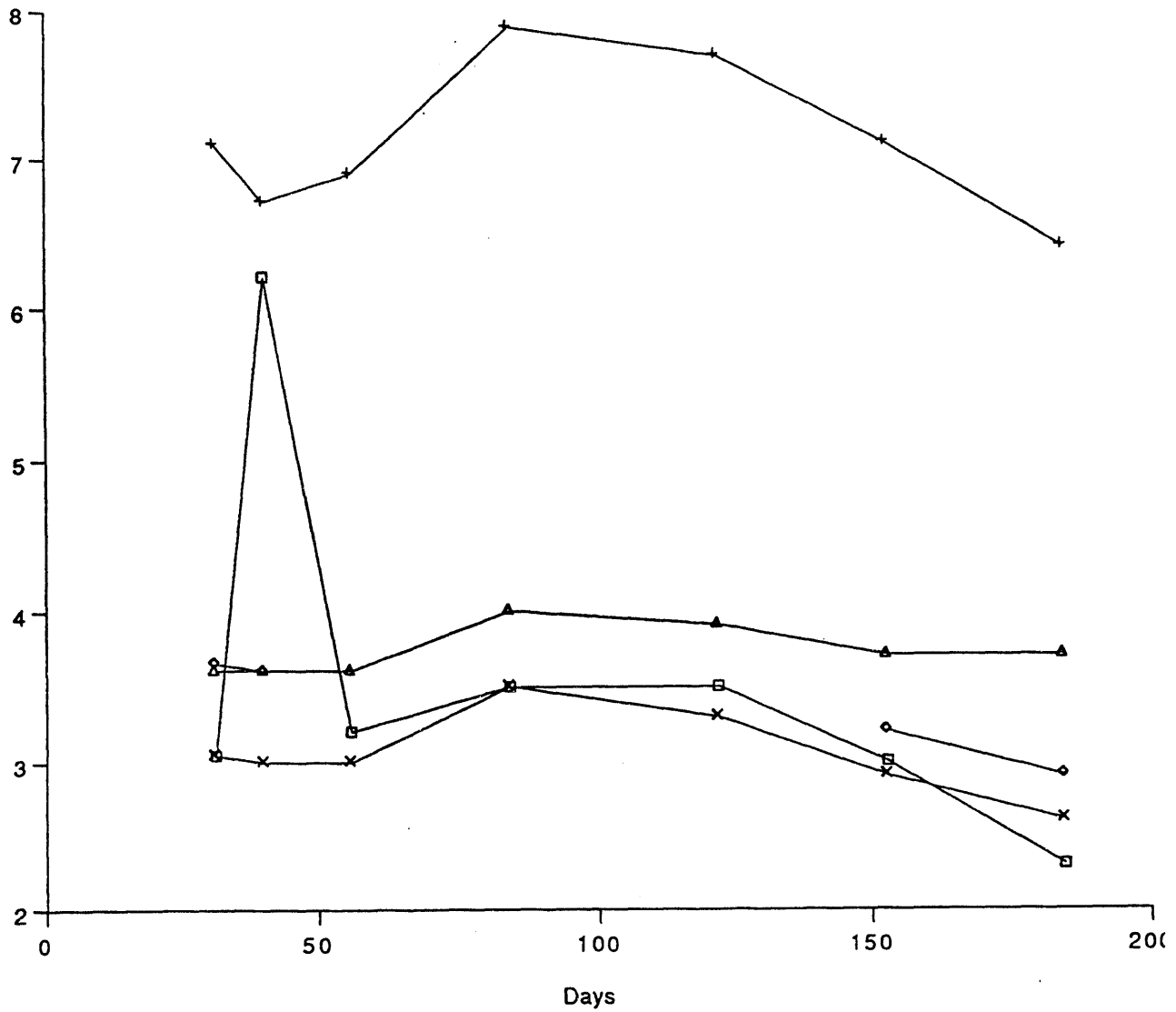
Uranium concentrations are in milligrams/liter.



- + — Bottle 16
- x — Bottle 17
- — Bottle 18
- ◇ — Bottle 19
- △ — Bottle 20

Exhibit 14. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 1-5

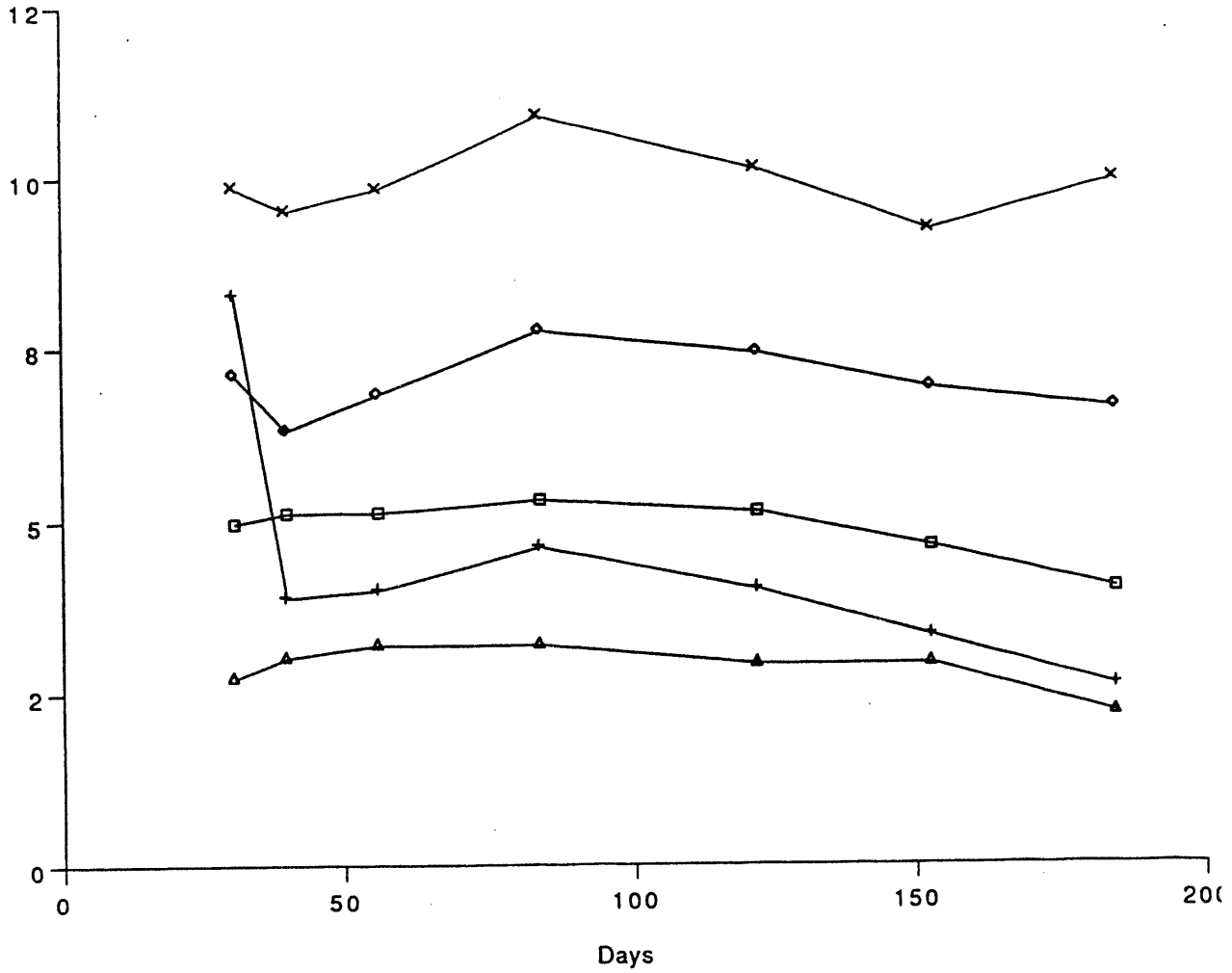
Uranium concentrations are in milligrams/liter.



- + — Bottle 1
- x — Bottle 2
- — Bottle 3
- ◇ — Bottle 4
- △ — Bottle 5

Exhibit 15. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 6-10

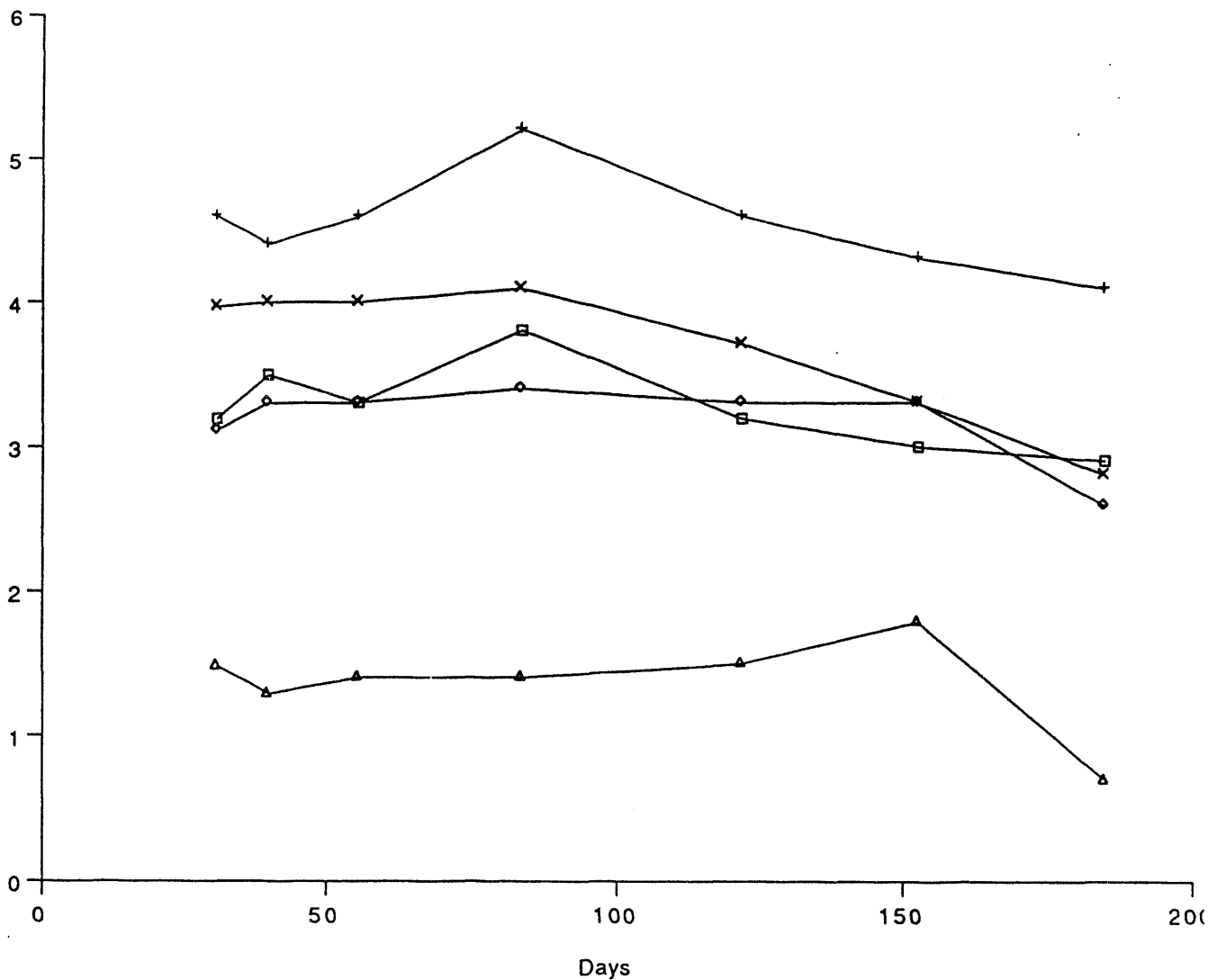
Uranium concentrations are in milligrams/liter.



