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REPORT ON THE INTERCOMPARISON RUN FOR THE DETERMINATION OF TRACE AND MINOR ELEMENTS IN LICHEN MATERIAL

IAEA-336

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Appendices

1. INTRODUCTION

In the last few years, a variety of biological materials have been proposed and used as biomonitors of environmental pollution. For example, lichens have been used in air pollution studies to monitor the levels of trace metals and organic compounds [1-3]. During analyses of these biomonitors, it is desirable to have included the analysis of a quality control or certified reference material that matches the sample as closely as possible with respect to its matrix and the concentrations of the constituents of interest to demonstrate the reproducibility and/or accuracy of the method. In the case of biomonitors, the number and type of control or reference materials is very limited. The present intercomparison exercise was organized to characterize a lichen material (identified as IAEA-336) which could then be used as a quality control material for the analysis of lichen biomonitors. The material was characterized for its contents of major and minor elements using both nuclear and non-nuclear analytical techniques. The recommended and information values for this lichen were established using standard statistical techniques that had been used by the Agency in many of its intercomparison evaluations.

The lichen material described in this report was produced for the International Atomic Energy Agency (IAEA) by the Instituto de Ciencias e Engenharia Nucleares (ITN) in Sacavém, Portugal [4]. This report presents the analytical results that were submitted for this lichen by the participants of the intercomparison exercise together with a summary of the statistical evaluation of the results. Individual participants should be able to use the results to assess their performance relative to the other participants of the exercise and modify their laboratory's analytical procedures where necessary.

2. SCOPE OF THE INTERCOMPARISON

The intercomparison was publicized both by letters sent out to individuals and laboratories that had previously participated in such intercomparisons and through advertisement in the IAEA's Analytical Quality Control Services Catalogue.

Each participant received one bottle of the lichen material, along with an information sheet and a reporting form. Participants were asked to determine as many elements as possible from the following list: Al, As, Br, Cd, Cl, Co, Cu, Hg, I, K, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Si, Sm, Th, U, and Zn. Participants were also invited to submit results for any additional analytes which they had determined.

In addition, each participant received a sample of IAEA-359, cabbage, to be analyzed in the same analytical run as the lichen material. The intent was to use the IAEA-359 as a quality control material to be used in the evaluation of each laboratory's results for the lichen. Unfortunately the intercomparison exercise to characterize IAEA-359 was not completed in time for this intercomparison and therefore it could not be used as a quality control material. For this reason results submitted for IAEA-359 have not been included in this evaluation but will be combined and evaluated together with the results from the original IAEA-359 characterization exercise to determine overall recommended and information values for the analytes reported.

In total, 42 laboratories from 26 countries submitted results for this intercomparison. The participants reported data for up to 47 major, minor and trace elements. The cut-off date for

inclusion of results to be considered for this evaluation was December 1994.

3. DESCRIPTION OF THE MATERIAL

The epiphytic lichen *Evernia prunastri (L.) Ach.* was collected by hand. The lichen was taken from the *Cistus ladanifer* and *Quercus* species of tree. The collection areas in Portugal were Gavião (centre of Portugal), Ourique and Serra do Cladeirão (south of Portugal) which were expected to yield lichen with low levels of metal contamination. Approximately 25 kg of raw lichen material were collected. After removal of debris and other epiphytic lichen species, the remaining lichen was washed in deionized water and oven-dried at 40 ^{o}C for 24 hours. The lichen was ground using a Teflon "mikro-dismembrator" mill. The material which passed through a 125 µm sieve was mixed in a rotating plastic drum and yielded sufficient material for approximately 800 bottles each containing 20g. In order to confer long-term stability by inhibiting microbial growth, the material was sterilized by irradiation to a dose of 12 kGy using a ^{60}Co radiation source.

To evaluate the homogeneity of this material, a number of bottles were selected at random for analysis using non-destructive techniques. A total of 20 bottles were sampled for analysis using INAA and 12 for X-ray fluorescence with two sub-samples, each weighing approximately 100 mg, taken from each bottle to evaluate in-bottle homogeneity. The results, obtained by a one-way variance analysis of the INAA measurements (two sub-samples per bottle), indicated good in-bottle and between bottle homogeneity for K, Na, Mn, Br, As, La and Sm [4]. The overall relative standard deviation of the 40 results averaged approximately 6% with a range of 3-10%. Of this 6% total standard deviation, approximately 4% on average was attributed to the inhomogeneity of the sample, with a maximum of 6% for As. Additional measurements using X-ray fluorescence for Br, Ca, Fe, Sr, and Zn indicated that the relative standard deviation arising from inhomogeneity ($s_{hom}^2 = s_{total}^2 - s_{meas}^2$) ranged from 1.1% for Ca up to 4.4 % for Sr [5]. Although the relative standard deviation due to the heterogeneity of the sample will vary from element to element, it was deemed prudent to include an extra component of relative uncertainty of 5% in the calculation of the confidence intervals of *all* the information and recommended values.

4. ANALYSES AND REPORTING

The participants were requested to determine the total element content in suitably sized portions (greater than or equal to 100 mg) of the sample by their usual analytical techniques. They were requested to make at least three, but preferably six, separate determinations of each element and to report the results together with a short description of the analytical method used. A reporting form, attached to the information sheet, was provided for this purpose. For each element, the participants were requested to report:

1. the average weight of the sample taken for analysis;
2. the limit of detection of the analytical method used, defined as the concentration equivalent to 3 times the standard deviation of the analytical blank signal;
3. the method code corresponding to the analytical method. (This was to be selected from a list supplied with the reporting form, which is given on page 18);
4. the water content of the material. (Participants were requested to determine the moisture content of an independent sub-sample by drying it for two hours at 100 □C and to report

all analytical results on a dry weight basis).

5. EVALUATION OF THE RESULTS

The reported results were first evaluated by a computer program, used by the IAEA for intercomparisons [6, 7]. For each analyte, all laboratory means were subjected to four statistical tests applied consecutively (Dixon's, Grubb's, coefficient of skewness, and coefficient of kurtosis). Any result failing one, or more, test was rejected as an outlier and the remaining data were re-tested until no further outliers were detected. These tests were applied at a significance level of 95%. The remaining laboratory means were used to calculate an overall mean, standard deviation, standard error and 95% confidence interval of the mean for each analyte.

After the overall means were calculated, a number of additional criteria were used to establish a mean as a recommended, information value. These criteria included: concentration of the analyte, number of results submitted, number of different analytical methods applied, standard deviation of the accepted laboratories' means and percentage of laboratories eliminated as outliers. In addition, any biases between the analytical methods were considered and, if they were significant (t- test), no recommended or information value was given.

6. CRITERIA FOR ESTABLISHING RECOMMENDED AND INFORMATION VALUES

Recommended values for individual elements were assigned if the results for the analyte in question met all the following criteria;

1) the overall mean was based on at least 10 accepted laboratory means;
2) two or more independent analytical methods were used to provide the overall mean;
3) the percentage of laboratory means rejected as outliers was less than or equal to 20% of the entire set of means;
4) the relative uncertainty of the overall mean (relative standard deviation) did not exceed 20% for trace elements (defined here as < 500 mg/kg) and 10% for major elements (> 500 mg/kg);
5) No statistically significant bias could be detected between the different methods.

Information values for individual elements were assigned if the results for the analyte in question failed to meet any of the above criteria but met all the following criteria;

1) the relative uncertainty of the mean was less than 30%;
2) the percentage of total outlying results did not exceed 30%;
3) the overall mean was based on a minimum of 5 laboratories means;
4) two or more analytical methods were used to provide the overall mean.
5) No statistically significant bias could be detected between the different methods.

7. RESULTS AND DISCUSSION

7.1 General

The results of this intercomparison are presented in three ways:

- as a summary table
- as tables of results for each analyte sorted alphabetically
- as graphical representations of results (S-plots) for each analyte sorted alphabetically.

The summary table (Appendix I) provides details concerning the total number of laboratories that submitted results for each element, the range of laboratory means reported, the number of outliers rejected together with the mean, range of the accepted laboratories' means, their standard deviation [%] and the corresponding confidence intervals. It should be noted that data presented in this appendix has not been rounded up (as is the case for data presented in this report and on the reference sheet).

Appendix II lists all the results reported by all the laboratories. The terms used in the tables are defined as follows:

Mean:	This is the overall mean value calculated from the accepted laboratories' means. It is only reported when an information or a recommended value is assigned.
Upper Limit:	This is the upper limit of the 95% confidence interval of the recommended or information value. It should be noted that the confidence interval was calculated from the combination of the standard deviation of the mean value and an additional 5% to account for any variation due to sample inhomogeneity.
Lower Limit:	This is the lower limit of the 95% confidence interval of the recommended or information value. It should be noted that the confidence interval was calculated from the combination of the standard deviation of the mean value and an additional 5% to account for any variation due to sample inhomogeneity.
Type:	This refers to the status assigned to the mean value for each element determined according to the intercomparison evaluation criteria (see above). The letter "R" corresponds to "recommended" value, the letter "I" corresponds to "information" value and the letter "N" indicates that the final set of results failed both set of criteria.
Lab. ID (#):	For anonymity, each laboratory was assigned a unique code number where the code sequence was randomized to ensure that it did not correspond to the list of participants given in Appendix IV of this report. A sub-code (A, B, C, etc.) was added when a laboratory reported two or more sets of results for the same element using different methods.
Value 1 - Value 6:	This corresponds to the number of results that a participant submitted for a particular element in the intercomparison.
Det. limit:	This value is the detection limit reported by the individual participant for each element. Participants were requested to report it based on 3 times the standard deviation of the analytical blank.
Method:	This is a method code used to identify the method employed in the individual laboratories. A detailed description of each method code is given on page 18. *Note: The assignment of a method code is based on the information provided*

by the participant.

Lab mean: The arithmetic mean calculated from all individual results supplied by a laboratory for a given element.

Stdev: This is the standard deviation of a laboratory's result.

Z-score: This is a parameter whose value indicates the level of agreement that a laboratory mean has with the overall mean. It is defined as the difference between the individual laboratory mean and the accepted recommended value divided by the standard deviation of the population (IUPAC recommendation [8]).

Outlying values are indicated with gray shading in Appendix II and denoted by a solid symbol graphically in Appendix III. The graphical presentation (S-plots) of all the laboratory means reported for a particular element are presented in Appendix III. The laboratory means are plotted in ascending concentration on the y-axis with their corresponding laboratory code noted along the x-axis. The error bars represent the standard deviation of the calculated laboratory mean. The code above the error bar represents the method code used by the particular laboratory (see page 18). The overall mean of all accepted laboratories' means is given and the expanded confidence interval (to include the 5% assigned to inhomogeneity) is shaded. S-plots are presented for all elements where 5 or more accepted laboratory means exist. In the case of elements for which no recommended or information values are assigned, no confidence interval is given.

7.2 Discussion of results for Elemental analyses

Although only 42 laboratories took part in this intercomparison, it was only possible to establish recommend values for 19 elements and information values for 14 elements following the criteria stated in section 6.

7.2.1 Elements with Recommended Values

Arsenic

Of the twenty laboratories that reported results for As, three laboratories were excluded as outliers. The following techniques were used by the laboratories that provided the accepted results: Graphite furnace AAS with and without Zeeman background correction (2), AAS using the hydride generation technique (2), RNAA (2), INAA (9) and ICP-MS (2). No bias between the various methods was found and all method means were within 10% of the overall mean. The recommended value for As is 0.63 mg/kg with a confidence interval of 0.55 - 0.71 mg/kg.

Barium

Of the twelve laboratories that reported results for Ba, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: ICP-OES (3), ICP-MS (2) and INAA (6). Although the mean value of the ICP-OES results appears different from the mean values obtained by INAA and ICP-MS (Figure 1), statistical evaluation showed no significant difference between the methods. The recommended value for Ba is 6.4 mg/kg with a confidence interval of 5.3 - 7.5 mg/kg.

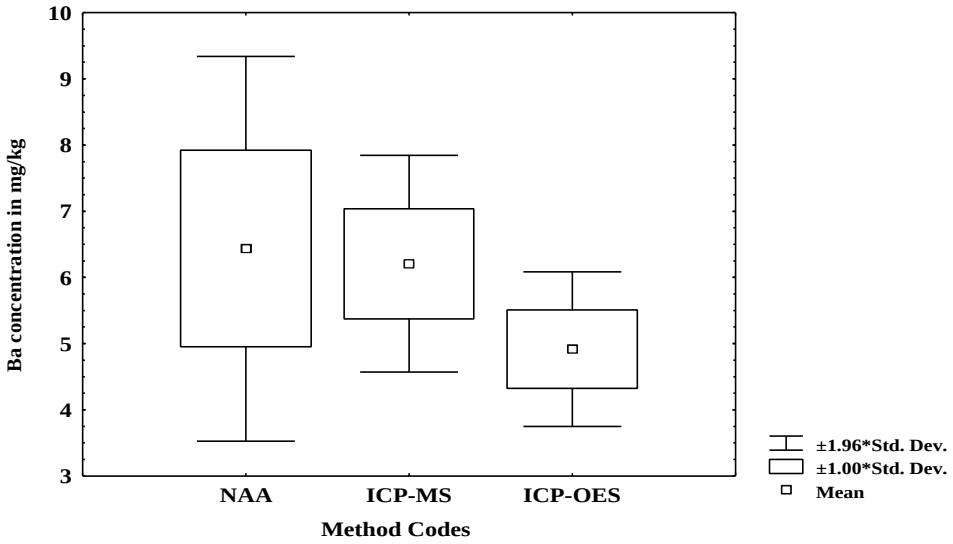

Figure 1 *Mean Ba results obtained by different methods*

Bromine

Of the nineteen laboratories that reported results for Br, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: NAA (13), XRF (3) and PIXE (2). The recommended value for Br is 12.9 mg/kg with a confidence interval of 11.2 - 14.6 mg/kg.

Cerium

Of the fourteen laboratories that reported results for Ce, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: NAA (8) and ICP-MS (5). It is noteworthy that this is the first time that it has been possible to establish a recommended value for Ce in an IAEA biological reference material. This is due to the participation of laboratories using ICP-MS. In the past, the lack of a second independent method to complement NAA always prevented such a classification. The recommended value for Ce is 1.28 mg/kg with a confidence interval from 1.11 - 1.45 mg/kg.

Cobalt

Of the twenty-one laboratories that reported results for Co, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: NAA (15), ICP-MS (2), Voltammetry (1) and graphite furnace AAS (1). The recommended value for Co is 0.29 mg/kg with a confidence interval of 0.24 - 0.34 mg/kg.

Cesium

Of the sixteen laboratories that reported results for Cs, three laboratories were rejected as outliers. The following techniques were used to provide the accepted results: NAA (11) and ICP-MS (2). The standard deviation of 6.3% for the laboratories' mean of all accepted results showed that these measurements seem to be well under control even when performed with different techniques and in different laboratories. The recommended value for Cs is 0.110 mg/kg with a confidence interval of 0.097 - 0.123 mg/kg.

Copper

Of the twenty-six laboratories that reported results for Cu, five laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame (4) and graphite furnace (2) AAS, Voltammetry (1), XRF (3), PIXE (2), RNAA (1), INAA (1), ICP-MS (2), SS-MS

(1) and ICP-OES (4). The recommended value for Cu is 3.6 mg/kg with a confidence interval of 3.1 - 4.1 mg/kg.

Iron

Of the thirty-nine laboratories that reported results for Fe, four laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame AAS (5), XRF (2), PIXE (4), NAA (15), ICP-MS (2), and ICP-OES (7). No significant difference between destructive and non-destructive techniques was observed which is an indication that complete dissolution of this matrix could be achieved with the usual digestion techniques. The recommended value for Fe is 430 mg/kg with a confidence interval of 380 - 480 mg/kg.

Mercury

Of the fifteen laboratories that reported results for Hg, no laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: cold vapour AAS (5), RNAA (3) and INAA (7). The recommended value for Hg is 0.20 mg/kg with a confidence interval of 0.16 - 0.24 mg/kg.

Potassium

Of the twenty-eight laboratories that reported results for K, four laboratories (all of which used X-ray techniques) were rejected as outliers. The following techniques were used to provide the accepted results: flame AAS (7), XRF (2), PIXE (1), NAA (13), and ICP-OES (1). The recommended value for K is 1840 mg/kg with a confidence interval of 1640 - 2040 mg/kg.

Lanthanum

Of the thirteen laboratories that reported results for La, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: INAA (10) and ICP-MS (2). The recommended value for La is 0.66 mg/kg with a confidence interval of 0.56 - 0.76 mg/kg.

Manganese

Of the thirty-three laboratories that reported results for Mn, four laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame AAS (5), XRF (4), PIXE (4), INAA (9), ICP-MS (2), and ICP-OES (5). The good agreement between the techniques can be seen in Figure 2. The recommended value for Mn is 63 mg/kg with a confidence interval of 56 - 70 mg/kg.

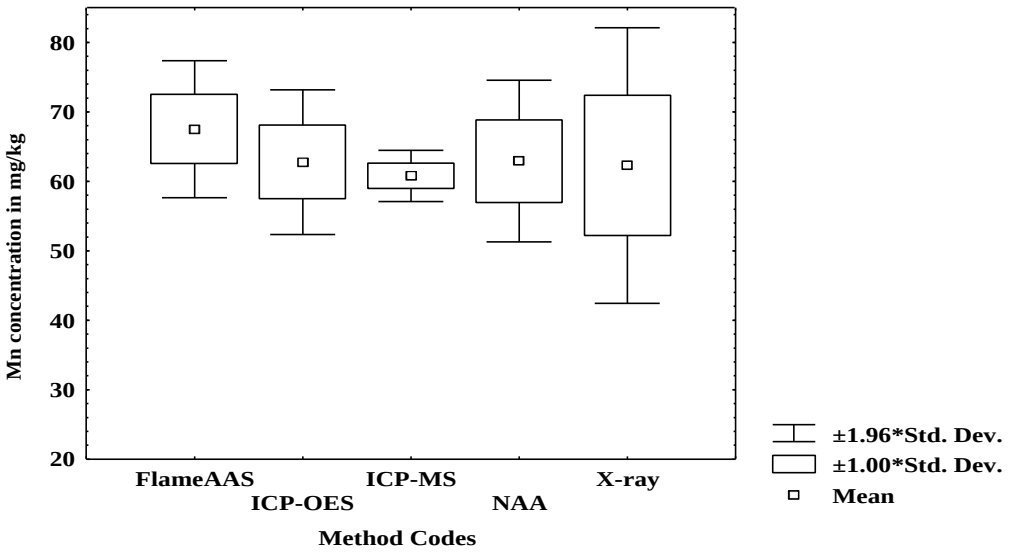

Figure 2 *Mean Mn results from all analytical techniques used*

Sodium

Of the twenty-five laboratories that reported results for Na, five laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame AAS (4), NAA (14), ICP-MS (1) and ICP-OES (1). The high number of outlying results (3 from 7) for flame AAS was surprising and might be an indication that contamination problems are not under control. The recommended value for Na is 320 mg/kg with a confidence interval of 280 - 360 mg/kg.

Antimony

Of the thirteen laboratories that reported results for Sb, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: RNAA (2), INAA (9), and ICP-OES (1). The recommended value for Sb is 0.073 mg/kg with a confidence interval of 0.063 - 0.083 mg/kg.

Selenium

Of the fifteen laboratories that reported results for Se, three laboratories were rejected as outliers. The following techniques were used to provide the accepted results: hydride generation AAS (1), RNAA (1), INAA (9) and ICP-MS (1). The recommended value for Se is 0.22 mg/kg with a confidence interval of 0.18 - 0.26 mg/kg.

Samarium

Of the fifteen laboratories that reported results for Sm, no laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: NAA (13) and ICP-MS (2). The recommended value for Sm is 0.106 mg/kg with a confidence interval of 0.092 - 0.120 mg/kg.

Strontium

Of the twenty-one laboratories that reported results for Sr, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: XRF (2), PIXE (2), INAA (6), ICP-MS (3) and ICP-OES (6). The mean values of these different techniques were in good agreement (Figure 3) although for PIXE and XRF large standard deviations were reported. The recommended value for Sr is 9.3 mg/kg with a confidence interval of 8.2 - 10.4 mg/kg.

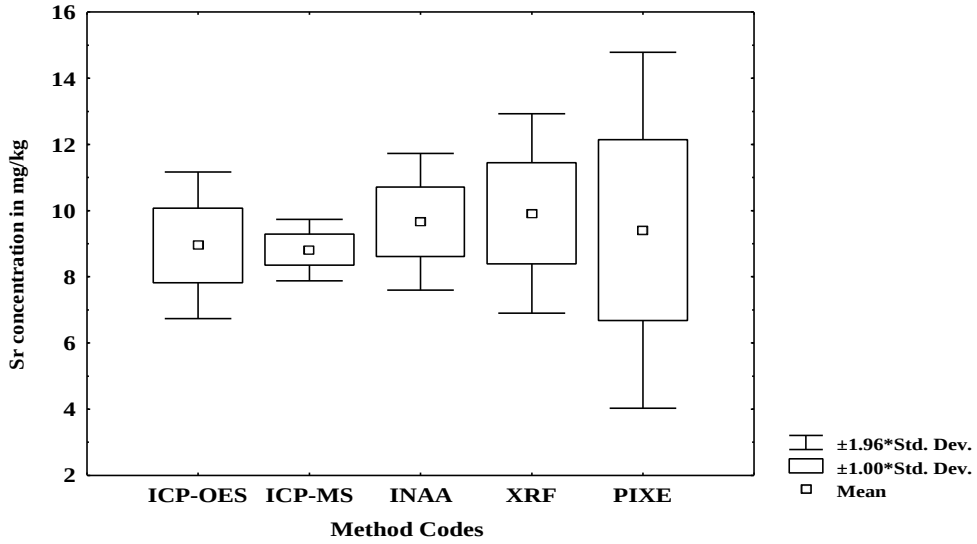

Figure 3 *Mean Sr results for different analytical techniques*

Thorium

Of the seventeen laboratories that reported results for Th, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: INAA (14) and ICP-MS (2). The recommended value for Th is 0.14 mg/kg with a confidence interval of 0.12 - 0.16 mg/kg.

Zinc

Of the forty laboratories that reported results for Zn, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame AAS (4), Voltammetry (1), TRXRF (1), XRF (3), PIXE (4), INAA (15), RNAA (1), fast neutron NAA (1), ICP-MS (2), SS-MS (1) and ICP-OES (5). Except for the individual results for SS-MS and Voltammetry, the mean values for all the methods fall within the confidence interval (Figure 4). The recommended value for Zn is 30.4 mg/kg with a confidence interval of 27.0 - 33.8 mg/kg.

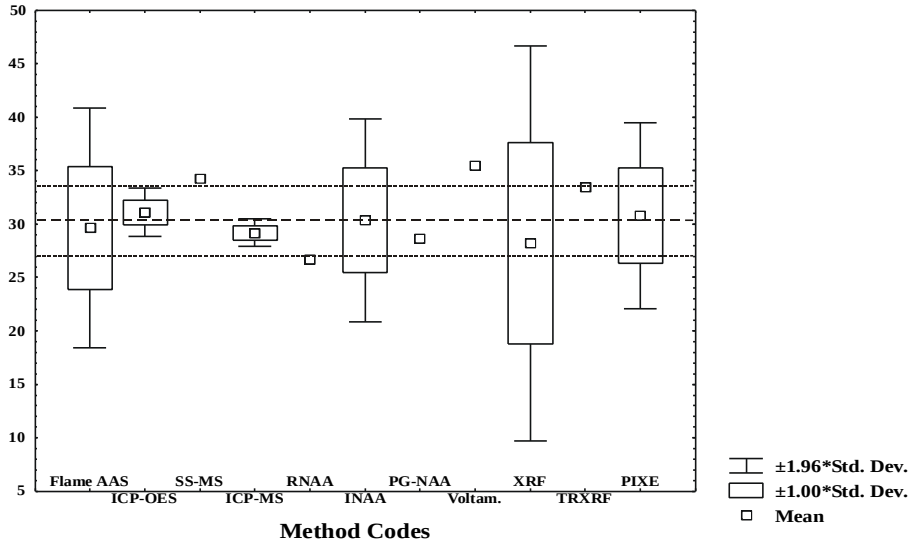

Figure 4 *Mean Zn results obtained by different analytical methods*

7.2.2 Elements with assigned information values

It should be noted that the following results which have been classified as information values, should be used with caution. In some cases information values were assigned to analytes because too few results were reported for them to be classified as recommended values or the results were obtained using only one technique. In some cases, there may be large uncertainties in the mean value or there may have been too many outliers which indicates that problems exist with the analysis of these elements.

Aluminium

Of the sixteen laboratories that reported results for Al, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: AAS (1), XRF (1), PIXE (1), NAA (7), ICP-MS (2), and ICP-OES (3). The comparison of the standard deviation of the mean results obtained by destructive and non-destructive techniques in Figure 5 indicates problems in the determination of Al. Possible reasons for the high standard deviation of the destructive techniques are: contamination from the laboratory environment and/or problems in the dissolution of the samples. The information value for Al is 680 mg/kg with a confidence interval of 570 - 790 mg/kg.

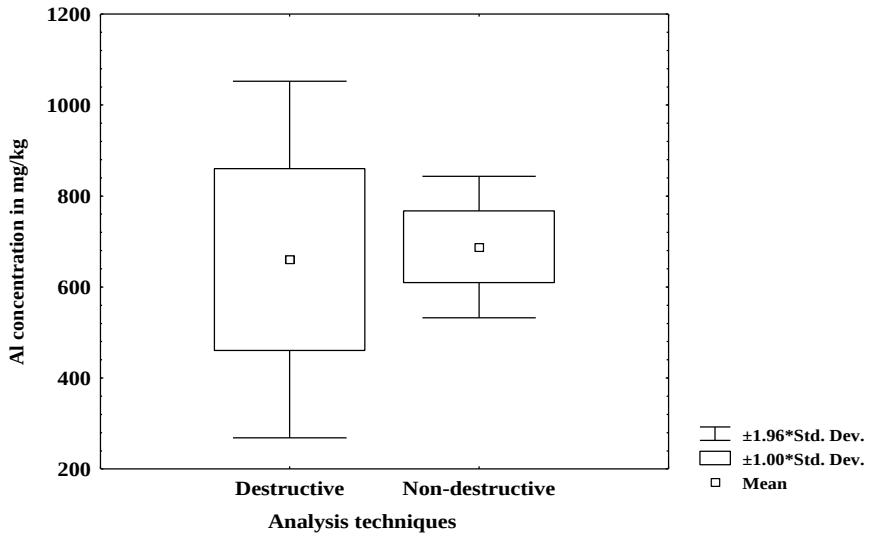

Figure 5 *Comparison of mean Al results obtained by destructive and non-destructive techniques*

Cadmium

Of the sixteen laboratories that reported results for Cd, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: flame (2) and graphite furnace AAS (5), Voltammetry (1), RNAA (1), ICP-MS (3), SS-MS (1), and ICP-OES (1). The AAS results, both flame and graphite furnace, were found to be significantly different from the results of the remaining techniques and therefore the Cd value was classified as an information value. The information value for Cd is 0.117 mg/kg with a confidence interval of 0.100 - 0.134 mg/kg.

Chlorine

Of the fifteen laboratories that reported results for Cl, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: INAA (8), PIXE (1), XRF (3) and unspecified (1). Because the standard deviation of all laboratories' mean values was 13.1% and the Cl concentration was higher than 500 mg/kg, the Cl value was assigned as an information value. The information value for Cl is 1900 mg/kg with a confidence interval of 1600 - 2200 mg/kg.

Chromium

Of the twenty-two laboratories that reported results for Cr, no laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: flame (2) and graphite furnace (2) AAS, PIXE (1), NAA (13), ICP-MS (3), and ICP-OES (1). Because the standard deviation of all laboratories' mean values was 27.5% the Cr value was assigned as an information value. In addition a large discrepancy was noted between results derived from destructive and non-destructive techniques (Figure 6), and application of the t-test confirmed that the two mean values were significantly different. The information value for Cr is 1.06 mg/kg with the confidence interval 0.89 - 1.23 mg/kg.

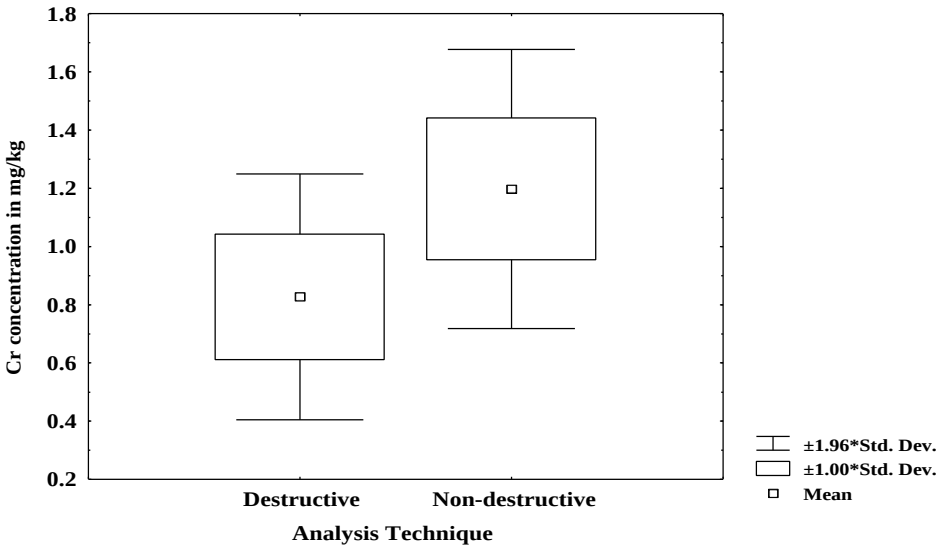

Figure 6 *Comparison of Cr results derived from destructive and non-destructive analysis techniques*

Europium

Of the ten laboratories that reported results for Eu, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: INAA (8) and RNAA (1). The information value for Eu is calculated to be 0.023 mg/kg. The confidence interval ranges from 0.019 - 0.027 mg/kg.

Lutetium

Of the five laboratories that reported results for Lu, no laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: NAA (4) and ICP-MS (1). Although the standard deviation was 26%, Lu meets the criteria for an information value. The information value for Lu is 0.0066 mg/kg with a confidence interval of 0.0042 - 0.0090 mg/kg.

Neodymium

Of the five laboratories that reported results for Nd, no laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: NAA (4) and ICP-MS (1). Because fewer than ten laboratories reported results for Nd, it was classified as an information value. The information value for Nd is 0.60 mg/kg with a confidence interval of 0.42 - 0.78 mg/kg.

Phosphorus

Of the thirteen laboratories that reported results for P, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: Colourimetry (2), ICP-OES (5), ICP-MS (1), PIXE (1), XRF (2) and INAA (1). Because the standard deviation of the mean exceeded 20%, the P value was classified as an information value. The information value for P is 610 mg/kg with a confidence interval of 490 - 730 mg/kg.

Lead

Pb failed the IAEA's rigid classification criteria to be considered as an information value for this intercomparison exercise because the number of rejected laboratory means exceeded 30% (8 out of 22). But since Pb is an important toxic element it was decided to incorporate the results obtained from a Co-ordinated Research Project on "Validation and application of plants as biomonitors of trace element atmospheric pollution, analyzed by nuclear and related techniques" for the final evaluation (these participants are listed in Appendix IV). Of the 29 laboratories that reported results,

6 laboratories were rejected as outliers, most of them using X-ray related techniques. The following techniques were used to provide the accepted results: flame (2) and graphite furnace (6) AAS, ICP-OES (5), ICP-MS (4), SS-MS (1), PIXE (3), TRXRF (1) and Voltammetry (1). The information value for Pb is 4.9 mg/kg with a confidence interval of 4.3 - 5.5 mg/kg.

Rubidium

Of the seventeen laboratories that reported results for Rb, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: X-ray related techniques (4), NAA (10) and ICP-MS (2). Because there was a significant difference between the mean of the X-ray related techniques (1.99 ± 0.15 mg/kg) and the means of NAA (1.69 ± 0.15 mg/kg) and ICP-MS (1.66 ± 0.04 mg/kg), the Rb value was classified as an information value. The information value for Rb is 1.76 mg/kg with a confidence interval of 1.54 - 1.98 mg/kg.

Scandium

Of the fourteen laboratories that reported results for Sc, one laboratory was rejected as an outlier. The following techniques were used to provide the accepted results: RNAA (1), INAA (11) and fast neutron NAA (1). Because all the techniques were NAA related it was decided to classify Sc as an information value. The information value for Sc is 0.17 mg/kg with a confidence interval of 0.15 - 0.19 mg/kg.

Terbium

Of the seven laboratories that reported results for Tb, no laboratory was rejected as outlier. The following techniques were used to provide the accepted results: INAA (6) and ICP-MS (1). Because less than ten laboratories reported results for Tb, the mean was assigned as an information value. The information value for Tb is 0.014 mg/kg with a confidence interval of 0.012 - 0.016 mg/kg.

Vanadium

Of the ten laboratories that reported results for V, two laboratories were rejected as outliers. The following techniques were used to provide the accepted results: ICP-MS (2), ICP-OES (1) and INAA (4) and RNAA (1). Because there was a statistically significant difference between the mean of the INAA results and those of the destructive techniques (RNAA, ICP-MS and ICP-OES) and only 8 laboratories' means were accepted, the value for V was classified as an information value. The information value for V is 1.47 mg/kg with a confidence interval of 1.25 - 1.69 mg/kg.

Ytterbium

Of the five laboratories that reported results for Yb, no laboratory was rejected as an outlier. The following techniques were used to provide accepted results: NAA (4) and ICP-MS (1). Because fewer than ten laboratories reported results for Yb, the value for Yb was classified as an information value. The information value for Yb is 0.037 mg/kg with the confidence interval of 0.025 - 0.049 mg/kg.

7.2.3 Elements which failed both Recommended and Information Value Acceptance Criteria

A total of 27 elements failed to meet the minimum criteria to be assigned as a recommended or an information value. Of these 27, a total of 18 failed because too few (less than 5) laboratories reported results (Au, Be, Bi, Er, Ga, Gd, Ho, I, Li, Mo, Pr, S, Sn, Tl, Tm, W, Y, Zr), a total of 7 failed because the standard deviation of the means was too high (Ag, Ca, Mg, Ni, Si, Ti, U) while 2 failed (Hf and Ta) because results from only one method (NAA) were accepted after outlier tests.

Of all the elements which failed to even the minimum criteria for acceptance as an

information value the results for four important and frequently measured elements merit further analysis to find a possible explanation for their unexpected failure.

Calcium

Results for calcium was produced by twenty-nine laboratories none of which was rejected as an outlier. The range of the results was large (standard deviation of 26.8% at a concentration of about 2800 mg/kg) and exceeded the limit for the overall mean to be assigned as an information value. The mean values of all methods are presented in Figure 7 which clearly demonstrates the large variation reported by various laboratories using different analytical techniques. This large variation could not be ascribed to inhomogeneity as this was evaluated using samples from the original bulk material and found to contribute less than 2% to the overall uncertainty of the measurements. An additional problem with the calcium data is that it is not normally distributed as can be seen in Figure 8 where a fitted normal distribution is superimposed on the calcium histogram data. Without detailed information on the methods applied (e.g. details on calibration standards, reference materials, reagents, blanks) it is not possible to identify and exclude biased values from this population.

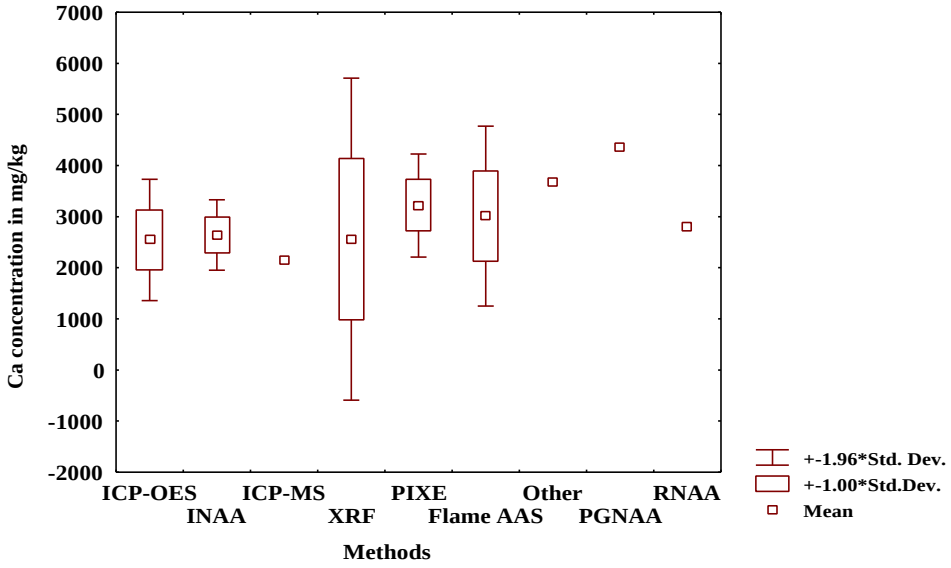

Figure 7 *Mean Ca results obtained with different methods*

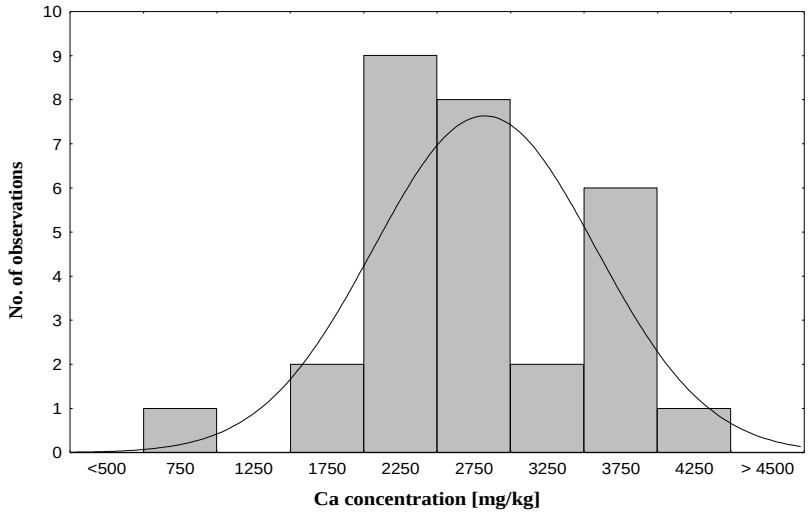

Figure 8 *Histogram showing the distribution of the Ca values*

Magnesium

Magnesium is another element where surprisingly poor agreement was observed among the laboratories. Of the twenty-one laboratories that submitted results, only one mean was excluded as an outlier. The set of accepted results was normally distributed and yielded an overall standard deviation of the consensus mean of 33.4% which exceeded the limit to be assigned as an information value. The evaluation of the standard deviation of means associated with various analytical methods yielded a range from 13% for ICP-OES to 80% for ICP-MS. INAA, XRF and flame AAS also showed high variances (Figure 9).

