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Elemental Composition of Fish Otoliths: Results of a Laboratory Intercomparison Exercise

by

Vincent S. Zdanowicz

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INTRODUCTION

Within the past decade, otolith elemental analysis has become increasingly utilized in studies of fishery biology. Based on the premise that differences in habitat chemistry are manifested in otoliths as differences in chemical composition, otolith elemental analysis has been used to address important questions in fishery research. However, this application has been hindered by the lack of an adequate standard for analytical quality control. As a newly emerging research technique, otolith elemental analysis requires further standardization between laboratories in order to optimize its usefulness.

Standardization can be achieved through the use of Certified Reference Materials (CRMs) and participation in intercomparison exercises. A CRM is a substance, one or more of whose properties is sufficiently well characterized to be used for the calibration of an apparatus or the assessment of a measurement method. Results of CRM analyses can be used to validate a laboratory's analytical results. They also provide a basis for comparing data generated at different laboratories or using different analytical methods. At the time of this exercise, no otolith CRMs were available, although one has since been developed through a collaboration between the Western Australian Marine Research Laboratories and Japan's National Institute for Environmental Studies (Yoshinaga, et al., 2000).

With the appearance of an increasing number of published studies and the absence of a suitable means of assessing the quality of their results, a laboratory intercomparison exercise was conducted in 1999 in an attempt to benchmark the status of otolith elemental analyses being conducted by investigators in the otolith research community. In the absence of a suitable otolith CRM, participation in intercomparison exercises can provide participants with a basis for direct comparison of their results with those of other laboratories.

SAMPLE DESCRIPTION

Three samples were used for the exercise: RMRS1, a fish otolith powder; SRM915a, a powdered, high purity calcium carbonate Standard Reference Material (SRM) produced by the National Institute of Standards and Technology (NIST); and SRMSOLN, a solution containing SRM915a dissolved in 1% nitric acid (1 mg $CaCO_3$/ml), spiked in the low ng/ml range with a mixture of metals.

RMRS1 was composed of ground, sieved, homogenized otoliths of red snapper (*Lutjanus campechanus*) from the Gulf of Mexico. Its true elemental composition was unknown. The otoliths were initially ground at NIST using their cryogenic homogenization technique (Zeisler, et al., 1983). This technique yields a powder containing particles of a range of sizes, including relatively large particles, so coarser fractions were ground (acid-rinsed, agate mortar and pestle) at the Howard Laboratory until all the powder passed through a 100 mesh screen (acid-rinsed, nylon). Enough material was obtained to produce 36 bottles of powder, each containing 450-500 mg. Homogeneity was assessed by analyzing three replicate samples from every sixth bottle, including the first and last, and comparing the concentrations of eight elements

(Li, Na, Mg, K, Ca, Mn, Sr and Ba) using the analysis of variance (ANOVA) (Zar, 1984). No statistical differences in composition were found between bottles, so the powder was judged to be homogeneous.

SRM915a was provided as an analytical reference material. Although it is not certified for trace metal content, some data on trace metal levels are provided in its Certificate of Analysis. Also, it is very pure, so it can be used to provide a clean matrix match for calibration standards/spikes, or possibly as a calibration standard for probe analyses.

SRMSOLN was intended to provide information on interlaboratory variability due to sample preparation methods. It was spiked with measurable levels of a suite of metals, some of whose concentrations in the powdered samples were expected to be below the detection limits of some laboratories. It was thought that laser laboratories that analyze solutions might also find it useful.

DETAILS OF THE EXERCISE

The exercise was performance-based. No particular sample preparation or instrumental procedure was specified. Participants were to use their routine procedures, including solid state (probe) methods, total dissolution techniques, isotope dilution, atomic absorption/emission, and others. Participants were invited to use more than one procedure and report more than one set of results.

Each participant was sent approximately 500 mg of RMRS1, 500 mg of SRM915a and 30 ml of the SRMSOLN. A copy of the Certificate of Analysis for SRM915a was included. Participants were asked to prepare five replicates of each material for analysis using their routine sample preparation method(s), to analyze the prepared samples using their routine analytical procedure(s), and to measure as many, or as few, elements / isotopes as they wished. For the powders, they were asked to calculate dry weight concentrations for each element using their routine procedure. For the solution, they were asked to report results in ng/ml and not to perform a blank correction.

A formatted spreadsheet was provided for reporting the analytical results and the details of laboratory procedures, including sample preparation methods, calibration methods, etc. Analytical results were compiled at the Howard Laboratory and each set of results was assigned a unique identification number (Lab #). When all results had been received, each participant was sent a listing of raw data in order to verify that no transcription errors were made in the compilation of the data.

The true composition of the otolith powder was unknown, and no attempt was made to assign consensus or “correct” values to any analyte. The purpose of the exercise was to provide a basis for direct comparison of results among laboratories.

METHODS

Two data sets were used for this report. The Raw (unedited) data set contains all verified, final data, including LTVs ("Less Than" Values). The Raw data set was

used only to construct the tables in Appendix B. The Quantitative (edited) data set contains all verified, final data, not including LTVs. The Quantitative data set was used for summary statistics appearing in the tables in Appendix B, for data plots in Appendix C, and for all computations and statistical evaluations and any tables resulting from them (text Tables 1 through 10 and Appendix D Tables D1 through D6).

Tables

Replicate data for 28 elements are compiled in the tables in Appendix B. Data are listed as received with respect to significant figures, or using four decimal places, whichever was fewer. Also shown are the mean, standard deviation and the %CV (coefficient of variation, computed as 100 x laboratory standard deviation / laboratory mean). For SRM915a, values listed as "Ref" are from the Certificate of Analysis. For SRMSOLN, values listed as "Actual" are the total concentrations present in the solution. For example, Mg is present in SRM915a at 1 ug/g dry weight. SRMSOLN contains 1 mg of the SRM per ml of solution. This contributes 1 ng Mg per ml of solution to the total solution concentration. Another 3.5 ng Mg per ml was contributed by the spike. Thus, the "Actual" concentration was 4.5 ng Mg per ml solution. For elements present in the spike solution but not listed on the Certificate of Analysis, the amount contributed by the SRM was assumed to be zero.

Figures

Replicate data for 18 elements are plotted versus Lab # in the data plots in Appendix C. Elements with fewer than four sets of results for at least one sample were not plotted.

Z-scores and P-scores

Z-scores and p-scores were computed for each laboratory for each element in each sample (Appendix D).

Z-scores were calculated as

$$z = (x_L - X) / S$$

where x_L = the laboratory mean, X = the accepted value and S = the target value for the standard deviation.

P-scores were calculated as

$$p = s_L / S$$

where s_L = the laboratory standard deviation and S = the target value for the standard deviation.

For this exercise, the value used for X was the overall mean for the element in the sample and the value used for S was the overall standard deviation for the element in the sample. The term "overall" indicates that all data (from the Quantitative data set) submitted by all participants were used.

RESULTS

Samples were distributed in January 1999 to 29 laboratories in ten countries, listed in Appendix A. Results were due by July 1999. Sixteen sets of results were received from fourteen laboratories from eight countries. Data were received for 28 elements. Results are compiled and summarized in tables and plots in Appendices B and C. Z-scores and p-scores are given in Appendix D.

Methods used and isotopes measured by the participants are listed in Appendix E. Two laboratories conducted solid state analyses by microprobe; they did not analyze the SRMSOLN sample. Fourteen laboratories dissolved the powders; they also analyzed the SRMSOLN sample. In almost every case, the dissolution procedure was performed using nitric acid in an open vessel at room temperature. Most laboratories used quadrupole inductively coupled plasma mass spectrometry (QICPMS) for the solution analyses, one used High Resolution ICPMS. Atomic absorption spectrophotometry (AAS) was next most frequently used, followed by ICPAES (atomic emission spectrometry).

After excluding LTVs and elements with fewer than five sets of results, 11 elements remained as candidates for evaluation for SRM915a, 15 for the RMRS1 and SRMSOLN samples. Three factors greatly influenced the evaluation of the results: 1) few certified or reference data were available for SRM 915a; 2) the composition of RMRS1 was unknown; and 3) no attempt was made to derive consensus or “correct” values for any analyte in any sample. Nevertheless, an attempt was made to assess the accuracy and precision of the results and the extent of agreement between laboratories.

Accuracy

Certified or informational values are available for six elements in SRM915a. In general, informational values differ from certified values in that they have not been determined by two independent methods, nor subjected to rigorous statistical evaluation. An informational value is a "value of a property, not certified but provided because it is believed to be reliable and to provide information important to the certified material" (Taylor, 1985). Quantitative results for sodium were submitted by only one laboratory, so accuracy was evaluated based on the five elements listed below.

Mg - 1.0 ug/g (informational value)
Ca - 40.0 % (certified value)
Mn - 0.6 ug/g (informational value)
Cu - 0.95 ug/g (average of 0.9 ug/g and 1 ug/g informational values)
Sr - 2.1 ug/g (informational value)

The measure used was % Recovery, computed as 100 x laboratory mean / certificate value. State-of-the-art accuracy in trace element analysis requires recoveries within 20% of the true concentration at analyte levels less than 1 ppm, within 10% of the true concentration at analyte levels greater than 1 ppm and within 5% of the true concentration at analyte levels in the percent range (greater than 1000 ppm). In this report, three cut-off levels were used to categorize results: ±10%, ±20% and > 150%, corresponding to "good," "acceptable," and "poor" or "unacceptable" accuracy.

Of the five elements listed above, good accuracy was achieved by all the laboratories only for Ca, present in the SRM at 40.0% (400,000 ppm, Table 1). All the laboratories (nine of nine) had recoveries within 10% of the certificate value. Results for the other four elements were generally poor. For Mg, present at 1 ug/g, only one (of nine) laboratory had a recovery within 10% of the certificate value; eight of nine recoveries were >150% of the certificate value. For Mn (0.6 ug/g) and Cu (0.95 ug/g), only one laboratory had a recovery within 10% of the certificate value; three had recoveries within 20% of the certificate value, but recoveries for three laboratories were >150% of the certificate value. For Sr (2.1 ug/g), the best recovery was 147%; seven of eight laboratories had recoveries >150% of the certificate value.

Recovery results for the SRMSOLN sample are summarized in Table 2. Again, the best accuracy was achieved only for Ca, present in the sample at 400,000 ng/ml. All the laboratories (10 of 10) had recoveries within 20% of the actual value, while eight of 10 recoveries were within 10% of the actual value. Results for the other four elements were slightly better than for the SRM, but still poor. For Mg, present at 4.5 ng/ml, no recoveries were within 10% of the actual value, while three (of 11) were within 20% of the actual value, and only four of 11 were >150% of the actual value. For Mn (4.1 ng/ml), more than half the laboratories (seven of 13) had recoveries within 10% of the actual value, and only three recoveries were >150% of the actual value. For Cu (4.4 ng/ml), three laboratories had recoveries within 10% of the actual value; five had recoveries within 20% of the actual value, and only two laboratories had recoveries >150% of the actual value. For Sr (5.6 ng/ml), one laboratory had a recovery within 10% of the actual value; two had recoveries within 20% of the actual value, and three laboratories had recoveries >150% of the actual value. Thus, for the SRMSOLN sample, there was a slight improvement over SRM915a recoveries.

Accuracy was not assessed for the RMRS1 sample, since its composition was unknown and consensus values were not derived for any element in the sample.

Although most laboratories used QICPMS to measure elemental concentrations, other techniques were also used. However, accuracy does not appear to be related to the instrumental methods used, nor to the specific isotopes measured. For example, best recoveries were obtained for Ca in the SRM915a and SRMSOLN samples. Ca was measured using ICPAES, Flame AA, and ICPMS (five different isotopes) with equally good results. These good recoveries were most likely related to the high concentrations of Ca in these samples, resulting in greater ease of measurement. Worst recoveries, in general, were obtained for Mg, which was also was measured by ICPAES, Graphite Furnace AA, and ICPMS (three different isotopes). Recoveries in the SRMSOLN sample were better than in the SRM915a sample, but again this was most likely due to higher concentrations of Mg in the SRMSOLN sample.

Precision

Analytical precision was assessed based on intralaboratory %CV. For each sample, elements with five or more sets of data were evaluated. Thus, 11 elements were evaluated for SRM915a and 15 for the RMRS1 and SRMSOLN samples. Cut-off levels used to categorize results were 10%, 20% and 50%, corresponding to "good," "acceptable," and "poor" or "unacceptable" precision.

Table 3 shows the precision results for SRM915a. Best results were obtained for Ca; all nine laboratories had CVs of 10% or less. This is not unexpected, however, since precision usually improves with concentration. If the sample weights reported in Appendix E were used for SRM915a as well as for RMRS1, Ca concentrations in solution would be more than high enough to promote good precision. This would not be true for other elements, however. For elements present in SRM915a in ug/g levels, they would be present in solutions prepared from SRM915a in ng/ml concentrations and precision would not be expected to be as high as for Ca. This, in fact, was observed. Nevertheless, for Mg, Mn, Cu and Sr (elements for which accuracy was assessed), precision results were much better than accuracy results - that is, most laboratories had CVs < 20%, and for Cu and Sr most CVs were < 10%. The worst precision for this group was for Mg, where two laboratories had CVs > 50%. Precision results for Co, Ni, Zn, Ba and Pb were comparable to results for the "accuracy group"; half the laboratories (for Co and Ni, almost all the laboratories) had CVs < 20% and many laboratories had CVs < 10%. Ba was the worst of this group; three laboratories had CVs > 50%. The worst results were for Cr. Of four laboratories, only one had a CV < 20%; one laboratory had a CV > 50%.

Results for SRMSOLN are given in Table 4. Best precision was again obtained for Ca (all laboratories had CVs < 10%), but comparably high precision was also achieved for Mg, Mn, Cu and Sr (the "accuracy group"), as well as for Li, Co, Zn, Cd, Ba and Pb. For Cr, Ni, As and Rb, most laboratories had CVs < 20%, and for As and Rb, one laboratory had CVs > 50%. Thus, compared to SRM915a, there was substantial improvement in the precision of measurements of the SRMSOLN sample. This is interesting, considering SRMSOLN was prepared from SRM915a. However, this improvement can likely be attributed to two factors. First, because this sample was spiked, the concentrations of many of these elements were much higher (relatively) in the SRMSOLN sample than in solutions of SRM915a prepared by the participants, promoting better precision. Second, the SRMSOLN sample was prepared as a single large sample by the organizer and aliquots were sent to the participants. Thus, the variability caused by sample preparation procedures was removed from the measurements. A similar result was observed in an intercomparison exercise conducted by NOAA and refereed by NRC Canada in the mid 1980s.

Precision results for RMRS1 are given in Table 5. Results for the “accuracy group” of elements (Mg, Ca, Mn, Cu and Sr) were mixed. For Ca and Sr, all laboratories had CVs < 20%, with all laboratories except one within 10%. For Mg and Mn, all laboratories except two had CVs < 20%, but for Mn only five of 12 laboratories had CVs < 10%; for Mg, eight of 11 laboratories had CVs < 10%, but one laboratory had a CV > 50%. And for Cu, only four of seven laboratories had CVs < 20%, while only two of seven were < 10%. Results for the other elements were also mixed. For Na, K and

Ba almost all laboratories had CVs < 20%; most were < 10%. For Li, Co and Ni most laboratories had CVs < 20%, although for Co one laboratory had a CV > 50%. And for Cr, Zn, Rb and Pb few laboratories had CVs < 20%; several CVs were > 50%. These results are also consistent with the trend toward better precision with increasing concentration. In fish otoliths, elemental concentrations may vary with species and geographic location, but certain elements consistently appear to occur in high abundance, while others occur at low abundance (Table 6, Zdanowicz, unpublished data). Thus, higher precision was generally obtained for the more abundant elements (Na, Mg, K, Ca and Sr) than for those occurring in otoliths at low ug/g levels.

As above, precision does not appear to be related to the methods used. A more significant result is the increase in precision obtained in the measurement of the solution sample relative to the precision in the measurement of the powder samples.

Agreement Among Laboratories

In order to gauge the extent of agreement between laboratories, two measures were used, z-scores and p-scores, listed in Appendix D. Z-scores are related to accuracy; a z-score is the number of standard deviations from some accepted value a laboratory's mean value is. P-scores are related to precision; a p-score is the number of multiples of some accepted value a laboratory's standard deviation is. Results were assessed by element and by laboratory.

Z-Scores

Z-scores were calculated for each element in each sample for each laboratory. Then, for each element in each sample, means (AVZ) and standard deviations (SDZ) of the z-scores were computed and an interval calculated which ranged from (AVZ-SDZ) to (AVZ+SDZ). Z-scores that fell within those intervals were designated "in agreement."

SUMMARY BY ELEMENT (Table 7). For each sample, by comparing z-scores for an element, agreement among laboratories in the measurement of that element could be assessed. It was expressed as Percentage of Laboratories in Measurement Agreement (%LMA), computed as 100 x number of laboratories with z-scores in agreement / the total number of laboratories for which z-scores were calculated. For example, z-scores were calculated for Mg in SRM915a for nine laboratories. Z-scores for eight laboratories were in agreement, as defined above. Thus, for that sample, %LMA was 89% (Mg results were in agreement 89% of the time).

- for the SRM915a sample, z-scores were computed for 11 elements. %LMA ranged from 75% to 92%, and was 80% or higher for eight elements and 90% or higher for two elements.
- for the SRMSOLN sample, z-scores were computed for 15 elements. %LMA ranged from 50% to 92%, and was 80% or higher for nine elements and 90% or higher for three elements.
- for the RMRS1 sample, z-scores were computed for 15 elements. %LMA ranged from 71% to 89%, and was 80% or higher for 12 elements and 90% or higher for 0 elements.

SUMMARY BY LABORATORY (Table 8). For each sample, by examining z-scores for all elements measured by a laboratory, measurement performance of individual laboratories could be assessed. It was expressed as Percentage of Elements in Measurement Agreement (%EMA), and computed as 100 x number of elements with z-scores in agreement / the total number of elements measured by that laboratory for which z-scores were calculated. For example, Lab 1 measured 12 elements in SRMSOLN. Z-scores for two of those elements were in agreement, as defined above. Thus, for that sample, %EMA for Lab 1 was 17% (Lab 1 was in agreement with other laboratories 17% of the time).

- for the SRM915a sample, z-scores were computed for at least one element for 15 laboratories. %EMA ranged from 0% to 100%, and was 80% or higher for nine laboratories, and 90% or higher for eight laboratories.
- for the SRMSOLN sample, z-scores were computed for at least one element for 14 laboratories. %EMA ranged from 17% to 100%, and was 80% or higher for 10 laboratories, and 90% or higher for six laboratories.
- for the RMRS1 sample, z-scores were computed for at least one element for 16 laboratories. %EMA ranged from 20% to 100%, and was 80% or higher for 10 laboratories, and 90% or higher for nine laboratories.

On the whole, the extent of measurement agreement among laboratories was moderate. %LMA ranged from 50% to 92%, and was 80% or higher in 29 of 41 instances (71% of the time), and 90% or higher in five of 41 instances (12% of the time). %EMA ranged from 0 to 100%, and was 80% or higher in 29 of 45 instances (64% of the time), and 90% or higher in 23 of 45 instances (51% of the time).

P-Scores

As above, p-scores were calculated for each element in each sample for each laboratory. Then, for each element in each sample, means (AVP) and standard deviations (SDP) of the p-scores were computed and an interval calculated which ranged from (AVP-SDP) to (AVP+SDP). P-scores that fell within those intervals were designated "in agreement."

SUMMARY BY ELEMENT (Table 9). For each sample, by comparing p-scores for an element, agreement among laboratories in precision for that element could be assessed. It was expressed as Percentage of Laboratories in Precision Agreement (%LPA), computed as 100 x number of laboratories with p-scores in agreement / the total number of laboratories for which p-scores were calculated. For example, p-scores were calculated for Mg in SRM915a for seven laboratories. P-scores for six laboratories were in agreement, as defined above. Thus, for that sample, %LPA was 86% (precision of Mg results were in agreement 86% of the time).

- for the SRM915a sample, p-scores were computed for 11 elements. %LPA ranged from 71% to 90%, and was 80% or higher for nine elements and 90% or higher for two elements.
- for the SRMSOLN sample, p-scores were computed for 15 elements. % LPA ranged from 75% to 90%, and was 80% or higher for 12 elements and 90% or

higher for one element.

- for the RMRS1 sample, p-scores were computed for 15 elements. % LPA ranged from 80% to 92%, and was 80% or higher for 15 elements and 90% or higher for two elements.

SUMMARY BY LABORATORY (Table 10). For each sample, by examining p-scores for all elements measured by a laboratory, precision performance of individual laboratories could be assessed. It was expressed as Percentage of Elements in Precision Agreement (%EPA), and computed as 100 x number of elements with p-scores in agreement / the total number of elements measured by that laboratory for which p-scores were calculated. For example, Lab 1 measured 12 elements in SRMSOLN. P-scores for three of those elements were in agreement, as defined above. Thus, for that sample, %EPA for Lab 1 was 25% (Lab 1 was in agreement with other laboratories 25% of the time).

- for the SRM915a sample, p-scores were computed for at least one element for 14 laboratories. %EPA ranged from 0% to 100%, and was 80% or higher for 12 laboratories, and 90% or higher for nine laboratories.
- for the SRMSOLN sample, p-scores were computed for at least one element for 11 laboratories. %EPA ranged from 25% to 100%, and was 80% or higher for 10 laboratories, and 90% or higher for eight laboratories.
- for the RMRS1 sample, p-scores were computed for at least one element for 15 laboratories. %EPA ranged from 25% to 100%, and was 80% or higher for 12 laboratories, and 90% or higher for eight laboratories.

On the whole, the extent of precision agreement among laboratories was greater than that observed for measurement agreement. %LPA ranged from 71% to 92%, and was 80% or higher in 36 of 41 instances (88% of the time), and 90% or higher in five of 41 instances (12% of the time). %EPA ranged from 0% to 100%, and was 80% or higher in 34 of 40 instances (85% of the time), and 90% or higher in 25 of 40 instances (63% of the time).

DISCUSSION

Three questions are of particular importance to participants in an intercomparison exercise. How accurate are my results? How good is my precision? How do my results compare with results from other labs?

Accuracy can be evaluated using %Recovery, as defined earlier. In this exercise, accuracy could be assessed for only five elements in two of the three samples (Tables 1 and 2). Results were not encouraging. Good accuracy was achieved only for Ca, the major component of the samples. This result is reassuring, however, since Ca is measured in many studies of otolith chemistry. Recoveries for the other four elements (Mg, Mn, Cu, and Sr), were widely scattered and generally poor.

SRM915a is not a particularly good reference material for otolith analyses. First of all, levels of Mg and Sr, two important elements in otolith studies, are much lower in SRM915a than in otoliths. Nevertheless, good accuracy for Mg and Sr in SRM915a would indicate that otolith analysts can measure these two elements at low levels in a

high Ca matrix, thus providing some suggestion that Mg and Sr measurements in otoliths might be done properly. Second, SRM915a is not a good matrix match for otoliths, which contain protein and other constituents not present in SRM915a. Finally, SRM915a contains few trace elements. Consideration was given to including limestone CRMs in the exercise, but they were rejected because they contain alumino-silicate phases. Alumino-silicates would have insured the presence of more trace elements for use in guaging accuracy, but would not have improved the matrix match. At the time of this exercise, there was no reference material available that was composed of otoliths, so SRM915a was, all things considered, the best reference material available. However, now that the Japanese otolith CRM is available, there will be little reason to use SRM915a as a reference material for otolith analyses in the future.

One method of evaluating precision is by using %CV, as defined earlier. In general, precision results were much better than accuracy results, although there is ample room for improvement. Best precision was obtained for Ca in all three samples (Tables 3-5). For elements of high abundance in otoliths (Na, K and Sr), %CVs were generally good (< 10%). However, for elements of low (Mg) or trace concentrations in otoliths (Li, Cr, Mn, Co, Ni, Cu, Zn, As, Rb, Cd, Ba and Pb), %CVs ranged from 1-2% to almost 200% in the two powder samples (%CVs were lower in the SRMSOLN sample). For Mg and Ba, two elements important in otolith research, precision was generally not good for SRM915a (containing trace levels of these elements), but was much improved for RMRS1, an otolith powder which contains higher levels. This result, too, is encouraging and suggests that otolith analysts generally achieve acceptable precision in measuring Mg and Ba in otolith samples.

Finally, regarding comparability of results, the main objective of this exercise was to provide the participants with a basis for direct comparison of their results with those of other laboratories. This can be accomplished by simple inspection of the data tables and plots. This inspection reveals that in many cases, there was considerable agreement among laboratories in their measurement results.

An attempt was also made to summarize the extent of agreement among participating laboratories in their measurement and precision results in a concise form, so z-scores and p-scores were used.

Z-scores are commonly used as an indicator of accuracy of results. However, their "goodness" as an indicator depends on several factors:

a) well characterized samples of known composition are analyzed

b) a "true" or statistically derived consensus value is used for X

c) the target value used for S is meaningfully related to X - typically X/10 for trace level elements and X/20 for percent level elements, or S is related to a confidence interval around X

d) the exercise produces a well behaved data set - one where the vast majority of laboratories submit quantitative results of high accuracy and precision

Under these circumstances, z-scores will scatter around zero with most values ranging between -2 and +2, and they will be good indicators of accuracy.

In this exercise, z-scores are not good indicators of accuracy. Other measures show that most results submitted by participants were not very accurate or precise, yet most z-scores ranged between -2 and +2. This is because the value used for X was the overall mean (of all Quantitative data, including obvious outliers) and the value used for

S was the standard deviation of that overall mean. As stated earlier, no attempt was made to assign consensus or "correct" values to any analyte. The main consequence of this was that there was no objective rationale for excluding any data from consideration, even obvious outliers. The inclusion of outliers in X and S caused their values to be excessively large and the values of the z-scores to be lower than they otherwise would have been (in the presence of large outliers, S increases much more rapidly than X). Consequently, the z-scores computed here are deceptively low.

Nevertheless (and accuracy notwithstanding), by computing "agreement intervals," they can be used to show the extent of agreement among laboratories. As already mentioned, there was only moderate agreement among participants. In only 29 of 41 instances were 80% of the laboratories in agreement in the measurement of specific elements (Table 7), and in only five of 41 instances were 90% of the laboratories in agreement. For elements important in otolith studies (Mg, Ca, Sr and Ba), agreement among laboratories averaged 81%. In a mature area of analytical chemistry, one where the great majority of practitioners are highly experienced in the subject analyses, one would expect 90% agreement among laboratories much more often than in five of 41 instances. With respect to individual laboratory performance (Table 8), there were 29 instances out of 45 where 80% of measurement results submitted by an individual laboratory agreed with results submitted by all laboratories, and there were 23 instances out of 45 where 90% of measurement results submitted by an individual laboratory agreed with results submitted by all laboratories. This suggests that a laboratory either was in agreement with the other laboratories, or it was not. For example, results submitted by Labs 2 and 10 were generally not in agreement with results submitted by the other laboratories. Labs 2 and 10 conducted solid state analyses using microprobe methods. Thus, not surprisingly, there appear to be significant differences between microprobe and dissolution methods.