- + — Bottle 6
- x — Bottle 7
- — Bottle 8
- ◇ — Bottle 9
- △ — Bottle 10

Exhibit 16. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 11-15

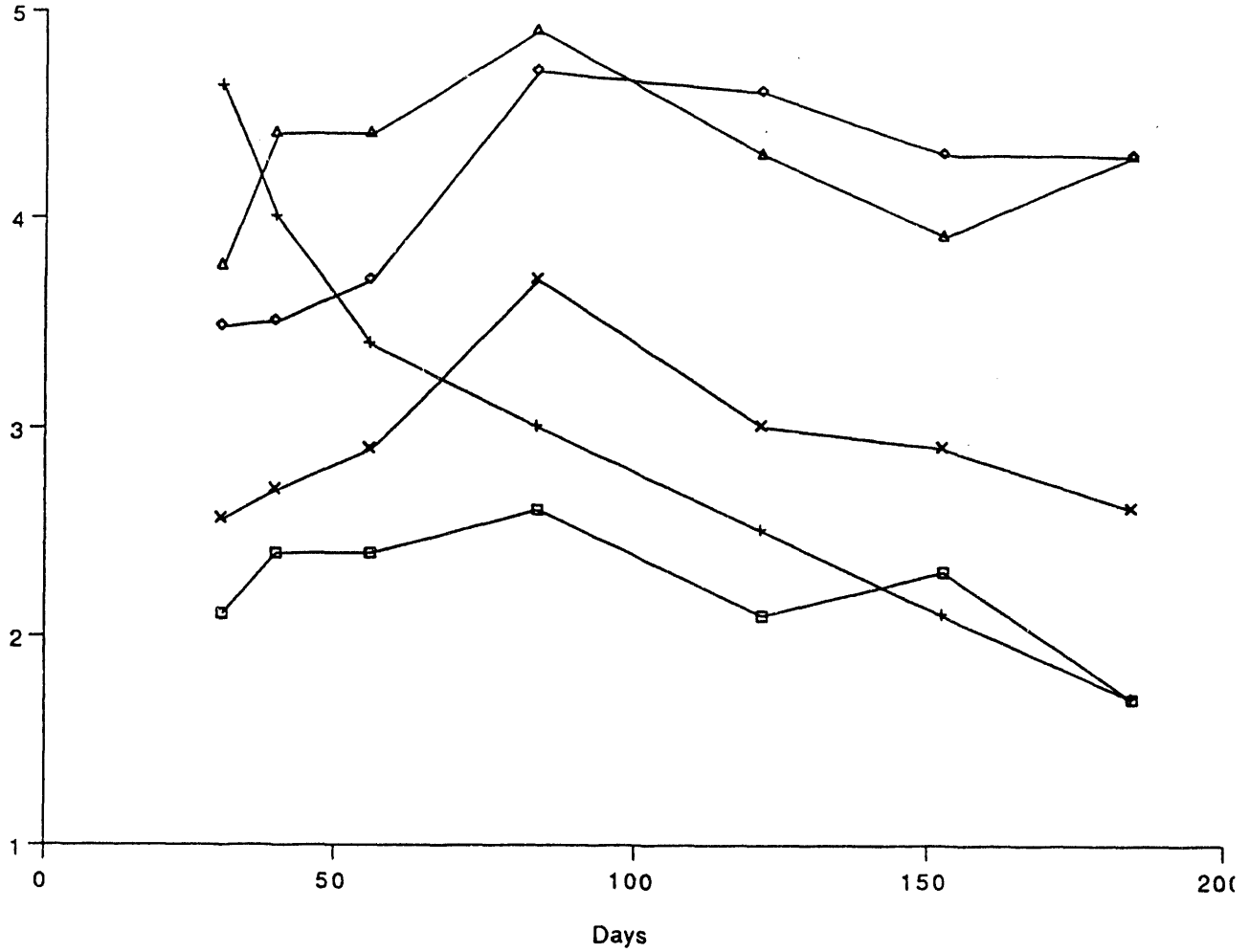
Uranium concentrations are in milligrams/liter.



- + — Bottle 11
- x — Bottle 12
- — Bottle 13
- ◇ — Bottle 14
- △ — Bottle 15

Exhibit 17. Time Plot of Uranium Concentrations as Measured by Chemchek Method for Bottles 16-20

Uranium concentrations are in milligrams/liter.



- + — Bottle 16
- x — Bottle 17
- — Bottle 18
- ◇ — Bottle 19
- △ — Bottle 20

Exhibit 18. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Full Model for Prepared Levels of Factors

Response: Pu-C

Summary of Fit

Rsquare	0.940763
Root Mean Square Error	0.097458
Mean of Response	0.219333
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	0.03327311	0.016637	7.0506
Pure Error	2	0.00471918	0.002360	Prob>F
Total Error	4	0.03799229		0.1242

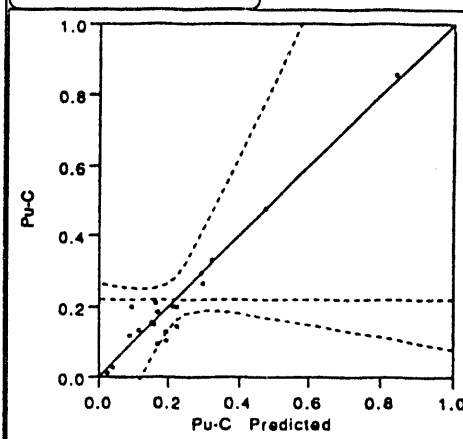
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.0886855	0.19745	0.45	0.6766
NO3	0.0083988	0.0471	0.14	0.8985
NO2	-0.055403	0.09918	-0.56	0.6062
CH	-0.188198	0.22238	-0.85	0.4450
AL(OH)4	-0.165755	0.36999	-0.45	0.6773
CO3	0.1222274	0.39392	0.31	0.7718
SO4	-0.416053	0.29031	-1.43	0.2251
Temp[H-L]	-0.013266	0.05902	-0.22	0.8332
NO3*NO2*OH	-0.101412	0.04581	-2.21	0.0913
OH*NO3	0.0346263	0.03253	1.06	0.3471
OH*NO2	0.0945039	0.07799	1.21	0.2923
OH*AL(OH)4	0.3006492	0.42722	0.70	0.5204
OH*CO3	0.4810464	0.44321	1.09	0.3388
OH*SO4	0.8408519	0.32654	2.58	0.0616
OH*Temp[H-L]	0.0011492	0.0616	0.02	0.9860
NO2*NO3	0.0668344	0.03974	1.68	0.1679

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00017529	0.0185	0.8985
NO2	1	1	0.00296405	0.3121	0.6062
CH	1	1	0.00680266	0.7162	0.4450
AL(OH)4	1	1	0.00190627	0.2007	0.6773
CO3	1	1	0.00091443	0.0963	0.7718
SO4	1	1	0.01950753	2.0538	0.2251
Temp	1	1	0.00047983	0.0505	0.8332
NO3*NO2*OH	1	1	0.04653712	4.8996	0.0913
OH*NO3	1	1	0.01076228	1.1331	0.3471
OH*NO2	1	1	0.01394520	1.4682	0.2923
OH*AL(OH)4	1	1	0.00470380	0.4952	0.5204
OH*CO3	1	1	0.01118880	1.1780	0.3388
OH*SO4	1	1	0.06298087	6.6309	0.0616
OH*Temp	1	1	0.00000331	0.0003	0.9860
NO2*NO3	1	1	0.02687124	2.8291	0.1679

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	15	0.60337130	0.040225	4.2350
Error	4	0.03799229	0.009498	Prob>F
C Total	19	0.64136359		0.0864

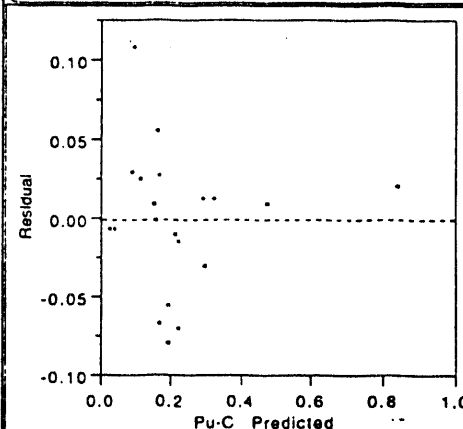


Exhibit 19.

Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Full Model for Prepared Levels of Factors

Response: Pu-ICP/MS

Summary of Fit

Rsquare	0.968709
Root Mean Square Error	0.048169
Mean of Response	0.139992
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	0.00586891	0.002934	1.7200
Pure Error	2	0.00341217	0.001706	Prob>F
Total Error	4	0.00928108		0.3676

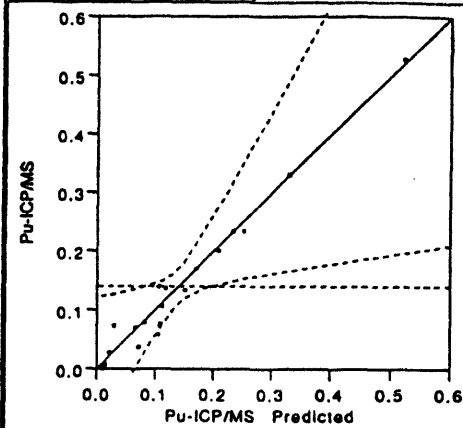
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.0520327	0.09759	0.53	0.6222
NO3	0.0187443	0.02328	0.81	0.4659
NO2	-0.016022	0.04902	-0.33	0.7602
CH	-0.165532	0.10991	-1.51	0.2065
AL(OH)4	-0.241485	0.18287	-1.32	0.2571
CO3	0.3359482	0.1947	1.73	0.1595
SO4	-0.479909	0.14349	-3.34	0.0287
Temp(H-L)	-0.010176	0.02917	-0.35	0.7448
NO3*NO2*OH	-0.067279	0.02264	-2.97	0.0411
OH*NO3	0.0141948	0.01608	0.88	0.4272
OH*NO2	0.0558649	0.03855	1.45	0.2209
OH*AL(OH)4	0.4127433	0.21116	1.95	0.1223
OH*CO3	0.2666085	0.21906	1.22	0.2905
OH*SO4	0.6840315	0.16139	4.24	0.0133
OH*Temp(H-L)	0.0070073	0.03045	0.23	0.8293
NO2*NO3	0.0367212	0.01964	1.87	0.1349

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00150420	0.6483	0.4659
NO2	1	1	0.00024788	0.1068	0.7602
CH	1	1	0.00526276	2.2682	0.2065
AL(OH)4	1	1	0.00404605	1.7438	0.2571
CO3	1	1	0.00690807	2.9773	0.1595
SO4	1	1	0.02595519	11.1863	0.0287
Temp	1	1	0.00028233	0.1217	0.7448
NO3*NO2*OH	1	1	0.02048208	8.8275	0.0411
OH*NO3	1	1	0.00180863	0.7795	0.4272
OH*NO2	1	1	0.00487308	2.1002	0.2209
OH*AL(OH)4	1	1	0.00886521	3.8208	0.1223
OH*CO3	1	1	0.00343683	1.4812	0.2905
OH*SO4	1	1	0.04167944	17.9632	0.0133
OH*Temp	1	1	0.00012290	0.0530	0.8293
NO2*NO3	1	1	0.00811187	3.4961	0.1349

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	15	0.28732532	0.019155	8.2555
Error	4	0.00928108	0.002320	Prob>F
C Total	19	0.29660640		0.0272

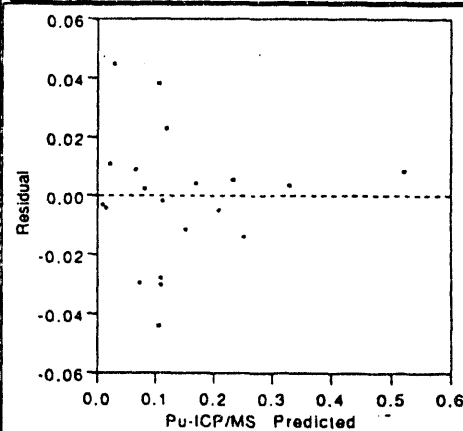


Exhibit 20.

Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Full Model for Prepared Levels of Factors

Response: U-ICP/MS

Summary of Fit

Rsquare	0.957628
Root Mean Square Error	1.061414
Mean of Response	5.026917
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	4.4239566	2.21198	53.6626
Pure Error	2	0.0824403	0.04122	Prob>F
Total Error	4	4.5063969		0.0183

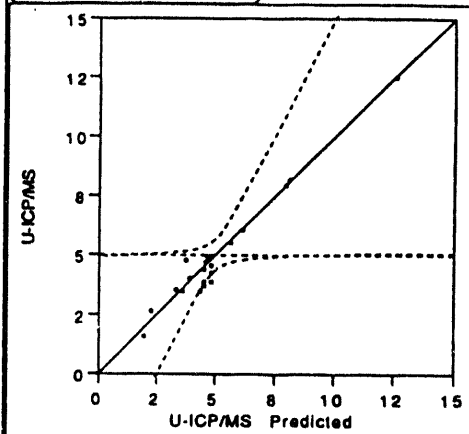
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.9940572	2.15042	0.93	0.4063
NO3	0.2924231	0.51298	0.57	0.5991
NO2	-0.928052	1.08013	-0.86	0.4387
OH	4.6632103	2.42192	1.93	0.1265
AL(OH)4	5.1619831	4.02956	1.28	0.2694
CO3	-0.636887	4.29021	-0.15	0.8892
SO4	-1.73634	3.16178	-0.55	0.6121
Temp(H-L)	-0.363993	0.64279	-0.57	0.6015
NO3*NO2*OH	-0.295897	0.49897	-0.59	0.5851
OH*NO3	-0.984577	0.35427	-2.78	0.0499
OH*NO2	-0.209135	0.84942	-0.25	0.8176
OH*AL(OH)4	-0.532675	4.65286	-0.11	0.9144
OH*CO3	-2.672816	4.82703	-0.55	0.6093
OH*SO4	3.0219802	3.55631	0.85	0.4433
OH*Temp(H-L)	0.1479238	0.67091	0.22	0.8363
NO2*NO3	0.2910382	0.43275	0.67	0.5381

