**Certificate**

# AMIS0565

# Certified Reference Material

## Lepidolite

## *Certificate of Analysis*

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# SUMMARY STATISTICS

## *Recommended Concentrations and Limits (at two Standard Deviations)*

## *Certified Concentrations*

| Analyte | Method | [7]Certified (µ) | [9]Two Standard Deviation (2s) ± | Unit |
|---|---|---|---|---|
| Li | FUS[1] | 5348 | 620 | ppm |
| Li | 4A_MICP[2] | 5424 | 468 | ppm |
| Be | 4A_MICP | 366 | 96 | ppm |
| Cs | 4A_MICP | 643 | 112 | ppm |
| Ga | 4A_MICP | 44 | 5 | ppm |
| Fe | FUS | 4584 | 710 | ppm |
| Fe | 4A_MICP | 4441 | 559 | ppm |
| Nb | 4A_MICP | 54 | 12 | ppm |
| Rb | FUS | 4202 | 337 | ppm |
| Sn | 4A_MICP | 14 | 4 | ppm |
| Ta | 4A_MICP | 46 | 14 | ppm |
| C | Combustion/LECO[3] | 5398 | 379 | ppm |
| LOI | LOI[4] | 3.67 | 0.43 | % |
| SG | SG[5] | 2.68 | 0.11 | Dimensionless |
| Ag | 4A_MICP | 1 | 0.1 | ppm |
| Al | FUS | 76324 | 2282 | ppm |
| As | 4A_MICP | 6 | 2 | ppm |
| Ba | 4A_MICP | 33 | 5 | ppm |
| Bi | 4A_MICP | 37 | 7 | ppm |
| Ca | 4A_MICP | 17232 | 1108 | ppm |
| Co | 4A_MICP | 2 | 0.3 | ppm |
| Cu | 4A_MICP | 11 | 2 | ppm |
| Dy | 4A_MICP | 0.4 | 0.1 | ppm |
| Er | 4A_MICP | 0.1 | 0.1 | ppm |
| Gd | 4A_MICP | 0.3 | 0.2 | ppm |
| Hf | 4A_MICP | 6 | 2 | ppm |
| K | 4A_MICP | 28920 | 1380 | ppm |
| La | 4A_MICP | 1 | 0.5 | ppm |
| Mg | FUS | 2599 | 286 | ppm |
| Mn | 4A_MICP | 1508 | 250 | ppm |
| Mo | 4A_MICP | 1 | 0.1 | ppm |
| Na | 4A_MICP | 15529 | 1269 | ppm |
| Nd | 4A_MICP | 1 | 1 | ppm |
| Ni | 4A_MICP | 5 | 1 | ppm |
| P | 4A_MICP | 1996 | 202 | ppm |
| Pb | 4A_MICP | 31 | 5 | ppm |
| Pr | 4A_MICP | 0.4 | 0.1 | ppm |
| Sb | 4A_MICP | 4 | 1 | ppm |
| Sc | 4A_MICP | 1 | 0.2 | ppm |
| Sm | 4A_MICP | 0.3 | 0.1 | ppm |
| Sr | 4A_MICP | 70 | 10 | ppm |
| Tb | 4A_MICP | 0.1 | 0.04 | ppm |
| Th | 4A_MICP | 4 | 1 | ppm |
| Tl | 4A_MICP | 24 | 4 | ppm |
| U | 4A_MICP | 7 | 1 | ppm |
| V | 4A_MICP | 8 | 2 | ppm |
| W | 4A_MICP | 8 | 2 | ppm |
| Y | 4A_MICP | 2 | 0.4 | ppm |
| Yb | 4A_MICP | 0 | 0.1 | ppm |
| Zn | 4A_MICP | 74 | 14 | ppm |
| Zr | 4A_MICP | 25 | 6 | ppm |

# Major Oxides

## *Certified Concentrations (at two Standard Deviations)*

| Analyte | Method | [7]Certified (μ) | [9]Two Standard Deviation (2s) ± | Unit |
|---|---|---|---|---|
| $Al_2O_3$ | XRF[6] | 14.60 | 0.39 | % |
| $Al_2O_3$ | FUS | 14.39 | 0.46 | % |
| CaO | XRF | 2.38 | 0.14 | % |
| $Fe_2O_3$ | XRF | 0.66 | 0.05 | % |
| $K_2O$ | XRF | 3.57 | 0.15 | % |
| MgO | XRF | 0.43 | 0.04 | % |
| MgO | FUS | 0.43 | 0.05 | % |
| MnO | XRF | 0.21 | 0.01 | % |
| $Na_2O$ | XRF | 2.13 | 0.058 | % |
| $P_2O_5$ | XRF | 0.45 | 0.05 | % |
| $SiO_2$ | XRF | 69.79 | 1.27 | % |

## 1. Certified Concentrations and Uncertainties

AMIS0565 is a new standard material, developed and certified in June 2018. Table 1 gives the certified concentrations, combined and expanded uncertainty for certified reference material. Table 2 shows the certified major oxides concentrations, two standard deviations, combined and expanded uncertainty.

**Table 1.** Certified concentrations, two standard deviations, combined and expanded uncertainty.

| Analyte | Method | [7]Certified (μ) | *N* | n | k | % RSD | [8]Combined uncertainty (uc) | [9]Two Standard Deviation (2s) ± | [10]Expanded uncertainty (U) ± | Unit |
|---|---|---|---|---|---|---|---|---|---|---|
| Li | FUS[1] | 5348 | 5 | 40 | 2.78 | 6 | 310 | 620 | 860 | ppm |
| Li | 4A_MICP[2] | 5424 | 7 | 56 | 2.45 | 4 | 234 | 468 | 572 | ppm |
| Be | 4A_MICP | 366 | 8 | 61 | 2.36 | 13 | 48 | 96 | 113 | ppm |
| Cs | 4A_MICP | 643 | 6 | 44 | 2.57 | 9 | 56 | 112 | 144 | ppm |
| Ga | 4A_MICP | 44 | 7 | 53 | 2.45 | 6 | 2 | 5 | 6 | ppm |
| Fe | FUS | 4584 | 4 | 32 | 3.18 | 8 | 355 | 710 | 1131 | ppm |
| Fe | 4A_MICP | 4441 | 7 | 56 | 2.45 | 6 | 280 | 559 | 684 | ppm |
| Nb | 4A_MICP | 54 | 6 | 47 | 2.57 | 11 | 6 | 12 | 16 | ppm |
| Rb | FUS | 4202 | 3 | 22 | 4.30 | 4 | 169 | 337 | 726 | ppm |
| Sn | 4A_MICP | 14 | 7 | 54 | 2.45 | 13 | 2 | 4 | 5 | ppm |
| Ta | 4A_MICP | 46 | 6 | 44 | 2.57 | 16 | 7 | 14 | 18 | ppm |
| C | Combustion/LECO[3] | 5398 | 4 | 32 | 3.18 | 4 | 189 | 379 | 603 | ppm |
| LOI | LOI[4] | 3.67 | 10 | 78 | 2.26 | 6 | 0.22 | 0.43 | 0.5 | % |
| SG | SG[5] | 2.68 | 7 | 53 | 2.45 | 2 | 0.054 | 0.11 | 0.1 | Dimensionless |
| Ag | 4A_MICP | 1 | 5 | 33 | 2.78 | 15 | 0.1 | 0.1 | 0.2 | ppm |
| Al | FUS | 76324 | 3 | 22 | 4.30 | 1 | 1141 | 2282 | 4910 | ppm |
| As | 4A_MICP | 6 | 8 | 62 | 2.36 | 14 | 1 | 2 | 2 | ppm |
| Ba | 4A_MICP | 33 | 8 | 62 | 2.36 | 8 | 3 | 5 | 6 | ppm |
| Bi | 4A_MICP | 37 | 8 | 62 | 2.36 | 10 | 4 | 7 | 9 | ppm |
| Ca | 4A_MICP | 17232 | 8 | 61 | 2.36 | 3 | 554 | 1108 | 1310 | ppm |
| Co | 4A_MICP | 2 | 4 | 32 | 3.18 | 8 | 0.2 | 0.3 | 0.5 | ppm |
| Cu | 4A_MICP | 11 | 5 | 40 | 2.78 | 9 | 1 | 2 | 3 | ppm |
| Dy | 4A_MICP | 0.4 | 3 | 24 | 4.30 | 16 | 0.07 | 0.1 | 0.3 | ppm |
| Er | 4A_MICP | 0.1 | 3 | 24 | 4.30 | 24 | 0.03 | 0.1 | 0.1 | ppm |
| Gd | 4A_MICP | 0.3 | 3 | 24 | 4.30 | 33 | 0.1 | 0.2 | 0.5 | ppm |
| Hf | 4A_MICP | 6 | 7 | 53 | 2.45 | 12 | 1 | 2 | 2 | ppm |
| K | 4A_MICP | 28920 | 7 | 53 | 2.45 | 2 | 690 | 1380 | 1689 | ppm |
| La | 4A_MICP | 1 | 6 | 44 | 2.57 | 18 | 0.3 | 0.5 | 0.7 | ppm |
| Mg | FUS | 2599 | 3 | 23 | 4.30 | 6 | 143 | 286 | 616 | ppm |
| Mn | 4A_MICP | 1508 | 8 | 62 | 2.36 | 8 | 125 | 250 | 296 | ppm |
| Mo | 4A_MICP | 1 | 5 | 39 | 2.78 | 9 | 0.1 | 0.1 | 0.2 | ppm |
| Na | 4A_MICP | 15529 | 7 | 55 | 2.45 | 4 | 635 | 1269 | 1553 | ppm |
| Nd | 4A_MICP | 1 | 4 | 32 | 3.18 | 25 | 0.3 | 1 | 1 | ppm |