Using the same approach for precision, p-scores were used to summarize the results. As with z-scores, the "goodness" of p-scores as indicators of precision depends on the same factors as described above. And, as above, those conditions were not met in this exercise, so p-scores, too, are deceptively low. However, "agreement intervals" can be used to show the extent of agreement between laboratories. As already mentioned, on the whole, the extent of precision agreement between laboratories was greater than that observed for measurement agreement. In 36 of 41 instances, 80% of the laboratories were in agreement, although in only five of 41 instances were 90% of the laboratories in agreement (Table 9). For elements important in otolith studies (Mg, Ca, Sr and Ba), agreement among laboratories averaged 86%. The incidence of 90% agreement will most likely improve as this area of measurement matures. Regarding individual laboratory performance (Table 10), there were 34 instances out of 40 where 80% of precision results submitted by an individual laboratory agreed with results submitted by all laboratories, and there were 25 instances out of 40 where 90% of precision results submitted by an individual laboratory agreed with results submitted by all laboratories. As above, this suggests that a laboratory either was in agreement with the other laboratories, or it was not. For example, results submitted by Lab 1 were generally not in agreement with results submitted by the other laboratories.

CONCLUSIONS

The main objective of this exercise was to provide participants with a basis for direct comparison of their results with those of other laboratories. On that basis, the exercise was a success. Data tables and plots contained in this report can be used to achieve that end. Another valuable goal would have been to evaluate the accuracy and precision of the participants' results and provide summaries of these characteristics. In these areas, the exercise was, at best, only partially successful. Use of samples of wholly or largely unknown composition, combined with the lack of consensus values for the analytes, severely hindered the evaluation of accuracy. For those analytes for which concentration values were available, accuracy was generally poor. Precision, in contrast, could be assessed for all the analytes. Results, however, were only slightly better. Levels of agreement among laboratories observed in this exercise spanned a fairly broad range. This is normally not considered a good situation, since comparability of results is essential if different studies are to be compared. Thus, there is considerable room for improvement in this area of analytical chemistry.

Results of this exercise reflect the fact that this is not a mature area of analytical chemistry - one where the great majority of practitioners are highly experienced in the subject analyses. Currently, the number of analysts in this field is relatively small, partially accounting for the small number of participants in this exercise. However, that number is growing, and as it increases, so will the need increase for methods of assessing the accuracy, precision and comparability of data generated at different laboratories using different analytical methods. Intercomparison exercises will partially fill that need, but acceptable control over the quality of otolith analyses will not be achieved without the use of suitable otolith CRMs. One such CRM now exists and others surely will follow. However, more intercomparison exercises are also needed that employ well characterized samples of known composition, so that "true" or consensus values for analytes of interest are available, allowing meaningful assessments of accuracy and precision to be made.

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Table 1. Percent Recovery for SRM915a.

LAB #	Mg	Ca	Mn	Cu	Sr
1	---	---	---	---	---
2	---	---	4556	---	524
3	---	93	128	---	---
4	260	99	133	86	157
5	---	---	---	---	163
6	---	104	---	---	---
7	383	---	129	311	---
8	3684	96	138	45	231
9	247	98	75	211	147
10	---	100	---	---	---
11	1870	---	583	347	---
12	---	101	73	72	163
13	16258	---	180	130	---
14	830	---	119	97	170
15	366	96	90	74	159
16	104	99	112	120	---
# Laboratories	9	9	12	10	8
# with % Recovery within 10% of Actual	1	9	1	1	0
# with % Recovery within 20% of Actual	1	9	3	3	0
# with % Recovery > 150% of Actual	8	0	3	3	7

Table 2. Percent Recovery for SRMSOLN.

LAB #	Mg	Ca	Mn	Cu	Sr
1	---	94	1154	258	177
2	---	---	---	---	---
3	---	98	103	---	---
4	89	89	103	70	116
5	132	---	---	---	150
6	1188	99	145	---	152
7	125	---	74	106	---
8	88	94	93	97	144
9	125	96	103	144	70
10	---	---	---	---	---
11	1408	---	221	170	---
12	---	93	107	87	126
13	2076	117	151	131	---
14	164	---	78	69	94
15	146	100	95	89	129
16	115	99	97	106	---
# Laboratories	11	10	13	11	9
# with % Recovery within 10% of Actual	0	8	7	3	1
# with % Recovery within 20% of Actual	3	10	7	5	2
# with % Recovery > 150% of Actual	4	0	3	2	3

Table 3. Percent CV for SRM915a.

LAB #	Mg	Ca	Cr	Mn	Co	Ni	Cu	Zn	Sr	Ba	Pb
1	---	---	---	---	---	---	---	---	---	---	---
2	---	---	---	15	---	48	---	---	33	87	---
3	---	4	---	28	---	---	---	---	---	8	---
4	---	2	---	---	20	---	---	---	4	81	---
5	---	---	---	---	---	---	---	---	3	---	---
6	---	1	---	---	---	---	---	---	---	---	---
7	68	---	---	8	11	---	5	---	---	---	26
8	4	7	---	13	6	---	6	22	23	7	---
9	23	5	38	3	7	9	4	6	8	14	22
10	---	0	---	---	---	---	---	---	---	---	---
11	---	---	---	---	---	---	---	---	---	---	---
12	---	2	---	8	4	13	5	19	3	31	2
13	9	---	12	4	4	5	3	7	---	59	1
14	57	---	47	16	2	2	45	113	2	8	14
15	7	3	---	10	12	---	0	---	6	34	11
16	8	1	63	14	14	12	21	123	---	7	33
# Laboratories	7	9	4	10	9	6	8	6	8	10	7
# with % CV < 10%	4	9	0	5	6	3	6	2	6	4	2
# with % CV < 20%	4	9	1	9	9	5	6	3	6	5	4
# with % CV > 50%	2	0	1	0	0	0	0	0	0	3	0

Table 4. Percent CV for SRMSOLN.

LAB #	Li	Mg	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb
1	---	---	6	24	33	8	13	7	9	96	51	7	9	---	5
2	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
3	2	---	1	---	2	---	---	---	---	---	---	---	---	1	---
4	2	---	2	21	10	14	---	---	---	---	0	10	---	4	1
5	---	3	---	---	---	---	---	---	---	---	---	2	---	---	---
6	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
7	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
8	---	6	1	---	3	1	---	20	6	---	---	4	---	1	---
9	3	10	1	2	6	2	41	7	9	3	2	3	---	2	7
10	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
11	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
12	---	---	1	---	3	4	3	4	5	---	---	4	2	5	4
13	---	18	0	7	5	7	11	7	9	8	8	---	5	2	0
14	4	4	---	3	2	2	1	10	5	4	1	1	---	1	1
15	14	3	2	4	2	4	---	2	3	---	1	1	2	2	1
16	1	4	1	3	1	1	4	1	18	3	0	---	---	1	0
# Laboratories	6	7	9	7	10	9	6	8	8	5	7	8	4	9	8
# with % CV < 10%	6	6	9	5	9	8	3	7	7	4	6	8	4	9	8
# with % CV < 20%	6	7	9	5	9	9	5	8	8	4	6	8	4	9	8
# with % CV > 50%	0	0	0	0	0	0	0	0	0	1	1	0	0	0	0

Table 5. Percent CV for RMRS1.

LAB #	Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	Rb	Sr	Ba	Pb
1	---	---	15	---	11	---	35	14	40	45	44	25	12	20	---
2	---	---	---	---	---	---	22	60	41	---	107	25	3	9	---
3	3	---	---	---	4	---	12	---	---	---	---	---	5	6	---
4	32	6	7	4	5	43	2	25	---	---	3	46	1	2	---
5	---	2	6	---	---	---	---	---	---	---	---	---	4	4	---
6	---	6	64	6	1	---	---	---	---	---	---	---	2	5	---
7	---	0	1	---	---	---	6	7	---	7	---	---	1	---	19
8	---	---	30	---	8	---	14	7	---	7	10	---	5	15	---
9	37	4	5	4	1	17	19	13	47	41	160	140	3	6	195
10	---	23	---	23	1	---	---	---	---	---	---	---	8	---	---
11	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
12	---	---	---	---	2	---	8	7	15	16	4	---	2	3	24
13	---	---	10	---	---	24	13	11	10	22	33	4	0	2	21
14	9	---	8	---	---	30	11	3	5	---	64	17	3	3	141
15	---	---	2	1	3	---	0	0	---	---	---	---	3	4	---
16	12	1	5	2	1	70	3	7	---	17	41	16	1	2	29
# Laboratories	5	7	11	6	10	5	12	11	6	7	9	7	15	13	6
# with % CV < 10%	2	6	8	5	9	0	5	6	1	2	3	1	14	11	0
# with % CV < 20%	3	6	9	5	10	1	10	8	3	4	3	3	15	13	1
# with % CV > 50%	0	0	1	0	0	1	0	1	0	0	3	1	0	0	2

Table 6. Typical concentrations of selected elements in otoliths of four pelagic species (blackfin tuna, bluefin tuna, bluefish, and cod).

ELEMENT	RANGE (ug/g)
Li	0.1 - 0.6
Na	2000 - 4000
Mg	10 - 60
K	300 - 1000
Ca (%)	35 - 39
Mn	0.5 - 5
Sr	1200 - 2800
Ba	0.6 - 4

Table 7. Measurement agreement among laboratories - summary by element.

	Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
SRM915a																		
# Labs	---	---	9	---	9	6	12	9	7	10	7	---	---	8	---	10	8	---
# Within Interval	---	---	8	---	7	5	11	7	6	8	6	---	---	7	---	9	6	---
% LMA	---	---	89	---	78	83	92	78	86	80	86	---	---	88	---	90	75	---
SRMSOLN																		
# Labs	6	---	11	---	10	8	13	10	7	11	9	5	7	9	5	10	9	---
# Within Interval	3	---	9	---	8	6	12	9	6	10	8	4	5	6	3	8	7	---
% LMA	50	---	82	---	80	75	92	90	86	91	89	80	71	67	60	80	78	---
RMRS1																		
# Labs	5	8	12	7	10	5	13	11	7	9	9	---	7	15	---	13	6	---
# Within Interval	4	6	10	5	8	4	11	9	6	8	8	---	6	12	---	10	5	---
% LMA	80	75	83	71	80	80	85	82	86	89	89	---	86	80	---	77	83	---

%LMA = 100 x # Within Interval / # Labs - see text.

Table 8. Measurement agreement among elements - summary by laboratory.

LAB #	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
SRM915a																
# Elements	0	4	3	10	1	1	5	8	11	1	5	9	9	10	8	10
# Within Interval	---	1	2	9	1	0	3	7	10	1	3	9	7	10	8	9
% EMA	---	25	67	90	100	0	60	88	91	100	60	100	78	100	100	90
SRMSOLN																
# Elements	12	0	4	12	2	5	6	8	14	0	5	10	13	13	13	13
# Within Interval	2	---	3	11	2	4	6	8	12	---	3	10	8	11	11	13
% EMA	17	---	75	92	100	80	100	100	86	---	60	100	62	85	85	100
RMRS1																
# Elements	10	7	5	12	4	6	7	8	15	4	6	9	11	12	7	14
# Within Interval	2	3	5	12	4	5	7	6	14	2	3	9	10	9	7	14
% EMA	20	43	100	100	100	83	100	75	93	50	50	100	91	75	100	100

%EMA = 100 x # Within Interval / # Elements - see text.

Table 9. Precision agreement among laboratories - summary by element.

	Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
SRM915a																		
# Labs	---	---	7	---	9	4	10	9	6	8	6	---	---	8	---	10	7	---
# Within Interval	---	---	6	---	8	3	9	8	5	7	5	---	---	7	---	9	5	---
% LPA	---	---	86	---	89	75	90	89	83	88	83	---	---	88	---	90	71	---
SRMSOLN																		
# Labs	6	---	7	---	9	7	10	9	6	8	8	5	7	8	4	9	8	---
# Within Interval	5	---	6	---	8	6	9	8	5	7	6	4	6	6	3	8	7	---
% LPA	83	---	86	---	89	86	90	89	83	88	75	80	86	75	75	89	88	---
RMRS1																		
# Labs	5	7	11	6	10	5	12	11	6	7	9	---	7	15	---	13	6	---
# Within Interval	4	6	9	5	8	4	10	10	5	6	8	---	6	12	---	12	5	---
% LPA	80	86	82	83	80	80	83	91	83	86	89	---	86	80	---	92	83	---

%LPA = 100 x # Within Interval / # Labs - see text.

Table 10. Precision agreement among elements - summary by laboratory.

LAB #	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
SRM915a																
# Elements	0	4	3	4	1	1	5	8	11	1	0	9	9	10	8	10
# Within Interval	---	0	3	4	1	1	3	7	11	1	---	9	8	9	8	8
% EPA	---	0	100	100	100	100	60	88	100	100	---	100	89	90	100	80
SRMSOLN																
# Elements	12	0	4	9	2	0	0	8	14	0	0	10	13	13	13	13
# Within Interval	3	---	4	8	2	---	---	7	13	---	---	9	12	13	12	12
% EPA	25	---	100	89	100	---	---	88	93	---	---	90	92	100	92	92
RMRS1																
# Elements	10	7	5	12	4	6	7	8	15	4	0	9	11	11	7	14
# Within Interval	3	4	5	12	4	5	7	7	14	1	---	9	9	9	7	14
% EPA	30	57	100	100	100	83	100	88	93	25	---	100	82	82	100	100

%EPA = 100 x # Within Interval / # Elements - see text.

APPENDIX A

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APPENDIX B

DATA TABLES

Lithium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3	0.323	0.329	0.315	0.309	0.331	0.321	0.009	2.9
4	0.32	0.51	< 2			0.42	0.13	32.4
5								
6								
7								
8								
9	0.52	0.71	0.46	0.37	0.26	0.46	0.17	36.7
10								
11								
12								
13								
14	0.4	0.4	0.3	0.3	0.3	0.3	0.0	9.4
15								
16	0.312	0.346	0.257	0.317	0.278	0.302	0.035	11.6

SRM915a (ug / g) Ref : < 0.05

LAB #						Mean	SD	%CV
1								
2								
3								
4	< 0.2							
5								
6								
7								
8								
9	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01			
10								
11								
12								
13								
14	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2			
15								
16	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			

SRMSOLN (ng / ml) Actual : < 3.52

LAB #						Mean	SD	%CV
1								
2								
3	3.7465	3.7068	3.7068	3.7601	3.5513	3.6943	0.0834	2.3
4	2.9	3				3.0	0.1	2.4
5								
6								
7								
8								
9	3.85	3.61	3.67	3.66	3.78	3.71	0.10	2.6
10								
11								
12								
13								
14	2.8	2.8	2.6	2.6	2.6	2.7	0.1	3.7
15	3.1	3.4	2.3	2.9	2.7	2.9	0.4	14.4
16	3.27	3.21	3.24	3.26	3.31	3.26	0.04	1.1

Beryllium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	0.247	0.185	0.251	0.205	0.209	0.219	0.028	12.9
13								
14								
15								
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	0.365	0.267	0.286	0.340	0.339	0.320	0.041	12.9
13								
14								
15								
16								

SRMSOLN (ng / ml) Actual : 3.51

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	3.850	3.501	3.615	3.876	4.325	3.833	0.317	8.3
13								
14								
15								
16								

Boron

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	8.059	5.339	5.789	5.652	5.294	6.027	1.155	19.2
13								
14								
15								
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	14.281	15.570	21.557	19.000	22.134	18.508	3.507	18.9
13								
14								
15								
16								

SRMSOLN (ng / ml)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12	10.128	10.008	9.499	10.386	11.572	10.319	0.771	7.5
13								
14								
15								
16								

Sodium

RMRS1	(ug / g)							
LAB #						Mean	SD	%CV
1								
2								
3								
4	2560	2860	2800			2740	159	5.8
5	2703	2750	2738	2847	2851	2778	67	2.4
6	2360	2360	2500	2690	2560	2494	140	5.6
7	2464	2458	2472	2479		2468	9	0.4
8								
9	2855	2637	2854	2905	2843	2819	104	3.7
10	1784.242	1769.404	1097.995	1187.022	1339.109	1435.55	323.29	22.5
11	1493							
12								
13								
14								
15								
16	2609	2647	2666	2603	2662	2637	29	1.1

SRM915a	(ug / g)		Ref :	0.45				
LAB #						Mean	SD	%CV
1								
2								
3								
4	< 40							
5								
6								
7	< 5	< 5	< 5	< 5				
8								
9	< 1	< 1	< 1	< 1	< 1			
10	< 109.9	< 109.9	< 109.9	< 109.9	< 109.9			
11	6.4							
12								
13								
14								
15								
16								

SRMSOLN	(ng / ml)		Actual :	3.95				
LAB #						Mean	SD	%CV
1								
2								
3								
4	< 0.02							
5	13.58	13.07	14.08	13.32	14.29	13.90	0.51	3.7
6	200.3							
7	< 5							
8								
9	< 20	< 20	< 20	< 20	< 20			
10								
11	12.4							
12								
13								
14								
15								
16								

Magnesium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	36	29	25	30	35	31	5	14.6
2								
3								
4	29	32	< 100			31	2	7.0
5	19.25	20.12	22.06	22.22	21.74	21.08	1.32	6.3
6	32.4		20.2	46.6	6.8	26.5	17.0	64.1
7	28.3	28.2	27.7	28.2		28.1	0.3	1.0
8	9.8296	14.3291	11.1990	20.2632	12.3593	13.5960	4.0758	30.0
9	23	21	22	24	22	22	1	5.1
10								
11	203							
12								
13	137.949	174.661	155.846	156.609		156.266	14.991	9.6
14	30.5	31.0	31.0	36.8	31.9	32.2	2.6	8.1
15	23	23	23	22		23	1	2.2
16	23.8	25.3	23.3	23.3	22.2	23.6	1.1	4.8

SRM915a (ug / g) Ref : 1.0

LAB #						Mean	SD	%CV
1								
2								
3								
4	2.6	< 20				2.6		
5								
6								
7	2.4	7.7	2.7	2.5		3.8	2.6	67.6
8	37.9841	36.5248	38.5487	35.4739	35.6755	36.8414	1.3739	3.7
9	2.75	3.06	2.63	2.30	1.60	2.47	0.56	22.5
10								
11	18.7							
12								
13	151.998	173.169				162.584	14.970	9.2
14	6.4	16.8	6.2	5.8	6.3	8.3	4.7	57.0
15	3.4	3.6	4.0	3.5	3.8	3.7	0.2	6.6
16	1.06	0.94	1.06	1.17	0.98	1.04	0.09	8.4

SRMSOLN (ng / ml) Actual : 4.5

LAB #						Mean	SD	%CV
1								
2								
3								
4	4	< 8				4		
5	5.70	6.10	5.90	5.80	6.00	5.90	0.16	2.7
6	53.2							
7	5.6							
8	4.09	4.00	4.29	3.64	3.79	3.96	0.25	6.4
9	6.4	5.0	5.7	5.7	5.1	5.6	0.6	10.1
10								
11	63.1							
12								
13	81	105				93	17	18.2
14	7.7	7.4	7.3	7.1	7.1	7.3	0.3	3.8
15	6.8	6.6	6.7	6.3	6.4	6.6	0.2	3.2
16	5.41	5.06	4.87	5.16	5.27	5.16	0.20	3.9

Aluminum

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8	7.8720	10.1937	8.6834	8.7006	9.4935	8.9886	0.8846	9.8
9								
10								
11								
12	1.615	1.640	2.021	1.820	2.214	1.862	0.255	13.7
13								
14								
15								
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8	10.4450	9.6611	9.3092	9.2281	9.6501	9.6587	0.4811	5.0
9								
10								
11								
12	0.611	0.884	1.182	1.015	1.123	0.963	0.227	23.6
13								
14								
15								
16								

SRMSOLN (ng / ml) Actual : 3.48

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8	4.15	4.18	3.90	4.84		4.27	0.40	9.4
9								
10								
11								
12	3.645	4.091	3.575	3.907	4.905	4.024	0.534	13.3
13								
14								
15	4.4	4.3	4.3	4.0	4.1	4.2	0.2	3.9
16								

Silicon

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	1320	1390	1285	1430		1356	66	4.8
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	1580	1770	1773	1603	1608	1667	96	5.8
16								

SRMSOLN (ng / ml)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								

Potassium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	328	341	314			328	14	4.1
5								
6	337	323	360	366	324	342	20	5.9
7								
8								
9	409	369	393	399	388	392	15	3.8
10	489.733	624.587	390.367	< 190.8	390.367	473.764	110.925	23.4
11	43							
12								
13								
14								
15	292	294	288	287		290	3	1.1
16	403	385	408	394	408	399	10	2.5

SRM915a (ug / g) Ref : < 0.4

LAB #						Mean	SD	%CV
1								
2								
3								
4	< 8							
5								
6								
7								
8								
9	< 1	< 1	< 1	< 1	< 1			
10	< 187.8	< 187.8	< 187.8	< 187.8	< 187.8			
11	3.9							
12								
13								
14								
15								
16								

SRMSOLN (ng / ml) Actual : < 3.93

LAB #						Mean	SD	%CV
1								
2								
3								
4	4							
5								
6	71.8							
7								
8								
9	< 20	< 20	< 20	< 20	< 20			
10								
11	11.2							
12								
13								
14								
15								
16								

Calcium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	400,000	330,000	300,000	380,000	360,000	354,000	39,749	11.2
2								
3	390,907	405,832	373,236	363,198	392,686	385,172	16,893	4.4
4	366,000	398,000	362,000			375,333	19,732	5.3
5								
6	393,000	388,000	383,000	388,000	386,000	387,600	3,647	0.9
7								
8	350,566	400,882	352,505	319,880	364,344	357,635	29,228	8.2
9	399,600	394,500	393,380	396,200	396,600	396,056	2,370	0.6
10	389,048	384,681	393,931	389,799	395,403	390,572	4,250	1.1
11								
12	387,845	397,408	406,060	384,946	385,907	392,433	9,085	2.3
13								
14								
15	370,000	380,000	360,000	360,000		367,500	9,574	2.6
16	374,610	383,190	376,760	381,040	372,470	377,614	4,445	1.2

SRM915a (ug / g) Ref : 400,000

LAB #						Mean	SD	%CV
1								
2								
3	367,144	360,921	356,171	379,994	390,131	370,872	13,992	3.8
4	401,000	389,000				395,000	8,485	2.1
5								
6	420,000	412,000	411,000	413,000	416,000	414,400	3,647	0.9
7								
8	421,443	355,785	401,030	370,706	375,333	384,859	26,155	6.8
9	390,800	374,100	379,500	412,400	412,300	393,820	17,957	4.6
10	400,321	398,906	401,343	398,584	401,329	400,097	1,307	0.3
11								
12	400,683	403,100	397,367	408,308	413,410	404,574	6,350	1.6
13								
14								
15	370,000	400,000	390,000	380,000	380,000	384,000	11,402	3.0
16	391,770	391,770	396,060	400,340	398,200	395,628	3,833	1.0

SRMSOLN (ng / ml) Actual : 400,003

LAB #						Mean	SD	%CV
1	377,952	393,542	350,910			374,134	21,571	5.8
2								
3	390,994	397,412	388,991	394,127	391,796	392,664	3,229	0.8
4	351,000	360,000				355,500	6,364	1.8
5								
6	397,920							
7								
8	373,592	379,390	377,366	373,284	377,153	376,157	2,633	0.7
9	393,000	386,000	384,000	383,000	381,000	385,400	4,615	1.2
10								
11								
12	368,202	373,927	371,584	370,764	371,757	371,247	2,065	0.6
13	466,440	466,391	468,457			467,096	1,179	0.3
14								
15	403,000	407,000	406,000	395,000	387,000	399,600	8,473	2.1
16	391,770	402,490	393,910	396,060	391,770	395,200	4,446	1.1

Vanadium

RMRS1 (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRM915a (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRMSOLN (ng / ml) Actual : 3.47

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	3.3	3.4	3.3	3.2	3.2	3.3	0.1	2.6
16								

Chromium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	0.08	0.15				0.12	0.05	43.0
5								
6								
7								
8								
9	0.19	0.19	0.19	0.23	0.14	0.19	0.03	17.1
10								
11								
12								
13	0.258	0.258	0.410	0.371		0.324	0.078	24.1
14	0.8	0.8	0.5	0.6	0.4	0.6	0.2	29.7
15								
16	0.097	0.035	0.121	0.024	0.034	0.062	0.044	70.2

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	0.16							
5								
6								
7								
8								
9	0.21	0.59	0.40	0.30	0.51	0.40	0.15	38.4
10								
11	2.9							
12								
13	0.336	0.398				0.367	0.044	11.9
14	0.7	0.3	0.3	0.3	0.3	0.4	0.2	46.6
15								
16	0.066	0.054	0.175	0.057	0.059	0.082	0.052	63.5

SRMSOLN (ng / ml) Actual : 3.50

LAB #						Mean	SD	%CV
1	11.3245	9.5690	6.8549			9.2494	2.2519	24.3
2								
3								
4	4.1	5.5				4.8	1.0	20.6
5								
6								
7								
8								
9	5.29	5.10	5.19	5.04	5.33	5.19	0.12	2.4
10								
11	2.3							
12								
13	6.84	7.46	6.54			6.95	0.47	6.8
14	3.8	3.9	3.8	3.7	3.7	3.8	0.1	2.5
15	4.7	4.8	4.5	4.5	4.4	4.6	0.2	3.6
16	4.74	4.67	4.55	4.75	4.91	4.72	0.13	2.8

Manganese

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	23	18	17	27	38	25	9	34.6
2	42	28	31			34	7	21.9
3	1.028	0.998	0.756	0.897	0.932	0.922	0.106	11.5
4	0.74	0.76				0.75	0.01	1.9
5	< 1	< 1	< 1	< 1	< 1			
6								
7	1.05	1.12	1.03	0.98		1.05	0.06	5.6
8	0.6952	0.8408	0.6232	0.6052	0.6531	0.6835	0.0943	13.8
9	0.78	1.05	0.83	0.78	0.63	0.81	0.15	18.6
10								
11	2							
12	0.534	0.482	0.526	0.462	0.563	0.514	0.041	7.9
13	0.897	1.211	1.138	1.197		1.111	0.146	13.1
14	0.67	0.71	0.74	0.86	0.67	0.73	0.08	10.9
15	0.6	0.6	0.6	0.6		0.6	0.0	0.0
16	0.641	0.681	0.670	0.677	0.688	0.671	0.018	2.7

SRM915a (ug / g) Ref : 0.6

LAB #						Mean	SD	%CV
1								
2	32	25	25			27	4	14.8
3	0.673	0.660	0.663	1.149	0.703	0.770	0.213	27.6
4	0.8							
5	< 1	< 1	< 1	< 1	< 1			
6								
7	0.74	0.86	0.76	0.73		0.77	0.06	7.7
8	0.9482	0.9226	0.7473	0.7151	0.8015	0.8269	0.1041	12.6
9	0.43	0.44	0.45	0.47	0.45	0.45	0.02	3.4
10								
11	3.5							
12	0.386	0.445	0.457	0.476	0.433	0.439	0.034	7.7
13	1.045	1.113				1.079	0.048	4.5
14	0.65	0.91	0.68	0.64	0.69	0.71	0.11	15.7
15	0.5	0.6	0.6	0.5	0.5	0.5	0.1	10.1
16	0.602	0.693	0.824	0.651	0.600	0.674	0.092	13.7