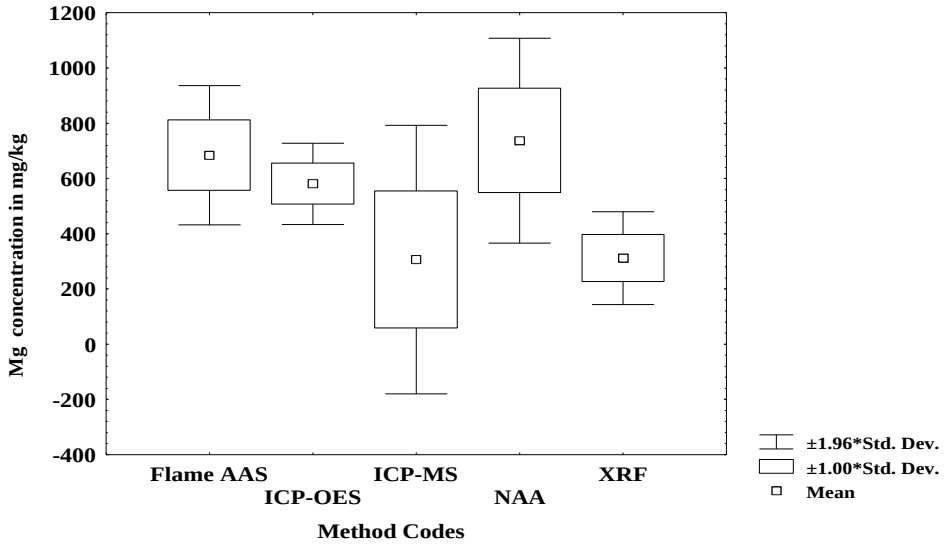

Figure 9 *Mean Mg results obtained by different Methods*

Nickel

Of the eighteen laboratories that submitted results for nickel, one result was rejected as an outlier. Further analysis of the data indicated that the results from the nine methods used for analysis could be sorted into three distinct groups each with a different mean of means. PIXE, TRXRF and Voltammetry yielded a group mean of 3.1 ± 0.2 mg/kg. INAA and XRF yielded a mean of 1.52 ± 0.46 mg/kg while the remaining destructive techniques (flame and graphite furnace AAS, ICP-MS and ICP-OES) yielded a group mean of 1.07 ± 0.27 mg/kg. While these two latter means are not significantly different, the first group was excluded on the basis of a t-test. An additional problem is that the relative standard deviation for the all the accepted laboratory means for Ni exceeded 50 %. Under these circumstances it was not possible to give an informational value for Ni.

Uranium

Ten laboratories reported results for U and none was rejected as an outlier. The relative standard deviation of the mean was 36% which exceeds the criterion for it to be classified as an information value. If a t-test had been performed in addition to the four standard outlier tests, one laboratory mean which was very low would have been rejected which would have reduced the relative standard deviation of the nine remaining laboratories to 26.2 %. This would have led to uranium qualifying as an information value of 0.04 mg/kg with a confidence interval of 0.03 - 0.05 mg/kg for U.

7.3 General Remarks

As is often the case with intercomparisons organized by the IAEA, this intercomparison exercise had a high percentage of the results (78%) produced by nuclear (NAA 50.4%) and nuclear related techniques (X-ray techniques 13.6%, ICP-MS 13.9%). The percentage of results produced by AAS and AES techniques accounted for only 19.9% while the remaining 2% were obtained by a number of different techniques. Overall there was good agreement between the laboratories as evidenced by only 9% of the results being rejected by the outlier tests. However the percentage of outliers attributable to a particular method varied considerably (Figure 10). From the large number of outliers, it would appear that the X-ray related techniques continue to have difficulties when analyzing biological matrices. In the case of ICP-MS, the results reported by one laboratory were consistently rejected and therefore accounted for almost all the outliers for this method. For this intercomparison it was possible to assign recommended and information values for some elements (mainly the rare earth elements) for the first time, due to the submission of results by ICP-MS laboratories. In previous intercomparisons this was not possible due to the fact that only one method (NAA) was capable of measuring these elements at such low levels.

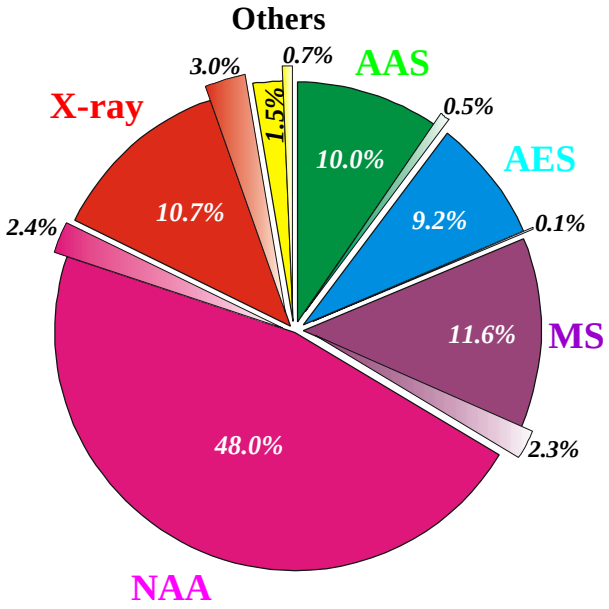

Figure 10 *Percentage of the methods used and their outliers*

8. RECOMMENDATIONS ON INDIVIDUAL DATA REVIEW

To provide feedback to each participant, a report format has been developed that lists the results of the laboratory together with the recommended value and its confidence interval. For ease of comparison we have calculated a Z-score based on the recommended value. Although the Z-score is routinely used in proficiency tests to evaluate the performance of laboratories, we wish to emphasize that because this was an intercomparison exercise, the Z-score can only be used as a relative indicator of how a laboratory's result compares to the mean of accepted laboratory means. For example, a Z-score of "0" means that there is little or no difference between the laboratory value and the recommended value. A Z-score of +1 or -1 would indicate a laboratory mean that was higher or lower than the recommended value respectively. Results that have a Z-score of 3 or greater were always rejected by the outlier tests and would indicate that a problem exists with the analysis and that the laboratory should look into possible causes for the poor result and initiate corrective actions.

It should be noted here that the results for the IAEA-359 cabbage reference material, which was distributed together with the IAEA-336 lichen, are not summarized in this report. The results

for IAEA-359 will be compiled together with two additional sets of intercomparison results for the same material in a separate IAEA report to be issued at a later date.

9. CONCLUSIONS

A lichen material, IAEA-336, prepared from the species *Evernia prunastri*, has been characterized through an international intercomparison exercise. Results for the lichen indicate that it has very low levels of most elements which is consistent with the low level of pollution found in the environment from where it originated. Recommended and information values have been assigned for 19 and 14 elements respectively. Under the current IAEA procedures for assigning recommended or information values, it was not possible to define the metrological traceability of the trace element concentrations specified in IAEA-336. Therefore, IAEA-336 can not be used for the purposes of establishing a chain of traceability or for the purposes of calibration (as is the case with most natural matrix reference materials). However, the material is suitable to be used as a quality control material to monitor the performance of an instrument or for method development purposes.

10. ACKNOWLEDGMENTS

The efforts of the participating laboratories listed in Appendix IV of this report, which contributed their time and facilities to the present work, are gratefully acknowledged, as are the efforts of the following individuals: Shirley Clements (IAEA-NAHU) for her invaluable help in the data preparation, Edith Wehrstein and Norbert Haselberger (IAEA-NAAL) for their work on the homogeneity studies and Andreas Bleise (IAEA-NAHU) for his assistance with the final formatting of this report. The authors also wish to acknowledge the work of M. Carmo Freitas in the collection and preparation of the material and for the collection of the preliminary data from the participating laboratories.

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Method Codes for the Intercomparison, IAEA-336, Lichen

A1: atomic absorption spectroscopy (AAS), flame
A2: AAS, electrothermal
A3: AAS, electrothermal + Zeeman background correction
A4: AAS, cold vapour
A5: AAS, hydride generation

C1: colourimetry
C2: gas/liquid chromatography with electron capture detection
C3: catalytic method for iodine

E1: atomic emission spectroscopy (AES), flame
E2: ES, Flame photometry
E3: ES, ES, DC arc
E4: ES, direct current plasma (DCP-OES)
E5: ES, inductively coupled plasma (ICP-OES)
E6: ICP-OES, hydride generation

F1: fluorimetry

G1: gamma-spectroscopy (prompt) following proton activation (PIGME)

I1: ion-specific electrode

M1: mass spectrometry (MS), spark source (SS-MS)
M2: MS, inductively coupled plasma source (ICP-MS)

N1: thermal neutron activation analysis (NAA), radiochemical
N2: thermal NAA, instrumental (INAA)
N3: fast NAA, radiochemical (RNAA)
N4: fast NAA, instrumental (INAA)
N5: NAA, prompt gamma (PGNAA)

P1: photon activation analysis (PAA), radiochemical
P2: PAA, instrumental

V1: voltammetry / polarography

X1: X-ray fluorescence analysis (XRF)
X2: total reflection XRF (TRXRF)
X3: particle induced X-ray emission analysis (PIXE)
X4: other X-ray methods

Z: other methods

APPENDIX I

SUMMARY TABLE OF THE RESULTS

FOR THE INTERCOMPARISON

(For explanation of the terms used in the tables, please refer to Section 7)

Analyte	Reported results [mg/kg]				Accepted results [mg/kg]					
	No. Lab. means	Range of laboratories' means		Outlier [%]	No. Lab means	Range of laboratories' means		Mean	Standard deviation [%]	Expanded Confidence Interval
Ag	5	0.0095	0.0298	0	5	0.0095	0.0298	0.021	44.0	
Al	16	73.3	949.7	6.3	15	402.2	949.7	675.0	21.2	567.9 - 782.1
As	20	0.143	0.849	15.0	17	0.488	0.746	0.634	10.6	0.559 - 0.710
Au	4	0.0017	0.0033	0	4	0.0017	0.0033	0.0025	35.4	
Ba	12	1.10	8.41	8.3	11	4.50	8.41	6.39	20.2	5.27 - 7.52
Be	2	0.02	0.02							
Bi	1	0.01								
Br	19	3.40	18.0	5.2	18	8.85	18.0	12.93	16.1	11.22 - 14.64
Ca	29	817.5	4350	0	29	817.5	4350	2823.1	26.8	
Cd	16	0.040	0.145	12.5	14	0.085	0.145	0.1174	15.3	0.1011 - 0.1339
Ce	14	0.308	1.64	7.1	13	0.917	1.64	1.275	13.4	1.102 - 1.448
Cl	15	377	3650	13.3	13	1472	2333	1914.5	13.1	1656.5 - 2172.5
Co	21	0.079	0.527	9.5	19	0.172	0.422	0.294	20.2	0.252 - 0.336
Cr	22	0.362	1.643	0	22	0.362	1.643	1.056	27.5	0.886 - 1.226
Cs	16	0.046	0.140	18.8	13	0.094	0.12	0.1103	6.3	0.0975 - 0.1230
Cu	26	0.82	10.2	19.2	21	2.32	5.19	3.63	18.8	3.14 - 4.12
Er	1	0.0357								
Eu	10	0.0107	0.0284	10.0	9	0.0188	0.0284	0.0232	14.6	0.0194 - 0.0269
Fe	39	0.98	664.0	10.3	35	289.1	556.7	427.8	12.3	380.7 - 475.0
Ga	3	0.101	0.565	0	3	0.101	0.565	0.31	74.6	
Gd	2	0.136	0.891							
Hf	7	0.0345	0.0633	14.3	6	0.0537	0.0633	0.057	6.4	
Hg	15	0.143	0.292	0	15	0.143	0.292	0.2036	20.6	0.1718 - 0.2356
Ho	1	0.01								
I	4	2.94	3.41	0	4	2.94	3.41	3.19	6.0	
K	28	1072	2634	14.3	24	1618	2145	1835.8	7.8	1635.4 - 2036.1

Analyte	Reported results [mg/kg]				Accepted results [mg/kg]					
	No. Lab. means	Range of laboratories' means		Outlier [%]	No. Lab means	Range of laboratories' means		Mean	Standard deviation [%]	Expanded Confidence Interval
La	13	0.123	0.856	7.7	12	0.537	0.856	0.663	15.0	0.566- 0.759
Li	4	0.26	0.44	0	4	0.26	0.44	0.364	20.8	
Lu	5	0.004	0.009	0	5	0.004	0.009	0.0066	26.4	0.0042 - 0.0090
Mg	21	131	2817	4.8	20	131	1000	583.5	33.4	
Mn	33	11.8	100.5	12.1	29	45	75.2	63.4	10.9	56.4 - 70.4
Mo	4	0.055	0.0265	0	4	0.055	0.0265	0.153	56.2	
Na	25	141	1280	20.0	20	267	386	323.4	11.1	285.6 - 361.2
Nd	5	0.490	0.800	0	5	0.490	0.800	0.600	21.6	0.419 - 0.782
Ni	18	0.63	4.03	5.6	17	0.63	3.22	1.651	53.6	
P	13	21.48	883.2	7.7	12	374.7	883.2	608.1	25.4	489.2 - 727.1
Pb	29	0.0244	10.75	20.7	23	4.39	6	4.9	8.6	4.3 -5.5
Pr	1	0.14								
Rb	17	0.338	2.2		16	1.45	2.2	1.763	11.0	1.548 - 1.978
S	5	499.2	4700	20.0	4	499.2	870.6	167.6	22.9	
Sb	13	0.058	0.163	7.7	12	0.058	0.090	0.0729	13.5	0.0624 - 0.0834
Sc	14	0.1088	0.2051	7.1	13	0.1388	0.2051	0.1727	9.5	0.1514 - 0.1940
Se	15	0.013	0.725	20	12	0.156	0.303	0.223	17.2	0.189 - 0.257
Si	7	172.2	1896.7	0	7	172.2	1896.7	1181.0	60.2	
Sm	15	0.0814	0.1373	0	15	0.0814	0.1373	0.1062	15.2	0.0918 - 0.1206
Sn	2	5.23	8.88							
Sr	21	1.96	14.8	9.5	19	7.47	11.35	9.30	12.7	8.17 - 10.43
Ta	7	0.0128	6.48	14.3	6	0.0128	0.0172	0.0153	10.9	
Tb	7	0.0120	0.0163	0	7	0.0120	0.0163	0.0141	11.1	0.0119 - 0.0164
Th	17	0.026	0.18	5.9	16	0.110	0.18	0.142	12.8	0.124 - 0.160
Ti	12	8.09	160	8.3	11	8.09	80.1	50.1	40.5	
Tl	3	0.007	0.0111	0	3	0.007	0.0111	0.0089	23.2	

Analyte	Reported results [mg/kg]				Accepted results [mg/kg]					
	No. Lab. means	Range of laboratories' means		Outlier [%]	No. Lab means	Range of laboratories' means		Mean	Standard deviation [%]	Expanded Confidence Interval
Tm	3	0.0043	0.0116	0	3	0.0043	0.0116	0.0070	56.4	
U	10	0.0116	0.0537	0	10	0.0116	0.0537	0.0378	36.1	
V	10	0.33	2.933	20	8	1.25	1.716	1.471	11.3	1.248 - 1.693
W	1	0.18								
Y	3	0.043	0.347	0	3	0.043	0.347	0.236	71.3	
Yb	5	0.0238	0.0453	0	5	0.0238	0.0453	0.0373	23.9	0.0250 - 0.0495
Zn	40	8.5	43.58	5	38	18.0	36.6	30.36	14.9	26.94 - 33.79
Zr	2	2.917	2.983							

APPENDIX II

DATA TABLE OF THE

INDIVIDUAL LABORATORY RESULTS

SORTED BY ANALYTE

(For explanation of the terms used in the tables, please refer to Section 7)

Element : Ag **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5		0.0218	0.0293	0.0306	0.0358	0.0321	0.0263	0.0155	N2	0.029	0.005
20	C	0.037	0.036	0.025	0.021			0.02	N2	0.030	0.008
28		0.020	0.015	0.012	0.009	0.012		0.008	M2	0.014	0.004
29	A	0.009	0.009	0.010	0.010	0.008	0.011	0.002	M2	0.010	0.001
41	A	0.0182	0.0215	0.0224	0.0199	0.0219	0.0243	0.0113	N2	0.021	0.002

Element: Al **Mean: 680** **Upper Limit: 790** **Lower Limit: 570** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		602	609	619	557	601	609	3.5	E5	600	21.8	-0.5
3		598	602	671	631	671	653	0.41	N2	638	32.7	-0.3
5		647	673	649		568	618	19.6	N2	631	40.3	-0.3
6		633	676	679	710	654	658	30	N2	668	26.3	0.0
11		559.85	553.35	531.44	635.97	679.80		0.48	M2	592	62.9	-0.6
12	A	760	760	790				10	N2	770	17.3	0.7
14		646	549	521	639			40	X1	589	63.2	-0.6
16	A	493	483	490	482	487	485	0.0075	E5	487	4.2	-1.3
17		810	792	819	817	828	851	1.9	N2	820	19.6	1.0
21	B	711	716	707				5	E5	711	4.5	0.3
22		531	650	724	710	655	628	90	X3	650	68.9	-0.2
28		394	386	409	403	419		0.3	M2	402	12.8	-1.9
29		72	63	87	82	65	71	1	M2	73.3	9.4	-4.2
31		950	950	945	955	948	950	0.001	A1	950	3.3	1.9
35		710	737	746	757	746		5	N2	739	17.8	0.4
40		880	930	880	890	840	860		N1	880	30.3	1.4

Element: As **Mean: 0.63** **Upper Limit: 0.71** **Lower Limit:0.55** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
2	B	0.64	0.67	0.64	0.66	0.74	0.65	0.17	A2	0.667	0.038	0.5
5		0.592	0.577	0.554	0.515	0.62	0.548	0.052	N2	0.568	0.038	-1.0
6		0.59	0.59	0.56	0.54	0.55	0.52	0.1	N2	0.558	0.028	-1.1
7	C	0.694	0.693	0.689	0.687	0.69	0.703	0.0005	A5	0.693	0.006	0.9
8	D	0.723	0.696	0.679	0.731	0.70	0.698	0.03	A5	0.704	0.019	1.0
10	A	0.667	0.631	0.661	0.648	0.633	0.625	0.002	N1	0.644	0.017	0.1
10	B	0.677	0.695	0.661	0.702	0.736	0.651	0.05	N2	0.687	0.031	0.8
11		0.56	0.525	0.558	0.6	0.60		0.315	M2	0.568	0.031	-1.0
12	A	0.64	0.68	0.65				0.04	N2	0.657	0.021	0.3
13	B	0.54	0.54	0.44	0.39	0.53		0.2	A3	0.488	0.069	-2.2
17		0.722	0.722	0.786	0.74	0.757	0.749	0.047	N2	0.746	0.024	1.6
20	C	0.862	0.803	0.794	0.923	0.81	0.896	0.04	N2	0.849	0.053	3.2
23		0.64	0.63	0.6	0.64	0.64	0.66	0.056	N2	0.635	0.020	0.0
28		0.699	0.681	0.67	0.68	0.68		0.005	M2	0.682	0.011	0.7
29		0.16	0.13	0.17	0.11			0.08	M2	0.143	0.028	-7.3
35		0.53	0.56	0.57	0.57	0.57		0.1	N2	0.560	0.017	-1.1
36		0.15	0.08	0.22				0.1	N2	0.150	0.070	-7.2
38	A	0.671	0.74	0.645					N2	0.685	0.049	0.8
40		0.59	0.64	0.60	0.59	0.62	0.55		N1	0.598	0.031	-0.5
41	A	0.651	0.628	0.616	0.643	0.66	0.687	0.0336	N2	0.648	0.025	0.2

Element: Au **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
17		0.002	0.0023	0.00205	0.00159	0.00119	0.00189	0.00028	N2	0.0018	0.0004
20	C	0.002	0.0019	0.0019	0.0082	0.0024		0.0001	N2	0.0033	0.0028
38	A	0.0025	0.0032	0.0043					N2	0.0033	0.0009
40		0.0007	0.0024	0.0007	0.0027	0.0019			N1	0.0017	0.0009

Element: Ba **Mean: 6.4** **Upper Limit:7.5** **Lower Limit: 5.3** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		7	6.66	7.05	5.96	7.12	7.14	1.11	N2	6.82	0.46	0.3
7	D	5.7	5.7	5.6	5.5	5.5		0.05	E5	5.60	0.10	-0.6
10	B	7.1	7.2	9.6	7.2	4.7	5.7	2.8	N2	6.92	1.66	0.4
11		5.62	5.33	5.68	5.7	5.75		0.189	M2	5.62	0.17	-0.6
13	C	4.7	4.4	4.3	5.1	4.7	4.7	1	E5	4.65	0.28	-1.4
17		10.2	6.93	7.25	8.47	8.2	8.62	0.94	N2	8.28	1.16	1.5
20	C	6.55	6.76	6.93	6.87	7.06	7.19	0.75	N2	6.89	0.23	0.4
21	B	4	5					0.5	E5	4.50	0.71	-1.5
23		6.4	5.7	5.84	4.81	5.96	6.4	2.05	N2	5.85	0.59	-0.4
28		6.9	6.91	6.78	6.6	6.8		0.006	M2	6.80	0.12	0.3
29		1.21	1.03	1.11	1.11	1.04	1.12	0.02	M2	1.10	0.07	-4.1
38	A	8.98	8.31	8.42	7.93				N2	8.41	0.43	1.6

Element: Be **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
28		0.024	0.023	0.015	0.018	0.02		0.006	M2	0.02	0.0036742
29		0.02	0.02	0.02				0.01	M2	0.02	3.293E-10

Element: Bi **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
28		0.013	0.012	0.01	0.009	0.012		0.004	M2	0.01	0.001643

Element: Br **Mean: 12.9** **Upper Limit: 14.6** **Lower Limit: 11.2** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		10	10.2	11.5	10.9	10.8	11.4	2.3	N2	10.80	0.61	-1.0
5		16.114	15.122	15.600	14.312	16.544	15.227	0.105	N2	15.49	0.79	1.2
6		10.6	10.5	10.9	10.7	10.5	10.1	0.2	N2	10.55	0.27	-1.1
9		13.3	12.6	13.3	12.8	13.1	14	0.31	N2	13.18	0.49	0.1
10	B	12.2	12.1	11.8	12.1	11.5	11.5	0.13	N2	11.87	0.31	-0.5
14		12	12.6	12.7	11.5			0.5	X1	12.20	0.56	-0.4
17		12.3	12.2	12.3	12.3	12	12.3	0.035	N2	12.23	0.12	-0.3
18		3.20	3.10	3.20	3.20	3.10	4.60	0.7	X3	3.40	0.59	-4.6
20	C	12.8	12.8	12.9	13.5	13.3	13.6	0.07	N2	13.15	0.36	0.1
23		12.7	12.4	12.5	13	13.1	13.4	0.15	N2	12.85	0.38	0.0
24		19	18	18	17	18		5	X1	18.00	0.71	2.4
25		11	16	17	15	14		1.5	X3	14.60	2.30	0.8
27	B	14	15.3	12.2	12.9	12.6	13.2	0.39	X3	13.37	1.13	0.2
32		12	6.7	8.9	7.8			2	X1	8.85	2.28	-2.0
35		11.7	12.2	11.7	11.7	12.4		0.2	N2	11.94	0.34	-0.5
36		13.0	13.7	11.4	11.8	14.0	12.1	3	N2	12.67	1.06	-0.1
37		13.8	14.9	14.1	13.7	14.5	13.2	0.5	N4	14.03	0.61	0.5
38	A	15.36	15.36	15.27					N2	15.33	0.05	1.1
40		11.7	12.1	11.5	12	11.2	11.8		N1	11.72	0.33	-0.6

Element: Ca **Mean: 2800** **Upper Limit: 3250** **Lower Limit:2350** **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
1		2374	2394	2427	2398	2439	2434	1.1	E5	2411	26.1
3		2410	2260	2270	2450	2330	2420	55	N2	2357	81.4
5		3661	3333.8		3129.8			2463.8	N2	3375	268.0
7	D	2470	2450	2500	2490	2500		0.1	E5	2482	21.7
9		2508	2307	2677	2321	2461	2478	890	N2	2459	136.0
10	B	2508	2563	2432	2580	2496	2527	26	N2	2518	52.8
11		2121	2138	2102	2080	2359		6.66	M2	2160	113.3
13	C	2390	2330	2360				10	E5	2360	30.0
14		3150	3049	2847	2872			100	X1	2980	144.9
16	B	1960	1876	1885	1874	1869	1896	0.00175	E5	1893	34.0
17		2560	2500	2620	2590	2430	2570	32	N2	2545	68.9
18		2910	2880	2860	2870	2910	2900	40	X3	2888	21.4
20	C	2725	2687	2927	2703	2810	2659	80	N2	2752	100.0
21	B	2410	2380	2340	2350	2350	2430	1	E5	2377	36.7
21	C	2550	2430					1	E5	2490	84.9
22		3340	3500	3270	3430	3340	2910	0.3	X3	3298	206.4
24		3900	3900	3900	3800	3900		3	X1	3880	44.7
25		3920	3920	3750	3990	3900		58.5	X3	3896	88.5
27	A	4024	3311	4081				417	A1	3805	429.1
27	B	2978	2633	2761	2784	2805	2737	41	X3	2783	112.8
31		1840	2000	2050	1990	2000	2000	0.002	A1	1980	71.8
32		780	830	800	860			22	X1	818	35.0
33	A	3760	3870	3550	3760	3650	3440	2	A1	3672	157.4
33	C	3820	3860	3970	3650	3700	3850	25	E5	3808	116.2
34	B	3659	3666	3683					Z	3669	12.3
38	A	1990	2400	2810	2530	2370	2810		N2	2485	309.4
38	B	5100	3600						N5	4350	1060.7
39		2550	2500		2510	2730		12.9	A1	2573	107.2
40		2940	3000	2600	2800	2700			N1	2808	165.3

Element: Cd **Mean: 0.117** **Upper Limit: 0.134** **Lower Limit: 0.100** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		0.12	0.07	0.09				0.01	E5	0.093	0.025	-1.3
2	A	0.13	0.11	0.12	0.11	0.12	0.12	0.11	A1	0.118	0.008	0.0
2	B	0.132	0.125	0.137		0.135	0.129	0.017	A2	0.132	0.005	0.8
4		0.106	0.103	0.097	0.099	0.099			A3	0.101	0.004	-0.9
7	A	0.128	0.125	0.125	0.13	0.125		0.0004	A3	0.127	0.002	0.5
7	E	0.112	0.115	0.113	0.118	0.111	0.114	0.001	M1	0.114	0.002	-0.2
7	F	0.121	0.112	0.103	0.127	0.132	0.12	0.001	V1	0.119	0.010	0.1
8	B	0.14	0.138	0.138	0.138	0.144	0.148	0.025	A2	0.141	0.004	1.3
10	A	0.119	0.11	0.109	0.079	0.104	0.115	0.007	N1	0.106	0.014	-0.6
11		0.09	0.12	0.12	0.12			0.015	M2	0.113	0.015	-0.3
12	A	0.091	0.083	0.081				0.003	M2	0.085	0.005	-1.8
13	B	0.136	0.15	0.139	0.14	0.14	0.133	0.02	A3	0.140	0.006	1.2
28		0.11	0.118	0.111	0.099	0.12		0.008	M2	0.112	0.008	-0.3
29		0.047	0.045	0.053	0.044			0.006	M2	0.047	0.004	-3.9
31		0.016	0.016	0.016	0.016	0.016	0.16	0.001	V1	0.040	0.059	-4.3
41	B	0.147	0.143	0.148	0.145	0.141	0.146		A1	0.145	0.003	1.5

Element: Ce **Mean: 1.28** **Upper Limit: 1.45** **Lower Limit:1.11** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		1.29	1.46	1.34	1.38	1.53	1.34	0.37	N2	1.39	0.09	0.7
5		1.4324	1.2318	1.2211	1.2012	1.2953	1.2331	0.029	N2	1.27	0.09	0.0
6		1.28	1.39	1.42				0.15	N2	1.36	0.07	0.5
9		1.22	1.16	1.17	1.28	1.18	1.31	0.09	N2	1.22	0.06	-0.3
10	B	0.831	0.184	0.242	0.208	0.131	0.25	0.058	N2	0.31	0.26	-5.6
12	A	1.15	1.31	1.29				0.2	N2	1.25	0.09	-0.1
12	B	0.93	0.91	0.91				0.005	M2	0.92	0.01	-2.1

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Element: Ce contd. **Mean: 1.28** **Upper Limit: 1.45** **Lower Limit:1.11** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
17		1.31	1.64	1.32	1.38	1.35	1.28	0.0083	N2	1.38	0.13	1.0
20	C	1.28	1.32	1.29	1.3	1.39	1.34	0.02	N2	1.32	0.04	0.0
23		1.15	1.13	1.1	1.16	1.1	1.1	0.032	N2	1.12	0.03	-1.0
28		1.06	1.19	1.35				0.002	M2	1.20	0.15	-0.4
38	A	1.74	1.57	1.61					N2	1.64	0.09	2.1
40		1.36	1.47	1.33	1.41	1.29	1.29		N1	1.36	0.07	0.5
41	A	1.213	1.144	1.147	1.111	1.151	1.103	0.0658	N2	1.14	0.04	-0.8

Element: Cl **Mean:1900** **Upper Limit: 2200** **Lower Limit: 1600** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		2280	2230	2340	2340	2450	2360	42	N2	2333	75	1.7
5		1701.8	1817.3	1744.6	1618.7	1646.8	1646	30.4	N2	1696	75	-0.9
6		1712	1897	1818	1865	1830	1765	104	N2	1815	67	-0.4
10	B	1950	2020	1978	1970	1968	1934	11	N2	1970	29	0.2
11		<361	377	<356	<353	<365		332	M2	377		-6.1
12	B	2200	2100	2200				20	N2	2167	58	1.0
13	D	1700	1690	1600	1730			5	Z	1680	56	-0.9
14		1439	1421	1509	1519			50	X1	1472	49	-1.8
17		1980	1940	2010	2010	2090	2090	4.3	N2	2020	60	0.4
18		2200	2140	2100	2160	2250	2140	28	X3	2165	53	1.0
24		2300	2300	2100	2100	2200		14	X1	2200	100	1.1
32		2100	1490	1708	1792			246	X1	1773	253	-0.6
35		1730	1810	1740	1770	1760		30	N2	1762	31	-0.6
36		1710	1805	1995				450	N2	1837	145	-0.3
38	B	5300	2000						N5	3650	2333	6.9

Element: Co **Mean: 0.29** **Upper Limit:0.34** **Lower Limit: 0.24** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		0.3	0.28	0.3	0.28	0.29	0.27	0.05	N2	0.287	0.012	-0.1
5		0.3295	0.3409	0.3326	0.3232	0.3445	0.325	0.0017	N2	0.333	0.009	0.7
6		0.29	0.31	0.29	0.3	0.32	0.3	0.01	N2	0.302	0.012	0.1
7	F	0.382	0.347	0.395	0.395	0.355	0.384	0.005	V1	0.376	0.021	1.4
9		0.303	0.317	0.317	0.335	0.334	0.344	0.035	N2	0.325	0.015	0.5
10	B	0.291	0.284	0.272	0.285	0.268	0.275	0.005	N2	0.279	0.009	-0.2
11		0.241	0.231	0.259	0.25	0.282		0.096	M2	0.253	0.019	-0.7
12	B	0.31	0.3	0.28				0.02	N2	0.297	0.015	0.0
13	B	0.45	0.41	0.44	0.4	0.39	0.44	0.2	A3	0.422	0.025	2.1
16	B	0.51	0.52	0.55	0.53	0.54	0.51	0.025	E5	0.527	0.016	3.9
17		0.268	0.29	0.298	0.271	0.265	0.289	0.0065	N2	0.280	0.014	-0.2
20	C	0.286	0.281	0.293	0.287	0.283	0.289	0.002	N2	0.287	0.004	-0.1
23		0.28	0.28	0.29	0.29	0.28	0.29	0.0068	N2	0.285	0.005	-0.1
28		0.269	0.279	0.27	0.26	0.27		0.004	M2	0.270	0.007	-0.4
29		0.082	0.074	0.082	0.082	0.073	0.08	0.006	M2	0.079	0.004	-3.6
30		0.38	0.35	0.4	0.38	0.35	0.41	0.02	N2	0.378	0.025	1.4
36		0.184	0.169	0.162				0.04	N2	0.172	0.011	-2.1
37		0.23	0.206	0.207	0.221	0.24	0.24	0.05	N4	0.224	0.015	-1.2
38		0.153	0.242	0.226	0.222				N2	0.211	0.039	-1.4
40		0.33	0.35	0.32	0.32	0.34	0.3		N1	0.327	0.018	0.6
41	A	0.2822	0.2779	0.2716	0.274	0.2817	0.2819	0.000872	N2	0.278	0.005	-0.3

Element: Cr **Mean: 1.06** **Upper Limit: 1.23** **Lower Limit: 0.89** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
2	A	0.64	0.68	0.68	0.72	0.68	0.7	0.27	A1	0.68	0.03	-1.3
5		1.358	1.326	1.313	1.301	1.382	1.345	0.024	N2	1.34	0.03	1.0
6		1.36	1.38	1.27				0.2	N2	1.34	0.06	1.0
7	A	1	0.98	0.93	0.89	0.94		0.002	A3	0.95	0.04	-0.4
8	B	0.939	0.786	0.839	0.826	0.852	0.913	0.2	A2	0.86	0.06	-0.7
10	B	1.15	1.06	1.55	1.15	1.02	0.94	0.09	N2	1.15	0.21	0.3
11		0.73	0.6	0.902	0.669			0.525	M2	0.73	0.13	-1.1
12	B	1.08	1.05	1.41				0.2	N2	1.18	0.20	0.4
16	B	1.07	1.1	1.12	1.08	1.06	1.09	0.0025	E5	1.09	0.02	0.1
17		0.901	0.975	0.978	0.772	1.06	1.1	0.11	N2	0.96	0.12	-0.3
19		1.33	1.36	1.2	1.18	1.27	1.12	0.1	N2	1.24	0.09	0.6
20	C	1.15	1.2	1.18	1.17	1.19	1.19	0.04	N2	1.18	0.02	0.4
22		1.34	1.5	1.86	1.83			0.2	X3	1.63	0.25	2.0
23		1.1	1.07	1.08	1.07	1.07	1.1	0.046	N2	1.08	0.01	0.1
28		0.94	1.04	0.94	0.93	0.93		0.006	M2	0.96	0.05	-0.3
29		0.38	0.36	0.37	0.36	0.34		0.01	M2	0.36	0.01	-2.4
36		1.59	1.72	1.62				0.2	N2	1.64	0.07	2.0
37		1.05	0.96	1.05	1.04	0.86	0.75	0.1	N4	0.95	0.12	-0.4
38	A	1.01	1.05	1.03				0.1	N2	1.03	0.02	-0.1
40		0.8	1.1	1	1.1	0.8	1.1		N1	0.98	0.15	-0.2
41	A	1.06	1.21	1.02	1.003	1.056	1.013	0.248	N2	1.06	0.08	0.0
41	B	0.932	0.839	0.852	0.805	0.771	0.835		A1	0.84	0.05	-0.7