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.3680904	0.3250	0.5991
NO2	1	1	0.8316889	0.7382	0.4387
OH	1	1	4.1765640	3.7072	0.1265
AL(OH)4	1	1	1.8487706	1.6410	0.2694
CO3	1	1	0.0248278	0.0220	0.8892
SO4	1	1	0.3397627	0.3016	0.6121
Temp	1	1	0.3612553	0.3207	0.6015
NO3*NO2*OH	1	1	0.3961883	0.3517	0.5851
OH*NO3	1	1	8.7014549	7.7236	0.0499
OH*NO2	1	1	0.0682932	0.0606	0.8176
OH*AL(OH)4	1	1	0.0147857	0.0131	0.9144
OH*CO3	1	1	0.3454201	0.3066	0.6093
OH*SO4	1	1	0.8134912	0.7221	0.4433
OH*Temp	1	1	0.0547672	0.0486	0.8363
NO2*NO3	1	1	0.5095515	0.4523	0.5381

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	15	101.84785	6.78986	6.0269
Error	4	4.50640	1.12660	Prob>F
C Total	19	106.35425		0.0476

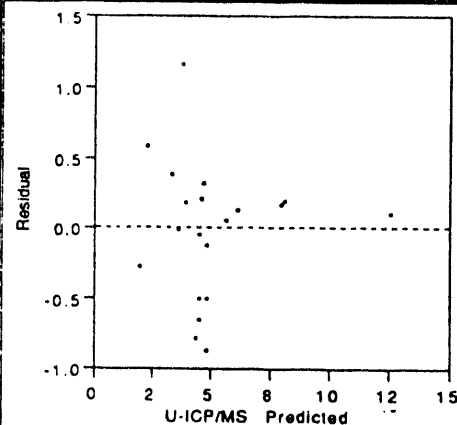


Exhibit 21. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Full Model for Prepared Levels of Factors

Response: U-Ck

Summary of Fit

Rsquare	0.939606
Root Mean Square Error	1.032505
Mean of Response	4.118518
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	4.1440305	2.07202	34.4654
Pure Error	2	0.1202373	0.06012	Prob>F
Total Error	4	4.2642678		0.0282

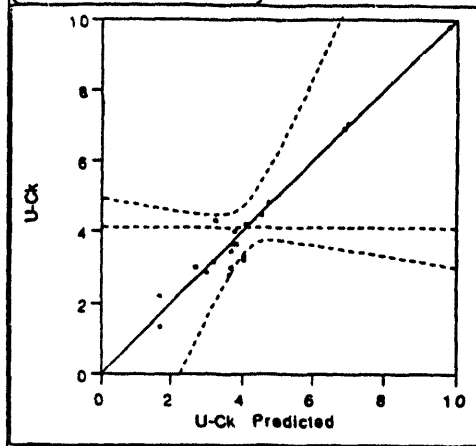
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.5289894	2.09186	0.73	0.5053
NO3	0.1965537	0.49901	0.39	0.7138
NO2	-0.886209	1.05071	-0.85	0.4455
OH	4.1699709	2.35596	1.77	0.1514
AL(OH)4	5.1031681	3.91982	1.30	0.2629
CO3	-0.714103	4.17336	-0.17	0.8724
SO4	-1.346679	3.07567	-0.44	0.6641
Temp(H-L)	-0.270722	0.62529	-0.43	0.6674
NO3*NO2*OH	-0.264369	0.48538	-0.54	0.6149
OH*NO3	-0.756044	0.34462	-2.19	0.0933
OH*NO2	-0.082196	0.82628	-0.10	0.9255
OH*AL(OH)4	-2.193051	4.52614	-0.48	0.6533
OH*CO3	-2.325375	4.69556	-0.50	0.6464
OH*SO4	2.0898659	3.45945	0.60	0.5784
OH*Temp(H-L)	0.0915666	0.65263	0.14	0.8952
NO2*NO3	0.2843569	0.42097	0.68	0.5364

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.1653969	0.1551	0.7138
NO2	1	1	0.7618101	0.7146	0.4455
OH	1	1	3.3397595	3.1328	0.1514
AL(OH)4	1	1	1.8068953	1.6949	0.2629
CO3	1	1	0.0312129	0.0293	0.8724
SO4	1	1	0.2044386	0.1918	0.6641
Temp	1	1	0.1998373	0.1875	0.6674
NO3*NO2*OH	1	1	0.3162571	0.2967	0.6149
OH*NO3	1	1	5.1308094	4.8128	0.0933
OH*NO2	1	1	0.0105494	0.0099	0.9255
OH*AL(OH)4	1	1	0.2502800	0.2348	0.6533
OH*CO3	1	1	0.2614541	0.2453	0.6464
OH*SO4	1	1	0.3890509	0.3649	0.5784
OH*Temp	1	1	0.0209855	0.0197	0.8952
NO2*NO3	1	1	0.4864248	0.4563	0.5364

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	15	66.342829	4.42286	4.1488
Error	4	4.264268	1.06607	Prob>F
C Total	19	70.607097		0.0894

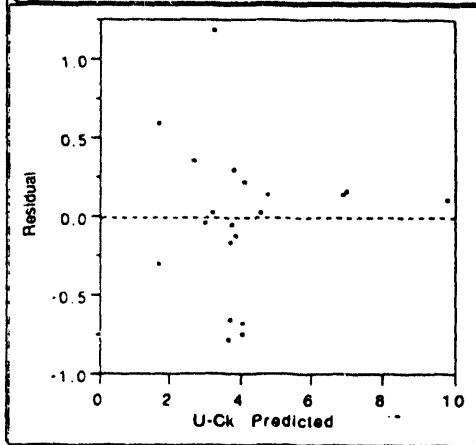


Exhibit 22.

Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Full Model for 6-month Levels of Factors

Response: Pu-C

Summary of Fit

Rsquare	0.928858
Root Mean Square Error	0.106799
Mean of Response	0.219355
Observations (nr Sum Wgts)	20

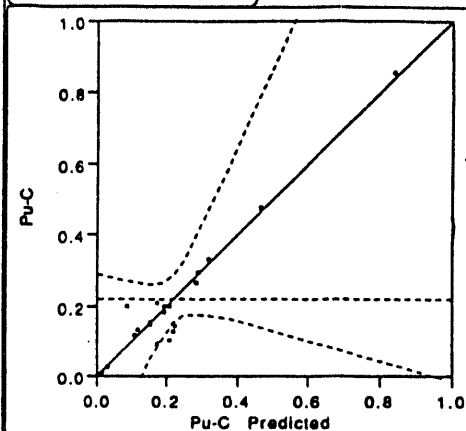
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.1036293	0.20328	0.51	0.6370
NO3	-0.006262	0.05918	-0.11	0.9208
NO2	-0.003653	0.1227	-0.03	0.9777
CH	-0.104164	0.20092	-0.52	0.6315
AL(OH)4	-0.672481	0.87303	-0.77	0.4841
CO3	-0.206844	0.53939	-0.38	0.7209
SO4	-0.157722	0.3851	-0.41	0.7031
Temp(H-L)	0.0240488	0.07619	0.32	0.7680
NO3*NO2*OH	-0.064879	0.04981	-1.30	0.2626
OH*NO3	0.0325376	0.03473	0.94	0.4019
OH*NO2	0.02748	0.08247	0.33	0.7557
OH*AL(OH)4	0.3409315	0.54262	0.63	0.5639
OH*CO3	0.4722752	0.53109	0.89	0.4241
OH*SO4	0.5738349	0.37669	1.52	0.2025
OH*Temp(H-L)	-0.032812	0.0703	-0.47	0.6650
NO2*NO3	0.0548509	0.04848	1.13	0.3211

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00012771	0.0112	0.9208
NO2	1	1	0.00001011	0.0009	0.9777
CH	1	1	0.00306554	0.2688	0.6315
AL(OH)4	1	1	0.00876753	0.5933	0.4841
CO3	1	1	0.00167734	0.1471	0.7209
SO4	1	1	0.00191322	0.1677	0.7031
Temp	1	1	0.00113651	0.0996	0.7680
NO3*NO2*OH	1	1	0.01935408	1.6968	0.2626
OH*NO3	1	1	0.01001018	0.8776	0.4019
OH*NO2	1	1	0.00126628	0.1110	0.7557
OH*AL(OH)4	1	1	0.00450266	0.3948	0.5639
OH*CO3	1	1	0.00901961	0.7908	0.4241
OH*SO4	1	1	0.02645107	2.3191	0.2025
OH*Temp	1	1	0.00248451	0.2178	0.6650
NO2*NO3	1	1	0.01460361	1.2803	0.3211

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	15	0.59568192	0.039712	3.4817	
Error	4	0.04562396	0.011406		0.1183
C Total	19	0.64130588			

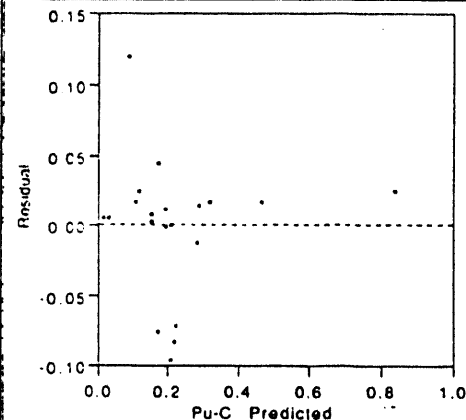


Exhibit 23. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Full Model for 6-month Levels of Factors

Response: Pu-ICP/MS

Summary of Fit

Rsquare	0.949982
Root Mean Square Error	0.060901
Mean of Response	0.139992
Observations (or Sum Wgts)	20

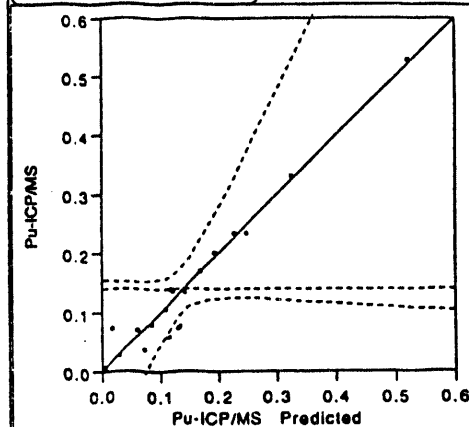
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.0358613	0.11592	0.31	0.7725
NO3	0.0213911	0.03375	0.63	0.5606
NO2	0.0262376	0.06997	0.37	0.7267
CH	-0.083335	0.11457	-0.73	0.5073
AL(OH)4	-0.545923	0.49784	-1.10	0.3344
CO3	0.1895808	0.30758	0.62	0.5710
SO4	-0.359888	0.2196	-1.64	0.1768
Temp(H-L)	0.0106209	0.04344	0.24	0.8189
NO3*NO2*OH	-0.038268	0.0284	-1.35	0.2491
OH*NO3	0.0084575	0.01981	0.43	0.6913
OH*NO2	0.0055611	0.04703	0.12	0.9116
OH*AL(OH)4	0.3902198	0.30943	1.26	0.2758
OH*CO3	0.2160551	0.30285	0.71	0.5150
OH*SO4	0.5063448	0.2148	2.36	0.0779
OH*Temp(H-L)	-0.017023	0.04009	-0.42	0.8930
NO2*NO3	0.0219854	0.02764	0.80	0.4709

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00149019	0.4018	0.5606
NO2	1	1	0.00052153	0.1406	0.7267
CH	1	1	0.00196211	0.5290	0.5073
AL(OH)4	1	1	0.00445999	1.2025	0.3344
CO3	1	1	0.00140903	0.3799	0.5710
SO4	1	1	0.00995024	2.6828	0.1768
Temp	1	1	0.00022167	0.0598	0.8189
NO3*NO2*OH	1	1	0.00673338	1.8155	0.2491
OH*NO3	1	1	0.00067833	0.1824	0.6913
OH*NO2	1	1	0.00005186	0.0140	0.9116
OH*AL(OH)4	1	1	0.00589866	1.5904	0.2758
OH*CO3	1	1	0.00188767	0.5090	0.5150
OH*SO4	1	1	0.02060938	5.5567	0.0779
OH*Temp	1	1	0.00066872	0.1803	0.6930
NO2*NO3	1	1	0.00234619	0.6326	0.4709

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	15	0.28177072	0.018785	5.0847	
Error	4	0.01483568	0.003709		0.0641
C Total	19	0.29660640			

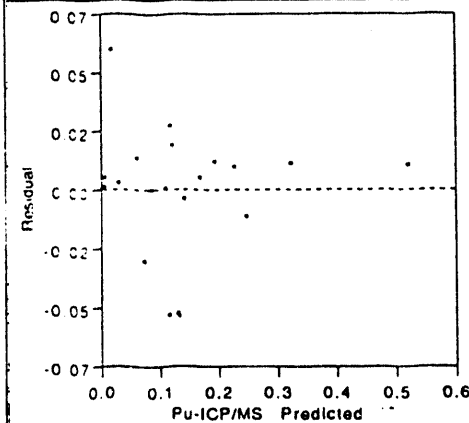


Exhibit 24. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Full Model for 6-month Levels of Factors

Response: U-ICP/MS

Summary of Fit

Rsquare	0.953086
Root Mean Square Error	1.116857
Mean of Response	5.026917
Observations (or Sum Wgts)	20