SRMSOLN (ng / ml) Actual : 4.1

LAB #						Mean	SD	%CV
1	63.0716	46.4464	31.7875			47.1018	15.6523	33.2
2								
3	4.2461	4.2105	4.2297	4.2213	4.0874	4.1990	0.0638	1.5
4	3.9	4.5				4.2	0.4	10.1
5	< 1	< 1	< 1	< 1	< 1			
6	5.9							
7	3							
8	3.96	3.80	3.86	3.69	3.73	3.81	0.11	2.8
9	4.68	4.05	4.11	4.02	4.15	4.20	0.27	6.4
10								
11	9.0							
12	4.321	4.313	4.280	4.291	4.622	4.365	0.144	3.3
13	6.05	6.53	5.92			6.17	0.32	5.2
14	3.26	3.19	3.17	3.12	3.15	3.18	0.05	1.6
15	3.9	3.9	3.9	3.9	3.7	3.9	0.1	2.3
16	3.95	3.99	3.93	3.99	3.92	3.95	0.03	0.8

Iron

RMRS1 (ug / g)

LAB #			Mean	SD	%CV
1					
2					
3					
4	2.4	2.1	2.3	0.2	9.4
5					
6					
7					
8					
9					
10	< 357.5				
11					
12					
13					
14					
15					
16					

SRM915a (ug / g) Ref : < 10

LAB #		Mean	SD	%CV
1				
2				
3				
4	5.4			
5				
6				
7				
8				
9				
10	< 320.2			
11				
12				
13				
14				
15				
16				

SRMSOLN (ng / ml) Actual : < 13.5

LAB #			Mean	SD	%CV
1					
2					
3					
4	6.3	< 7			
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					

Cobalt

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	7	6	5	7	7	6	1	14.0
2	9	4	3			5	3	60.3
3								
4	0.04	0.057				0.05	0.01	24.8
5								
6								
7	1.95	2.33	2.07	2.14		2.12	0.16	7.5
8	0.4102	0.4460	0.3983	0.3623	0.4006	0.4035	0.0300	7.4
9	2.32	2.61	2.41	2.20	1.83	2.28	0.29	12.7
10								
11								
12	0.304	0.336	0.316	0.308	0.358	0.325	0.023	6.9
13	1.46	1.89	1.62	1.70		1.67	0.18	10.7
14	1.14	1.13	1.18	1.20	1.11	1.15	0.04	3.2
15	0.4	0.4	0.4	0.4		0.4	0.0	0.0
16	0.333	0.357	0.383	0.384	0.397	0.371	0.025	6.9

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	0.3	0.4				0.4	0.1	20.2
5								
6								
7	1.43	1.81	1.72	1.48		1.61	0.18	11.4
8	0.4225	0.3857	0.4062	0.3707	0.3717	0.3913	0.0226	5.8
9	1.70	1.82	1.74	1.65	1.48	1.68	0.13	7.5
10								
11								
12	0.385	0.366	0.390	0.361	0.392	0.379	0.015	3.8
13	1.919	2.034				1.977	0.081	4.1
14	1.20	1.23	1.25	1.21	1.26	1.23	0.03	2.1
15	0.4	0.5	0.5	0.5	0.4	0.5	0.1	11.9
16	0.455	0.469	0.631	0.499	0.478	0.506	0.071	14.1

SRMSOLN (ng / ml) Actual : 3.46

LAB #						Mean	SD	%CV
1	14.3716	14.8576	12.6550			13.9614	1.1572	8.3
2								
3								
4	3.3	4				4	0	13.6
5								
6								
7	3.6							
8	3.49	3.44	3.47	3.42	3.40	3.45	0.04	1.1
9	6.43	6.21	6.38	6.25	6.49	6.35	0.12	1.9
10								
11								
12	3.678	3.680	3.576	3.882	3.973	3.758	0.164	4.4
13	4.72	5.27	4.61			4.87	0.35	7.3
14	3.60	3.52	3.51	3.44	3.42	3.50	0.07	2.0
15	4.0	3.9	3.9	3.8	3.6	3.8	0.2	3.9
16	3.77	3.74	3.78	3.87	3.80	3.79	0.05	1.3

Nickel

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	63	44	33	71	96	61	25	39.9
2	28	14	15			19	8	41.1
3								
4								
5								
6								
7								
8								
9	1.02	2.52	1.36	1.11	0.93	1.39	0.65	46.9
10								
11	0.6							
12	0.143	0.106	0.119	0.098	0.109	0.115	0.017	15.1
13	2.476	3.115	2.652	2.769		2.753	0.270	9.8
14	2.4	2.3	2.4	2.5	2.2	2.4	0.1	5.3
15								
16	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2	43	19	21			28	13	48.1
3								
4								
5								
6								
7								
8								
9	0.78	0.89	0.87	0.87	0.72	0.83	0.07	8.7
10								
11	1.0							
12	0.288	0.225	0.256	0.250	0.317	0.267	0.036	13.4
13	3.356	3.591				3.474	0.166	4.8
14	2.4	2.5	2.5	2.6	2.5	2.5	0.1	2.1
15								
16	0.687	0.616	0.839	0.691	0.757	0.718	0.084	11.7

SRMSOLN (ng / ml) Actual : 3.45

LAB #						Mean	SD	%CV
1	157.359	154.219	123.473			145.017	18.724	12.9
2								
3								
4								
5								
6								
7								
8								
9	10.56	5.09	4.90	4.87	4.98	6.08	2.50	41.2
10								
11	4.5							
12	3.764	3.742	3.663	3.779	4.000	3.789	0.126	3.3
13	3.69	4.27	3.47			3.81	0.41	10.8
14	4.9	4.9	4.9	4.8	4.8	4.9	0.0	0.9
15								
16	8.87	8.24	8.60	9.09	8.75	8.71	0.32	3.7

Copper

RMRS1	(ug / g)							
LAB #						Mean	SD	%CV
1	8	4	3	5	9	6	3	44.6
2								
3								
4	< 0.3	< 0.3						
5								
6								
7	2.44	2.87	2.60	2.49		2.60	0.19	7.4
8	0.9106	1.0626	1.0130	0.9285	1.0199	0.9869	0.0647	6.6
9	2.32	4.85	2.66	2.33	1.97	2.83	1.16	40.9
10								
11	2.6							
12	0.152	0.127	0.107	0.103	0.113	0.120	0.020	16.4
13	0.246	0.411	0.304	0.385		0.337	0.076	22.5
14	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			
15								
16	0.316	0.485	0.384	0.331	0.377	0.379	0.066	17.5

SRM915a	(ug / g)		Ref :	0.95				
LAB #						Mean	SD	%CV
1								
2								
3								
4	0.82							
5								
6								
7	2.85	3.16	2.92	2.87		2.95	0.14	4.8
8	0.4657	0.4214	0.4192	0.3926	0.4234	0.4244	0.0262	6.2
9	2.03	2.07	2.01	2.06	1.86	2.01	0.08	4.2
10								
11	3.3							
12	0.681	0.735	0.640	0.676	0.711	0.689	0.036	5.3
13	1.207	1.264				1.236	0.040	3.3
14	0.7	1.7	0.7	0.7	0.8	0.9	0.4	45.0
15	0.7	0.7	0.7	0.7	0.7	0.7	0.0	0.0
16	0.99	1.54	0.93	1.16	1.08	1.14	0.24	21.1

SRMSOLN	(ng / ml)		Actual :	4.42				
LAB #						Mean	SD	%CV
1	10.6344	12.1385	11.4861			11.4197	0.7542	6.6
2								
3								
4	3.1	< 4						
5								
6								
7	4.7							
8	3.98	3.95	3.92	3.82	5.84	4.30	0.86	20.1
9	7.08	6.44	6.20	5.95	6.13	6.36	0.44	6.9
10								
11	7.5							
12	3.646	3.884	3.780	3.868	4.038	3.843	0.144	3.7
13	5.44	6.25	5.62			5.77	0.43	7.4
14	3.6	3.0	2.9	2.9	2.9	3.1	0.3	10.3
15	4.0	4.0	4.0	3.9	3.8	3.9	0.1	2.3
16	4.74	4.66	4.61	4.73	4.68	4.68	0.05	1.2

Zinc

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	11	5	5	7	13	8	4	44.3
2	114	12	28			51	55	106.9
3								
4	2.5	2.6				2.6	0.1	2.8
5								
6								
7	< 5	< 5	< 5	< 5				
8	0.7998	0.7489	0.8484	0.9698	0.8353	0.8404	0.0819	9.8
9	0.85	6.18	0.70	0.18	0.14	1.61	2.57	159.8
10								
11								
12	0.297	0.302	0.322	0.305	0.323	0.310	0.012	3.8
13	0.722	1.533	0.948	1.372		1.144	0.374	32.7
14	< 0.5	< 0.5	0.5	1.4	< 0.5	0.9	0.6	63.5
15								
16	3.37	4.69	2.91	1.63	5.29	3.58	1.45	40.6

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	1.5							
5								
6								
7	< 5	< 5	< 5	< 5				
8	1.1107	0.7172	0.6710	0.7377	0.7612	0.7995	0.1771	22.1
9	0.26	0.26	0.28	0.30	0.28	0.28	0.02	6.1
10								
11								
12	0.717	1.025	0.676	0.731	0.929	0.816	0.153	18.7
13	1.679	1.856				1.768	0.125	7.1
14	0.8	4.9	1.1	0.5	0.9	1.6	1.8	113.2
15								
16	1.48	13.14	4.39	0.27	1.87	4.23	5.20	123.0

SRMSOLN (ng / ml) Actual : 3.49

LAB #						Mean	SD	%CV
1	16.8977	16.4182	14.1497			15.8219	1.4679	9.3
2								
3								
4	2.7	< 4						
5								
6								
7	< 5							
8	3.55	3.45	3.47	3.41	3.93	3.56	0.21	5.9
9	3.22	2.70	2.65	2.59	2.70	2.77	0.25	9.1
10								
11								
12	3.763	4.029	3.896	3.964	4.346	4.000	0.217	5.4
13	7.44	8.97	8.44			8.28	0.78	9.4
14	2.7	2.5	2.5	2.6	2.4	2.5	0.1	5.4
15	4.9	5.1	5.1	5.0	4.8	5.0	0.1	2.6
16	10.3	10.5	9.5	8.9	13.7	10.6	1.9	17.7

Gallium

RMRS1 (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRM915a (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRMSOLN (ng / ml) Actual : 3.49

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	3.4	3.4	3.4	3.4	3.2	3.4	0.1	2.7
16								

Arsenic

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9	0.38	0.56	0.60	0.65	0.60	0.56	0.10	18.3
10								
11								
12								
13	0.118	0.124	0.119	0.121		0.121	0.003	2.2
14	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			
15								
16	0.075	0.038	0.072	0.074	0.042	0.060	0.018	30.7

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2	20	16	16			17	2	13.3
3								
4								
5								
6								
7								
8								
9	0.48	0.49	0.46	0.45	0.37	0.45	0.05	10.3
10								
11								
12								
13	0.123	0.127				0.125	0.003	2.3
14	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			
15								
16	0.035	0.043	0.040	0.030	0.034	0.037	0.005	13.8

SRMSOLN (ng / ml) Actual : 3.48

LAB #						Mean	SD	%CV
1	0.8853	0.6966	< 0.0429			0.7910	0.1335	16.9
2								
3								
4								
5								
6								
7								
8								
9	2.95	2.95	3.02	3.00	3.15	3.01	0.08	2.8
10								
11								
12								
13	3.74	3.96	3.36			3.69	0.30	8.2
14	2.3	2.3	2.1	2.2	2.2	2.2	0.1	3.7
15								
16	2.78	2.74	2.93	2.82	2.87	2.83	0.08	2.7

Rubidium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	6	4	3	5	5	5	1	24.8
2	3	2	2			2	1	24.7
3								
4	0.026	0.051				0.039	0.018	45.9
5								
6								
7								
8								
9	0.02	0.21	0.03	0.02	0.02	0.06	0.08	139.9
10								
11								
12								
13	0.0237	0.0238	0.0217	0.0238		0.0233	0.0010	4.4
14	0.03	0.02	0.03	0.04	0.03	0.03	0.01	16.8
15								
16	0.0199	0.0273	0.0207	0.0195	0.0188	0.0213	0.0034	16.2

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2	2	2	2			2	0	0.0
3								
4	< 0.02							
5								
6								
7								
8								
9	< 0.010	< 0.010						
10								
11								
12								
13	0.0031	0.0041				0.0036	0.0007	19.6
14	< 0.010	0.10	< 0.010	< 0.010	< 0.010			
15								
16	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			

SRMSOLN (ng / ml) Actual : 3.49

LAB #						Mean	SD	%CV
1	3.0206	1.1387	1.5952			1.9182	0.9816	51.2
2								
3								
4	3.2	3.2				3.2	0.0	0.0
5								
6								
7								
8								
9	3.43	3.47	3.53	3.39	3.47	3.46	0.05	1.6
10								
11								
12								
13	3.41	3.38	2.94			3.24	0.26	8.1
14	2.69	2.67	2.62	2.63	2.61	2.64	0.04	1.4
15	3.7	3.8	3.7	3.7	3.7	3.7	0.0	1.2
16	3.22	3.24	3.25	3.24	3.25	3.24	0.01	0.4

Strontium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	1981	1627	1498	1857	1598	1712	200	11.7
2	2849	2707	2872			2809	89	3.2
3	2231.91	2339.64	2111.65	2059.77	2151.43	2178.88	109.68	5.0
4	1870	1910	1910			1897	23	1.2
5	1956	1822	1981	1961	1992	1942	69	3.5
6	1820	1830	1860	1890	1880	1856	30	1.6
7	1889	1845	1871	1884		1872	20	1.1
8	2165.0	1910.3	2065.4	1947.3	1959.3	2009.5	104.3	5.2
9	2058	1923	2069	2083	2054	2037	65	3.2
10	2054.98118	2021.15	2376.34	2257.94	2012.70	2144.62	163.72	7.6
11								
12	2328	2384	2404	2299	2309	2345	47	2.0
13	2033.731	2055.99	2038.21	2036.25		2041.05	10.13	0.5
14	2654	2619	2720	2809	2637	2688	77	2.9
15	1962	2091	1971	1993		2004	59	3.0
16	1988	2022	1995	2029	1974	2002	23	1.2

SRM915a (ug / g) Ref : 2.1

LAB #						Mean	SD	%CV
1								
2	15	10	8			11	4	32.8
3								
4	3.4	3.2				3.3	0.1	4.3
5	3.26	3.36	3.50	3.52	3.45	3.42	0.11	3.2
6								
7	< 1	< 1	< 1	< 1				
8	3.9255	5.75	4.3695	3.8845	6.3	4.8	1.1	22.9
9	2.90	2.80	3.00	3.30	3.40	3.08	0.26	8.4
10	< 219.1	< 219.1	< 219.1	< 219.1	< 219.1			
11								
12	3.405	3.480	3.410	3.539	3.279	3.423	0.098	2.8
13								
14	3.5	3.6	3.6	3.5	3.6	3.6	0.1	1.8
15	3.3	3.5	3.5	3.0	3.4	3.3	0.2	6.2
16	< 5.3	< 5.3	< 5.3	< 5.3	< 5.3			

SRMSOLN (ng / ml) Actual : 5.58

LAB #						Mean	SD	%CV
1	10.3383	10.1979	9.0164			9.8509	0.7261	7.4
2								
3								
4	6.9	6				6	1	9.9
5	8.50	8.60	8.29	8.24	8.19	8.36	0.18	2.1
6	8.5							
7	< 1							
8	8.15	7.97	8.30	8.27	7.54	8.05	0.31	3.9
9	4.10	3.90	3.80	3.80	3.80	3.88	0.13	3.4
10								
11								
12	6.900	6.941	6.847	6.932	7.567	7.038	0.298	4.2
13								
14	5.3	5.3	5.2	5.2	5.2	5.3	0.1	1.5
15	7.3	7.2	7.2	7.1	7.2	7.2	0.1	1.0
16								

Silver

RMRS1 (ug / g)

LAB #					Mean	SD	%CV
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13	0.0298	0.0354	0.0303	0.0329	0.0321	0.0026	8.1
14							
15							
16							

SRM915a (ug / g)

LAB #					Mean	SD	%CV
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13	0.0309	0.0322			0.0316	0.0009	2.9
14							
15							
16							

SRMSOLN (ng / ml) Actual : 3.48

LAB #					Mean	SD	%CV
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13	3.97	4.31	3.77		4.02	0.27	6.8
14							
15							
16							

Cadmium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7	< 0.1	< 0.1	< 0.1	< 0.1				
8								
9								
10								
11								
12	0.007	0.008	0.007	0.010	0.010	0.008	0.002	18.3
13	0.0212	0.0267	0.0249	0.0231		0.0240	0.0024	9.9
14	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010			
15								
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7	< 0.1	< 0.1	< 0.1	< 0.1				
8								
9								
10								
11								
12	0.013	0.017	0.011	0.013	0.022	0.015	0.005	29.6
13	0.0231	0.0242				0.0237	0.0008	3.3
14	2.92	2.95	2.92	2.93	2.95	2.93	0.02	0.6
15								
16								

SRMSOLN (ng / ml) Actual : 3.50

LAB #						Mean	SD	%CV
1	1.9745	2.3765	2.2311			2.1940	0.2035	9.3
2								
3								
4								
5								
6								
7	2.6							
8								
9								
10								
11								
12	3.069	3.079	2.958	3.064	3.172	3.068	0.076	2.5
13	4.09	4.41	3.98			4.16	0.22	5.4
14	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010			
15	3.5	3.5	3.6	3.5	3.4	3.5	0.1	2.0
16								

Cesium

RMRS1 (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRM915a (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRMSOLN (ng / ml) Actual : 3.50

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	3.8	3.8	3.9	3.8	3.8	3.8	0.0	1.2
16								

Barium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1	12	11	10	9	7	10	2	19.6
2	7	6	7			7	1	8.7
3	3.766	3.936	3.565	3.422	3.498	3.637	0.210	5.8
4	3.7	3.7	3.8			3.7	0.1	1.5
5	3.14	3.14	3.42	3.42	3.37	3.30	0.15	4.4
6	10.6	11.3				11.0	0.5	4.5
7								
8	0.1651	0.1867	0.1799	0.2383	0.1743	0.1888	0.0288	15.2
9	3.69	4.08	4.22	4.38	4.22	4.12	0.26	6.4
10								
11								
12	3.933	4.240	4.128	3.984	4.144	4.086	0.125	3.1
13	3.423	3.481	3.298	3.391		3.398	0.077	2.3
14	3.8	3.8	4.0	4.1	3.8	3.9	0.1	3.4
15	3.7	4.0	3.8	3.7		3.8	0.1	3.7
16	3.74	3.70	3.68	3.63	3.54	3.66	0.08	2.1

SRM915a (ug / g) Ref : << 10

LAB #						Mean	SD	%CV
1								
2	4	1	1			2	2	86.6
3	0.105	0.111	0.110	0.128	0.108	0.112	0.009	8.0
4	0.16	0.59				0.38	0.30	81.1
5	< 1.000	< 1.000	< 1.000	< 1.000	< 1.000			
6								
7								
8	4.8317	4.0476	4.3831	4.1662	4.2242	4.3305	0.3051	7.0
9	0.12	0.11	0.13	0.15	0.16	0.13	0.02	14.1
10								
11								
12	0.038	0.074	0.063	0.075	0.098	0.070	0.022	31.2
13	0.117	0.283				0.200	0.117	58.7
14	0.1	0.1	0.1	0.1	0.1	0.1	0.0	8.1
15	0.1	0.1	0.2	0.2	0.2	0.2	0.1	34.2
16	0.133	0.150	0.139	0.133	0.155	0.142	0.010	7.1

SRMSOLN (ng / ml) Actual : << 13.5

LAB #						Mean	SD	%CV
1								
2								
3	3.2119	3.2190	3.1820	3.1716	3.1271	3.1823	0.0367	1.2
4	3.5	3.3				3.4	0.1	4.2
5	< 1.000	< 1.000	< 1.000	< 1.000	< 1.000			
6	6.5							
7								
8	3.59	3.56	3.56	3.50	3.51	3.55	0.04	1.0
9	3.53	3.60	3.52	3.45	3.48	3.52	0.06	1.6
10								
11								
12	3.872	3.973	3.895	4.010	4.359	4.022	0.197	4.9
13	2.42	2.42	2.35			2.40	0.04	1.7
14	2.7	2.7	2.7	2.7	2.7	2.7	0.0	0.9
15	3.8	3.8	3.9	3.8	4.0	3.9	0.1	2.3
16	3.37	3.46	3.37	3.36	3.40	3.39	0.04	1.2

Thallium

RMRS1 (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRM915a (ug / g)

LAB #	Mean	SD	%CV
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

SRMSOLN (ng / ml) Actual : 3.53

LAB #						Mean	SD	%CV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15	3.5	3.5	3.7	3.6	3.6	3.6	0.1	2.3
16								

Lead

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	< 0.04	< 0.04	< 0.4					
5								
6								
7	0.17	0.22	0.15	0.22		0.19	0.04	18.7
8								
9	0.0191	0.9368	0.0785	0.0068	0.0031	0.2089	0.4081	195.4
10								
11								
12	0.035	0.028	0.027	0.046	0.028	0.033	0.008	24.0
13	0.0210	0.0300	0.0201	0.0291		0.0251	0.0052	20.8
14	< 0.05	< 0.05	0.06	< 0.05	46.53	23.30	32.86	141.1
15								
16	0.0368	0.0327	0.0156	0.0282	0.0368	0.0300	0.0088	29.3

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	0.29							
5								
6								
7	0.66	0.47	0.61	0.36		0.53	0.14	26.0
8								
9	0.25	0.23	0.28	0.36	0.38	0.30	0.07	22.2
10								
11								
12	0.370	0.380	0.375	0.381	0.366	0.374	0.006	1.7
13	0.342	0.335				0.339	0.005	1.5
14	0.34	0.40	0.47	0.35	0.36	0.39	0.05	13.6
15	0.5	0.4	0.4	0.4	0.4	0.4	0.0	10.6
16	0.340	0.396	0.676	0.352	0.360	0.425	0.142	33.4

SRMSOLN (ng / ml) Actual : 3.50

LAB #						Mean	SD	%CV
1	3.6009	3.5034	3.2497			3.4513	0.1813	5.3
2								
3								
4	3.02	3.06				3.04	0.03	0.9
5								
6								
7	3							
8								
9	4.00	4.04	3.64	3.46	3.50	3.73	0.27	7.3
10								
11								
12	3.714	3.776	3.698	3.756	4.072	3.803	0.153	4.0
13	2.77	2.77	2.76			2.77	0.01	0.2
14	3.15	3.11	3.09	3.07	3.08	3.10	0.03	1.1
15	3.9	4.0	4.0	4.0	4.0	4.0	0.0	1.1
16	3.56	3.57	3.57	3.56	3.54	3.56	0.01	0.4

Uranium

RMRS1 (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	< 0.001	< 0.001						
5								
6								
7	< 0.1	< 0.1	< 0.1	< 0.1				
8								
9								
10								
11								
12								
13	0.0367	0.0363	0.0316	0.0350		0.0349	0.0023	6.6
14								
15								
16								

SRM915a (ug / g)

LAB #						Mean	SD	%CV
1								
2								
3								
4	< 0.001							
5								
6								
7	< 0.1	< 0.1	< 0.1	< 0.1				
8								
9								
10								
11								
12								
13	0.0314	0.0365				0.0340	0.0036	10.6
14								
15								
16								

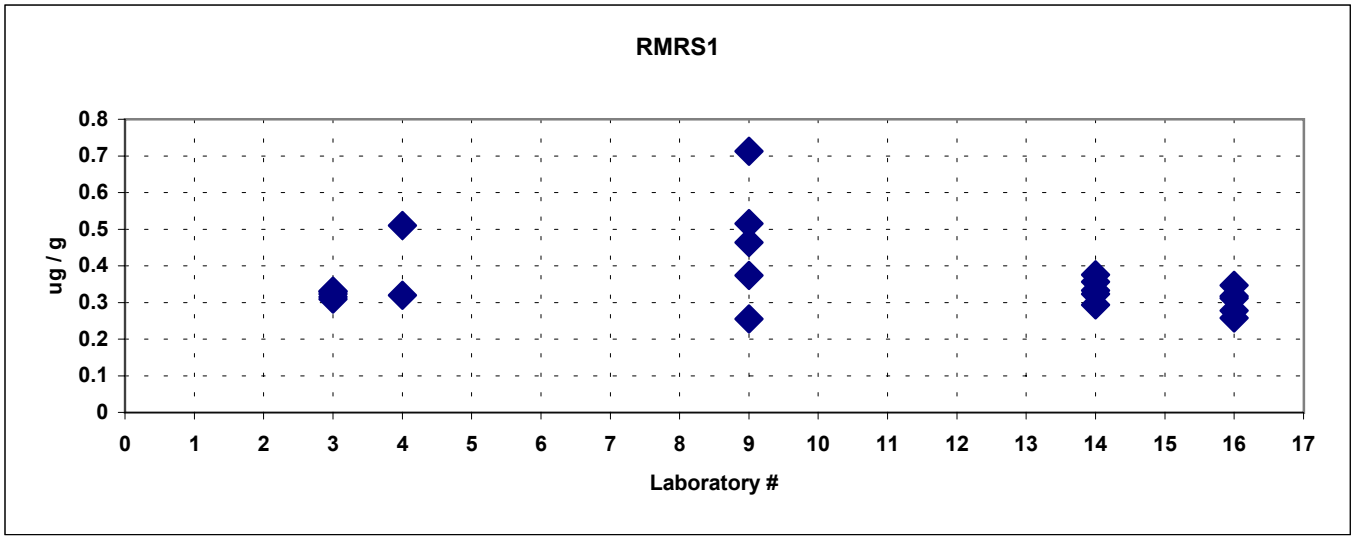
SRMSOLN (ng / ml) Actual : 3.52

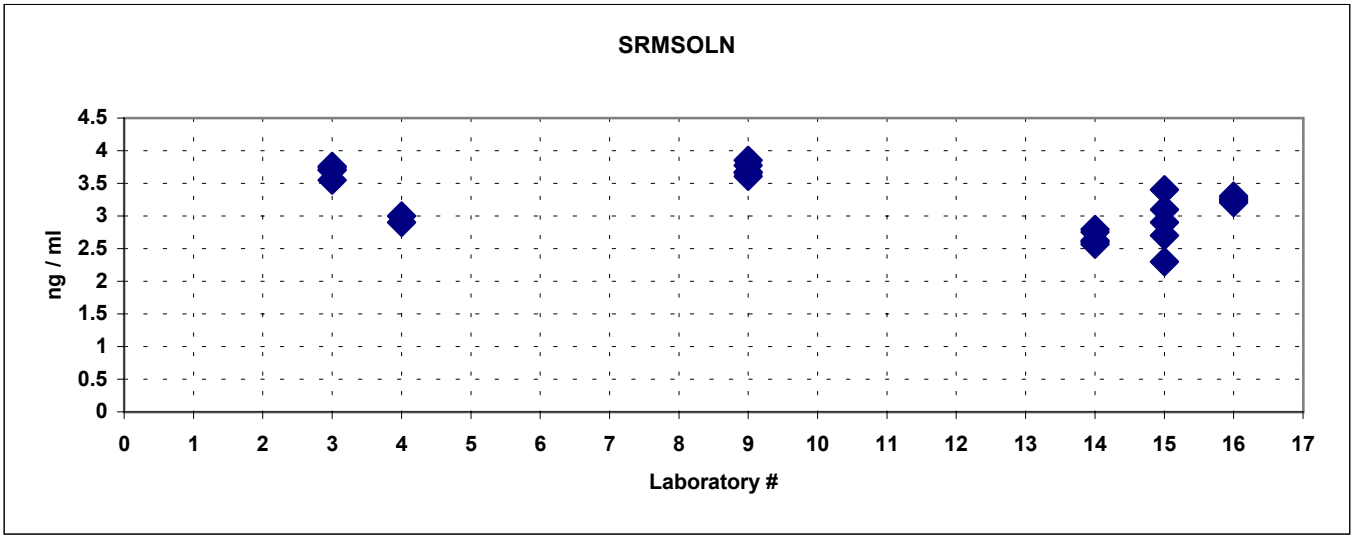
LAB #						Mean	SD	%CV
1								
2								
3								
4	3.4	3.1				3.3	0.2	6.5
5								
6								
7	2.7							
8								
9								
10								
11								
12								
13	2.56	2.71	2.61			2.63	0.08	2.9
14								
15	3.5	3.4	3.5	3.5	3.5	3.5	0.0	1.3
16								

