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# FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
# FERNALD, OHIO

# COMPARABILITY OF TOTAL URANIUM DATA AS MEASURED BY IN-SITU GAMMA SPECTROMETRY AND FOUR LABORATORY METHODS

## ADDENDUM TO JULY, 1997 COMPARABILITY OF IN-SITU GAMMA SPECTROMETRY AND LABORATORY DATA REPORT

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1

# TABLE OF CONTENTS

List of Tables . . . . . ii
List of Figures . . . . . iii
List of Acronyms and Abbreviations . . . . . iv

Introduction . . . . . 1

Analytical Methods . . . . . 1

WAC Sample Locations . . . . . 2

Analytical Data . . . . . 2

Comparison of HPGe and Laboratory Data . . . . . 3
- Closeness of Laboratory and HPGe Data . . . . . 4
- Correlation of Laboratory and HPGe Data . . . . . 6
- Linear Regression Analysis . . . . . 6

Heterogeneity of Total Uranium Concentrations . . . . . 7

Conclusions . . . . . 8

## LIST OF TABLES

Table 1A Comparison of Alpha Spectrometry and HPGe Total Uranium Data
Table 1B Comparison of Gamma Spectrometry and HPGe Total Uranium Data
Table 1C Comparison of ICP/MS and HPGe Total Uranium Data
Table 1D Comparison of BromoPADAP and HPGe Total Uranium Data
Table 1E Comparison of Four Method Average with HPGe Total Uranium Data
Table 2A Closeness of Alpha Spectrometry and HPGe Total Uranium Data
Table 2B Closeness of Gamma Spectrometry and HPGe Total Uranium Data
Table 2C Closeness of ICP/MS and HPGe Total Uranium Data
Table 2D Closeness of BromoPADAP and HPGe Total Uranium Data
Table 2E Closeness of Four Method Average and HPGe Total Uranium Data
Table 3 Linear Regression Parameters

E 2149

## LIST OF FIGURES


| | |
|---|---|
| Figure 1 | Sewage Treatment Plant, Areas 18 and 19 |
| Figure 2A | Correlation Between 31 cm HPGe and Alpha Spectrometry Data for Total Uranium (w/o PBC-03-01) |
| Figure 2B | Correlation Between 100 cm HPGe and Alpha Spectrometry Data for Total Uranium (w/o PBC-03-01) |
| Figure 3A | Correlation Between 31 cm HPGe and Gamma Spectrometry Data for Total Uranium (w/o PBC-03-01) |
| Figure 3B | Correlation Between 100 cm HPGe and Gamma Spectrometry Data for Total Uranium (w/o PBC-03-01) |
| Figure 4A | Correlation Between 31 cm HPGe and ICP/MS Data for Total Uranium (w/o PBC-03-01) |
| Figure 4B | Correlation Between 100 cm HPGe and ICP/MS Data for Total Uranium (w/o PBC-03-01) |
| Figure 5A | Correlation Between 31 cm HPGe and BromoPADAP Data for Total Uranium (w/o PBC-03-01) |
| Figure 5B | Correlation Between 100 cm HPGe and BromoPADAP Data for Total Uranium (w/o PBC-03-01) |
| Figure 6A | Correlation Between 31 cm HPGe and Four Method Average Data for Total Uranium (w/o PBC-03-01) |
| Figure 6B | Correlation Between 100 cm HPGe and Four Method Average Data for Total Uranium (w/o PBC-03-01) |




4

## LIST OF ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| ASL | analytical support level |
| BromoPADAP | 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol |
| cm | centimeter |
| FEMP | Fernald Environmental Management Project |
| ft | feet |
| HPGe | high purity germanium detector |
| ICP/MS | inductively coupled plasma/mass spectrometry |
| pCi/g | picocuries per gram |
| ppm | parts per million |
| TPU | total propagated uncertainty |
| WAC | waste acceptance criteria |

2149

## INTRODUCTION

In July 1997, a report entitled "Comparability of in-Situ Gamma Spectrometry and Laboratory Data" was issued that assessed the comparability of high-purity germanium detector (HPGe) measurements with laboratory data generated from the analysis of physical samples. Results demonstrated that total uranium data generated by the HPGe were very comparable to total uranium data generated by laboratory alpha spectrometry analysis of physical samples. Results further showed that HPGe total uranium data met all proposed quality control acceptance criteria for use at analytical support levels (ASL) B and D. However, the report noted that additional data were needed to demonstrate comparability of HPGe and alpha spectrometry data at concentrations of total uranium near or exceeding 1000 ppm in order for the HPGe to be reliably used for waste acceptance criteria (WAC) activities.

This report extends the range of comparability to allow the HPGe to be used with confidence for total uranium WAC attainment measurements. Further, this report strengthens the case for the routine use of HPGe by demonstrating that HPGe data are comparable to laboratory gamma spectrometry data, laboratory bromoPADAP data, and laboratory inductively coupled plasma/mass spectrometry (ICP/MS) data as well as with laboratory alpha spectrometry data.

## ANALYTICAL METHODS

In the July 1997 HPGe comparability report, HPGe data were compared to laboratory alpha spectrometry data for total uranium. Alpha spectrometry was the method originally chosen at the Fernald Environmental Management Project (FEMP) for comparison with the HPGe because FEMP personnel had considerable expertise using that method and because alpha spectrometry is an established laboratory method routinely used by radioanalytical laboratories to generate high quality uranium isotopic data. However, FEMP personnel also have considerable experience in analyzing uranium by gamma spectrometry, by ICP/MS, and with a colorimetric method, bromoPADAP. Thus, to strengthen the case for the routine use of HPGe, the same samples originally analyzed by alpha spectrometry have been subsequently reanalyzed by these other three additional methods to demonstrate comparability of HPGe with multiple analytical methods. Further, new physical samples collected to extend the range of comparability to WAC levels of total uranium (1030 ppm) have also been analyzed by all four methods.

6

Appendix B contains brief descriptions of bromoPADAP and ICP/MS. Detailed descriptions of HPGe in-situ gamma spectrometry, laboratory alpha spectrometry and laboratory gamma spectrometry are contained in Appendix E of the July 1997 HPGe comparability report and are not duplicated here.

## WAC SAMPLE LOCATIONS

Based upon historical data, the dried sludge beds in the Sewage Treatment Plant area were selected as the FEMP site most likely to contain a sizeable area of "soil" containing uranium concentrations near, or in excess of, 1,000 ppm. A schematic map of the sludge drying beds in the Sewage Treatment Plant is shown in Figure 1.

Eight exploratory HPGe readings were first taken in the study area to delineate a general pattern of uranium contamination. The exploratory HPGe locations are depicted by solid triangles in Figure 1. HPGe measured uranium-238 concentrations are shown in pCi/g in parentheses below the triangles. (Uranium-238 concentrations may be converted to approximate total uranium concentrations in ppm by multiplying pCi/g by 3.0). The data show that total uranium concentrations increase to the south in the sludge drying beds. Accordingly, physical samples were collected (solid circles, Figure 1) in the southern half of the dried sludge beds.

Because the sludge beds contained high concentrations of uranium, 15 samples (and one duplicate) were collected for each of the two locations. The rationale for taking 15 samples is provided in Section 2.2.2.3 of the July 1997 HPGe comparability report. Figure 2-5 of that same report presents the sample numbering scheme, which was also used for this study. The location of the HPGe measurements were at physical sample location 1, the very center point in the concentric circular rings. As with the original study, HPGe measurements were made at both 100 cm (1 m) and 31 cm (1 ft) detector heights with a data acquisition time of 900 seconds.

## ANALYTICAL DATA

Appendix A contains laboratory alpha spectrometry, laboratory gamma spectrometry, laboratory bromoPADAP, laboratory ICP/MS, HPGe, and soil moisture data. The moisture data were used to calculate laboratory results on a wet weight basis. HPGe data are also shown on a wet weight, or "as is," basis to provide a common ground for comparing the different data sets.

7

Total uranium data from HPGe measurements, alpha spectrometry, and gamma spectrometry have been converted to ppm from pCi/g using the equations presented in Section 2.2.2 of the July 1997 HPGe comparability report. The bromoPADAP method measures ppm directly, while ICP/MS data are the sum of masses of individual isotopes. Total propagated uncertainties (TPUs) are displayed for each analytical method. The laboratory TPUs are presented as 1.96 σ values while the HPGe uncertainties reflect 1.96 σ counting errors. Laboratory TPUs are greater than HPGe 1.96 σ counting errors because the former represents total system uncertainty, while the latter represents only one component of system uncertainty.

Lastly, Appendix A contains a column representing the average for each sample of the concentrations determined by the four different analytical methods. For example, for PBC-1-1, the average of 8.0 ppm in the last column is the average of 7.4 ppm (alpha spectrometry), 7.2 ppm (gamma spectrometry), 11.3 ppm (bromoPADAP), and 5.9 ppm (ICP/MS). The standard deviation of 2.3 ppm is the 1.0 σ standard deviation associated with the four measurement data values given above.

The data in Appendix A are slightly different than the total uranium data in Appendix A of the comparability study issued in July 1997 for two reasons. First, more attention has been given to the significant digits in the results reported by the laboratory when a computation like the conversion from pCi/g to ppm pr the calculation of a weighted average is performed, slight differences may be apparent if rounded results are used instead of unrounded values. Secondly, a formatting error (when data are pulled from the sitewide database and displayed in a customized report like Appendix A, a program is written which pulls the data and formats the report) in the final version of Appendix A in the July 1997 study resulted in some of the total uranium data varying slightly from correct values.

## COMPARISON OF HPGe AND LABORATORY DATA

Tables 1A through 1E summarize the data in Appendix A. Using the weighting factors shown in Table 2-4 of the July 1997 HPGe comparability report and the computational method described in Section 3.2.2. of the July 1997 HPGe comparability report, weighted means and standard deviations for each sample collection area are shown in Tables 1A-1E. For each sample location in each area from which sample duplicates were collected, the average of the data for the duplicates was used in the weighted mean/standard deviation calculations. By comparing the weighted average of laboratory data for each

8

area with the HPGe data for that same area, an assessment of the degree of closeness of the data sets can be made. The section below contains such assessments.

Closeness of Laboratory and HPGe Data

Tables 2A through 2E summarize the closeness between laboratory data weighted means (Tables 1A-1E) and HPGe data. The closeness between weighted means and HPGe data is expressed as the percent relative deviation:

$$\%\,RelativeDeviation = [\frac{(\bar{x}_1 - \bar{x}_2)}{\bar{x}}]x100$$

where:

$\bar{x}_1$ is the mean of the laboratory data weighted to simulate HPGe measurements at a given detector height

$\bar{x}_2$ is the mean of duplicates for HPGe measurements at a given detector height

$\bar{x}$ is the average of the two means

The percent relative deviations shown in Tables 2A-2E are shown as negative and positive values in order to provide a sense of possible bias. However, the averages of the relative deviations shown at the bottom of Tables 2A-2E are calculated based upon the absolute value of the relative deviations.

Adopting criteria proposed in the July 1997 HPGe comparability report (Section 3.2.2) for interpreting percent relative deviations, when the relative deviation between HPGe and laboratory data is less than 20%, the data are defined as being very similar; when the relative deviation is greater than 20% but less than 35%, the data are defined as having acceptable similarity. When the relative deviation is greater than 35%, the data are defined as dissimilar.

Excluding sample PBC-03-01 for reasons delineated in the July 1997 HPGe comparability report, the averages of the relative deviations for all of the HPGe/laboratory comparisons are less than 20%. This indicates that on an average basis, HPGe and laboratory data for total uranium are very similar. In this regard, the average of the relative deviations for three of the laboratory methods/HPGe comparisons are remarkably similar. Thus, the average percent relative deviations for alpha spectrometry/HPGe are

10.98 and 11.12 (for 31 cm and 100 cm detector heights); for gamma spectrometry/HPGe they are 8.55 and 11.47; and for bromoPADAP, they are 9.36 and 11.75. Further, the average percent relative deviations for the "four method average"/HPGe comparison is 9.27 and 11.86 respectively, for 31 cm and 100 cm HPGe detector heights. The degree of closeness is not as good for ICP/MS and HPGe data as for the other three laboratory methods. The average percent relative deviation for the ICP/MS and HPGe comparisons are 15.56 and 16.67 for 31 cm and 100 cm detector heights.

The discussion above dealt with the averages of the percent relative deviations. Examination of Tables 2A-2E shows that a large majority of the individual percent relative deviations for given sampling areas are below 20%. Some areas have percent relative deviations between 20% and 35% (acceptable similarity), but very few instances occur where percent relative deviations are greater than 35%.

By using four different analytical methods to analyze the physical samples, laboratory analytical effects on the closeness of data can be differentiated from analyte heterogeneity effects. For example, BromoPADAP data for PBC-01 have percent relative deviations of 26.09 and 37.56, respectively, for 31 cm and 100 cm HPGe detector heights. Alpha spectrometry data, on the other hand, have percent relative deviations of -18.18 and -7.79 for PBC-01. Conversely, every analytical method has a percent relative deviation in excess of 20% for PBC-07 at a 1.0 m detector height. This indicates that either the HPGe detector is biased low (the percent relative deviations are all positive), or that the HPGe is detecting gamma rays from low contamination areas not reflected in the weighted average of the physical samples. Use of the average of the four laboratory methods (Table 2E) should average out, to a large extent, data variability due to analytical methods. The percent relative deviations in Table 2E should then mostly reflect either HPGe detector bias, analyte heterogeneity in the soil, or as explained in a later section possible limitations in the physical sample grid pattern.

This report contains data from two areas, PBC-18 and PBC-19, not included in the original July 1997 HPGe comparability report. As noted earlier, samples in these locations contain high concentrations of uranium. Examination of the data in Appendix A reveals that total uranium concentrations are very heterogeneous in Areas PBC-18 and PBC-19. However, this heterogeneity does not affect the overall degree of closeness of HPGe and laboratory data. For example, Table 2A shows that the average percent of relative deviations for alpha spectrometry and HPGe data are 10.98 and 11.12, respectively, for 31 cm and 100 cm HPGe detector heights. The July 1997 HPGe comparability report, without data

from Areas PBC-18 and PBC-19, had corresponding average percent relative deviations of 11.2 and 10.3 (31 cm and 100 cm) for alpha spectrometry and HPGe data.

Correlation of Laboratory and HPGe Data

Figures 2 through 6 show x,y scatter plots of the data in Tables 1A through 1E. These figures show both of the individual HPGe measurements (two measurements were taken at the same detector height except for Area PBC-06 in which six HPGe measurements were taken at each detector height. The minimum and maximum values of those six measurements in PBC-06 were used in the figures) for a given area plotted against the weighted averages of the laboratory measurements for the area. Error bars for the HPGe measurements are not derived from standard deviations in Tables 1A-1E, but represent the 1.96 $\sigma$ counting errors listed for each HPGe measurement in Appendix A. Error bars for laboratory data represent upper and lower 97.5% confidence limits based upon the weighted standard deviations in Tables 1A-1E.