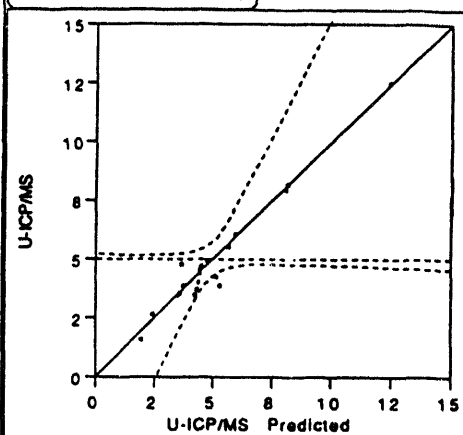
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.3238031	2.12582	0.62	0.5672
NO3	0.666673	0.61889	1.08	0.3420
NO2	-0.8303	1.28316	-0.65	0.5528
OH	4.5925852	2.10118	2.19	0.0941
AL(OH)4	7.2140891	9.12982	0.79	0.4736
CO3	-1.751503	5.64087	-0.31	0.7717
SO4	-1.309375	4.02723	-0.33	0.7614
Temp(H-L)	-0.720271	0.79672	-0.90	0.4171
NO3*NO2*OH	-0.261715	0.52085	-0.50	0.6417
OH*NO3	-1.04869	0.36321	-2.89	0.0447
OH*NO2	-0.188056	0.86248	-0.22	0.8381
OH*AL(OH)4	-0.921155	5.67453	-0.16	0.8789
OH*CO3	-2.240333	5.55391	-0.40	0.7073
OH*SO4	2.6922449	3.93923	0.68	0.5319
OH*Temp(H-L)	0.3587005	0.73521	0.49	0.6512
NO2*NO3	0.2119289	0.50693	0.42	0.6974

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	1.447436	1.1604	0.3420
NO2	1	1	0.522278	0.4187	0.5528
OH	1	1	5.959156	4.7774	0.0941
AL(OH)4	1	1	0.778814	0.6244	0.4736
CO3	1	1	0.120269	0.0964	0.7717
SO4	1	1	0.131859	0.1057	0.7614
Temp	1	1	1.019481	0.8173	0.4171
NO3*NO2*OH	1	1	0.314938	0.2525	0.6417
OH*NO3	1	1	10.398390	8.3363	0.0447
OH*NO2	1	1	0.059302	0.0475	0.8381
OH*AL(OH)4	1	1	0.032870	0.0264	0.8789
OH*CO3	1	1	0.202966	0.1627	0.7073
OH*SO4	1	1	0.582641	0.4671	0.5319
OH*Temp	1	1	0.296916	0.2380	0.6512
NO2*NO3	1	1	0.218008	0.1748	0.6974

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	15	101.36477	6.75765	5.4175	
Error	4	4.98948	1.24737		0.0572
C Total	19	106.35425			

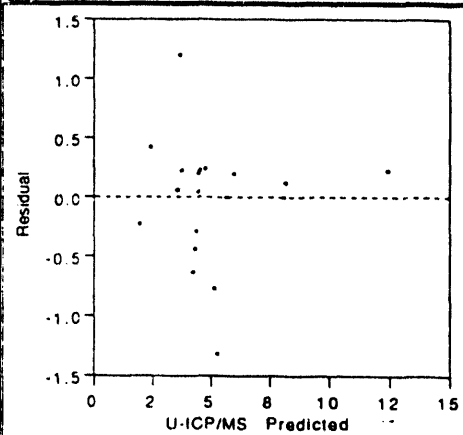


Exhibit 25. Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Full Model for 6-month Levels of Factors

Response: U-Ck

Summary of Fit

Rsquare	0.932744
Root Mean Square Error	1.089578
Mean of Response	4.118518
Observations (or Sum Wgts)	20

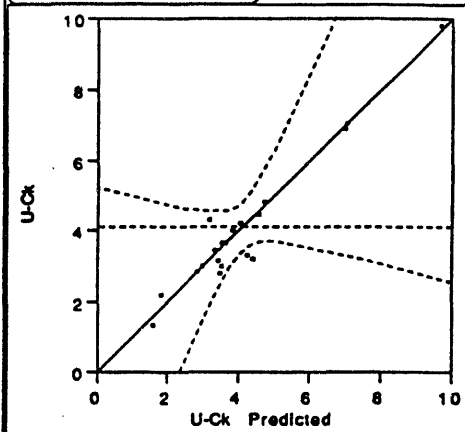
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.0489805	2.0739	0.51	0.6386
NO3	0.4452068	0.60377	0.74	0.5018
NO2	-0.88686	1.25182	-0.71	0.5178
CH	4.2300737	2.04986	2.06	0.1080
AL(OH)4	6.3490917	8.90683	0.71	0.5153
CO3	-1.227602	5.5029	-0.22	0.8344
SO4	-0.501571	3.92887	-0.13	0.9046
Temp(H-L)	-0.479491	0.77726	-0.62	0.5707
NO3*NO2*OH	-0.197787	0.50813	-0.39	0.7169
OH*NO3	-0.785505	0.35434	-2.22	0.0909
OH*NO2	-0.04278	0.84142	-0.05	0.9619
OH*AL(OH)4	-2.715921	5.53594	-0.49	0.6494
OH*CO3	-2.539805	5.41826	-0.47	0.6637
OH*SO4	1.2782826	3.84302	0.33	0.7561
OH*Temp(H-L)	0.1793993	0.71725	0.25	0.8148
NO2*NO3	0.2103896	0.49455	0.43	0.6924

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.6455015	0.5437	0.5018
NO2	1	1	0.5958575	0.5019	0.5178
CH	1	1	5.0555239	4.2584	0.1080
AL(OH)4	1	1	0.6032454	0.5081	0.5153
CO3	1	1	0.0590811	0.0498	0.8344
SO4	1	1	0.0193485	0.0163	0.9046
Temp	1	1	0.4517838	0.3806	0.5707
NO3*NO2*OH	1	1	0.1798708	0.1515	0.7169
OH*NO3	1	1	5.8340351	4.9142	0.0909
OH*NO2	1	1	0.0030689	0.0026	0.9619
OH*AL(OH)4	1	1	0.2857385	0.2407	0.6494
OH*CO3	1	1	0.2608544	0.2197	0.6637
OH*SO4	1	1	0.1313487	0.1106	0.7561
OH*Temp	1	1	0.0742696	0.0626	0.8148
NO2*NO3	1	1	0.2148526	0.1810	0.6924

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	15	65.858372	4.39056	3.6983	
Error	4	4.748725	1.18718		0.1075
C Total	19	70.607097			

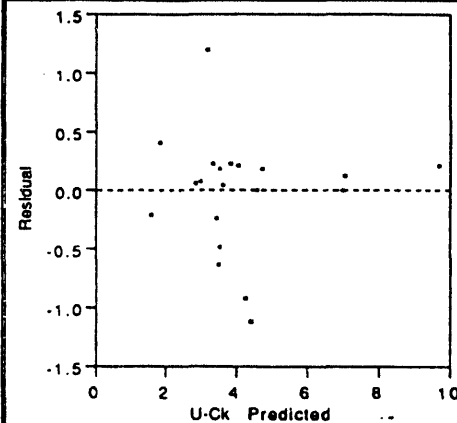


Exhibit 26. Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Reduced Model for Prepared Levels of Factors

Response: Pu-C

Summary of Fit

Rsquare	0.876128
Root Mean Square Error	0.093954
Mean of Response	0.219333
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	6	0.07416237	0.012360	7.0169
Pure Error	3	0.00528454	0.001762	Prob>F
Total Error	9	0.07944690		0.0692

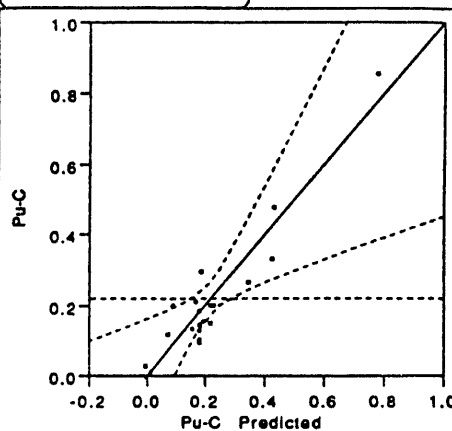
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	-0.030532	0.14335	-0.21	0.8361
NO3	0.0066067	0.04539	0.15	0.8875
NO2	-0.054301	0.09461	-0.57	0.5801
Cl	-0.024326	0.09842	-0.25	0.8103
CO3	0.5754091	0.17827	3.23	0.0104
SO4	-0.351579	0.2395	-1.47	0.1762
NO3*NO2*OH	-0.095184	0.04046	-2.35	0.0431
OH*NO3	0.0340099	0.03131	1.09	0.3057
OH*NO2	0.0886685	0.07297	1.22	0.2552
OH*SO4	0.7019696	0.18025	3.89	0.0037
NO2*NO3	0.0649147	0.03666	1.77	0.1104

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00018702	0.0212	0.8875
NO2	1	1	0.00290786	0.3294	0.5801
Cl	1	1	0.00053928	0.0611	0.8103
CO3	1	1	0.09197172	10.4189	0.0104
SO4	1	1	0.01902178	2.1548	0.1762
NO3*NO2*OH	1	1	0.04884843	5.5337	0.0431
OH*NO3	1	1	0.01041328	1.1797	0.3057
OH*NO2	1	1	0.01303300	1.4764	0.2552
OH*SO4	1	1	0.13388216	15.1666	0.0037
NO2*NO3	1	1	0.02767306	3.1349	0.1104

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	0.56191668	0.056192	6.3656
Error	9	0.07944690	0.008827	Prob>F
C Total	19	0.64136359		0.0051

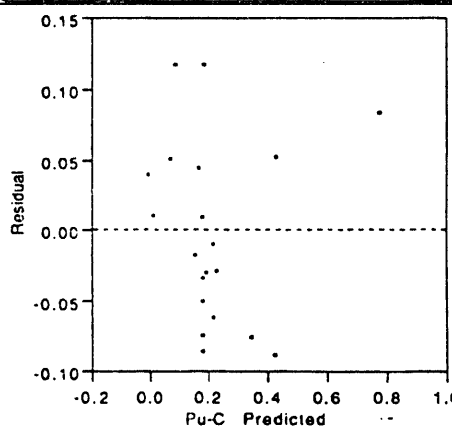


Exhibit 27.

Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Reduced Model for Prepared Levels of Factors

Response: Pu-ICP/MS

Summary of Fit

Rsquare	0.942637
Root Mean Square Error	0.049301
Mean of Response	0.139982
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	4	0.01305447	0.003264	2.4726
Pure Error	3	0.00395973	0.001320	Prob>F
Total Error	7	0.01701420		0.2415

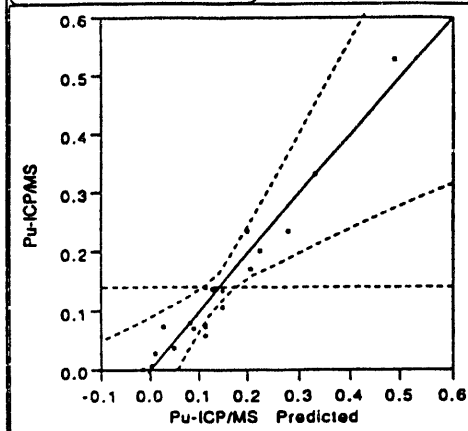
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	-0.007911	0.08223	-0.10	0.9261
NO3	0.0189192	0.02382	0.79	0.4531
NO2	-0.014686	0.04967	-0.30	0.7760
CH	-0.087766	0.06019	-1.46	0.1882
AL(OH)4	-0.186864	0.16956	-1.10	0.3069
CO3	0.5931328	0.09828	6.04	0.0005
SO4	-0.444807	0.12693	-3.50	0.0099
NO3*NO2*OH	-0.063304	0.02136	-2.96	0.0210
OH*NO3	0.0138045	0.01643	0.84	0.4286
OH*NO2	0.052043	0.03835	1.36	0.2169
OH*AL(OH)4	0.3107492	0.13285	2.34	0.0519
OH*SO4	0.6111202	0.09962	6.13	0.0005
NO2*NO3	0.0351515	0.01926	1.82	0.1108

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00153359	0.6310	0.4531
NO2	1	1	0.00021252	0.0874	0.7760
CH	1	1	0.00516720	2.1259	0.1882
AL(OH)4	1	1	0.00295197	1.2145	0.3069
CO3	1	1	0.08852758	36.4221	0.0005
SO4	1	1	0.02984679	12.2796	0.0099
NO3*NO2*OH	1	1	0.02134998	8.7838	0.0210
OH*NO3	1	1	0.00171527	0.7057	0.4286
OH*NO2	1	1	0.00447548	1.8413	0.2169
OH*AL(OH)4	1	1	0.01329843	5.4713	0.0519
OH*SO4	1	1	0.09147676	37.6355	0.0005
NO2*NO3	1	1	0.00809301	3.3296	0.1108

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	12	0.27959220	0.023299	9.5858
Error	7	0.01701420	0.002431	Prob>F
C Total	19	0.29660640		0.0031