APPENDIX C

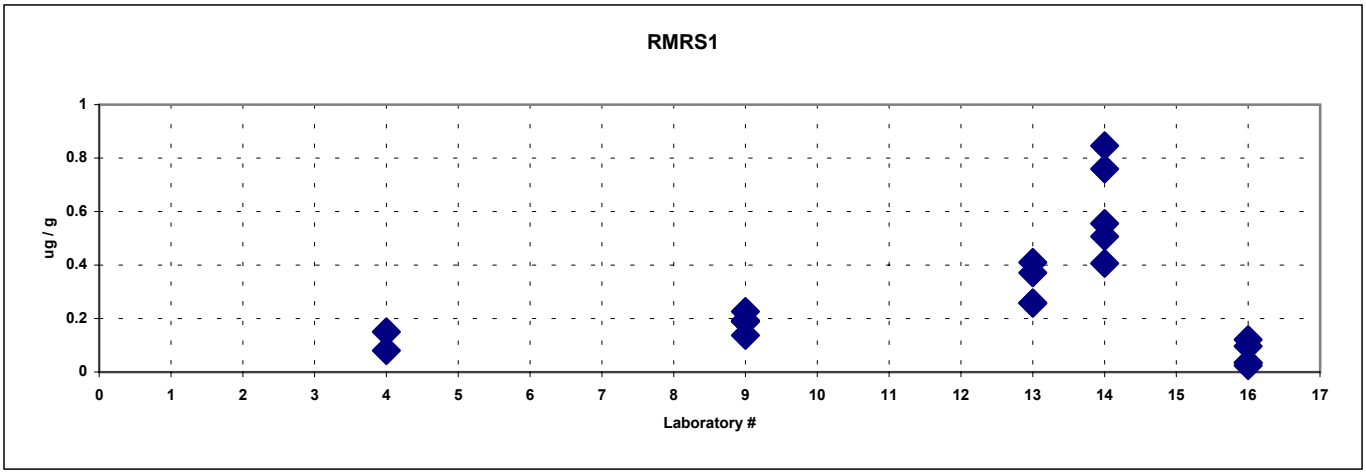
DATA PLOTS

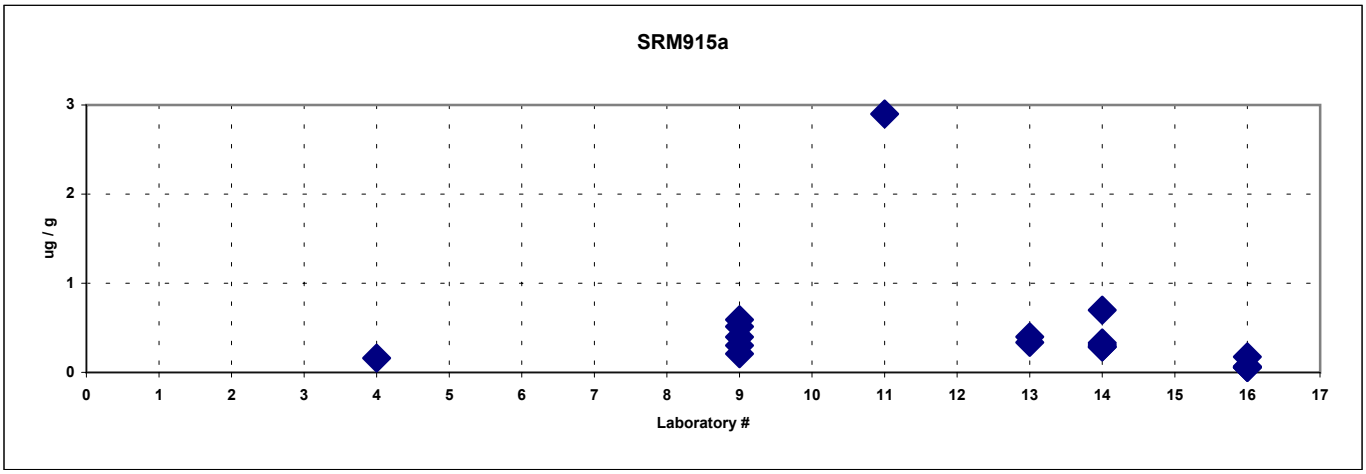
Lithium

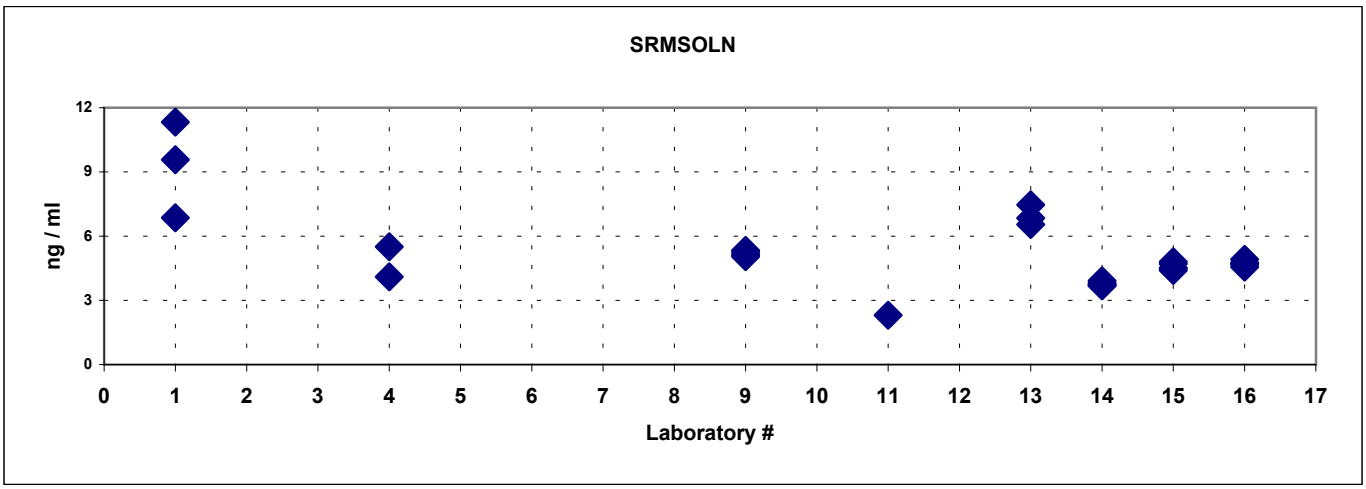

RMRS1
ug / g
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

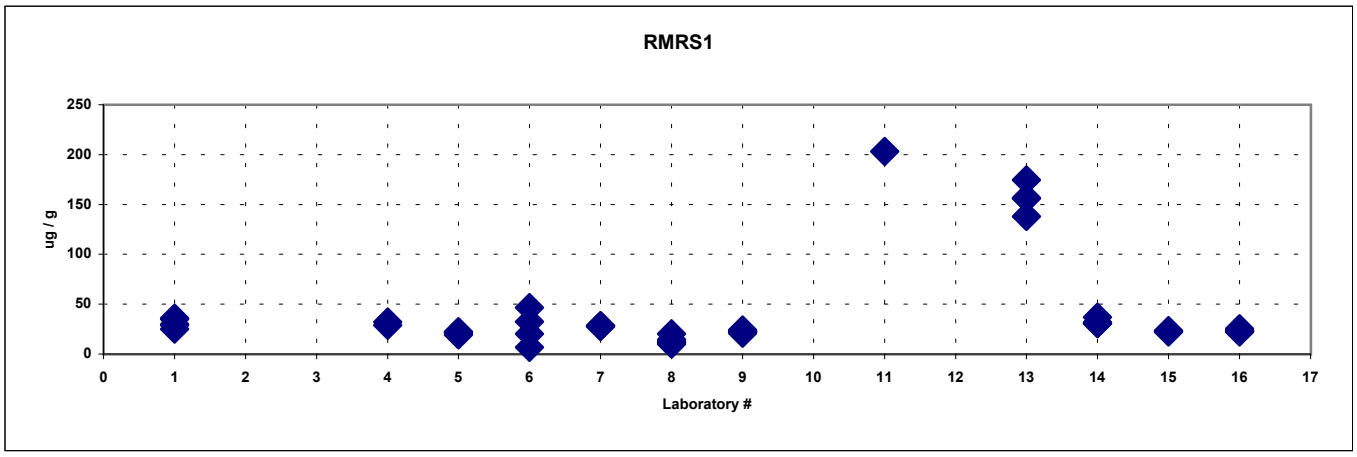
Sodium

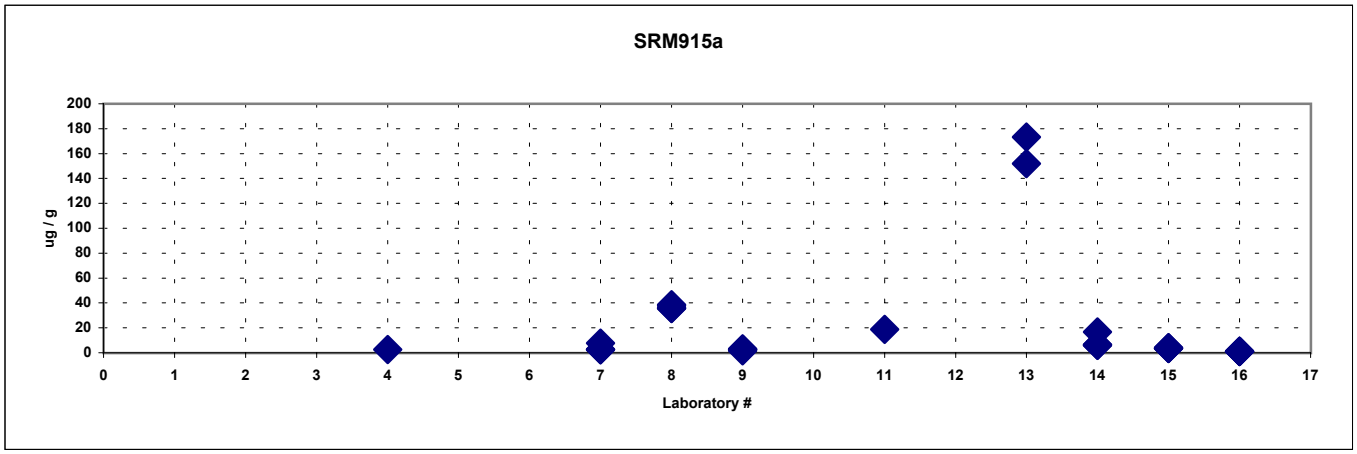

RMRS1
ug / g
1
0.8
0.6
0.4
0.2
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

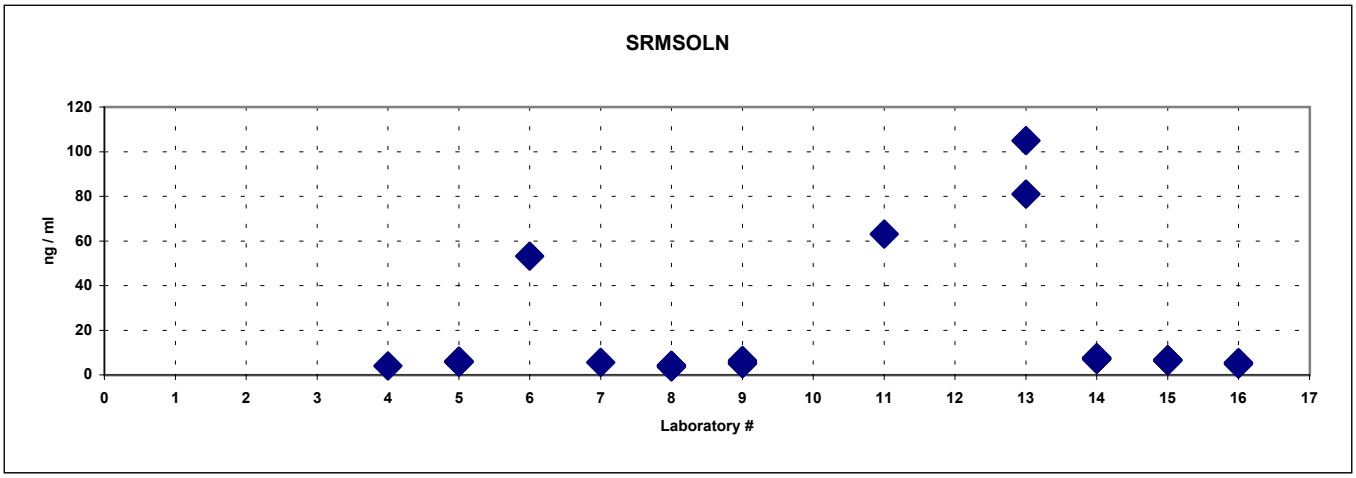

SRM915a
ug / g
3
2
1
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
12
9
6
3
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

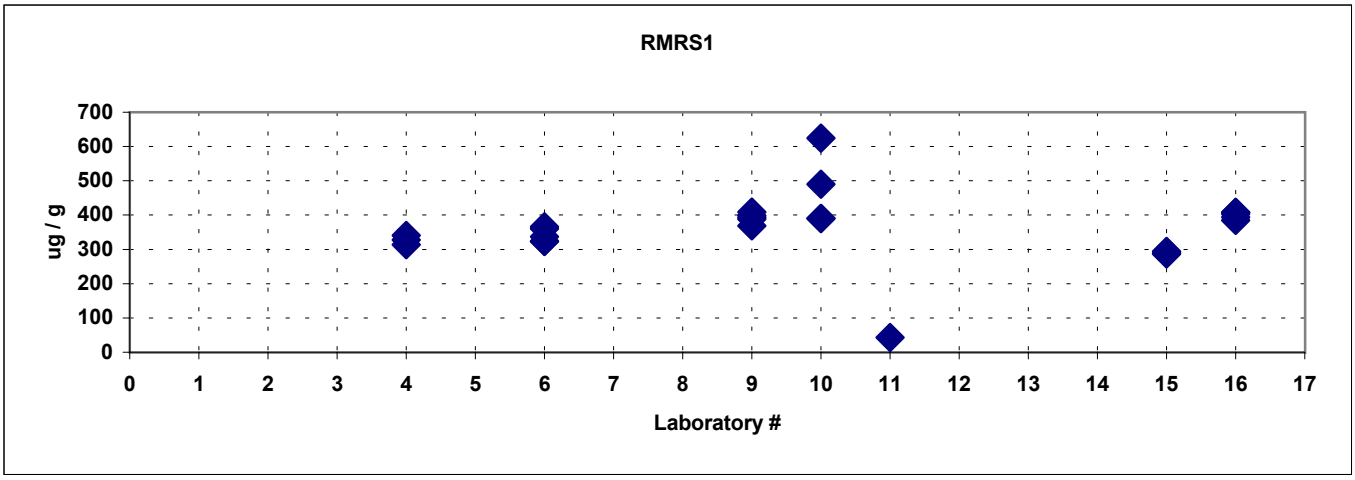
Magnesium

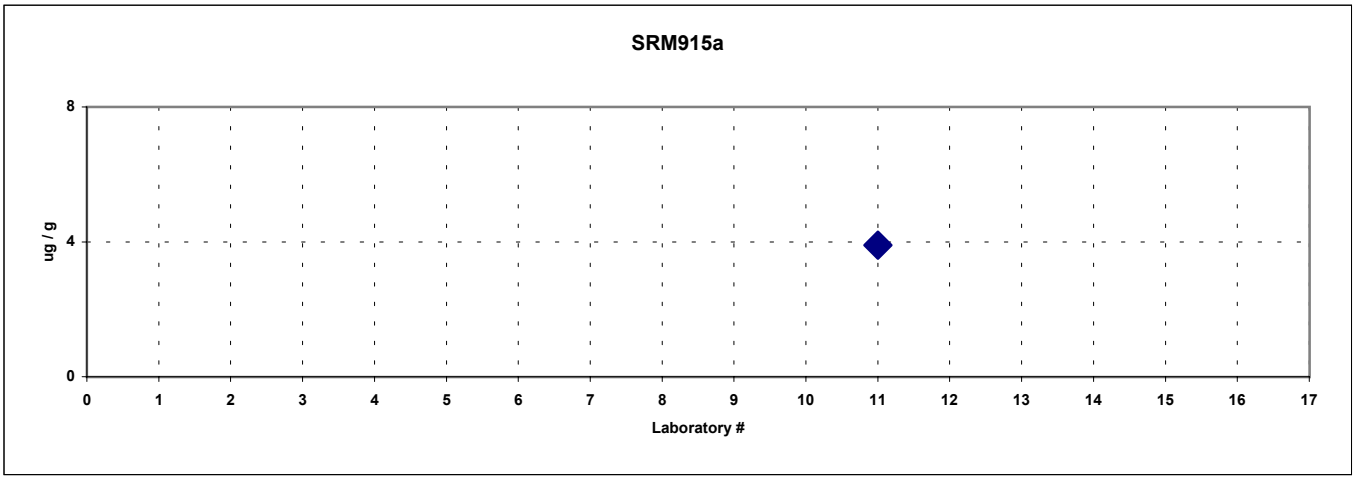

RMRS1
ug / g
250
200
150
100
50
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

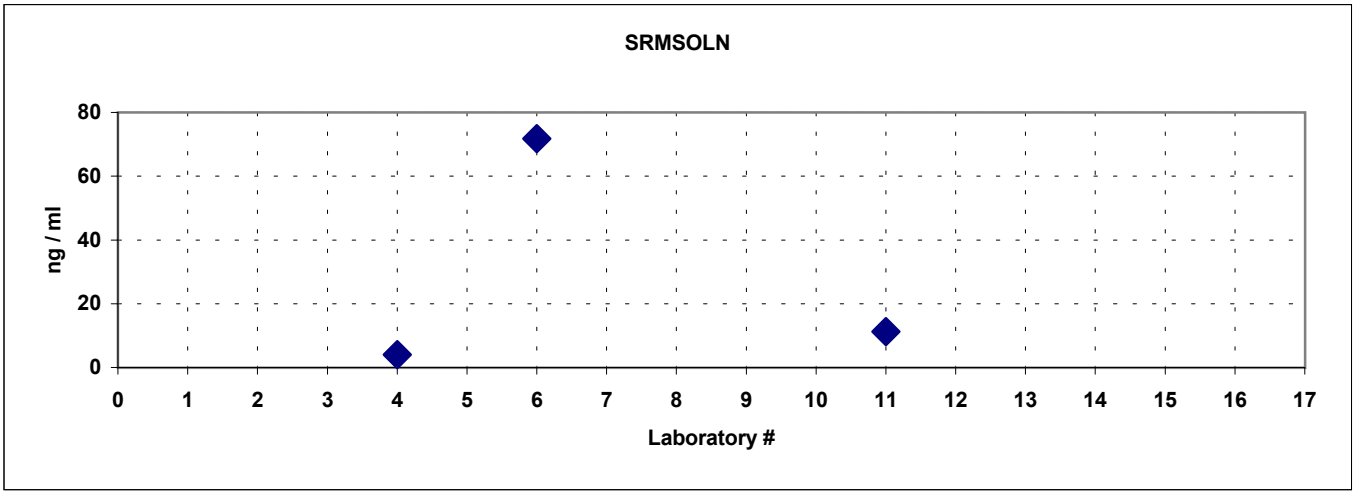

SRM915a
ug / g
200
180
160
140
120
100
80
60
40
20
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
120
100
80
60
40
20
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

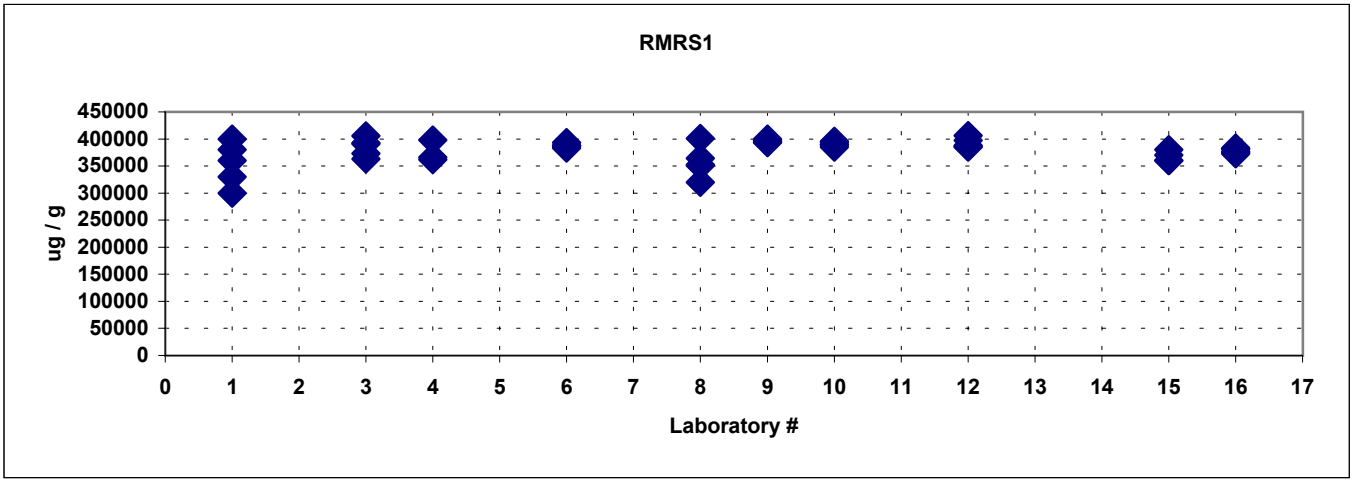
Potassium

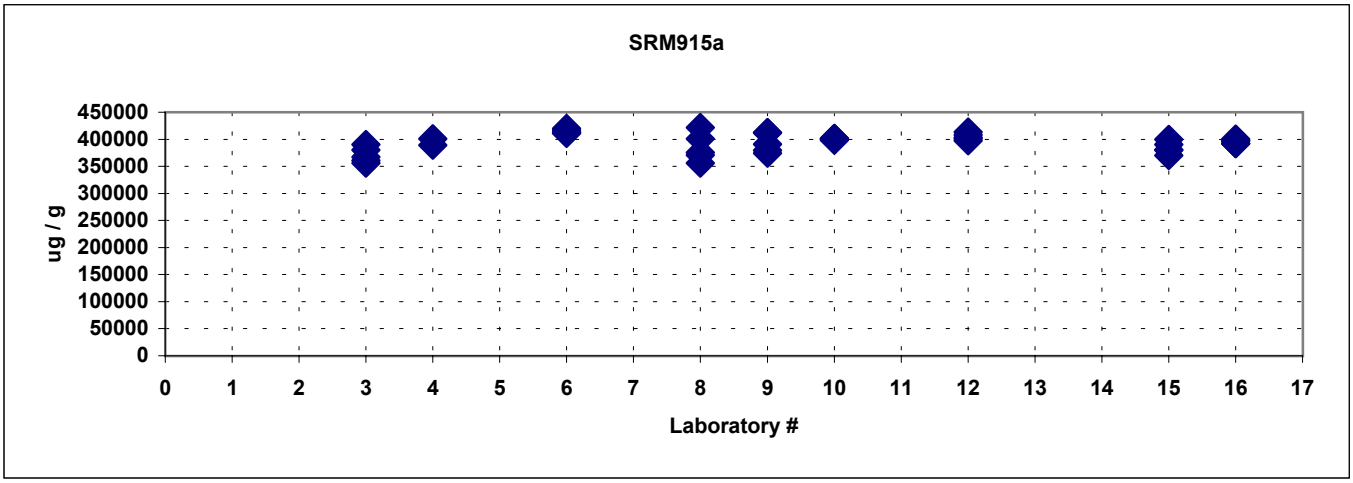

RMRS1
ug / g
700
600
500
400
300
200
100
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

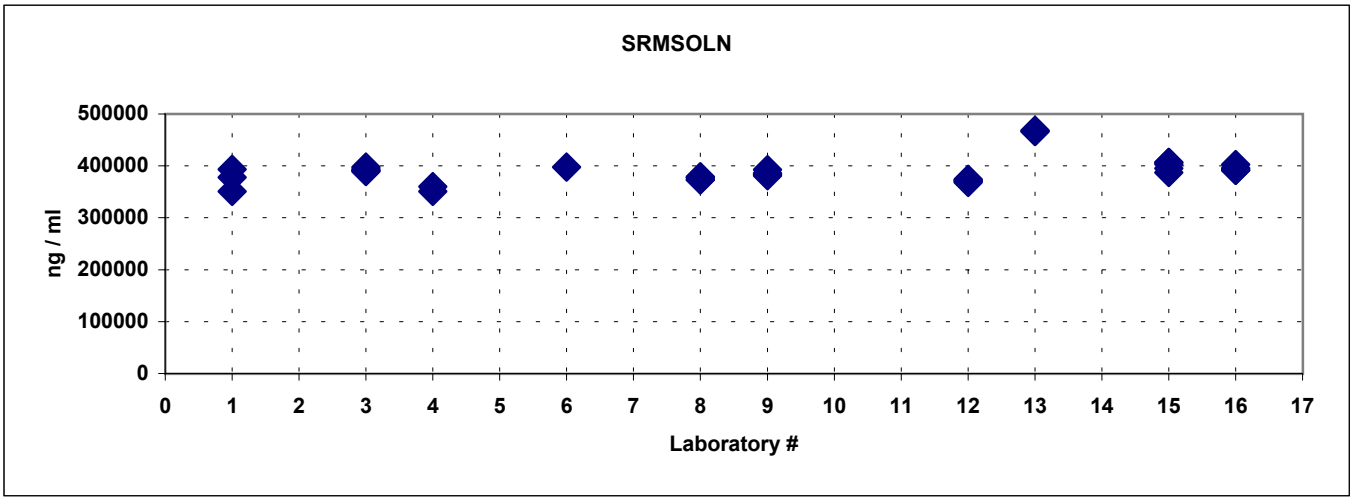

SRM915a
ug / g
8
4
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
80
60
40
20
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