Two points are particularly noteworthy based upon examination of Figures 2 through 6. First, the figures clearly demonstrate that linear correlation of HPGe and laboratory data can be extended to total uranium concentrations that exceed WAC limits (1030 ppm). In this regard, Areas PBC-18 and PBC-19 are bisected by the line having a slope of 1,0. Secondly, the figures clearly demonstrate that the linear correlation of HPGe with laboratory data is independent of the particular laboratory analytical method.

Linear Regression Analysis

Linear regression analyses of the data in Tables 1A-1E were carried out using the regression tools built into a commercial spreadsheet. Table 3 contains the correlation coefficient, slope and intercept calculated from these regression analyses. Linear regressions have been performed for:

- HPGe vs. alpha spectrometry data at 31 and 100 cm detector heights
- HPGe vs. gamma spectrometry data at 31 cm and 100 cm detector heights.
- HPGe vs. ICP/MS data at 31 and 100 cm detector heights.
- HPGe vs. bromoPADAP data at 31 and 100 cm detector heights.
- HPGe vs. "four method average" data at 31 and 100 cm detector heights

11

For each regression analysis, HPGe data comprised "y" coordinates while laboratory data comprised "x" coordinates.

The 31 cm data show very good linear regression characteristics: very high correlation coefficients (greater than 0.99), line slopes near 1.0, and intercepts near zero. Using criteria outlined in Section 3.3 of the July 1997 HPGe comparability report, 31 cm HPGe data display a high degree of comparability with laboratory data for total uranium for all four laboratory methods even extending from near background concentrations (5 ppm) to WAC exceedance concentrations (1000 ppm). The comparability of 100 cm HPGe data with laboratory data is also good, but not as good as the 31 cm data. Correlation coefficients are not quite as high, line slopes are a little more removed from 1.0, and intercepts are a little further removed from 0.0. These differences are consistent across all four analytical methods, and a possible reason for such differences is discussed later in the report.

## HETEROGENEITY OF TOTAL URANIUM CONCENTRATIONS

Areas PBC-18 and PBC-19 are excellent areas to examine to effect of analyte heterogeneity on HPGe readings. In area PBC-18, total uranium concentrations (four method average) range from a low of 560 ppm to a high of 2000 ppm. In area PBC-19, total uranium concentrations (four method average) vary from a low of 448 ppm to a high of 1780 ppm. HPGe measurements of total uranium (depending upon detector height) fall approximately in the middle of these low and high readings for each area. One of the principal strengths of HPGe is that it gives a very representative average reading for a given area (the size of which depends upon the detector height). Taking into account the total uranium concentration of the individual samples, HPGe measured total uranium concentrations appear subjectively to be very representative of areas PBC-18 and PBC-19.

Areas PBC-18 and PBC-19 also offer the opportunity to examine the effect of analyte heterogeneity with respect to physical samples. Clearly, if physical samples were the basis to provide information on the average total uranium concentration, the probability of one, two, or even three samples providing a reliable, representative average does not appear high. Further, one, two, or even three samples taken in each area may not provide reliable information as to the range of uranium concentrations in those areas.

The major point to be made is that both in-situ measurements and laboratory measurements have strengths and limitations in heterogeneous areas. The strengths and limitations in heterogeneous areas do not reflect upon the validity of the analytical measurement, but rather indicate the necessity to plan a sampling strategy that maximizes the strengths and minimizes the limitations given the intended use of the data.

## CONCLUSIONS

The following conclusions are the most important ones to be drawn from the data and discussions in this report:

1. HPGe can be used to accurately measure total uranium concentrations over a wide range of values from near background to greater than WAC levels (1030 ppm).

2. HPGe shows good comparability of data with data measured by a variety of laboratory analytical methods. Thus comparability is independent of the analytical method used.

3. HPGe can yield very representative average total uranium concentrations in areas in which uranium is very heterogeneously distributed.

13

DRAFT 2149

TABLE 1A

COMPARISON OF ALPHA SPECTROMETRY AND HPGe TOTAL URANIUM DATA

| HPGe Setting | Area | Alpha Spectrometry | | HPGe | |
|---|---|---|---|---|---|
| | | Weighted Mean(ppm) | Weighted Std. Dev. (ppm) | Mean (ppm) | Std. Dev. (Ppm) |
| 31 cm(1 ft) | PBC-01 | 7.5 | 0.3 | 9.0 | 0.6 |
| | PBC-02 | 30 | 1.4 | 31.2 | 1.9 |
| | PBC-03 | 1078 | 216 | 291 | 10.4 |
| | PBC-03* | 288 | 11.2 | 291 | 10.4 |
| | PBC-04 | 58 | 1.9 | 56.6 | 1.5 |
| | PBC-05 | 36 | 3.8 | 42.2 | 0.8 |
| | PBC-06 | 84 | 2.6 | 76.7 | 1.3 |
| | PBC-07 | 160 | 8.6 | 141 | 1.3 |
| | PBC-08 | 5.1 | 0.5 | 5.8 | 0.3 |
| | PBC-09 | 5.9 | 0.2 | 6.6 | 1.2 |
| | PBC-10 | 65 | 3.1 | 80.9 | 0.4 |
| | PBC-18 | 1141 | 120 | 1064 | 2.1 |
| | PBC-19 | 894 | 71 | 1047 | 4.6 |
| 1.0 m (3.28 ft.) | PBC-01 | 7.4 | 0.4 | 8.0 | ** |
| | PBC-02 | 26 | 1.4 | 26.0 | 2.8 |
| | PBC-03 | 447 | 130 | 287 | 7.9 |
| | PBC-03* | 285 | 16.7 | 287 | 7.9 |
| | PBC-04 | 56 | 3.1 | 58.6 | 1.8 |
| | PBC-05 | 45 | 5.4 | 47.9 | 1.7 |
| | PBC-06 | 88 | 3.8 | 75.9 | 2.9 |
| | PBC-07 | 174 | 13.4 | 140 | 2.0 |
| | PBC-08 | 4.9 | 0.6 | 5.3 | 0.3 |
| | PBC-09 | 6.2 | 0.2 | 6.9 | 0.5 |
| | PBC-10 | 55 | 3.6 | 74.0 | 0.4 |
| | PBC-18 | 1053 | 140 | 891 | 2.1 |
| | PBC-19 | 764 | 82 | 871 | 8.4 |

* Sample PBC-03-01 excluded from data set

** Only one HPGe measurement. Second measurement did not detect any gamma rays for uranium-238 and was excluded as a valid measurement

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TABLE 1B

COMPARISON OF GAMMA SPECTROMETRY AND HPGe TOTAL URANIUM DATA

| HPGe Setting | Area | Gamma Spectrometry | | HPGe | |
|---|---|---|---|---|---|
| | | Weighted Mean (ppm) | Weighted Std. Dev. (ppm) | Mean (ppm) | Std. Dev. (ppm) |
| 31 cm(1 ft) | PBC-01 | 8.2 | 0.9 | 9.0 | 0.6 |
| | PBC-02 | 34 | 2.6 | 31.2 | 1.9 |
| | PBC-03 | 1183 | 241 | 291 | 10.4 |
| | PBC-03* | 301 | 15 | 291 | 10.4 |
| | PBC-04 | 57 | 3.5 | 56.6 | 1.5 |
| | PBC-05 | 37 | 3.2 | 42.2 | 0.8 |
| | PBC-06 | 86 | 5.3 | 76.7 | 1.3 |
| | PBC-07 | 173 | 10 | 141 | 1.3 |
| | PBC-08 | 5.4 | 0.5 | 5.8 | 0.3 |
| | PBC-09 | 6.8 | 0.4 | 6.6 | 1.2 |
| | PBC-10 | 71 | 3.0 | 80.9 | 0.4 |
| | PBC-18 | 1185 | 97 | 1064 | 2.1 |
| | PBC-19 | 1029 | 82 | 1047 | 4.6 |
| 1.0 m (3.28 ft.) | PBC-01 | 8.6 | 1.0 | 8.0 | ** |
| | PBC-02 | 26 | 2.3 | 26.0 | 2.8 |
| | PBC-03 | 485 | 146 | 287 | 7.9 |
| | PBC-03* | 306 | 23 | 287 | 7.9 |
| | PBC-04 | 61 | 4.3 | 58.6 | 1.8 |
| | PBC-05 | 45 | 4.7 | 47.9 | 1.7 |
| | PBC-06 | 96 | 5.0 | 75.9 | 2.9 |
| | PBC-07 | 190 | 16 | 140 | 2.0 |
| | PBC-08 | 4.6 | 0.7 | 5.3 | 0.3 |
| | PBC-09 | 7.0 | 0.7 | 6.9 | 0.5 |
| | PBC-10 | 67 | 3.6 | 74.0 | 0.4 |
| | PBC-18 | 1197 | 129 | 891 | 2.1 |
| | PBC-19 | 918 | 99 | 871 | 8.4 |

* Sample PBC-03-01 excluded from data set

** Only one HPGe measurement. Second measurement did not detect any gamma rays for uranium-238 and was excluded as a valid measurement.

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TABLE 1C
COMPARISON OF ICP/MS AND HPGe TOTAL URANIUM DATA

| HPGe Setting | Area | ICP/MS | | HPGe | |
|---|---|---|---|---|---|
| | | Weighted Mean (ppm) | Weighted Std. Dev. (ppm) | Mean (ppm) | Std. Dev. (ppm) |
| 31 cm(1 ft) | PBC-01 | 7.1 | 0.7 | 9.0 | 0.6 |
| | PBC-02 | 35.2 | 1.9 | 31.2 | 1.9 |
| | PBC-03 | 1028 | 208 | 291 | 10.4 |
| | PBC-03* | 268 | 14.9 | 291 | 10.4 |
| | PBC-04 | 55.5 | 1.7 | 56.6 | 1.5 |
| | PBC-05 | 39.2 | 3.0 | 42.2 | 0.8 |
| | PBC-06 | 95.1 | 3.2 | 76.7 | 1.3 |
| | PBC-07 | 160 | 9.1 | 141 | 1.3 |
| | PBC-08 | 3.2 | 0.2 | 5.8 | 0.3 |
| | PBC-09 | 6.3 | 0.4 | 6.6 | 1.2 |
| | PBC-10 | 67.2 | 3.5 | 80.9 | 0.4 |
| | PBC-18 | 1120 | 90 | 1064 | 2.1 |
| | PBC-19 | 915 | 86 | 1047 | 4.6 |
| 1.0 m (3.28 ft.) | PBC-01 | 8.5 | 0.8 | 8.0 | ** |
| | PBC-02 | 31.5 | 2.6 | 26.0 | 2.8 |
| | PBC-03 | 422 | 126 | 287 | 7.9 |
| | PBC-03* | 267 | 19.0 | 287 | 7.9 |
| | PBC-04 | 55 | 2.8 | 58.6 | 1.8 |
| | PBC-05 | 47.1 | 4.0 | 47.9 | 1.7 |
| | PBC-06 | 99.6 | 3.7 | 75.9 | 2.9 |
| | PBC-07 | 180 | 13.6 | 140 | 2.0 |
| | PBC-08 | 3.5 | 0.2 | 5.3 | 0.3 |
| | PBC-09 | 6.3 | 0.5 | 6.9 | 0.5 |
| | PBC-10 | 56.7 | 4.0 | 74.0 | 0.4 |
| | PBC-18 | 1100 | 116 | 891 | 2.1 |
| | PBC-19 | 787 | 100 | 871 | 8.4 |

* Sample PBC-03-01 excluded from data set

** Only one HPGe measurement. Second measurement did not detect any gamma rays for uranium-238 and was excluded as a valid measurement

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TABLE 1D
COMPARISON OF BROMOPADAP AND HPGe TOTAL URANIUM DATA

| HPGe Setting | Area | BromoPADAP | | HPGe | |
|---|---|---|---|---|---|
| | | Weighted Mean (ppm) | Weighted Std. Dev. (ppm) | Mean (ppm) | Std. Dev. (ppm) |
| 31 cm(1 ft) | PBC-01 | 11.7 | 0.3 | 9.0 | 0.6 |
| | PBC-02 | 31.6 | 1.6 | 31.2 | 1.9 |
| | PBC-03 | 984 | 188 | 291 | 10.4 |
| | PBC-03* | 297 | 12.5 | 291 | 10.4 |
| | PBC-04 | 60.4 | 2.0 | 56.6 | 1.5 |
| | PBC-05 | 41.2 | 2.5 | 42.2 | 0.8 |
| | PBC-06 | 83.6 | 2.5 | 76.7 | 1.3 |
| | PBC-07 | 157 | 10.1 | 141 | 1.3 |
| | PBC-08 | 5.3 | 0.2 | 5.8 | 0.3 |
| | PBC-09 | 6.8 | 0.2 | 6.6 | 1.2 |
| | PBC-10 | 66.4 | 3.0 | 80.9 | 0.4 |
| | PBC-18 | 1126 | 91 | 1064 | 2.1 |
| | PBC-19 | 880 | 101 | 1047 | 4.6 |
| 1.0 m (3.28 ft.) | PBC-01 | 11.7 | 0.5 | 8.0 | ** |
| | PBC-02 | 28.2 | 2.1 | 26.0 | 2.8 |
| | PBC-03 | 433 | 114 | 287 | 7.9 |
| | PBC-03* | 293 | 18.1 | 287 | 7.9 |
| | PBC-04 | 58.0 | 3.2 | 58.6 | 1.8 |
| | PBC-05 | 47.1 | 3.5 | 47.9 | 1.7 |
| | PBC-06 | 87.8 | 3.3 | 75.9 | 2.9 |
| | PBC-07 | 179 | 15.2 | 140 | 2.0 |
| | PBC-08 | 5.3 | 0.4 | 5.3 | 0.3 |
| | PBC-09 | 6.7 | 0.2 | 6.9 | 0.5 |
| | PBC-10 | 58.9 | 3.6 | 74.0 | 0.4 |
| | PBC-18 | 1137 | 124 | 891 | 2.1 |
| | PBC-19 | 857 | 113 | 871 | 8.4 |

* Sample PBC-03-01 excluded from data set

** Only one HPGe measurement. Second measurement did not detect any gamma rays for uranium-238 and was excluded as a valid measurement