Element: Cs **Mean: 0.110** **Upper Limit:0.123** **Lower Limit: 0.097** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.164	0.1291	0.1259	0.156	0.1376	0.1276	0.0036	N2	0.140	0.016	4.3
6		0.11	0.11	0.1				0.015	N2	0.107	0.006	-0.5
9		0.102	0.11	0.107	0.113	0.107	0.1	0.02	N2	0.107	0.005	-0.5
10	B	0.092	0.105	0.091	0.092	0.091	0.095	0.012	N2	0.094	0.005	-2.3
11		0.074	<0.068	<0.067	0.07	0.085		0.063	M2	0.076	0.008	-4.9
12	A	0.13	0.11	0.12				0.05	N2	0.120	0.010	1.4
12	B	0.11	0.1	0.13				0.02	M2	0.113	0.015	0.4
17		0.123	0.126	0.124	0.119	0.107	0.0993	0.0049	N2	0.116	0.011	0.9
20	C	0.115	0.115	0.117	0.112	0.111	0.111	0.007	N2	0.114	0.003	0.5
23		0.11	0.11	0.11	0.12	0.11	0.12	0.0063	N2	0.113	0.005	0.4
28		0.109	0.112	0.105	0.102	0.106		0.006	M2	0.107	0.004	-0.5
29		0.05	0.045	0.043				0.001	M2	0.046	0.004	-9.3
37		0.127	0.13	0.11	0.12	0.097	0.13	0.01	N4	0.119	0.013	1.3
38	A	0.126	0.111	0.107	0.11	0.104	0.0969	0.01	N2	0.109	0.010	-0.2
40		0.08	0.09	0.1	0.09	0.16			N1	0.104	0.032	-0.9
41	A	0.109	0.106	0.1135	0.1068	0.1083	0.1213	0.00249	N2	0.111	0.006	0.1

Element: Cu **Mean: 3.6** **Upper Limit: 4.1** **Lower Limit: 3.1** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		3.7	4.7	4.4				0.01	E5	4.27	0.51	0.9
2	A	3.52	3.57	3.65	3.68	3.65	3.59	0.34	A1	3.61	0.06	0.0
4		2.84	2.54	3.07	3.06	3.27			A1	2.96	0.28	-1.0
7	A	3.29	3.21	3.22	3.44	3.52		0.002	A3	3.34	0.14	-0.4
7	E	3.48	3.51	3.49	3.43	3.38	3.5	0.002	M1	3.47	0.05	-0.2
7	F	3.36	3.56	3.12	3.46	3.35	3.21	0.005	V1	3.34	0.16	-0.4
8	B	4.1	3.69	3.3	3.67	3.49	3.41	0.1	A2	3.61	0.28	0.0
10	A	3.42	3.18	3.14	3.19	3.08	3.14	0.055	N1	3.19	0.12	-0.6
11		3.01	3.06	3.05	3.15	3.08		0.08	M2	3.07	0.05	-0.8
13	C	3.56	3.01	2.94	3.32	3.25	3.03	1	E5	3.19	0.24	-0.6
14		4.7	3.8	4.7	4.8			0.3	X1	4.50	0.47	1.3
15		3.8						0.1	X1	3.80		0.3
16	B	3.48	3.53	3.64	3.48	3.49	3.48	0.0005	E5	3.52	0.06	-0.2
18		3.5	2.8	5.2	5.3	3.1	3.1	0.3	X3	3.83	1.12	0.3
21	B	6	4	7	6	4	4	0.2	E5	5.17	1.33	2.3
22		6.08	8.47	6.77	5.73	3.95	5.07	0.2	X3	6.01	1.54	3.5
24		8	7	6	7	6		0.5	X1	6.80	0.84	4.6
25		11	14	8	10	8		1.5	X3	10.20	2.49	9.6
27	A	3.29	3.3					1.15	A1	3.30	0.01	-0.5
27	B	2.36	2.37	2.63	2.33	2.11	2.1	0.21	X3	2.32	0.20	-1.9
28		3.35	3.23	3.37	3.4	3.33		0.01	M2	3.34	0.06	-0.4
29		1.13	1.06	1.13	1.04	1.02	1.04	0.06	M2	1.07	0.05	-3.7
31		0.783	0.817	0.833	0.800	0.850	0.817	0.001	V1	0.82	0.02	-4.1
36		4.16	6.21					3	N2	5.19	1.45	2.3
39		3.59	3.38		3.68	3.43		0.305	A1	3.52	0.14	-0.2
42	A	3.9	3.7	3.7	3.6	3.5	3.7	0.8	X2	3.68	0.13	0.1

Element: Er **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
28		0.036	0.037	0.034				0.0004	M2	0.0357	0.0015

Element: Eu **Mean: 0.023** **Upper Limit: 0.027** **Lower Limit: 0.019** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.02	0.02	0.02	0.02	0.02	0.02	0.00635	N2	0.019	0.001	-1.3
6		0.02	0.02	0.02				0.001	N2	0.020	0.000	-0.9
10		0.023	0.018	0.018	0.02	0.019	0.02	0.0018	N2	0.020	0.002	-1.0
12	A	0.01	0.011	0.011				0.0018	M2	0.011	0.001	-3.7
17		0.0272	0.0279	0.0301	0.0287	0.0268	0.0299	0.00047	N2	0.028	0.001	1.5
20	C	0.031	0.024	0.021	0.028	0.022	0.022	0.005	N2	0.025	0.004	0.4
23		0.025	0.024	0.024	0.024	0.023	0.023	0.0015	N2	0.024	0.001	0.2
38	A	0.0293	0.0262	0.0273				0.0015	N2	0.028	0.002	1.3
40		0.027	0.019	0.023	0.024	0.021	0.024		N1	0.023	0.003	-0.1
41	A	0.0235	0.02268	0.02304	0.02197	0.02265	0.02236	0.0017	N2	0.023	0.001	-0.1

Element: Fe **Mean: 430** **Upper Limit: 480** **Lower Limit: 380** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		427	435	428	442	434	434	0.4	E5	433	5	0.1
3		413	440	443	410	447	417	32	N2	428	17	0.0
5		491.3	500.6	492	481.7	513	486	1.4	N2	494	11	1.3
6		480	472	451				5	N2	468	15	0.8
7		413	416	419	420	423		0.3	E5	418	4	-0.2
8	A	469	450	435	449	447	467	20	A1	453	13	0.5
9		426	434	446	434	439	440	28	N2	437	7	0.2
10		412	416	392	409	395	383	7.1	N2	401	13	-0.5

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Element: Fe contd. **Mean: 430** **Upper Limit: 480** **Lower Limit: 380** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
11		433.69	415.42	407.39	413.89	430.93		0.546	M2	420	11	-0.1
12	B	460	450	470				20	N2	460	10	0.6
13		438	383	407	408	401	400	3	E5	406	18	-0.4
14		656	619	525	596			10	X1	599	55	3.3
15		443						0.5	X1	443		0.3
16		366	374	363	363	363	371	0.005	E5	367	5	-1.2
17		417	438	433	434	440	423	2.3	N2	431	9	0.1
18		491	396	422	394	406	398	0.8	X3	418	37	-0.2
19		397	405	385	400	414	401	5	N2	400	10	-0.5
20		425	424	432	435	426	439	2	N2	430	6	0.0
21	B	413	410	393	400	392		3	E5	402	10	-0.5
21	C	417	407	410				3	E5	411	5	-0.3
22		482	513	479	501	500	465	1	X3	490	18	1.2
23		445	433	438	444	438	449	0.0006	N2	441	6	0.3
24		663	674	661	664	658		1	X1	664	6	4.5
25		340	360	340	320	380		2.2	X3	348	23	-1.5
27	B	368	379					42	A1	374	8	-1.0
27	A	401	369	368	372	349	368	0.47	X3	371	17	-1.1
28		384	382	381	365	367		0.3	M2	376	9	-1.0
29		134	123	123				1	M2	127	6	-5.7
30		434	469	458	449	454	425	20	N2	448	16	0.4
31		290	300	280	290	285	290	0.002	A1	289	7	-2.6
32		475	479	518	518			20	X1	498	24	1.3
33	A	580	537	526	530	560	526		A1	543	22	2.2
33	C	508	580	570	600	540	542	100	E5	557	33	2.4
36		319	296	438	435			200	N2	372	75	-1.1
37		460	492	441	441	450	486	5	N4	462	22	0.6
38		447	362	427	369	409		5	N2	403	37	-0.5
39		448.4	463		450.2	454		1.45	A1	454	7	0.5
40		0.8	1.1	1	1.1	0.8	1.1	1.45	N1	1	0	-8.1
41		427.9	425.8	424.2	424.6	437.1	433.1	6.6	N2	429	5	0.0

Element: Ga **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
20	C	0.267	0.295	0.193	0.405	0.267	0.226	0.1	N2	0.276	0.073
28		0.55	0.57	0.57	0.563	0.572		0.001	M2	0.565	0.009
29		0.11	0.097	0.11	0.087			0.001	M2	0.101	0.011

Element: Gd **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5				0.932	0.806	0.908	0.917	0.056	N2	0.891	0.057
20	C	0.159	0.136	0.118		0.107	0.143	0.056	N2	0.133	0.021

Element: Hf **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5		0.0571	0.0603	0.0582	0.0603	0.0606	0.0558	0.0023	N2	0.059	0.002
6		0.05	0.061	0.05				0.01	N2	0.054	0.006
10	B	0.049	0.062	0.044	0.06	0.055	0.053	0.082	N2	0.054	0.007
17		0.0583	0.0575	0.0552	0.0535	0.0511	0.0592	0.0025	N2	0.056	0.003
20	C	0.069	0.057	0.065	0.059	0.062	0.068	0.003	N2	0.063	0.005
23		0.058	0.056	0.058	0.058	0.061	0.061	0.0039	N2	0.059	0.002
40		0.058	0.034	0.023	0.023	0.029	0.04	0.0039	N1	0.035	0.013

Element: Hg **Mean: 0.20** **Upper Limit: 0.24** **Lower Limit: 0.16** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
4		0.17	0.19	0.19	0.19				A4	0.185	0.010	-0.4
5		0.2526	0.2575	0.2514	0.2159	0.2571	0.2515	0.0089	N2	0.248	0.016	1.0
7	B	0.177	0.174	0.171	0.171	0.178		0.0002	A4	0.174	0.003	-0.7
8	C	0.225	0.215	0.24	0.208	0.233	0.226	0.02	A4	0.225	0.012	0.5
10	A	0.165	0.162	0.162	0.16	0.174	0.168	0.002	N1	0.165	0.005	-0.9
10	B	0.146	0.14	0.151	0.16	0.135	0.125	0.028	N2	0.143	0.012	-1.4
17		0.131	0.155	0.128	0.229	0.131	0.129	0.0053	N2	0.151	0.040	-1.3
20	A	0.201	0.205	0.199				0.0001	A4	0.202	0.003	0.0
20	B	0.233	0.219	0.204				0.0001	N1	0.219	0.015	0.4
23		0.22	0.19	0.21	0.24	0.21	0.19	0.0156	N2	0.210	0.019	0.2
26	A	0.5	0.25	0.25	0.25	0.25	0.25	0.292	A4	0.292	0.102	2.1
36		0.22	0.16					0.1	N2	0.190	0.042	-0.3
38	A	0.184	0.178	0.173				0.1	N2	0.178	0.006	-0.6
40		0.25	0.25	0.29	0.29	0.24	0.3		N1	0.270	0.026	1.6
41	A	0.214	0.192	0.224	0.208	0.207	0.185	0.0672	N2	0.205	0.014	0.0

Element: Ho **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
28		0.012	0.013	0.011				0.002	M2	0.01	0.001

Element: I **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
12	B	3.3	3.2	3.2				1	N2	3.23	0.06
17		3.49	3.09	3.44	4.27	3.01	3.14	0.16	N2	3.41	0.47
20	B	3.08	2.76	2.99				0.0005	N1	2.94	0.17
40		3.1	3.3	3.1				0.0005	N1	3.17	0.12

Element: K **Mean: 1840** **Upper Limit: 2040** **Lower Limit: 1640** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
4		1950	1900	1950	2020	1932			A1	1950	44	0.8
5		1970.6	1875.4	1833.5	1799.6	1922.7	1754.2	53.3	N2	1859	80	0.2
6		1704	1583	1679	1648	1632	1585	18	N2	1639	49	-1.4
7	D	1750	1810	1750	1760	1730		0.1	E5	1760	30	-0.5
9		1826	1741	1799	1836	1808	1797	180	N2	1801	33	-0.2
10	B	1837	1861	1774	1791	1803	1751	120	N2	1803	41	-0.2
12	B	1890	2000	2000				20	N2	1963	64	0.9
13	A	1690	1620	1780				100	A1	1697	80	-1.0
14		2607	2543	2659	2725			100	X1	2634	77	5.6
16	A	1606	1607	1665	1612	1639	1625	0.125	A1	1626	23	-1.5
17		1950	2050	1970	1970	1840	1940	170	N2	1953	68	0.8
18		2050	1990	2020	1970	2010	1990	22	X3	2005	28	1.2
20	C	1827	1826	1822	1931	1879	1917	10	N2	1867	49	0.2
21	A	1580	1610	1610	1730	1810	1740	0.3	A1	1680	93	-1.1
22		2560	2580	2300	2520	2470	2150	0.02	X3	2430	170	4.2
23		1730	1780	1790	1840	1800	1800	250	N2	1790	36	-0.3
24		2200	2200	1900	1900	1900		3	X1	2020	164	1.3
25		1060	1070	840	1320	1070		197	X3	1072	170	-5.3
27	A	1846	1798	1855				300	A1	1833	31	0.0
27	B	1385	948	1120	1241	582	1232	465	X3	1085	286	-5.2
30		1500	1500	1800	1700	2000	1600	0.03	N2	1683	194	-1.1
31		1800	1800	1810	1815	1800	1800	0.001	A1	1804	7	-0.2
32		2150	2080	2290	2060			156	X1	2145	104	2.2
35		1980	2070	2100	2030	2060		400	N2	2048	45	1.5
38	A	1800	1820	1850				400	N2	1823	25	-0.1
38	B	1930							N5	1930		0.7
39		1718	1755		1812	1756		8.61	A1	1760	39	-0.5
40		1660	1630	1640	1580	1640	1560	8.61	N1	1618	39	-1.5

Element: La **Mean: 0.66** **Upper Limit: 0.76** **Lower Limit: 0.56** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		0.86	0.84	0.93	0.81	0.84	0.86	0.07	N2	0.857	0.040	1.9
5		0.77	0.666	0.64	0.664	0.66	0.5	0.093	N2	0.650	0.087	-0.1
9		0.69	0.724	0.676	0.733	0.734	0.669	0.062	N2	0.704	0.029	0.4
10	B	0.367	0.082	0.117	0.058	0.06	0.055	0.016	N2	0.123	0.122	-5.4
11		0.596	0.491	0.519	0.531	0.549		0.0004	M2	0.537	0.039	-1.3
12	B	0.71	0.75	0.75				0.02	N2	0.737	0.023	0.7
17		0.625	0.723	0.648	0.636	0.645	0.616	0.01	N2	0.649	0.038	-0.1
20	C	0.642	0.605	0.609	0.562	0.639	0.639	0.025	N2	0.616	0.031	-0.5
23		0.61	0.58	0.6	0.67	0.62	0.64	0.025	N2	0.620	0.032	-0.4
28		0.561	0.51	0.68				0.0008	M2	0.584	0.087	-0.8
35		0.59	0.58	0.59	0.56	0.6		0.15	N2	0.584	0.015	-0.8
38	A	0.877	0.82	0.781				0.15	N2	0.826	0.048	1.6
41	A	0.626	0.578	0.585	0.579	0.6	0.594	0.0075	N2	0.594	0.018	-0.7

Element: Li **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
11		0.367	0.36	0.365	0.408	0.399		0.09	M2	0.380	0.022
16	B	0.374	0.381	0.365	0.384			3.5E-6	M1	0.376	0.008
28		0.46	0.43	0.43	0.42	0.47		0.01	M2	0.442	0.022
29		0.27	0.26	0.25					M2	0.260	0.010

Element: Lu **Mean: 0.0066** **Upper Limit: 0.0090** **Lower Limit: 0.0042** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.0104	0.0078	0.0057	0.00791	0.00561	0.00613	0.005	N2	0.0073	0.0018	0.4
10	B	0.011	0.01	0.008	0.009	0.007	0.008	0.002	N2	0.0088	0.0015	1.3
17		0.0052	0.0073	0.0071	0.0063	0.00668	0.00576	0.00039	N2	0.0064	0.0008	-0.1
23		0.0068	0.0067	0.0074	0.0061	0.0062	0.0067	0.0027	N2	0.0067	0.0005	0.0
28		0.005	0.005	0.002				0.0008	M2	0.0040	0.0017	-1.5

Element: Mg **Mean: 580** **Upper Limit: 690** **Lower Limit: 470** **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
1		609	622	614	596	624	625	3.4	E5	615	11
3		596	594	643	625	594	632	44	N2	614	22
4		530	630	620	620	590			A1	598	41
5		530.5	606.3	803.3	473.3	479.9	618.7	13.6	N2	585	123
7	D	654	656	647	647	651		0.1	E5	651	4
11		459.19	461.71	447.91	498.84	542.87		0.21	M2	482	39
13	C	562	564	561				10	E5	562	2
14		386	374	336	392			10	X1	372	25
16	B	432	419	416	418	439	425	0.00025	E5	425	9
17		616	741	727	810	770	829	25	N2	749	76
21	B	643	610	651	630	635	640	0.3	E5	635	14
21	C	590	600					0.3	E5	595	7
24		270	344	204	255	181		58	X1	251	64
27	A	884	880	909				29	A1	891	16
29		142	132	133	131	131	118	0.1	M2	131	8
31		720	725	715	720	722	720	0.001	A1	720	3
33	A	565	616	560	616	520	560	40	A1	573	37
33	C	580	586	554	555	596	615	2	E5	581	24
36		940	1060					200	N2	1000	85
39		631	601	675	649			3.59	A1	639	31
40		3300	2600	3000	2500	2500	3000	3.59	N1	2817	331

Element: Mn **Mean: 63** **Upper Limit: 70** **Lower Limit: 56** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		64.8	65.9	65.9	65.3	66.4	66.3	0.1	E5	65.8	0.6	0.4
2	A	70.8	72.4	74	72.9	73.7	73.3	0.34	A1	72.9	1.2	1.4
3		71	70	72	73	71	74	0.39	N2	71.8	1.5	1.2
4		68.42	73.31	72.51	72.49	67.74			A1	70.9	2.6	1.1
5		52.5	59.2	54	48.7	49.5	54.7	1.4	N2	53.1	3.8	-1.5
6		58	59	58	58	58	55	0.5	N2	57.7	1.4	-0.8
7	D	62.1	61.4	61.4	61.7	61.3		0.1	E5	61.6	0.3	-0.3
10	B	63.8	68.2	66.4	65.7	66.2	64.9	0.54	N2	65.9	1.5	0.4
11		57.59	59.39	56.59	60.42	63.32		4	M2	59.5	2.6	-0.6
12	B	66	65	66				0.1	N2	65.7	0.6	0.3
13	C	65	68	67	67.6	68.1		2	E5	67.1	1.3	0.5
14		73.9	75.1	73.1	78.7			10	X1	75.2	2.5	1.7
15		55						0.5	X1	55.0		-1.2
16	B	53	55	54	53	55	54	0.0025	E5	54.0	0.9	-1.4
17		69.8	66.2	69.3	68.2	71.9	71.4	0.048	N2	69.5	2.1	0.9
18		70	60	63	62	65	59	0.4	X3	63.2	4.0	0.0
21	C	68	65	66	64	64	65	0.5	E5	65.3	1.5	0.3
22		68.7	69.3	57	71	68.5	68.2	0.1	X3	67.1	5.1	0.5
23		60.2	61.6	61.2	61.5	60.7	59.6	48.0E-6	N2	60.8	0.8	-0.4
24		86	86	83	84	83		2	X1	84.4	1.5	3.1
25		71	73	70	72	74		6	X3	72.0	1.6	1.3
27	B	56.9	54	52.8	56.7	52.4	53.5	0.57	X3	54.4	2.0	-1.3
28		62.4	60	64	61	63.2		0.07	M2	62.1	1.6	-0.2
29		16.7	16.9	17.2	17	16.8	17.1	0.01	M2	17.0	0.2	-6.7
31		100	98	100	105	100	100	0.001	A1	100.5	2.3	5.4
32		50	36	40	54			22	X1	45.0	8.4	-2.7
35		58	60.8	57.6	59.3	59.2		2	N2	59.0	1.3	-0.6
36		11	12.6					2.4	N2	11.8	1.1	-7.5
39		66.94	66.81		68.44	65.29		0.187	A1	66.9	1.3	0.5
40		63	64	63	62	63	62	0.187	N1	62.8	0.8	-0.1
41	B	67.99	66.66	65.56	68.24	68.33	66.27		A1	67.2	1.2	0.6
42	A	71.6	65	65.2	64.1	66.5	65.6	2.4	X2	66.3	2.7	0.4
42	B	63.7	64.4	63.2	37.2	65.2	64.6	2	A1	59.7	11.1	-0.5

Element: Mo **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
10	A	0.151	0.161	0.152	0.14	0.134	0.11	0.025	N1	0.141	0.018
11		0.059	0.052	0.054	0.055			0.009	M2	0.055	0.003
20	C	0.308	0.376	0.164	0.181	0.192	0.366	0.03	N2	0.265	0.097
38	A	0.151						0.03	N2	0.151	

Element: Na **Mean: 320** **Upper Limit: 360** **Lower Limit: 280** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		320	314	329	326	335	347	14	N2	329	12	0.1
4		300	290	300	290	280			A1	292	8	-0.9
5		374.6	367.4	364.6	343	377.34	343.9	0.36	N2	362	15	1.1
6		293	283	296	292	290	277	1	N2	289	7	-1.0
7	D	280	283	282	281	283		0.1	E5	282	1	-1.2
9		323	314	330	319	318	342	5.7	N2	324	10	0.0
10	B	308	309	300	305	295	290	8.4	N2	301	8	-0.6
11		264.75	271.56	249.33	286.04	303.94		8.49	M2	275	21	-1.3
12	B	310	320	330				0.02	N2	320	10	-0.1
13	A	348	360	377				300	A1	362	15	1.1
14		138	129	147	150			10	X1	141	9	-5.1
16	A	321	332	326	327	330	328	0.025	A1	327	4	0.1
17		350	355	355	354	367	376	1.8	N2	360	10	1.0
20	C	328	324	328	342	339	345	0.15	N2	334	9	0.3
21	A	296	291	292	279	284	297	0.2	A1	290	7	-0.9
23		386	384	386	396	395	372	0.00022	N2	387	9	1.8
27	A	1318	1127	1395				32	A1	1280	138	27
30		344	348	375	378	355	346	5	N2	358	15	1.0
31		510	510	505	508	507	510	0.001	A1	508	2	5.2
35		325	325	310	314	319		10	N2	319	7	-0.1
36		610	610	855	855	1045	1045	600	N2	837	195	14
37		386	370	422	375	384	358	1	N4	383	22	1.6
38	A	317	304	312				1	N2	311	7	-0.3
39		689	631	651	701			15.3	A1	668	33	9.6
40		270	270	270	260	270	260	15.3	N1	267	5	-1.6

Element: Nd **Mean: 0.60** **Upper Limit: 0.42** **Lower Limit: 0.78** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.581	0.532	0.419	0.4185	0.487	0.502	0.077	N2	0.4899	0.0638	-0.9
17		0.894	0.923	0.582				0.15	N2	0.7997	0.1891	1.5
20	C	0.657	0.691	0.524	0.77	0.731	0.607	0.06	N2	0.6633	0.0887	0.5
23		0.53	0.49	0.54	0.54	0.53	0.52	0.23	N2	0.5250	0.0187	-0.6
28		0.462	0.53	0.58				0.0008	M2	0.5240	0.0592	-0.6

Element: Ni **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
1		1.6	1.2	1.2				0.01	E5	1.33	0.23
2	A	1.04	0.99	1.14	0.96	1.14	0.94	0.27	A1	1.04	0.09
7	F	3.36	3.36	3.01	2.93	2.97	3.36	0.005	V1	3.17	0.22
8	B	1.35	1.62	1.43	1.35	1.94	1.51	0.2	A2	1.53	0.22
11		1.245	0.863	0.786	1	0.922		0.252	M2	0.96	0.18
13	B	1.09	1.15	1.11	1.19	1.21		0.3	A3	1.15	0.05
14		2.3	1.9					0.2	X1	2.10	0.28
15		1						0.1	X1	1.00	
16	B	1.23	1.18	1.18	1.22	1.18	1.18	0.0025	E5	1.20	0.02
22		2.77	4.06	2.9	3.02	2.84	2.12	0.6	X3	2.95	0.63
25		3						1.2	X3	3.00	
27	B	2.95	3.56	2.67	2.44	6.63	5.93	0.32	X3	4.03	1.80
28		0.98	0.97	0.98	0.98	0.94		0.01	M2	0.97	0.02
29		0.6	0.7	0.6				0.2	M2	0.63	0.06
38	A	1.69	1.62	1.45				0.01	N2	1.59	0.12
41	B	0.833	1.171	0.825	0.723	0.764	0.747		A1	0.84	0.17
41	A	1.09	1.21	1.44	1.37	1.84		1.58	N2	1.39	0.29
42	A	4.5	2.4	3.7	2.5	2.2	4	1	X2	3.22	0.97

Element: P **Mean: 610** **Upper Limit: 730** **Lower Limit: 490** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
7	D	565	576	573	588	556		0.1	E5	572	12	-0.2
11		875	842	787	903	1009		86	M2	883	83	1.8
13	C	499	513	495				20	E5	502	9	-0.7
14		565	547	697				50	X1	603	82	0.0
16	B	530	540	515	525			0.05	E5	528	10	-0.5
18		590	695	613	619	886	646	46	X3	675	110	0.4
21	B	570	575					20	E5	573	4	-0.2
24		763	567	520	554	548		67	X1	590	98	-0.1
31		21.1	22.2	21.8	21.5	21.2	21.1	0.001	C1	21	0	-3.8
33	B	430	344	350				10	C1	375	48	-1.5
33	C	436	440	408	400	403	405	25	E5	415	18	-1.2
34	A	830	827	825					C1	827	3	1.4
36		720	770	840	690			200	N2	755	66	1.0

Element: Pb **Mean: 4.9** **Upper Limit: 5.5** **Lower Limit: 4.3** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		4.4	6.2	5.4				0.01	E5	5.33	0.90	1.0
2	A	4.88	5.02	5.17	5.2	5.4	5.07	1.35	A1	5.12	0.18	0.5
2	B	5.03	4.99	5.03				0.17	A2	5.02	0.02	0.2
4		4.24	4.36	4.27	4.62	4.47			A3	4.39	0.16	-1.2
7	A	4.89	5.08	4.98	5.1	4.82		0.006	A3	4.97	0.12	0.1
7	E	4.42	4.99	4.66	4.74	4.56	4.63	0.002	M1	4.67	0.19	-0.6
7	F	5.18	5.26	4.83	4.91	4.86	5.13	0.005	V1	5.03	0.18	0.2
8	B	4.51	4.55	4.57	5	5.1	4.52	0.25	A2	4.71	0.27	-0.5
11		4.81	4.54	4.78	4.73	4.73		0.024	M2	4.72	0.11	-0.5
13	B	4.21	4.06	5.31	5.03	6.24	5.32	0.2	A3	5.03	0.80	0.2
14		10	8	7.7	7.5			0.5	X1	8.30	1.15	8.0
16	B	4.86	4.84	4.92	4.83	5.02	4.82	0.005	E5	4.88	0.08	-0.1
18		6.7	6.1	6.3	8.6	7	7.8	1.6	X3	7.08	0.95	5.1
22		5.57	3.57	3.79	2.92	4.8		0.3	X3	4.13	1.05	-1.9
25		6			6			5.4	X3	6.00	0.00	2.5
27	B	5.87	7.17	5.34	5.55	6.01	5.39	1.09	X3	5.89	0.68	2.3
28		4.7	4.73	4.8	4.7	4.6		0.01	M2	4.71	0.07	-0.5
29		1.66	1.68	1.53	1.61	1.56		0.01	M2	1.61	0.06	-7.8
31		0.264	0.233	0.25	0.233	0.233	0.25	0.001	V1	0.24	0.01	-11.0
32		13	9	10	11			5	X1	10.75	1.71	13.7
41	B	4.868	5.226	5.02	4.871	5.313	4.951		A1	5.04	0.19	0.3
42	A	4.5	4.8	4.9	5.5	4.9	5.2	1.3	X2	4.97	0.34	0.1
44*		4.99	5.02	5.16	4.81	4.71	4.79	0.01	A3	4.91	0.17	0.0
45*		6.75	6.07	6.96	5.96	7.08	6.2	2.215	X3	6.50	0.49	3.7
46*		4.64	4.76	5.07	5.12	5.03	5.08	0.52	E5	4.95	0.20	0.1
47*		4.65	4.15	4.05				0.0003	M2	4.28	0.32	-1.5
48*		4.56	4.60	4.66	4.61	4.64	4.73	1	E5	4.63	0.06	-0.7
49*		4.09	5.75	5.1				3.125	E5	4.98	0.84	0.1
50*		4.51	4.26	5.05	4.96	5.48			M2	4.85	0.48	-0.2

* Additional data from a CRP was included in the evaluation

Element: Pr **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
28		0.137	0.139	0.146				0.0004	M2	0.141	0.005

Element: Rb **Mean: 1.76** **Upper Limit: 1.98** **Lower Limit: 1.54** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		1.917	1.867	1.967	1.836	2.068	1.901	0.088	N2	1.93	0.08	0.8
9		1.71	1.59	1.68	1.54	1.5	1.75	0.63	N2	1.63	0.10	-0.7
10	B	1.61	1.74	1.53	1.55	1.84	1.6	0.3	N2	1.65	0.12	-0.6
11		1.59	1.57	1.61	1.74	1.66		0.0012	M2	1.63	0.07	-0.7
17		1.57	1.48	1.68	1.93	1.62	1.89	0.14	N2	1.70	0.18	-0.4
18		2.1	1.6	1.1	2.3	1.7	3	1	X3	1.97	0.66	1.1
20	C	1.76	1.73	1.74	1.76	1.79	1.75	0.1	N2	1.76	0.02	0.0
23		1.65	1.69	1.71	1.65	1.54	1.64	0.18	N2	1.65	0.06	-0.6
24		2	2	3	2	2		2	X1	2.20	0.45	2.3
27	B	2.39	2.05	2.72	1.77	1.45	1.38	0.47	X3	1.96	0.53	1.0
28		1.73	1.73	1.65	1.66	1.67		0.004	M2	1.69	0.04	-0.4
29		0.34	0.36	0.35	0.3	0.34	0.34	0.06	M2	0.34	0.02	-7.3
36		1.4	1.8	1.4				1	N2	1.53	0.23	-1.2
37		2	2	2.03	1.84	1.85	1.96	0.5	N4	1.95	0.08	0.9
38	A	1.72	1.7	1.68				1	N2	1.70	0.02	-0.3
40		1.5	1.7	1.2	1.6	1.4	1.3	0.5	N1	1.45	0.19	-1.6
42	A	1.6	1.9	1.8	2	1.8	1.9	0.9	X2	1.83	0.14	0.4

Element: S **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
7	D	734	717	704	726	714		0.1	E5	719	11
11		551	396	450	480	619		254	M2	499	87
18		783	861	797	799	964	809	30	X3	836	68
24		959	894	815	847	838		27	X1	871	57
38	B	7000	2400					30	N5	4700	3253

Element: Sb **Mean: 0.073** **Upper Limit: 0.083** **Lower Limit: 0.063** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		0.07	0.08	0.06				0.01	E5	0.070	0.010	-0.3
5		0.0909	0.0864	0.0951	0.0816	0.0935	0.0919	0.0036	N2	0.090	0.005	1.7
6		0.07	0.08	0.07	0.07	0.07	0.07	0.02	N2	0.072	0.004	-0.1
10	A	0.074	0.069	0.069	0.075	0.076	0.07	0.008	N1	0.072	0.003	-0.1
10	B	0.068	0.047	0.05	0.068	0.066	0.05	0.008	N2	0.058	0.010	-1.5
10	A	0.048	0.053	0.049	0.073	0.073	0.054	0.0008	N2	0.058	0.012	-1.5
17		0.0701	0.0671	0.0708	0.0788	0.0909	0.0998	0.0047	N2	0.080	0.013	0.7
20	C	0.075	0.078	0.07	0.069	0.067	0.072	0.005	N2	0.072	0.004	-0.1
23		0.063	0.062	0.063	0.082	0.087	0.096	0.0098	N2	0.076	0.015	0.3
37		0.195	0.174	0.206	0.141	0.12	0.14	0.01	N4	0.163	0.034	9.1
38	A	0.0967	0.102	0.0815	0.0778	0.0947	0.0763	0.0098	N2	0.088	0.011	1.5
40		0.072	0.075	0.073	0.076	0.065	0.084	0.01	N1	0.074	0.006	0.1
41	A	0.0638	0.0651	0.0638	0.0659	0.065	0.0702	0.00473	N2	0.066	0.002	-0.7

Element: Sc **Mean: 0.17** **Upper Limit: 0.19** **Lower Limit: 0.15** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		0.189	0.179	0.181	0.172	0.189	0.17	0.006	N2	0.180	0.008	0.4
5		0.2047	0.2073	0.204	0.1996	0.2132	0.2016	0.0004	N2	0.205	0.005	2.0
6		0.19	0.19	0.18				0.0015	N2	0.187	0.006	0.8
9		0.147	0.147	0.131	0.131	0.141	0.136	0.004	N2	0.139	0.007	-2.1
10	B	0.166	0.168	0.16	0.168	0.16	0.158	0.0011	N2	0.163	0.005	-0.6
12	B	0.166	0.169	0.178				0.002	N2	0.171	0.006	-0.1
17		0.188	0.185	0.184	0.185	0.179	0.182	0.00033	N2	0.184	0.003	0.7
19		0.158	0.16	0.147	0.161	0.168	0.156	0.005	N2	0.158	0.007	-0.9
20	C	0.181	0.181	0.186	0.185	0.18	0.185	0.0005	N2	0.183	0.003	0.6
23		0.17	0.17	0.17	0.17	0.17	0.17	0.001	N2	0.170	0.000	-0.2
37		0.18	0.174	0.184	0.163	0.174	0.185	0.0005	N4	0.177	0.008	0.2
38	A	0.117	0.1155	0.1077	0.108	0.102	0.103	0.001	N2	0.109	0.006	-3.9
40		0.155	0.164	0.153	0.162	0.156	0.151	0.0005	N1	0.157	0.005	-1.0
41	A	0.1743	0.1718	0.1698	0.1694	0.1729	0.1733	0.00047	N2	0.172	0.002	0.0

Element: Se **Mean: 0.22** **Upper Limit: 0.26** **Lower Limit: 0.18** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.2597	0.272	0.248	0.2358	0.2642	0.286	0.023	N2	0.2610	0.0177	1.0
6		0.22	0.19	0.22				0.1	N2	0.2100	0.0173	-0.3
7	C	0.217	0.222	0.219	0.22	0.224		0.0005	A5	0.2204	0.0027	-0.1
10	A	0.201	0.21	0.212	0.208	0.207	0.21	0.005	N1	0.2080	0.0038	-0.4
10	B	0.211	0.203	0.166	0.23	0.198	0.159	0.06	N2	0.1945	0.0272	-0.7
17		0.217	0.222	0.224	0.192	0.195	0.214	0.016	N2	0.2107	0.0138	-0.3
19		0.24	0.25	0.22				0.03	N2	0.2367	0.0153	0.4
20	C	0.218	0.225	0.208	0.229	0.212	0.214	0.018	N2	0.2177	0.0080	-0.1
23		0.27	0.28	0.21	0.26	0.29	0.27	0.053	N2	0.2633	0.0280	1.0
26	B	0.7161	0.7335	0.7281	0.7238	0.7228	0.7269	0.00244	F1	0.7252	0.0058	13
29		0.25	0.34	0.32				0.1	M2	0.3033	0.0473	2.1
36		0.014	0.012					0.01	N2	0.0130	0.0014	-5.5
37		0.61	0.5	0.41	0.47	0.6	0.72	0.05	N4	0.5517	0.1127	8.5
38	A	0.165	0.146	0.158				0.01	N2	0.1563	0.0096	-1.7
41	A	0.185	0.211	0.169	0.188	0.19	0.232	0.095	N2	0.1958	0.0222	-0.7

Element: Si **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
11		153	175	153	179	201		3	M2	172	20
14		1053	1051	1080	1052			100	X1	1059	14
16	B	1387	1666	1515	1723			0.025	E5	1573	152
18		1800	1900	1870	1840	2160	1810	48	X3	1897	134
21	B	1920	1840	1870				5	E5	1877	40
22		211	195	312	270	359	363	25	X3	285	72
31		1400	1410	1400	1415	1405	1400	0.01	C1	1405	6

Element: Sm **Mean: 0.106** **Upper Limit: 0.120** **Lower Limit: 0.092** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		0.12	0.15	0.15	0.13	0.13	0.14	0.01	N2	0.137	0.012	1.9
5		0.0898	0.1019	0.0797	0.1446	0.09523	0.0972	0.0063	N2	0.101	0.022	-0.3
6		0.09	0.11	0.09	0.09	0.09	0.08	0.01	N2	0.092	0.010	-0.9
9		0.14	0.136	0.132	0.137	0.139	0.14	0.007	N2	0.137	0.003	1.9
10	B	0.095	0.097	0.091	0.096	0.093	0.095	0.0022	N2	0.095	0.002	-0.7
12	A	0.109	0.118	0.105				0.0022	N2	0.111	0.007	0.3
12	B	0.091	0.089	0.087				0.002	M2	0.089	0.002	-1.1
17		0.117	0.129	0.115	0.113	0.109	0.115	0.0012	N2	0.116	0.007	0.6
20	C	0.119	0.118	0.103	0.107	0.115	0.125	0.004	N2	0.115	0.008	0.5
23		0.12	0.11	0.11	0.1	0.1		0.0022	N2	0.108	0.008	0.1
28		0.103	0.101	0.1				0.002	M2	0.101	0.002	-0.3
30		0.11	0.11	0.12	0.12	0.11	0.12	0.01	N2	0.115	0.005	0.5
35		0.099	0.1	0.105	0.098	0.102		0.01	N2	0.101	0.003	-0.3
38	A	0.0804	0.0852	0.0787				0.01	N2	0.081	0.003	-1.5
40		0.104	0.05	0.108	0.102	0.102	0.1	0.01	N1	0.094	0.022	-0.7