| Analyte | Method | [7]Certified (μ) | *N* | n | k | % RSD | [8]Combined uncertainty (uc) | [9]Two Standard Deviation (2s) ± | [10]Expanded uncertainty (U) ± | Unit |
|---|---|---|---|---|---|---|---|---|---|---|
| Ni | 4A_MICP | 5 | 8 | 60 | 2.36 | 14 | 1 | 1 | 2 | ppm |
| P | 4A_MICP | 1996 | 7 | 54 | 2.45 | 5 | 101 | 202 | 247 | ppm |
| Pb | 4A_MICP | 31 | 8 | 57 | 2.36 | 8 | 3 | 5 | 6 | ppm |
| Pr | 4A_MICP | 0.4 | 3 | 23 | 4.30 | 21 | 0.1 | 0.1 | 0.3 | ppm |
| Sb | 4A_MICP | 4 | 6 | 46 | 2.57 | 14 | 0.5 | 1 | 1 | ppm |
| Sc | 4A_MICP | 1 | 4 | 31 | 3.18 | 12 | 0.1 | 0.2 | 0.3 | ppm |
| Sm | 4A_MICP | 0.3 | 3 | 24 | 4.30 | 22 | 0.1 | 0.1 | 0 | ppm |
| Sr | 4A_MICP | 70 | 7 | 55 | 2.45 | 7 | 5 | 10 | 12 | ppm |
| Tb | 4A_MICP | 0.1 | 4 | 32 | 3.18 | 22 | 0.02 | 0.04 | 0.1 | ppm |
| Th | 4A_MICP | 4 | 6 | 46 | 2.57 | 11 | 0.4 | 1 | 1 | ppm |
| Tl | 4A_MICP | 24 | 7 | 53 | 2.45 | 8 | 2 | 4 | 5 | ppm |
| U | 4A_MICP | 7 | 6 | 45 | 2.57 | 10 | 1 | 1 | 2 | ppm |
| V | 4A_MICP | 8 | 6 | 46 | 2.57 | 13 | 1 | 2 | 3 | ppm |
| W | 4A_MICP | 8 | 7 | 52 | 2.45 | 13 | 1 | 2 | 2 | ppm |
| Y | 4A_MICP | 2 | 7 | 54 | 2.45 | 13 | 0.2 | 0.4 | 0.5 | ppm |
| Yb | 4A_MICP | 0 | 4 | 25 | 3.18 | 22 | 0.03 | 0.1 | 0.1 | ppm |
| Zn | 4A_MICP | 74 | 7 | 53 | 2.45 | 9 | 7 | 14 | 17 | ppm |
| Zr | 4A_MICP | 25 | 8 | 63 | 2.36 | 12 | 3 | 6 | 8 | ppm |

**Table 2.** Certified major oxides concentrations, two standard deviations, combined and expanded uncertainty.

| Analyte | Method | [7]Certified (μ) | *N* | n | k | % RSD | [8]Combined uncertainty (uc) | [9]Two Standard Deviation (2s) ± | [10]Expanded uncertainty (U) ± | Unit |
|---|---|---|---|---|---|---|---|---|---|---|
| $Al_2O_3$ | XRF[6] | 14.60 | 9 | 70 | 2.31 | 1 | 0.19 | 0.39 | 0.5 | % |
| $Al_2O_3$ | FUS | 14.39 | 3 | 23 | 4.30 | 2 | 0.23 | 0.46 | 1 | % |
| CaO | XRF | 2.38 | 9 | 68 | 2.31 | 3 | 0.1 | 0.14 | 0.2 | % |
| $Fe_2O_3$ | XRF | 0.66 | 8 | 61 | 2.36 | 4 | 0.02 | 0.05 | 0.06 | % |
| $K_2O$ | XRF | 3.57 | 9 | 69 | 2.31 | 2 | 0.1 | 0.15 | 0.2 | % |
| MgO | XRF | 0.43 | 8 | 62 | 2.36 | 5 | 0.02 | 0.04 | 0.05 | % |
| MgO | FUS | 0.43 | 3 | 23 | 4.30 | 6 | 0.02 | 0.05 | 0.1 | % |
| MnO | XRF | 0.21 | 8 | 57 | 2.36 | 3 | 0.01 | 0.01 | 0.01 | % |
| $Na_2O$ | XRF | 2.13 | 6 | 48 | 2.57 | 1 | 0.03 | 0.058 | 0.08 | % |
| $P_2O_5$ | XRF | 0.45 | 9 | 72 | 2.31 | 5 | 0.02 | 0.05 | 0.05 | % |
| $SiO_2$ | XRF | 69.79 | 9 | 68 | 2.31 | 1 | 1 | 1.27 | 1 | % |

1. *FUS is Fusion by ICP finish*
2. *4A_MICP is Multi acid digestion with ICP Finish*
3. *Combustion/LECO is analysis by combustion infra-red*
4. *LOI is Loss on Ignition*
5. *SG is specific gravity*
6. *XRF is X-ray Fluorescence*
7. *The certified value μ, is an unweighted grand mean of the means of N accepted sets of data from different laboratories and n number of test sample replicates. The certified value is traceable to SI units and is reported on a dry basis.*
8. *The combined uncertainty of the certified value is the within-laboratory reproducibility standard deviation derived from the analysis of variance of results from N number of laboratories and n number of sample replicates.*
9. *The two standard deviations (2s) is calculated as for example: uc x 2 = 0.23 x 2=0.46%. See section 27, page 15 for recommended use in quality control.*
10. *Expanded uncertainty (U) at a confidence level of 95% is determined by multiplication of the combined uncertainty (uc) with a coverage factor (k) found from N-1 degrees of freedom (see Appendix 7 for t-distribution table). Example: U = 2.36 x 0.23=0.54%.*
11. *Informational values are only to be used as indicative values*

## 2. Statistical Comparison of Means

A comparison of means for replicate data for the same element concentration determined by different analytical methods is firstly done equating the variances between the two data sets; if the variances are found to be equal (F-test, *p*-value>0.05), then an equal variance t-test is applied. Should the variances be statistically significant, i.e. *p*<0.05, then an unequal variance t-test is performed. For either t-test, if the obtained *p*-value >0.05, the null hypothesis that the means (certified values) are equal is accepted (**Error! Reference source not found.**). This gives the analyst confidence in the certified values reported by different analytical methods on the same analyte.

**Table 3.** The results of a two-sample equal variance t-test (two-tailed) for oxides/elements by XRF and ICP Fusion demonstrating equal variances and equal means.

| Method | Certified value | Method | Certified value | p-value (t-test) | t-test outcome |
|---|---|---|---|---|---|
| $Al_2O_3$ XRF | 5224 ppm | $Al_2O_3$ Fus | 5348 ppm | 0.50 | Accept $H_0$; certified values are equal |
| MgO XRF | 0.43 % | MgO Fus | 0.43 % | 0.98 | Accept $H_0$; certified values are equal |
| Fe Fus | 4584 ppm | Fe 4A_MICP | 4441 ppm | 0.46 | Accept $H_0$; certified values are equal |
| Li Fus | 5348 ppm | Li 4A_MICP | 5424 ppm | 0.59 | Accept $H_0$; certified values are equal |

## 3. Intended Use

AMIS0565 is a matrix matched Certified Reference Material, fit for use as a control sample in routine assay laboratory quality control when inserted within runs of test samples and measured in parallel to test samples. This material can also be used for method development, use as independent calibration verification check standard (*i.e.* if not used as a calibration standard in an instrument calibration), or for validation of accuracy in a method validation exercise (see Appendix 3). The recommend procedure for the use of this CRM as a control standard in laboratory quality control is to develop a Shewhart chart, where a mean value and corresponding 1, 2 and 3 standard deviations are derived from replicate measurements of the CRM (see Appendix 4). This CRM can also be used to assess inter-laboratory or instrument bias and establish within-laboratory precision and within-laboratory reproducibility. The certified concentrations and expanded uncertainty for this material are property values based on an inter-laboratory measurement campaign and reflect consensus results from the laboratories that participated in the exercise.

## 4. Abbreviations and Symbols

Abbreviations and symbols used in this document are shown in Table 4.

**Table 4.** Abbreviations, symbols and descriptions.

| Abbreviation/Symbol | Description |
|---|---|
| Alpha (α) | Significance level (denoted by alpha, 'α') of 0.05 or 5% |
| ANOVA | Analysis of variance by statistical means |
| BIF | Banded iron formation |
| CRM | Certified reference material |
| *df* | Degrees of freedom, typically, *n*-1, or *N*-1 |
| $F_{calc}$ | Calculated F statistic from ANOVA or Fisher's test |
| F-critical or $F_{crit}$ | F-critical value from F-distribution table |
| GOI | Gain on ignition |
| $H_0$ | Null hypothesis |
| $H_1$ | Alternate hypothesis |
| g/t | Grams per tonne |
| *k* | Coverage factor, *e.g. k*=2 for 95% level of confidence |
| LOC | Level of confidence or confidence level |
| LOD | Limit of detection |
| LOQ | Limit of quantitation |
| LOI | Loss on ignition |
| MS | Mean squares (ANOVA) |
| MSb | Mean squares between(ANOVA) |
| MSw | Mean squares within (ANOVA) |
| *N* | Number of labs |
| *n* | Number of replicates |
| μ | Property or certified value of a CRM |

**Table 4** Continued.

| Abbreviation/Symbol | Description |
|---|---|
| *p* | '*p*-value' a measure of the strength of evidence against $H_0$ |
| P | Total number of data points in ANOVA |
| ppm | Parts per million. Equivalent to g/t |
| RSD | Relative standard deviation usually expressed as % at a 68% LOC |
| Replicates | Replication is the repetition of an experimental condition so that the variability associated with an analysis can be estimated (ASTM E1847) |
| *s* | Standard deviation |
| $s_r$ | Within laboratory repeatability as derived from ANOVA |
| $s_s$ | Between laboratory standard deviation as derived from ANOVA |
| SS | Sum of squares in ANOVA |
| SST | Total variation in ANOVA |
| SSB | Between group (laboratory) variance |
| SSW | Within group (laboratory) variance |
| 2s | Two times standard deviation |
| SI | Standard International system of units |
| $t_{calc}$ | Calculated t statistic from a one-sample, two-tailed t-test |
| t-critical or $t_{crit}$ | t-critical value at given alpha and degrees of freedom |
| Tonne | A metric ton, is a unit of mass equaling 1000 kilograms |
| =TINV(5%,*df*) | MS Excel function for t-critical value at LOC 95% and *df* |
| *U* | Expanded uncertainty at a given k |
| *u* | Standard uncertainty at k=1 |
| $u_c$ | Combined standard uncertainty at $k$=1 |
| µm | Micron, is an SI derived unit of length equaling $1\times10^{-6}$ of a meter |

## 5. Uncertified Concentration Values

Appendix 1 gives uncertified concentrations for other elements present in the CRM.