DRAFT

2149

TABLE 1E

COMPARISON OF FOUR METHOD AVERAGE WITH HPGe TOTAL URANIUM DATA

| HPGe Setting | Area | Four Method Average | | HPGe | |
|---|---|---|---|---|---|
| | | Weighted Mean(ppm) | Weighted Std. Dev. (ppm) | Mean (ppm) | Std. Dev. (Ppm) |
| 31 cm(1 ft) | PBC-01 | 8.6 | 0.3 | 9.0 | 0.6 |
| | PBC-02 | 32.5 | 1.7 | 31.2 | 1.9 |
| | PBC-03 | 1068 | 213 | 291 | 10.4 |
| | PBC-03* | 289 | 13 | 291 | 10.4 |
| | PBC-04 | 60.5 | 3.3 | 56.6 | 1.5 |
| | PBC-05 | 38.4 | 3.1 | 42.2 | 0.8 |
| | PBC-06 | 87.2 | 3.2 | 76.7 | 1.3 |
| | PBC-07 | 162 | 9 | 141 | 1.3 |
| | PBC-08 | 4.8 | 0.2 | 5.8 | 0.3 |
| | PBC-09 | 6.4 | 1.0 | 6.6 | 1.2 |
| | PBC-10 | 67.5 | 2.9 | 80.9 | 0.4 |
| | PBC-18 | 1143 | 98 | 1064 | 2.1 |
| | PBC-19 | 929 | 83 | 1047 | 4.6 |
| 1.0 m (3.28 ft.) | PBC-01 | 9.0 | 0.3 | 8.0 | ** |
| | PBC-02 | 28.1 | 1.9 | 26.0 | 2.8 |
| | PBC-03 | 447 | 129 | 287 | 7.9 |
| | PBC-03* | 288 | 19 | 287 | 7.9 |
| | PBC-04 | 60.6 | 4.2 | 58.6 | 1.8 |
| | PBC-05 | 46.1 | 4.2 | 47.9 | 1.7 |
| | PBC-06 | 93.0 | 3.7 | 75.9 | 2.9 |
| | PBC-07 | 181 | 14 | 140 | 2.0 |
| | PBC-08 | 4.6 | 0.2 | 5.3 | 0.3 |
| | PBC-09 | 6.5 | 1.1 | 6.9 | 0.5 |
| | PBC-10 | 59.5 | 3.3 | 74.0 | 0.4 |
| | PBC-18 | 1122 | 125 | 891 | 2.1 |
| | PBC-19 | 831 | 97 | 871 | 8.4 |

* Sample PBC-03-01 excluded from data set

** Only one HPGe measurement. Second measurement did not detect any gamma rays for uranium-238 and was excluded as a valid measurement

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TABLE 2A

CLOSENESS OF ALPHA SPECTROMETRY AND HPGe TOTAL URANIUM DATA

| Area | % Relative Deviation of Means (31 cm)* | % Relative Deviation of Means (100 cm)* |
|---|---|---|
| PBC-01 | -18.18 | -7.79 |
| PBC-02 | -3.92 | 0.00 |
| PBC-03# | 114.97 | 43.60 |
| PBC-03** | -1.04 | -0.70 |
| PBC-04 | 2.44 | -4.54 |
| PBC-05 | -15.86 | -6.24 |
| PBC-06 | 9.09 | 14.77 |
| PBC-07 | 12.62 | 21.66 |
| PBC-08 | -12.84 | -7.84 |
| PBC-09 | -11.20 | -10.69 |
| PBC-10 | -21.80 | -29.46 |
| PBC-18 | 6.98 | 16.67 |
| PBC-19 | -15.77 | -13.09 |

Average (using PBC-03) = 20.47 Average (using PBC-03) = 14.69

Average (using PBC-03**) = 10.98 Average (using PBC-03**) = 11.12

* Negative signs indicate laboratory data are less than HPGe data; positive signs indicate laboratory data are greater than HPGe data. Signs are not taken into account in the computation of the above averages which appear at the bottom of each column.

** Sample PBC-03-01 excluded from data set.

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TABLE 2B

CLOSENESS OF GAMMA SPECTROMETRY AND HPGE TOTAL URANIUM DATA

| Area | % Relative Deviation of Means (31 cm)* | % Relative Deviation of Means (100 cm)* |
|---|---|---|
| PBC-01 | -9.30 | 7.23 |
| PBC-02 | 8.59 | 0.00 |
| PBC-03# | 121.03 | 51.30 |
| PBC-03** | 3.38 | 6.41 |
| PBC-04 | 0.70 | 4.01 |
| PBC-05 | -13.13 | -6.24 |
| PBC-06 | 11.43 | 23.39 |
| PBC-07 | 20.38 | 30.30 |
| PBC-08 | -7.14 | -14.14 |
| PBC-09 | 2.99 | 1.44 |
| PBC-10 | -13.03 | -9.93 |
| PBC-18 | 10.76 | 29.31 |
| PBC-19 | -1.73 | 5.25 |

Average (using PBC-03) = 18.35 Average (using PBC-03) = 15.21
Average (using PBC-03**) = 8.55 Average (using PBC-03**) = 11.47

* Negative signs indicate laboratory data are less than HPGe data; positive signs indicate laboratory data are greater than HPGe data. Signs are not taken into account in the computation of the above averages which appear at the bottom of each column.

** Sample PBC-03-01 excluded from data set.

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TABLE 2C
CLOSENESS OF ICP/MS AND HPGE TOTAL URANIUM DATA

| Area | % Relative Deviation of Means (31 cm)* | % Relative Deviation of Means (100 cm)* |
|---|---|---|
| PBC-01 | -23.60 | 6.06 |
| PBC-02 | 12.05 | 19.13 |
| PBC-03# | 111.75 | 38.08 |
| PBC-03** | -8.23 | -7.22 |
| PBC-04 | -1.96 | -6.34 |
| PBC-05 | -7.37 | -1.68 |
| PBC-06 | 21.42 | 27.01 |
| PBC-07 | 12.62 | 25.00 |
| PBC-08 | -57.78 | -40.91 |
| PBC-09 | -4.65 | -9.09 |
| PBC-10 | -18.50 | -26.47 |
| PBC-18 | 5.13 | 20.99 |
| PBC-19 | -13.46 | -10.13 |

Average (using PBC-03) = 24.19 Average (using PBC-03) = 19.24
Average (using PBC-03**) = 15.56 Average (using PBC-03**) = 16.67

* Negative signs indicate laboratory data are less than HPGe data; positive signs indicate laboratory data are greater than HPGe data. Signs are not taken into account in the computation of the above averages which appear at the bottom of each column.

** Sample PBC-03-01 excluded from data set.

DRAFT 2149

TABLE 2D

CLOSENESS OF BROMOPAPAP AND HPGE TOTAL URANIUM DATA

| Area | % Relative Deviation of Means (31 cm)* | % Relative Deviation of Means (100 cm)* |
|---|---|---|
| PBC-01 | 26.09 | 37.56 |
| PBC-02 | 1.27 | 8.12 |
| PBC-03# | 108.71 | 40.56 |
| PBC-03** | 2.04 | 2.07 |
| PBC-04 | 6.50 | -1.03 |
| PBC-05 | -2.40 | -1.68 |
| PBC-06 | 8.61 | 14.54 |
| PBC-07 | 10.74 | 24.45 |
| PBC-08 | -9.01 | 0.00 |
| PBC-09 | 2.99 | -2.94 |
| PBC-10 | -19.69 | -22.72 |
| PBC-18 | 5.66 | 24.26 |
| PBC-19 | -17.33 | -1.62 |

Average (using PBC-03) = 18.25 Average (using PBC-03) = 14.96

Average (using PBC-03**) = 9.36 Average (using PBC-03**) = 11.75

* Negative signs indicate laboratory data are less than HPGe data; positive signs indicate laboratory data are greater than HPGe data. Signs are not taken into account in the computation of the above averages which appear at the bottom of each column.

** Sample PBC-03-01 excluded from data set.

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TABLE 2E

CLOSENESS OF FOUR METHOD AVERAGE AND HPGE TOTAL URANIUM DATA

| Area | % Relative Deviation of Means (31 cm)* | % Relative Deviation of Means (100 cm)* |
|---|---|---|
| PBC-01 | -4.55 | 11.76 |
| PBC-02 | 4.08 | 7.76 |
| PBC-03# | 114.35 | 43.60 |
| PBC-03** | -0.69 | 0.35 |
| PBC-04 | 6.66 | 3.36 |
| PBC-05 | -9.43 | -3.83 |
| PBC-06 | 12.81 | 20.25 |
| PBC-07 | 13.86 | 25.55 |
| PBC-08 | -18.87 | -14.14 |
| PBC-09 | -3.08 | -5.97 |
| PBC-10 | -18.06 | -21.72 |
| PBC-18 | 7.16 | 22.95 |
| PBC-19 | -11.94 | -4.70 |

Average (using PBC-03) = 18.74 Average (using PBC-03) =15.47

Average (using PBC-03**) =9.27 Average (using PBC-03**)=11.86

* Negative signs indicate laboratory data are less than HPGe data; positive signs indicate laboratory data are greater than HPGe data. Signs are not taken into account in the computation of the above averages which appear at the bottom of each column.

** Sample PBC-03-01 excluded from data set.

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TABLE 3
LINEAR REGRESSION PARAMETERS

2149

| ANALYTE | HPGe DETECTOR HEIGHT | CORRELATION COEFFICIENT | SLOPE of REGRESSION LINE | INTERCEPT of REGRESSION LINE | INTERCEPT as PERCENT of FRL* |
|---|---|---|---|---|---|
| Alpha Spectrometry | 31 cm | 0.991 | 1.012 | 2.2 | 2.68 |
| | 100 cm | 0.985 | 0.938 | 7.1 | 8.65 |
| Gamma spectrometry | 31 cm | 0.997 | 0.947 | 1.4 | 1.71 |
| | 100 cm | 0.990 | 0.813 | 9.4 | 11.5 |
| BromoPADAP | 31 cm | 0.991 | 1.035 | -0.94 | -0.47 |
| | 100 cm | 0.988 | 0.860 | 9.0 | 11.1 |
| ICP/MS | 31 cm | 0.994 | 1.023 | 0.51 | 0.62 |
| | 100 cm | 0.983 | 0.902 | 8.9 | 10.8 |
| Average of Four Methods | 31 cm | 0.994 | 1.007 | 0.35 | 0.43 |
| | 100 cm | 0.987 | 0.877 | 8.2 | 10.0 |