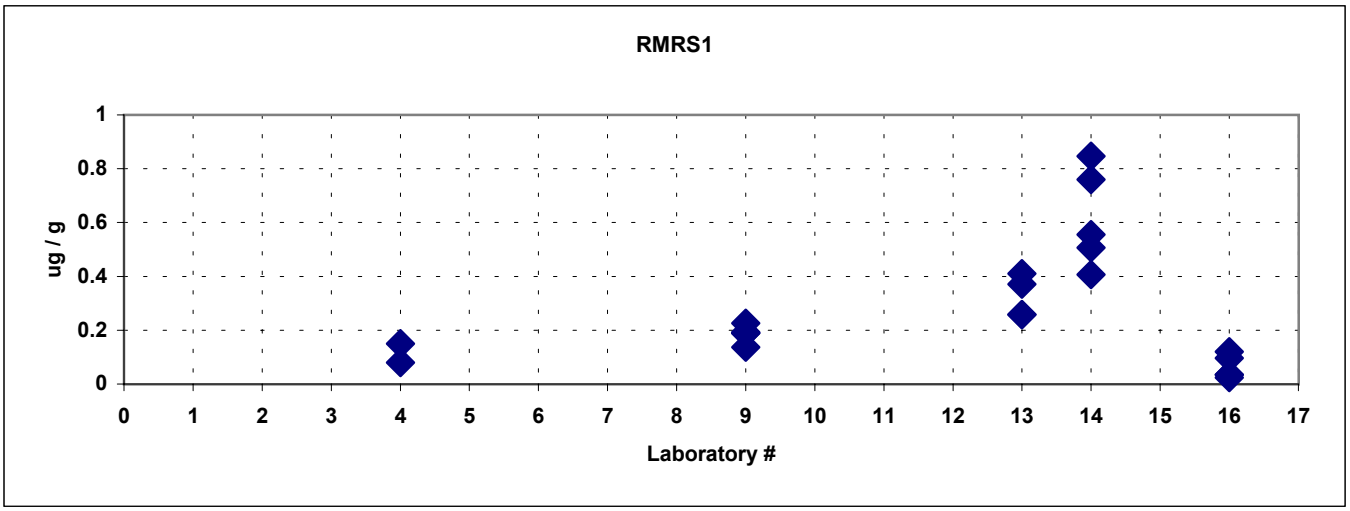
Calcium

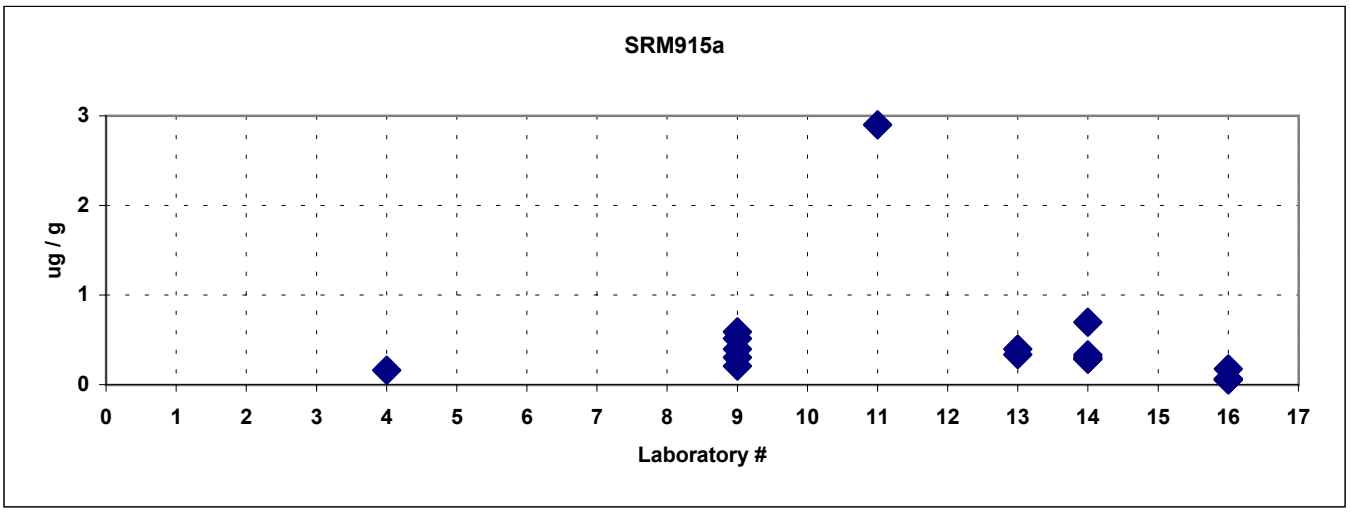

RMRS1
ug / g
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

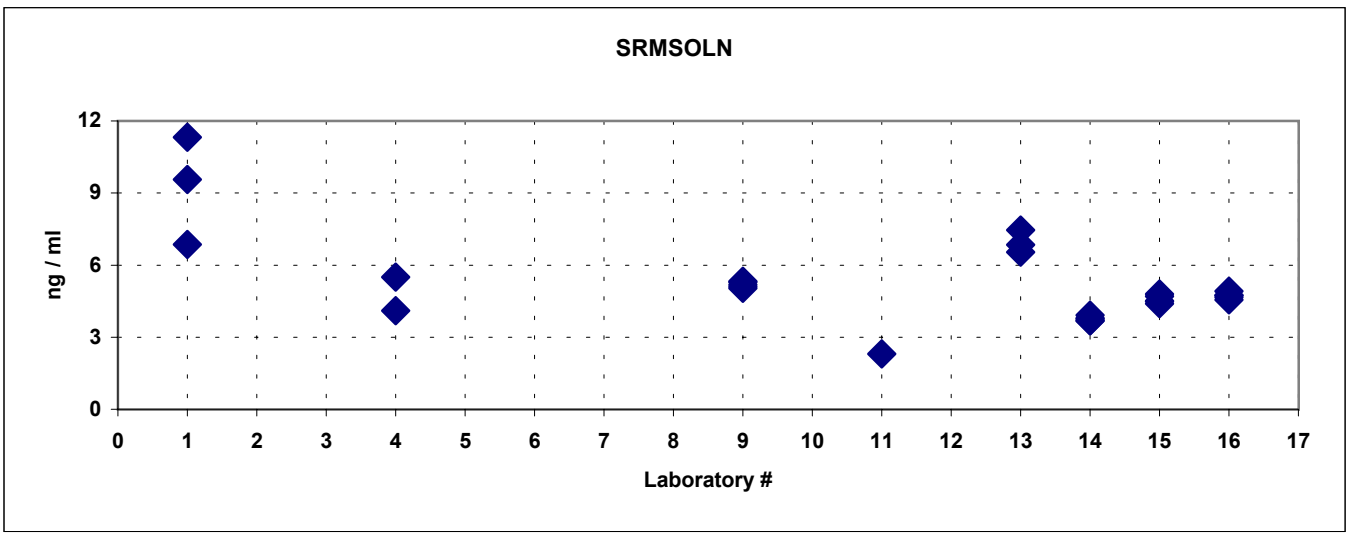

SRM915a
ug / g
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
500000
400000
300000
200000
100000
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

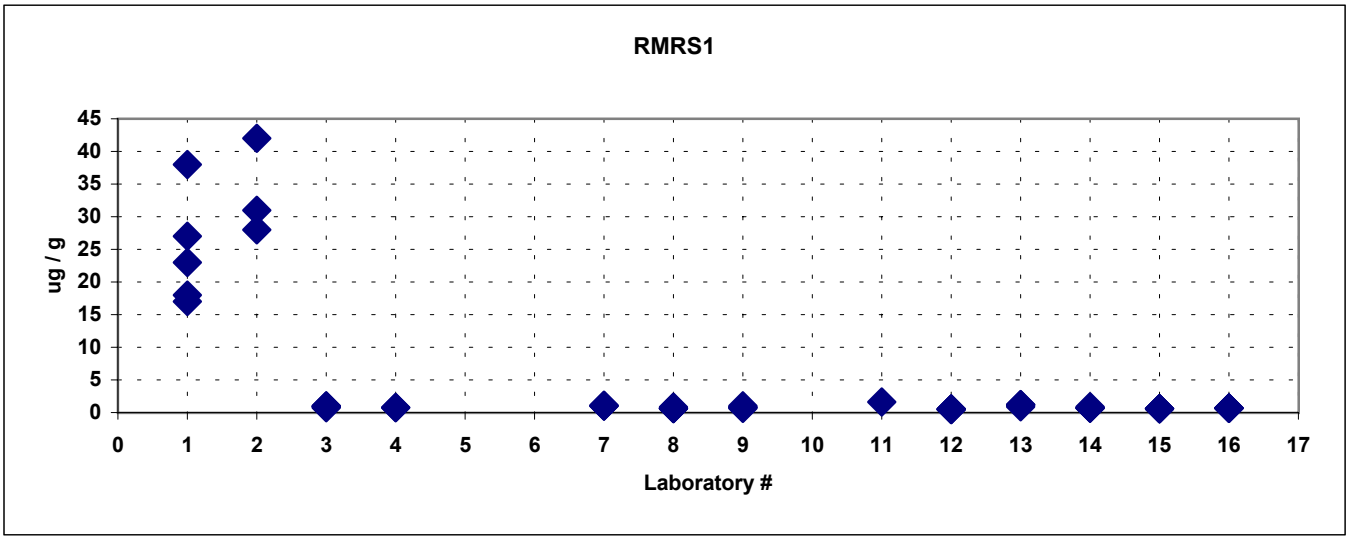
Chromium

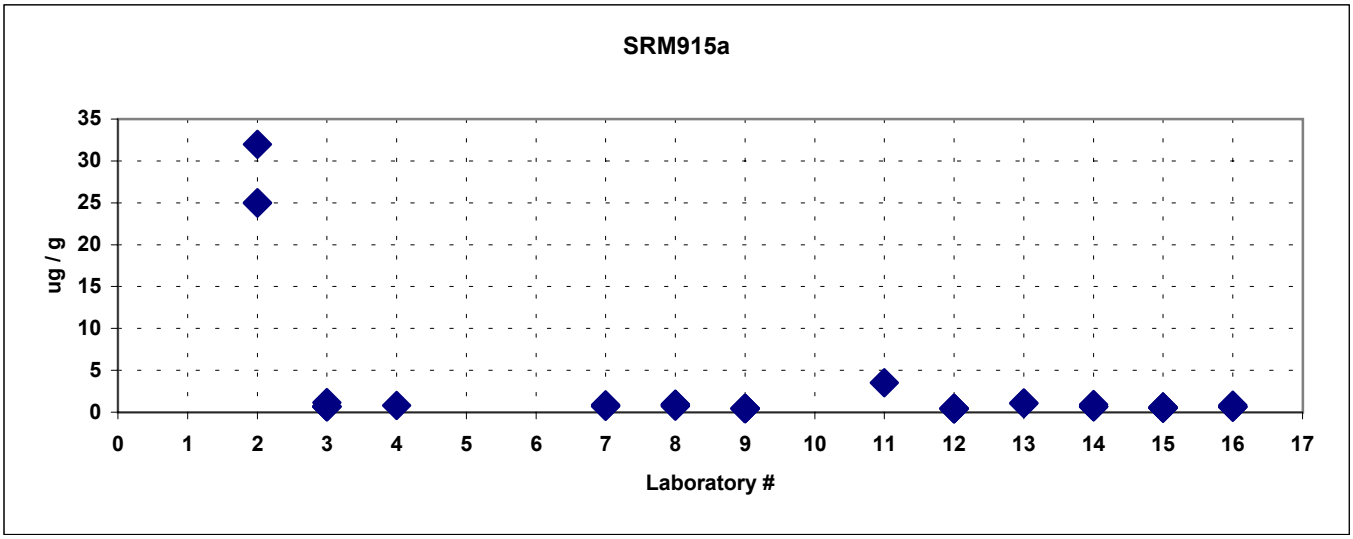

RMRS1
ug / g
1
0.8
0.6
0.4
0.2
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