## 6. Units

All results for major oxides are reported as oxides in percentages. All results for major elements analyses reported in percentages or ppm. Results for Au and the platinum group elements are reported in g/t or ppm. Specific gravity (SG) is the ratio of the density of a substance to the density of a reference substance, *i.e.* equivalently; it is the ratio of the mass of a substance to the mass of a reference substance for the same given volume. Since specific gravity is a ratio of densities its units are therefore dimensionless.

## 7. Analytical and Physical Methods

A complete list of analytical and physical methods as generic method codes with a brief description of the methods is available on the AMIS web site www.amis.co.za

## 8. Origin of Material

The CRM can be defined as being sourced from stockpiled and in situ material from Desert Lion Energy's Rubicon pegmatite located near Karibib in Namibia. Rubicon is a classic example of a highly zoned Li-Cs-Ta (LCT) type pegmatite.

## 9. Approximate Mineral and Chemical Composition

Major Minerals: Quartz, Albite, Lepidolite, Microcline.
Minor/Accessory Minerals: Beryl, Petalite, Apatite and Chlorite.

## 10. Quantitative Analysis by X-Ray Diffraction

Both natural and synthetic materials have a specific chemistry and atomic arrangement, known as phases. Phases can be identified and quantified using X-ray diffraction (XRD) which produces a plot of the intensity of X-rays scattered at different angles by crystalline phases in a material. Essentially, an X-ray diffraction pattern is the sum of the diffraction patterns produced by each phase. Simply put, an X-ray diffraction pattern is a fingerprint that allows the identification of what is in a target sample material. Knowledge of the mineral phase composition is useful in method development with techniques such as ICP-OES and XRF as potential matrix effects and spectral interferences can be recognised and accounted for. X-ray diffraction is effective in that it allows the identification of different phases of compounds that are identical in chemistry, but have a distinctly different the atoms, e.g. quartz, cristobalite, and glass are all different phases of $SiO_2$. Where quantitative XRD results do not correspond to results of other analytical techniques, it should be borne in mind that even though the data are quantitative they are meant to be used for indicative purposes in development of other analytical methods. Mineral names may not reflect the actual compositions of minerals identified, but rather the mineral group.

### 10.1 Sample preparation for X-Ray Diffraction

The sample was micro milled for 10 minutes, with ethanol as the grinding liquid. The resultant sample was lightly pressed into a back-packed sample holder.

## 10.2 XRD Results

Note: The XRD results are un-validated and do not account for amorphous material. These results are not certified and should be used for informational purposes only.

1. Albite was used in this Rietveld refinement. Further work would be required to better define the plagioclase mineral.

2. Microcline was used in this Rietveld refinement. Further work would be required to better define the feldspar-group mineral.

3. Mica group, where X=Ba, Ca, Cs, ($H_3O$), K, Na, ($NH_4$); Y=Al, $Cr^{3+}$, $Fe^{2+}$, $Fe^{3+}$, Li, Mg, $Mn^{2+}$, $Mn^{3+}$, $V^{3+}$ Zn; Z=Al, Be, $Fe^{3+}$, Si. It was assumed that the peak at 10.3°2Θ/10.0Å arose from muscovite. Further work would be required to confirm this.

4. Chlorite group, where A=Al, $Fe^{2+}$, $Fe^{3+}$, Li, Mg, $Mn^{2+}$, Ni, Zn; Z=Al, B, $Fe^{3+}$, Si. It was assumed that the peaks at 7.3°2Θ/14.1Å and 14.5°2Θ/7.07Å arose from chamosite. Further work would be required to confirm this.

5. It was assumed that at least some of the peak at 32.2°2Θ/3.23Å arose from rutile. Further work would be required to confirm this.

6. Tr = trace, i.e. the mineral giving rise to this peak is assumed to be present in trace amounts.

Table 5**.** Mineral species identified and quantified two sub-samples of AMIS0558 using Rietveld Refinement.

| **Mineral** | **Composition** | Weight % |
|---|---|---|
| Quartz | $SiO_2$ | 31 |
| Plagioclase[1] | (Na, Ca) Al (Al, Si)$Si_2O_8$ | 44 |
| Petalite | $LiAl(Si_4O_{10})$ | 5 |
| K feldpsar[2] | $KAlSi_3O_8$ | 7 |
| Mica group[3] | $XY_{2\text{-}3}Z_4O_{10}(OH, F)_2$ | 13 |
| Chlorite group[4] | $A_{4\text{-}6}Z_4O_{10}(OH, O)_8$ | <1 |
| Rutile[5] | $TiO_2$ | <1 |
| Unassigned peak[6] | 12.9°2Θ/7.95Å | Tr |
| Total | | 100 |

## 11. Health and Safety

The material is a very fine powder coloured Very Light Grey (5Y 8/1). Safety precautions for handling fine particulate matter are recommended, such as the use of safety glasses, breathing protection, gloves and a laboratory coat.

## 12. Method of Preparation

The particle size distribution for this material was shown to have a nominal top size of 54µm (at least 95% passing 54µm). The procedure of preparation in brief is as follows: the material was crushed, dry-milled and air-classified to <54µm. It was then blended in a bi-conical mixer, systematically divided and sealed into 1kg Laboratory Packs. Explorer Packs are then subdivided from the Laboratory Packs as required. Final packaged units were then selected on a random basis and submitted for analysis to an independent laboratory accredited with the ISO17025:2005 standard of general requirements for the competence of testing and calibration laboratories. The results obtained from this laboratory are then evaluated statistically by AMIS for homogeneity.

## 13. Particle Size Determination

The particle size was determined by laser diffraction. In this method, particles are passed through a focused laser beam that scatter light at an angle inversely proportional to their size. The intensity of light is measured and converted to a volume in particle size distribution. The results for this standard are presented in Table 6.

**Table 6.** Particle Size Determination by laser diffraction.

| Size | Vol Under % | Size | Vol Under % | Size | Vol Under % | Size | Vol Under % | Size | Vol Under % |
|---|---|---|---|---|---|---|---|---|---|
| 0.10 | 0.00 | 8.00 | 47.81 | 50.00 | 94.55 | 106.00 | 99.28 | 200.00 | 99.99 |
| 0.25 | 0.00 | 9.00 | 51.01 | 55.00 | 95.61 | 110.00 | 99.36 | 225.00 | 100.00 |
| 0.50 | 1.44 | 10.00 | 53.97 | 60.00 | 96.42 | 115.00 | 99.45 | 250.00 | 100.00 |
| 0.60 | 2.47 | 12.00 | 59.32 | 65.00 | 97.06 | 120.00 | 99.53 | 300.00 | 100.00 |
| 0.75 | 4.16 | 15.00 | 66.10 | 70.00 | 97.57 | 125.00 | 99.60 | 400.00 | 100.00 |
| 1.00 | 7.09 | 18.00 | 71.66 | 75.00 | 97.98 | 130.00 | 99.65 | 500.00 | 100.00 |
| 2.00 | 17.71 | 20.00 | 74.80 | 80.00 | 98.31 | 135.00 | 99.70 | 600.00 | 100.00 |
| 3.00 | 25.53 | 25.00 | 81.07 | 85.00 | 98.57 | 140.00 | 99.74 | 700.00 | 100.00 |
| 4.00 | 31.46 | 30.00 | 85.61 | 90.00 | 98.79 | 150.00 | 99.81 | 800.00 | 100.00 |
| 5.00 | 36.33 | 35.00 | 88.92 | 95.00 | 98.98 | 160.00 | 99.88 | 900.00 | 100.00 |
| 6.00 | 40.55 | 40.00 | 91.36 | 100.00 | 99.13 | 170.00 | 99.91 | 1000.00 | 100.00 |
| 7.00 | 44.34 | 45.00 | 93.18 | 105.00 | 99.25 | 175.00 | 99.92 | 1100.00 | 100.00 |

## 14. Handling

The material is packaged in Laboratory Packs and Explorer Packs that must be shaken or otherwise agitated before use. The analyte concentrations are quoted on a dry basis; therefore the user needs to determine the moisture content in order to convert any obtained assay values to an air-dry basis (see Appendix 5 for an example calculation).

## 15. Storage information

The material should be stored in a cool dry place, in such a way that it does not compromise the integrity of the CRM. The material should be stored in conditions which will ensure it does not absorb moisture.