Element: Sn **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
16	B	5.29	5.35	5.35	5.1	5.14	5.16	0.125	E5	5.23	0.11
42	A	8.3	9	9	10.1	8.4	8.5	0.8	X2	8.88	0.67

Element: Sr **Mean: 9.3** **Upper Limit: 10.4** **Lower Limit: 8.2** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		8.6	8.9	8.7	9.1	9	8.9	0.1	E5	8.87	0.19	-0.4
5		12.65	13.04	10.07	8.76	12.33	10.33	39.53	N2	11.20	1.72	1.6
7	D	8.6	8.5	8.6	8.4	8.4		0.05	E5	8.50	0.10	-0.7
10	B	13.2	8.8	12.1	13.2	8.8	7.7	0.58	N2	10.63	2.48	1.1
11		8.07	7.67	8.44	8.46	8.77		426	M2	8.28	0.42	-0.9
12	A	9.4	9	9.2				0.7	M2	9.20	0.20	-0.1
13	C	11	11.4	11.2	11.1	11.2		1	E5	11.18	0.15	1.6
14		9	9.4	8.6	8.3			1	X1	8.83	0.48	-0.4
16	B	7.88	8.23	8.28	7.6			0.0005	E5	8.00	0.32	-1.1
17		9.82	8.17		9.48			1.1	N2	9.16	0.87	-0.1
18		8.8	6.6	6.9	7.2	8.5	6.8	1.2	X3	7.47	0.94	-1.5
20	C	9.5	8.62	9.39	10.7	10.2	9.53	1	N2	9.66	0.72	0.3
21	B	9.3	9.1	9.5	8.3	8	8.3	0.5	E5	8.75	0.63	-0.5
21	C	8.4	8.5	8.4				0.5	E5	8.43	0.06	-0.7
23		9.12	8.29	8.08	9.26	7.79	9.06	1.44	N2	8.60	0.62	-0.6
24		11	12	11	10	11		1	X1	11.00	0.71	1.4
27	B	12.2	11.3	10.9	11.3	10.8	11.6	0.93	X3	11.35	0.51	1.7
28		8.99	8.91	9.1	8.8	8.9		0.006	M2	8.94	0.11	-0.3
29		1.9	2	1.8	2	2.1		0.01	M2	1.96	0.11	-6.2
32		16	12.3	17.9	13			2	X1	14.80	2.62	4.6
38	A	8.42	9.06	8.69				0.01	N2	8.72	0.32	-0.5

Element: Ta **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5		0.016	0.0164	0.0146	0.0153	0.015	0.0142	0.0085	N2	0.0153	0.0008
10	B	0.02	0.019	0.017	0.018	0.015	0.014	0.0043	N2	0.0172	0.0023
17		0.0105	0.015					0.004	N2	0.0128	0.0032
20	C	0.0149	0.0133	0.0143	0.0124	0.0137	0.015	0.002	N2	0.0139	0.0010
23		0.015	0.017	0.015	0.017	0.017	0.018	0.0036	N2	0.0165	0.0012
38	A	0.0165	0.0148	0.0167				0.002	N2	0.0160	0.0010
40		6.4	6.7	6.4	6.5	6.4		0.0036	N1	6.48	0.13

Element: Tb **Mean: 0.014** **Upper Limit: 0.016** **Lower Limit: 0.013** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.0161	0.0114	0.0156	0.0165	0.0158	0.0148	0.0046	N2	0.015	0.002	0.6
10	B	0.018	0.021	0.013	0.013	0.012	0.011	0.0046	N2	0.015	0.004	0.3
17		0.0132	0.0175	0.00965	0.0116	0.00971		0.0023	N2	0.012	0.003	-1.2
20	C	0.0131	0.0127	0.0141	0.0131	0.0151	0.0135	0.002	N2	0.014	0.001	-0.3
23		0.0148	0.0107	0.0158	0.0175	0.0143	0.0171	0.0024	N2	0.015	0.002	0.6
28		0.012	0.013	0.011				0.002	M2	0.012	0.001	-1.4
38	A	0.0163	0.0163	0.0164				0.0024	N2	0.016	0.000	1.4

Element: Th **Mean: 0.14** **Upper Limit: 0.16** **Lower Limit: 0.12** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.1665	0.166	0.1669	0.1589	0.1738	0.1645	0.0239	N2	0.166	0.005	1.3
6		0.13	0.13	0.12	0.13	0.14	0.12	0.03	N2	0.128	0.008	-0.7
9		0.144	0.14	0.149	0.149	0.151	0.149	0.014	N2	0.147	0.004	0.3
10	B	0.028	0.024	0.025	0.02	0.026	0.033	0.0068	N2	0.026	0.004	-6.4
11		0.112	0.109	0.105	0.11	0.116		0.0003	M2	0.110	0.004	-1.7
12	B	0.12	0.133	0.128				0.02	N2	0.127	0.007	-0.8
17		0.137	0.151	0.156	0.149	0.143	0.144	0.0016	N2	0.147	0.007	0.3
20	B	0.132	0.142	0.152	0.143	0.138	0.136	0.0005	N1	0.141	0.007	-0.1
20	C	0.145	0.148	0.15	0.148	0.145	0.154	0.002	N2	0.148	0.003	0.4
23		0.14	0.14	0.14	0.16	0.14	0.15	0.0049	N2	0.145	0.008	0.2
28		0.14	0.143	0.143				0.0008	M2	0.142	0.002	0.0
30		0.19	0.18	0.19	0.17	0.17	0.18	0.02	N2	0.180	0.009	2.1
35		0.154	0.132	0.132	0.158	0.141		0.03	N2	0.143	0.012	0.1
37		0.19	0.16	0.16	0.17	0.16	0.15	0.01	N4	0.165	0.014	1.3
38	A	0.135	0.127	0.129				0.03	N2	0.130	0.004	-0.6
40		0.12	0.125	0.12	0.122	0.112	0.115	0.01	N1	0.119	0.005	-1.3
41	A	0.131	0.126	0.124	0.125	0.134	0.126	0.0194	N2	0.128	0.004	-0.8

Element: Ti **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5		59.7	66.2	59.8		41.1	57.3	35.2	N2	56.8	9.4
10	B	46	53	45	60	45	62	4	N2	51.8	7.7
11		24.59	24.03	23.82	23.86	27.44		0.089	M2	24.7	1.5
13	C	8.41	8.13	7.89	8.01	8.01		1	E5	8.1	0.2
14		87.3	70.8	86.5	75.8			10	X1	80.1	8.1
16	B	52.4	50.5	48.6	51.5			0.004	E5	50.8	1.6
17		68.5	49.3	67.5	67.9	65.1	56.3	4.3	N2	62.4	7.9
18		56	52	52	49	50	51	2	X3	51.7	2.4
22		42.6	40.9	34.9	36.2	36.8	33.2	0.3	X3	37.4	3.6
25		77	61	76	66	70		13.6	X3	70.0	6.7
27	B	66.1	50.8	61.6	56.7	53	58.2	2.33	X3	57.7	5.6
32		130	150	150	210			45	X1	160	35

Element: Tl **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
7	E	0.0107	0.0112	0.011	0.0114	0.0108	0.0116	0.0002	M1	0.011	0.000
11		0.007	0.011	0.008				0.006	M2	0.009	0.002
28		0.008	0.009	0.005	0.006	0.007		0.004	M2	0.007	0.002

Element: Tm **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5		0.0039	0.0069	0.006	0.0062	0.0028			N2	0.005	0.002
20	C	0.0078	0.013	0.014	0.0096	0.014	0.011	0.004	N2	0.012	0.003
28		0.005	0.005	0.003				0.002	M2	0.004	0.001

Element: U **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
5			0.047		0.0488	0.0469	0.048	0.0116	N2	0.048	0.001
11		0.033	0.037	0.035	0.034	0.039		70.0E-6	M2	0.036	0.002
12	A	0.026	0.026	0.024				0.001	M2	0.025	0.001
20	B	0.0456	0.0458	0.05	0.0478	0.0455	0.0452	10.0E-6	N1	0.047	0.002
20	C	0.0575	0.0527	0.0586	0.0569	0.0572	0.0392	0.005	N2	0.054	0.007
28		0.049	0.052	0.047	0.047	0.05		0.005	M2	0.049	0.002
29		0.012	0.01	0.016	0.01	0.01		0.004	M2	0.012	0.003
31		0.031	0.028	0.043	0.043	0.031	0.044	0.005	Z	0.037	0.007
38	A	0.0243						0.004	N2	0.024	
41	A	0.0522	0.0393	0.0415	0.045	0.0506	0.0526	0.0244	N2	0.047	0.006

Element: V **Mean: 1.47** **Upper Limit: 1.69** **Lower Limit: 1.25** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
3		2.83	2.94	3.05	2.83	2.96	2.99	0.24	N2	2.93	0.09	8.8
5		1.51	1.52	1.59		1.182	1.38	0.42	N2	1.44	0.16	-0.2
10	B	1.44	1.44	1.62	1.51	1.59	1.48	0.11	N2	1.51	0.08	0.3
11		1.19	1.25	1.21	1.29	1.31		0.027	M2	1.25	0.05	-1.3
16	B	1.28	1.31	1.29	1.3	1.24	1.26	0.0025	E5	1.28	0.03	-1.1
17		1.72	1.71	1.7	1.53	1.77	1.65	0.043	N2	1.68	0.08	1.3
28		1.45	1.44	1.42	1.38	1.42		0.007	M2	1.42	0.03	-0.3
29		0.34	0.31	0.3	0.34	0.31	0.38	0.005	M2	0.33	0.03	-6.8
35		1.47	1.74	1.82	1.84	1.71		0.3	N2	1.72	0.15	1.5
40		1.6	1.5	1.3				0.007	N1	1.47	0.15	0.0

Element: W **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
38	A	0.185	0.179					0.005	N2	0.182	0.004

Element: Y **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
11		0.312	0.297	0.319	0.335	0.333		0.006	M2	0.319	0.016
17		0.0435	0.0365	0.0431	0.048	0.0428	0.0411	0.0024	N2	0.043	0.004
28		0.335	0.351	0.356				0.0008	M2	0.347	0.011

Element: Yb **Mean: 0.037** **Upper Limit: 0.049** **Lower Limit: 0.025** **Type: I**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
5		0.0258	0.0246	0.0266	0.0244	0.02	0.0215	0.0135	N2	0.024	0.003	-1.5
10	B	0.046	0.044	0.034	0.046	0.04	0.038	0.004	N2	0.041	0.005	0.5
20	C	0.0435	0.0406	0.046	0.0452	0.0412	0.0423	0.002	N2	0.043	0.002	0.7
23		0.046	0.044	0.046	0.044	0.046	0.046	0.013	N2	0.045	0.001	0.9
28		0.033	0.034	0.031				0.002	M2	0.033	0.002	-0.5

Element: Zn **Mean: 30.4** **Upper Limit: 33.8** **Lower Limit: 27.0** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
1		32.8	32.7	31	30.8	32.3	32.1	0.2	E5	32.0	0.9	0.4
2	A	31.2	31.2	31.2	32	32.7	31.2	0.34	A1	31.6	0.6	0.3
3		33.9	33.1	30.6	30.4	34.1	31	2.9	N2	32.2	1.7	0.4
4		32.25	33.83	33.98	34.01	33.59			A1	33.5	0.7	0.7
5		36.326	36.332	36.094	35.066	37.54	35.5	0.00067	N2	36.1	0.8	1.3
6		34	35	34	35	36	34	2.5	N2	34.7	0.8	0.9
7	D	31.4	29.5	31.4	30.6	31.3		0.2	E5	30.8	0.8	0.1
7	E	36.9	34.5	33.1	33.3	32.7	34.4	0.004	M1	34.2	1.5	0.8
7	F	35.2	35.6	35.3	36.1	34.6	35.9	0.005	V1	35.5	0.5	1.1
9		32.9	31.5	30.5	30.6	29.4	31.5	1.7	N2	31.1	1.2	0.2
10	B	28.9	29.7	28.2	30.6	28.7	27.5	0.35	N2	28.9	1.1	-0.3
11		29.56	29.35	29.25	29.37	30.72		1.86	M2	29.7	0.6	-0.2
12	B	29	30	31				1	N2	30.0	1.0	-0.1

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Element: Zn contd. **Mean: 30.4** **Upper Limit: 33.8** **Lower Limit: 27.0** **Type: R**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev	Z-Score
13	C	31.7	27.7	29.2	30.6	30.9	29.4	3	E5	29.9	1.4	-0.1
14		44.5	42.4	43.3	44.1			3	X1	43.6	0.9	2.9
15		30						0.1	X1	30.0		-0.1
16	B	30	29.45	30.5	31.06	30.5	29.8	0.00375	E5	30.2	0.6	0.0
17		31.2	31.3	30.9	30.8	31.2	31	0.13	N2	31.1	0.2	0.2
18		35	30	32	31	32	31	0.3	X3	31.8	1.7	0.3
19		36.7	37.3	36	35.3	34.6	34	0.03	N2	35.7	1.3	1.2
20	C	31.3	31	31.5	32.1	30.7	31.5	0.09	N2	31.4	0.5	0.2
21	B	33	33	32	34	31		0.1	E5	32.6	1.1	0.5
22		36.1	39.9	34.3	40.6	35	30.5	0.6	X3	36.1	3.8	1.3
23		28	27.5	27.6	28.2	28.5	29.3	0.32	N2	28.2	0.7	-0.5
24		37	38	36	36	36		0.5	X1	36.6	0.9	1.4
25		25	27	27	26	22		1.1	X3	25.4	2.1	-1.1
27	B	31.9	29.7	28.4	30.8	28.6	29.6	0.23	X3	29.8	1.3	-0.1
28		28.72	28.7	29	28.6	28.6		0.02	M2	28.7	0.2	-0.4
29		8.6	8.7	8.4	8.2	8.6		0.06	M2	8.5	0.2	-4.8
30		31.7	33.4	40.5	38.3	35.8	37.2	1	N2	36.2	3.2	1.3
32		18	12	16	26			10	X1	18.0	5.9	-2.7
33	A	21	24	21	21	20	20	0.2	A1	21.2	1.5	-2.0
35		20.7	21.2	20	20.1	21.7		8	N2	20.7	0.7	-2.1
36		21.5	20.5	19.5	18.1			5	N2	19.9	1.5	-2.3
37		29.1	28	29.4	29.3	29.4	26.6	1	N4	28.6	1.1	-0.4
38	A	28.96	30.77	29.5	28.8	28.6		0.2	N2	29.3	0.9	-0.2
39		32.17	31.41		32.64	33		0.372	A1	32.3	0.7	0.4
40		26	28	25	28	26	25	5	N1	26.3	1.4	-0.9
41	A	30.56	29.96	29.76	29.72	30.44	30.61	0.098	N2	30.2	0.4	0.0
42	A	32.7	34.5	33.6	35	31.1	33.7	0.4	X2	33.4	1.4	0.7

Element: Zr **Type: N**

Lab. ID		Value 1	Value 2	Value 3	Value 4	Value 5	Value 6	Det.Limit	Method	Lab. Mean	Stdev
20	C	3.5	1.5	4.3	3	3	2.6	2	N2	2.98	0.93
38	A	3.13	2.95	2.67					N2	2.92	0.23

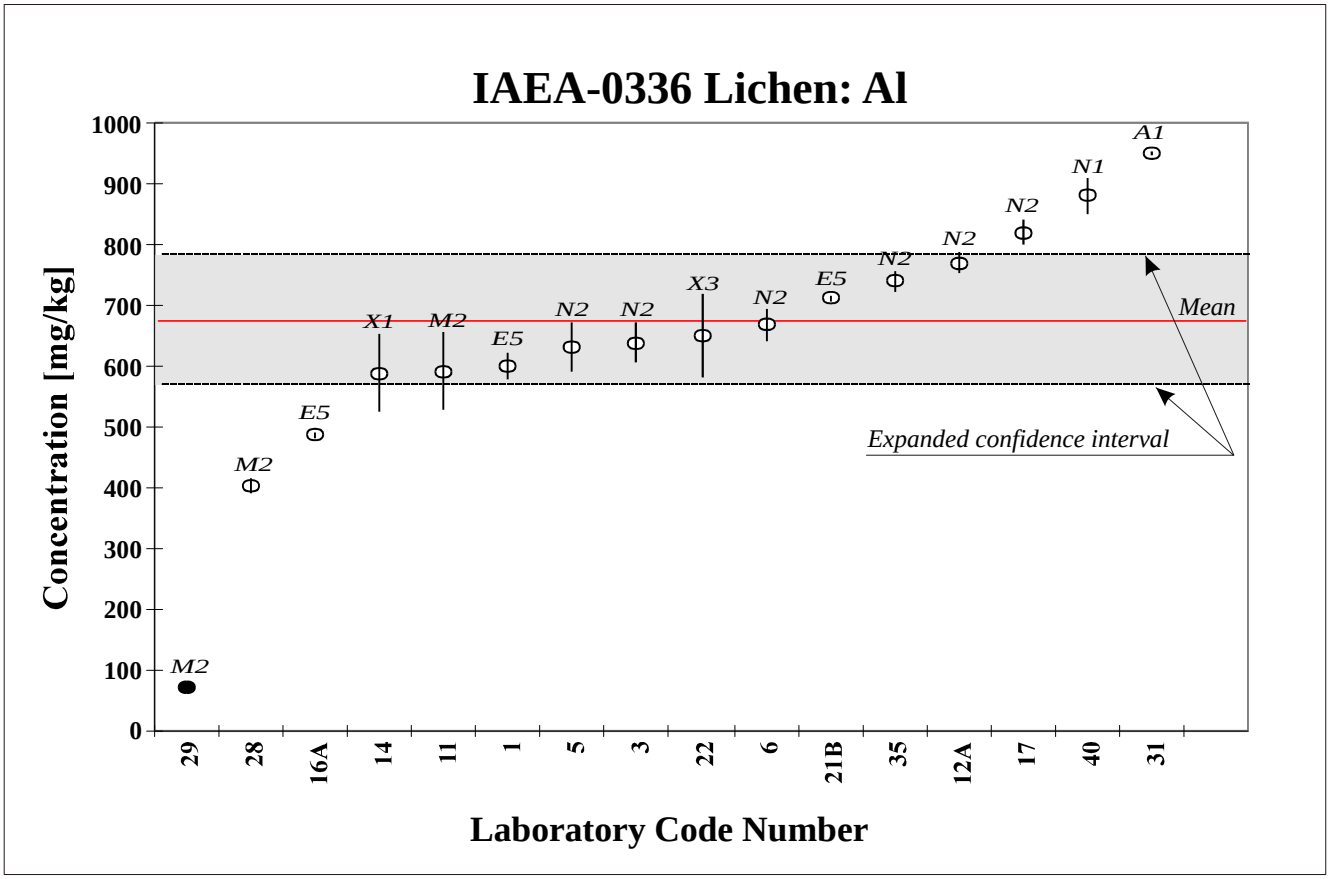
APPENDIX III

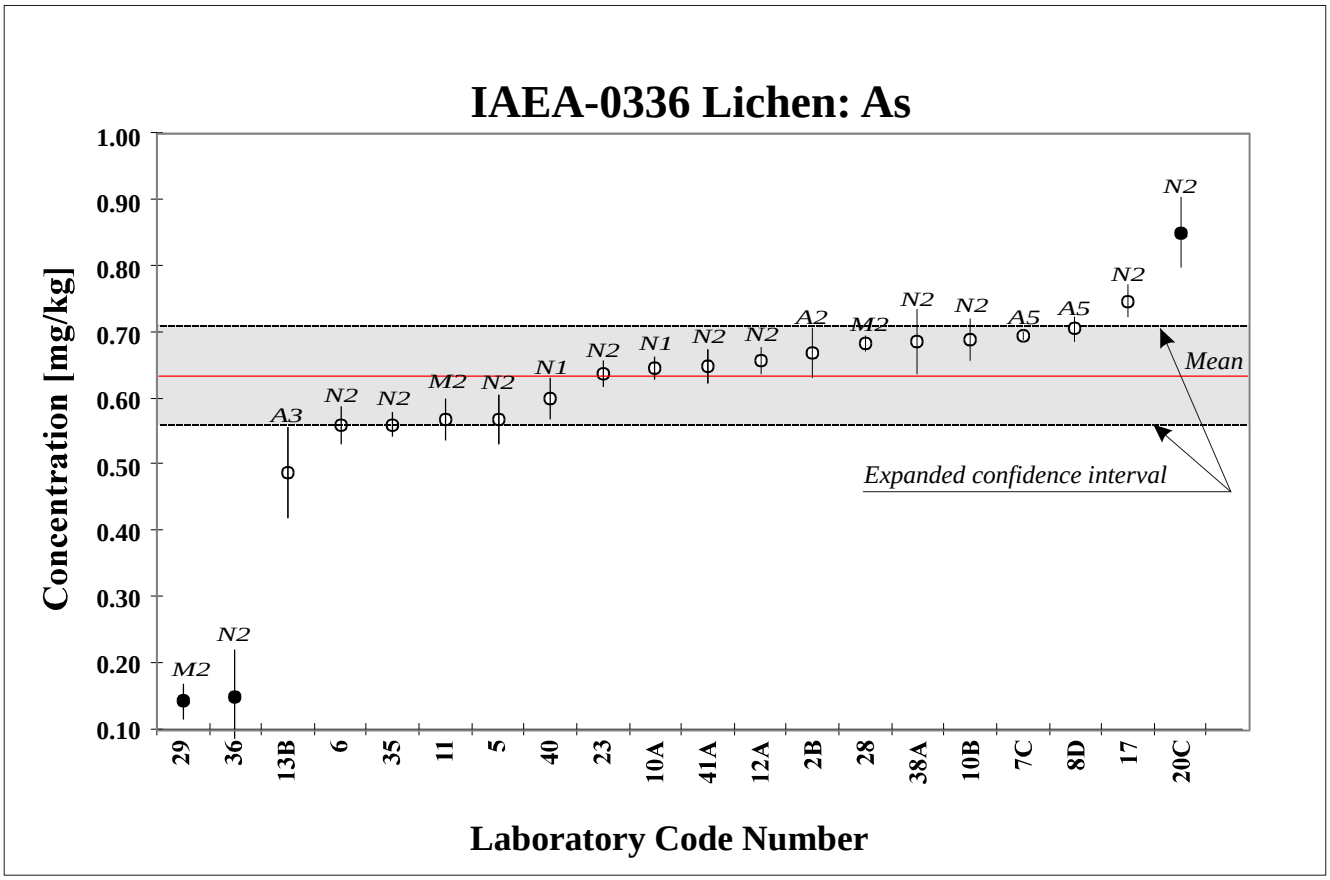
GRAPHICAL PRESENTATION (S-PLOTS)

OF THE RESULTS

SORTED BY ANALYTE

(For a description of the terms and codes used, please refer to Section 7)


IAEA-0336 Lichen: Al
Concentration [mg/kg]
1000
900
800
700
600
500
400
300
200
100
0
Mean
Expanded confidence interval
29 28 16A 14 11 1 5 3 22 6 21B 35 12A 17 40 31
Laboratory Code Number


IAEA-0336 Lichen: As
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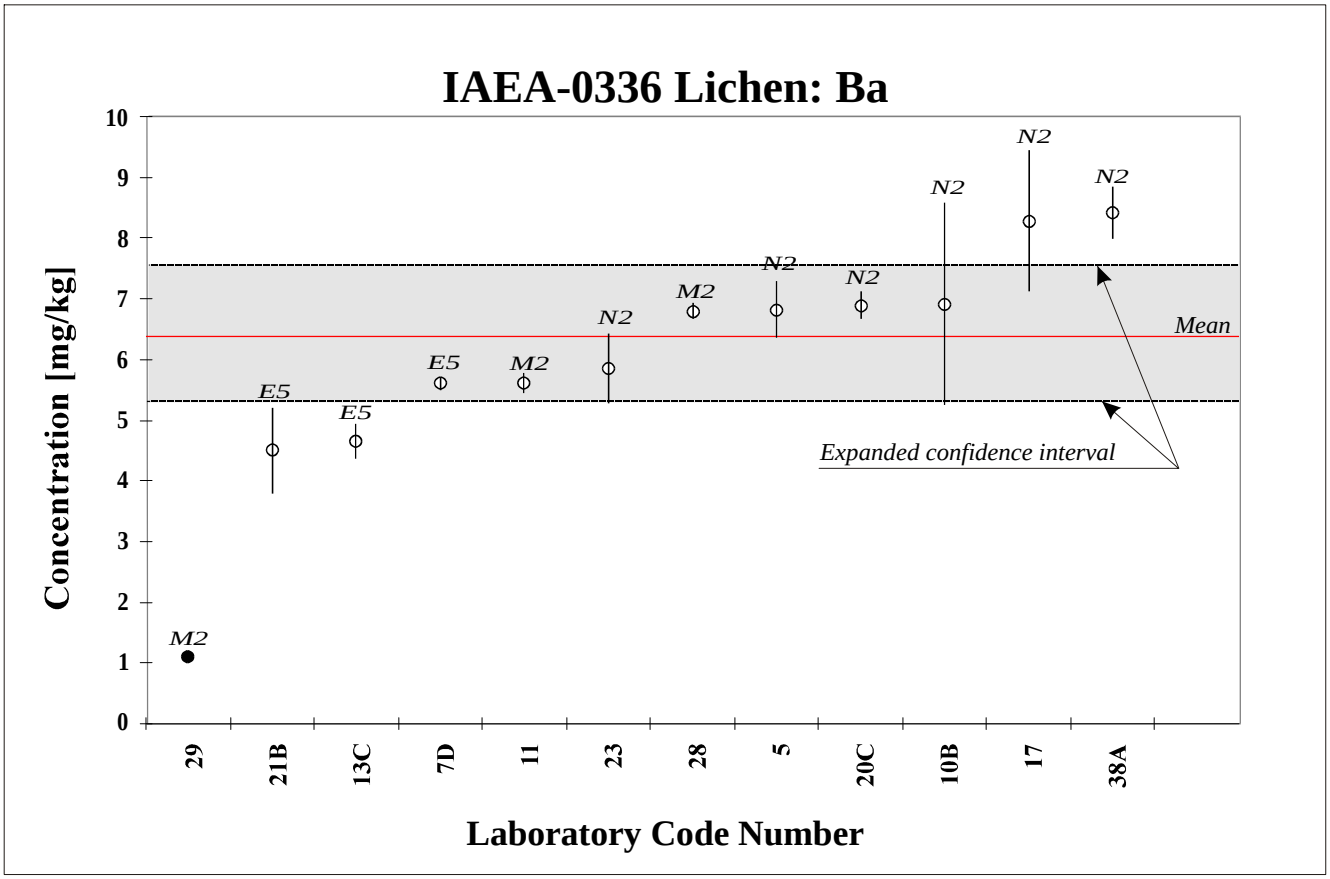
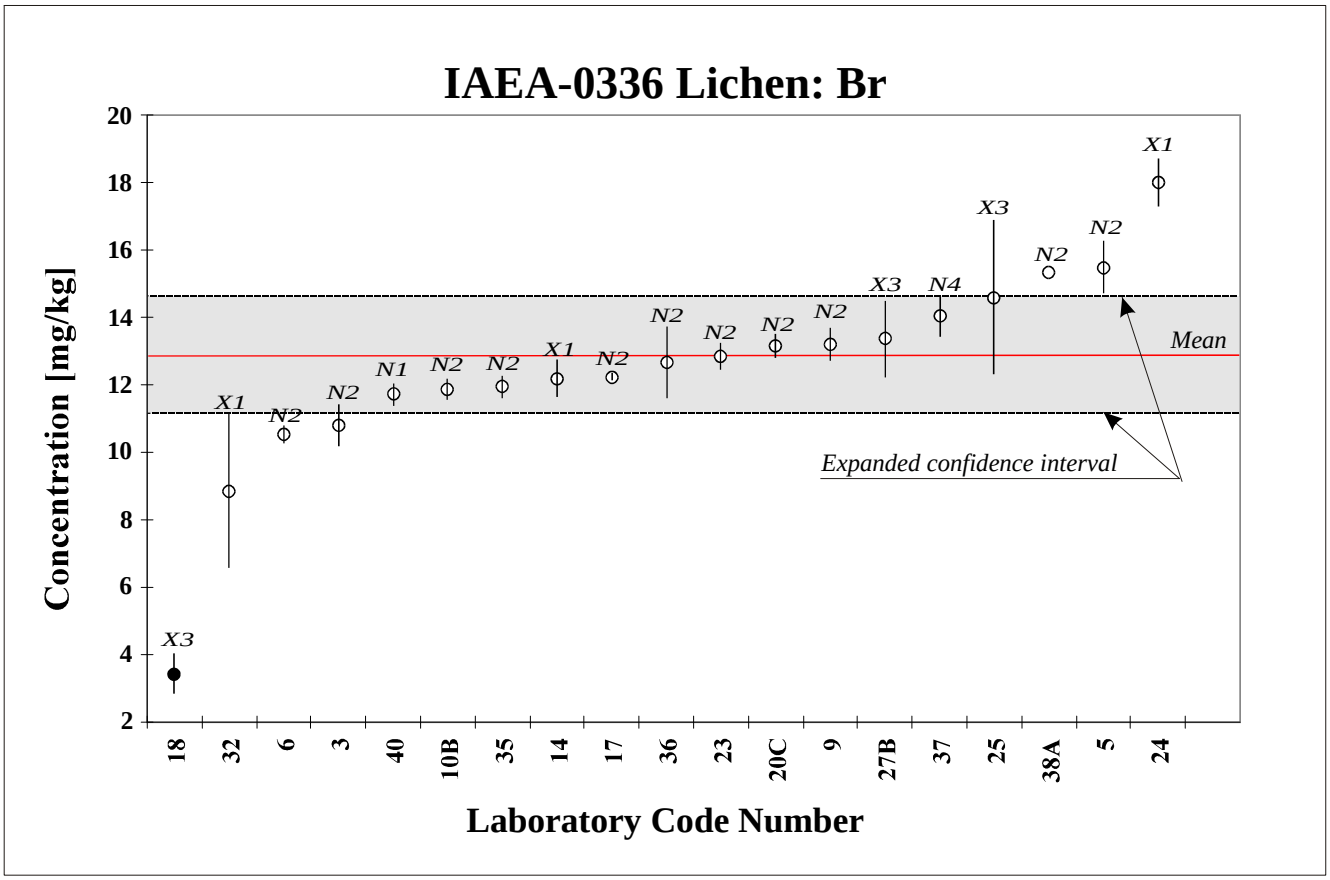
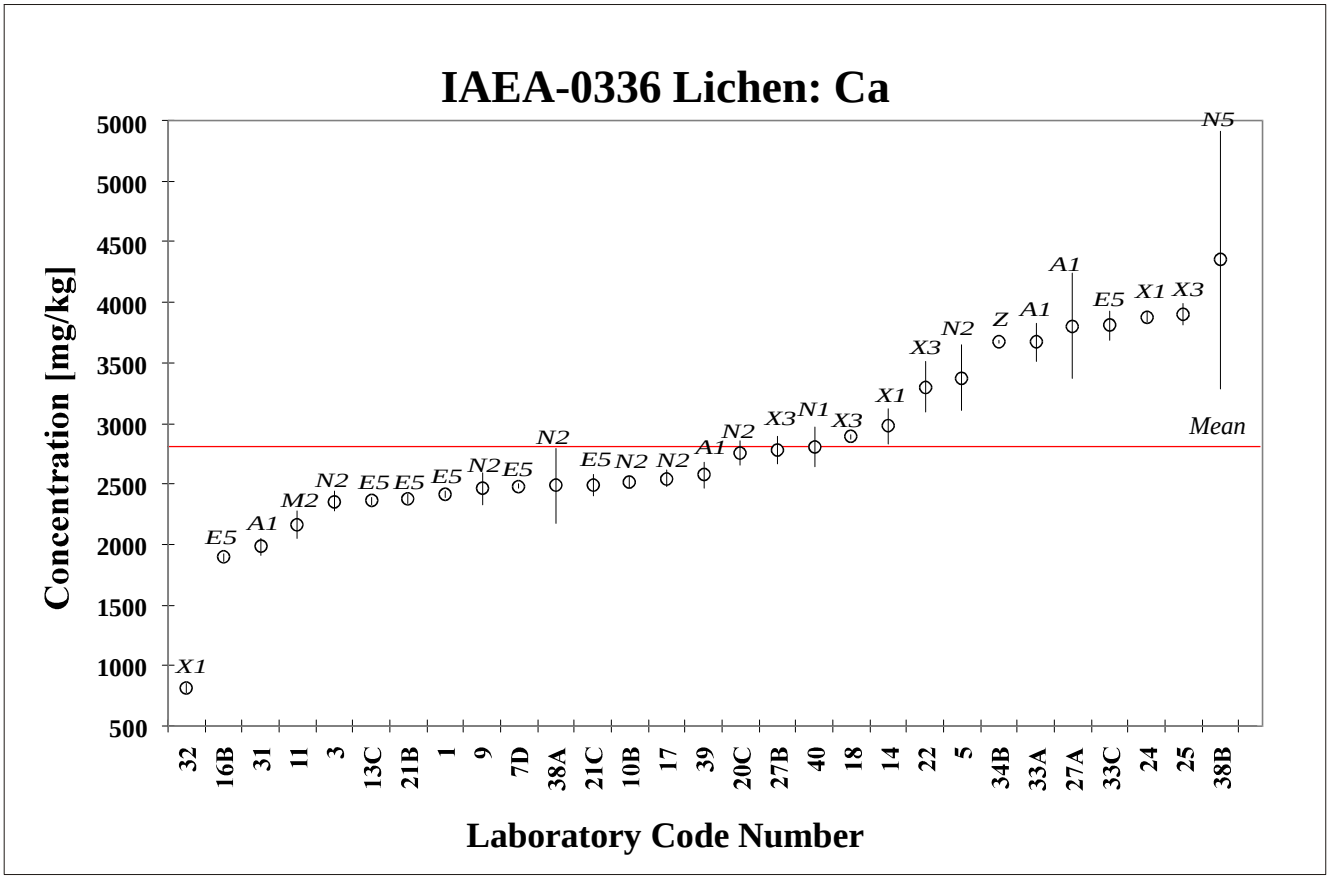
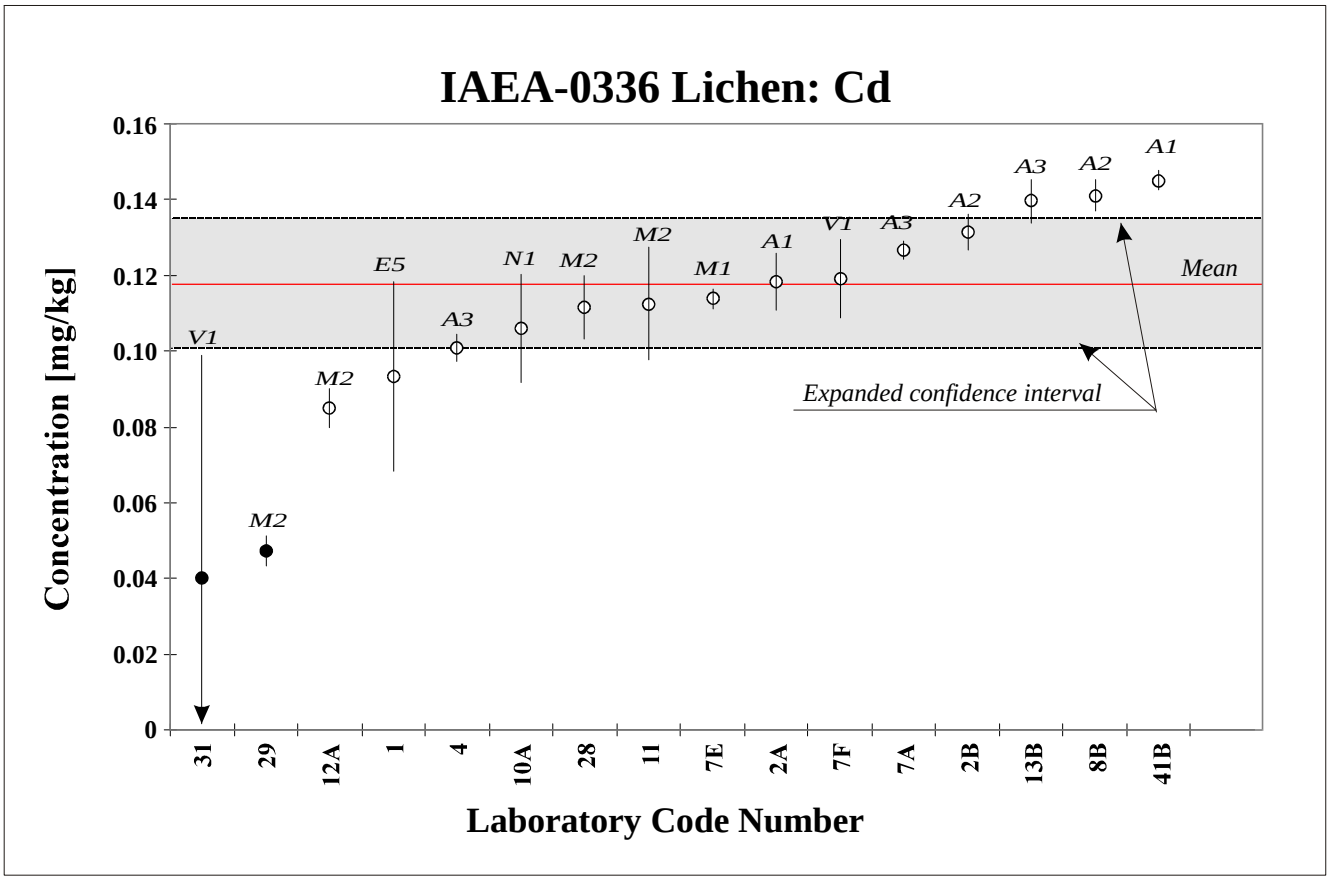
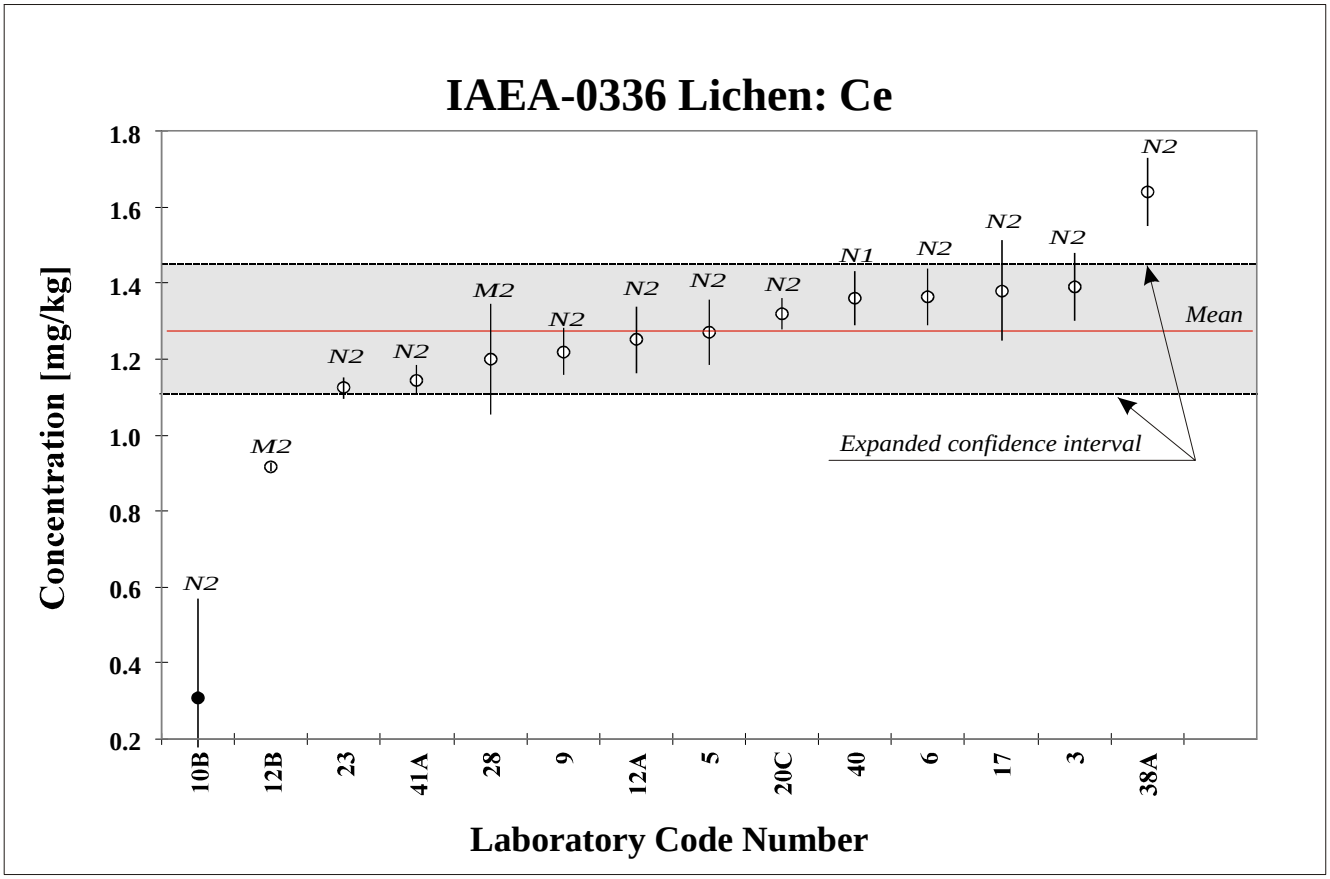
0.90
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
Mean
Expanded confidence interval
29 36 13B 6 35 11 5 40 23 10A 41A 12A 2B 28 38A 10B 7C 8D 17 20C
Laboratory Code Number