* Calculation is based upon an FRL of 82 ppm

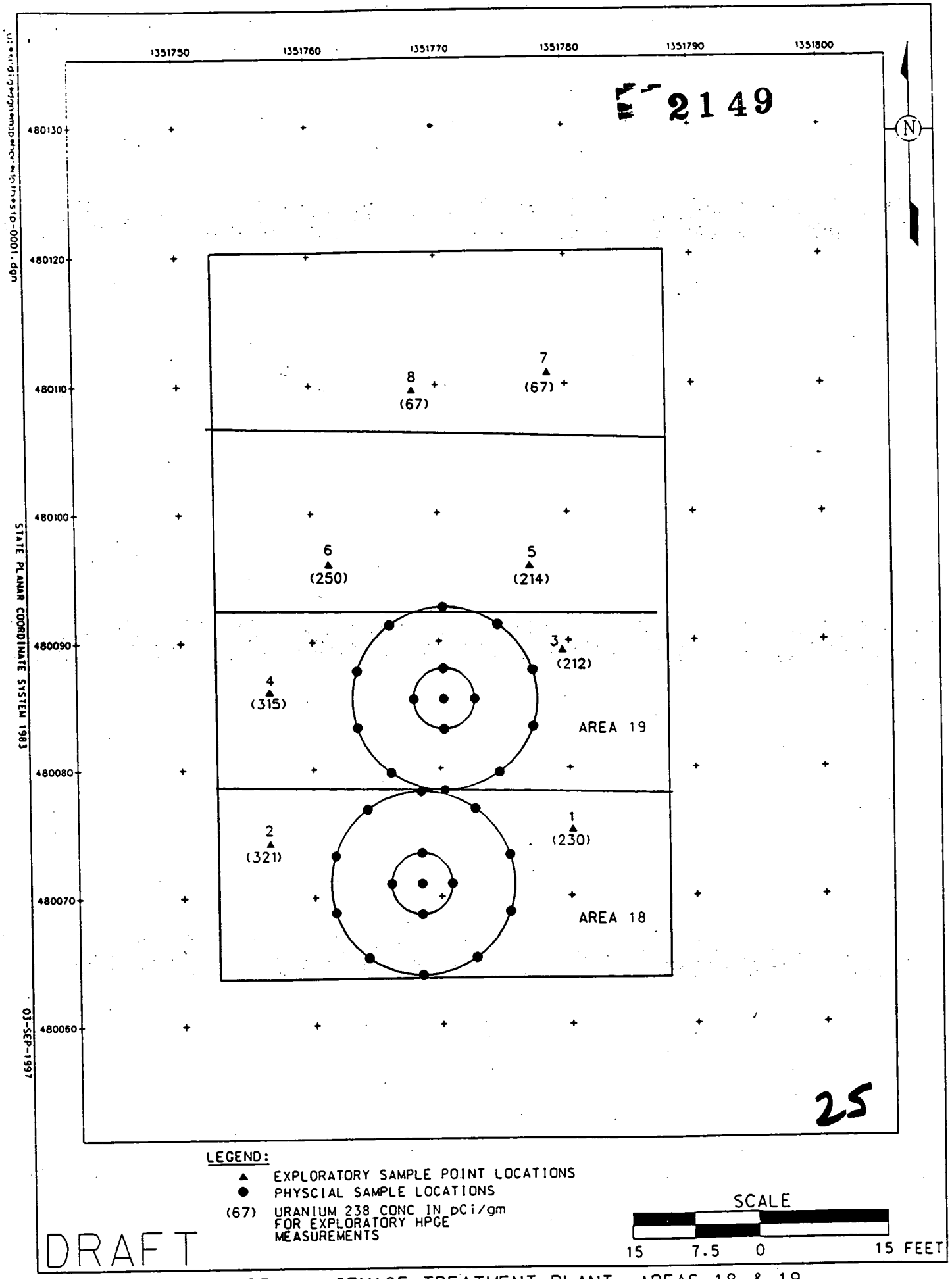


FIGURE 1. SEWAGE TREATMENT PLANT, AREAS 18 & 19

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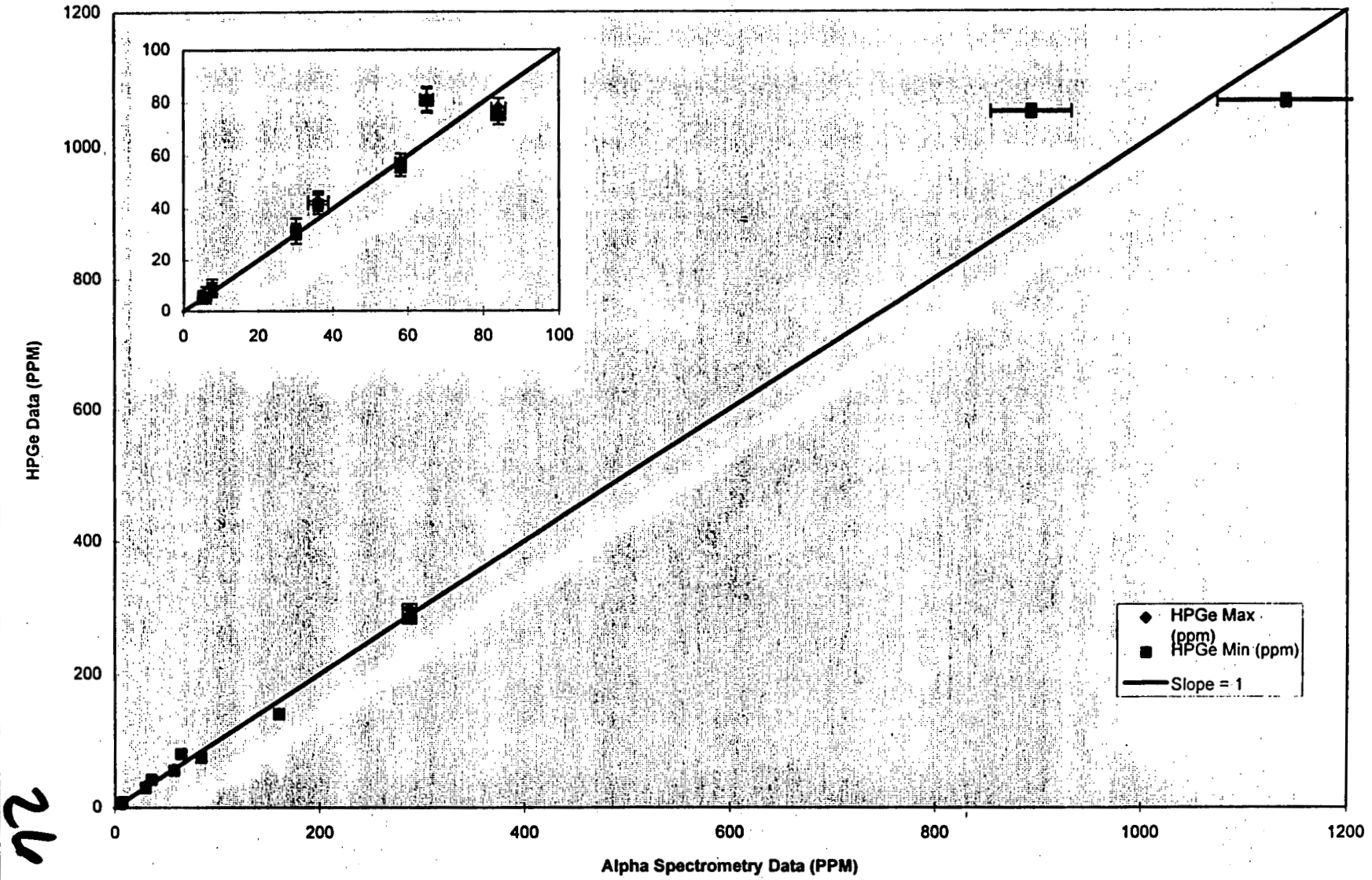
**Figure 2A**

**Correlation Between 31 cm HPGe and Alpha Spectrometry Data for Total Uranium (w/o PBC-03-01)**




2149

21

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**Figure 2B**

**Correlation Between 100 cm HPGe and Alpha Spectrometry Data for Total Uranium (w/o PBC-03-01)**

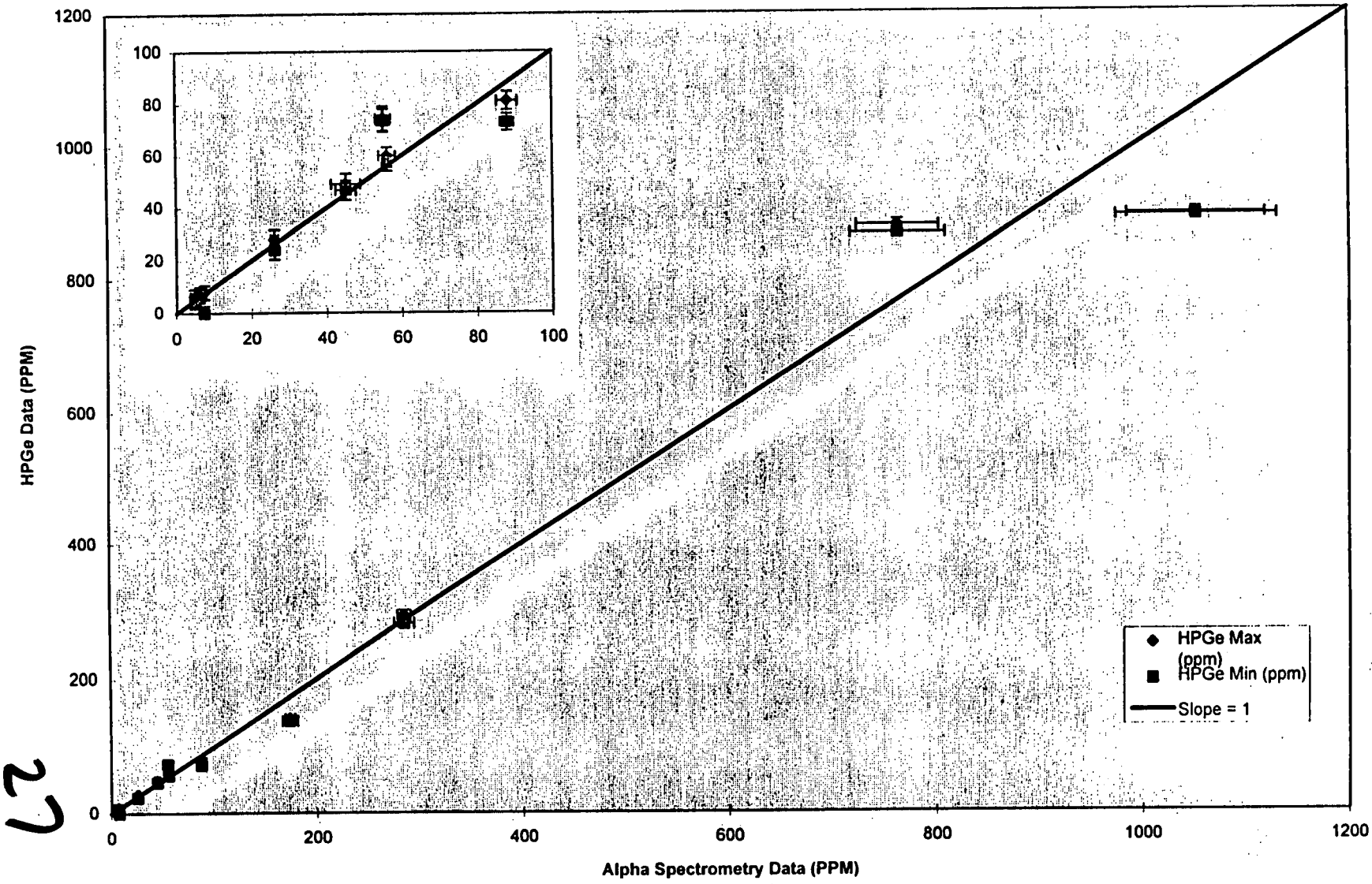

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**Figure 3A**

**Correlation Between 31 cm HPGe and Gamma Spectrometry Data for Total Uranium (w/o PBC-03-01)**

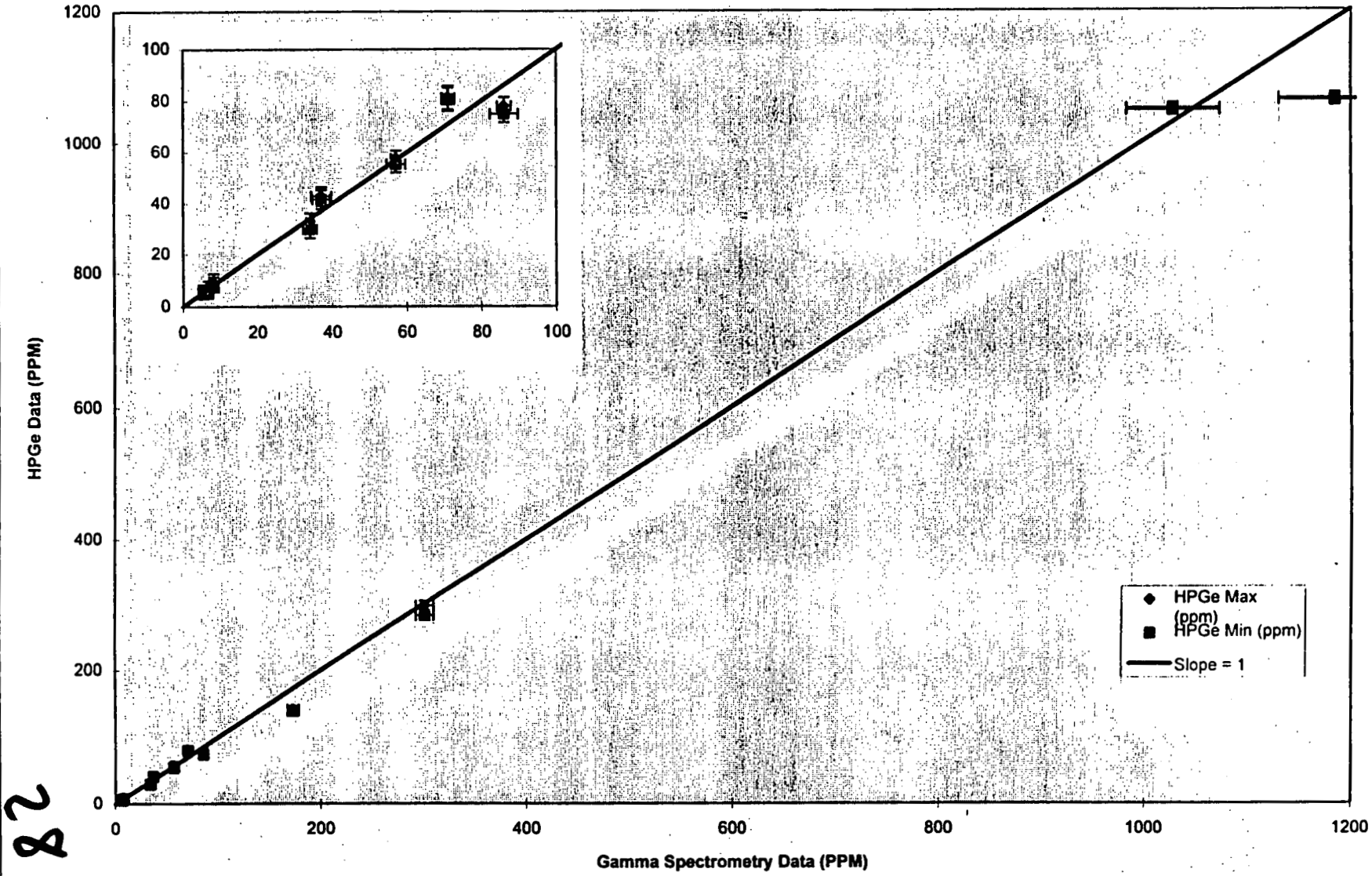

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**Figure 3B**

**Correlation Between 100 cm HPGe and Gamma Spectrometry Data for Total Uranium (w/o PBC-03-01)**

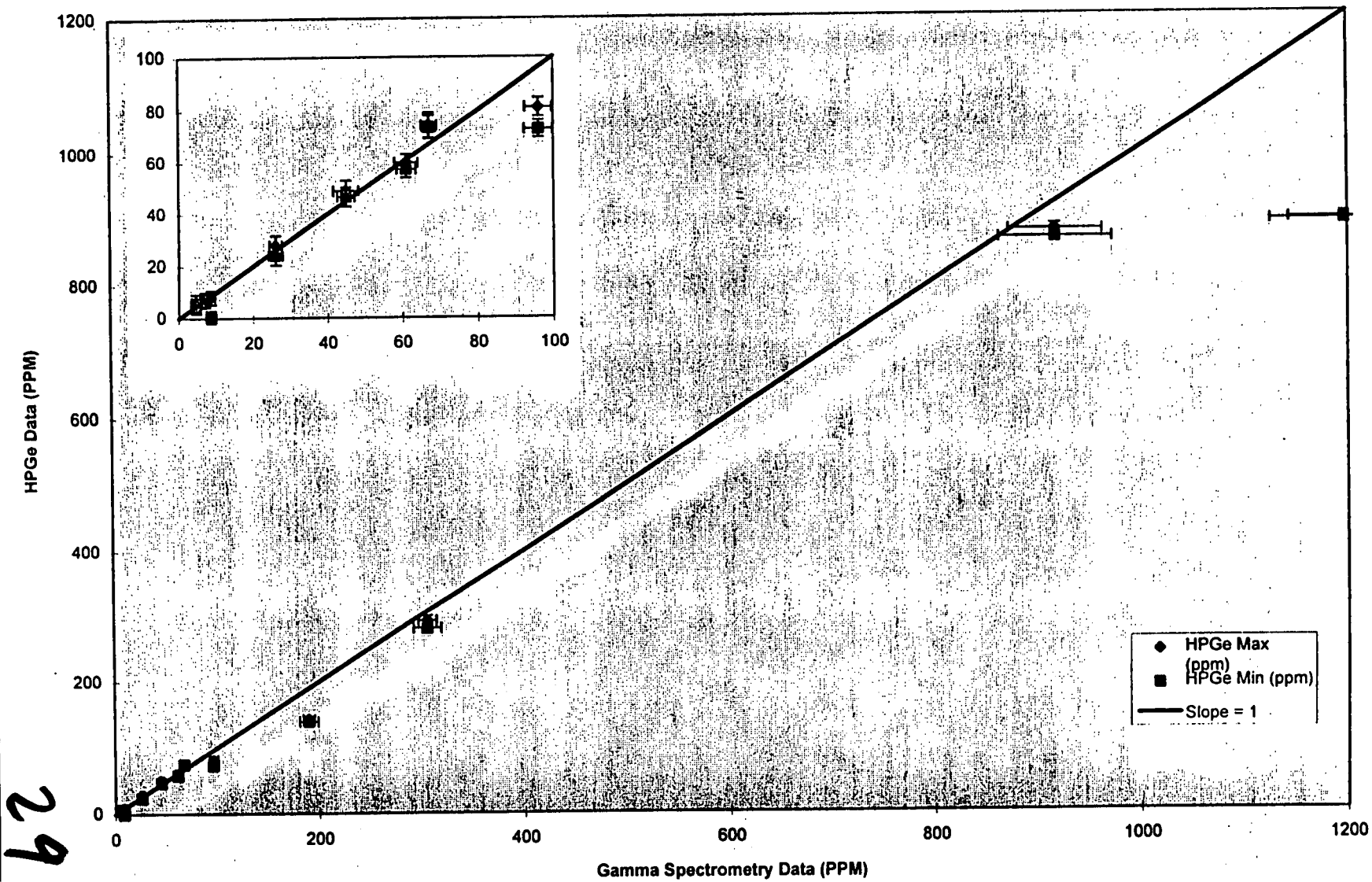

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**Figure 4A**
**Correlation Between 31 cm HPGe and ICP/MS Data for Total Uranium (w/o PBC-03-01)**