## 16. Methods of Analysis Requested

The following methods were requested of the participating laboratories for the development of this CRM:

- Multi element scan to include all elements especially Li, Be, Cs, Ga, Fe, Nb, Rb, Sn and Ta - 4-acid total digestion including HF and/or peroxide fusion finished with either ICP-OES or ICP-MS or AAS
- LOI and all major oxides excluding $U_3O_8$ with XRF finish. (Please specify the temperature for LOI)
- SG – gas pycnometer
- S and C Combustion/LECO

## 17. Information Requested of Participating Laboratories

The following information was requested of the participating laboratories for the development of this CRM:

- State aliquots used for all determinations.
- All results for major elements to be reported as oxides in percentages.
- All results for multi-element scans and fusion to be reported in ppm.

- Report all QC data, to include replicates, blanks and certified reference materials used.
- All Round robin samples must be treated the same as routine test samples.
- All results must be reported to maximum decimal places i.e. dependent on laboratories capabilities.
- Please ensure moisture content is determined and calculated. All results should be corrected by the moisture correction factor and this factor should be stated in the laboratory results.
- Please use the excel template provided by AMIS. If you require a copy, please email any of the email addresses below and ensure all uncertainties are added to the results.
- Please send PDF of all results.
- Ensure correct PPE is used i.e. gloves, dust masks and protective clothing.
- Analysis should be done under controlled environmental conditions.

## 18. Certification of Mean and Estimation of Measurement Uncertainty

The samples used in this certification process have been selected in such a way as to represent the entire batch of material and were taken from the final packaged units; therefore, all sources of uncertainty are included in the combined standard uncertainty determination. Initially the data submitted by all of the laboratories are subjected to a z-score test, equation [1] to exclude outliers and the remaining data sets examined for their normality in distribution. This is followed by the exclusion of further outliers as defined by the IUPAC Harmonised Protocol of 1995 in which both Cochran and a Grubbs test are applied until all outliers are identified, equations [2] and [3]. A grand mean and standard deviation is re-calculated using all remaining data (Thompson, 2008; Carr, 2011) (see Appendix 2). These data are then subjected to an analysis of variance (ANOVA) as per equations, [9],[10],[11],[12] and [13] in Appendix 2. The mean squares for data within and between laboratories derived from ANOVA are used to compute the within-laboratory reproducibility, or combined standard uncertainty as shown in Appendix 2, equation [14], [15] and [16]. The Horwitz function is applied to assess the performance of the data under consideration with respect to precision as relative standard deviation (equations [4], [5] and [6]). Should the ratio of the observed %RSD and a calculated %RSD be <2, the observed %RSD is accepted (Horwitz & Albert, 2006). A final certified value is then found by calculating a grand mean of equally weighted individual laboratory means [7] (ISO Guide 35, 2003; Barwick & Pritchard, 2011).

An uncertainty statement on AMIS certificates is typically presented as:

The expanded uncertainty (U) is determined by multiplication of the combined uncertainty (uc) with a coverage factor (k) found from N-1 degrees of freedom and a t-critical value at a level of confidence of 95% (EURACHEM / CITAC Guide CG 4., (2012), (see Table 12, Appendix 7 for a t-critical table). N is the number of laboratory means used in the establishment of the certified value. Since the estimated values of the CRM approximate a normal distribution with combined uncertainty, uc, the certified value of the CRM is understood to lie in the interval defined by U with a level of confidence of 95 % (Thompson & Lowthian, 2011).

Appendix 2 gives detail on the principles used for certification of the reported assay values and estimation of measurement uncertainty.

## 19. Participating Laboratories

Sixteen laboratories were each given eight randomly selected packages of the CRM. Thirteen laboratories of the Sixteen laboratories submitted results in time for certification.

The 13 laboratories that provided results timeously are:

1. ALS Chemex Laboratory Group Johannesburg SA
2. Antech Zimbabwe
3. Bureau Veritas Minerals Ultra Trace Pty Ltd
4. "Dorfner Anzaplan Analysenzentrum und Anlagenplanungsgesellschaft mbH"
5. Intertek Perth
6. JAEC_CPAL
7. Koza Altin Isletmeleri
8. Set Point Laboratories (Isando) SA
9. SGS Mineral Services Lakefield (Canada)
10. SGS South Africa
11. Shiva Analyticals India
12. UIS Analytical Services (pty) Ltd
13. Université du Québec en Abitibi-Témiscamingue (UQAT)

## 20. Accepted Assay Data

Data from the 13 laboratories used for certification are set out in Table 7.