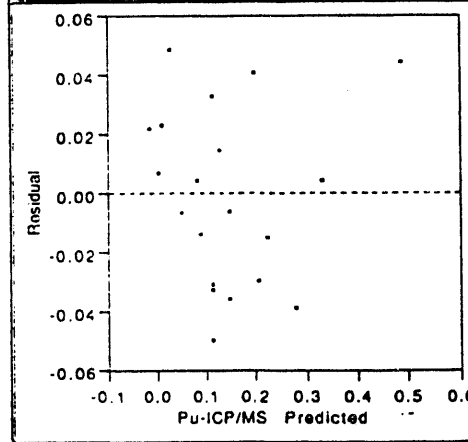


Exhibit 28. Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Reduced Model for Prepared Levels of Factors

Response: U-ICP/MS

Summary of Fit

Rsquare	0.817899
Root Mean Square Error	1.176167
Mean of Response	5.026917
Observations (or Sum Wgts)	20

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	11	19.222639	1.74751	36.2744
Pure Error	3	0.144524	0.04817	Prob>F
Total Error	14	19.367164		0.0065

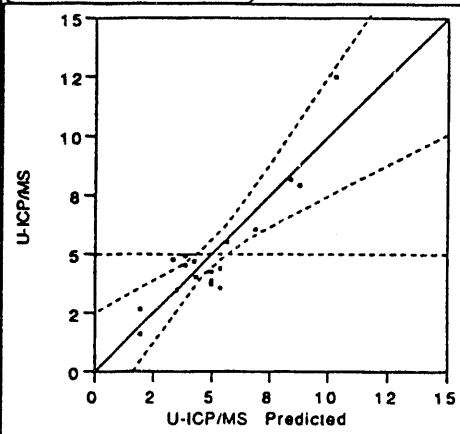
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	3.4037122	1.18661	2.87	0.0124
NO3	0.5093719	0.36702	1.39	0.1869
NO2	-0.998196	0.33677	-2.96	0.0103
OH	4.280973	0.77286	5.54	0.0001
CO3	-5.356014	2.1779	-2.46	0.0275
OH*NO3	-1.015264	0.29571	-3.43	0.0040

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	2.664617	1.9262	0.1869
NO2	1	1	12.153712	8.7856	0.0103
OH	1	1	42.444778	30.6822	0.0001
CO3	1	1	8.366543	6.0479	0.0275
OH*NO3	1	1	16.306727	11.7877	0.0040

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	86.98708	17.3974	12.5761
Error	14	19.36716	1.3834	Prob>F
C Total	19	106.35425		0.0001

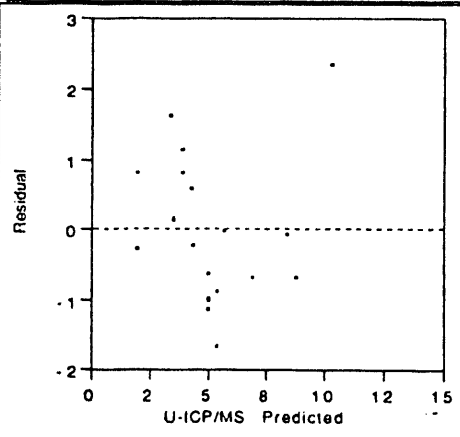


Exhibit 29.

Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Reduced Model for Prepared Levels of Factors

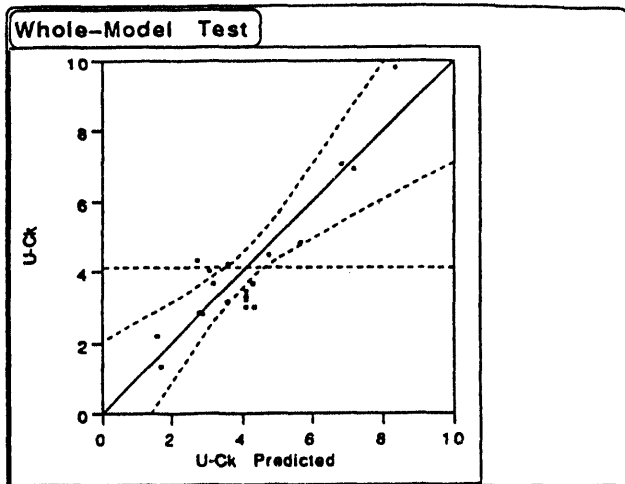
Response: U-Ck

Summary of Fit	
Rsquare	0.816193
Root Mean Square Error	0.962811
Mean of Response	4.118518
Observations (or Sum Wgts)	20

Lack of Fit				
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	11	12.857687	1.16888	29.1276
Pure Error	3	0.120389	0.04013	Prob>F
Total Error	14	12.978076		0.0090

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	2.6348227	0.97136	2.71	0.0168
NO3	0.4364556	0.30044	1.45	0.1683
NO2	-0.796524	0.27568	-2.89	0.0119
OH	3.5281802	0.63266	5.58	0.0001
CO3	-4.248078	1.78283	-2.38	0.0319
OH*NO3	-0.817527	0.24207	-3.38	0.0045

Effect Test					
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	1.956343	2.1104	0.1683
NO2	1	1	7.738826	8.3482	0.0119
OH	1	1	28.829746	31.0999	0.0001
CO3	1	1	5.263172	5.6776	0.0319
OH*NO3	1	1	10.573353	11.4059	0.0045



Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	5	57.629021	11.5258	12.4334	
Error	14	12.978076	0.9270	Prob>F	
C Total	19	70.607097		0.0001	

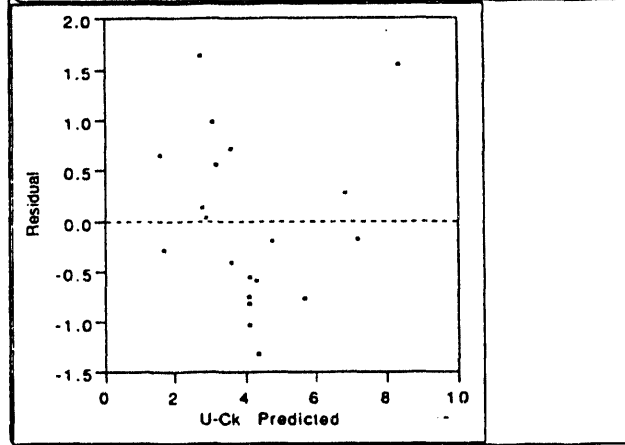


Exhibit 30.

Statistical Analysis of Average Plutonium Concentrations from Counting Analyses for Reduced Model for 6-month Levels of Factors

Response: Pu-C

Summary of Fit

Rsquare	0.898506
Root Mean Square Error	0.0902
Mean of Response	0.219355
Observations (or Sum Wgts)	20

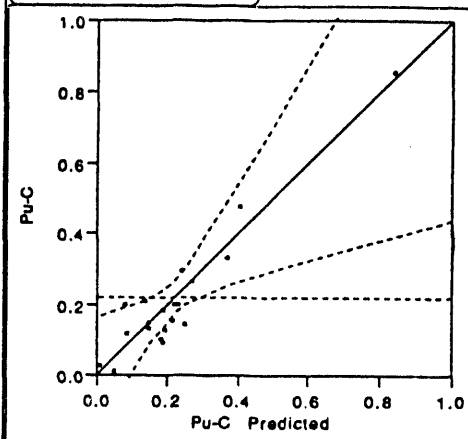
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.0188489	0.13793	0.14	0.8947
NO3	0.0083165	0.04517	0.18	0.8585
NO2	-0.005536	0.10027	-0.06	0.9573
CH	-0.084537	0.09398	-0.90	0.3946
CO3	-0.314051	0.35577	-0.88	0.4031
SO4	-0.191937	0.28656	-0.67	0.5218
NO3*NO2*OH	-0.083862	0.03651	-2.30	0.0507
OH*NO3	0.0273362	0.02769	0.99	0.3525
OH*NO2	0.0414638	0.0665	0.62	0.5503
OH*CO3	0.6060652	0.24538	2.47	0.0387
OH*SO4	0.6373994	0.18514	3.44	0.0088
NO2*NO3	0.0640476	0.04009	1.60	0.1488

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.00027575	0.0339	0.8585
NO2	1	1	0.00002480	0.0030	0.9573
CH	1	1	0.00658381	0.8092	0.3946
CO3	1	1	0.00633967	0.7792	0.4031
SO4	1	1	0.00365011	0.4486	0.5218
NO3*NO2*OH	1	1	0.04292846	5.2763	0.0507
OH*NO3	1	1	0.00792719	0.9743	0.3525
OH*NO2	1	1	0.00316328	0.3888	0.5503
OH*CO3	1	1	0.04963576	6.1007	0.0387
OH*SO4	1	1	0.09643845	11.8531	0.0088
NO2*NO3	1	1	0.02076690	2.5524	0.1488

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	11	0.57621696	0.052383	6.4384	
Error	8	0.06508892	0.008136		0.0069
C Total	19	0.64130588			

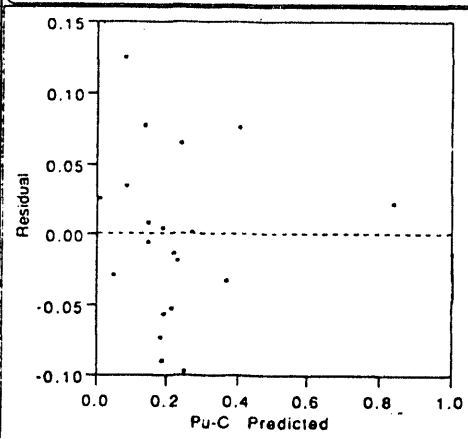


Exhibit 31. Statistical Analysis of Average Plutonium Concentrations from ICP/MS Analyses for Reduced Model for 6-month Levels of Factors

Response: Pu-ICP/MS

Summary of Fit

Rsquare	0.839796
Root Mean Square Error	0.062927
Mean of Response	0.139992
Observations (or Sum Wgts)	20

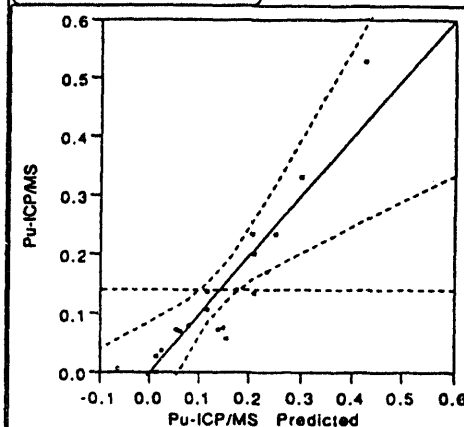
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.0600193	0.07636	0.79	0.4471
NO3	0.0253102	0.01012	2.50	0.0279
Cl	-0.06715	0.05052	-1.73	0.1101
AL(OH)4	-0.915016	0.40862	-2.24	0.0449
CO3	0.5263713	0.13083	4.02	0.0017
SO4	-0.436945	0.1855	-2.36	0.0363
OH*AL(OH)4	0.5708727	0.2316	2.46	0.0298
OH*SO4	0.5382312	0.12828	4.20	0.0012

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	0.02475092	6.2505	0.0279
Cl	1	1	0.01178589	2.9764	0.1101
AL(OH)4	1	1	0.01985570	5.0143	0.0449
CO3	1	1	0.06410055	16.1878	0.0017
SO4	1	1	0.02197137	5.5486	0.0363
OH*AL(OH)4	1	1	0.02405871	6.0757	0.0298
OH*SO4	1	1	0.06970782	17.6039	0.0012

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	7	0.24908878	0.035584	8.9863	
Error	12	0.04751762	0.003960		Prob>F
C Total	19	0.29660640			0.0006

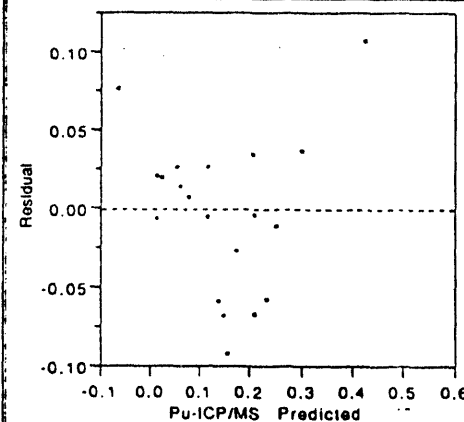


Exhibit 32.