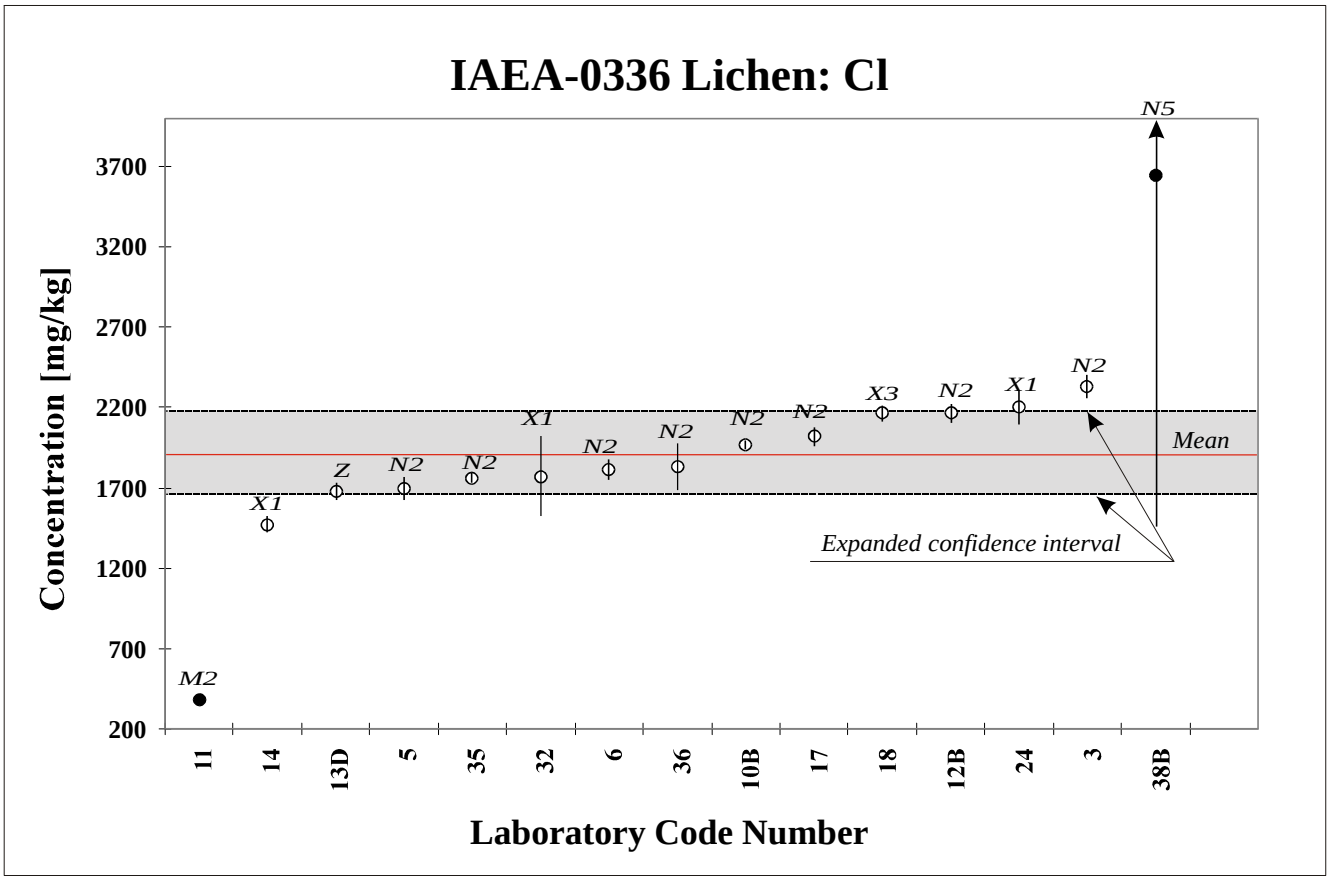

IAEA-0336 Lichen: Ba
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval
29
21B
13C
7D
11
23
28
5
20C
10B
17
38A

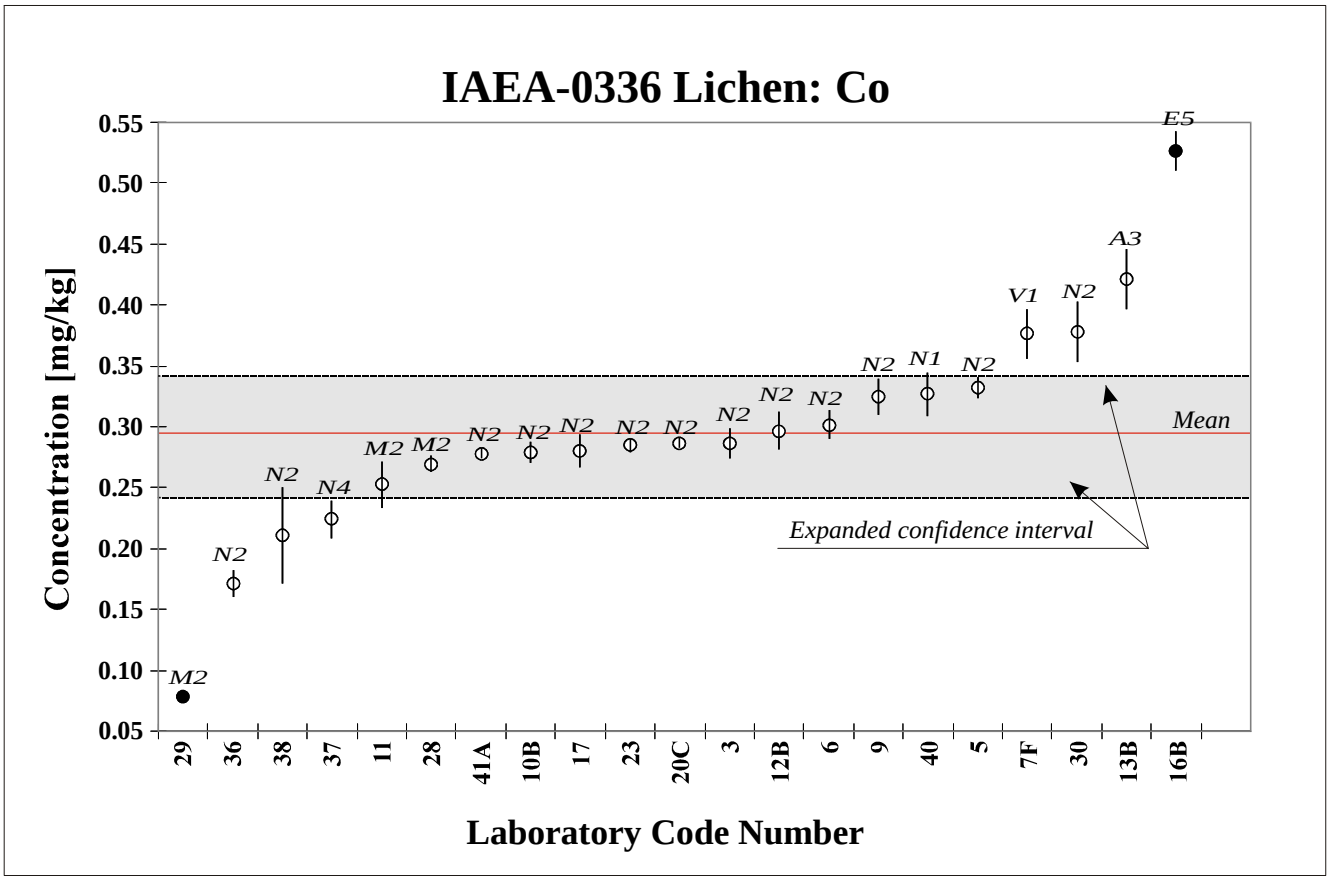

IAEA-0336 Lichen: Br
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval
18
32
6
3
40
10B
35
14
17
36
23
20C
9
27B
37
25
38A
5
24

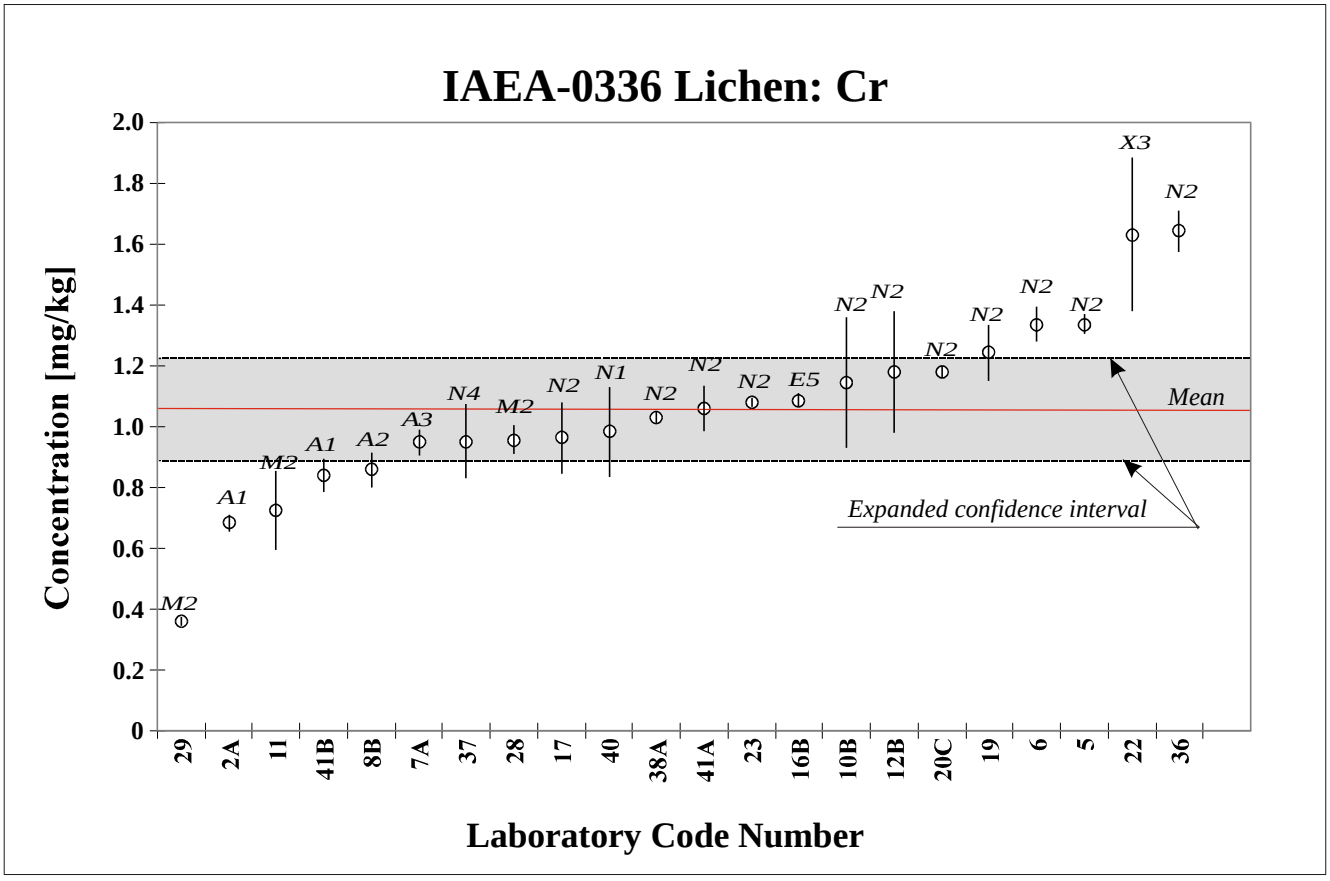

IAEA-0336 Lichen: Ca
Concentration [mg/kg]
5000
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
Mean
X1 E5 A1 M2 N2 E5 E5 E5 N2 E5 N2 E5 N2 N2 N2 A1 N2 X3 N1 X3 X1 X3 N2 Z A1 A1 E5 X1 X3 N5
32 16B 31 11 3 13C 21B 1 9 7D 38A 21C 10B 17 39 20C 27B 40 18 14 22 5 34B 33A 27A 33C 24 25 38B
Laboratory Code Number

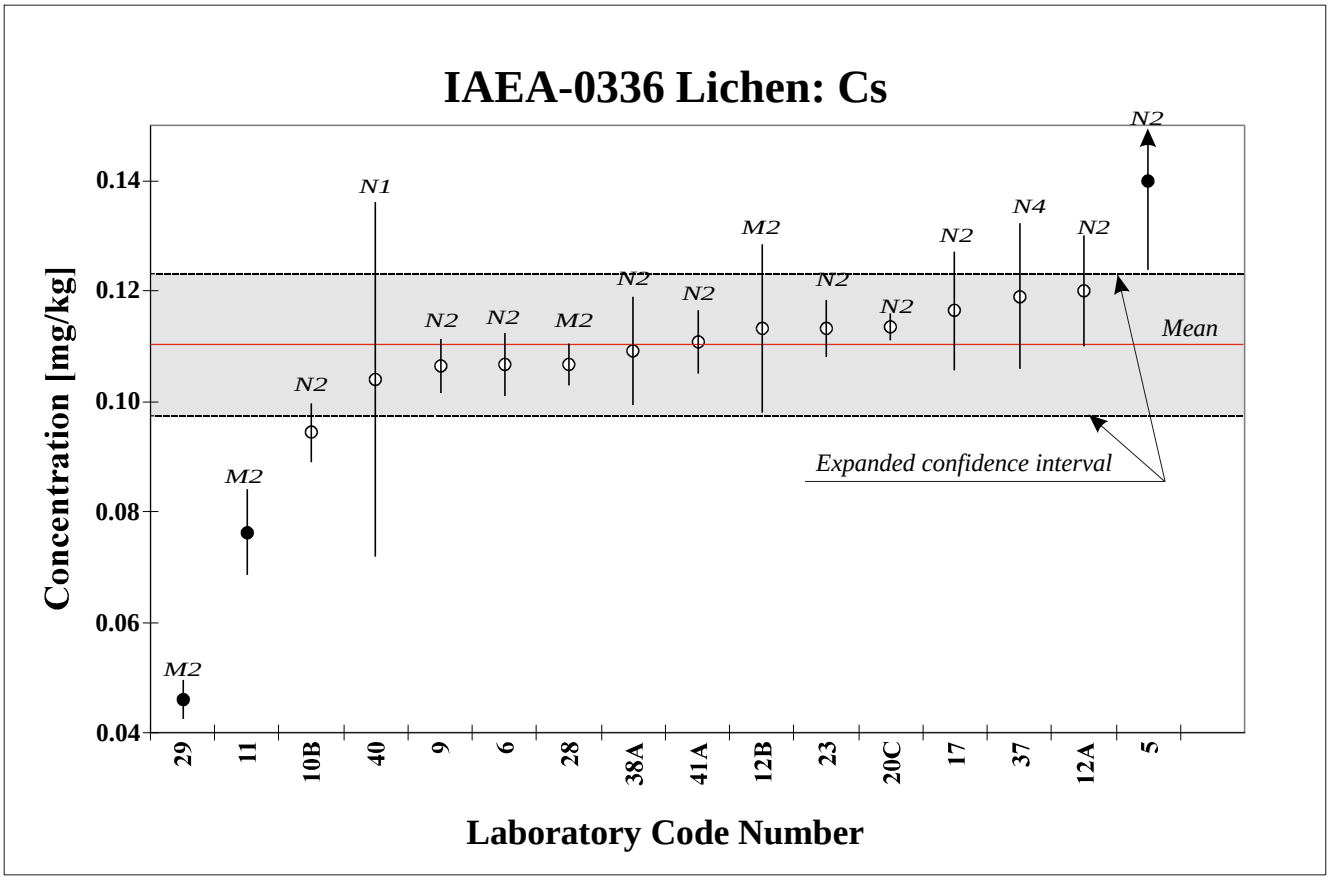

IAEA-0336 Lichen: Cd
Concentration [mg/kg]
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0
Mean
Expanded confidence interval
V1 M2 M2 E5 A3 N1 M2 M2 M1 A1 V1 A3 A2 A3 A2 A1
31 29 12A 1 4 10A 28 11 7E 2A 7F 7A 2B 13B 8B 41B
Laboratory Code Number

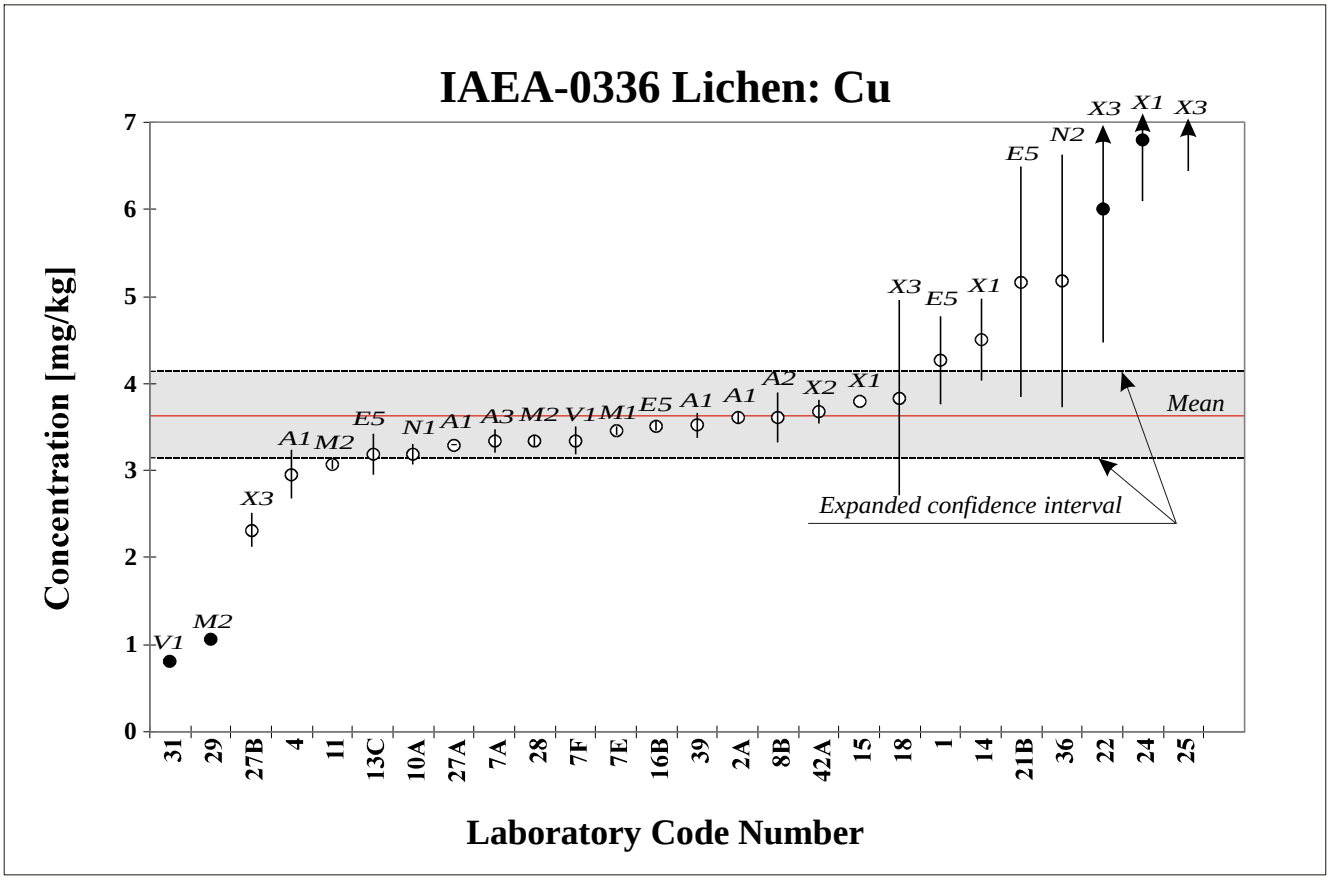

IAEA-0336 Lichen: Ce
Concentration [mg/kg]
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
N2
M2
N2
N2
M2
N2
N2
N2
N2
N1
N2
N2
N2
Mean
Expanded confidence interval
10B
12B
23
41A
28
9
12A
5
20C
40
6
17
3
38A
Laboratory Code Number

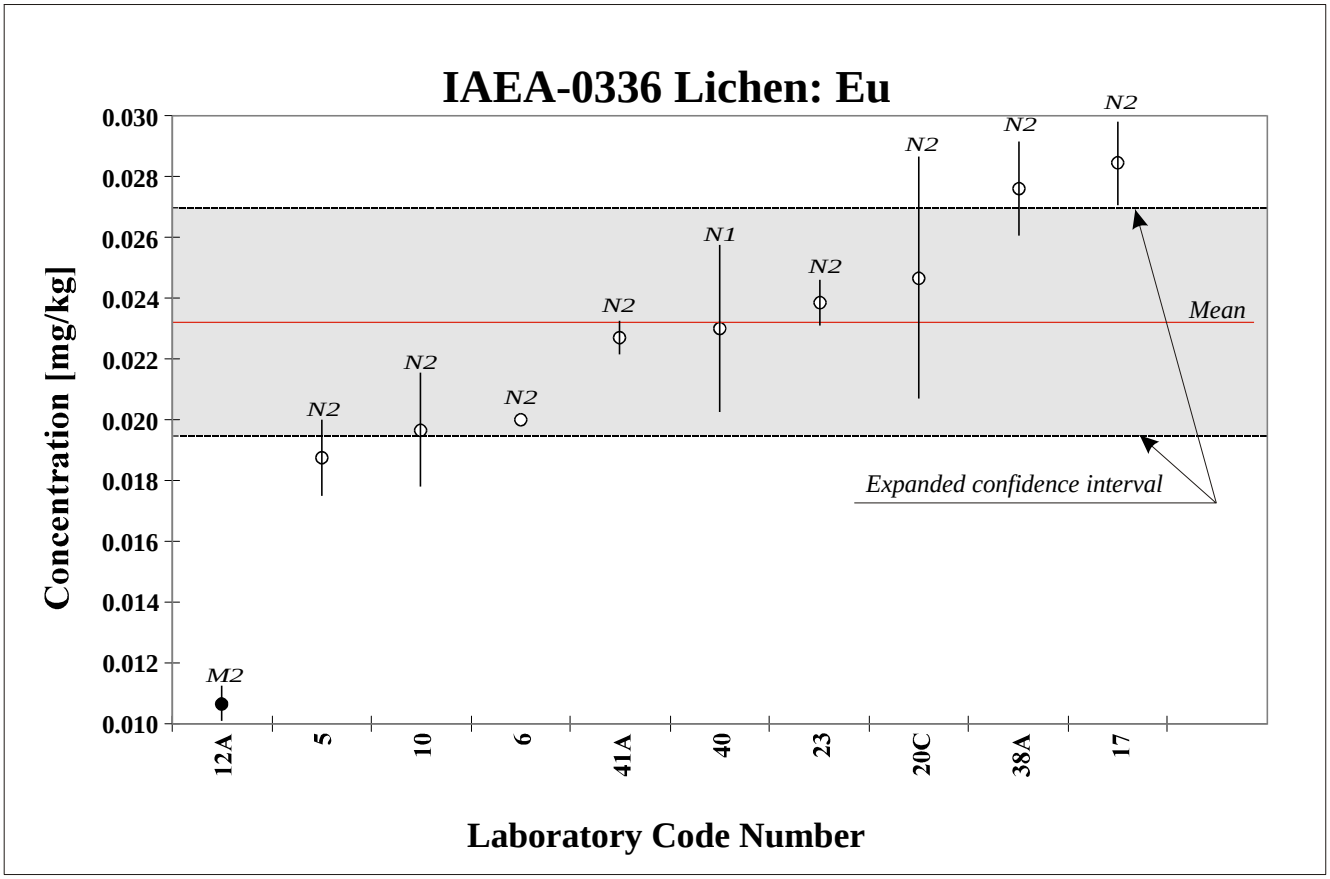

IAEA-0336 Lichen: Cl
Concentration [mg/kg]
3700
3200
2700
2200
1700
1200
700
200
N5
M2
X1
Z
N2
N2
X1
N2
N2
N2
N2
X3
N2
X1
N2
Mean
Expanded confidence interval
11
14
13D
5
35
32
6
36
10B
17
18
12B
24
3
38B
Laboratory Code Number

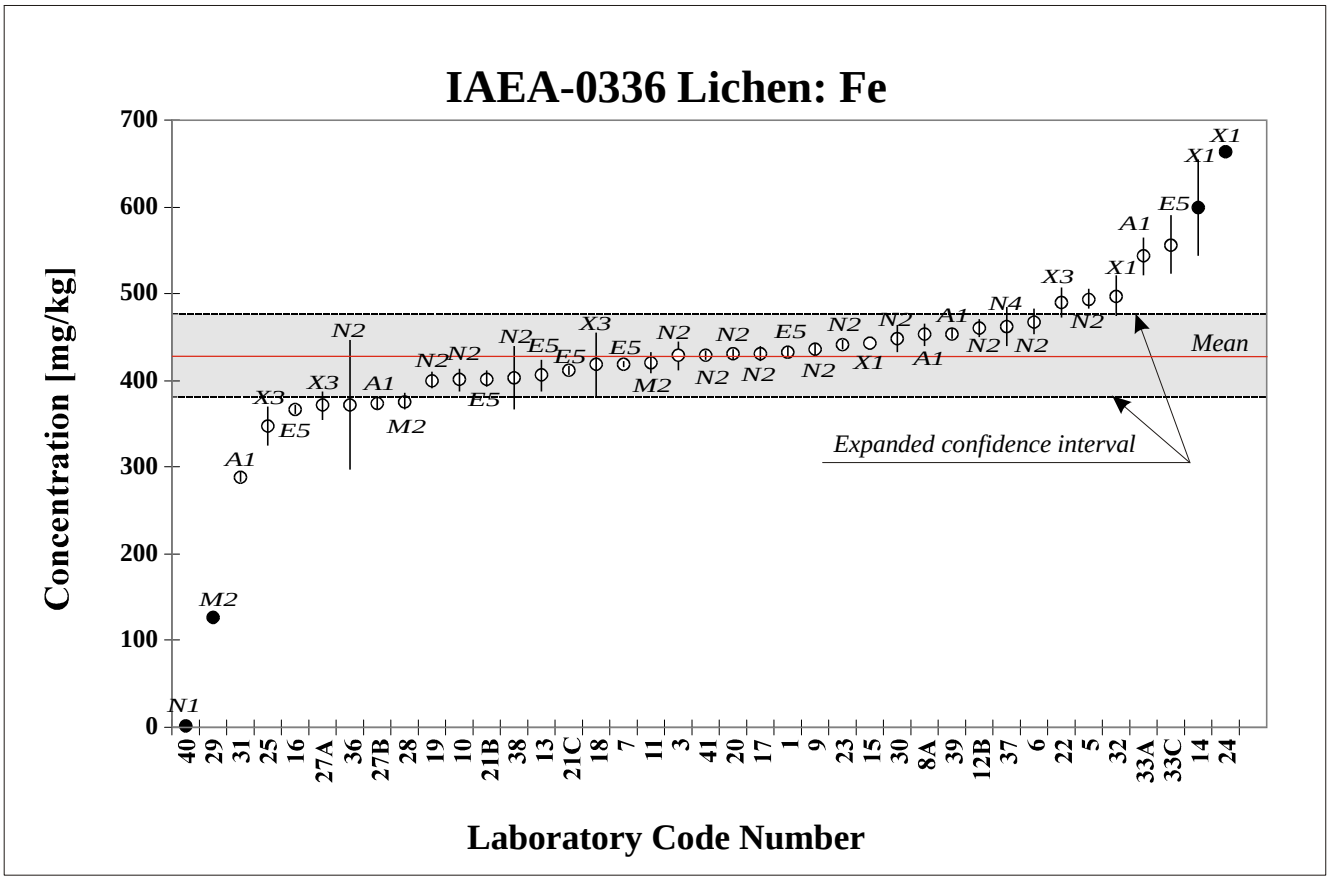

IAEA-0336 Lichen: Co
Concentration [mg/kg]
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
Mean
Expanded confidence interval
29 36 38 37 11 28 41A 10B 17 23 20C 3 12B 6 9 40 5 7F 30 13B 16B
Laboratory Code Number

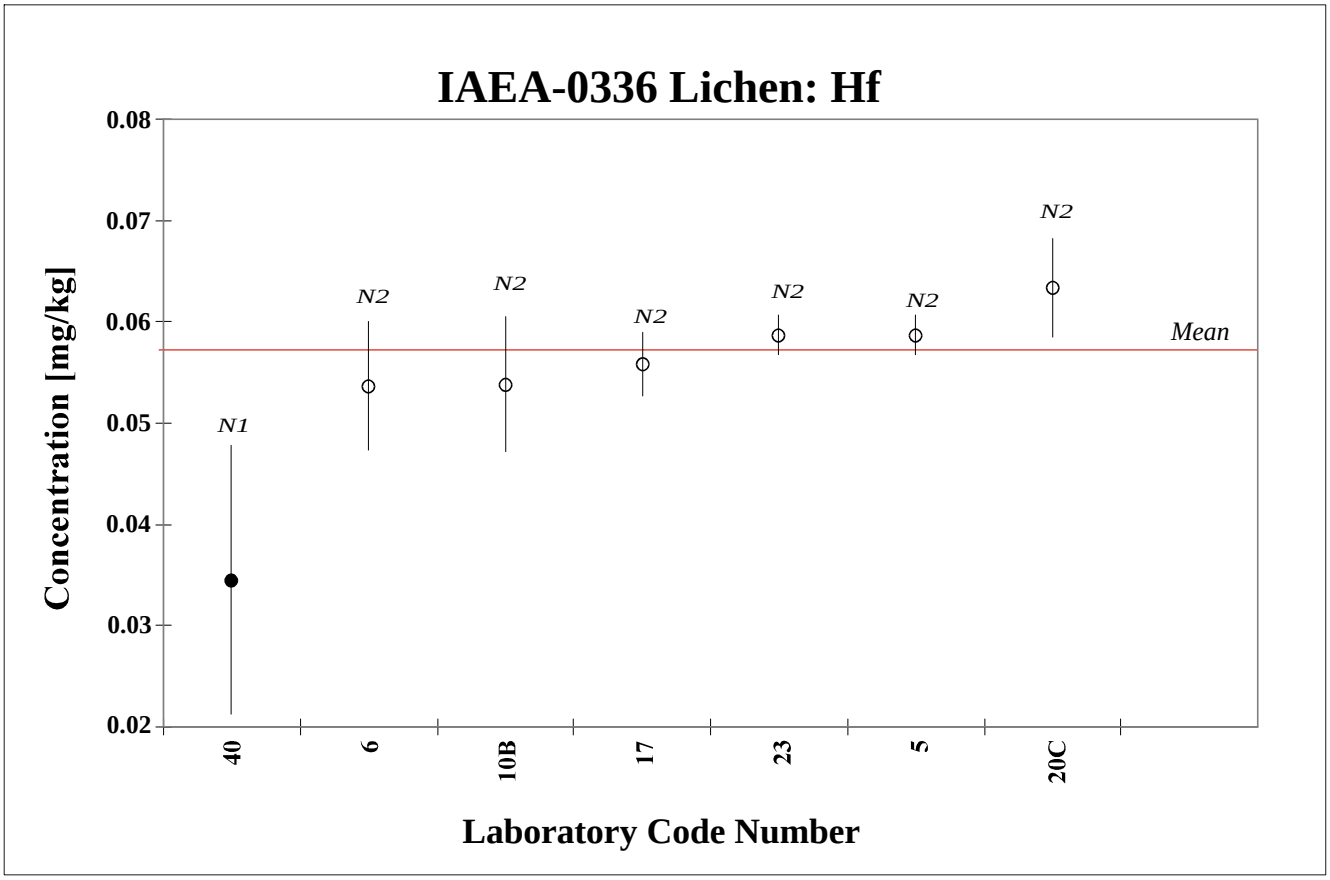

IAEA-0336 Lichen: Cr
Concentration [mg/kg]
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0
Mean
Expanded confidence interval
29 2A 11 41B 8B 7A 37 28 17 40 38A 41A 23 16B 10B 12B 20C 19 6 5 22 36
Laboratory Code Number