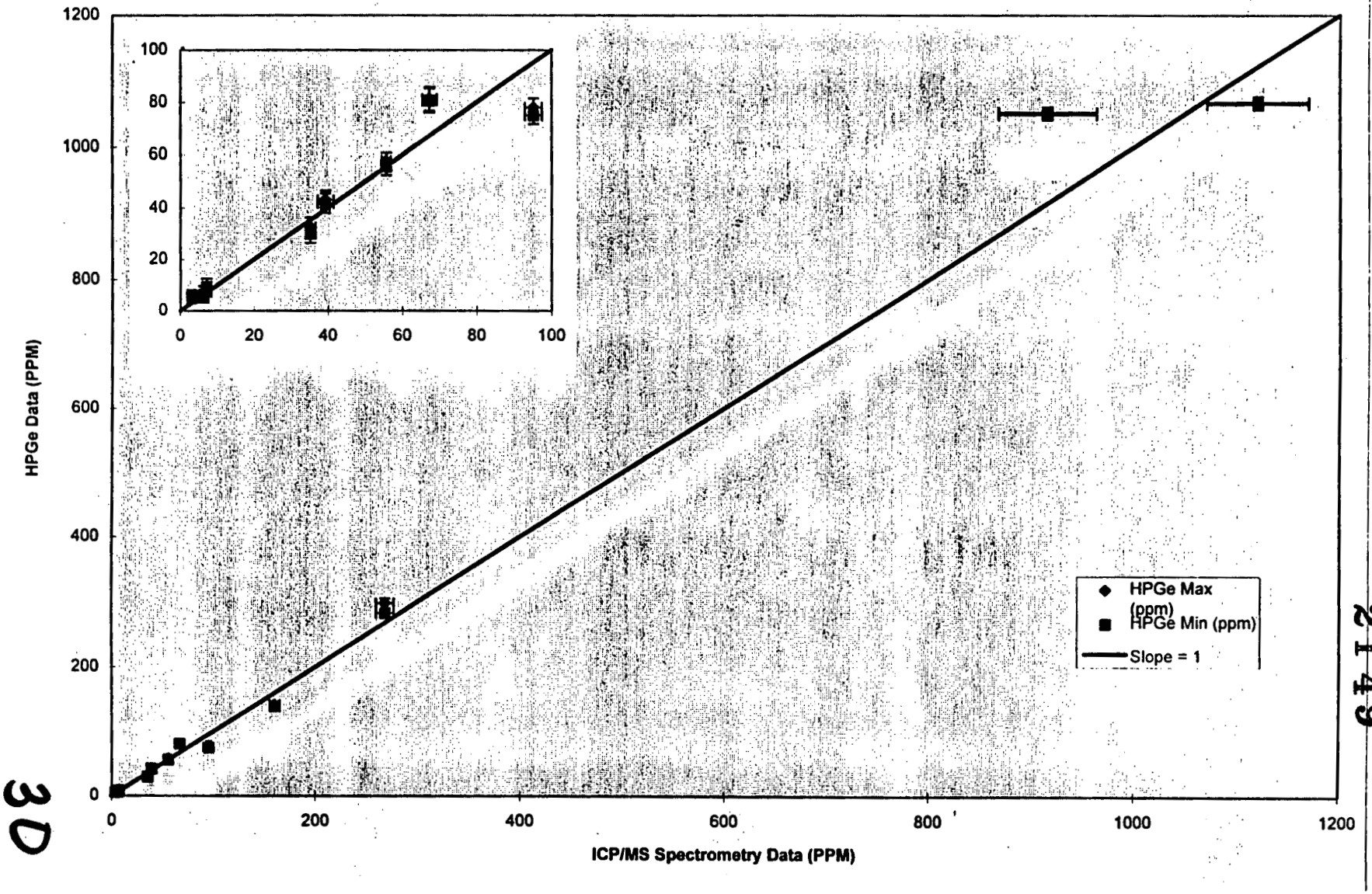

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**Figure 4B**

**Correlation Between 100 cm HPGe and ICP/MS Data for Total Uranium (w/o PBC-03-01)**

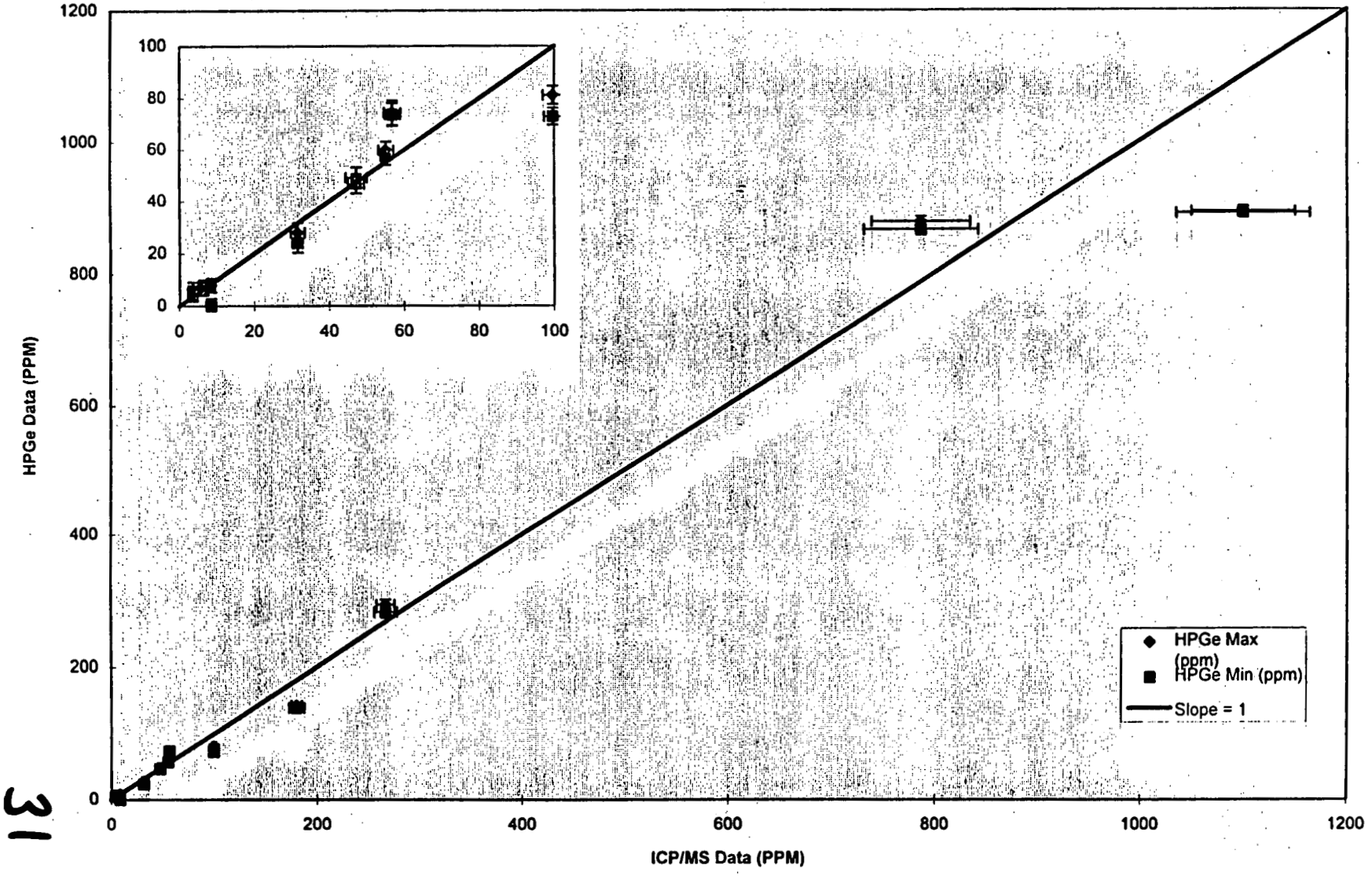

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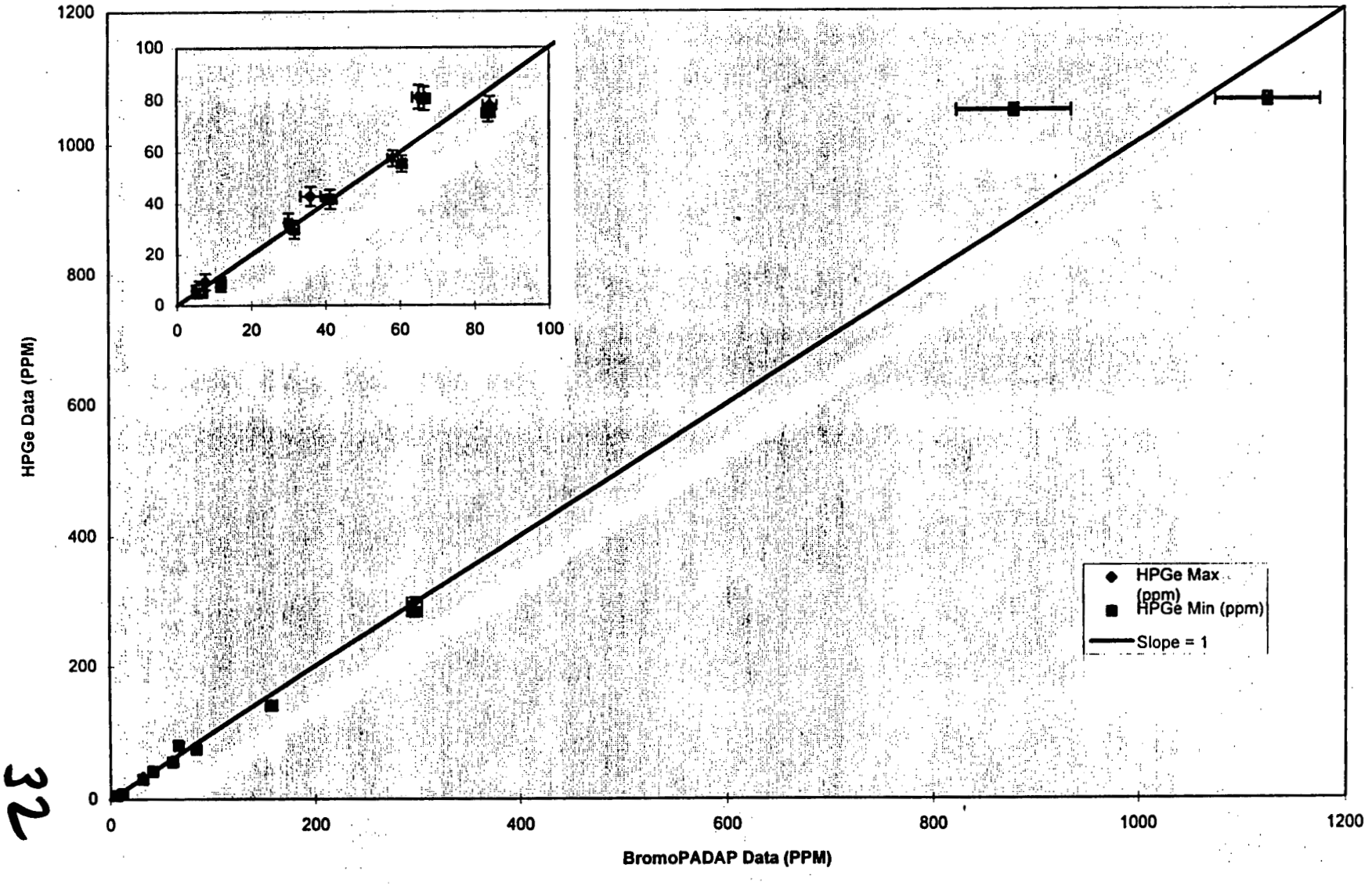
**Figure 5A**