Statistical Analysis of Average Uranium Concentrations from ICP/MS Analyses for Reduced Model for 6-month Levels of Factors

Response: U-ICP/MS

Summary of Fit

Rsquare	0.925882
Root Mean Square Error	0.810493
Mean of Response	5.026917
Observations (or Sum Wgts)	20

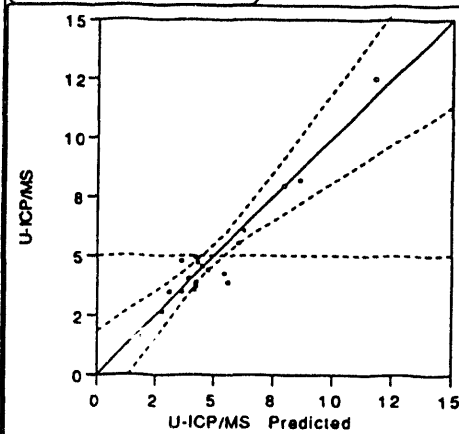
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	2.9457672	0.98164	3.00	0.0110
NO3	0.5616369	0.25859	2.17	0.0506
NO2	-1.059377	0.23706	-4.47	0.0008
CH	5.2955188	0.62235	8.51	0.0000
CO3	1.2679509	2.9936	0.42	0.6794
OH*NO3	-0.951932	0.18284	-5.21	0.0002
OH*CO3	-6.145458	2.03604	-3.02	0.0107
Temp (N)	-0.024562	0.00977	-2.51	0.0272

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
NO3	1	1	3.098745	4.7172	0.0506
NO2	1	1	13.117950	19.9695	0.0008
CH	1	1	47.560590	72.4016	0.0000
CO3	1	1	0.117848	0.1794	0.6794
OH*NO3	1	1	17.805771	27.1058	0.0002
OH*CO3	1	1	5.984568	9.1103	0.0107
Temp (N)	1	1	4.153598	6.3230	0.0272

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	7	98.47145	14.0674	21.4148	
Error	12	7.88279	0.6569		Prob>F
C Total	19	106.35425			0.0000

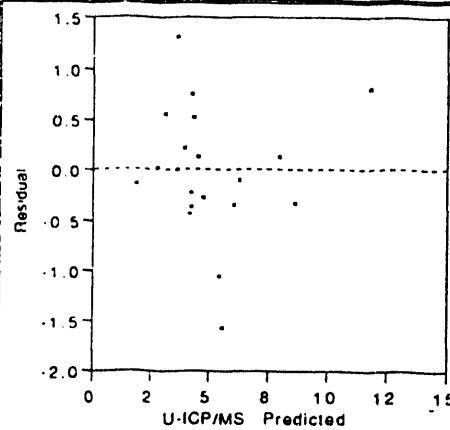


Exhibit 33.

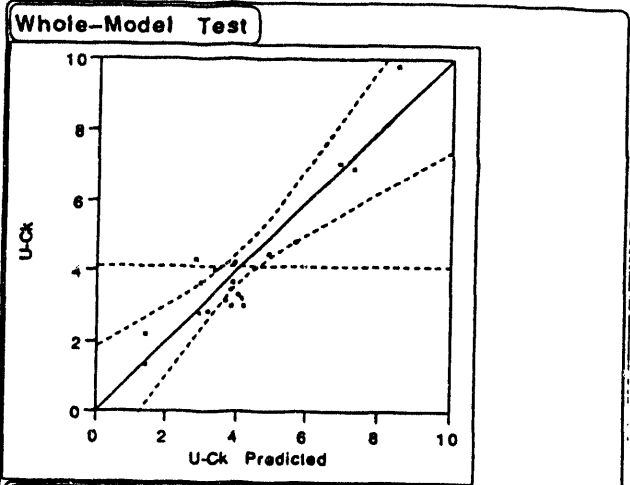
Statistical Analysis of Average Uranium Concentrations from Chemchek Analyses for Reduced Model for 6-month Levels of Factors

Response: U-Ck

Summary of Fit	
Rsquare	0.845933
Root Mean Square Error	0.881486
Mean of Response	4.118518
Observations (or Sum Wgts)	20

Parameter Estimates					
Term	Estimate	Std. Error	t Ratio	Prob> t	
Intercept	2.4414721	0.89223	2.74	0.0161	
NO3	0.4356965	0.28097	1.55	0.1433	
NO2	-0.78013	0.25674	-3.04	0.0088	
CH	3.3806179	0.53773	6.29	0.0000	
CO3	-5.112233	1.69431	-3.02	0.0092	
OH*NO3	-0.70198	0.19758	-3.55	0.0032	

Effect Test						
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F	
NO3	1	1	1.868455	2.4046	0.1433	
NO2	1	1	7.174132	9.2329	0.0088	
CH	1	1	30.711115	39.5243	0.0000	
CO3	1	1	7.074069	9.1041	0.0092	
OH*NO3	1	1	9.798384	12.6102	0.0032	



Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	5	59.728843	11.9458	15.3739	
Error	14	10.878254	0.7770		0.0000
C Total	19	70.607097			

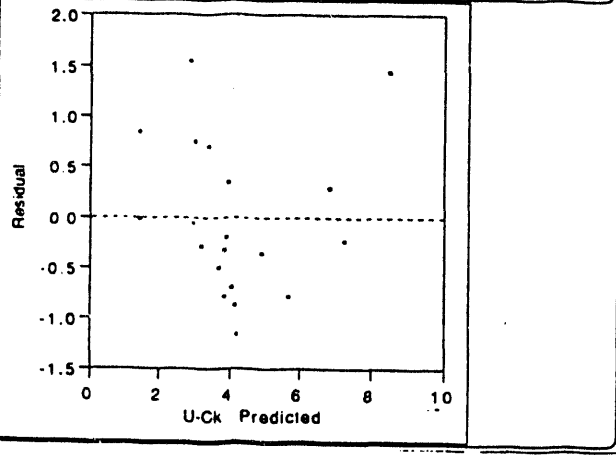


Exhibit 34. Statistical Analysis of Plutonium Concentration Measurements by Counting Method for Special Bottle # 23

Response: Conc (mg/L)

Summary of Fit

Rsquare	0.515497
Root Mean Square Error	0.280508
Mean of Response	1.950937
Observations (or Sum Wgts)	32

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.9528788	0.04993	39.11	0.0000
Time[14-185]	-0.089545	0.15477	-0.58	0.5690
Time[25-185]	-0.106212	0.15477	-0.69	0.5000
Time[31-185]	0.3837879	0.15477	2.48	0.0217
Time[40-185]	0.1337879	0.15477	0.86	0.3971
Time[56-185]	-0.149545	0.15477	-0.97	0.3449
Time[74-185]	-0.172879	0.15477	-1.12	0.2766
Time[81-185]	-0.176212	0.15477	-1.14	0.2677
Time[84-185]	-0.186212	0.15477	-1.20	0.2423
Time[122-185]	-0.192879	0.15477	-1.25	0.2264
Time[153-185]	0.0621212	0.18623	0.33	0.7420

Expected Mean Squares

The Mean Square per row by the Variance Component per column

EMS	Intercept	Time
Intercept	0	0
Time	0	2.90625

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est
Time	0.033419
Residual	0.078685

These estimates based on equating Mean Squares to Expected Value.

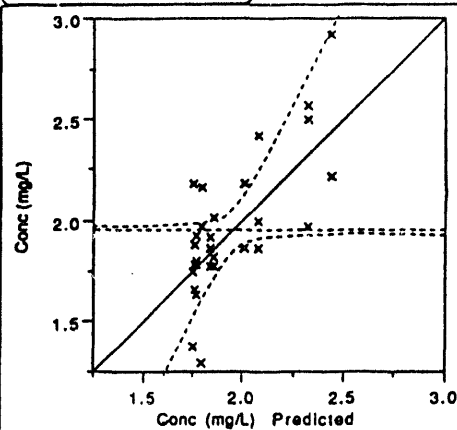
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Time	0.07868	21 Residual	

Tests wrt Random Effects

Source	SS	MS	Num	DF Num	F Ratio	Prob>F
Time	1.75809	0.17581	10	10	2.2343	0.0580

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	10	1.758085	0.175809	2.2343	
Error	21	1.6523833	0.078685		0.0580
C Total	31	3.4104719			

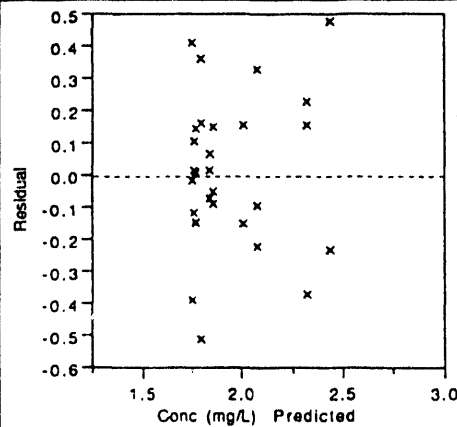


Exhibit 35. Statistical Analysis of Plutonium Concentration Measurements by ICP/MS Method for Special Bottle # 23

Response: Conc (mg/L)

Summary of Fit

Rsquare	0.375192
Root Mean Square Error	0.621052
Mean of Response	1.845217
Observations (or Sum Wgts)	23

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1.8814583	0.13067	14.40	0.0000
Time[31-153]	-0.311458	0.3369	-0.92	0.3699
Time[40-153]	-0.164792	0.3369	-0.49	0.6318
Time[56-153]	-0.374792	0.3369	-1.11	0.2834
Time[74-153]	-0.114792	0.3369	-0.34	0.7360
Time[81-153]	-0.178125	0.3369	-0.53	0.6047
Time[84-153]	-0.261458	0.3369	-0.78	0.4498
Time[122-153]	0.571875	0.3369	1.70	0.1103

Expected Mean Squares

The Mean Square per row by the Variance Component per column

BMS	Intercept	Time
Intercept	0	0
Time	0	2.86957

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est
Time	0.038545
Residual	0.385706

These estimates based on equating Mean Squares to Expected Value.

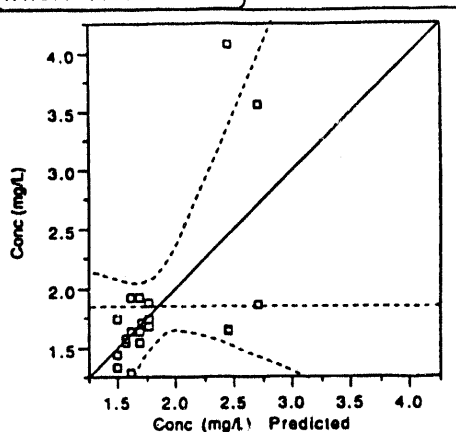
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Time	0.38571	15 Residual	

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob>F
Time	3.47419	0.49631	7	1.2868	0.3209

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	3.4741906	0.496313	1.2868
Error	15	5.7855833	0.385706	Prob>F
C Total	22	9.2597739		0.3209

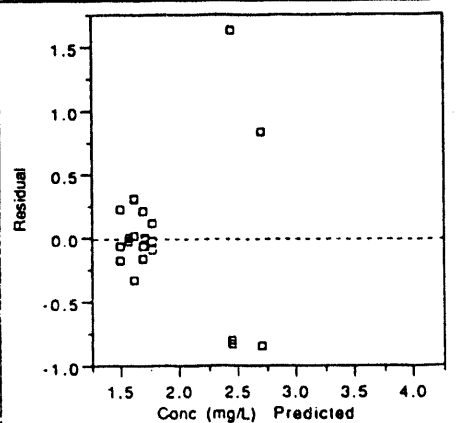


Exhibit 36.

Statistical Analysis of Uranium Concentration Measurements by ICP/MS Method for Special Bottle # 23

Response: Conc (mg/L)

Summary of Fit

Rsquare	0.834426
Root Mean Square Error	0.591442
Mean of Response	12.83077
Observations (or Sum Wgts)	26

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	12.777778	0.11694	109.27	0.0000
Time[31-185]	-1.877778	0.32306	-5.81	0.0000
Time[40-185]	-0.744444	0.32306	-2.30	0.0341
Time[56-185]	-0.477778	0.32306	-1.48	0.1574
Time[74-185]	1.222222	0.32306	3.78	0.0015
Time[81-185]	1.122222	0.32306	3.47	0.0029
Time[84-185]	0.788889	0.32306	2.44	0.0258
Time[122-185]	0.955556	0.32306	2.96	0.0088
Time[153-185]	-1.377778	0.38692	-3.56	0.0024

Expected Mean Squares

The Mean Square per row by the Variance Component per column

BMS	Intercept	Time
Intercept	0	0
Time	0	2.88462

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est
Time	1.177379
Residual	0.349804

These estimates based on equating Mean Squares to Expected Value.