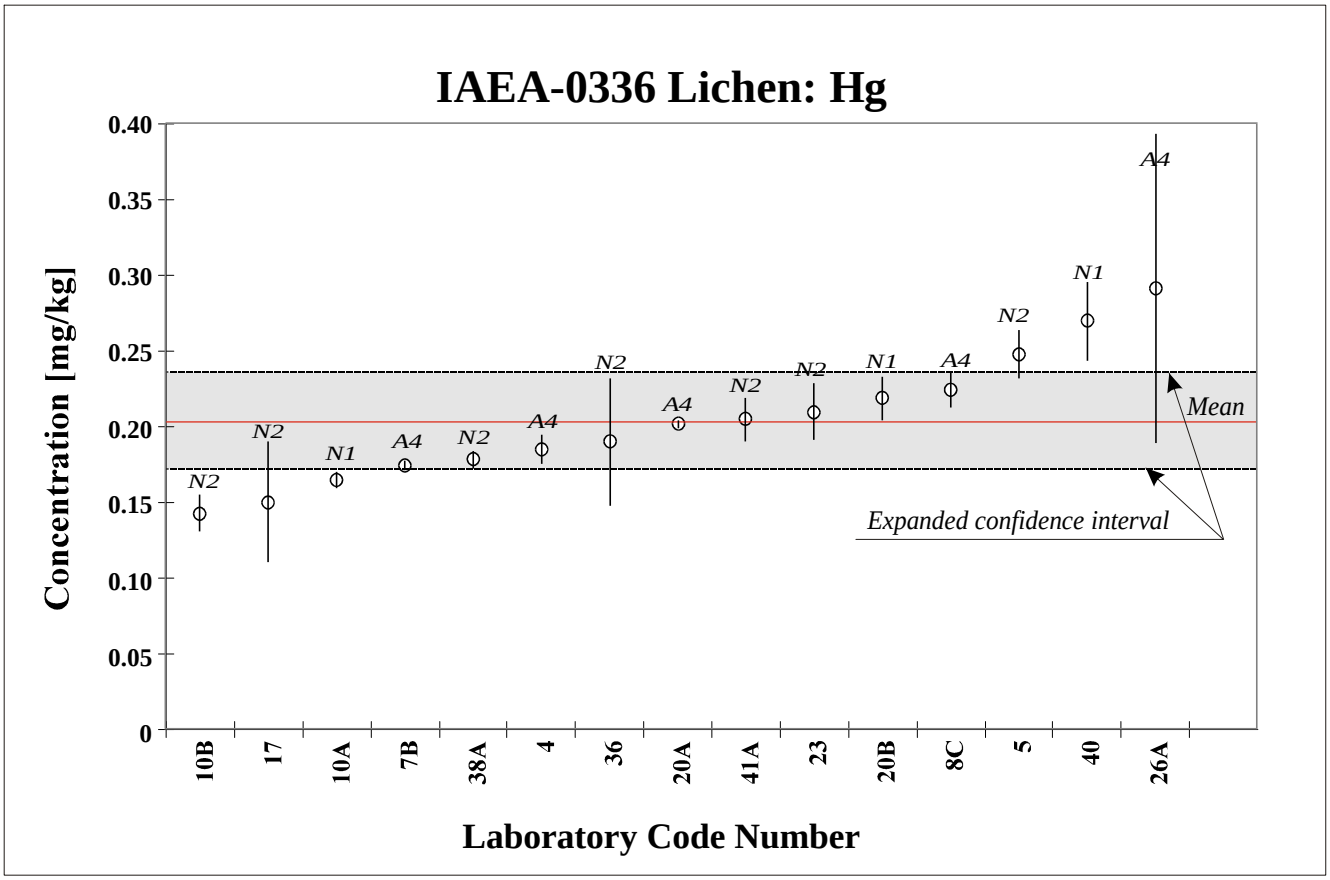

IAEA-0336 Lichen: Cs
Concentration [mg/kg]
0.14
0.12
0.10
0.08
0.06
0.04
Mean
Expanded confidence interval
29 11 10B 40 9 6 28 38A 41A 12B 23 20C 17 37 12A 5
Laboratory Code Number

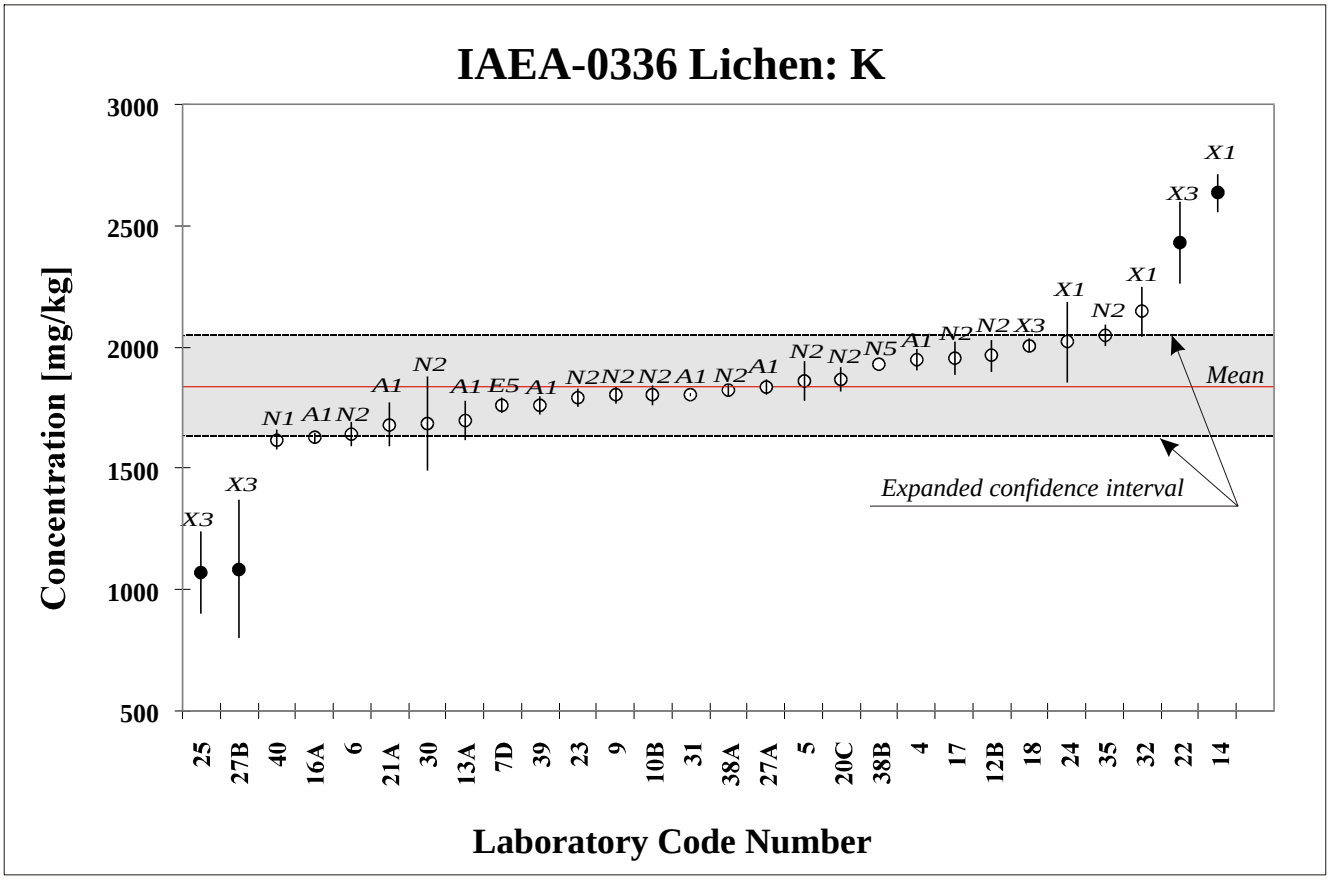

IAEA-0336 Lichen: Cu
Concentration [mg/kg]
7
6
5
4
3
2
1
0
Mean
Expanded confidence interval
31 29 27B 4 11 13C 10A 27A 7A 28 7F 7E 16B 39 2A 8B 42A 15 18 1 14 21B 36 22 24 25
Laboratory Code Number

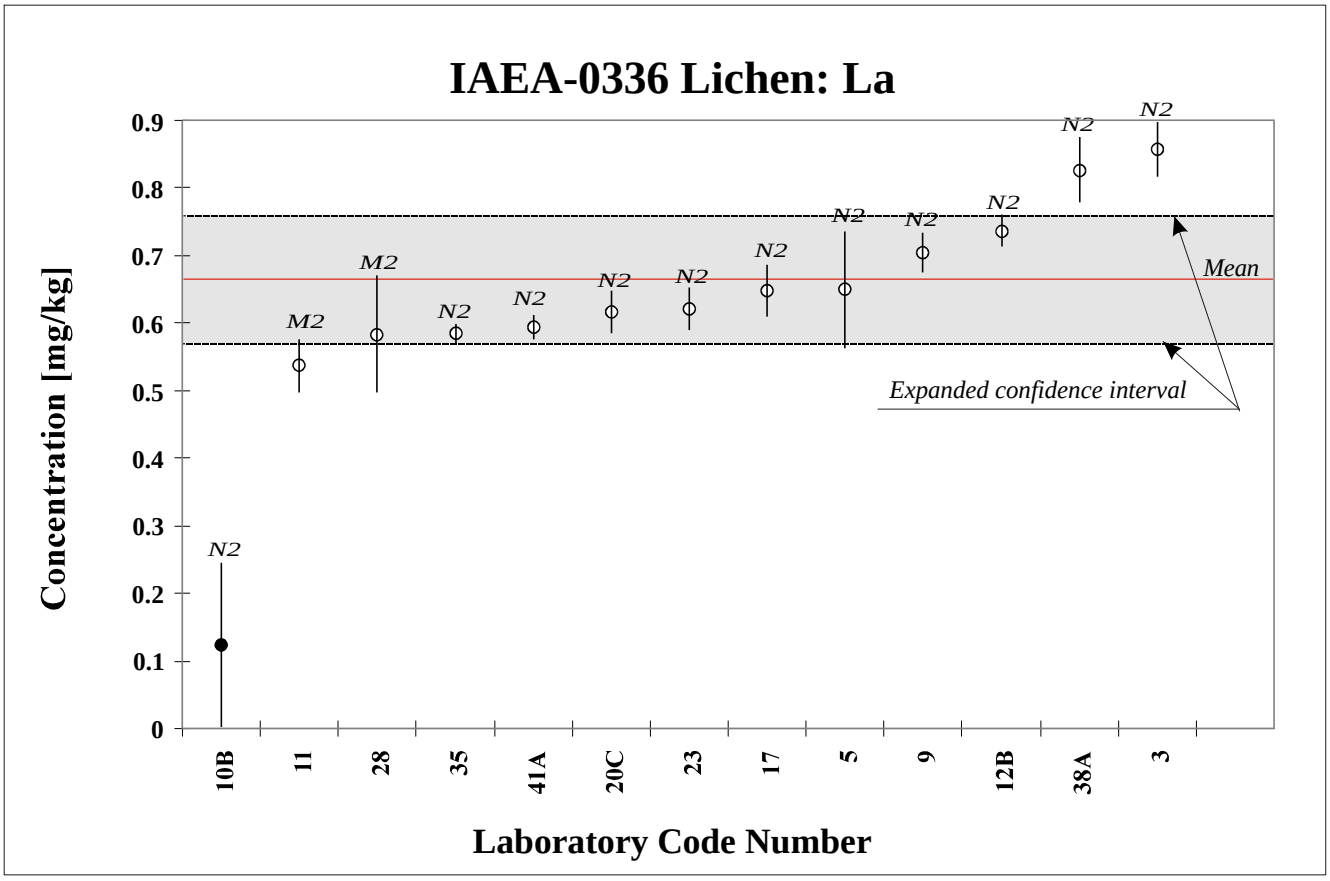

IAEA-0336 Lichen: Eu
Concentration [mg/kg]
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
Mean
Expanded confidence interval
M2
N1
N2
12A
5
10
6
41A
40
23
20C
38A
17
Laboratory Code Number

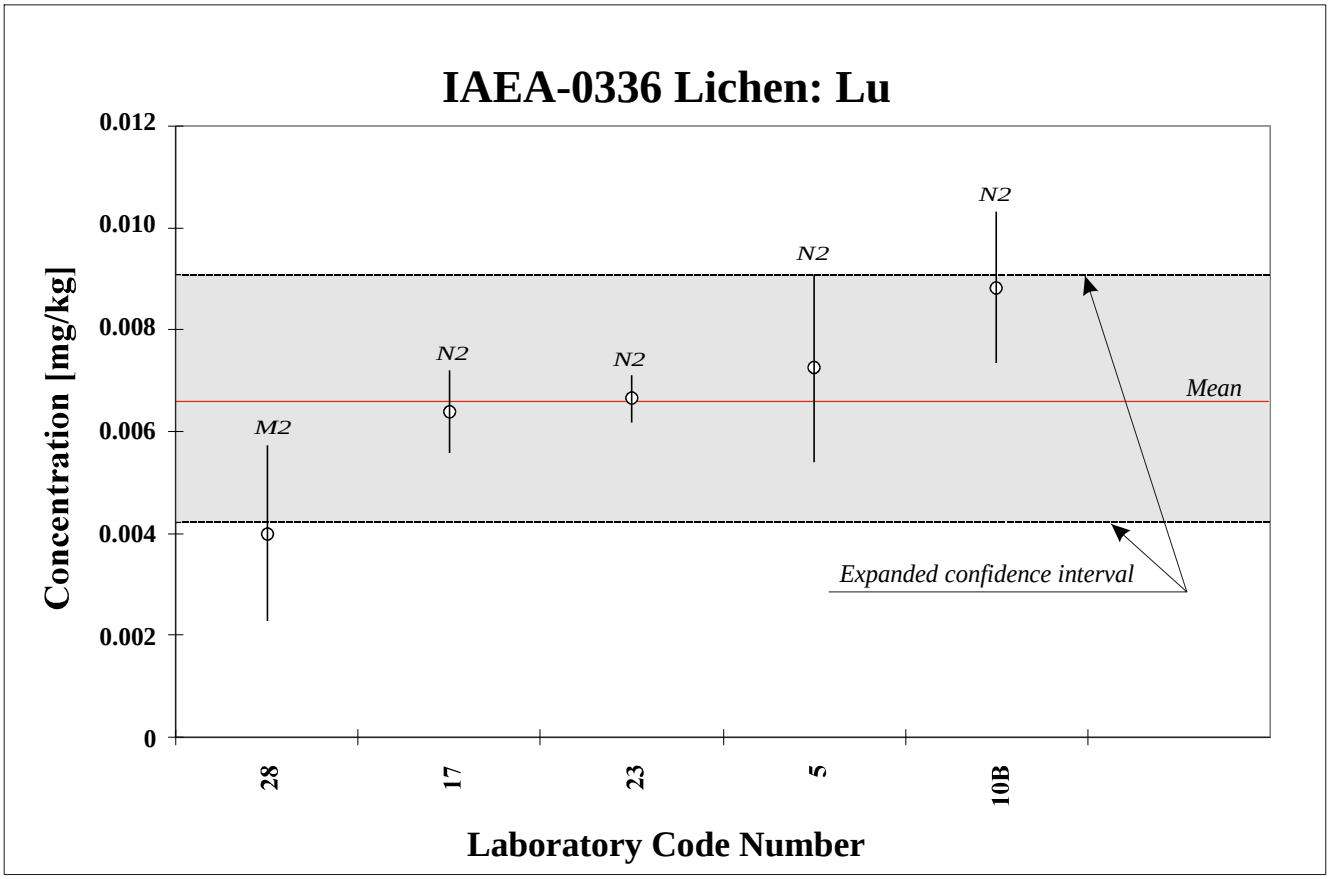

IAEA-0336 Lichen: Fe
Concentration [mg/kg]
700
600
500
400
300
200
100
0
Mean
Expanded confidence interval
N1
M2
A1
E5
X3
N2
X1
N4
40
29
31
25
16
27A
36
27B
28
19
10
21B
38
13
21C
18
7
11
3
41
20
17
1
9
23
15
30
8A
39
12B
37
6
22
5
32
33A
33C
14
24
Laboratory Code Number

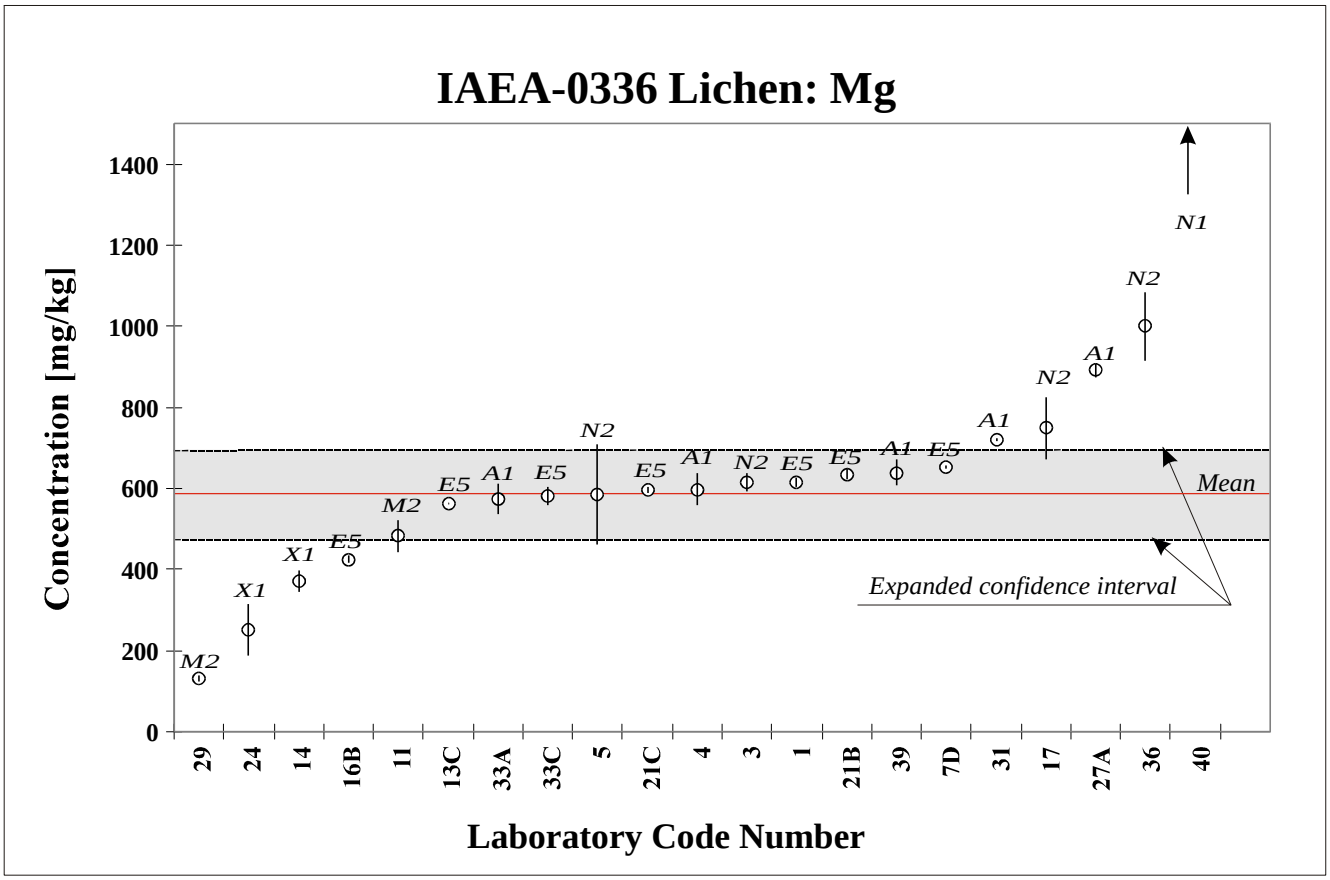

IAEA-0336 Lichen: Hf
Concentration [mg/kg]
0.08
0.07
0.06
0.05
0.04
0.03
0.02
N1
N2
N2
N2
N2
N2
N2
Mean
40
6
10B
17
23
5
20C
Laboratory Code Number

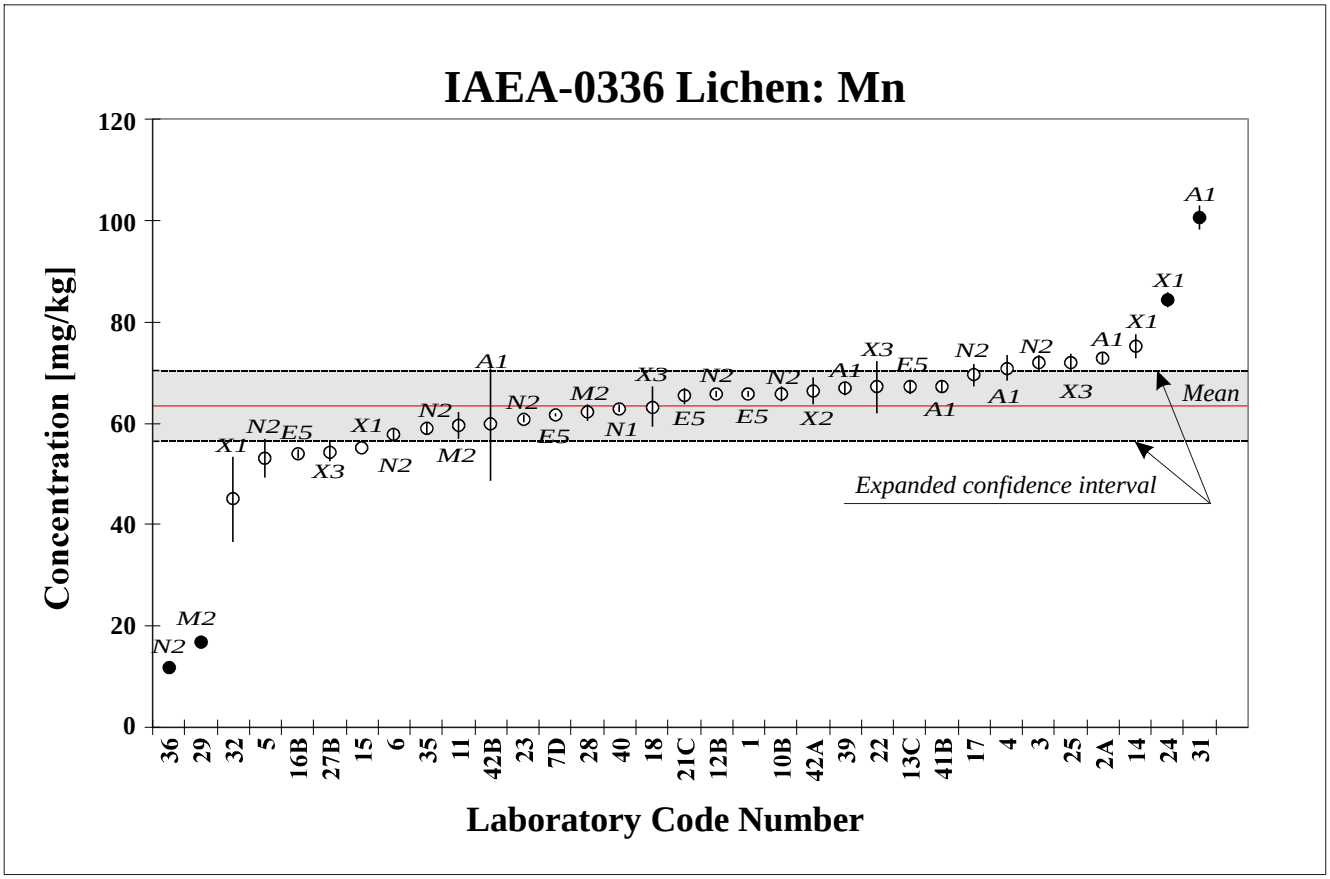

IAEA-0336 Lichen: Hg
Concentration [mg/kg]
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
N2
N2
N1
A4
N2
A4
N2
A4
N2
N2
N1
A4
N2
N1
A4
Mean
Expanded confidence interval
10B
17
10A
7B
38A
4
36
20A
41A
23
20B
8C
5
40
26A
Laboratory Code Number

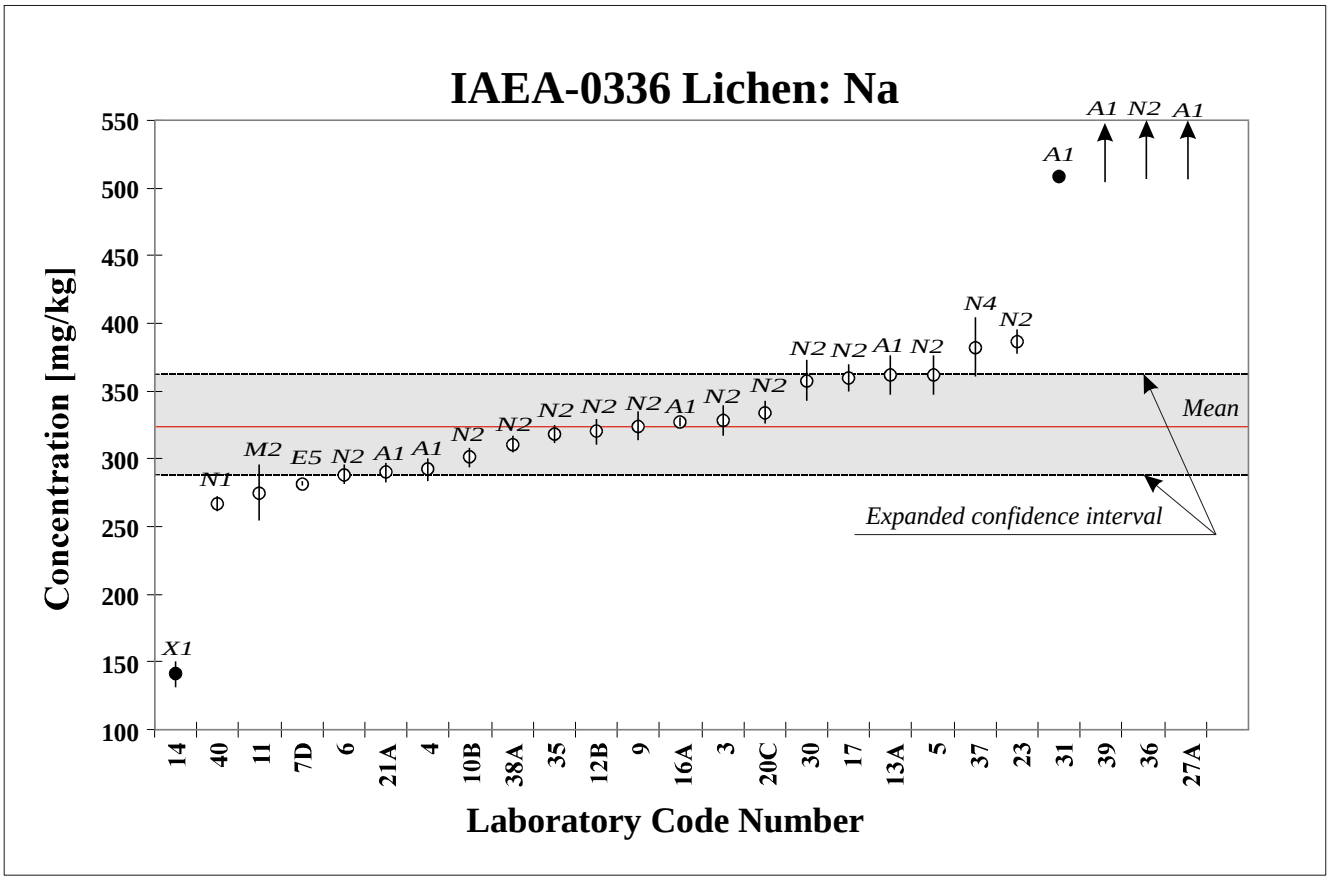

IAEA-0336 Lichen: K
Concentration [mg/kg]
3000
2500
2000
1500
1000
500
Mean
Expanded confidence interval
25 27B 40 16A 6 21A 30 13A 7D 39 23 9 10B 31 38A 27A 5 20C 38B 4 17 12B 18 24 35 32 22 14
Laboratory Code Number

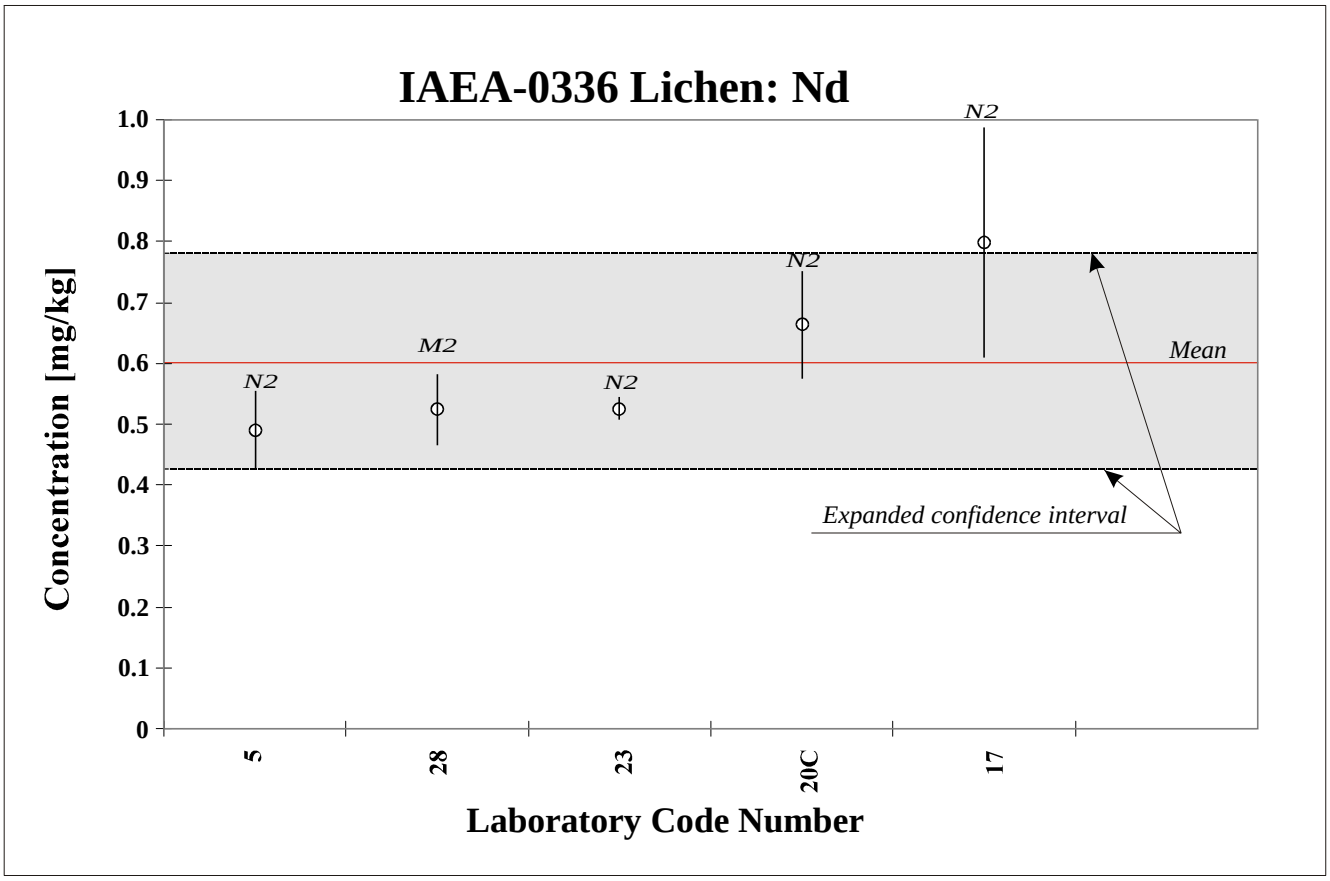

IAEA-0336 Lichen: La
Concentration [mg/kg]
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Mean
Expanded confidence interval
10B 11 28 35 41A 20C 23 17 5 9 12B 38A 3
Laboratory Code Number

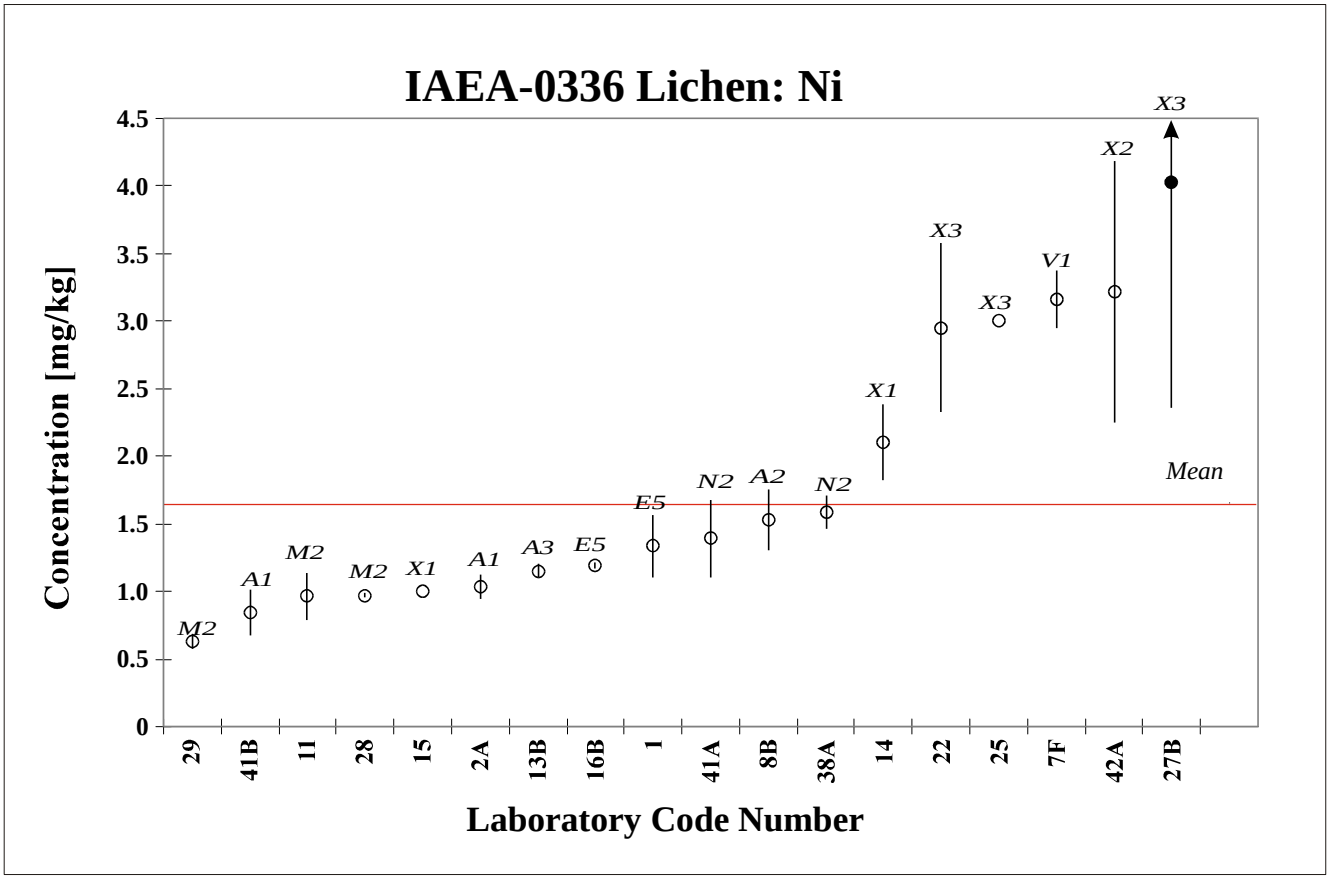

IAEA-0336 Lichen: Lu
Concentration [mg/kg]
0.012
0.010
0.008
0.006
0.004
0.002
0
M2
N2
N2
N2
N2
Mean
Expanded confidence interval
28
17
23
5
10B
Laboratory Code Number