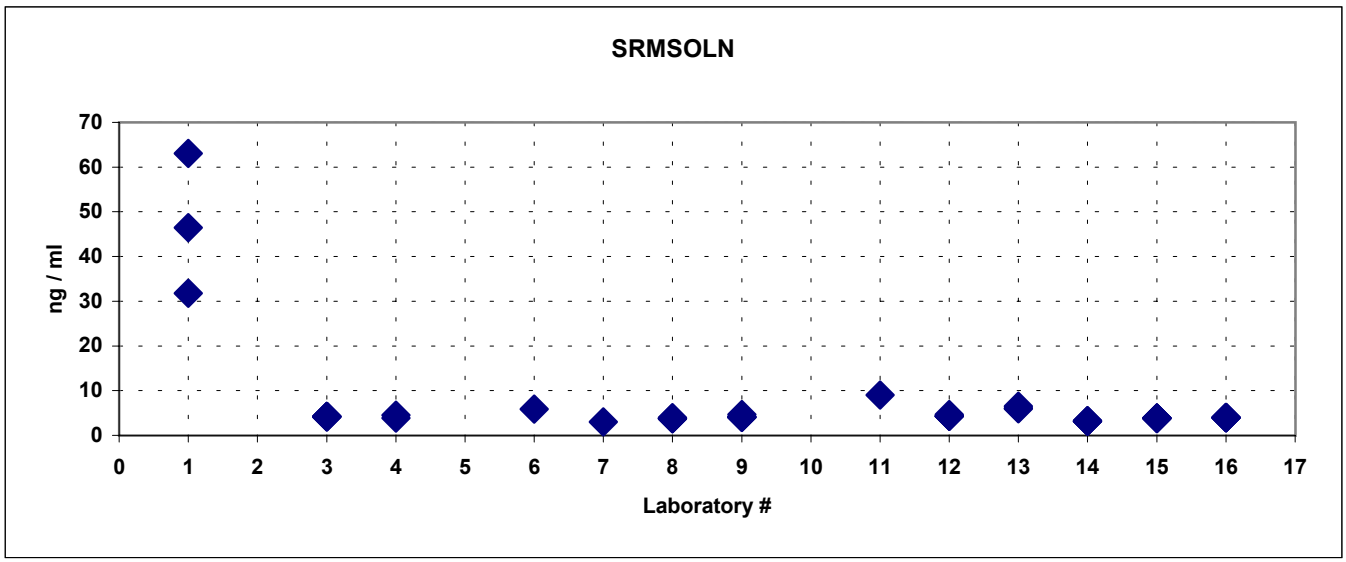

SRM915a
ug / g
3
2
1
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

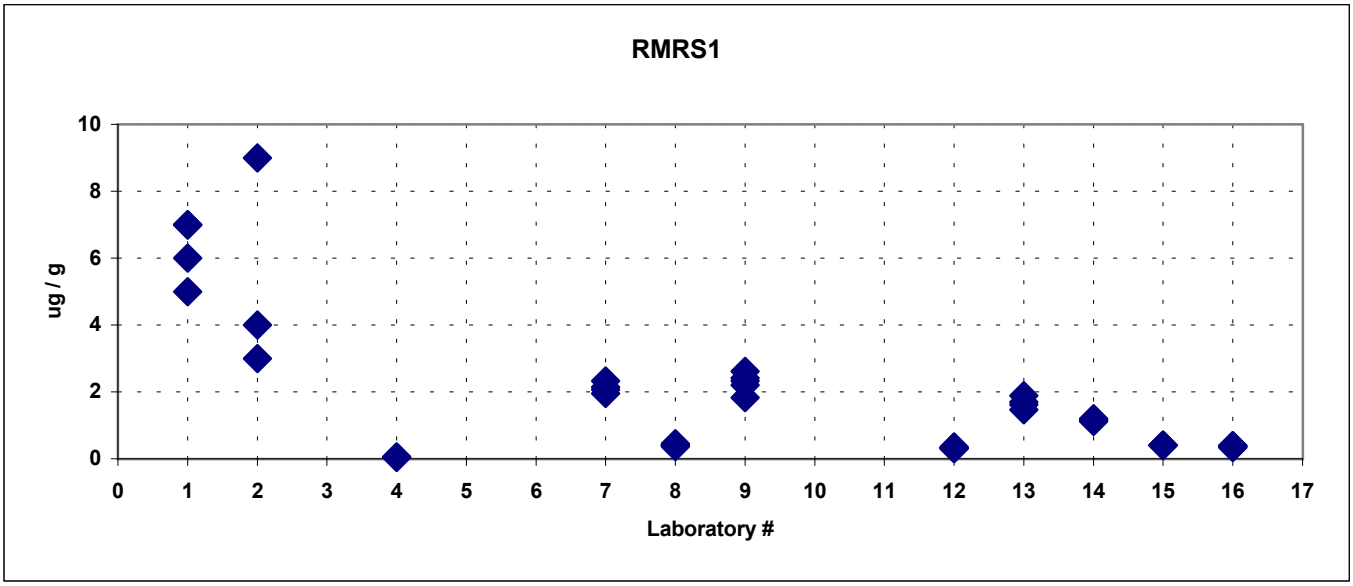

SRMSOLN
ng / ml
12
9
6
3
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

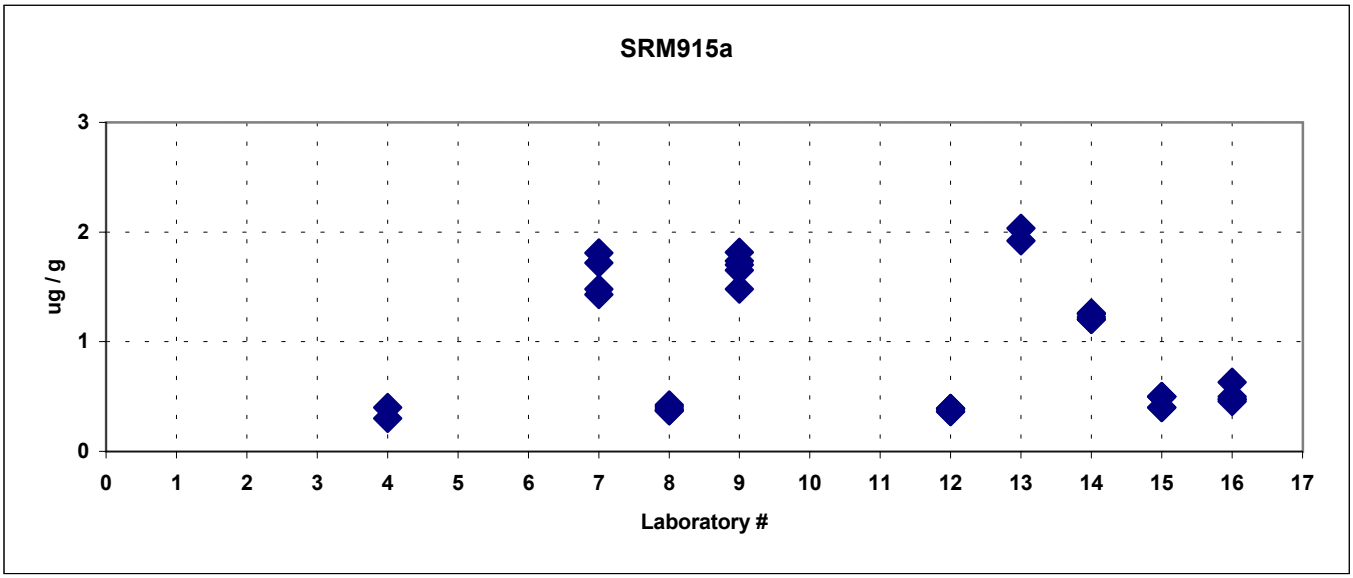
Manganese

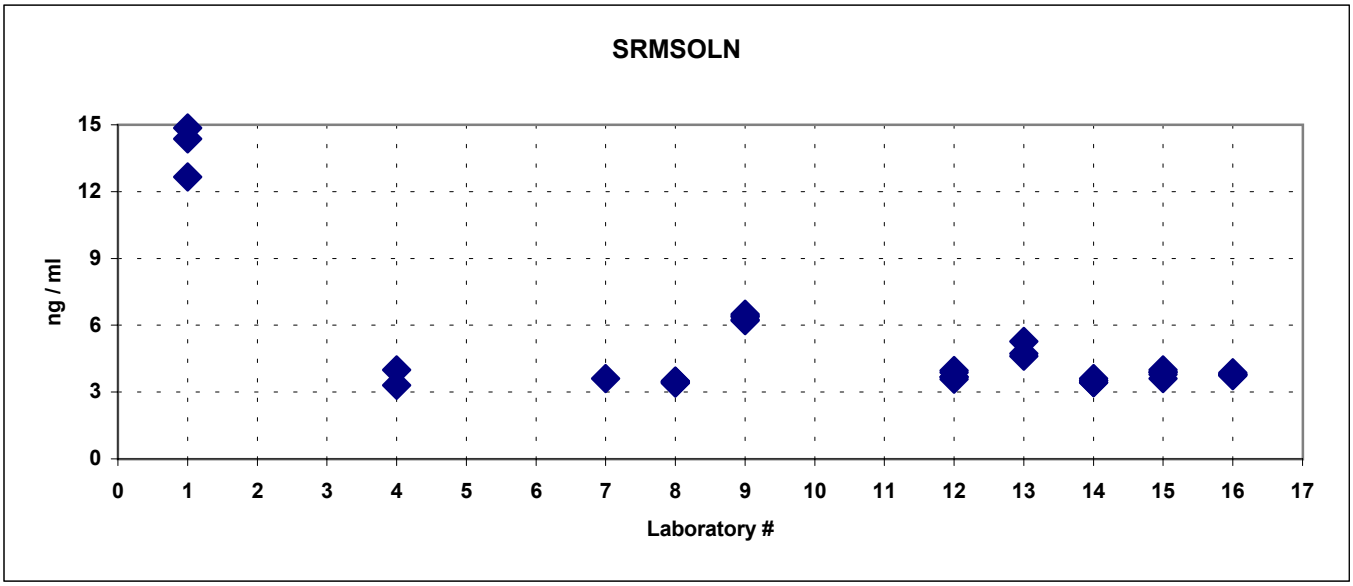

RMRS1
ug / g
Laboratory #


SRM915a
ug / g
Laboratory #

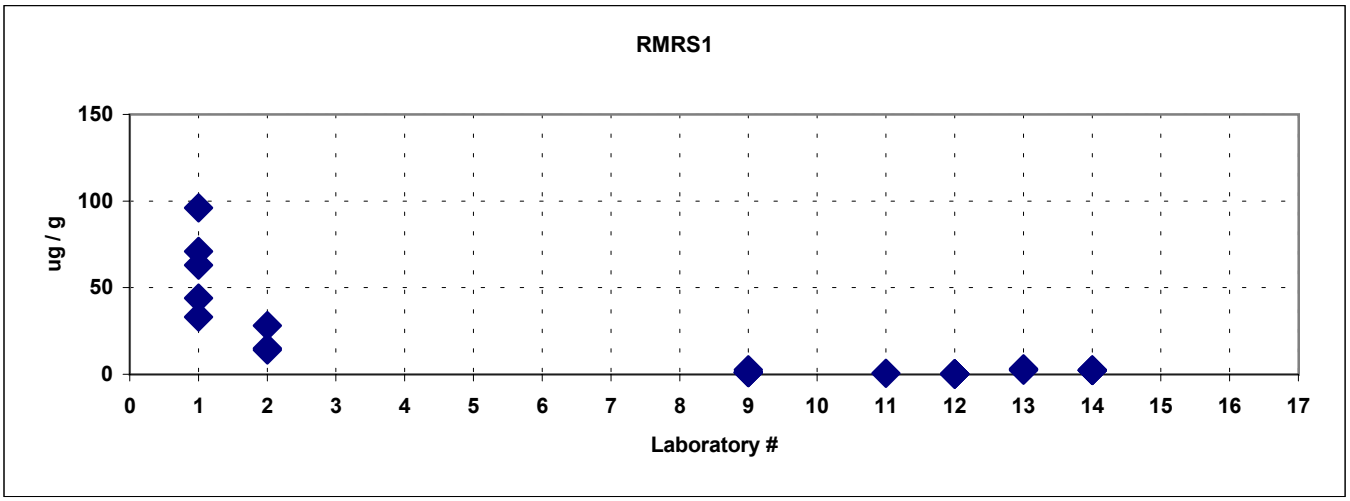

SRMSOLN
ng / ml
Laboratory #

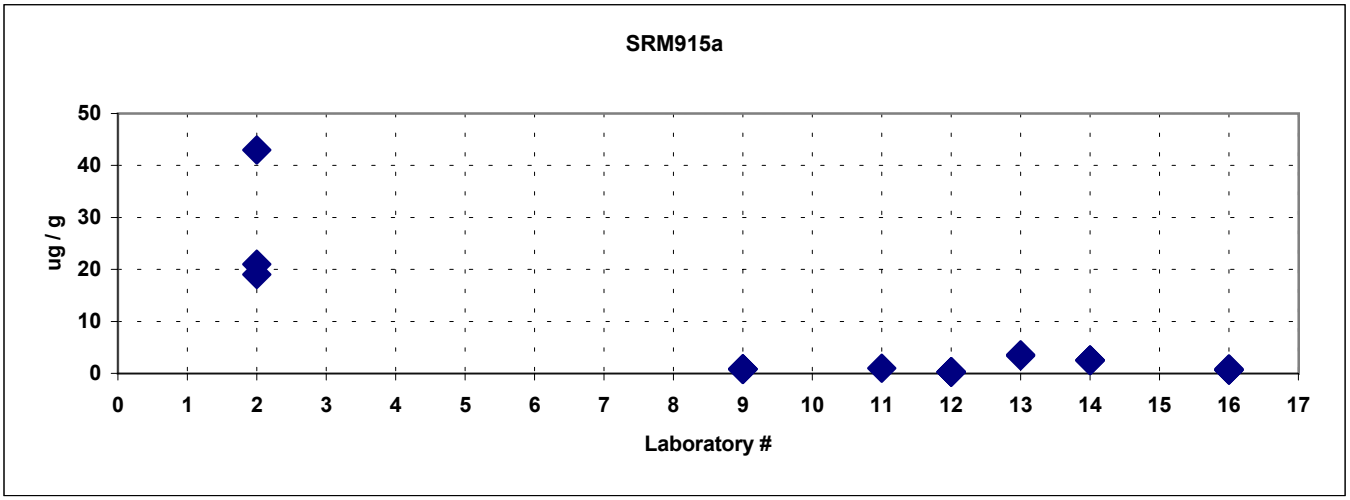

RMRS1
ug / g
Laboratory #

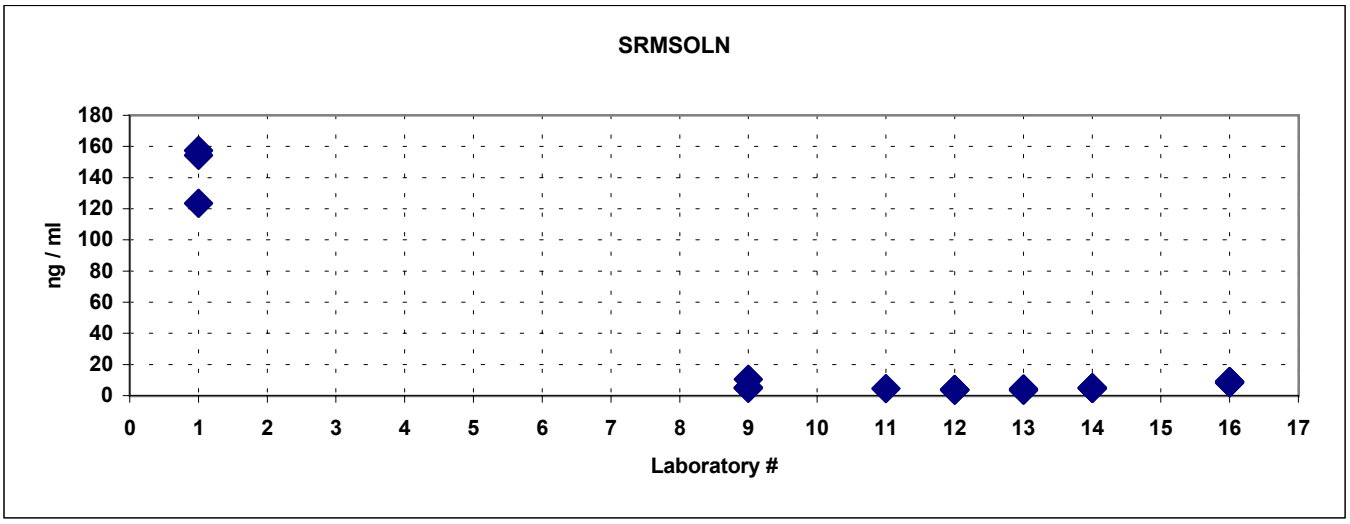

SRM915a
ug / g
Laboratory #


SRMSOLN
ng / ml
Laboratory #

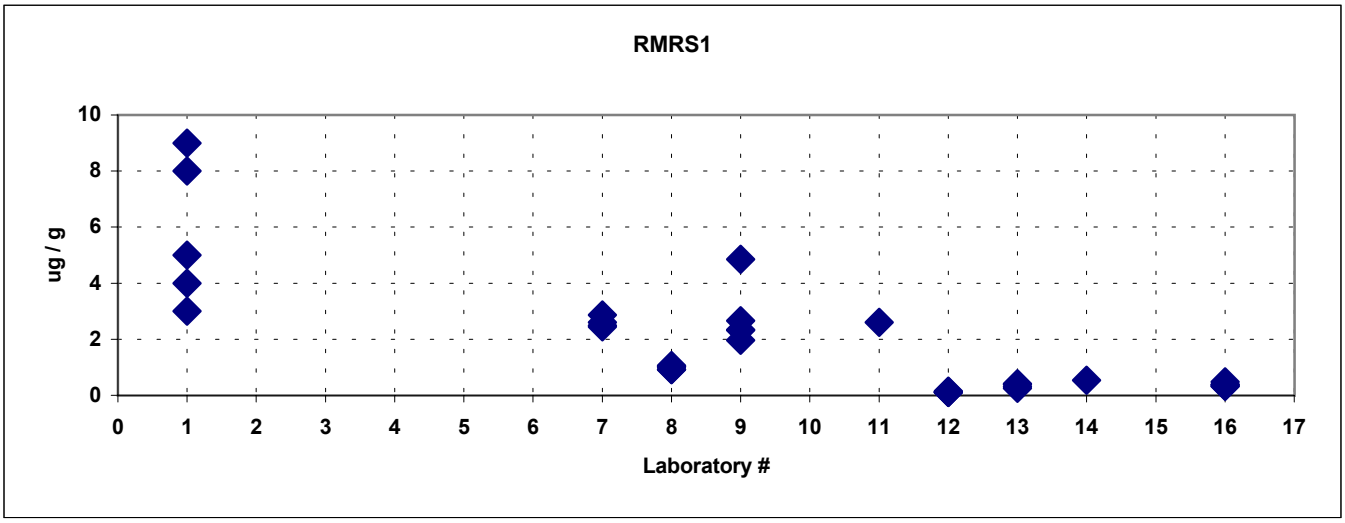
Nickel

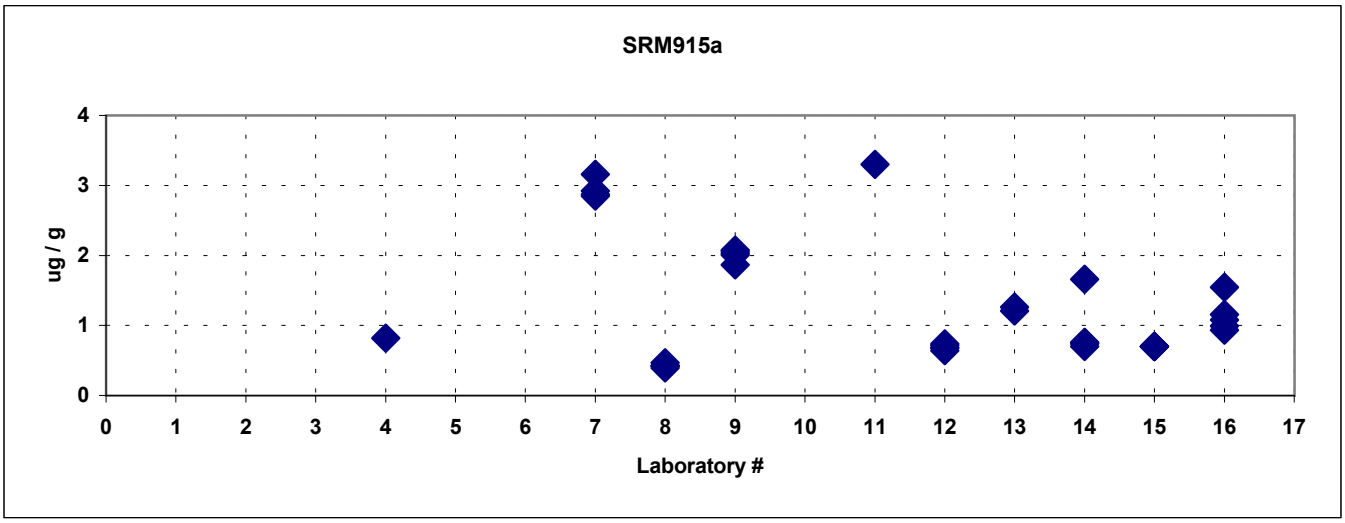

RMRS1
150
100
50
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

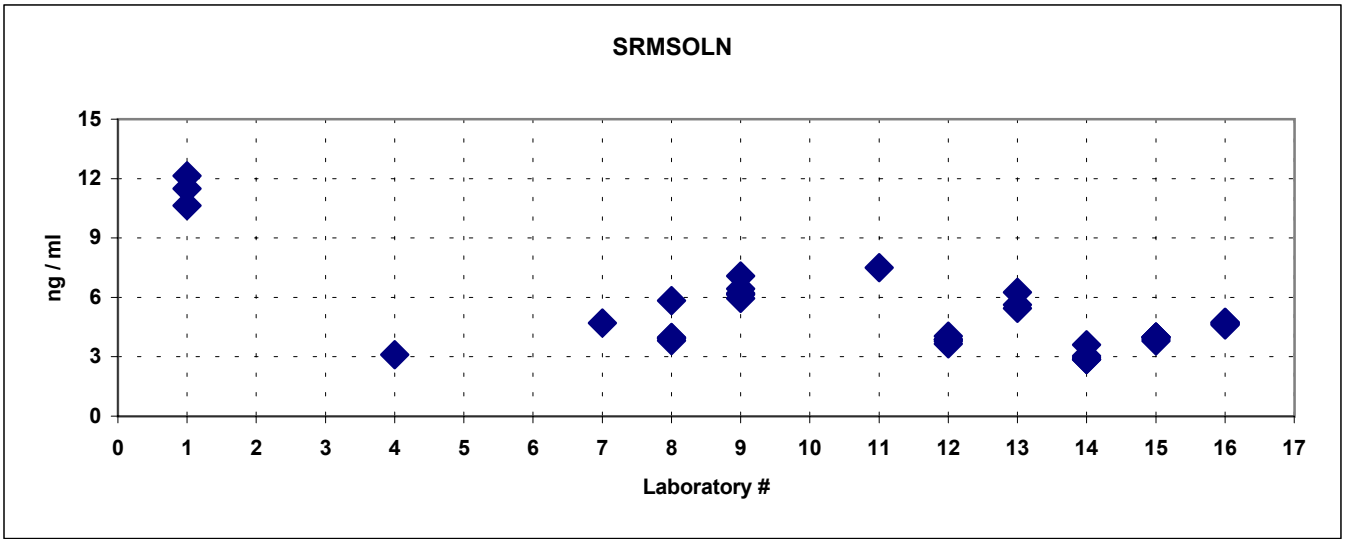

SRM915a
50
40
30
20
10
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

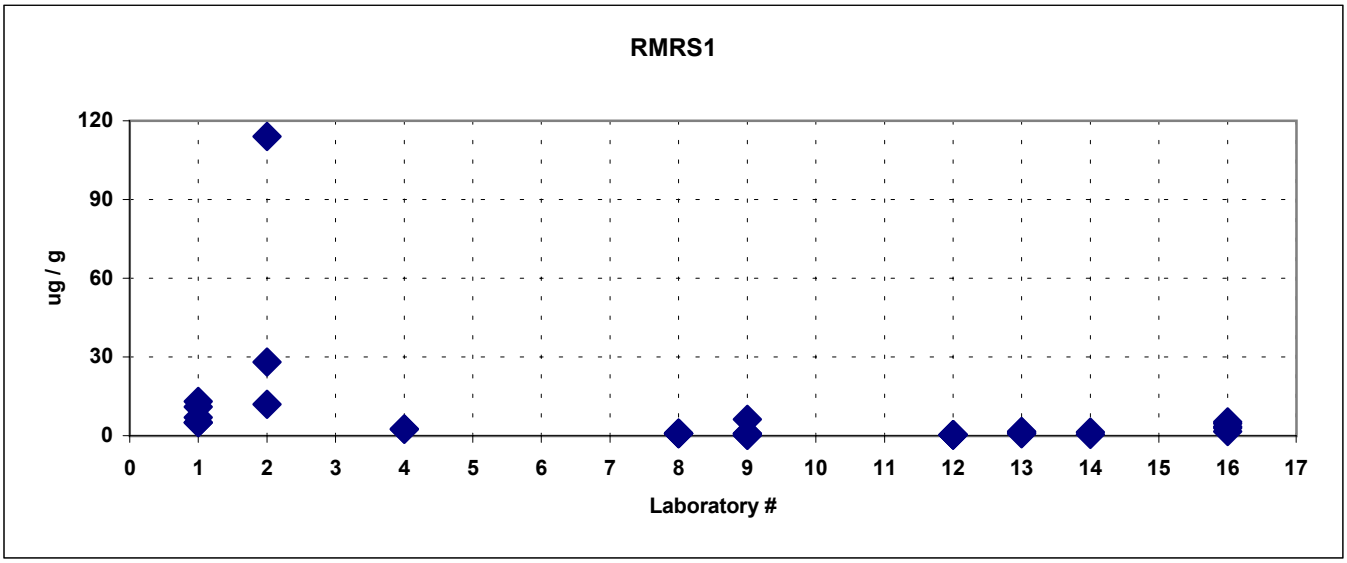

SRMSOLN
180
160
140
120
100
80
60
40
20
0
ng / ml
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

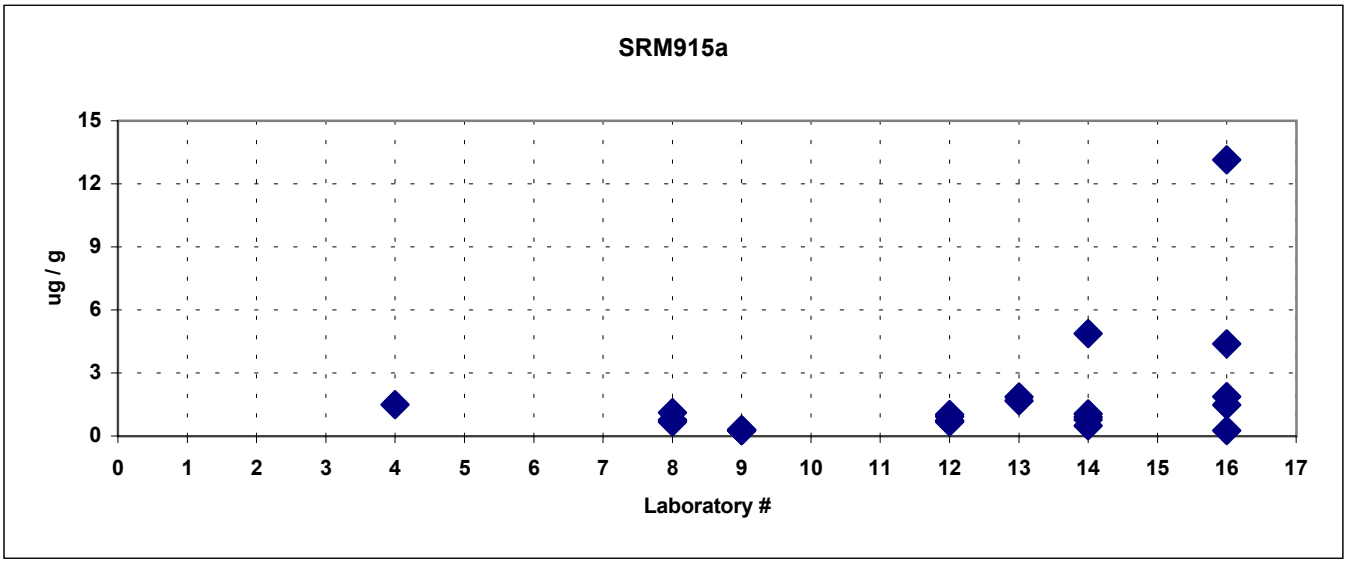
Copper

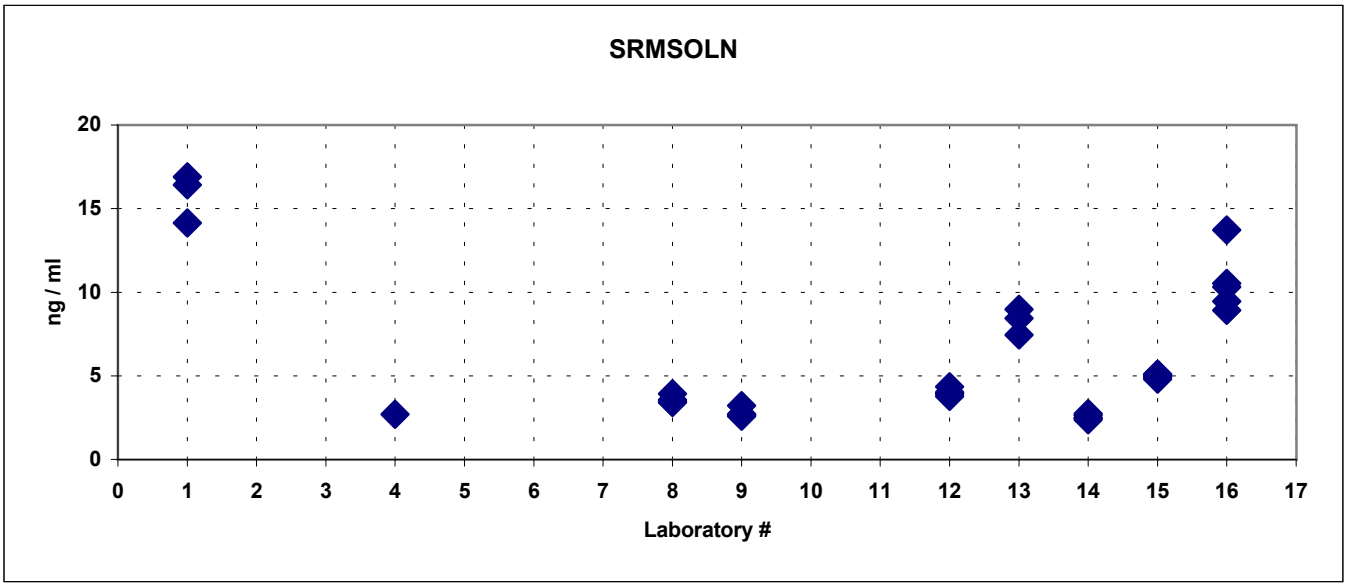

RMRS1
ug / g
0
2
4
6
8
10
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

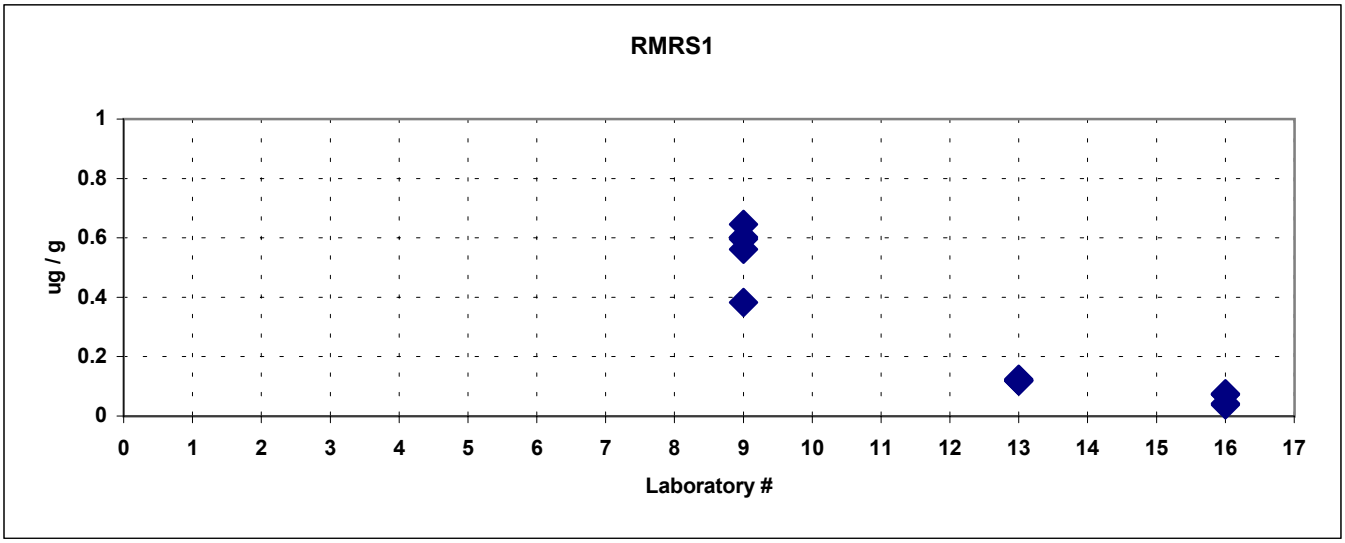

SRM915a
ug / g
0
1
2
3
4
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

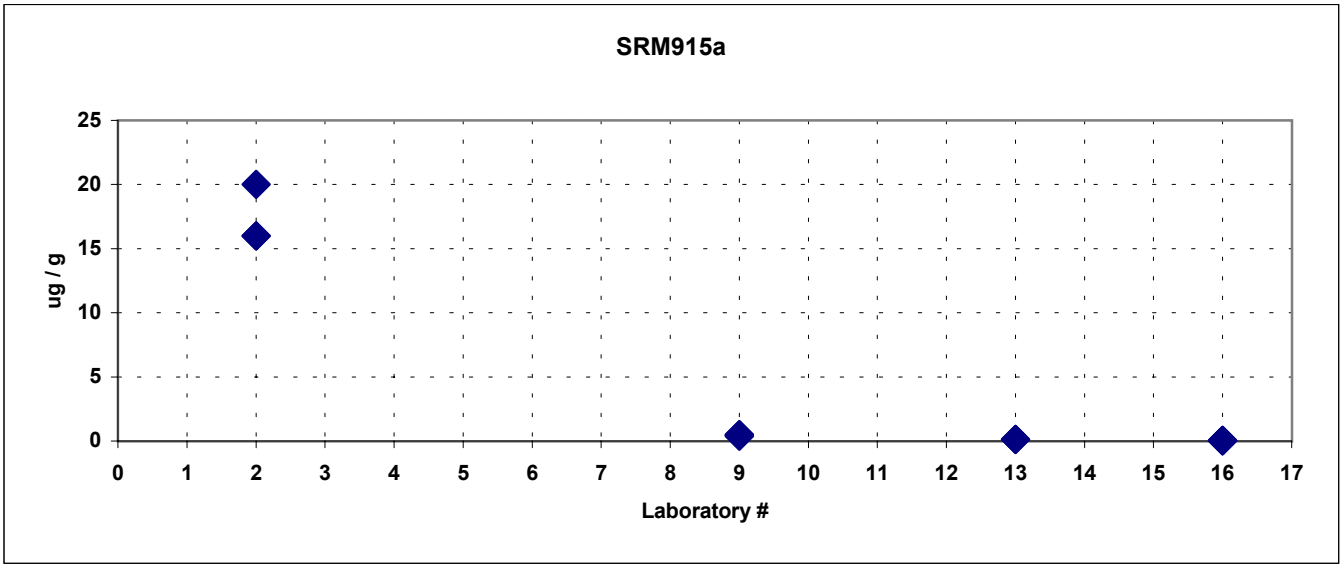

SRMSOLN
ng / ml
0
3
6
9
12
15
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

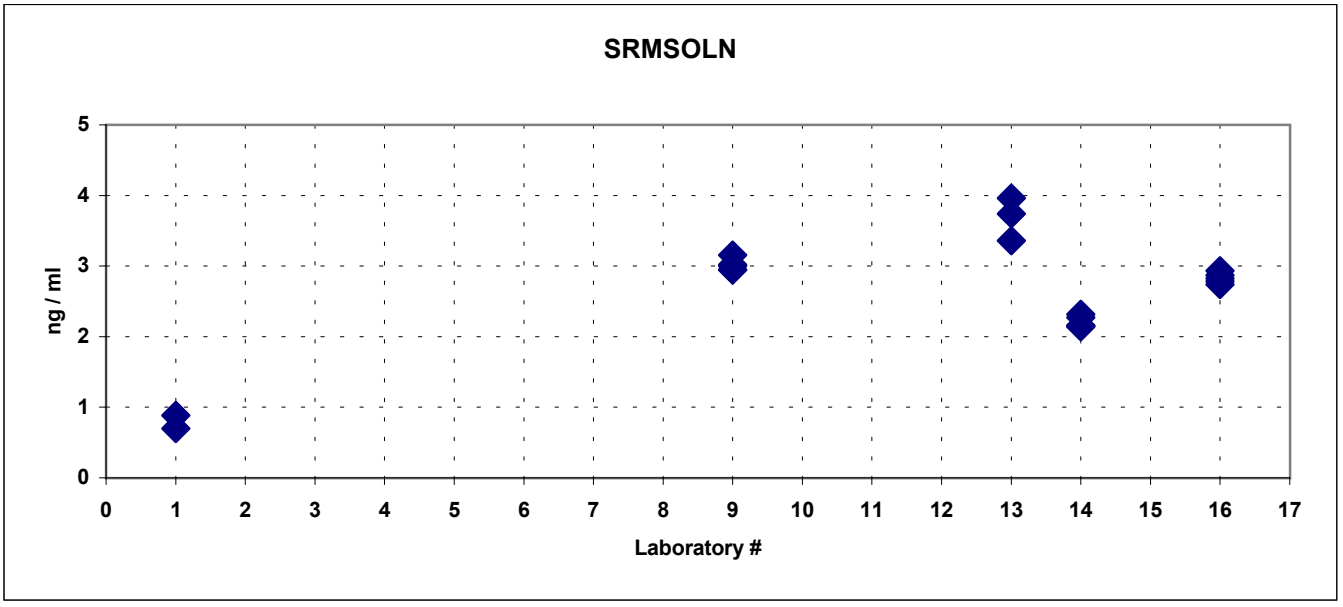

RMRS1
120
90
60
30
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRM915a
15
12
9
6
3
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

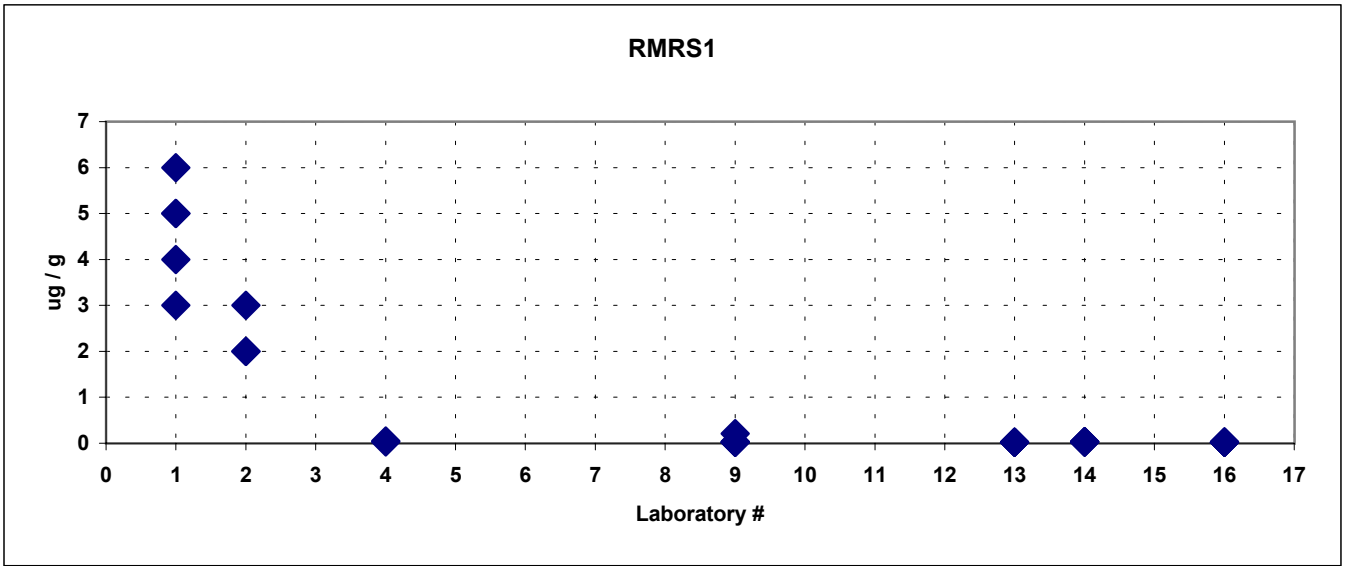

SRMSOLN
20
15
10
5
0
ng / ml
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