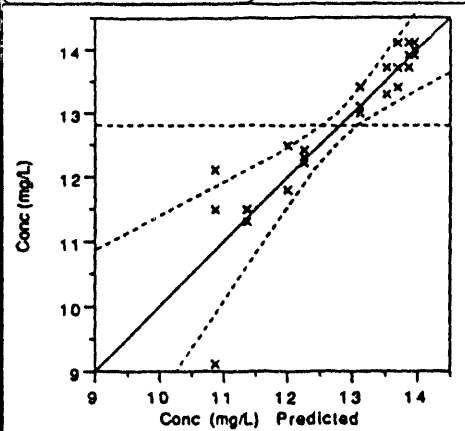
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Time	0.3498	17	Residual

Tests wrt Random Effects

Source	SS	MS	Num DF	Denom DF	F Ratio	Prob>F
Time	29.9687	3.74609	8	17	10.7091	0.0000

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	8	29.968718	3.74609	10.7091	
Error	17	5.946667	0.34980		0.0000
C Total	25	35.915385			

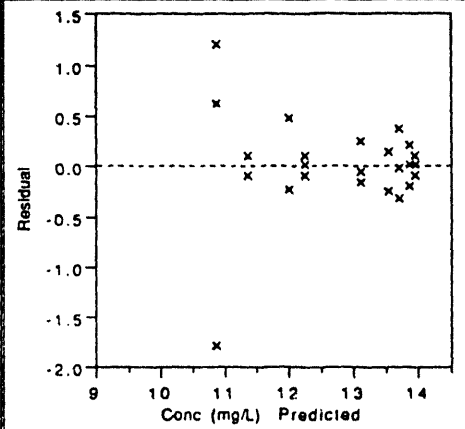


Exhibit 37. Statistical Analysis of Uranium Concentration Measurements by Chemchek Method for Special Bottle # 23

Response: Conc (mg/L)

Summary of Fit

Rsquare	0.812874
Root Mean Square Error	0.942514
Mean of Response	14.06957
Observations (or Sum Wgts)	23

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	14.235417	0.19831	71.78	0.0000
Time[31-153]	-1.26875	0.51128	-2.48	0.0254
Time[40-153]	0.93125	0.51128	1.82	0.0885
Time[56-153]	-2.502083	0.51128	-4.89	0.0002
Time[74-153]	0.33125	0.51128	0.65	0.5269
Time[91-153]	-0.26875	0.51128	-0.53	0.6068
Time[94-153]	-0.935417	0.51128	-1.83	0.0873
Time[122-153]	-0.102083	0.51128	-0.20	0.8444

Expected Mean Squares

The Mean Square per row by the Variance Component per column

EMS	Intercept	Time
Intercept	0	0
Time	0	2.86957

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est
Time	2.572085
Residual	0.888333

These estimates based on equating Mean Squares to Expected Value.

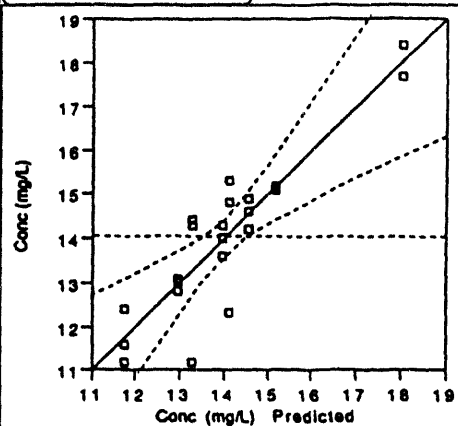
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Time	0.88833	15 Residual	

Tests wrt Random Effects

Source	SS	MS	Num DF	Denom DF	F Ratio	Prob>F
Time	57.8837	8.2691	7	15	9.3086	0.0002

Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	7	57.883696	8.26910	9.3086	
Error	15	13.325000	0.88833		0.0002
C Total	22	71.208696			

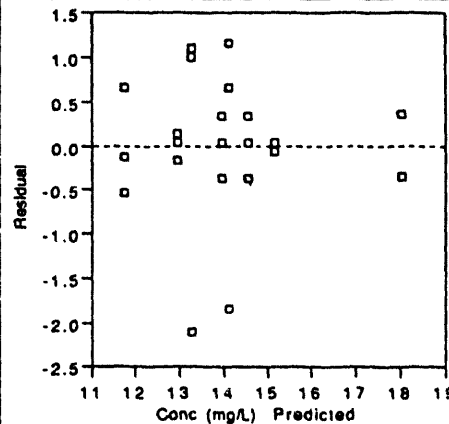
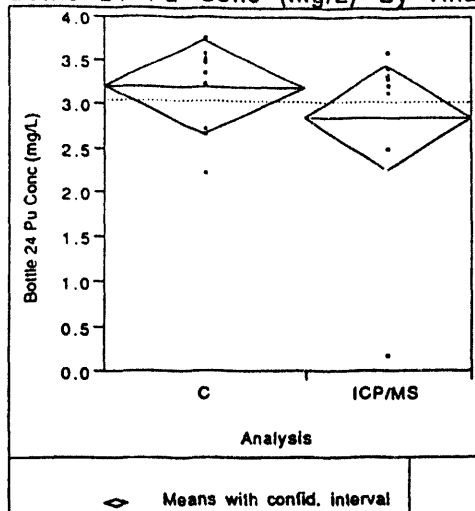


Exhibit 38. Statistical Comparison of Analytical Methods for Measuring Plutonium Concentration for Special Bottle # 24

Bottle 24 Pu Conc (mg/L) By Analysis



Means

Summary of Fit

Rsquare 0.04312
 Root Mean Square Error 0.830797
 Mean of Response 3.047278
 Observations (or Sum Wgts) 18

t-Test

t-Test	DF	Prob> t
0.84912585	16	0.4083

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.497662	0.497662	0.7210
Error	16	11.043586	0.690224	Prob>F
C Total	17	11.541248		0.4083

Mean Estimates

Level	number	Mean	Std. Error
C	10	3.19600	0.26272
ICP/MS	8	2.86137	0.29373

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
C	10	0.489176	0.3876000	0.3640000
ICP/MS	8	1.126939	0.7529375	0.5786250

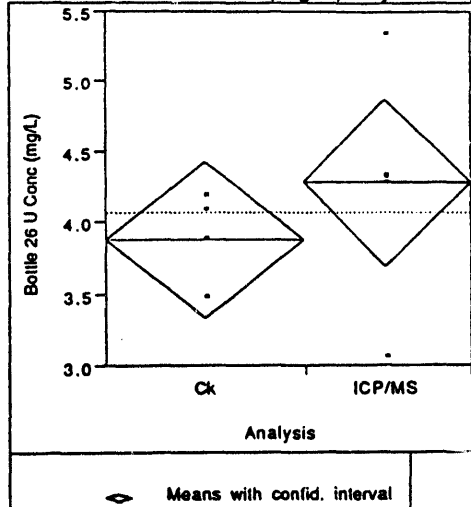
Test	F Ratio	DF Num	DF Den	Prob>F
O'Brien[.5]	1.1682	1	16	0.2958
Brown-Forsythe	0.3790	1	16	0.5468
Levene	1.8960	1	16	0.1875
Bartlett	4.9498	1	.	0.0261

Welch Anova testing Means Equal, allowing Std's Not Equal

F Ratio	DF Num	DF Den	Prob>F
0.6130	1	9.1084	0.4536

Exhibit 39. Statistical Comparison of Analytical Methods for Measuring Uranium Concentration for Special Bottle # 26

Bottle 26 U Conc (mg/L) By Analysis



Means

Summary of Fit

Rsquare	0.122925
Root Mean Square Error	0.589148
Mean of Response	4.065455
Observations (or Sum Wgts)	11

t-Test

t-1	Prob> t
1.1231129	9 0.2905

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	1	0.4378194	0.437819	1.2614	
Error	9	3.1238533	0.347095		0.2905
C Total	10	3.5616727			

Mean Estimates

Level	number	Mean	Std Error
Ck	6	3.88333	0.24052
ICP/MS	5	4.28400	0.26347

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Ck	6	0.3125167	0.2555556	0.2500000
ICP/MS	5	0.8117142	0.4816000	0.4740000

Test	F Ratio	DF Num	DF Den	Prob>F
O'Brien[.5]	1.7158	1	9	0.2227
Brown-Forsythe	0.7168	1	9	0.4191
Levene	0.7977	1	9	0.3950
Bartlett	3.3931	1	.	0.0655

Warning: Small sample sizes. Use Caution.

Welch Anova testing Means Equal, allowing Std's Not Equal				
F Ratio	DF Num	DF Den	Prob>F	
1.0843	1	4.9884	0.3456	

Exhibit 40. Statistical Analysis of Plutonium Concentrations by Analytical Method and Bottle for Bottles # 19 and 22

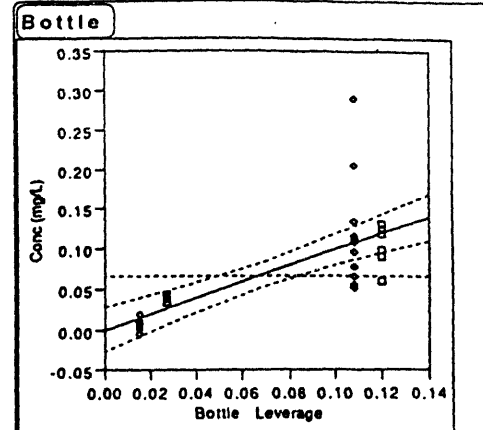
Response: Conc (mg/L)

Summary of Fit	
Rsquare	0.610532
Root Mean Square Error	0.043551
Mean of Response	0.067079
Observations (or Sum Wgts)	34

Lack of Fit				
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	0.00486943	0.004869	2.7089
Pure Error	30	0.05392674	0.001798	Prob>F
Total Error	31	0.05879616		0.1102

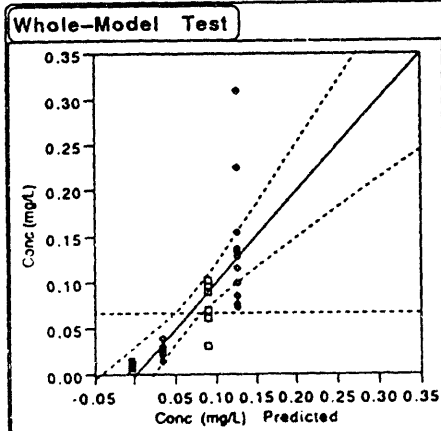
Parameter Estimates					
Term	Estimate	Std Error	t Ratio	Prob> t	
Intercept	0.0636909	0.00759	8.39	0.0000	
Bottle[19-22]	0.0463096	0.00752	6.16	0.0000	
Analytic[C-ICP/MS]	0.0191981	0.00764	2.51	0.0174	

Effect Test					
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
Bottle	1	1	0.07187414	37.8953	0.0000
Analytical	1	1	0.01196761	6.3099	0.0174

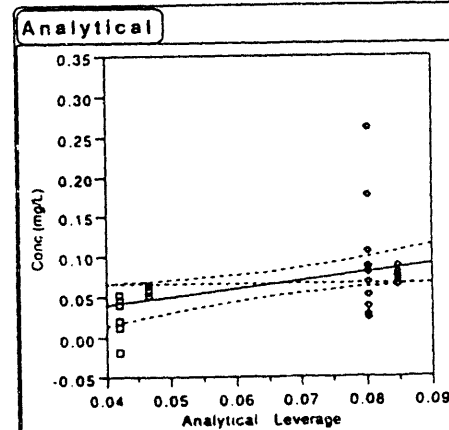
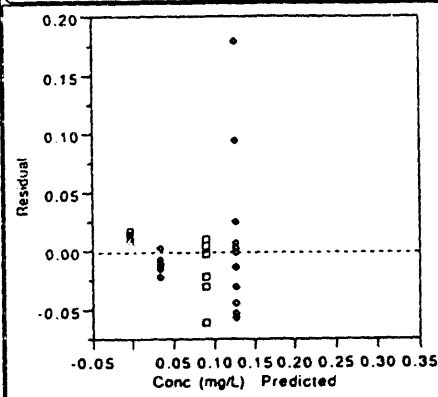


Effect Test				
Sum of Squares	F Ratio	DF	Prob>F	
0.07187414	37.8953	1	0.0000	

Least Squares Means				
Level	Least Sq Mean	Std Error	Mean	
19	0.110005541	0.0107990999	0.115647	
22	0.0173812873	0.0105721232	0.018511	



Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.09216931	0.046085	24.2979
Error	31	0.05879616	0.001897	Prob>F
C Total	33	0.15096547		0.0000



Effect Test				
Sum of Squares	F Ratio	DF	Prob>F	
0.01196761	6.3099	1	0.0174	

Least Squares Means				
Level	Least Sq Mean	Std Error	Mean	
C	0.0828890367	0.0097672128	0.087520	
ICP/MS	0.044498048	0.0116888827	0.037877	

Exhibit 41. Statistical Analysis of Uranium Concentrations by Analytical Method and Bottle for Bottles # 19 and 22

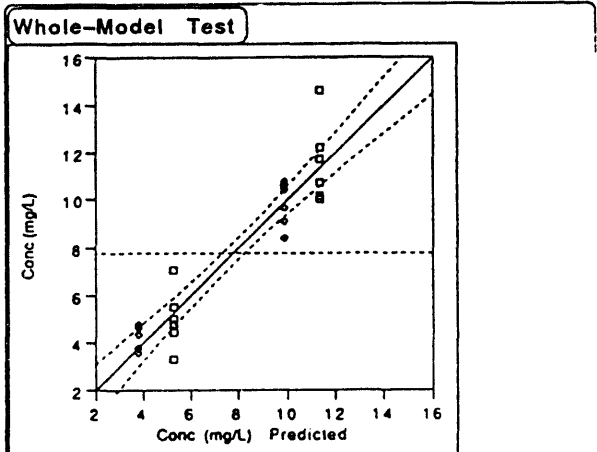
Response: Conc (mg/L)

Summary of Fit	
Rsquare	0.897708
Root Mean Square Error	1.11528
Mean of Response	7.781429
Observations (or Sum Wgts)	28