**Correlation Between 31 cm HPGe and BromoPADAP Data for Total Uranium (w/o PBC-03-01)**




32

2149

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**Figure 5B**

**Correlation Between 100 cm HPGe and BromoPADAP Data for Total Uranium (w/o PBC-03-01)**

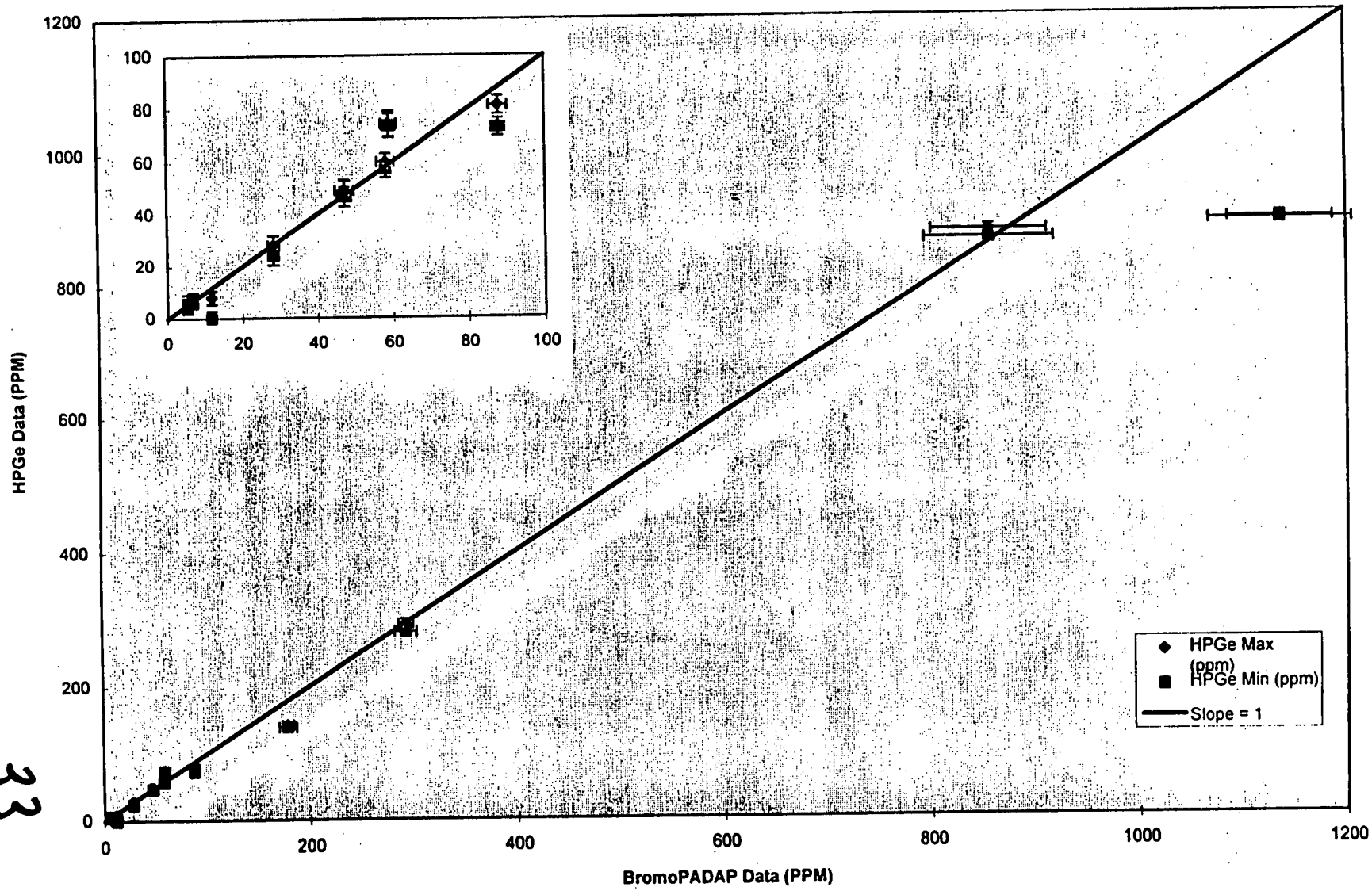

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**Figure 6A**

**Correlation Between 31 cm HPGe and Four Method Average Data for Total Uranium (w/o PBC-03-01)**

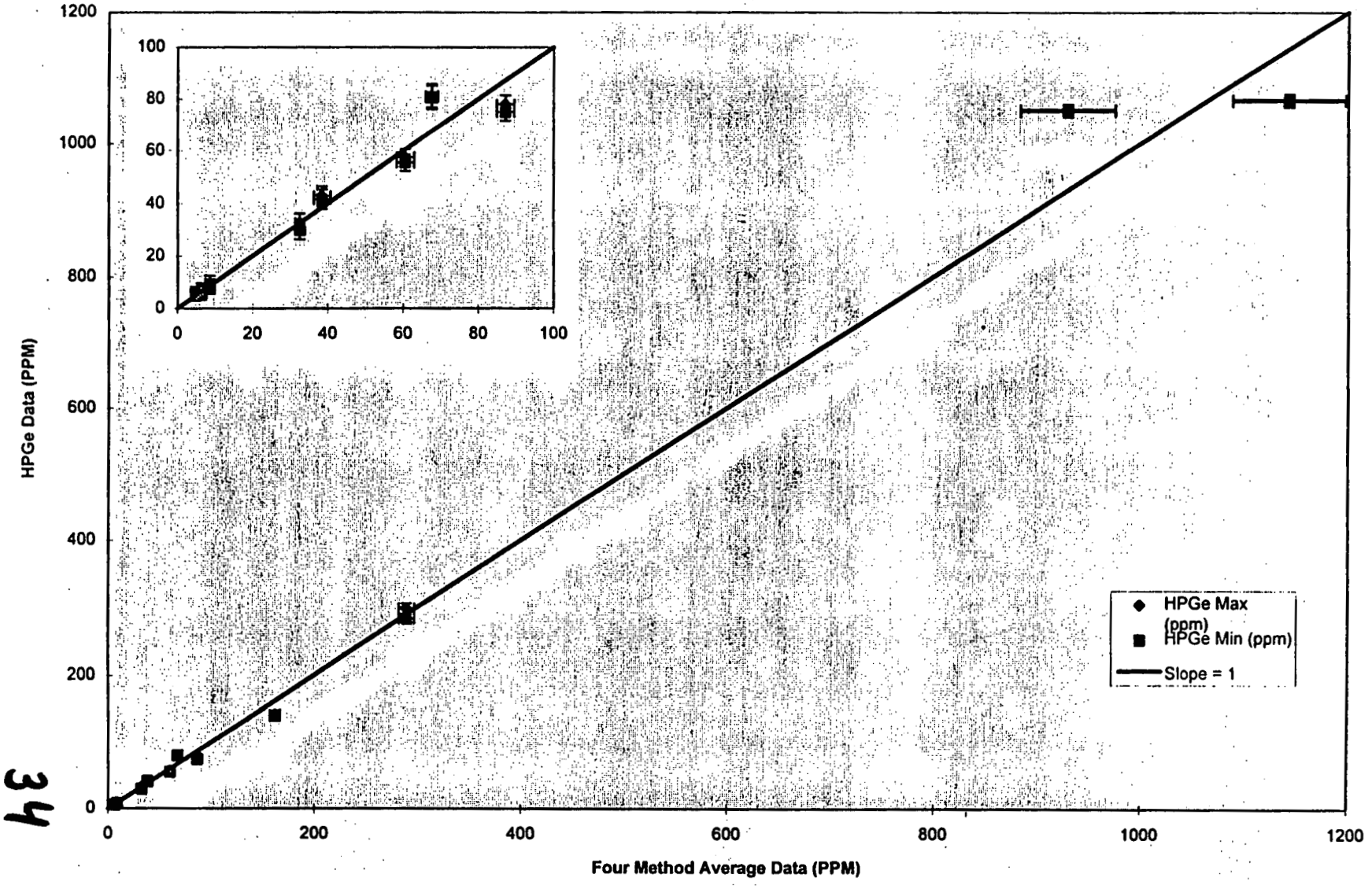

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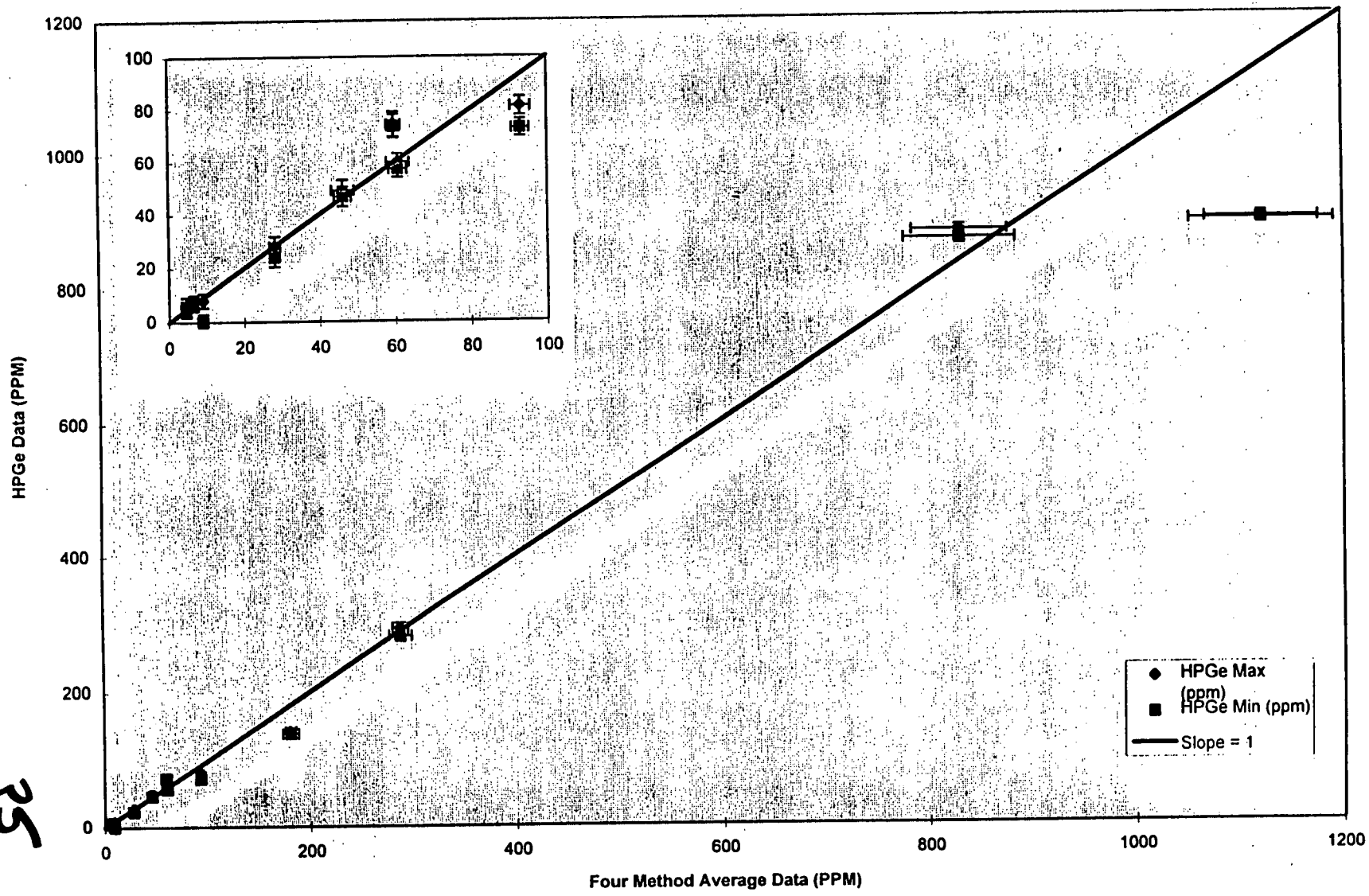
**Figure 6B**

**Correlation Between 100 cm HPGe and Four Method Average Data for Total Uranium (w/o PBC-03-01)**




35

# APPENDIX A

## TOTAL URANIUM RESULTS DETERMINED BY HPGe AND FOUR DIFFERENT LABORATORY METHODS

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA01-PBC | | | | | | | | | | | | | |
| PBC-1-1 | 1 m | 24.1 | 0.06 | 0.02 | | | | | | | | | | |
| PBC-1-1 | 1 m | 24.1 | 7.97 | 2.60 | | | | | | | | | | |
| PBC-1-1 | .31 m | 24.1 | 9.42 | 3.10 | | | | | | | | | | |
| PBC-1-1 | .31 m | 24.1 | 8.53 | 2.79 | | | | | | | | | | |
| PBC-1-1 | | 24.1 | | | 7.4 | 1.9 | 7.2 | 4.8 | 11.3 | .8 | 5.9 | .1 | 8.0 | 2.3 |
| PBC-1-2 | | 23.6 | | | 6.7 | 1.7 | 12 | 4.8 | 12.5 | .8 | 8.1 | .1 | 9.8 | 2.9 |
| PBC-1-3 | | 20.6 | | | 8.7 | 2.2 | 7.3 | 6 | 12.4 | .8 | 6.5 | .2 | 8.7 | 2.6 |
| PBC-1-4 | | 20.4 | | | 6.3 | 1.7 | 6.8 | 1.6 | 12.9 | .9 | 9.1 | .1 | 8.8 | 3.0 |
| PBC-1-5 | | 24.4 | | | 6.9 | 1.7 | 11 | 3.9 | 10.6 | .7 | 11.2 | .2 | 9.9 | 2.0 |
| PBC-1-6 | * | 20.6 | | | 8.3 | 2 | 6.2 | 2.6 | 11.2 | .8 | 8.2 | .1 | 8.5 | 2.1 |
| PBC-1-7 | * | 22.1 | | | 8.2 | 2 | 7.5 | 2.5 | 9 | .6 | 9.9 | .1 | 8.7 | 1.0 |

37

21149

* Duplicates



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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | Std Dev |
| Area: | AREA02-PBC | | | | | | | | | | | | | |
| PBC-2-1 | 1 m | 21.9 | 27.9 | 3.72 | | | | | | | | | | |
| PBC-2-1 | 1 m | 21.9 | 24.0 | 3.64 | | | | | | | | | | |
| PBC-2-1 | .31 m | 21.9 | 32.6 | 3.59 | | | | | | | | | | |
| PBC-2-1 | .31 m | 21.9 | 29.9 | 3.58 | | | | | | | | | | |
| PBC-2-1 | | 21.9 | | | 33 | 7.6 | 41 | 9.9 | 33.9 | 1.6 | 36.6 | .4 | 36.1 | 3.6 |
| PBC-2-2 | | 24.2 | | | 29 | 6.7 | 30 | 7 | 36 | 1.7 | 45.5 | .7 | 35.1 | 7.6 |
| PBC-2-3 | | 19.1 | | | 26 | 6.2 | 29 | 7 | 28.5 | 1.3 | 35.1 | .4 | 29.7 | 3.9 |
| PBC-2-4 | | 22 | | | 28 | 6.4 | 27 | 7.5 | 32 | 1.5 | 34.2 | .3 | 30.3 | 3.4 |
| PBC-2-5 | | 24.3 | | | 28 | 6.4 | 28 | 6.6 | 32.1 | 1.5 | 34.7 | .4 | 30.7 | 3.3 |
| PBC-2-6 | | 22.2 | | | 17 | 3.9 | 15 | 3.8 | 17.9 | .8 | 21.8 | .2 | 17.9 | 2.9 |
| PBC-2-7 | | 18.5 | | | 29 | 6.6 | 17 | 6.4 | 17 | .8 | 19.5 | .2 | 20.6 | 5.7 |
| PBC-2-8 | * | 19 | | | 24 | 6 | 21 | 6.9 | 31.1 | 1.4 | 29.1 | .3 | 26.3 | 4.6 |
| PBC-2-9 | | 19 | | | 20 | 4.6 | 23 | 6.8 | 21.3 | 1 | 21.7 | .2 | 21.5 | 1.2 |
| PBC-2-10 | | 30.1 | | | 25 | 5.6 | 27 | 3.6 | 27 | 1.2 | 27.5 | .2 | 26.6 | 1.1 |
| PBC-2-11 | * | 18.6 | | | 26 | 5.8 | 24 | 9.2 | 32 | 1.5 | 32.7 | .3 | 28.7 | 4.3 |