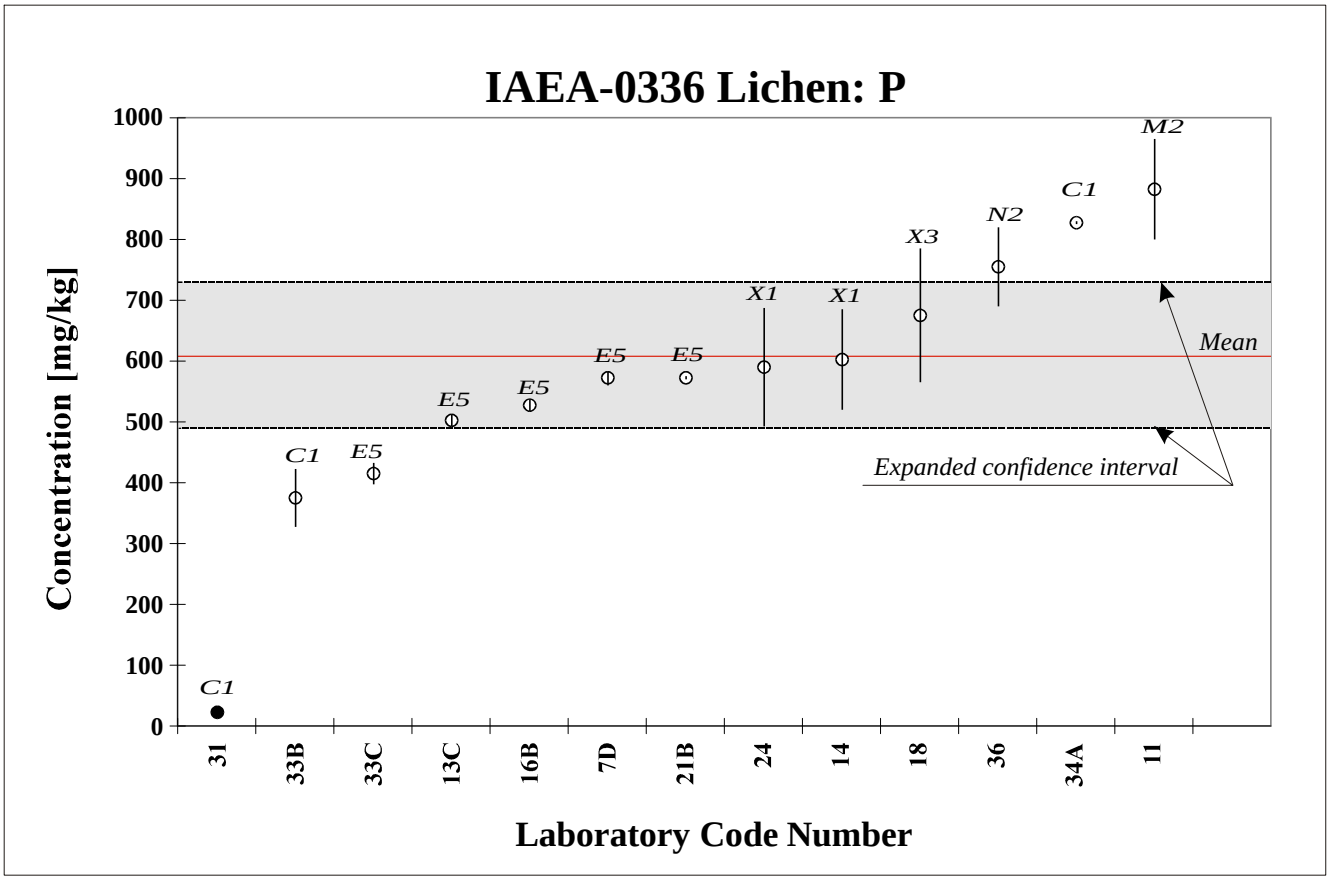

IAEA-0336 Lichen: Mg
Concentration [mg/kg]
1400
1200
1000
800
600
400
200
0
M2
X1
X1
E5
M2
E5
A1
E5
N2
E5
A1
N2
E5
E5
A1
E5
A1
N2
A1
N2
N1
Mean
Expanded confidence interval
29
24
14
16B
11
13C
33A
33C
5
21C
4
3
1
21B
39
7D
31
17
27A
36
40
Laboratory Code Number

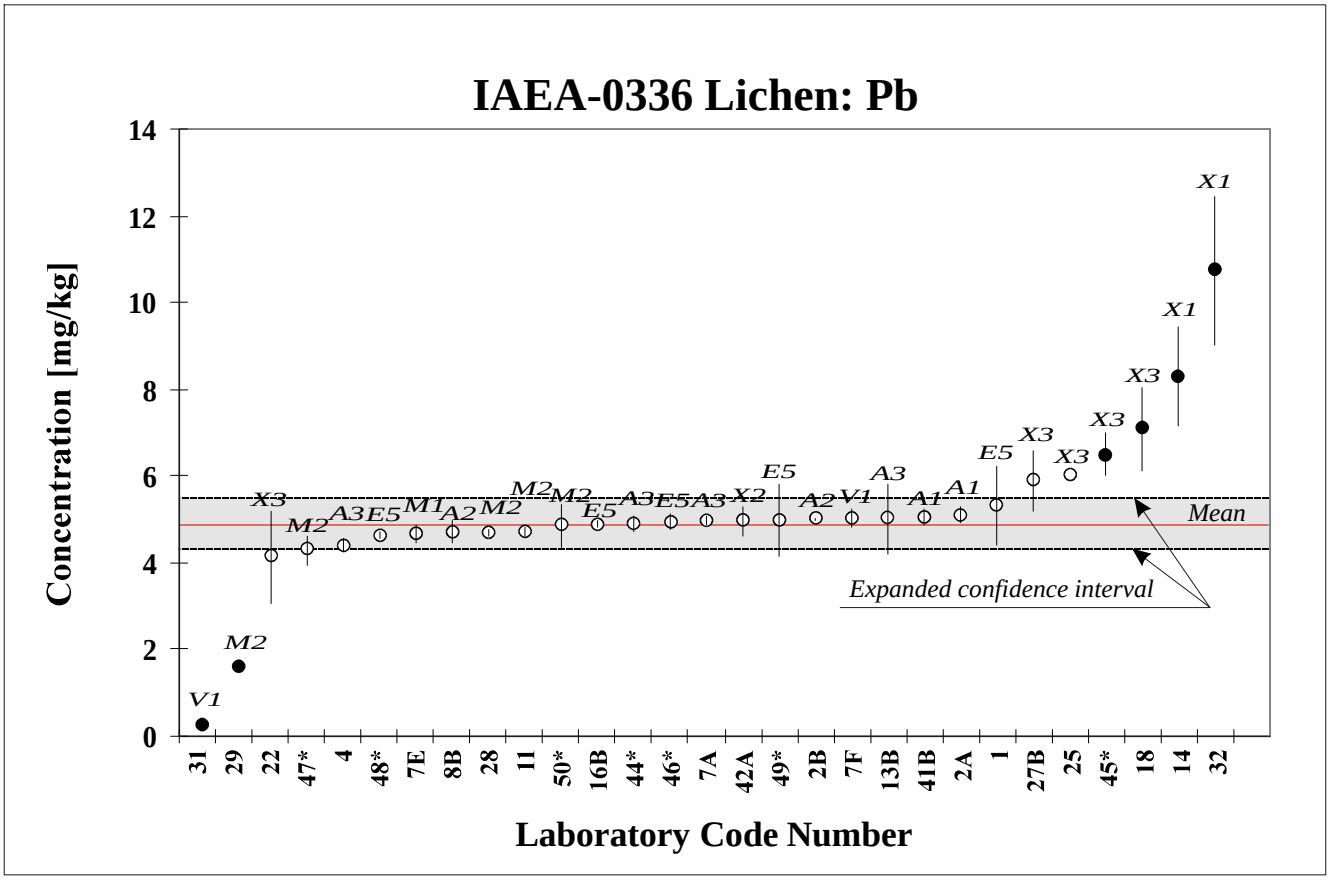

IAEA-0336 Lichen: Mn
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval

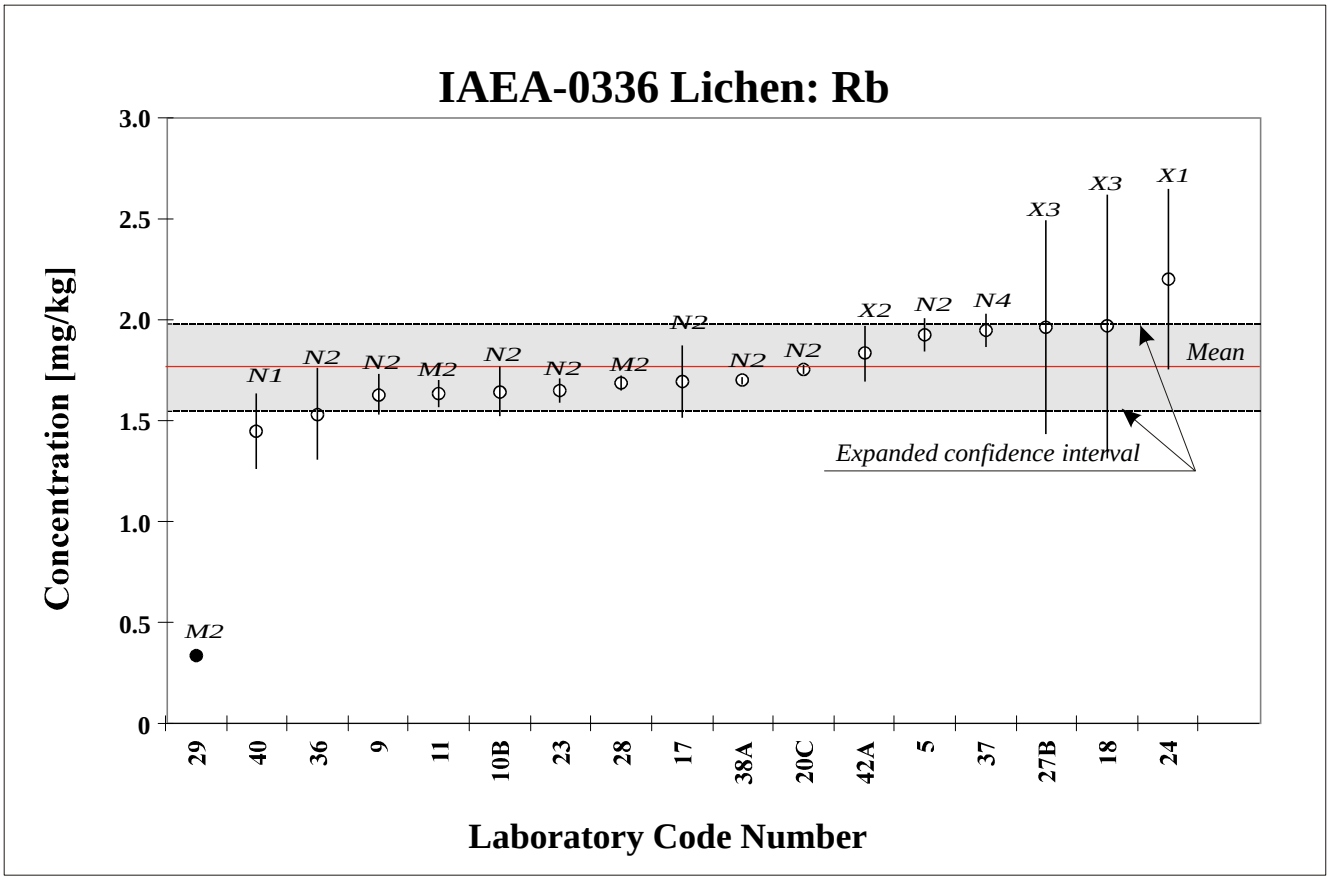

IAEA-0336 Lichen: Na
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval

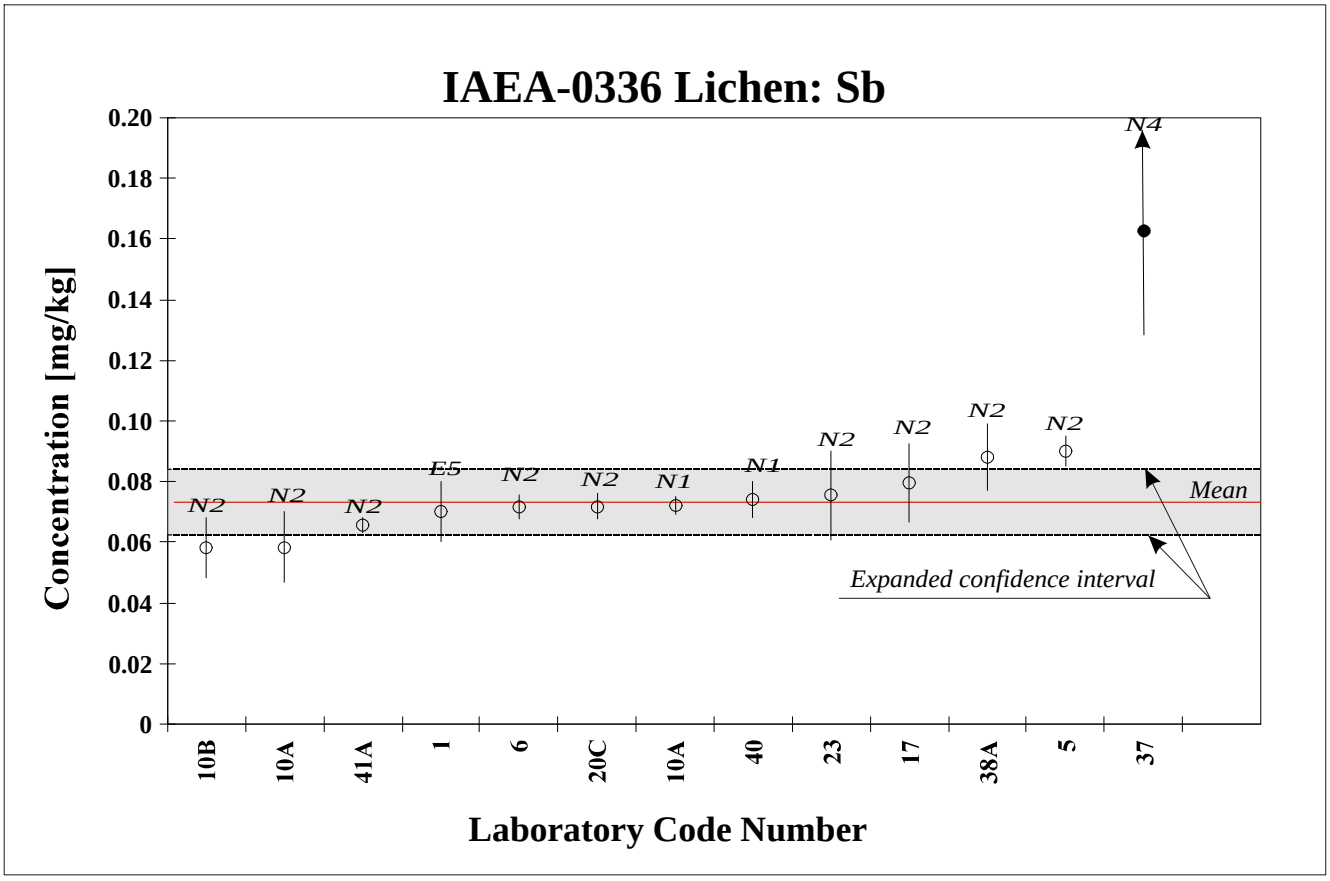

IAEA-0336 Lichen: Nd
Concentration [mg/kg]
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
N2
M2
N2
N2
N2
Mean
Expanded confidence interval
5
28
23
20C
17
Laboratory Code Number

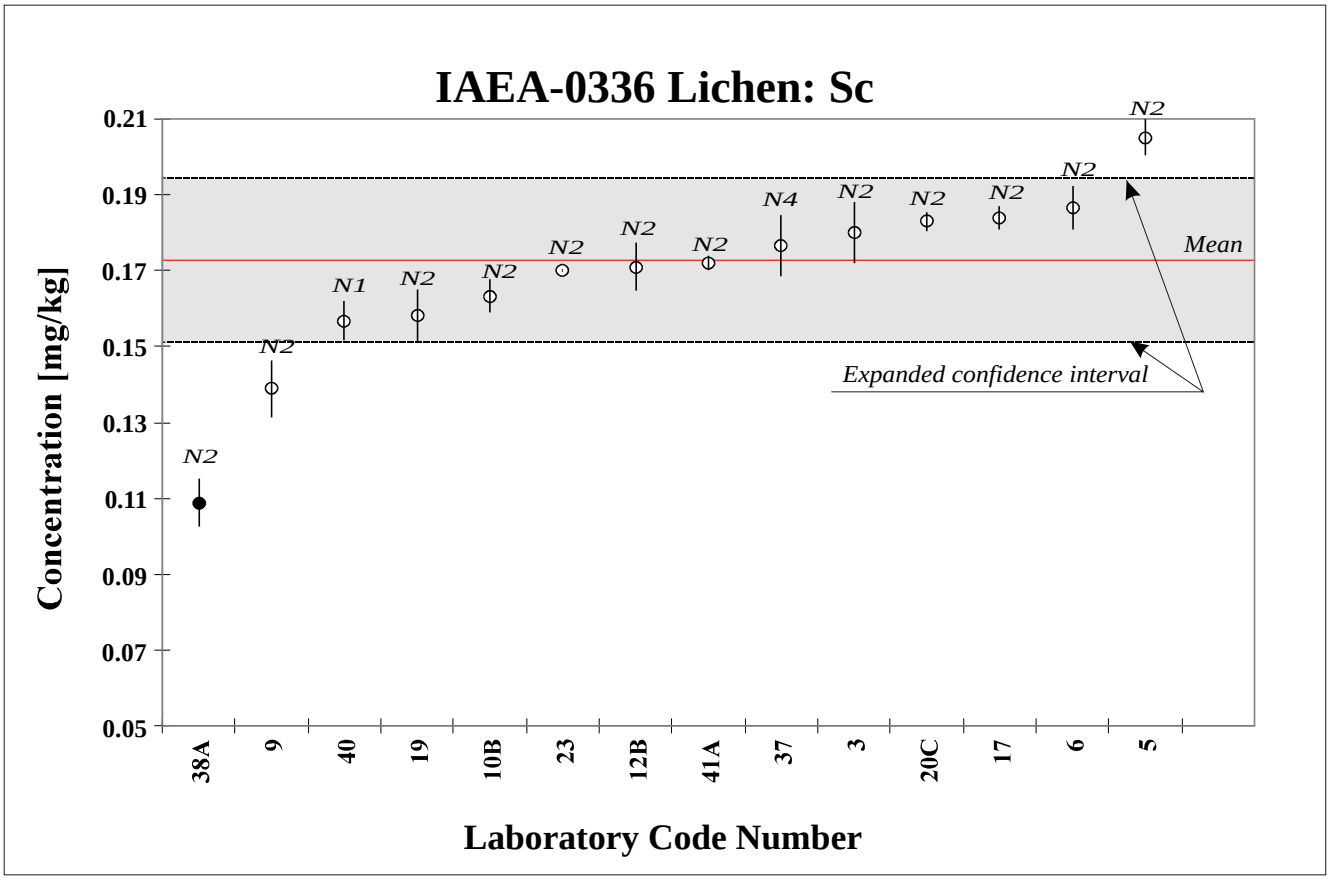

IAEA-0336 Lichen: Ni
Concentration [mg/kg]
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
M2
A1
M2
M2
X1
A1
A3
E5
E5
N2
A2
N2
X1
X3
X3
V1
X2
X3
Mean
29
41B
11
28
15
2A
13B
16B
1
41A
8B
38A
14
22
25
7F
42A
27B
Laboratory Code Number

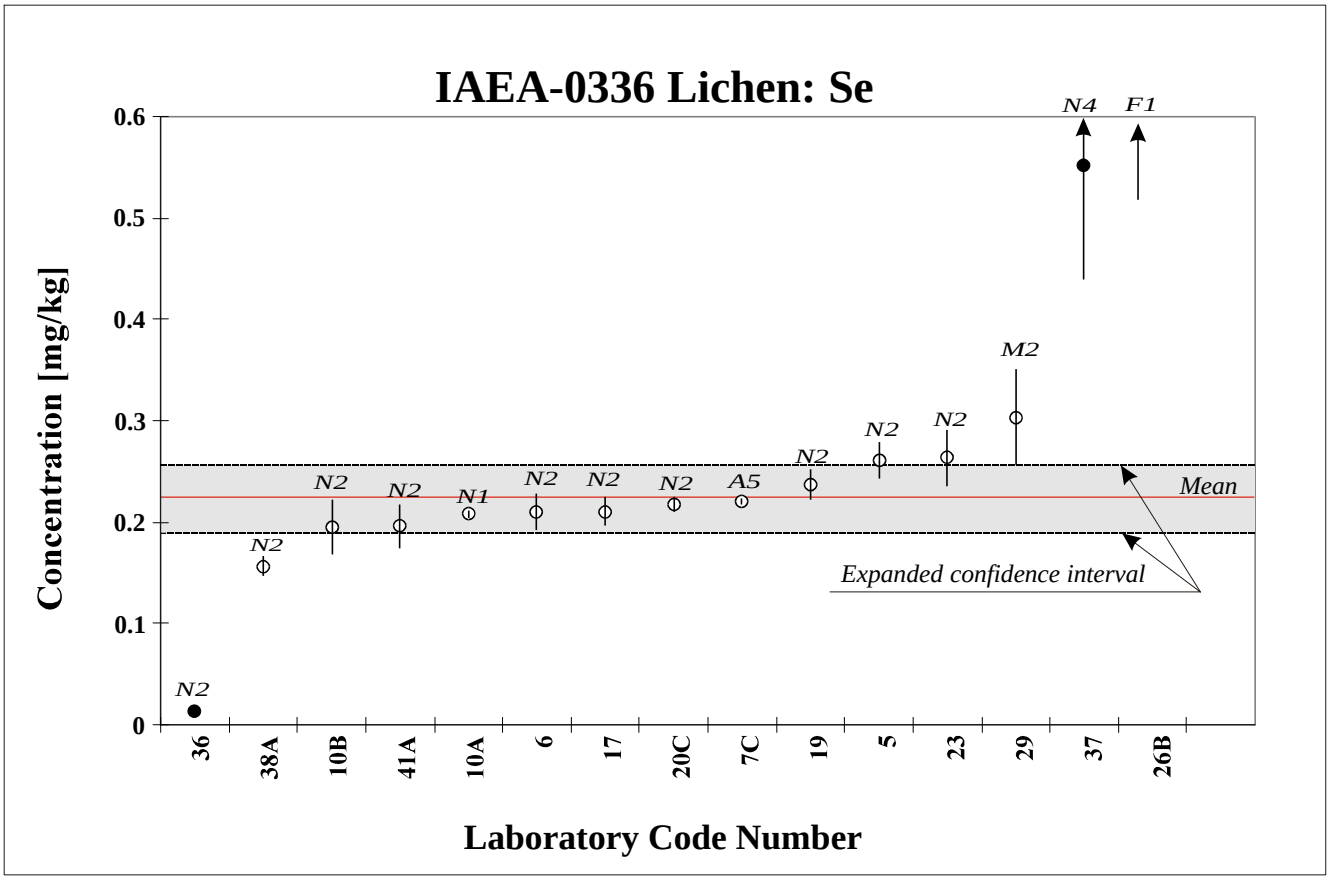

IAEA-0336 Lichen: P
Concentration [mg/kg]
1000
900
800
700
600
500
400
300
200
100
0
C1
C1
E5
E5
E5
E5
E5
X1
X1
X3
N2
C1
M2
Mean
Expanded confidence interval
31
33B
33C
13C
16B
7D
21B
24
14
18
36
34A
11
Laboratory Code Number

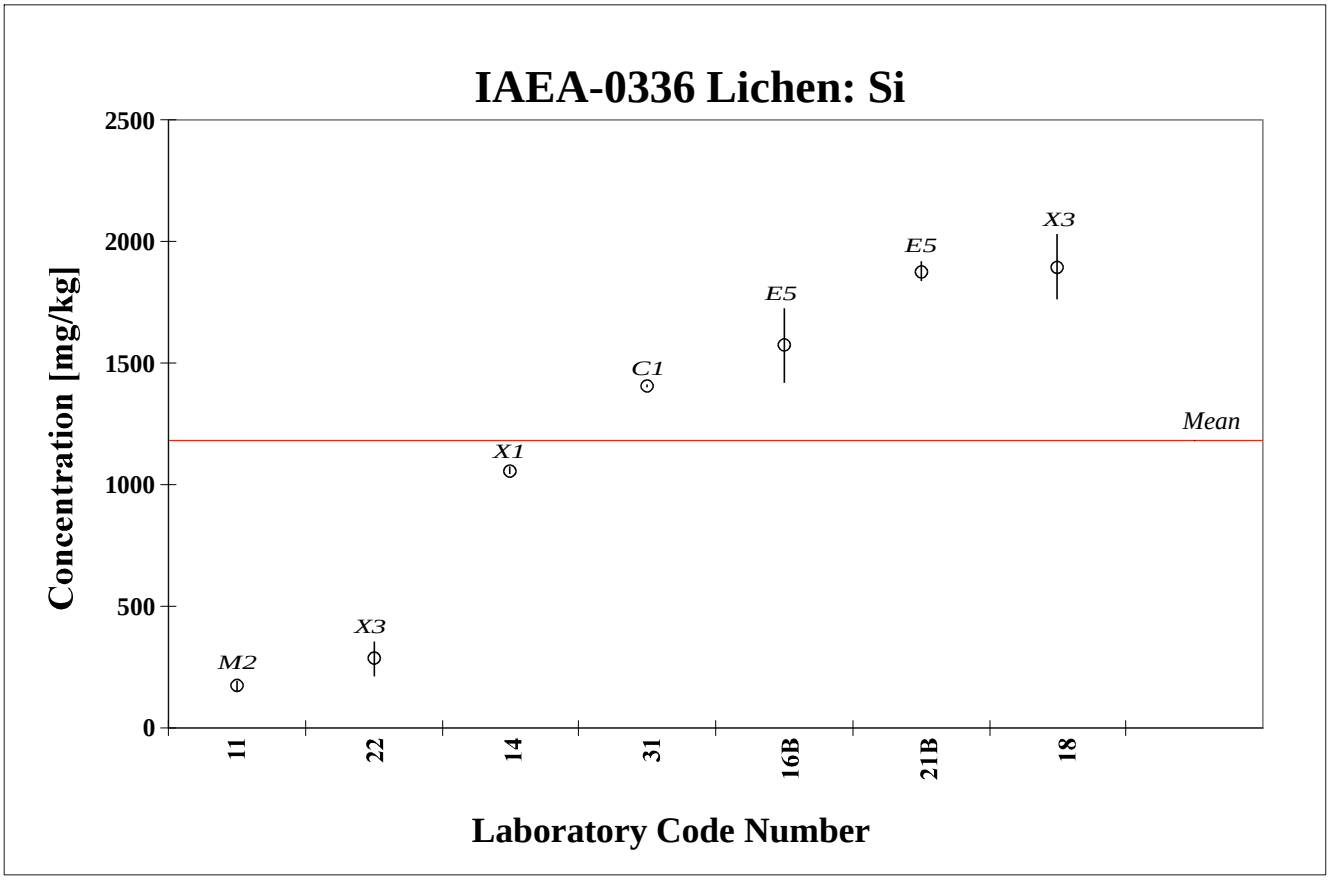

IAEA-0336 Lichen: Pb
Concentration [mg/kg]
14
12
10
8
6
4
2
0
V1
M2
X3
M2
A3
E5
M1
A2
M2
M2
M2
E5
A3
E5
A3
X2
E5
A2
V1
A3
A1
A1
E5
X3
X3
X3
X1
X1
Mean
Expanded confidence interval
31
29
22
47*
4
48*
7E
8B
28
11
50*
16B
44*
46*
7A
42A
49*
2B
7F
13B
41B
2A
1
27B
25
45*
18
14
32
Laboratory Code Number

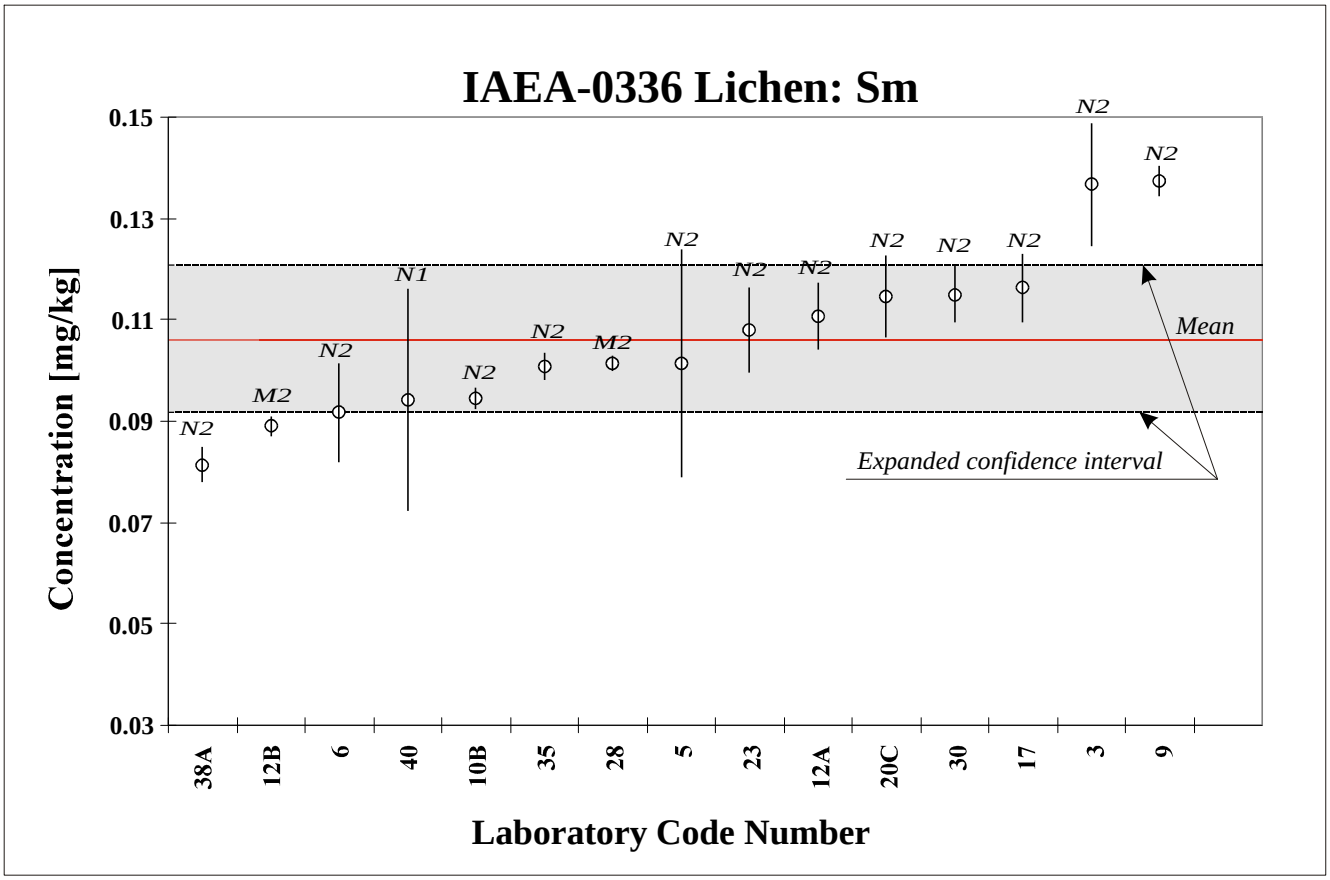

IAEA-0336 Lichen: Rb
Concentration [mg/kg]
3.0
2.5
2.0
1.5
1.0
0.5
0
M2
N1
N2
N2
M2
N2
N2
M2
N2
N2
X2
N2
N4
X3
X3
X1
Mean
Expanded confidence interval
29
40
36
9
11
10B
23
28
17
38A
20C
42A
5
37
27B
18
24
Laboratory Code Number

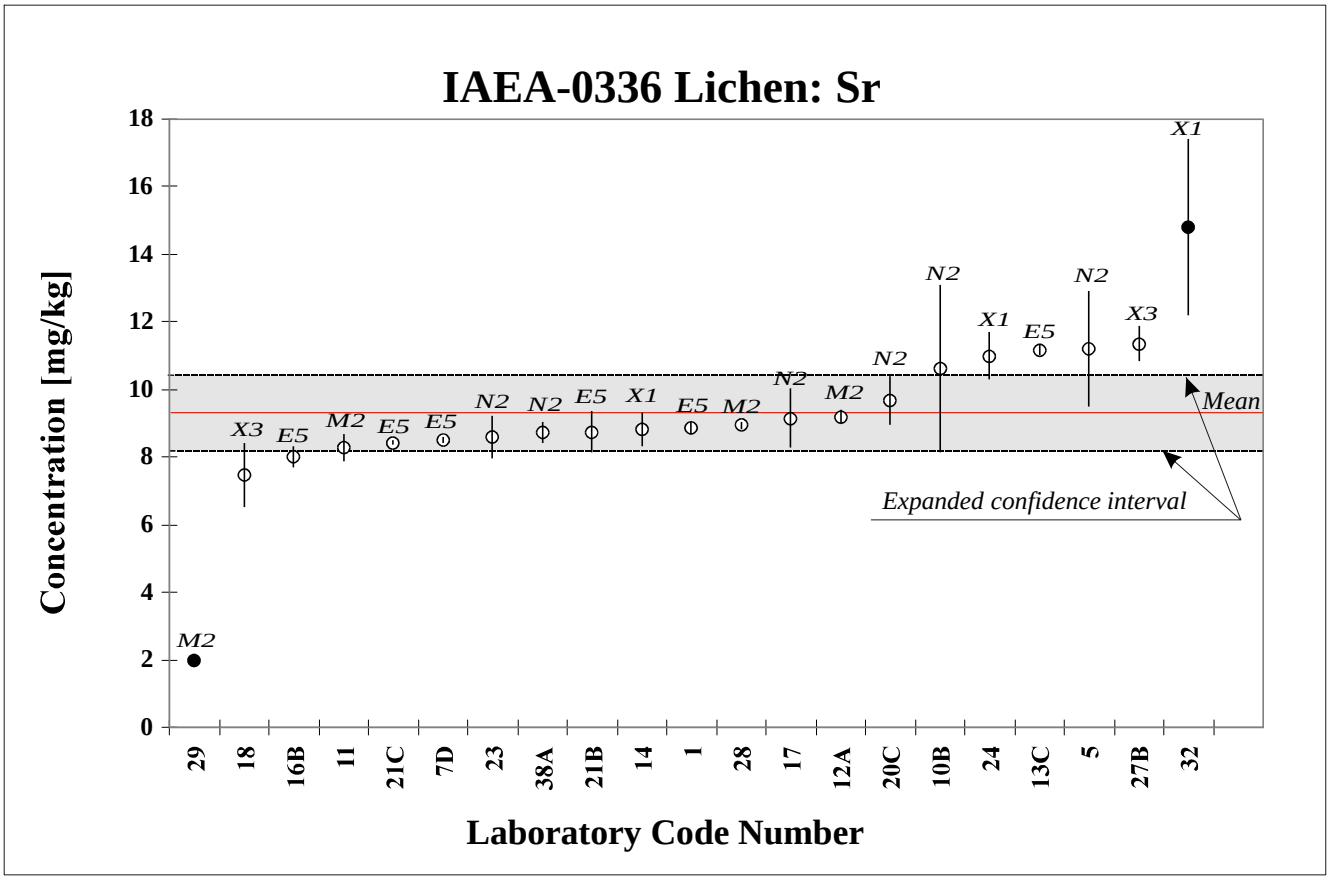

IAEA-0336 Lichen: Sb
Concentration [mg/kg]
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0
N2
N2
N2
E5
N2
N2
N1
N1
N2
N2
N2
N2
N4
Mean
Expanded confidence interval
10B
10A
41A
1
6
20C
10A
40
23
17
38A
5
37
Laboratory Code Number

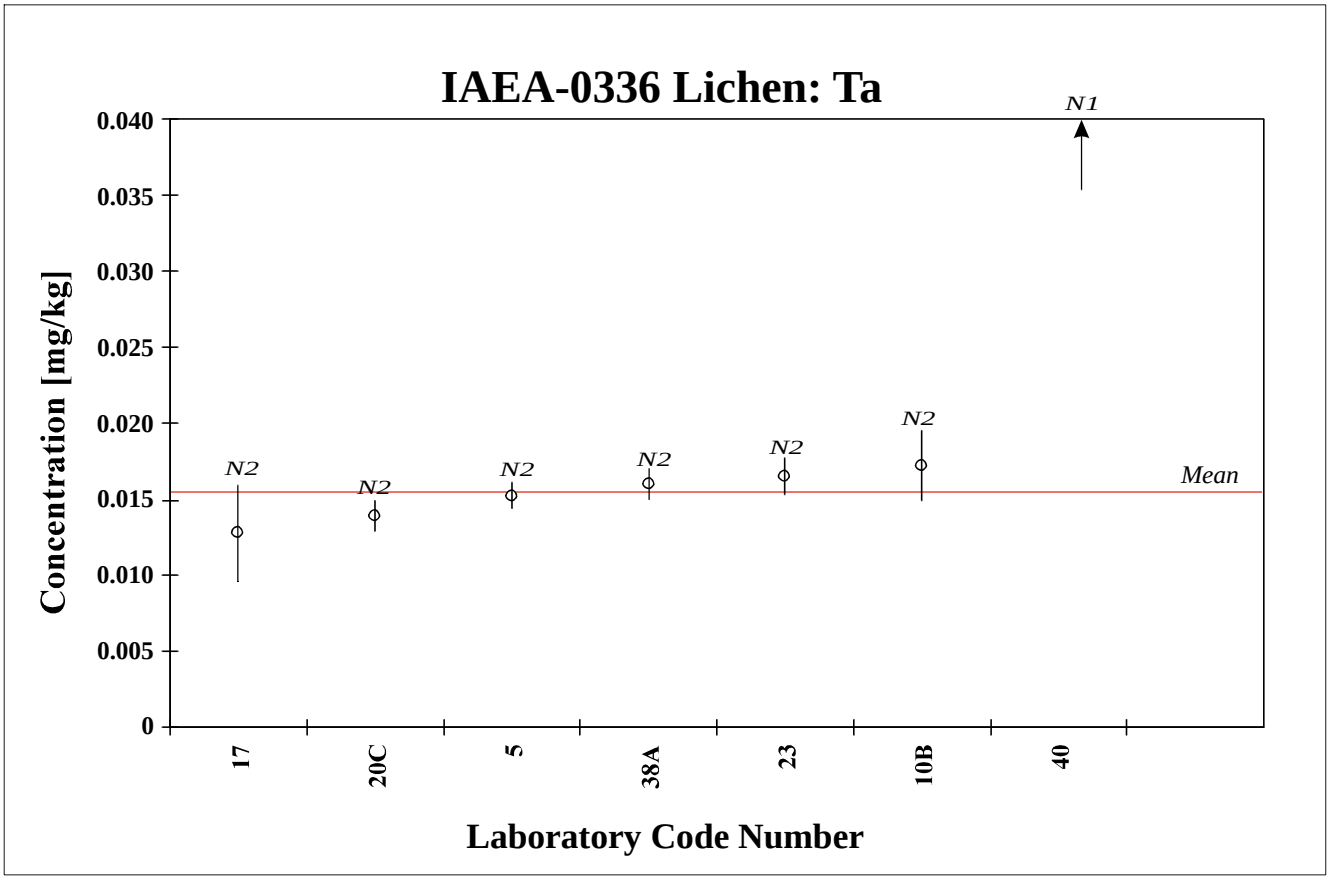

IAEA-0336 Lichen: Sc
Concentration [mg/kg]
0.21
0.19
0.17
0.15
0.13
0.11
0.09
0.07
0.05
Mean
Expanded confidence interval
N2
N1
N4
38A
9
40
19
10B
23
12B
41A
37
3
20C
17
6
5
Laboratory Code Number