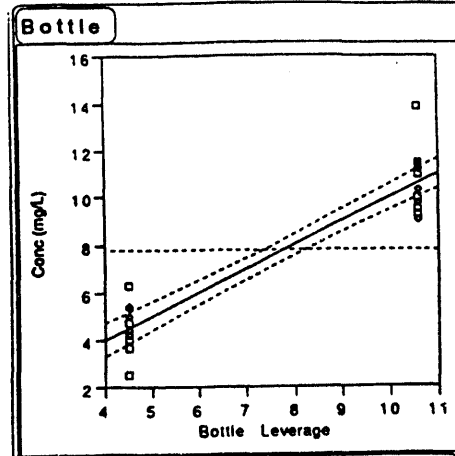
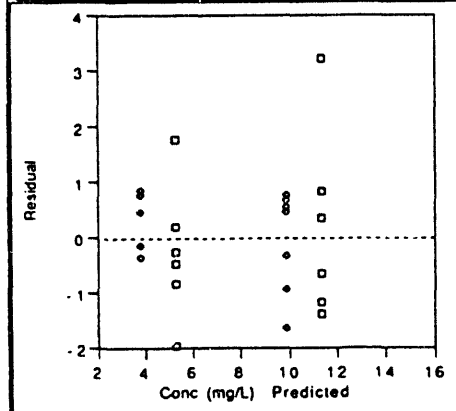
Lack of Fit				
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	1.389188	1.38919	1.1223
Pure Error	24	29.707026	1.23779	Prob>F
Total Error	25	31.096214		0.3000

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	7.6141237	0.21185	35.94	0.0000
Bottle[19-22]	-3.046134	0.21131	-14.42	0.0000
Analytic[CK-ICP/MS]	-0.703866	0.21131	-3.33	0.0027

Effect Test					
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
Bottle	1	1	258.47775	207.8048	0.0000
Analytical	1	1	13.80083	11.0953	0.0027

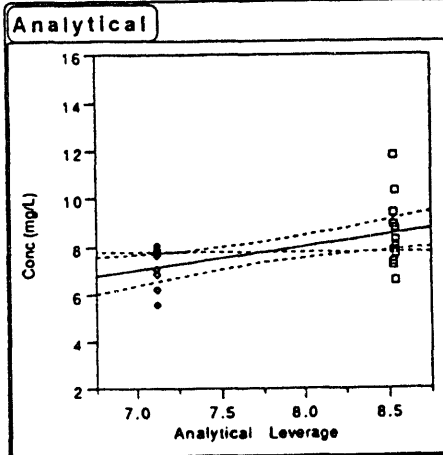


Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	272.89833	136.449	109.6992
Error	25	31.09621	1.244	Prob>F
C Total	27	303.99454		0.0000



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
258.47775	207.8048	1	0.0000

Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
19	4.56798969	0.3097496952	4.5138
22	10.66025773	0.2883083288	10.6133



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
13.800827	11.0953	1	0.0027

Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
Ck	6.910257732	0.2883083288	7.11333
ICP/MS	8.317989691	0.3097496952	8.55231

Exhibit 42. Statistical Analysis of Plutonium Concentrations by Analytical Method and Bottle for Bottles # 19 and 28

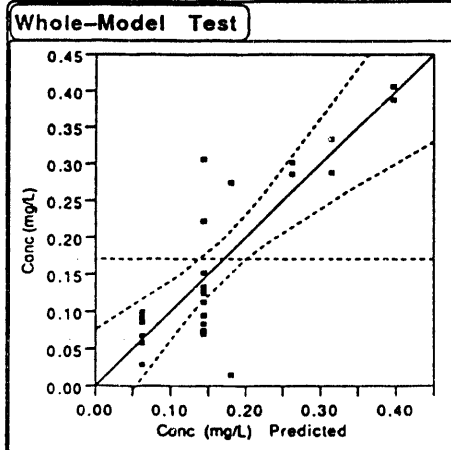
Response: Conc (mg/L)

Summary of Fit	
Rsquare	0.715378
Root Mean Square Error	0.067018
Mean of Response	0.170792
Observations (or Sum Wgts)	25

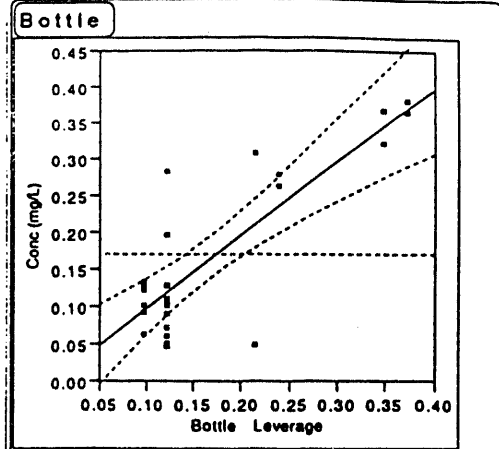
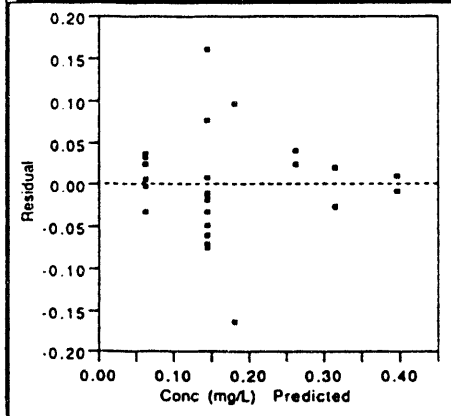
Lack of Fit				
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	0.00564627	0.002823	0.6049
Pure Error	19	0.08867258	0.004667	Prob>F
Total Error	21	0.09431884		0.5563

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.2265117	0.01675	13.52	0.0000
Bottle[19-28B]	-0.122927	0.01935	-6.35	0.0000
Bottle[28A-28B]	-0.005562	0.02559	-0.22	0.8301
Analytic[C-ICP/MS]	0.041011	0.01382	2.97	0.0073

Effect Test					
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
Bottle	2	2	0.21727039	24.1875	0.0000
Analytical	1	1	0.03957417	8.8112	0.0073

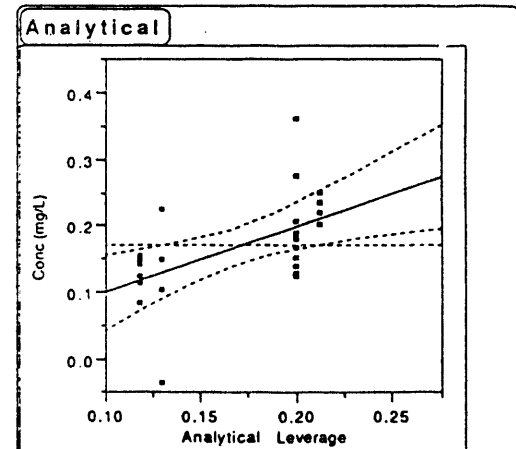


Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.23706421	0.079021	17.5940
Error	21	0.09431884	0.004491	Prob>F
C Total	24	0.33138306		0.0000



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
0.21727039	24.1875	2	0.0000

Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
19	0.1035850000	0.0167544277	0.115647
28A	0.2209500000	0.0335088554	0.220950
28B	0.3550000000	0.0335088554	0.355000



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
0.03957417	8.8112	1	0.0073

Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
C	0.2675226667	0.0208366531	0.193767
ICP/MS	0.1855006667	0.0225615234	0.136330

Exhibit 43. Statistical Analysis of Uranium Concentrations by Analytical Method and Bottle for Bottles # 19 and 28

Response: Conc (mg/L)

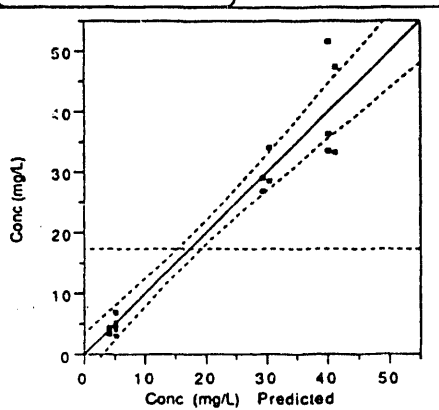
Summary of Fit	
Rsquare	0.944595
Root Mean Square Error	4.222788
Mean of Response	17.31727
Observations (or Sum Wgts)	22

Lack of Fit					
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Lack of Fit	2	6.51026	3.2551	0.1656	
Pure Error	16	314.46470	19.6540	Prob>F	
Total Error	18	320.97496		0.8488	

Parameter Estimates					
Term	Estimate	Std Error	t Ratio	Prob> t	
Intercept	25.034287	1.02518	24.42	0.0000	
Bottle[19-28B]	-20.47627	1.22533	-16.71	0.0000	
Bottle[28A-28B]	15.660557	1.49744	10.46	0.0000	
Analytic[Ch-ICP/MS]	-0.574221	0.90602	-0.63	0.5342	

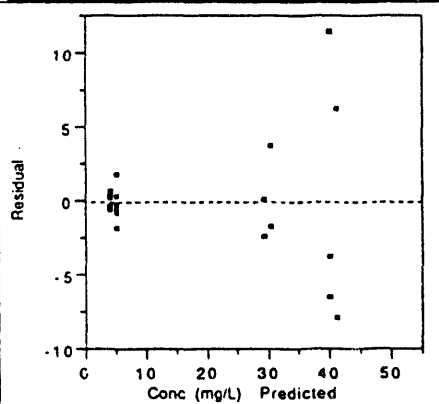
Effect Test					
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
Bottle	2	2	5472.0867	153.4350	0.0000
Analytical	1	1	7.1627	0.4017	0.5342

Whole-Model Test

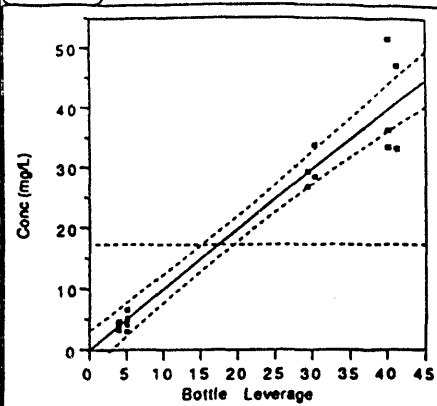


Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	3	5472.2727	1824.09	102.2934	
Error	18	320.9750	17.83	Prob>F	
C Total	21	5793.2476		0.0000	



Bottle



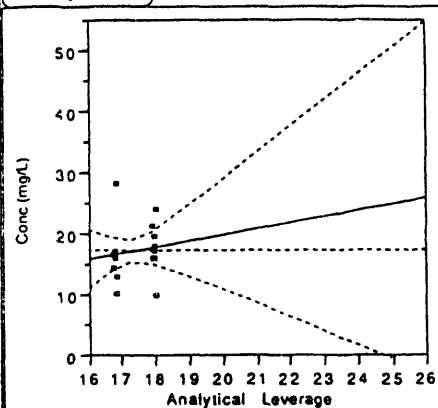
Effect Test

Sum of Squares	F Ratio	DF	Prob>F
5472.0867	153.4350	2	0.0000

Least Squares Means

Level	Least Sq Mean	Std Error	Mean
19	4.55801700	1.173262601	4.5138
28A	40.69484419	1.897161961	40.5800
28B	29.85000000	2.111394233	29.8500

Analytical



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
7.1627440	0.4017	1	0.5342

Least Squares Means

Level	Least Sq Mean	Std Error	Mean
Ch	24.46006610	1.311613744	17.2333
ICP/MS	25.60850803	1.422470779	17.4180

8.3 Simultaneous Tolerance Limits for Fitted Regression Models

From reference [3], for each of the fitted models, simultaneous, upper $100(1-\gamma)\%$ tolerance limits for the specie (plutonium or uranium) solubility over all salt solutions that are within the range of those studied here is given by

$$\mathbf{x}'\hat{\beta} + s \left\{ (pF_{\alpha/2;p,n-p})^{1/2} [\mathbf{x}'(\mathbf{X}'\mathbf{X})^{-1}\mathbf{x}]^{1/2} + z_{\gamma} \left(\frac{n-p}{\chi_{\alpha/2;n-p}^2} \right)^{1/2} \right\}$$

with probability at least $1-\alpha$,

- where
- n = 20, the number of salt solutions considered in the study,
 - \mathbf{x} = for a salt solution under consideration, the $(p \times 1)$ vector of values of the constituents' concentrations, including interactions, that appear in the fitted model with x_1 restricted to unity,
 - $\hat{\beta}$ = the estimated regression coefficients for the fitted model,
 - s = the root mean square error term from the fitted model,
 - p = the number of terms in the fitted model (main factors and/or interactions including the intercept term),
 - $F_{\alpha/2;p,n-p}$ = the upper $\alpha/2$ percentile point for the F-distribution with p and $(n-p)$ degrees of freedom,
 - \mathbf{X} = the $(n \times p)$ original design matrix for the constituents' concentrations and/or interactions in the fitted model with the first column equal to unity,
 - z_{γ} = the upper γ percentile point for the standard normal distribution, and
 - $\chi_{\alpha/2;n-p}^2$ = the lower $\alpha/2$ percentile point for the chi-square distribution with $n-p$ degrees of freedom.

**DATE
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5/4/94

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