**Table 7.** Data used to calculate the certified values after removal of outliers.

| 4A_MICP Ag ppm | FUS Al ppm | XRF $Al_2O_3$ % | FUS $Al_2O_3$ % | 4A_MICP As ppm | 4A_MICP Ba ppm | 4A_MICP Be ppm | 4A_MICP Bi ppm | Combustion/LECO C ppm | 4A_MICP Ca ppm | XRF CaO % | 4A_MICP Co ppm | 4A_MICP Cs ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.50 | 77155 | 14.72 | 14.56 | 6.00 | 35.00 | 416 | 34.20 | 5380 | 17300 | 2.37 | 1.60 | 611 |
| 0.50 | 77444 | 14.72 | 14.61 | 6.00 | 34.00 | 424 | 35.50 | 5280 | 17400 | 2.36 | 2.00 | 620 |
| 0.50 | 77254 | 14.70 | 14.58 | 6.00 | 34.00 | 425 | 36.10 | 5310 | 17200 | 2.36 | 1.70 | 618 |
| 0.50 | 76981 | 14.69 | 14.52 | 6.00 | 35.00 | 417 | 35.70 | 5360 | 17300 | 2.36 | 1.70 | 620 |
| 0.50 | 77498 | 14.69 | 14.62 | 6.00 | 34.00 | 417 | 34.50 | 5320 | 17300 | 2.36 | 1.60 | 617 |
| 0.40 | 77136 | 14.76 | 14.55 | 6.00 | 33.00 | 417 | 34.60 | 5350 | 17200 | 2.35 | 1.80 | 611 |
| 0.40 | 77326 | 14.70 | 14.59 | 6.00 | 34.00 | 410 | 36.60 | 5430 | 17300 | 2.37 | 1.60 | 615 |
| 0.40 | 76748 | 14.70 | 14.48 | 7.00 | 33.00 | 413 | 36.60 | 5330 | 17400 | 2.36 | 1.80 | 610 |
| 0.40 | 75600 | 14.80 | 14.28 | 6.95 | 34.18 | 271 | 31.63 | 5100 | 16600 | 2.30 | 2.00 | 695 |
| 0.49 | 74300 | 14.90 | 13.94 | 6.41 | 37.19 | 284 | 30.90 | 5200 | 16600 | 2.30 | 2.30 | 771 |
| 0.59 | 74500 | 14.90 | 14.04 | 6.97 | 34.19 | 271 | 31.91 | 5100 | 16300 | 2.30 | 1.80 | 779 |
| 0.55 | 75300 | 14.80 | 14.08 | 7.06 | 29.69 | 261 | 30.37 | 5200 | 16500 | 2.30 | 1.80 | 774 |
| 0.49 | 76800 | 14.80 | 14.23 | 7.27 | 37.01 | 294 | 30.61 | 5300 | 16200 | 2.31 | 1.90 | 669 |
| 0.57 | 76600 | 14.90 | 14.51 | 7.25 | 31.74 | 350 | 30.87 | 5200 | 16200 | 2.30 | 1.90 | 666 |
| 0.50 | 76700 | 14.80 | 14.47 | 5.24 | 32.01 | 374 | 30.91 | 5100 | 16400 | 2.31 | 1.90 | 666 |
| 0.52 | 76850 | 14.90 | 14.49 | 6.73 | 33.78 | 365 | 32.24 | 5500 | 16800 | 2.31 | 2.00 | 670 |
| 0.55 | 74730 | 14.70 | 14.50 | 5.00 | 30.00 | 365 | 35.40 | 5640 | 17100 | 2.35 | 1.70 | 662 |
| 0.55 | 75790 | 14.60 | 14.10 | 6.00 | 30.00 | 379 | 42.40 | 5620 | 16900 | 2.34 | 1.70 | 666 |
| 0.49 | 77380 | 14.60 | 14.30 | 6.00 | 30.00 | 383 | 38.80 | 5630 | 16900 | 2.34 | 1.70 | 670 |
| 0.55 | 77380 | 14.70 | 14.60 | 8.00 | 30.00 | 384 | 41.70 | 5640 | 16900 | 2.36 | 1.80 | 663 |
| 0.40 | 75790 | 14.70 | 14.60 | 6.00 | 30.00 | 358 | 42.60 | 5640 | 16800 | 2.37 | 1.80 | 580 |
| 0.41 | 74730 | 14.70 | 14.30 | 5.00 | 30.00 | 421 | 41.50 | 5660 | 17000 | 2.36 | 1.70 | 593 |
| 0.45 |  | 14.60 | 14.10 | 6.00 | 30.00 | 420 | 40.10 | 5610 | 17000 | 2.33 | 1.80 | 566 |
| 0.53 |  | 14.70 |  | 6.00 | 30.00 | 422 | 38.80 | 5650 | 18000 | 2.35 | 1.70 | 598 |
| 0.49 |  | 14.28 |  | 5.40 | 35.91 | 419 | 34.20 | 5400 | 18000 | 2.30 | 1.70 | 579 |
| 0.60 |  | 14.42 |  | 5.80 | 37.75 | 422 | 36.60 | 5400 | 17700 | 2.33 | 1.70 | 580 |
| 0.60 |  | 14.24 |  | 7.70 | 36.76 | 419 | 35.50 | 5400 | 17900 | 2.28 | 1.70 | 566 |
| 0.50 |  | 14.30 |  | 5.50 | 36.77 | 421 | 36.20 | 5400 | 18000 | 2.30 | 1.80 | 579 |
| 0.60 |  | 14.32 |  | 6.50 | 36.98 | 422 | 38.60 | 5400 | 17500 | 2.30 | 1.90 | 600 |
| 0.60 |  | 14.24 |  | 5.50 | 37.37 | 296 | 36.60 | 5400 | 17500 | 2.31 | 1.80 | 594 |
| 0.60 |  | 14.65 |  | 6.80 | 38.29 | 327 | 38.50 | 5400 | 17400 | 2.26 | 1.80 | 590 |
| 0.60 |  | 14.89 |  | 5.80 | 37.07 | 339 | 32.60 | 5400 | 17834 | 2.29 | 1.70 | 616 |
| 0.60 |  | 14.83 |  | 5.00 | 33.00 | 336 | 41.97 |  | 17719 | 2.43 |  | 622 |
|  |  | 14.90 |  | 5.00 | 35.00 | 338 | 41.69 |  | 17656 | 2.44 |  | 568 |
|  |  | 14.78 |  | 6.00 | 34.00 | 357 | 40.09 |  | 17360 | 2.42 |  | 593 |
|  |  | 14.81 |  | 5.00 | 35.00 | 334 | 41.36 |  | 18165 | 2.46 |  | 562 |
|  |  | 14.86 |  | 5.00 | 35.00 | 367 | 41.82 |  | 17798 | 2.47 |  | 643 |
|  |  | 14.84 |  | 5.00 | 34.00 | 333 | 41.39 |  | 17724 | 2.44 |  | 643 |
|  |  | 14.52 |  | 5.00 | 35.00 | 306 | 41.11 |  | 17109 | 2.44 |  | 639 |
|  |  | 14.42 |  | 6.85 | 35.00 | 322 | 41.93 |  | 15938 | 2.44 |  | 648 |
|  |  | 14.42 |  | 7.53 | 28.60 | 328 | 35.70 |  | 16959 | 2.34 |  | 658 |
|  |  | 14.32 |  | 7.85 | 28.60 | 465 | 36.90 |  | 16536 | 2.33 |  | 653 |
|  |  | 14.43 |  | 7.15 | 36.30 | 352 | 34.90 |  | 17190 | 2.33 |  | 637 |
|  |  | 14.45 |  | 7.89 | 32.10 | 363 | 36.50 |  | 17458 | 2.33 |  | 654 |
|  |  | 14.37 |  | 6.69 | 30.90 | 342 | 34.70 |  | 16955 | 2.36 |  |  |
|  |  | 14.43 |  | 7.07 | 27.80 | 338 | 35.90 |  | 16617 | 2.32 |  |  |
|  |  | 14.60 |  | 6.80 | 33.00 | 358 | 36.80 |  | 17000 | 2.34 |  |  |
|  |  | 14.70 |  | 7.90 | 34.00 | 359 | 34.80 |  | 17274 | 2.33 |  |  |
|  |  | 14.50 |  | 7.98 | 33.00 | 358 | 31.90 |  | 17500 | 2.46 |  |  |
|  |  | 14.80 |  | 6.93 | 34.00 | 350 | 30.30 |  | 16860 | 2.46 |  |  |
|  |  | 14.60 |  | 7.97 | 34.00 | 353 | 39.20 |  | 17276 | 2.46 |  |  |
|  |  | 14.60 |  | 7.64 | 34.00 | 358 | 37.50 |  | 17376 | 2.44 |  |  |
|  |  | 14.60 |  | 7.34 | 33.00 | 352 | 36.10 |  | 17386 | 2.44 |  |  |
|  |  | 14.70 |  | 6.73 | 34.00 | 393 | 31.60 |  | 17938 | 2.44 |  |  |
|  |  | 14.61 |  | 7.00 | 32.17 | 392 | 38.29 |  | 17488 | 2.47 |  |  |
|  |  | 14.61 |  | 7.00 | 32.30 | 399 | 37.74 |  | 18318 | 2.47 |  |  |
|  |  | 14.60 |  | 7.00 | 33.32 | 404 | 37.48 |  | 17679 | 2.37 |  |  |
|  |  | 14.61 |  | 7.00 | 31.07 | 404 | 37.48 |  | 17842 | 2.37 |  |  |
|  |  | 14.60 |  | 6.00 | 33.88 | 399 | 37.68 |  | 17710 | 2.36 |  |  |
|  |  | 14.61 |  | 7.00 | 31.55 | 401 | 38.99 |  | 17687 | 2.36 |  |  |
|  |  | 14.61 |  | 7.00 | 33.10 | 398 | 37.78 |  | 17810 | 2.36 |  |  |
|  |  | 14.59 |  | 6.00 | 33.97 |  | 39.47 |  |  | 2.37 |  |  |
|  |  | 14.40 |  |  |  |  |  |  |  | 2.37 |  |  |
|  |  | 14.23 |  |  |  |  |  |  |  | 2.36 |  |  |
|  |  | 14.40 |  |  |  |  |  |  |  | 2.53 |  |  |
|  |  | 14.46 |  |  |  |  |  |  |  | 2.54 |  |  |
|  |  | 14.35 |  |  |  |  |  |  |  | 2.51 |  |  |
|  |  | 14.52 |  |  |  |  |  |  |  | 2.50 |  |  |
|  |  | 14.44 |  |  |  |  |  |  |  |  |  |  |
|  |  | 14.42 |  |  |  |  |  |  |  |  |  |  |