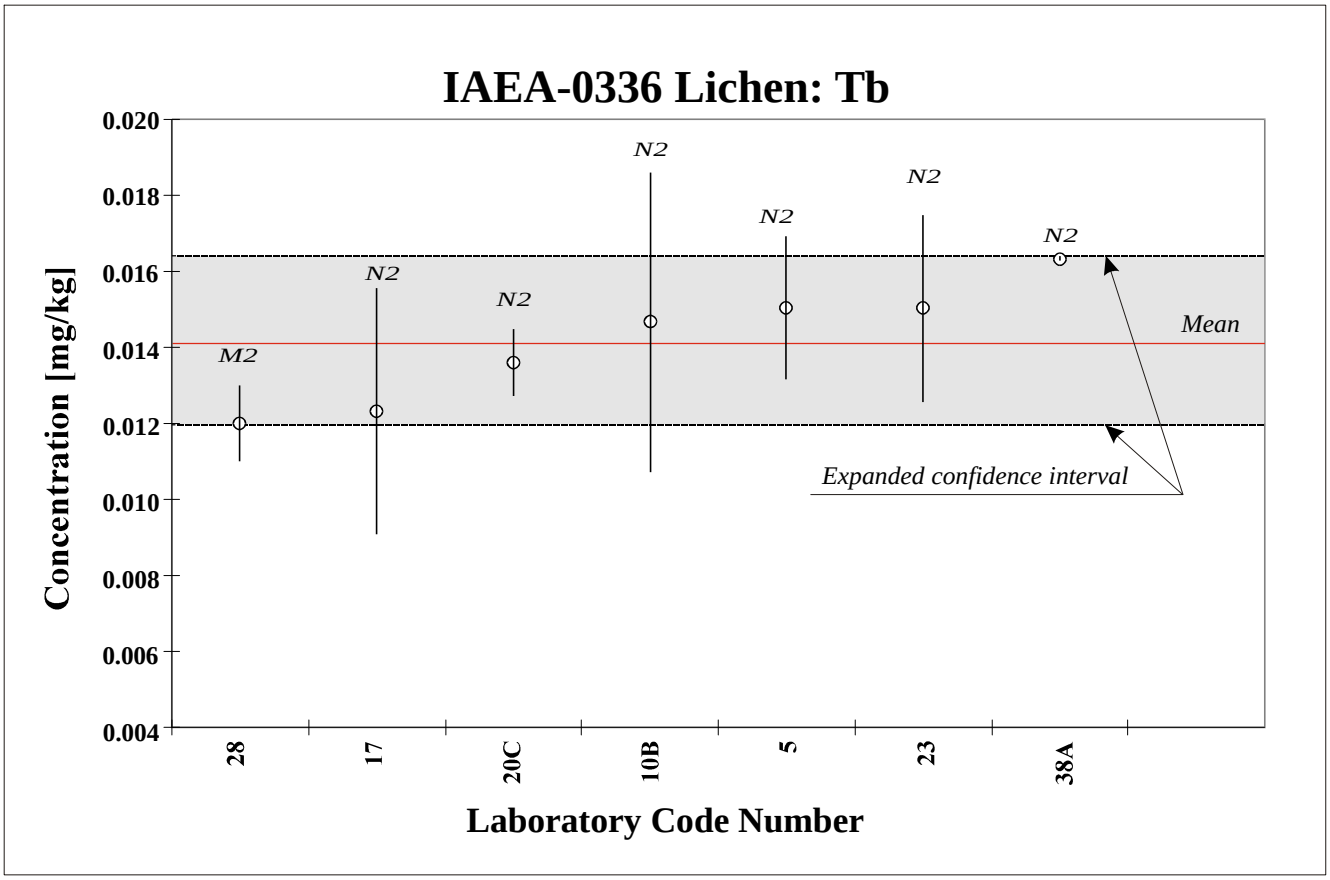

IAEA-0336 Lichen: Se
Concentration [mg/kg]
0.6
0.5
0.4
0.3
0.2
0.1
0
Mean
Expanded confidence interval
N2
N1
N4
F1
M2
A5
36
38A
10B
41A
10A
6
17
20C
7C
19
5
23
29
37
26B
Laboratory Code Number


IAEA-0336 Lichen: Si
Concentration [mg/kg]
2500
2000
1500
1000
500
0
M2
X3
X1
C1
E5
E5
X3
Mean
11
22
14
31
16B
21B
18
Laboratory Code Number


IAEA-0336 Lichen: Sm
Concentration [mg/kg]
0.15
0.13
0.11
0.09
0.07
0.05
0.03
N2
M2
N2
N1
N2
N2
M2
N2
N2
N2
N2
N2
N2
N2
N2
Mean
Expanded confidence interval
38A
12B
6
40
10B
35
28
5
23
12A
20C
30
17
3
9
Laboratory Code Number


IAEA-0336 Lichen: Sr
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval
0
2
4
6
8
10
12
14
16
18
29
18
16B
11
21C
7D
23
38A
21B
14
1
28
17
12A
20C
10B
24
13C
5
27B
32
M2
X3
E5
M2
E5
E5
N2
N2
E5
X1
E5
M2
N2
M2
N2
N2
X1
E5
N2
X3
X1


IAEA-0336 Lichen: Ta
Concentration [mg/kg]
Laboratory Code Number
Mean
0
0.005
0.010
0.015
0.020
0.025
0.030
0.035
0.040
17
20C
5
38A
23
10B
40
N2
N2
N2
N2
N2
N2
N1


IAEA-0336 Lichen: Tb
Concentration [mg/kg]
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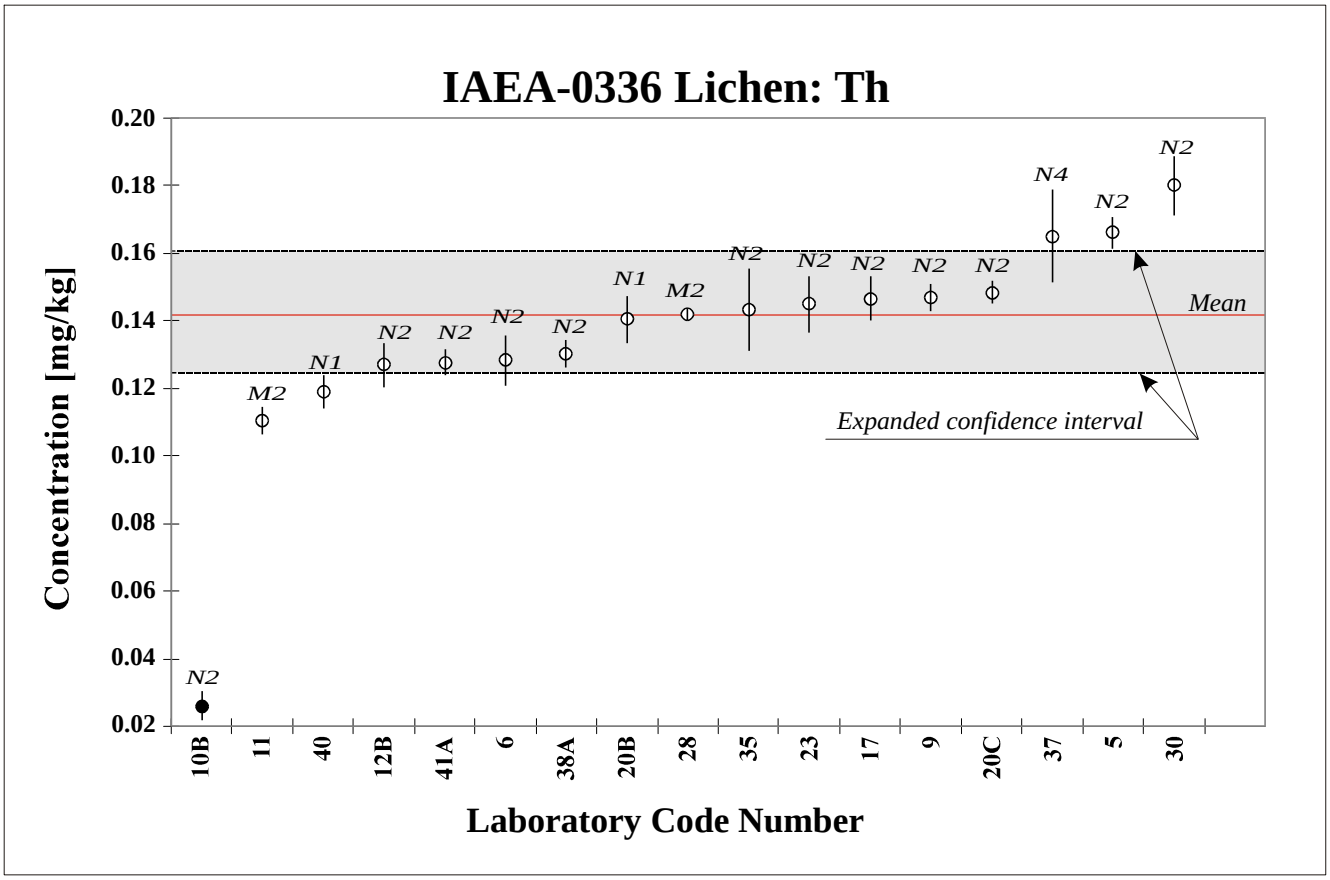
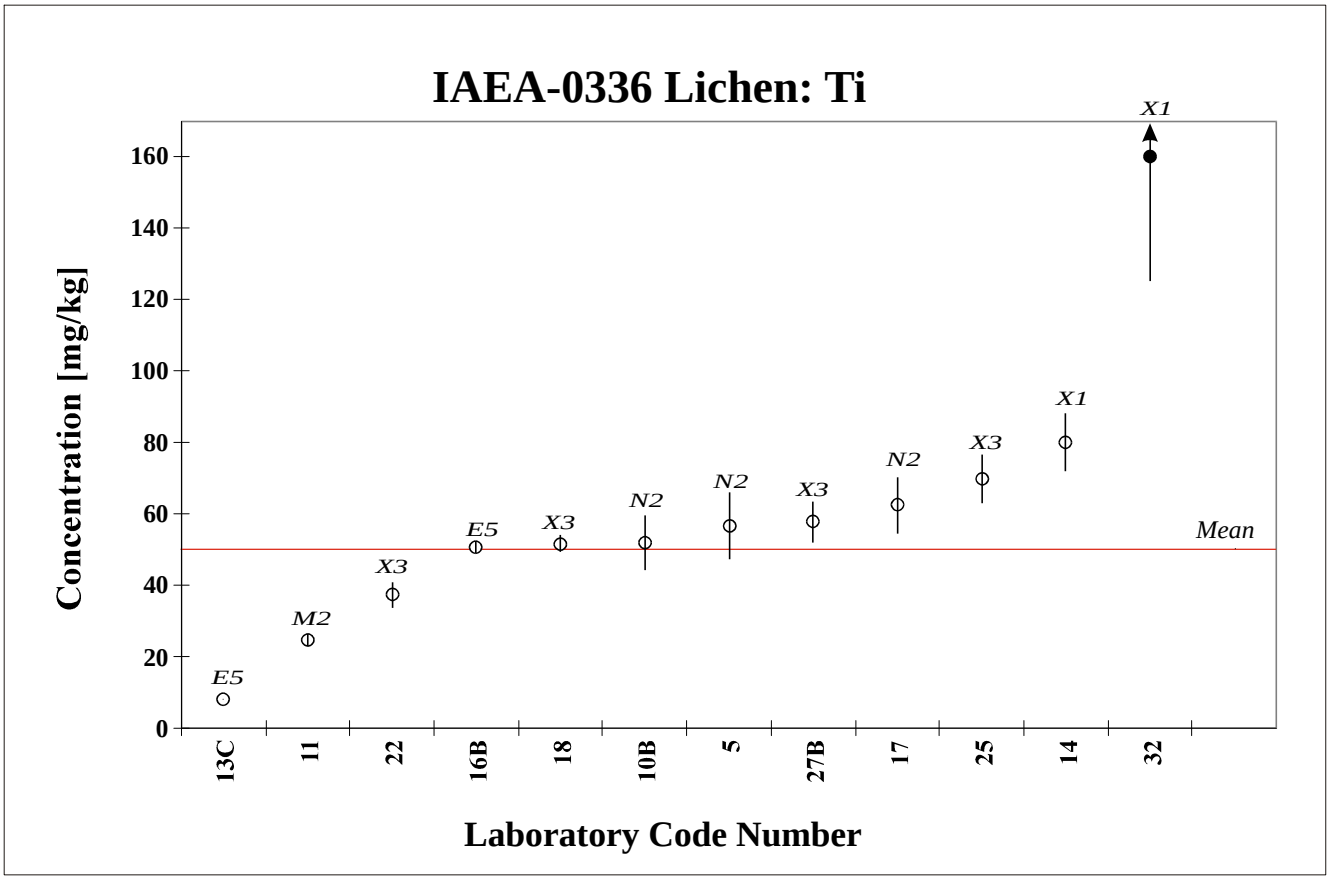
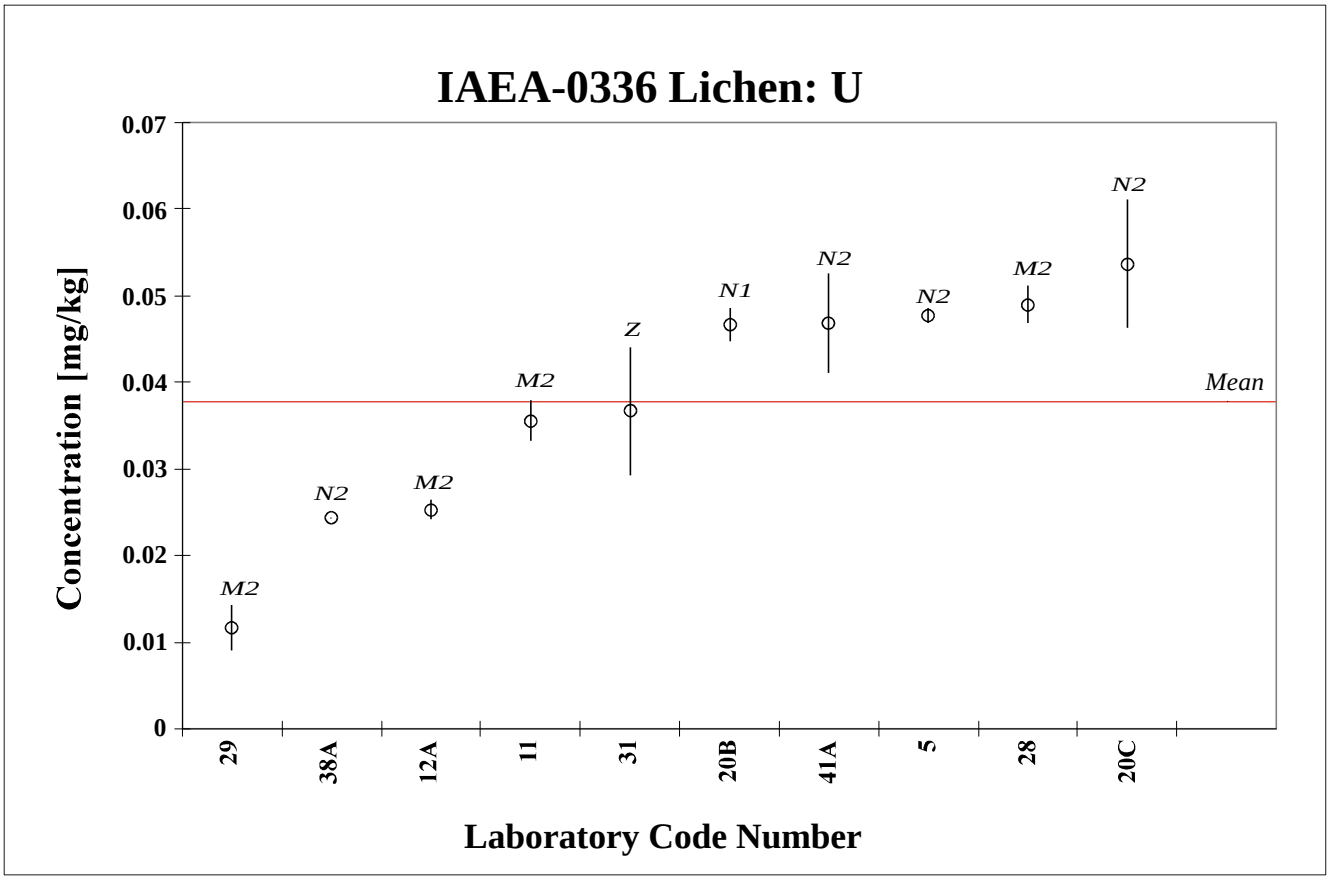
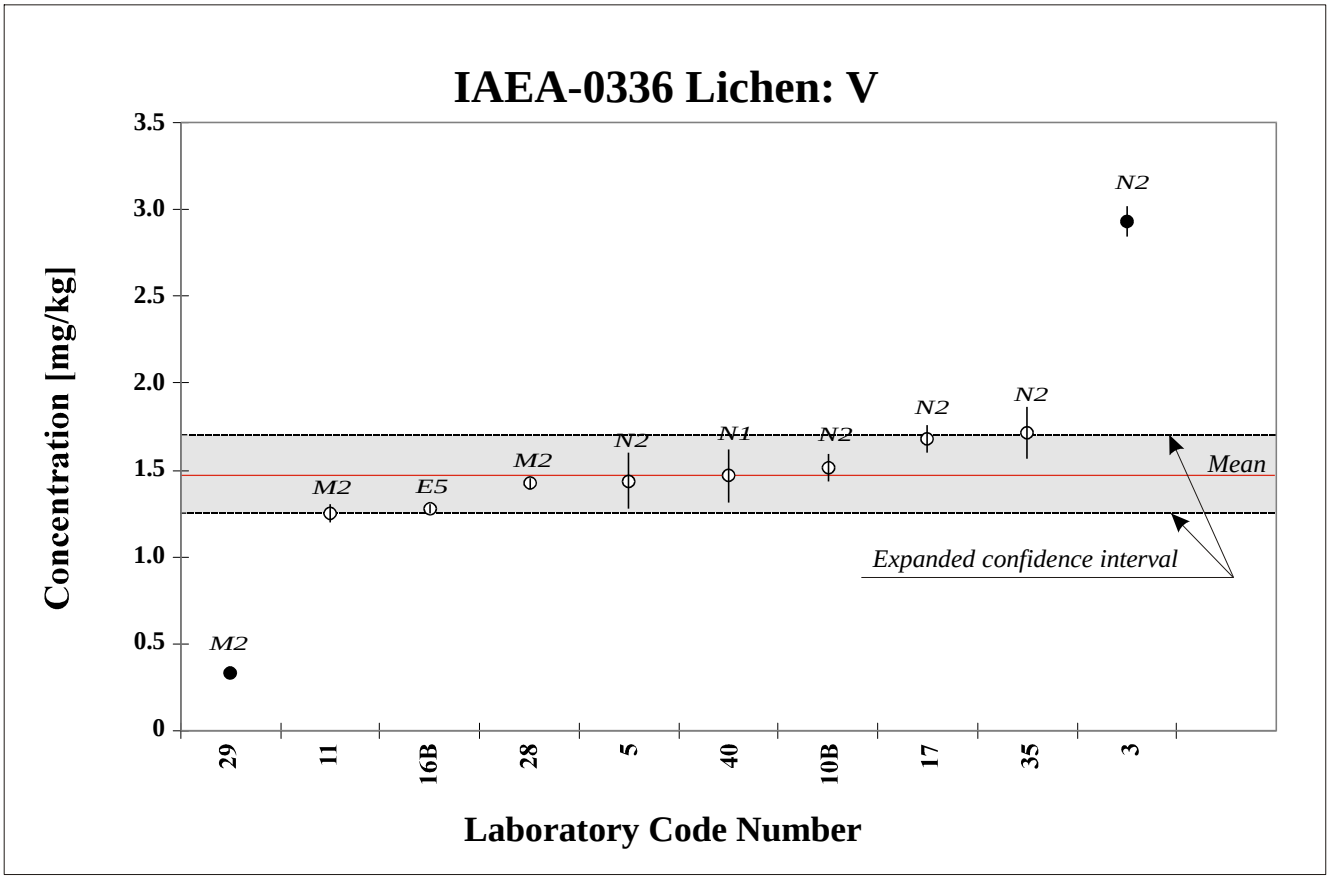
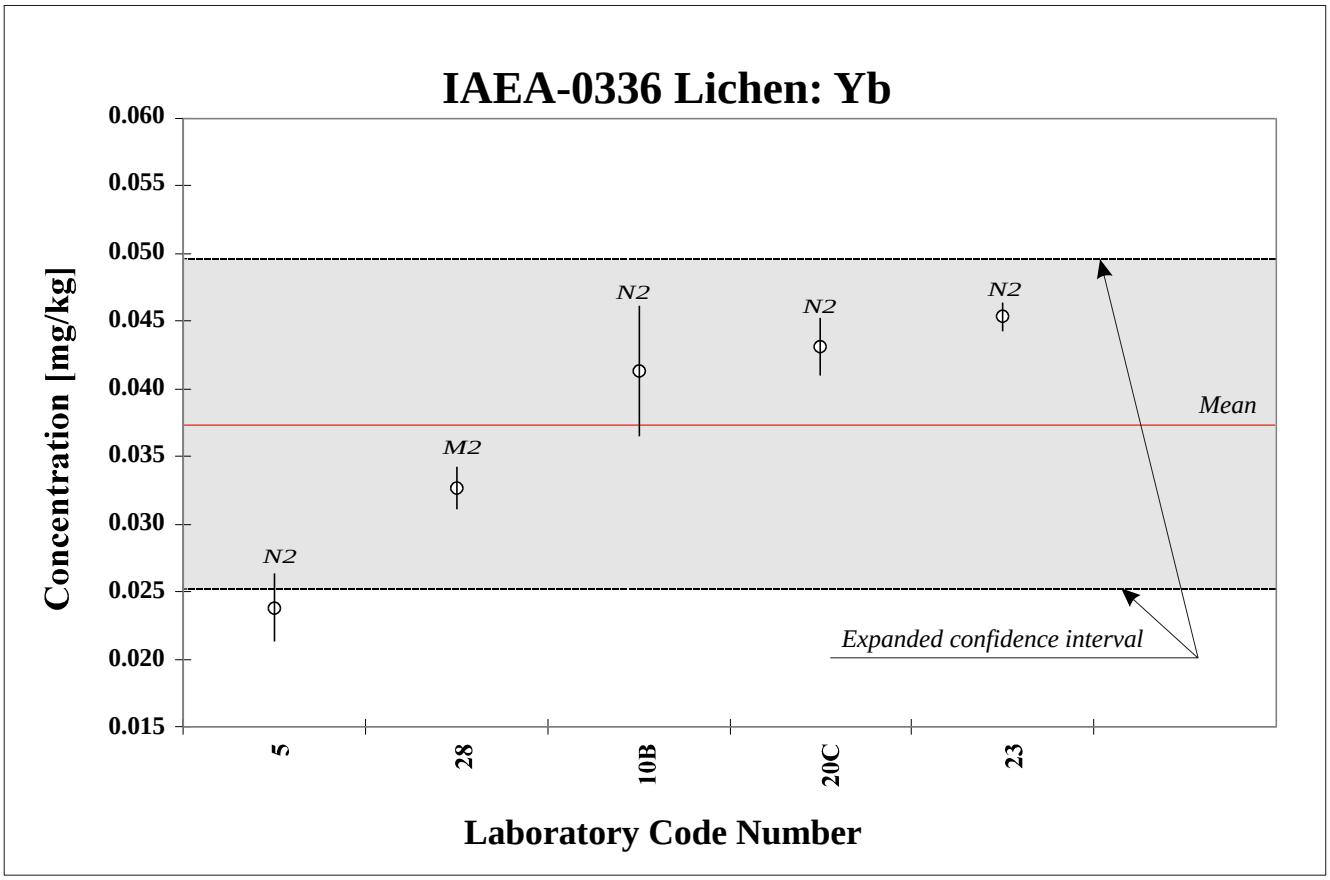
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
M2
N2
N2
N2
N2
N2
N2
Mean
Expanded confidence interval
28
17
20C
10B
5
23
38A
Laboratory Code Number


IAEA-0336 Lichen: Th
Concentration [mg/kg]
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
N2
M2
N1
N2
N2
N2
N2
N1
M2
N2
N2
N2
N2
N2
N4
N2
N2
Mean
Expanded confidence interval
10B
11
40
12B
41A
6
38A
20B
28
35
23
17
9
20C
37
5
30
Laboratory Code Number


IAEA-0336 Lichen: Ti
Concentration [mg/kg]
0
20
40
60
80
100
120
140
160
E5
M2
X3
E5
X3
N2
N2
X3
N2
X3
X1
X1
Mean
13C
11
22
16B
18
10B
5
27B
17
25
14
32
Laboratory Code Number


IAEA-0336 Lichen: U
Concentration [mg/kg]
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
M2
N2
M2
M2
Z
N1
N2
N2
M2
N2
Mean
29
38A
12A
11
31
20B
41A
5
28
20C
Laboratory Code Number


IAEA-0336 Lichen: V
Concentration [mg/kg]
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
M2
M2
E5
M2
N2
N1
N2
N2
N2
N2
Mean
Expanded confidence interval
29
11
16B
28
5
40
10B
17
35
3
Laboratory Code Number


IAEA-0336 Lichen: Yb
Concentration [mg/kg]
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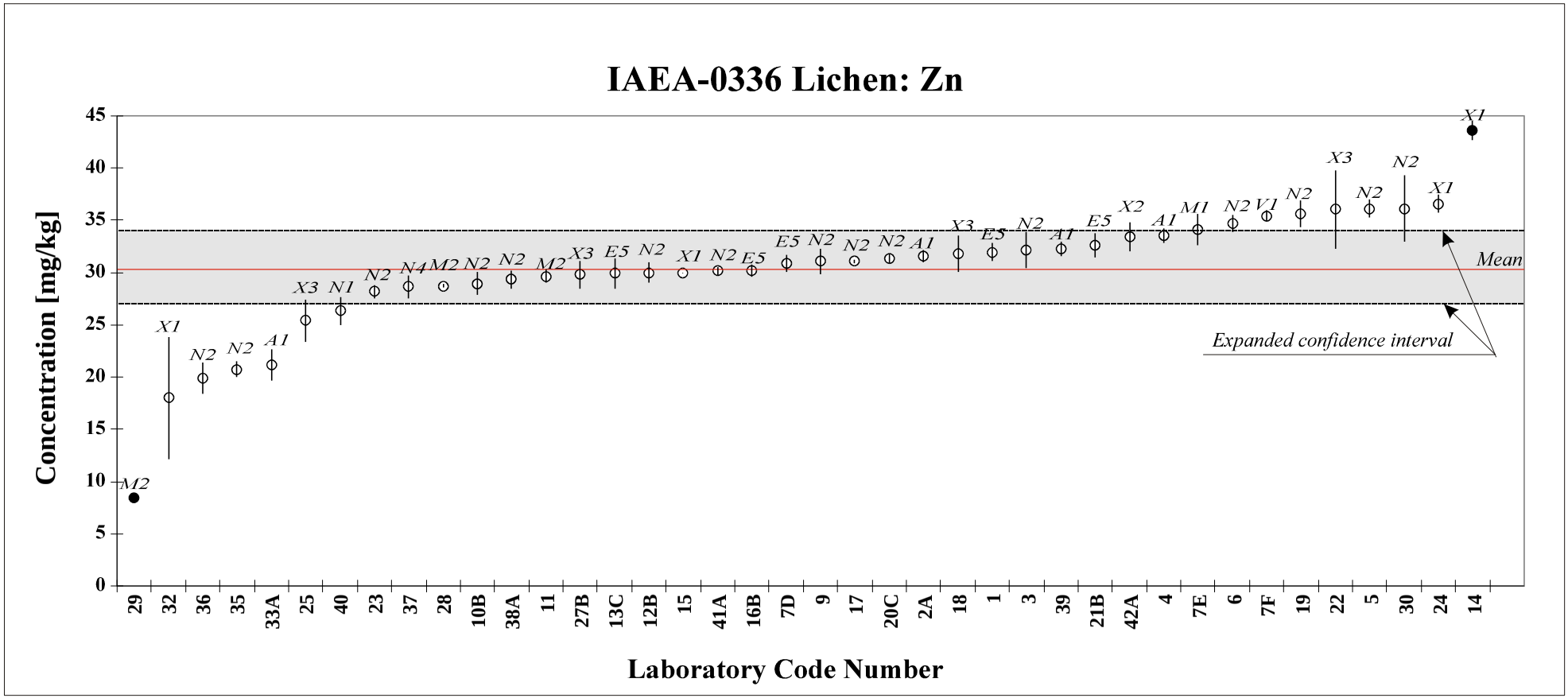
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
N2
M2
N2
N2
N2
Mean
Expanded confidence interval
5
28
10B
20C
23
Laboratory Code Number


IAEA-0336 Lichen: Zn
Concentration [mg/kg]
Laboratory Code Number
Mean
Expanded confidence interval
0
5
10
15
20
25
30
35
40
45
29 32 36 35 33A 25 40 23 37 28 10B 38A 11 27B 13C 12B 15 41A 16B 7D 9 17 20C 2A 18 1 3 39 21B 42A 4 7E 6 7F 19 22 5 30 24 14
M2 X1 N2 N2 A1 X3 N1 N2 N4 M2 N2 N2 M2 X3 E5 N2 X1 N2 E5 E5 N2 N2 N2 A1 X3 E5 N2 A1 E5 X2 A1 M1 N2 V1 N2 X3 N2 N2 X1 X1

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APPENDIX V

REFERENCE SHEET FOR

IAEA-336 LICHEN

International Atomic Energy Agency
Analytical Quality Control Services
Wagramer Strasse 5, P.O.Box 100, A-1400 Vienna, Austria

REFERENCE SHEET

REFERENCE MATERIAL
IAEA-336
TRACE AND MINOR ELEMENTS IN LICHEN

Date of issue: June 1999

Recommended Values
(Based on dry weight)

Element	Recommended Value mg/kg	Confidence Interval* mg/kg	N**
As	0.63	0.55 - 0.71	17
Ba	6.4	5.3 - 7.5	11
Br	12.9	11.2 - 14.6	18
Ce	1.28	1.11 - 1.45	13
Co	0.29	0.24 - 0.34	19
Cs	0.110	0.097 - 0.123	13
Cu	3.6	3.1 - 4.1	21
Fe	430	380 - 480	35
Hg	0.20	0.16 - 0.24	15
K	1840	1640 - 2040	24
La	0.66	0.56 - 0.76	12
Mn	63	56 - 70	29
Na	320	280 - 360	20
Sb	0.073	0.063 - 0.083	12
Se	0.22	0.18 - 0.26	12
Sm	0.106	0.092 - 0.120	15
Sr	9.3	8.2 - 10.4	19
Th	0.14	0.12 - 0.16	16
Zn	30.4	27.0 - 33.8	38

* *It should be noted that the confidence interval was calculated from the combination of the standard deviation of the mean value and an additional 5% to account for any variation due to sample inhomogeneity.*

** *Number of accepted laboratory means which were used to calculate the recommended values and confidence intervals.*

 Revision of the original reference sheet dated October 1994

Information Values
(Based on dry weight)

Element	Information Value mg/kg	Confidence Interval* mg/kg	N**
Al	680	570 - 790	15
Cd	0.117	0.100 - 0.134	14
Cl	1900	1600 - 2200	13
Cr	1.06	0.89 - 1.23	22
Eu	0.023	0.019 - 0.027	9
Lu	0.0066	0.0042 - 0.0090	5
Nd	0.60	0.42 - 0.78	5
P	610	490 - 730	12
Pb	4.9	4.3 - 5.5	23
Rb	1.76	1.54 - 1.98	16
Sc	0.17	0.15 - 0.19	13
Tb	0.014	0.012 - 0.016	7
V	1.47	1.25 - 1.69	8
Yb	0.037	0.025 - 0.049	5

* *It should be noted that the confidence interval was calculated from the combination of the standard deviation of the mean value and an additional 5% to account for any variation due to sample inhomogeneity.*

** *Number of accepted laboratory means which were used to calculate the information values and confidence intervals.*

The values listed above were established on the basis of statistically valid results submitted by laboratories which had participated in an international intercomparison exercise organized during 1992/1994. The details concerning the criteria for qualification as a recommended or an information value can be found in the report NAHRES-33 (IAEA/AL/79) "Intercomparison Run for the Determination of Trace and Minor Elements in Lichen Material IAEA-336" [1]. This report is available free of charge upon request.

Intended Use

This sample is intended to be used as a reference material for the measurement of trace and minor elements in lichens. It can also be used as a quality control material for the assessment of a laboratory's analytical work, for the validation of analytical methods and for quality assurance within a laboratory.

Origin and preparation of the material

The epiphytic lichen *Evernia prunastri (L.) Ach.* was selected and collected by hand. The lichen was collected from areas in Portugal remote from pollution sources. These areas included Gavião (center of Portugal), Ourique and Serra do Cladeirão (south of Portugal). The lichen was harvested from both the *Cistus ladanifer* and *Quercus* species of tree. About 25 kilograms were collected, separated from debris and other epiphytic lichen species by visual inspection, then washed in deionized water and oven-dried at 40 °C for 24 hours. The lichen was ground using a Teflon “Mikro-dismembrator” mill. The final material was passed through a 125 µm sieve and mixed in a rotating plastic drum. Sufficient material was obtained to produce 800 units of 20 g. The material was radiation-sterilized to a total dose of 12 kGy using a ^{60}Co source.

Homogeneity

Homogeneity tests were performed on two 100 mg sub-samples taken from each of 20 bottles. The homogeneity was evaluated based on the variation in the concentration of the elements As, Br, Fe, K, La, Mn, and Sm which were determined by instrumental neutron activation analysis using the k_0 method [2]. The results of a one way ANOVA test showed no significant difference between the within-bottle variance and the between-bottle variance. The relative standard deviation of these results varied from 3 to 11 %. Taking into account the measurement uncertainty, the relative uncertainty due to inhomogeneity was estimated to be between 3 and 6%. Additional measurements for Br, Ca, Fe, Sr, and Zn using X-ray fluorescence [3] supported these results. For these latter measurements the additional uncertainty due to inhomogeneity was estimated to be between 1 and 5 %. Although the degree of inhomogeneity was not the same for all the elements, an additional component of uncertainty (5%) was added in quadrature to expand the confidence interval of each analyte. The final confidence interval includes this additional uncertainty.

Dry weight determination

All recommended and information values are expressed on a dry weight basis. Therefore the dry weight must be determined at the time of analysis, using separate sub-samples of 500 mg dried to constant weight in a drying oven set to 100 °C. Subsequent weighings should differ by less than 5 mg

Instructions for use

The recommended minimum sample size for analysis is 100 mg. Analysts are reminded to take appropriate precautions in order to avoid contaminating the remaining material in the bottle. It is recommended that the material be stored in a dark place, below 20□C (refrigeration is advised).

Legal disclaimer

The IAEA makes no warranties, expressed or implied, with respect to the data contained in this reference sheet and shall not be liable for any damage that may result from the use of such data.

References

[1] HELLER-ZEISLER, S. F., ZEISLER, R., ZEILLER, PARR, R. M, E., RADECKI, Z., BURNS, K. I., DE REGGE, P., Intercomparison run for the determination of trace and minor elements in lichen material IAEA-336, NAHRES-33, (IAEA/AL/79), IAEA, Vienna, June 1999.

[2] FREITAS, M. C., CATARINO, F. M., BRANQUINHO, C., MAGUAS, C., Preparation of a lichen reference material, J. Radioanal. Nucl. Chem. **169** (1993) 47-55.

[3] STONE, S. F., FREITAS, M. C., PARR, R. M., ZEISLER, R., Elemental characterization of a candidate lichen research material-IAEA-336, Fresenius J. Anal. Chem. **352** (1995) 227-231.

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