38

21149

* Duplicates



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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA03-PBC | | | | | | | | | | | | | |
| PBC-3-1 | 1 m | 21.2 | 292.2 | 7.90 | | | | | | | | | | |
| PBC-3-1 | 1 m | 21.2 | 281.0 | 7.86 | | | | | | | | | | |
| PBC-3-1 | .31 m | 21.2 | 283.4 | 7.66 | | | | | | | | | | |
| PBC-3-1 | .31 m | 21.2 | 298.1 | 7.65 | | | | | | | | | | |
| PBC-3-1 | | 21.2 | | | 1900 | 430 | 2100 | 140 | 1700 | 42.3 | 1819 | 16.7 | 1880 | 168 |
| PBC-3-2 | | 25.9 | | | 270 | 63 | 250 | 23 | 260 | 6.1 | 218 | 5.3 | 250 | 23 |
| PBC-3-3 | | 26.1 | | | 240 | 54 | 230 | 23 | 251 | 5.9 | 203 | 2.2 | 231 | 21 |
| PBC-3-4 | | 25 | | | 300 | 68 | 310 | 27 | 314 | 7.4 | 253 | 16.8 | 294 | 28 |
| PBC-3-5 | | 17.4 | | | 360 | 81 | 390 | 32 | 386 | 9.1 | 403 | 25.5 | 385 | 18 |
| PBC-3-6 | | 22 | | | 240 | 53 | 250 | 23 | 269 | 6.3 | 233 | 3.8 | 248 | 16 |
| PBC-3-7 | | 21.7 | | | 450 | 100 | 460 | 39 | 456 | 11.4 | 411 | 6.4 | 444 | 23 |
| PBC-3-8 | | 24.1 | | | 170 | 39 | 160 | 18 | 148 | 3.5 | 174 | 2.6 | 163 | 12 |
| PBC-3-9 | | 22 | | | 250 | 17 | 250 | 24 | 259 | 6.1 | 242 | 2.3 | 250 | 7 |
| PBC-3-10 | | 17.3 | | | 230 | 53 | 260 | 25 | 263 | 6.2 | 240 | 2.6 | 248 | 16 |
| PBC-3-11 | | 18.2 | | | 350 | 80 | 510 | 44 | 373 | 8.8 | 366 | 3.8 | 400 | 74 |
| PBC-3-12 | | 23.1 | | | 280 | 66 | 340 | 29 | 281 | 6.6 | 268 | 2.8 | 292 | 32 |
| PBC-3-13 | | 27.3 | | | 240 | 56 | 280 | 27 | 246 | 5.8 | 217 | 2.7 | 246 | 26 |
| PBC-3-14 | | 28.6 | | | 280 | 64 | 290 | 26 | 278 | 6.6 | 246 | 2.6 | 274 | 19 |
| PBC-3-15 | * | 24.4 | | | 320 | 73 | 360 | 30 | 331 | 7.8 | 292 | 3.1 | 326 | 28 |
| PBC-3-16 | * | 21.1 | | | 300 | 70 | 300 | 26 | 241 | 5.7 | 220 | 1.8 | 265 | 41 |

39

* Duplicates



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2149

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA04-PBC | | | | | | | | | | | | | |
| PBC-4-1 | 1 m | 27.3 | 57.3 | 3.28 | | | | | | | | | | |
| PBC-4-1 | 1 m | 27.3 | 59.9 | 3.32 | | | | | | | | | | |
| PBC-4-1 | .31 m | 27.3 | 55.5 | 3.20 | | | | | | | | | | |
| PBC-4-1 | .31 m | 27.3 | 57.6 | 3.14 | | | | | | | | | | |
| PBC-4-1 | | 27.3 | | | 58 | 13 | 50 | 7.7 | 62 | 1.6 | 54.8 | .8 | 56.2 | 5.1 |
| PBC-4-2 | * | 24.9 | | | 49 | 11 | 54 | 11 | 52.5 | 1.4 | 50.8 | .6 | 51.6 | 2.2 |
| PBC-4-3 | | 26.3 | | | 59 | 14 | 66 | 11 | 62.4 | 1.6 | 58.1 | 1 | 61.4 | 3.6 |
| PBC-4-4 | | 26.6 | | | 63 | 14 | 73 | 11 | 64.1 | 1.7 | 61.3 | .7 | 65.3 | 5.2 |
| PBC-4-5 | | 23.1 | | | 38 | 8.9 | 40 | 10 | 41.4 | 1.1 | 39.3 | .6 | 39.7 | 1.4 |
| PBC-4-6 | | 26.3 | | | 48 | 11 | 46 | 8.3 | 46.5 | 1.2 | 40.9 | .4 | 45.4 | 3.1 |
| PBC-4-7 | | 25.3 | | | 44 | 10 | 53 | 10 | 45 | 1.2 | 48.9 | .4 | 47.7 | 4.1 |
| PBC-4-8 | | 29.5 | | | 73 | 16 | 76 | 11 | 75.2 | 1.8 | 67 | .7 | 72.8 | 4.1 |
| PBC-4-9 | | 26.7 | | | 64 | 15 | 84 | 12 | 66.3 | 1.7 | 62.7 | .6 | 69.3 | 9.9 |
| PBC-4-10 | | 24.6 | | | 55 | 12 | 57 | 8.3 | 58.1 | 1.5 | 60.2 | .7 | 57.6 | 2.2 |
| PBC-4-11 | * | 25 | | | 61 | 14 | 68 | 11 | 58.8 | 1.5 | 57 | 1.1 | 61.2 | 4.8 |

2149

40

* Duplicates

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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | TPU | Result ppm | Std Dev |
| **Area:** | **AREA05-PBC** | | | | | | | | | | | | | |
| PBC-5-1 | 1 m | 21.8 | 49.1 | 3.95 | | | | | | | | | | |
| PBC-5-1 | 1 m | 21.8 | 46.7 | 3.92 | | | | | | | | | | |
| PBC-5-1 | .31 m | 21.8 | 41.6 | 3.69 | | | | | | | | | | |
| PBC-5-1 | .31 m | 21.8 | 42.8 | 3.70 | | | | | | | | | | |
| PBC-5-L | | 21.8 | | | 30 | 6.9 | 33 | 6.9 | 37.1 | 1.7 | 33.8 | .3 | 33.5 | 2.9 |
| PBC-5-2 | | 24 | | | 37 | 8.4 | 35 | 5.9 | 40.4 | 1.1 | 37.3 | .3 | 37.4 | 2.2 |
| PBC-5-3 | | 24.9 | | | 36 | 8.1 | 34 | 4.9 | 40.6 | 1.1 | 37.9 | .4 | 37.1 | 2.8 |
| PBC-5-4 | | 23.9 | | | 32 | 7.5 | 34 | 8.9 | 40.6 | 1.1 | 39.4 | .3 | 36.5 | 4.2 |
| PBC-5-5 | | 25.7 | | | 53 | 12 | 63 | 9 | 57.3 | 1.5 | 61.6 | .6 | 58.7 | 4.5 |
| PBC-5-6 | | 27.8 | | | 70 | 16 | 63 | 10 | 65.9 | 1.7 | 68.4 | .7 | 66.8 | 3.1 |
| PBC-5-7 | | 25.4 | | | 67 | 16 | 70 | 9.7 | 64 | 1.7 | 64.1 | .8 | 66.3 | 2.8 |
| PBC-5-8 | | 22.3 | | | 28 | 6.3 | 29 | 7.8 | 34 | 1.6 | 37 | .3 | 32.0 | 4.2 |
| PBC-5-9 | | 21.5 | | | 72 | 17 | 50 | 8 | 50.5 | 1.3 | 52 | .5 | 56.1 | 10.6 |
| PBC-5-10 | * | 26 | | | 36 | 8.5 | 44 | 9.1 | 43.9 | 1.1 | 44.2 | .4 | 42.0 | 4.0 |
| PBC-5-11 | * | 27.6 | | | 48 | 11 | 54 | 8.8 | 53.1 | 1.4 | 55.9 | .8 | 52.8 | [illegible] |

21149

* Duplicates



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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU ppm | Result | TPU ppm | Result | TPU ppm | Result | TPU ppm | Result | TPU ppm | Result | Std Dev ppm |
| Area: | AREA06-PBC | | | | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 76.1 | 3.48 | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 74.4 | 3.48 | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 76.4 | 3.49 | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 81.2 | 3.52 | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 72.9 | 3.51 | | | | | | | | | | |
| PBC-6-1 | 1 m | 26.7 | 74.4 | 3.50 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 78.0 | 3.41 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 75.0 | 3.34 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 75.5 | 3.36 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 77.9 | 3.36 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 77.7 | 3.40 | | | | | | | | | | |
| PBC-6-1 | .31 m | 26.7 | 76.4 | 3.37 | | | | | | | | | | |
| PBC-6-1 | | 26.7 | | | 80 | 18 | 72 | 10 | 78.8 | 1.9 | 89.4 | 1.2 | 80.1 | 7.2 |
| PBC-6-2 | | 27.5 | | | 84 | 19 | 97 | 11 | 89.5 | 2.1 | 91.6 | .9 | 90.5 | 5.4 |
| PBC-6-3 | | 27 | | | 93 | 21 | 110 | 13 | 92.3 | 2.2 | 115 | 1.3 | 102.6 | 11.6 |
| PBC-6-4 | | 24.3 | | | 85 | 19 | 87 | 11 | 81.1 | 1.9 | 95 | 1.1 | 87.0 | 5.9 |
| PBC-6-5 | | 24.7 | | | 110 | 24 | 120 | 14 | 91.7 | 2.2 | 106 | 1.2 | 106.9 | 11.7 |
| PBC-6-6 | | 28.1 | | | 88 | 20 | 90 | 11 | 93.8 | 2.2 | 101 | .8 | 93.2 | 5.7 |
| PBC-6-7 | | 26.8 | | | 82 | 19 | 100 | 13 | 92.1 | 2.2 | 101 | .8 | 93.8 | 8.8 |
| PBC-6-8 | | 25.3 | | | 110 | 24 | 120 | 14 | 108.8 | 2.6 | 121 | 1.4 | 115.0 | 6.4 |
| PBC-6-9 * | | 24.1 | | | 73 | 16 | 80 | 7.9 | 70.5 | 1.8 | 80 | .9 | 75.9 | 4.9 |

* Duplicates



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2149

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| PBC-6-10 | | 26.5 | | | 78 | 18 | 89 | 12 | 81.6 | 1.9 | 94.4 | 1.1 | 85.8 | 7.4 |
| PBC-6-11 | * | 24.4 | | | 76 | 17 | 80 | 10 | 67.6 | 1.8 | 84.1 | .8 | 76.9 | 7.0 |

43

F-21149

* Duplicates

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA07-PBC | | | | | | | | | | | | | |
| PBC-7-1 | 1 m | 28.4 | 138.9 | 4.21 | | | | | | | | | | |
| PBC-7-1 | 1 m | 28.4 | 141.8 | 4.19 | | | | | | | | | | |
| PBC-7-1 | .31 m | 28.4 | 140.3 | 4.04 | | | | | | | | | | |
| PBC-7-1 | .31 m | 28.4 | 142.1 | 4.06 | | | | | | | | | | |
| PBC-7-1 | | 28.4 | | | 150 | 38 | 160 | 16 | 139 | 3.3 | 149 | 1.2 | 150 | 9 |
| PBC-7-2 | | 29.5 | | | 170 | 42 | 180 | 17 | 166 | 3.9 | 173 | 2.1 | 172 | 6 |
| PBC-7-3 | | 28.4 | | | 140 | 36 | 180 | 18 | 154 | 3.6 | 163 | 2.2 | 159 | 17 |
| PBC-7-4 | | 26.6 | | | 140 | 36 | 140 | 14 | 143 | 3.4 | 116 | 3.8 | 135 | 13 |
| PBC-7-5 | | 27.5 | | | 190 | 42 | 190 | 19 | 180 | 4.2 | 153 | 4.4 | 178 | 17 |
| PBC-7-6 | | 27 | | | 210 | 47 | 200 | 19 | 208 | 4.9 | 199 | 11.4 | 204 | 6 |
| PBC-7-7 * | * | 27.2 | | | 180 | 41 | 180 | 19 | 183 | 4.3 | 162 | 7.2 | 176 | 10 |
| PBC-7-8 | | 25 | | | 170 | 38 | 200 | 19 | 182 | 4.3 | 199 | 2.3 | 188 | 14 |
| PBC-7-9 | | 28.7 | | | 150 | 38 | 160 | 16 | 156 | 3.7 | 165 | 1.2 | 158 | 6 |
| PBC-7-10 | | 27 | | | 140 | 32 | 159 | 17 | 151 | 3.6 | 166 | 1.5 | 154 | 11 |
| PBC-7-11 | | 27.7 | | | 130 | 30 | 140 | 15 | 137 | 3.2 | 144 | 2.8 | 138 | 6 |
| PBC-7-12 | | 28.5 | | | 210 | 49 | 230 | 23 | 220 | 5.2 | 241 | 2.2 | 225 | 13 |
| PBC-7-13 | | 28.6 | | | 360 | 80 | 430 | 33 | 396 | 9.9 | 352 | 21 | 385 | 36 |
| PBC-7-14 | | 23.7 | | | 160 | 37 | 170 | 17 | 160 | 3.8 | 179 | 2.1 | 167 | 9 |
| PBC-7-15 | | 28.9 | | | 180 | 40 | 200 | 16 | 188 | 4.4 | 223 | 2 | 198 | 19 |
| PBC-7-16 | * | 27 | | | 170 | 39 | 190 | 19 | 190 | 4.5 | 202 | 1.6 | 188 | 13 |

* Duplicates

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91149

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| **Area:** | **AREA08-PBC** | | | | | | | | | | | | | |
| PBC-8-1 | 1 m | 29.9 | 5.47 | 3.63 | | | | | | | | | | |
| PBC-8-1 | 1 m | 29.9 | 5.09 | 3.10 | | | | | | | | | | |
| PBC-8-1 | .31 m | 29.9 | 5.65 | 2.65 | | | | | | | | | | |
| PBC-8-1 | .31 m | 29.9 | 6.02 | 2.18 | | | | | | | | | | |
| PBC-8-1 | | 29.9 | | | 4.9 | 1.2 | 6.3 | 1.6 | 5.3 | .4 | 2.9 | .1 | 4.9 | 1.4 |
| PBC-8-2 | * | 35.1 | | | 4.3 | 1.2 | 5.3 | 1.5 | 5.1 | .3 | 3.4 | .1 | 4.5 | .9 |
| PBC-8-3 | | 26.3 | | | 7.3 | 1.9 | 4.9 | 2.3 | 5.5 | .4 | 3.2 | .1 | 5.2 | 1.7 |
| PBC-8-4 | | 30.9 | | | 3.2 | 1 | 6.6 | 2.2 | 4.4 | .3 | 3.5 | .2 | 4.4 | 1.5 |
| PBC-8-5 | | 28.2 | | | 4 | 1.1 | 3.4 | 1.5 | 4.5 | .3 | 3.4 | 0 | 3.8 | .5 |
| PBC-8-6 | | 34 | | | 6 | 1.6 | 2.3 | .9 | 6.7 | .5 | 4.5 | .1 | 4.9 | 1.9 |
| PBC-8-7 | • | 32.6 | | | 3.7 | 1.1 | 4.5 | 1.6 | 5.8 | .4 | 3.4 | .5 | 4.4 | 1.1 |