## Assay Data (Cont.)

| 4A_MICP Cu ppm | 4A_MICP Dy ppm | 4A_MICP Er ppm | FUS Fe ppm | 4A_MICP Fe ppm | XRF $Fe_2O_3$ % | 4A_MICP Ga ppm | 4A_MICP Gd ppm | 4A_MICP Hf ppm | 4A_MICP K ppm | XRF $K_2O$ % | XRF $K_2O$ % | 4A_MICP La ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 10.00 | 0.45 | 0.15 | 4040 | 4700 | 0.66 | 42.60 | 0.40 | 6.60 | 29300 | 3.55 | 3.57 | 1.30 |
| 15.00 | 0.40 | 0.10 | 4470 | 4800 | 0.66 | 44.20 | 0.20 | 6.80 | 29700 | 3.55 | 3.60 | 1.20 |
| 10.00 | 0.40 | 0.10 | 4320 | 4800 | 0.66 | 43.40 | 0.20 | 6.80 | 29700 | 3.54 | 3.62 | 1.20 |
| 10.00 | 0.40 | 0.15 | 4280 | 4800 | 0.66 | 43.40 | 0.20 | 6.60 | 29700 | 3.55 | 3.61 | 1.30 |
| 10.00 | 0.40 | 0.10 | 4260 | 4700 | 0.65 | 43.80 | 0.20 | 6.40 | 29900 | 3.55 | 3.60 | 1.30 |
| 10.00 | 0.40 | 0.10 | 4200 | 4700 | 0.66 | 43.40 | 0.40 | 6.80 | 29400 | 3.54 | 3.60 | 1.20 |
| 10.00 | 0.40 | 0.10 | 4300 | 4700 | 0.66 | 43.40 | 0.20 | 6.40 | 29500 | 3.55 | 3.60 | 1.20 |
| 10.00 | 0.40 | 0.10 | 4370 | 4700 | 0.66 | 43.40 | 0.20 | 6.60 | 29200 | 3.54 | 3.57 | 1.20 |
| 10.76 | 0.32 | 0.09 | 5153 | 4500 | 0.66 | 42.70 | 0.27 | 5.70 | 28100 | 3.66 | 3.59 | 1.70 |
| 10.69 | 0.38 | 0.15 | 4875 | 4300 | 0.65 | 43.30 | 0.36 | 6.15 | 28200 | 3.64 | 3.56 | 1.60 |
| 10.86 | 0.37 | 0.12 | 5204 | 4200 | 0.67 | 44.20 | 0.34 | 6.66 | 28200 | 3.64 | 3.73 | 1.70 |
| 10.70 | 0.38 | 0.13 | 5138 | 4000 | 0.65 | 43.20 | 0.31 | 6.14 | 27900 | 3.65 | 3.68 | 1.50 |
| 10.68 | 0.35 | 0.11 | 4968 | 4500 | 0.66 | 41.70 | 0.28 | 5.84 | 28000 | 3.65 | 3.64 | 1.60 |
| 10.50 | 0.34 | 0.12 | 5058 | 4200 | 0.67 | 45.10 | 0.31 | 5.63 | 28100 | 3.65 | 3.69 | 1.70 |
| 10.60 | 0.40 | 0.13 | 5161 | 4100 | 0.66 | 43.90 | 0.29 | 5.92 | 28100 | 3.65 | 3.71 | 1.50 |
| 10.70 | 0.33 | 0.12 | 5019 | 4200 | 0.65 | 42.30 | 0.37 | 5.90 | 28300 | 3.67 | 3.72 | 1.30 |
| 13.20 | 0.47 | 0.16 | 4500 | 4100 | 0.63 | 46.90 | 0.43 | 5.40 | 28500 | 3.59 |  | 1.60 |
| 11.90 | 0.47 | 0.16 | 4400 | 4100 | 0.63 | 44.70 | 0.40 | 5.90 | 29000 | 3.57 |  | 1.80 |
| 11.20 | 0.49 | 0.17 | 4400 | 4100 | 0.63 | 44.20 | 0.46 | 5.60 | 28800 | 3.58 |  | 2.00 |
| 10.50 | 0.52 | 0.17 | 4500 | 4000 | 0.65 | 48.60 | 0.44 | 5.60 | 28800 | 3.59 |  | 1.90 |
| 11.40 | 0.46 | 0.17 | 4500 | 4100 | 0.63 | 47.40 | 0.46 | 6.20 | 28800 | 3.58 |  | 1.66 |
| 11.30 | 0.48 | 0.17 | 4400 | 4100 | 0.66 | 47.20 | 0.43 | 5.90 | 28500 | 3.58 |  | 1.67 |
| 11.30 | 0.50 | 0.17 | 4400 | 4000 | 0.64 | 43.30 | 0.45 | 6.10 | 29100 | 3.57 |  | 1.67 |
| 11.10 | 0.51 | 0.16 | 4400 | 4100 | 0.64 | 47.90 | 0.42 | 5.40 | 28900 | 3.60 |  | 1.69 |
| 10.60 |  |  | 4500 | 4300 | 0.63 | 47.35 |  | 7.39 | 29600 | 3.46 |  | 1.70 |
| 11.00 |  |  | 4580 | 4300 | 0.63 | 47.43 |  | 7.39 | 29700 | 3.50 |  | 1.70 |
| 10.10 |  |  | 4530 | 4200 | 0.62 | 47.76 |  | 7.38 | 29300 | 3.42 |  | 1.66 |
| 11.20 |  |  | 4670 | 4200 | 0.63 | 48.16 |  | 7.39 | 30000 | 3.44 |  | 1.64 |
| 10.90 |  |  | 4480 | 4300 | 0.62 | 48.18 |  | 7.16 | 30100 | 3.46 |  | 1.30 |
| 9.90 |  |  | 4400 | 4200 | 0.62 | 48.05 |  | 7.54 | 27900 | 3.47 |  | 1.30 |
| 10.30 |  |  | 4600 | 4200 | 0.62 | 47.65 |  | 7.34 | 29400 | 3.42 |  | 1.30 |
| 10.90 |  |  | 4610 | 4200 | 0.70 | 43.30 |  | 7.29 | 29200 | 3.44 |  | 1.40 |
| 10.90 |  |  |  | 4607 | 0.71 | 42.40 |  | 5.73 | 28887 | 3.54 |  | 1.40 |
| 11.10 |  |  |  | 4755 | 0.70 | 42.00 |  | 5.71 | 28439 | 3.46 |  | 1.30 |
| 10.60 |  |  |  | 4550 | 0.67 | 44.70 |  | 5.96 | 28456 | 3.52 |  | 1.30 |
| 11.60 |  |  |  | 4397 | 0.71 | 43.20 |  | 5.92 | 28066 | 3.42 |  | 1.30 |
| 10.30 |  |  |  | 4583 | 0.70 | 42.80 |  | 5.67 | 28791 | 3.50 |  | 1.15 |
| 10.50 |  |  |  | 4588 | 0.66 | 44.50 |  | 5.73 | 28902 | 3.50 |  | 1.12 |
| 10.50 |  |  |  | 4644 | 0.66 | 43.40 |  | 5.98 | 29115 | 3.48 |  | 1.06 |
| 10.80 |  |  |  | 4635 | 0.66 | 40.80 |  | 5.86 | 27644 | 3.56 |  | 1.05 |
|  |  |  |  | 4400 | 0.65 | 39.60 |  | 4.55 | 28307 | 3.50 |  | 1.47 |
|  |  |  |  | 4400 | 0.66 | 38.80 |  | 5.43 | 28684 | 3.52 |  | 1.27 |
|  |  |  |  | 4500 | 0.65 | 42.90 |  | 5.14 | 29187 | 3.53 |  | 1.24 |
|  |  |  |  | 4500 | 0.67 | 43.10 |  | 4.85 | 29624 | 3.52 |  | 1.13 |
|  |  |  |  | 4300 | 0.66 | 39.60 |  | 4.43 | 28688 | 3.51 |  |  |
|  |  |  |  | 4500 | 0.66 | 41.90 |  | 6.50 | 29330 | 3.52 |  |  |
|  |  |  |  | 4500 | 0.65 | 42.20 |  | 6.20 | 29452 | 3.51 |  |  |
|  |  |  |  | 4400 | 0.66 | 41.20 |  | 6.00 | 29362 | 3.57 |  |  |
|  |  |  |  | 4707 | 0.66 | 43.70 |  | 6.10 | 28399 | 3.58 |  |  |
|  |  |  |  | 4660 | 0.66 | 43.60 |  | 6.10 | 28257 | 3.58 |  |  |
|  |  |  |  | 4730 | 0.67 | 43.20 |  | 6.20 | 29739 | 3.58 |  |  |
|  |  |  |  | 4878 | 0.65 | 43.00 |  | 6.10 | 28033 | 3.55 |  |  |
|  |  |  |  | 4747 | 0.66 | 42.70 |  | 6.10 | 30371 | 3.56 |  |  |
|  |  |  |  | 4708 | 0.68 |  |  |  |  |  |  |  |
|  |  |  |  | 4754 | 0.69 |  |  |  |  |  |  |  |
|  |  |  |  | 4877 | 0.69 |  |  |  |  |  |  |  |
|  |  |  |  |  | 0.69 |  |  |  |  |  |  |  |
|  |  |  |  |  | 0.70 |  |  |  |  |  |  |  |
|  |  |  |  |  | 0.70 |  |  |  |  |  |  |  |
|  |  |  |  |  | 0.69 |  |  |  |  |  |  |  |
|  |  |  |  |  | 0.69 |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |