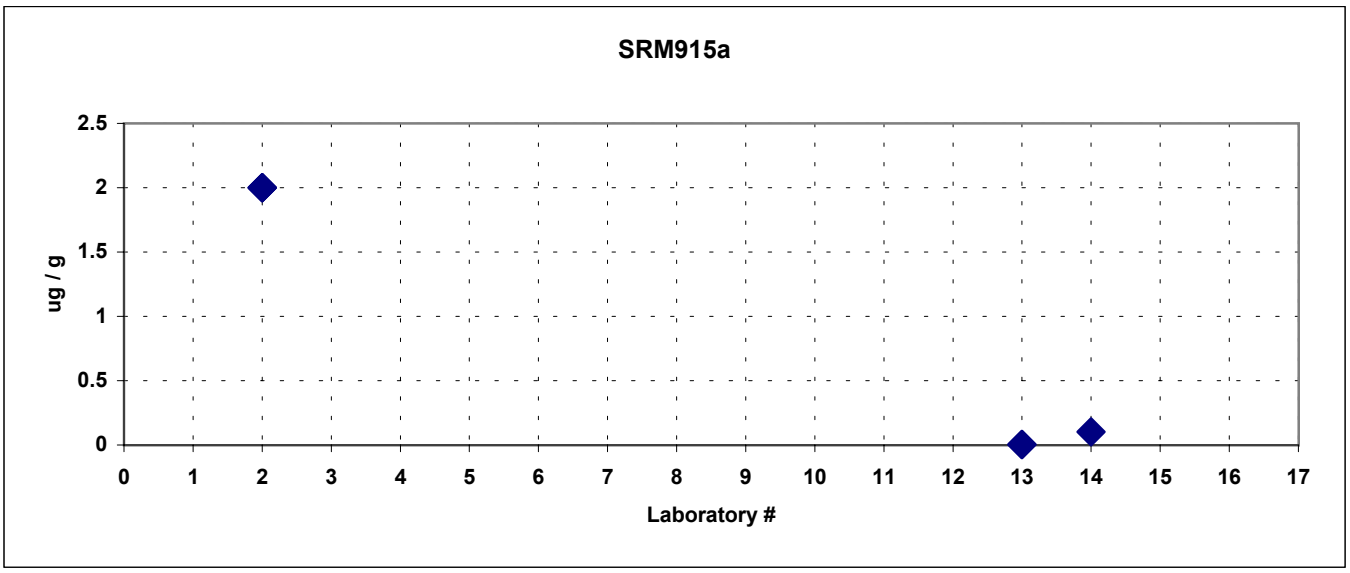

RMRS1
ug / g
Laboratory #

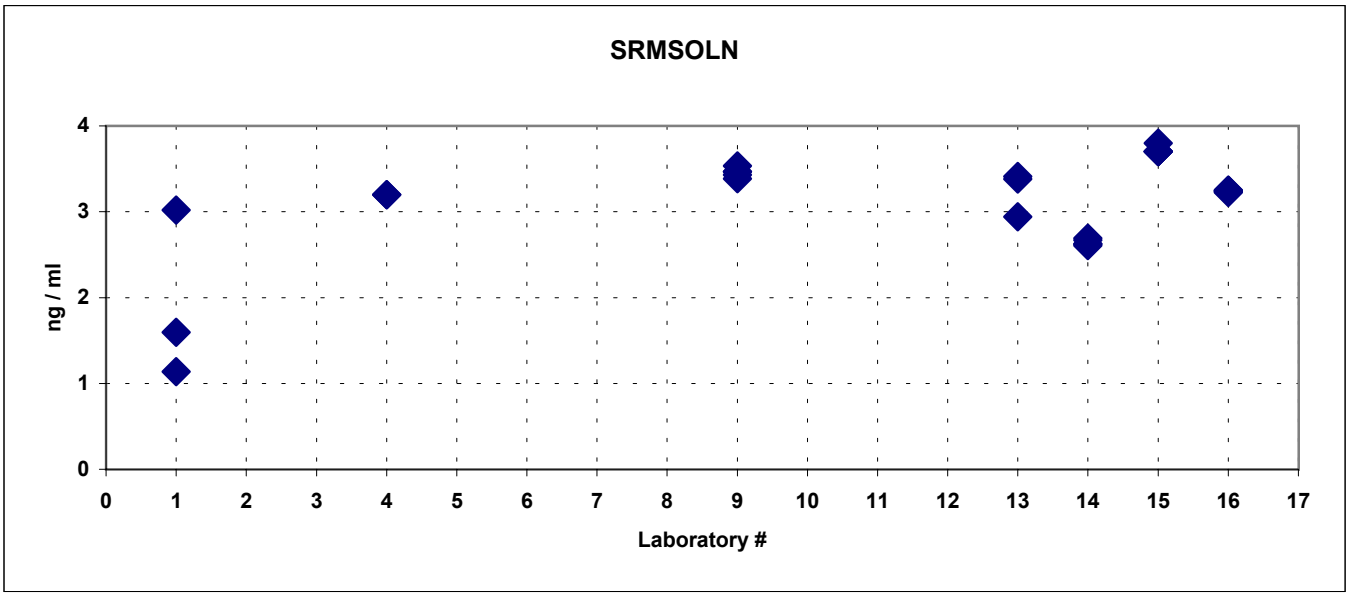

SRM915a
ug / g
Laboratory #

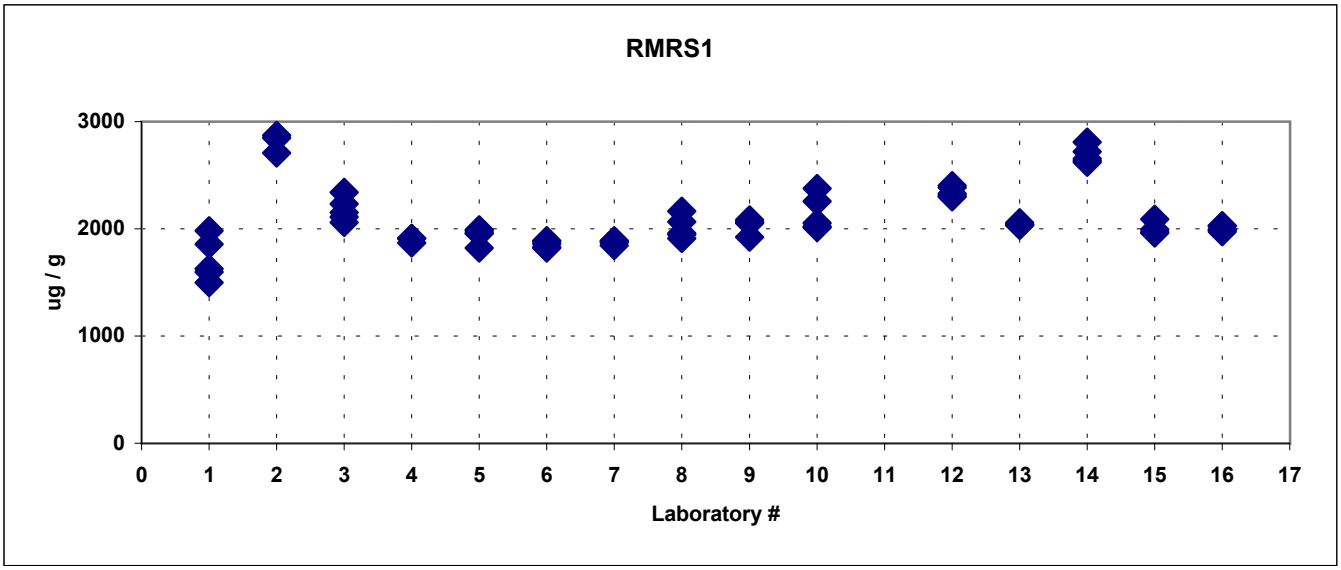

SRMSOLN
ng / ml
Laboratory #

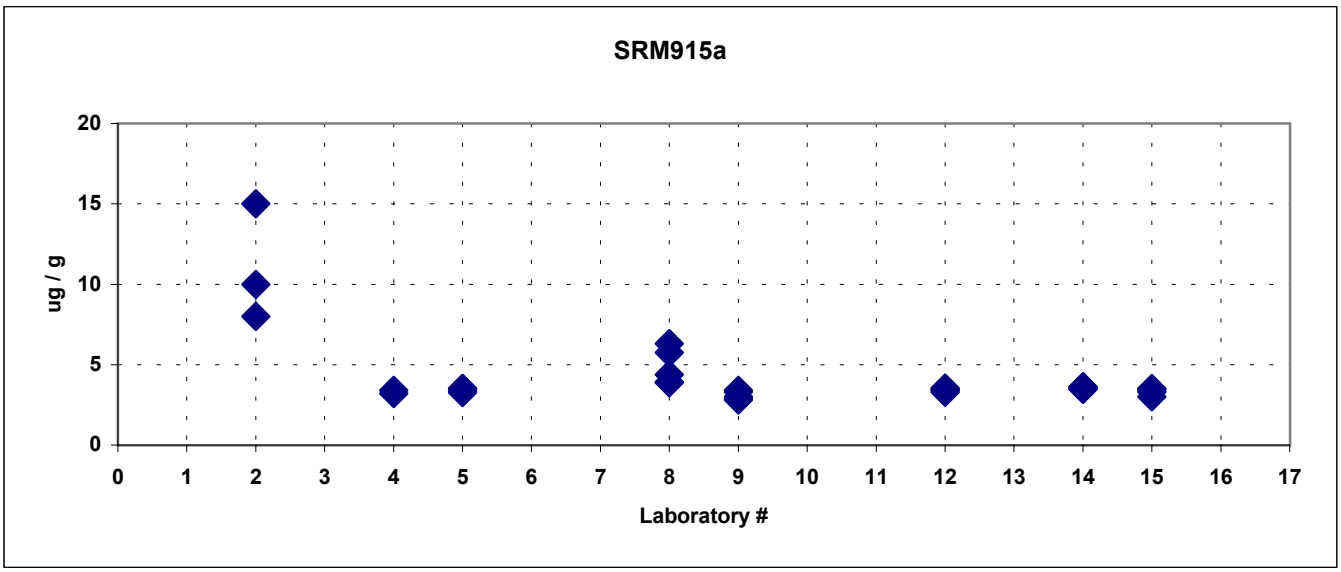
Rubidium


RMRS1
ug / g
Laboratory #


SRM915a
ug / g
Laboratory #


SRMSOLN
ng / ml
Laboratory #


RMRS1
3000
2000
1000
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #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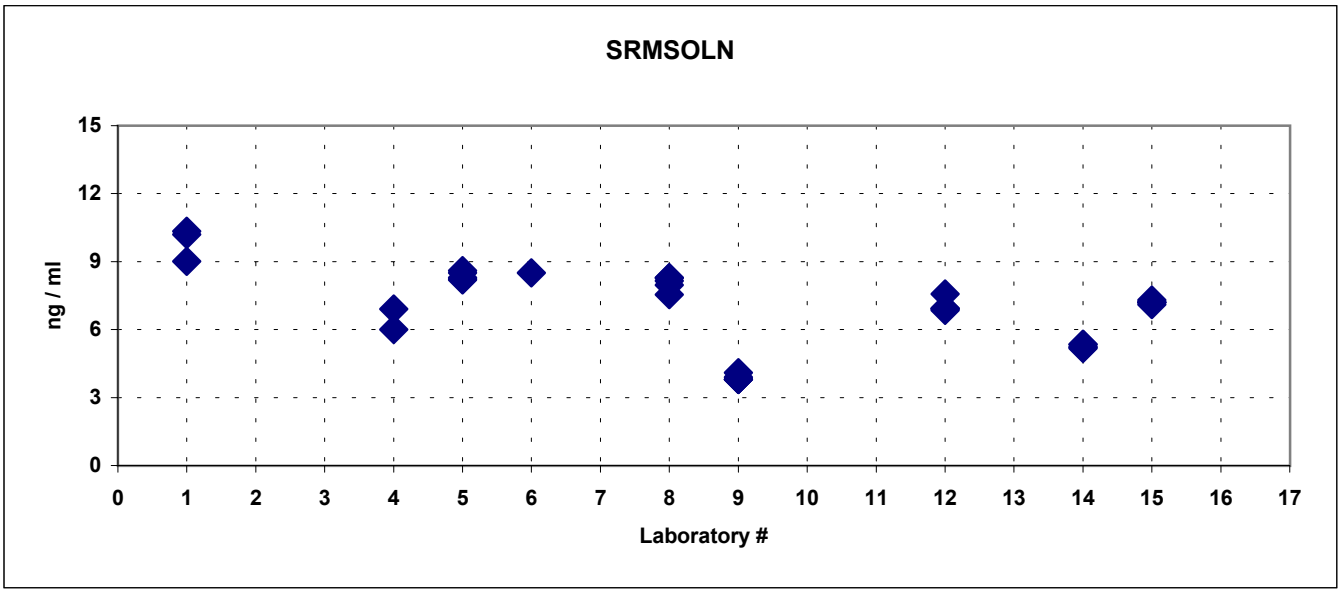
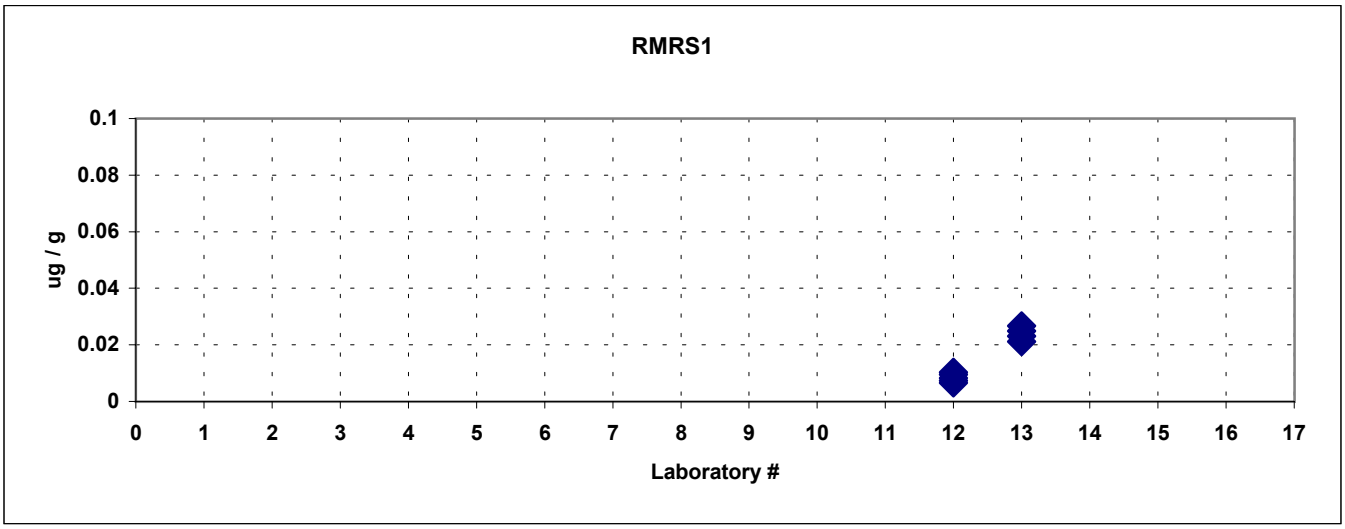
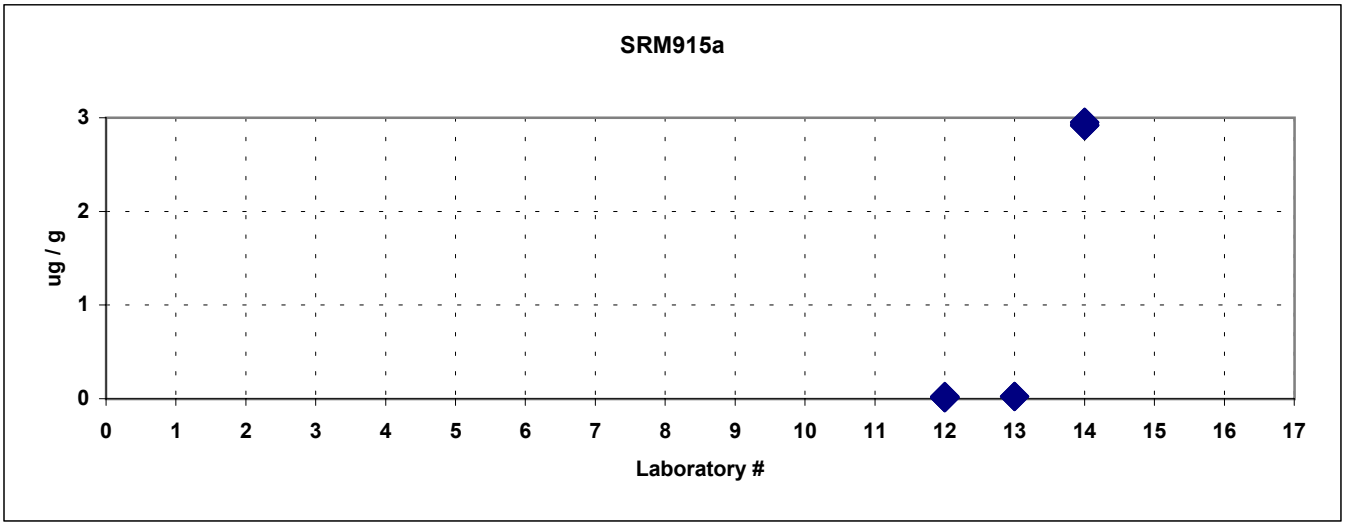
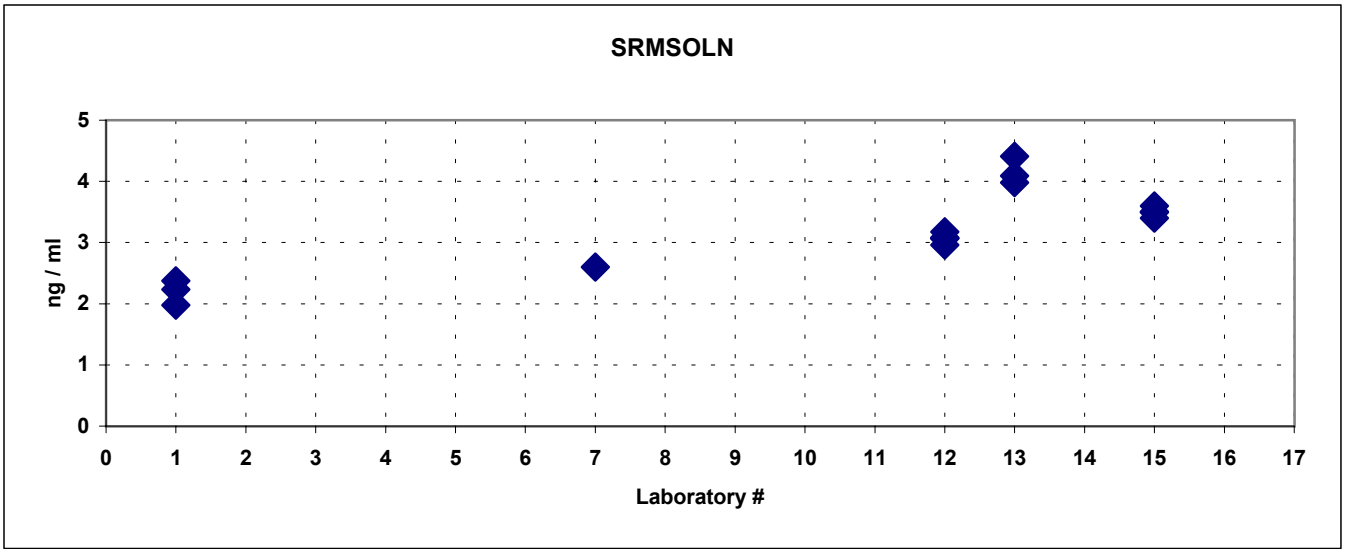
20
15
10
5
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

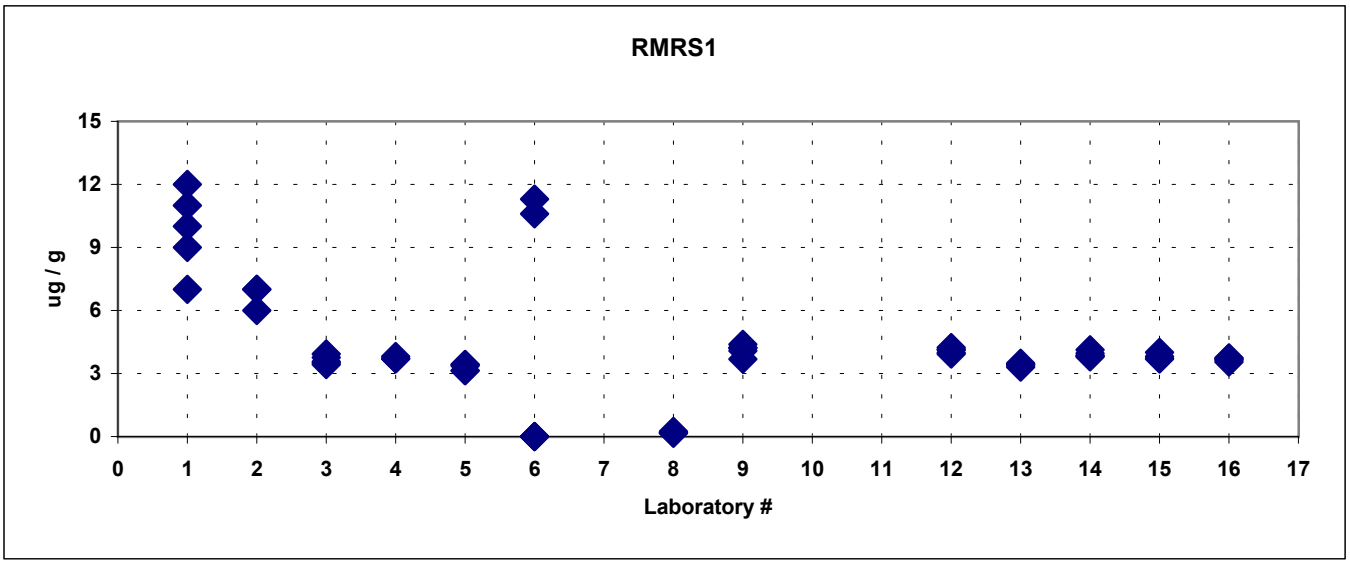

SRMSOLN
15
12
9
6
3
0
ng / ml
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

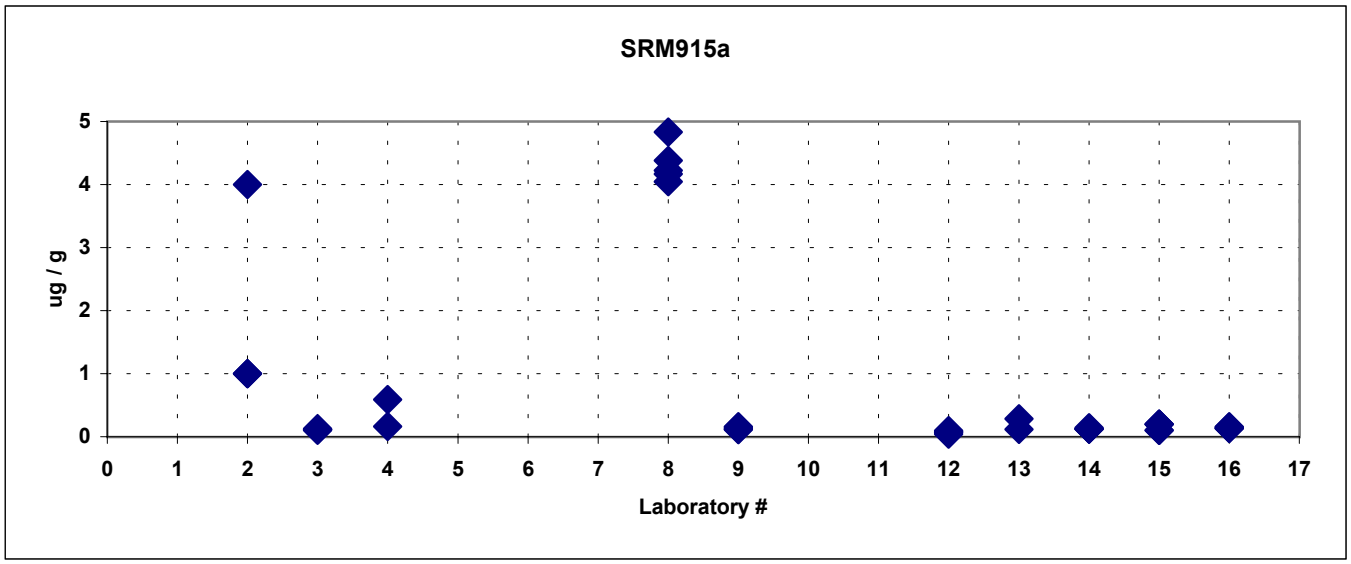
Cadmium

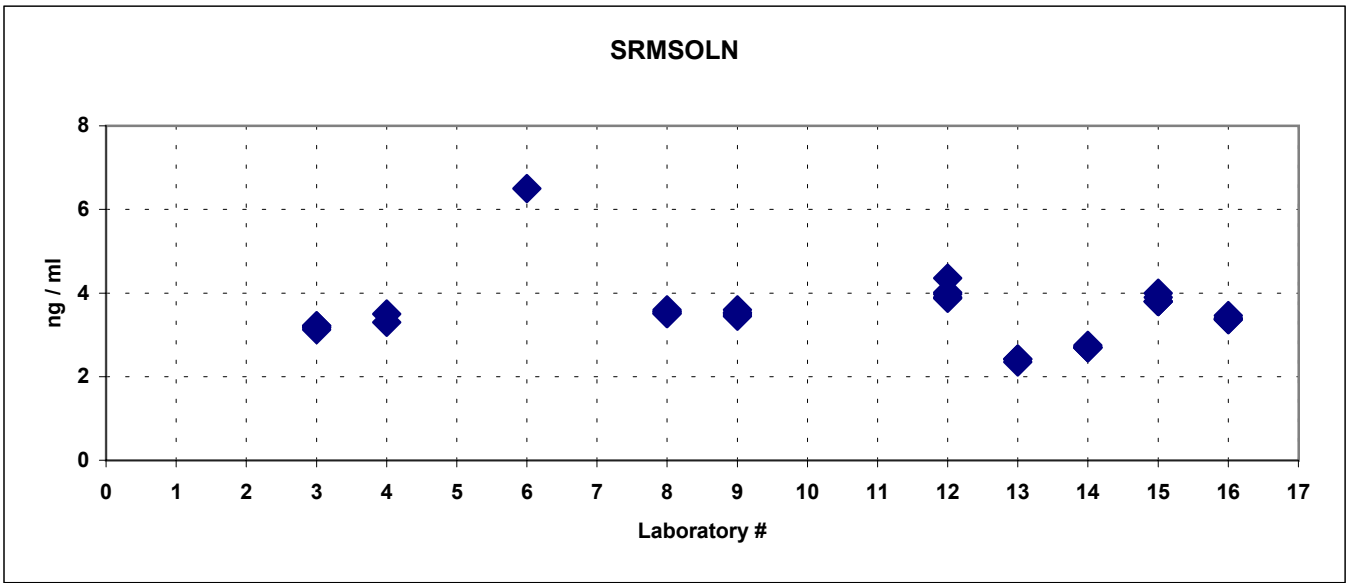

RMRS1
0.1
0.08
0.06
0.04
0.02
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