2149

45

* Duplicates



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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA09-PBC | | | | | | | | | | | | | |
| PBC-9-1 | 1 m | 29.3 | 7.26 | 2.61 | | | | | | | | | | |
| PBC-9-1 | 1 m | 29.3 | 6.55 | 2.58 | | | | | | | | | | |
| PBC-9-1 | .31 m | 29.3 | 5.75 | 2.46 | | | | | | | | | | |
| PBC-9-1 | .31 m | 29.3 | 7.41 | 2.30 | | | | | | | | | | |
| PBC-9-1 | | 29.3 | | | 5.8 | 1.8 | 6.7 | 2.1 | 7.1 | .5 | 6.1 | 0 | 6.4 | .6 |
| PBC-9-2 | | 27.3 | | | 5.8 | 1.4 | 7.2 | 1.7 | 6.5 | .4 | 8.1 | .1 | 6.9 | 1.0 |
| PBC-9-3 | | 26.3 | | | 5.9 | 1.4 | 6 | 2.5 | 5.9 | .4 | 5.1 | .1 | 5.7 | .4 |
| PBC-9-4 | • | 26.3 | | | 7.1 | 2.1 | 6.6 | 1.9 | 6.6 | .4 | 6 | .2 | 6.6 | .5 |
| PBC-9-5 | | 26.4 | | | 5.9 | 1.5 | 9.3 | 2.2 | 6.4 | .4 | 5.4 | .1 | 6.8 | 1.7 |
| PBC-9-6 | | 24.8 | | | 7.2 | 2.1 | 4.8 | 2 | 7.5 | .5 | 6.1 | .1 | 6.4 | 1.2 |
| PBC-9-7 | • | 25.6 | | | 5.7 | 1.4 | 9.1 | 6.7 | 7.6 | .5 | 7.6 | .1 | 7.5 | 1.4 |

• Duplicates



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## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| Area: | AREA10-PBC | | | | | | | | | | | | | |
| PBC-10-1 | 1 m | 20.8 | 74.3 | 4.76 | | | | | | | | | | |
| PBC-10-1 | 1 m | 20.8 | 73.7 | 4.75 | | | | | | | | | | |
| PBC-10-1 | .31 m | 20.8 | 81.2 | 4.47 | | | | | | | | | | |
| PBC-10-1 | .31 m | 20.8 | 80.6 | 4.48 | | | | | | | | | | |
| PBC-10-1 | | 20.8 | | | 73 | 16 | 77 | 12 | 72.9 | 1.9 | 77.2 | 1.1 | 75.0 | 2.4 |
| PBC-10-2 | | 19.1 | | | 48 | 11 | 52 | 11 | 48.5 | 1.3 | 51.9 | .8 | 50.1 | 2.1 |
| PBC-10-3 | | 17.4 | | | 62 | 15 | 61 | 11 | 61.5 | 1.6 | 64.6 | .5 | 62.3 | 1.6 |
| PBC-10-4 | | 18.8 | | | 67 | 15 | 65 | 11 | 77.8 | 2 | 62.8 | .5 | 68.2 | 6.7 |
| PBC-10-5 | | 21.8 | | | 72 | 16 | 87 | 13 | 69.1 | 1.8 | 68.7 | .9 | 74.2 | 8.7 |
| PBC-10-6 | | 16.7 | | | 41 | 9.3 | 66 | 13 | 38.3 | 1.8 | 35.4 | .3 | 45.2 | 14.1 |
| PBC-10-7 | | 18.8 | | | 30 | 6.9 | 38 | 10 | 39 | 1.8 | 31.3 | .4 | 34.6 | 4.6 |
| PBC-10-8 | | 18.8 | | | 31 | 7.3 | 61 | 13 | 32.9 | 1.5 | 27.6 | .2 | 38.1 | 15.4 |
| PBC-10-9 | | 16.8 | | | 55 | 12 | 96 | 16 | 51.6 | 1.3 | 40.1 | .5 | 60.7 | 24.4 |
| PBC-10-10 | * | 20.5 | | | 73 | 16 | 70 | 12 | 81.9 | 1.9 | 73.9 | .6 | 74.7 | 5.1 |
| PBC-10-11 | | 19.3 | | | 42 | 9.4 | 56 | 10 | 50 | 1.3 | 43.5 | 1 | 47.9 | 6.4 |
| PBC-10-12 | | 20.4 | | | 48 | 11 | 61 | 12 | 49.3 | 1.3 | 48 | .7 | 51.6 | 6.3 |
| PBC-10-13 | | 16.6 | | | 51 | 12 | 63 | 11 | 56.7 | 1.5 | 62 | 1 | 58.2 | 5.5 |
| PBC-10-14 | | 17.8 | | | 50 | 11 | 69 | 11 | 66.6 | 1.7 | 63.3 | 1 | 62.2 | 8.5 |
| PBC-10-15 | | 18.4 | | | 61 | 14 | 81 | 12 | 68.8 | 1.8 | 67.2 | .8 | 69.5 | 8.4 |
| PBC-10-16 | * | 15.2 | | | 56 | 13 | 63 | 12 | 65.1 | 1.7 | 75.8 | .6 | 65.0 | 8.2 |

* Duplicates



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21 49

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| Sample Id | Det Ht | Moisture % | HPGE Result (ppm) | HPGE TPU (ppm) | ALPHA Result (ppm) | ALPHA TPU (ppm) | GAMMA Result (ppm) | GAMMA TPU (ppm) | BRPADAP Result (ppm) | BRPADAP TPU (ppm) | ICP/MS Result (ppm) | ICP/MS TPU (ppm) | AVERAGE Result (ppm) | AVERAGE Std Dev (ppm) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Area: AREA18-PBC** | | | | | | | | | | | | | | |
| PBC-18-1 | 1 m | 8.9 | 892.5 | 7.97 | | | | | | | | | | |
| PBC-18-1 | 1 m | 8.9 | 889.5 | 8.00 | | | | | | | | | | |
| PBC-18-1 | .31 m | 8.9 | 1066 | 8.34 | | | | | | | | | | |
| PBC-18-1 | .31 m | 8.9 | 1063 | 8.36 | | | | | | | | | | |
| PBC-18-1 | | 8.9 | | | 1400 | 300 | 1300 | 120 | 1240 | 31 | 1260 | 17 | 1300 | 71 |
| PBC-18-2 | | 8.9 | | | 350 | 79 | 670 | 60 | 610 | 15 | 610 | 38 | 560 | 143 |
| PBC-18-3 | | 9.9 | | | 550 | 120 | 680 | 57 | 750 | 19 | 670 | 12 | 663 | 83 |
| PBC-18-4 | | 10.7 | | | 520 | 120 | 710 | 62 | 640 | 16 | 660 | 9 | 633 | 81 |
| PBC-18-5 | * | 15.8 | | | 1600 | 350 | 1500 | 130 | 1390 | 35 | 1520 | 66 | 1503 | 87 |
| PBC-18-6 | | 7.5 | | | 750 | 170 | 650 | 56 | 810 | 20 | 750 | 24 | 740 | 66 |
| PBC-18-7 | | 10.3 | | | 1700 | 390 | 1800 | 150 | 1960 | 49 | 1950 | 70 | 1853 | 125 |
| PBC-18-8 | | 13.8 | | | 1500 | 330 | 1700 | 140 | 1730 | 41 | 1480 | 15 | 1603 | 131 |
| PBC-18-9 | | 10.5 | | | 570 | 130 | 880 | 73 | 720 | 18 | 860 | 13 | 758 | 144 |
| PBC-18-10 | | 8.6 | | | 740 | 170 | 850 | 75 | 890 | 22 | 760 | 12 | 810 | 72 |
| PBC-18-11 | | 20.7 | | | 1600 | 350 | 2000 | 900 | 1760 | 43 | 1460 | 22 | 1705 | 232 |
| PBC-18-12 | | 9 | | | 1400 | 320 | 1700 | 130 | 1500 | 35 | 1550 | 19 | 1538 | 125 |
| PBC-18-13 | | 14.7 | | | 2200 | 500 | 2000 | 170 | 1930 | 47 | 1870 | 25 | 2000 | 144 |
| PBC-18-14 | | 11.8 | | | 810 | 180 | 1100 | 91 | 890 | 23 | 930 | 7 | 933 | 122 |
| PBC-18-15 | | 8 | | | 860 | 190 | 980 | 86 | 850 | 21 | 780 | 7 | 868 | 83 |
| PBC-18-16 | * | 16.3 | | | 1300 | 290 | 1700 | 140 | 1480 | 36 | 1380 | 20 | 1465 | 173 |

* Duplicates



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2149

## APPENDIX A - TOTAL URANIUM RESULTS DETERMINED BY HPGE AND FOUR DIFFERENT LABORATORY METHODS

| | | | HPGE | | ALPHA | | GAMMA | | BRPADAP | | ICP/MS | | AVERAGE | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Id | Det Ht | Moisture % | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | TPU | Result | Std Dev |
| | | | ppm | | ppm | | ppm | | ppm | | ppm | | ppm | |
| **Area:** | **AREA19-PBC** | | | | | | | | | | | | | |
| PBC-19-1 | 1 m | 37.2 | 877.3 | 7.80 | | | | | | | | | | |
| PBC-19-1 | 1 m | 37.2 | 865.4 | 7.77 | | | | | | | | | | |
| PBC-19-1 | .31 m | 37.2 | 1042 | 8.07 | | | | | | | | | | |
| PBC-19-1 | .31 m | 37.2 | 1051 | 8.06 | | | | | | | | | | |
| PBC-19-1 | .31 m | 37.2 | 1048 | 8.04 | | | | | | | | | | |
| PBC-19-1 | | 37.2 | | | 1000 | 230 | 1100 | 94 | 850 | 26 | 990 | 11 | 985 | 103 |
| PBC-19-2 | | 9.1 | | | 590 | 130 | 670 | 59 | 590 | 14 | 560 | 5 | 603 | 47 |
| PBC-19-3 | | 5.7 | | | 1500 | 340 | 1800 | 150 | 2040 | 48 | 1780 | 26 | 1780 | 221 |
| PBC-19-4 | | 22 | | | 800 | 180 | 860 | 69 | 730 | 20 | 790 | 7 | 795 | 53 |
| PBC-19-5 | | 14.6 | | | 590 | 130 | 870 | 71 | 570 | 15 | 680 | 8 | 678 | 137 |
| PBC-19-6 | | 8.1 | | | 460 | 100 | 530 | 50 | 580 | 15 | 410 | 4 | 495 | 75 |
| PBC-19-7 | | 6.3 | | | 500 | 110 | 600 | 58 | 640 | 15 | 520 | 8 | 565 | 66 |
| PBC-19-8 | | 16.5 | | | 1000 | 230 | 1600 | 130 | 1360 | 31 | 1220 | 20 | 1295 | 252 |
| PBC-19-9 | | 13.6 | | | 840 | 190 | 1000 | 95 | 990 | 22 | 770 | 14 | 900 | 113 |
| PBC-19-10 | | 7.2 | | | 500 | 110 | 630 | 56 | 620 | 15 | 560 | 10 | 578 | 60 |
| PBC-19-11 | | 12 | | | 600 | 140 | 670 | 63 | 630 | 16 | 570 | 10 | 618 | 43 |
| PBC-19-12 * | | 6.7 | | | 440 | 100 | 480 | 49 | 480 | 12 | 390 | 6 | 448 | 43 |
| PBC-19-13 | | 5.3 | | | 340 | 78 | 630 | 60 | 610 | 15 | 500 | 5 | 520 | 133 |
| PBC-19-14 | | 8.1 | | | 880 | 200 | 860 | 78 | 900 | 22 | 730 | 9 | 843 | 77 |
| PBC-19-15 | | 7.4 | | | 900 | 200 | 890 | 82 | 940 | 23 | 750 | 13 | 870 | 83 |
| PBC-19-16 * | | 6 | | | 500 | 110 | 610 | 58 | 480 | 12 | 340 | 8 | 483 | 111 |

* Duplicates

49



2149

# APPENDIX B

# DESCRIPTION OF BROMOPADAP AND ICP/MS ANALYTICAL METHODS

# APPENDIX B
# DESCRIPTION OF BROMOPADAP AND ICP/MS ANALYTICAL METHODS

The paragraphs below briefly describe the bromoPADAP and ICP/MS analytical methods. Detailed FEMP procedures exist for both methods: for bromoPADAP, the procedure is 256-S-6039 ("The Colorimetric (BrPADAP) Determination of Uranium Using an Auto Analyzer), and for ICP/MS The procedure is AC97-0043 ("Sample Preparation and Analysis for Uranium Determination by ICP/MS").

## BromoPADAP

BromoPADAP is used for the colorimetric determination of uranium in a variety of matrices in the concentration range of 1-100,000 ppm. The instrument consists of an autosampler, a proportionary pump, a flow cell colorimeter, and a personal computer loaded with peak detection, calibration, response factor, and reporting software.

The bromoPADAP is a classical colorimetric procedure for the determination of total uranium. In this procedure, the sample is rigorously digested with perchloric ($HClO_4$) and nitric ($HNO_3$) acid. Once solubilized, the uranium is reacted with 2-(5-bromo-2pyridylazo)-5-diethylaminophenol (bromoPADAP) to produce a colored complex which absorbs strongly at 580 nanometers. The amount of absorption is proportional to the concentration of uranium present. Uranium is separated from major interfering metal ions by extraction into trioctylphosphine oxide (TOPO) in cyclohexane. The extracting solution contains $F^-$ and $PO_4^{-3}$ ions to inhibit the extraction of Th and Zr along with uranium. Ascorbic acid in the extracting solution reduces $V^{+5}$ to $V^{+4}$, $Ce^{+4}$ to $Ce^{+3}$, $Cr^{+6}$ to $Cr^{+3}$, and $Fe^{+3}$ to $Fe^{+2}$. The interferences of these cations are much less severe at the lower valence states. A complexing solution is added during color development to prevent interference from traces of interfering elements. Sulfosalicylic acid masks the interferences of Be and Al. NaF reduces Th and Zr interferences and 1,2-diaminocyclohexylenedinitrilotetraacetic acid (Cy DTA) complexes many cations but not $UO_2^{+2}$. Quantificaiton of uranium is accomplished by comparing the absorbance of the unknown solution to the absorbances of standard uranium solutions.

ICP/MS

Inductively coupled plasma mass spectrometry (ICP/MS) is a relatively newly developed technique for the determination of total uranium. This procedure is an adaptation of the SW-846 6020 procedure for metals determination. The technique has proven to be a very sensitive and selective method. A soil sample is digested with $HClO_4$ and $HNO_3$, along with high heat. The digestate is then brought up in a 1% $HNO_3$ solution. The uranium in the $HNO_3$ solution is introduced into the ICP/MS instrument where it is nebulized and the resulting aerosol transported by argon gas into a plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences are assessed and valid corrections applied that include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. Standard uranium solutions are used to calibrate for this technique. The total uranium is based on the uranium-238 concentration. However, if concentrations of individual uranium are needed, ICP/MS can easily detect uranium-236, uranium-235, uranium-234, and uranium-233 as well as uranium-238.