## Assay Data (Cont.)

| FUS Li ppm | 4A_MICP Li ppm | LOI LOI % | LOI LOI % | FUS Mg ppm | XRF MgO % | FUS MgO % | 4A_MICP Mn ppm | XRF MnO % | 4A_MICP Mo ppm | 4A_MICP Na ppm | XRF $Na_2O$ % | 4A_MICP Nb ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5390 | 5330 | 3.53 | 4.01 | 2741 | 0.44 | 0.46 | 1550 | 0.21 | 0.90 | 15800 | 2.14 | 44.50 |
| 5390 | 5480 | 3.51 | 3.89 | 2723 | 0.45 | 0.45 | 1560 | 0.21 | 0.80 | 16000 | 2.15 | 44.50 |
| 5440 | 5310 | 3.48 | 4.06 | 2732 | 0.44 | 0.46 | 1560 | 0.21 | 0.76 | 16000 | 2.15 | 44.50 |
| 5510 | 5440 | 3.50 | 4.05 | 2741 | 0.45 | 0.46 | 1560 | 0.21 | 0.76 | 16000 | 2.15 | 46.50 |
| 5430 | 5330 | 3.52 | 3.96 | 2805 | 0.44 | 0.47 | 1580 | 0.21 | 0.68 | 16100 | 2.14 | 47.00 |
| 5520 | 5380 | 3.50 | 3.91 | 2790 | 0.45 | 0.46 | 1560 | 0.21 | 0.85 | 15800 | 2.15 | 46.00 |
| 5440 | 5340 | 3.53 | 3.39 | 2796 | 0.45 | 0.47 | 1550 | 0.21 | 0.75 | 15700 | 2.14 | 45.00 |
| 5420 | 5370 | 3.48 | 3.39 | 2500 | 0.44 | 0.41 | 1540 | 0.21 | 0.70 | 15600 | 2.14 | 46.00 |
| 4660 | 5047 | 3.36 | 3.39 | 2500 | 0.43 | 0.41 | 1523 | 0.20 | 0.87 | 14300 | 2.12 | 53.80 |
| 5390 | 5068 | 3.34 | 3.37 | 2500 | 0.44 | 0.41 | 1513 | 0.20 | 0.76 | 14400 | 2.16 | 57.80 |
| 5230 | 5078 | 3.28 | 3.31 | 2500 | 0.43 | 0.41 | 1501 | 0.21 | 0.78 | 14400 | 2.17 | 56.80 |
| 5080 | 4980 | 3.35 | 3.40 | 2500 | 0.43 | 0.41 | 1427 | 0.20 | 0.81 | 14500 | 2.16 | 57.20 |
| 5130 | 5040 | 3.31 | 3.45 | 2500 | 0.46 | 0.41 | 1521 | 0.20 | 0.83 | 14300 | 2.18 | 58.10 |
| 4970 | 5042 | 3.34 | 3.37 | 2500 | 0.43 | 0.41 | 1471 | 0.20 | 0.78 | 14400 | 2.15 | 59.30 |
| 4930 | 5068 | 3.27 | 3.80 | 2500 | 0.43 | 0.41 | 1492 | 0.20 | 0.70 | 14400 | 2.12 | 56.50 |
| 5130 | 5069 | 3.33 | 3.77 | 2538 | 0.44 | 0.42 | 1509 | 0.20 | 0.75 | 15600 | 2.17 | 65.60 |
| 5400 | 5610 | 3.80 | 3.69 | 2502 | 0.44 | 0.42 | 1560 | 0.21 | 0.75 | 15600 | 2.15 | 55.20 |
| 5750 | 5600 | 3.82 | 3.70 | 2520 | 0.43 | 0.42 | 1580 | 0.20 | 0.75 | 15700 | 2.17 | 61.00 |
| 5660 | 5630 | 3.78 | 3.66 | 2634 | 0.43 | 0.44 | 1570 | 0.20 | 0.78 | 15200 | 2.12 | 59.10 |
| 5850 | 5510 | 3.81 | 3.76 | 2442 | 0.44 | 0.41 | 1580 | 0.20 | 0.76 | 15600 | 2.13 | 59.90 |
| 5830 | 5590 | 3.85 | 3.70 | 2616 | 0.45 | 0.44 | 1580 | 0.21 | 0.77 | 15500 | 2.12 | 62.00 |
| 5790 | 5560 | 3.82 | 3.69 | 2514 | 0.45 | 0.42 | 1560 | 0.21 | 0.77 | 15600 | 2.14 | 61.60 |
| 5510 | 5570 | 3.83 |  | 2532 | 0.44 | 0.42 | 1580 | 0.20 | 0.88 | 15800 | 2.11 | 60.60 |
| 5490 | 5630 | 3.81 |  |  | 0.44 |  | 1580 | 0.20 | 0.71 | 15000 | 2.15 | 56.30 |
| 4860 | 5206 | 3.91 |  |  | 0.43 |  | 1631 | 0.21 | 0.72 | 15500 | 2.14 | 58.10 |
| 5206 | 5378 | 3.92 |  |  | 0.43 |  | 1626 | 0.21 | 0.69 | 15100 | 2.09 | 58.50 |
| 4737 | 5197 | 3.91 |  |  | 0.42 |  | 1591 | 0.20 | 0.71 | 15400 | 2.12 | 59.50 |
| 5216 | 5380 | 3.96 |  |  | 0.42 |  | 1614 | 0.20 | 0.98 | 15600 | 2.09 | 58.00 |
| 5139 | 5417 | 3.95 |  |  | 0.43 |  | 1635 | 0.21 | 0.76 | 14700 | 2.12 | 58.40 |
| 5376 | 5399 | 3.93 |  |  | 0.43 |  | 1623 | 0.21 | 0.78 | 14800 | 2.08 | 58.80 |
| 5231 | 5530 | 3.90 |  |  | 0.41 |  | 1607 | 0.21 | 0.79 | 14600 | 2.13 | 57.90 |
| 4832 | 5470 | 3.90 |  |  | 0.42 |  | 1590 | 0.21 | 0.90 | 15935 | 2.15 | 58.40 |
| 5579 | 5462 | 3.66 |  |  | 0.41 |  | 1573 | 0.21 | 0.80 | 15797 | 2.07 | 58.30 |
| 5523 | 5573 | 3.65 |  |  | 0.42 |  | 1567 | 0.21 | 0.80 | 15761 | 2.13 | 42.20 |
| 5509 | 5520 | 3.58 |  |  | 0.42 |  | 1534 | 0.21 | 0.80 | 15568 | 2.10 | 51.50 |
| 5558 | 5754 | 3.60 |  |  | 0.42 |  | 1623 | 0.21 | 0.80 | 16554 | 2.13 | 52.10 |
| 5643 | 6068 | 3.59 |  |  | 0.41 |  | 1472 | 0.22 | 0.70 | 15762 | 2.07 | 57.40 |
| 5423 | 5747 | 3.62 |  |  | 0.42 |  | 1544 | 0.22 | 0.80 | 15965 | 2.14 | 48.80 |
| 5385 | 5641 | 3.62 |  |  | 0.41 |  | 1579 | 0.22 | 0.80 | 15216 | 2.08 | 46.00 |
| 5366 | 5596 | 3.61 |  |  | 0.42 |  | 1325 | 0.22 |  | 15509 | 2.08 | 54.20 |
|  | 5412 | 3.74 |  |  | 0.47 |  | 1421 | 0.22 |  | 15673 | 2.15 | 53.60 |
|  | 5632 | 3.69 |  |  | 0.47 |  | 1342 | 0.21 |  | 15936 | 2.16 | 52.70 |
|  | 5601 | 3.74 |  |  | 0.47 |  | 1357 | 0.21 |  | 16072 | 2.15 | 55.50 |
|  | 5716 | 3.73 |  |  | 0.45 |  | 1368 | 0.21 |  | 15652 | 2.16 | 55.10 |
|  | 5580 | 3.74 |  |  | 0.44 |  | 1306 | 0.21 |  | 15962 | 2.16 | 54.70 |
|  | 5682 | 3.76 |  |  | 0.45 |  | 1291 | 0.21 |  | 15877 | 2.15 | 54.40 |
|  | 5554 | 3.73 |  |  | 0.44 |  | 1393 | 0.21 |  | 16170 | 2.16 | 54.90 |
|  | 5661 | 3.73 |  |  | 0.45 |  | 1580 | 0.21 |  | 16228 | 2.15 |  |
|  | 5253 | 3.58 |  |  | 0.45 |  | 1614 | 0.21 |  | 16252 |  |  |
|  | 5272 | 3.73 |  |  | 0.44 |  | 1644 | 0.21 |  | 16580 |  |  |
|  | 5365 | 3.63 |  |  | 0.45 |  | 1648 | 0.21 |  | 15559 |  |  |
|  | 5298 | 3.75 |  |  | 0.45 |  | 1601 | 0.21 |  | 16025 |  |  |
|  | 5374 | 3.81 |  |  | 0.45 |  | 1628 | 0.21 |  | 15670 |  |  |
|  | 5262 | 3.58 |  |  | 0.45 |  | 1628 | 0.21 |  | 16122 |  |  |
|  | 5338 | 3.59 |  |  | 0.44 |  | 1655 | 0.21 |  | 16393 |  |  |
|  | 5362 | 3.57 |  |  | 0.41 |  | 1287 | 0.21 |  |  |  |  |
|  |  |  |  |  | 0.41 |  | 1280 | 0.21 |  |  |  |  |
|  |  |  |  |  | 0.40 |  | 1367 |  |  |  |  |  |
|  |  |  |  |  | 0.40 |  | 1345 |  |  |  |  |  |
|  |  |  |  |  | 0.39 |  | 1255 |  |  |  |  |  |
|  |  |  |  |  | 0.39 |  | 1255 |  |  |  |  |  |
|  |  |  |  |  | 0.40 |  | 1249 |  |  |  |  |  |