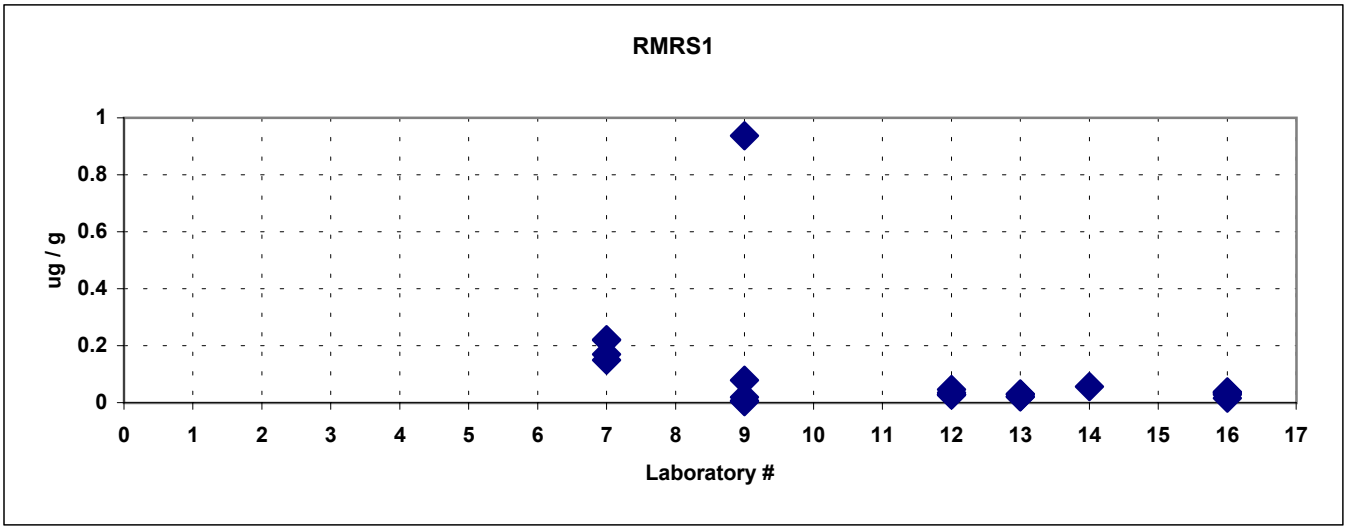

SRM915a
3
2
1
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

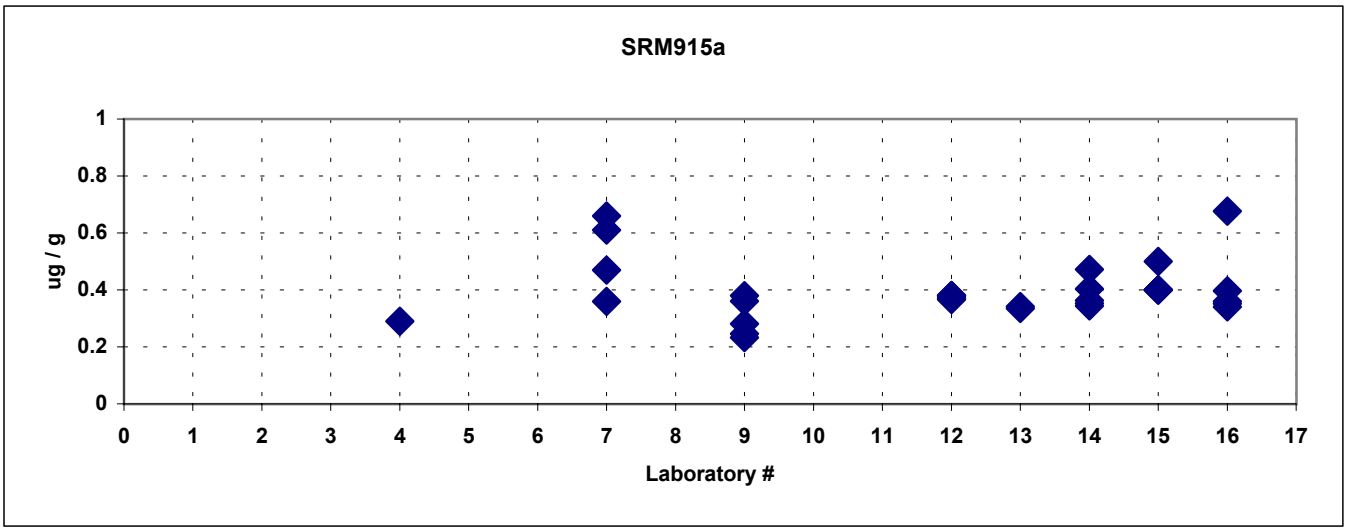

SRMSOLN
5
4
3
2
1
0
ng / ml
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

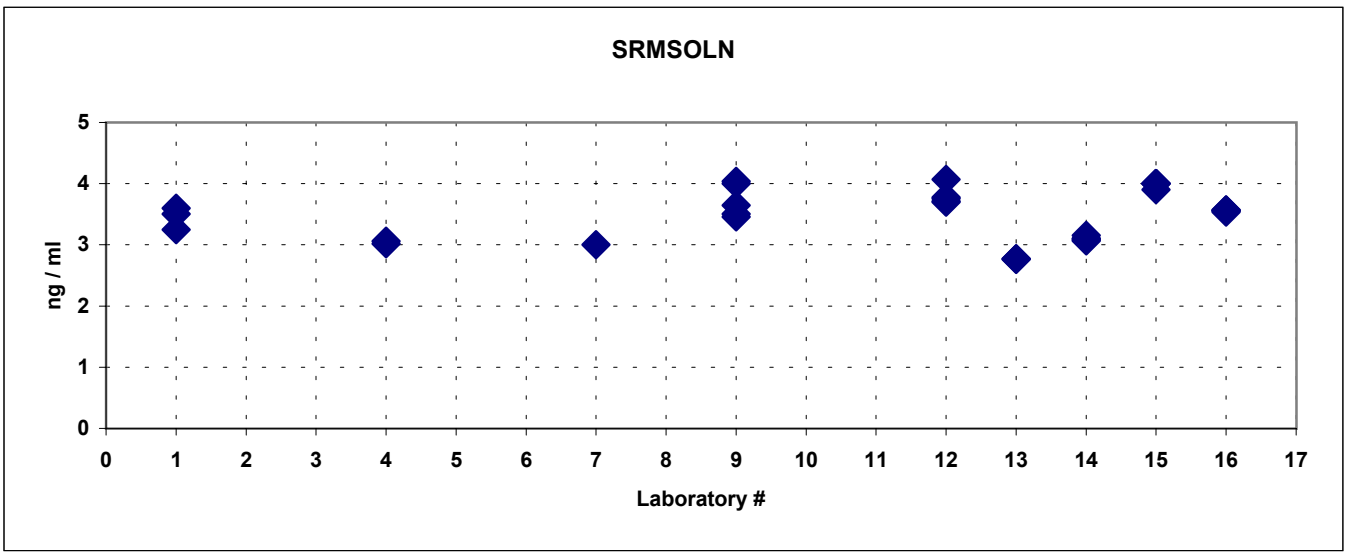

RMRS1
ug / g
Laboratory #

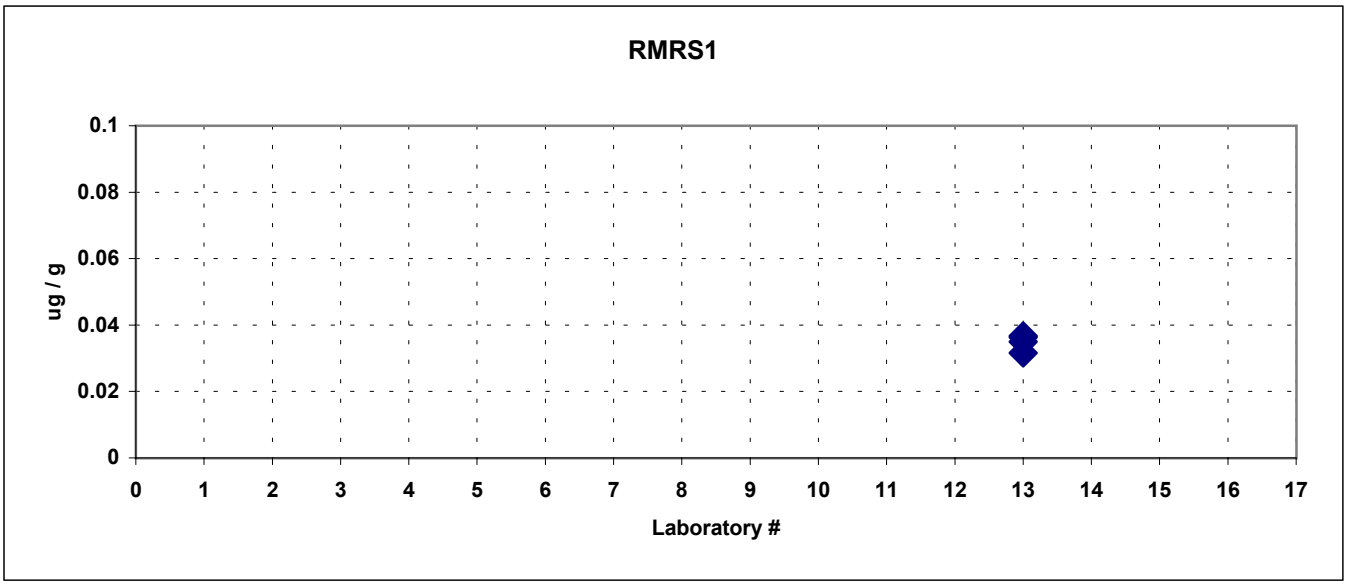

SRM915a
ug / g
Laboratory #

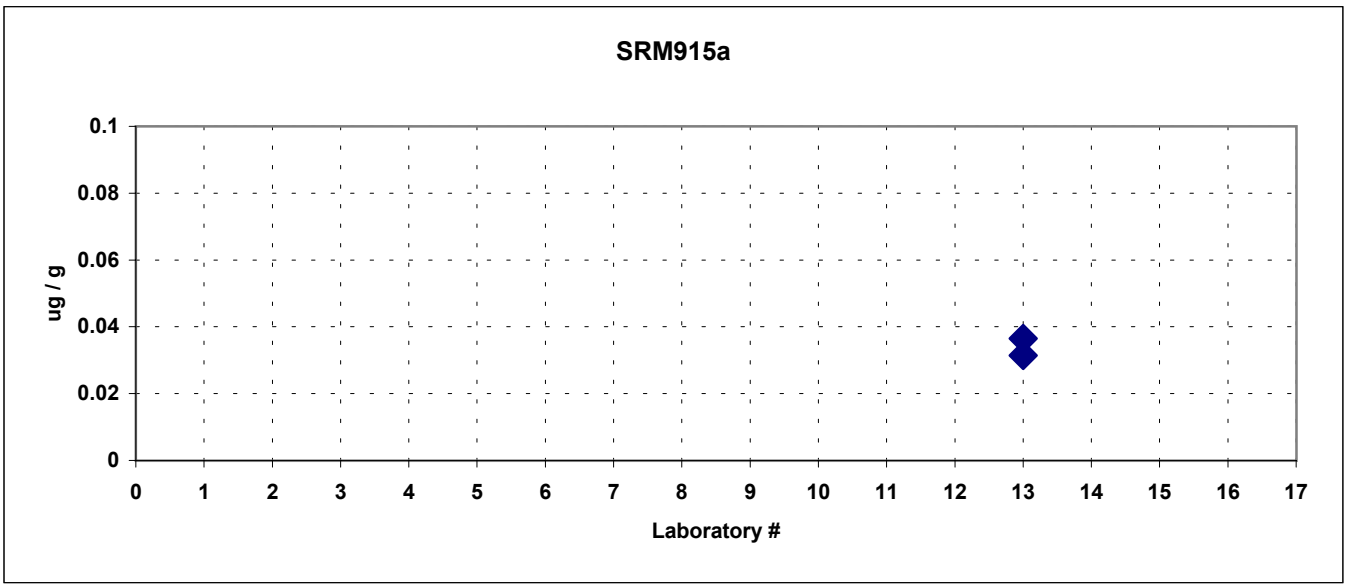

SRMSOLN
ng / ml
Laboratory #

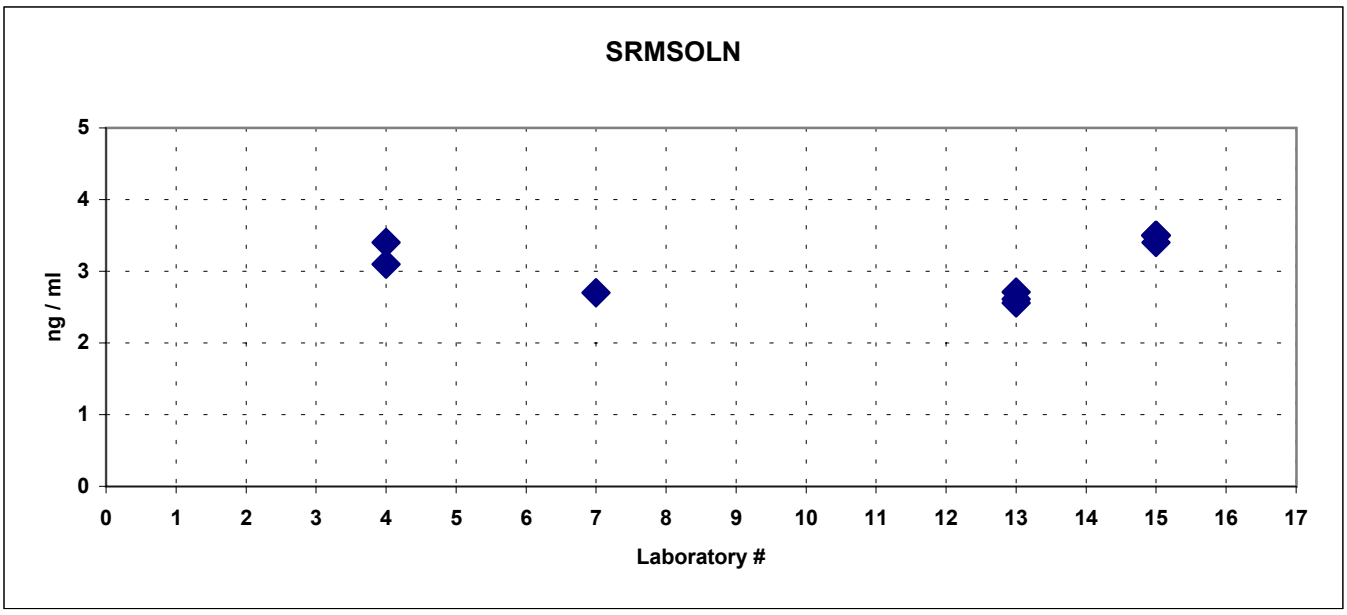

RMRS1
ug / g
1
0.8
0.6
0.4
0.2
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRM915a
ug / g
1
0.8
0.6
0.4
0.2
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
ng / ml
5
4
3
2
1
0
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


RMRS1
0.1
0.08
0.06
0.04
0.02
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRM915a
0.1
0.08
0.06
0.04
0.02
0
ug / g
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #


SRMSOLN
5
4
3
2
1
0
ng / ml
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory #

APPENDIX D

Z-SCORES
&
P-SCORES

Z-Scores for RMRS1

LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1				-0.2		-1.1		1.9	2.1	1.9	1.8	0.1		1.9	-1.3		2.1		
2								2.8	1.6	0.2		2.3		0.7	2.5		0.9		
3		-0.4				0.3		-0.4							0.3		-0.3		
4		0.5	0.6	-0.2	-0.4	-0.2	-0.7	-0.4	-0.8			-0.2		-0.6	-0.7		-0.2		
5			0.7	-0.4											-0.5		-0.4		
6			0.1	-0.3	-0.2	0.4									-0.8		2.5		
7			0.1	-0.2				-0.4	0.1		0.3				-0.8			-0.2	
8				-0.6		-1.0		-0.4	-0.7		-0.4	-0.3			-0.3		-1.6		
9		1.0	0.7	-0.4	0.3	0.8	-0.4	-0.4	0.2	-0.5	0.4	-0.3		-0.5	-0.2		-0.1	-0.2	
10			-2.0		1.2	0.5									0.2				
11			-1.8	3.7	-3.4			-0.3		-0.5	0.3								
12						0.6		-0.4	-0.7	-0.6	-0.8	-0.3			0.9		-0.1	-0.2	
13				2.6			0.2	-0.4	-0.1	-0.5	-0.7	-0.3		-0.6	-0.2		-0.4	-0.2	
14		-0.2		-0.2			1.5	-0.4	-0.3	-0.5	-0.6	-0.3		-0.6	2.0		-0.2	2.3	
15				-0.4	-0.8	-0.5		-0.4	-0.7						-0.3		-0.2		
16		-0.6	0.4	-0.4	0.4	-0.1	-0.9	-0.4	-0.7		-0.7	-0.2		-0.6	-0.3		-0.3	-0.2	

Z-Scores for SRM915a

LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1																			
2								3.7		2.5					2.9		0.8		
3						-1.3		-0.3									-0.5		
4				-0.4		0.1	-0.4	-0.2	-0.9		-0.5	0.0			-0.4		-0.3	-1.0	
5															-0.3				
6						1.2													
7				-0.4				-0.3	1.2		2.0							1.3	
8				0.5		-0.5		-0.2	-0.9		-1.0	-0.3			0.3		2.4		
9				-0.4		0.0	0.0	-0.3	1.3	-0.4	0.9	-0.5			-0.5		-0.4	-0.9	
10						0.4													
11				0.0			4.0	0.2		-0.4	2.4								
12						0.6		-0.3	-0.9	-0.4	-0.7	-0.3			-0.3		-0.5	-0.2	
13				3.7			-0.1	-0.2	1.8	-0.1	0.0	0.1					-0.4	-0.5	
14				-0.3			-0.1	-0.3	0.5	-0.2	-0.4	0.0			-0.3		-0.4	-0.1	
15				-0.4		-0.6		-0.3	-0.7		-0.7				-0.4		-0.4	0.3	
16				-0.5		0.1	-0.6	-0.3	-0.7	-0.4	-0.1	1.1					-0.4	0.3	

Z-Scores for SRMSOLN

LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1						-0.6	2.2	3.6	3.2	2.8	2.9	2.4	-2.1	-2.0	1.7	-1.5		-0.1	
2																			
3		1.0				0.1		-0.3									-0.4		
4		-0.6		-0.4		-1.3	-0.2	-0.3	-0.4		-0.9	-0.8		0.1	-0.3		-0.1	-1.1	
5				-0.3											0.8				
6				1.7		0.3		-0.1							0.9		4.5		
7				-0.3				-0.4	-0.5		-0.2					-0.9		-1.2	
8				-0.4		-0.6		-0.3	-0.5		-0.4	-0.6			0.6		0.1		
9		1.1		-0.3		-0.2	0.0	-0.3	0.5	-0.3	0.6	-0.8	0.5	0.6	-1.7		0.1	0.6	
10																			
11				2.1			-1.6	0.2		-0.4	1.1								
12						-0.7		-0.2	-0.4	-0.4	-0.6	-0.5			0.1	-0.2	0.8	0.8	
13				3.4		2.9	0.9	-0.1	0.0	-0.4	0.3	0.6	1.2	0.2		1.5	-1.6	-1.8	
14		-1.2		-0.3			-0.8	-0.3	-0.5	-0.4	-0.9	-0.8	-0.3	-0.8	-1.0		-1.1	-1.0	
15		-0.7		-0.3		0.3	-0.4	-0.3	-0.4		-0.5	-0.2		1.0	0.1	0.4	0.6	1.2	
16		0.1		-0.4		0.2	-0.3	-0.3	-0.4	-0.3	-0.2	1.1	0.3	0.2			-0.1	0.2	

P-Scores for RMRS1

LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1				0.1		1.8		0.8	0.4	1.0	1.2	0.2		0.6	0.7		0.7		
2								0.7	1.5	0.3		2.9		0.3	0.3		0.2		
3		0.1				0.8		0.0							0.4		0.1		
4		1.3	0.3	0.0	0.1	0.9	0.2	0.0	0.0			0.0		0.0	0.1		0.0		
5			0.1	0.0											0.2		0.1		
6			0.3	0.4	0.2	0.2									0.1		0.2		
7			0.0	0.0				0.0	0.1		0.1				0.1			0.0	
8				0.1		1.3		0.0	0.0		0.0	0.0			0.4		0.0		
9		1.6	0.2	0.0	0.2	0.1	0.1	0.0	0.1	0.0	0.5	0.1		0.0	0.2		0.1	0.0	
10			0.6		1.2	0.2									0.6				
11																			
12						0.4		0.0	0.0	0.0	0.0	0.0			0.2		0.0	0.0	
13				0.3			0.3	0.0	0.1	0.0	0.0	0.0		0.0	0.0		0.0	0.0	
14		0.3		0.1			0.8	0.0	0.0	0.0		0.0		0.0	0.3		0.1	3.5	
15				0.0	0.0	0.4		0.0	0.0						0.2		0.1		
16		0.3	0.1	0.0	0.1	0.2	0.2	0.0	0.0		0.0	0.1		0.0	0.1		0.0	0.0	

P-Scores for SRM915a

LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1																			
2								0.6		1.4					1.5		1.2		
3						0.8		0.0									0.0		
4						0.5			0.1						0.1		0.2		
5															0.0				
6						0.2													
7				0.1				0.0	0.3		0.2							1.4	
8				0.0		1.5		0.0	0.0		0.0	0.1			0.5		0.2		
9				0.0		1.0	0.2	0.0	0.2	0.0	0.1	0.0			0.1		0.0	0.7	
10						0.1													
11																			
12						0.4		0.0	0.0	0.0	0.0	0.1			0.0		0.0	0.1	
13				0.4			0.1	0.0	0.1	0.0	0.0	0.0					0.1	0.0	
14				0.1			0.3	0.0	0.0	0.0	0.5	0.7			0.0		0.0	0.5	
15				0.0		0.7		0.0	0.1		0.0				0.1		0.0	0.4	
16				0.0		0.2	0.1	0.0	0.1	0.0	0.3	2.1					0.0	1.4	

P-Scores for SRMSOLN																			
LAB #		Li	Na	Mg	K	Ca	Cr	Mn	Co	Ni	Cu	Zn	As	Rb	Sr	Cd	Ba	Pb	U
1						0.8	1.2	1.4	0.4	0.4	0.3	0.4	0.5	1.6	0.4	0.3		0.4	
2																			
3		0.2				0.1		0.0									0.1		
4		0.2				0.2	0.5	0.0	0.2					0.0	0.4		0.2	0.1	
5				0.0											0.1				
6																			
7																			
8				0.0		0.1		0.0	0.0		0.4	0.1			0.2		0.1		
9		0.2		0.0		0.2	0.1	0.0	0.0	0.1	0.2	0.1	0.1	0.1	0.1		0.1	0.7	
10																			
11																			
12						0.1		0.0	0.1	0.0	0.1	0.1			0.2	0.1	0.3	0.4	
13				0.7		0.0	0.3	0.0	0.1	0.0	0.2	0.2	0.3	0.4		0.3	0.1	0.0	
14		0.2		0.0			0.1	0.0	0.0	0.0	0.1	0.0	0.1	0.1	0.0		0.0	0.1	
15		0.9		0.0		0.3	0.1	0.0	0.1		0.0	0.0		0.1	0.0	0.1	0.1	0.1	
16		0.1		0.0		0.2	0.1	0.0	0.0	0.0	0.0	0.5	0.1	0.0			0.1	0.0	

APPENDIX E

LABORATORY METHODS

LABORATORY METHODS

DRYING		LAB #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Temperature	oC		70	70	---	---	200	---	200	18	105	45	105	105	---	---	105	25
Duration	hr		9	9	---	---	4	---	4	24	6	10	24	24	---	---	2	72
Vacuum (Y/N)	Y/N		N	N	---	---	Y	---	N	N	---	N	N	N	---	---	N	Y
SOLID STATE																		
Sample weight	mg		---	150	---	---	---	---	---	---	---	150	---	---	---	---	---	---
Binder/Flux weight	mg		---	150	---	---	---	---	---	---	---	150	---	---	---	---	---	---
Pelletizing Method																		
Binder			---	Spurr Resin	---	---	---	---	---	---	---	Spurr Resin	---	---	---	---	---	---
Fusion			---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
SOLUTION																		
Sample weight	mg		10	---	10	3	50	30	50	10	50	---	50	10	10	10	30	25
Reagents																		
HNO3			X	---	X	X	X	---	X	X	X	---	X	X	---	X	X	X
HCl			---	---	---	X	---	---	---	---	---	---	---	---	---	---	---	---
Other			---	---	---	---	---	X	---	---	---	---	---	---	---	---	---	---
Digestion Method																		
Open vessel			X	---	X	X	X	---	X	X	X	---	X	X	---	X	X	X
Closed vessel			---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Microwave oven			---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Heat			---	---	---	---	---	---	---	---	X	---	---	---	---	---	---	---
Room temp			X	---	X	X	X	X	X	X	---	---	X	X	---	X	---	X
INSTRUMENTATION																		
FAAS			---	---	---	---	---	---	---	---	Na,K,Ca	---	---	---	---	---	---	Na,K
GFAAS			---	---	---	---	---	---	X	---	---	---	X	---	---	---	---	---
ICPMS - Quadrupole			X	X	X	---	---	X	X	X	X	---	---	X	X	X	X	X
ICPMS - HiResolution			---	---	---	X	---	---	---	---	---	---	---	---	---	---	---	---
ICPAES			---	---	---	---	X	Ca	Na,Mg,Sr	---	---	---	---	---	---	---	---	---
WDEM			---	---	---	---	---	---	---	---	---	X	---	---	---	---	---	---
Laser (Nd:YAG)			---	X	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Calibration method																		
Standard additions			X	---	X	---	---	---	X	---	---	---	X	---	---	---	---	---
External standards			X	X	X	X	X	X	X	X	X	X	---	X	---	X	X	X
NOTES:				1			2											

Note 1 STD: NIST 610 & 612; Semi-quantitative; Ca Internal Standard
Note 2 Ultrasonic Nebulizer

ISOTOPES USED FOR QUANTIFICATION

Element	Li	Be	B	Na	Mg	Al	Si	K	Ca	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	As	Rb	Sr	Ag	Cd	Cs	Ba	Tl	Pb	U
Z	3	4	5	11	12	13	14	19	20	23	24	25	26	27	28	29	30	31	33	37	38	47	48	55	56	81	82	92
Lab #																												
1	-	-	-	-	25	-	-	-	43	-	52	55	-	59	60	65	64	-	75	85	88	-	114	-	138	-	208	-
2	-	-	-	-	25	-	-	-	43	-	52	55	-	59	60	65	64	-	75	85	88	-	114	-	138	-	208	-
3	7	-	-	-	-	-	-	-	48	-	-	55	-	-	-	-	-	-	-	-	86	-	-	-	138	-	-	-
4	7	-	-	23	24	-	-	39	44	-	52	55	57	59	-	65	68	-	-	85	88	-	-	-	138	-	208	238
5	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
7	-	-	-	-	-	-	-	-	-	-	-	55	-	59	-	65	66	-	-	-	-	-	111	-	-	-	208	238
8	-	-	-	-	25	27	-	-	43	-	-	55	-	59	-	65	68	-	-	-	86	-	-	-	137	-	-	-
9	7	-	-	23	26	-	-	39	42	-	53	55	-	59	62	65	66	-	75	85	88	-	-	-	138	-	208	-
10	-	-	-	23	-	-	-	39	40	-	-	-	56	-	-	-	-	-	-	-	88	-	-	-	-	-	-	-
11	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
12	-	9	11	-	-	27	-	-	44	-	-	55	-	59	62	63	68	-	-	-	88	-	114	-	138	-	208	-
13	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
14	7	-	-	-	24	-	-	-	-	-	52	55	-	59	58	63	66	-	75	85	86	-	-	-	138	-	208	238
15	6	-	-	23	24	27	29	39	42	51	53	55	-	59	62	63	66	71	75	85	88	-	111	133	138	205	208	238
16	7	-	-	-	26	-	-	-	44	-	53	55	-	59	62	65	68	-	75	85	88	-	-	-	138	-	208	-

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STANDARD
MAIL A

Publications and Reports of the Northeast Fisheries Science Center

The mission of NOAA's National Marine Fisheries Service (NMFS) is "stewardship of living marine resources for the benefit of the nation through their science-based conservation and management and promotion of the health of their environment." As the research arm of the NMFS's Northeast Region, the Northeast Fisheries Science Center (NEFSC) supports the NMFS mission by "planning, developing, and managing multidisciplinary programs of basic and applied research to: 1) better understand the living marine resources (including marine mammals) of the Northwest Atlantic, and the environmental quality essential for their existence and continued productivity; and 2) describe and provide to management, industry, and the public, options for the utilization and conservation of living marine resources and maintenance of environmental quality which are consistent with national and regional goals and needs, and with international commitments." Results of NEFSC research are largely reported in primary scientific media (*e.g.*, anonymously-peer-reviewed scientific journals). However, to assist itself in providing data, information, and advice to its constituents, the NEFSC occasionally releases its results in its own media. Those media are in four categories:

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