## Assay Data (Cont.)

| 4A_MICP | 4A_MICP | 4A_MICP | XRF | XRF | 4A_MICP | 4A_MICP | FUS | 4A_MICP | 4A_MICP | SG | XRF | 4A_MICP |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Nd | Ni | P | $P_2O_5$ | $P_2O_5$ | Pb | Pr | Rb | Sb | Sc | SG | $SiO_2$ | Sm |
| ppm | ppm | ppm | % | % | ppm | ppm | ppm | ppm | ppm | Dimensionless | % | ppm |
| 1.10 | 5.00 | 2000 | 0.45 | 0.46 | 33.00 | 0.30 | 3970 | 3.20 | 0.80 | 2.73 | 69.91 | 0.25 |
| 1.05 | 5.00 | 2000 | 0.45 | 0.47 | 31.00 | 0.28 | 4180 | 3.20 | 0.80 | 2.75 | 69.88 | 0.25 |
| 1.10 | 5.00 | 2000 | 0.45 | 0.46 | 32.00 | 0.28 | 4100 | 3.00 | 0.90 | 2.73 | 69.86 | 0.25 |
| 1.10 | 5.00 | 2050 | 0.45 | 0.46 | 33.00 | 0.30 | 4150 | 3.00 | 0.80 | 2.73 | 69.81 | 0.25 |
| 1.00 | 5.00 | 2100 | 0.45 | 0.48 | 32.00 | 0.28 | 4130 | 3.00 | 0.80 | 2.75 | 69.85 | 0.25 |
| 1.00 | 5.00 | 2000 | 0.45 | 0.45 | 31.00 | 0.28 | 4030 | 2.80 | 0.90 | 2.71 | 69.83 | 0.25 |
| 1.10 | 4.24 | 2050 | 0.45 | 0.47 | 31.00 | 0.30 | 4109 | 3.00 | 0.80 | 2.72 | 69.81 | 0.25 |
| 1.05 | 4.18 | 2000 | 0.45 | 0.45 | 31.00 | 0.30 | 4130 | 2.80 | 0.80 | 2.75 | 69.80 | 0.25 |
| 1.20 | 4.13 | 2106 | 0.41 | 0.47 | 29.72 | 0.30 | 4145 | 3.39 | 0.60 | 2.59 | 69.70 | 0.20 |
| 1.40 | 4.09 | 2120 | 0.41 | 0.45 | 29.07 | 0.42 | 4153 | 3.47 | 0.60 | 2.60 | 69.90 | 0.30 |
| 1.20 | 4.13 | 2163 | 0.41 | 0.47 | 28.94 | 0.33 | 4122 | 4.06 | 0.60 | 2.57 | 69.70 | 0.20 |
| 1.30 | 4.23 | 2064 | 0.41 | 0.45 | 27.97 | 0.32 | 4127 | 3.87 | 0.70 | 2.60 | 69.70 | 0.30 |
| 1.20 | 4.13 | 2153 | 0.41 |  | 28.83 | 0.32 | 4099 | 3.71 | 0.70 | 2.59 | 70.00 | 0.30 |
| 1.30 | 4.14 | 2126 | 0.41 |  | 29.02 | 0.35 | 4210 | 3.18 | 0.70 | 2.59 | 69.90 | 0.30 |
| 1.30 | 5.70 | 1980 | 0.41 |  | 28.71 | 0.32 | 4251 | 4.03 | 0.80 | 2.59 | 69.90 | 0.30 |
| 1.80 | 6.30 | 2000 | 0.41 |  | 29.45 | 0.42 | 4147 | 3.13 | 0.67 | 2.66 | 69.90 | 0.30 |
| 1.60 | 5.80 | 1970 | 0.44 |  | 36.60 | 0.42 | 4600 | 4.03 | 0.70 | 2.66 | 69.90 | 0.36 |
| 1.57 | 6.50 | 1930 | 0.44 |  | 36.20 | 0.41 | 4400 | 3.90 | 0.73 | 2.68 | 69.50 | 0.38 |
| 1.64 | 5.90 | 1960 | 0.44 |  | 36.60 | 0.42 | 4200 | 3.99 | 0.69 | 2.67 | 69.80 | 0.35 |
| 1.68 | 5.70 | 1970 | 0.44 |  | 35.80 | 0.42 | 4200 | 3.72 | 0.67 | 2.67 | 70.10 | 0.35 |
| 1.65 | 5.60 | 1920 | 0.44 |  | 32.30 | 0.44 | 4600 | 4.06 | 0.66 | 2.67 | 70.00 | 0.38 |
| 1.60 | 6.00 | 1960 | 0.44 |  | 35.00 | 0.43 | 4100 | 3.93 | 0.70 | 2.67 | 69.90 | 0.37 |
| 1.62 | 4.70 | 1937 | 0.44 |  | 33.00 | 0.44 |  | 3.85 | 0.66 | 2.67 | 69.60 | 0.36 |
| 1.60 | 4.80 | 2066 | 0.44 |  | 32.60 |  |  | 4.29 | 0.70 | 2.71 | 70.10 | 0.36 |
| 0.95 | 4.70 | 2002 | 0.44 |  | 34.20 |  |  | 3.96 | 0.80 | 2.63 | 68.45 |  |
| 0.93 | 4.50 | 2030 | 0.44 |  | 34.10 |  |  | 4.01 | 0.70 | 2.66 | 69.18 |  |
| 0.86 | 5.00 | 2073 | 0.43 |  | 35.30 |  |  | 3.82 | 0.80 | 2.70 | 68.58 |  |
| 0.86 | 4.70 | 2039 | 0.44 |  | 29.80 |  |  | 3.67 | 0.70 | 2.60 | 68.66 |  |
| 1.21 | 4.70 | 2030 | 0.44 |  | 33.70 |  |  | 3.72 | 0.80 | 2.59 | 70.73 |  |
| 1.06 | 4.50 | 2088 | 0.44 |  | 30.60 |  |  | 3.79 | 0.80 | 2.67 | 70.66 |  |
| 1.01 | 6.10 | 1883 | 0.43 |  | 30.50 |  |  | 3.63 | 0.70 | 2.68 | 70.53 |  |
| 0.96 | 6.10 | 1874 | 0.44 |  | 33.40 |  |  | 2.86 |  | 2.68 | 70.69 |  |
|  | 5.60 | 1885 | 0.47 |  | 30.70 |  |  | 2.62 |  | 2.68 | 70.50 |  |
|  | 5.80 | 1839 | 0.47 |  | 31.10 |  |  | 2.75 |  | 2.68 | 70.68 |  |
|  | 6.50 | 1942 | 0.47 |  | 30.70 |  |  | 3.70 |  | 2.68 | 70.71 |  |
|  | 5.40 | 1859 | 0.49 |  | 31.00 |  |  | 3.15 |  | 2.69 | 70.50 |  |
|  | 5.80 | 1895 | 0.49 |  | 25.20 |  |  | 3.12 |  | 2.69 | 69.36 |  |
|  | 5.80 | 1804 | 0.49 |  | 29.00 |  |  | 2.71 |  | 2.72 | 68.59 |  |
|  | 6.09 | 2123 | 0.48 |  | 28.40 |  |  | 3.70 |  | 2.69 | 68.95 |  |
|  | 6.12 | 2049 | 0.48 |  | 28.20 |  |  | 3.80 |  | 2.71 | 68.45 |  |
|  | 5.95 | 2055 | 0.48 |  | 28.10 |  |  | 3.90 |  | 2.72 | 68.97 |  |
|  | 6.37 | 2081 | 0.48 |  | 28.96 |  |  | 3.80 |  | 2.72 | 68.63 |  |
|  | 6.26 | 2059 | 0.48 |  | 33.24 |  |  | 3.70 |  | 2.69 | 68.80 |  |
|  | 6.11 | 2110 | 0.48 |  | 29.63 |  |  | 3.70 |  | 2.71 | 68.93 |  |
|  | 5.94 | 2083 | 0.48 |  | 29.74 |  |  | 3.60 |  | 2.72 | 70.10 |  |
|  | 5.36 | 2110 | 0.48 |  | 30.35 |  |  | 4.10 |  | 2.74 | 70.90 |  |
|  | 5.98 | 1873 | 0.48 |  | 29.05 |  |  |  |  | 2.70 | 69.90 |  |
|  | 5.48 | 1850 | 0.48 |  | 28.05 |  |  |  |  | 2.73 | 70.30 |  |
|  | 5.74 | 1859 | 0.46 |  | 30.93 |  |  |  |  | 2.74 | 70.00 |  |
|  | 5.20 | 1863 | 0.47 |  | 32.00 |  |  |  |  | 2.72 | 70.10 |  |
|  | 5.00 | 1905 | 0.46 |  | 32.00 |  |  |  |  | 2.75 | 70.90 |  |
|  | 5.72 | 1882 | 0.46 |  | 32.00 |  |  |  |  | 2.76 | 70.70 |  |
|  | 5.50 | 1866 | 0.45 |  | 33.00 |  |  |  |  | 2.70 | 69.69 |  |
|  | 5.20 | 1903 | 0.46 |  | 32.00 |  |  |  |  |  | 69.79 |  |
|  | 5.20 |  | 0.46 |  | 32.00 |  |  |  |  |  | 69.59 |  |
|  | 5.40 |  | 0.46 |  | 33.00 |  |  |  |  |  | 69.65 |  |
|  | 5.30 |  | 0.46 |  | 33.00 |  |  |  |  |  | 69.67 |  |
|  | 5.20 |  | 0.46 |  |  |  |  |  |  |  | 69.64 |  |
|  | 5.60 |  | 0.46 |  |  |  |  |  |  |  | 69.74 |  |
|  | 5.10 |  | 0.46 |  |  |  |  |  |  |  | 69.74 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |
|  |  |  |  |  |  |  |  |  |  |  | 70.30 |  |

## Assay Data (Cont.)

| 4A_MICP Sn ppm | 4A_MICP Sr ppm | 4A_MICP Ta ppm | 4A_MICP Tb ppm | 4A_MICP Th ppm | 4A_MICP Tl ppm | 4A_MICP U ppm | 4A_MICP V ppm | 4A_MICP W ppm | 4A_MICP Y ppm | 4A_MICP Yb ppm | 4A_MICP Zn ppm | 4A_MICP Zr ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 14.00 | 65.00 | 56.00 | 0.08 | 3.00 | 22.70 | 6.50 | 10.00 | 7.00 | 1.80 | 0.15 | 70.00 | 29.00 |
| 15.00 | 65.00 | 55.00 | 0.08 | 3.00 | 22.70 | 6.60 | 10.00 | 7.00 | 1.70 | 0.15 | 70.00 | 29.00 |
| 14.00 | 66.00 | 56.00 | 0.08 | 3.20 | 23.50 | 6.70 | 10.00 | 7.00 | 1.70 | 0.15 | 70.00 | 28.00 |
| 14.00 | 65.50 | 55.00 | 0.08 | 3.10 | 23.30 | 6.70 | 10.00 | 7.00 | 1.60 | 0.15 | 70.00 | 27.00 |
| 14.00 | 66.00 | 57.00 | 0.08 | 3.20 | 22.60 | 6.50 | 10.00 | 7.00 | 1.60 | 0.15 | 75.00 | 27.00 |
| 14.00 | 65.50 | 56.00 | 0.08 | 3.20 | 22.70 | 6.50 | 10.00 | 7.00 | 1.60 | 0.15 | 70.00 | 28.00 |
| 14.00 | 64.50 | 55.00 | 0.08 | 3.20 | 23.00 | 6.60 | 8.00 | 7.00 | 1.60 | 0.15 | 75.00 | 27.00 |
| 13.00 | 65.00 | 55.00 | 0.08 | 3.10 | 22.60 | 6.70 | 8.00 | 7.00 | 1.70 | 0.10 | 70.00 | 28.00 |
| 11.65 | 66.99 | 43.80 | 0.08 | 3.10 | 22.90 | 6.14 | 8.00 | 7.90 | 1.30 | 0.10 | 63.66 | 20.80 |
| 11.07 | 67.46 | 47.60 | 0.10 | 4.10 | 26.40 | 6.72 | 8.00 | 8.20 | 1.60 | 0.10 | 77.22 | 21.51 |
| 10.92 | 65.54 | 44.50 | 0.09 | 3.60 | 25.80 | 6.81 | 8.00 | 8.10 | 1.80 | 0.10 | 66.42 | 22.38 |
| 10.74 | 68.94 | 43.90 | 0.10 | 3.60 | 26.10 | 6.78 | 7.00 | 8.30 | 1.70 | 0.10 | 65.29 | 21.16 |
| 10.62 | 64.22 | 43.90 | 0.09 | 3.70 | 25.80 | 6.53 | 8.00 | 8.00 | 1.50 | 0.10 | 67.64 | 20.62 |
| 10.40 | 64.37 | 50.50 | 0.09 | 3.40 | 25.70 | 6.33 | 8.00 | 7.70 | 1.40 | 0.10 | 63.76 | 21.67 |
| 12.60 | 66.37 | 37.20 | 0.09 | 3.70 | 26.60 | 6.45 | 7.79 | 7.80 | 2.00 | 0.14 | 65.75 | 20.61 |
| 14.10 | 64.60 | 53.80 | 0.08 | 3.50 | 25.10 | 7.07 | 7.62 | 8.30 | 1.70 | 0.14 | 71.00 | 20.45 |
| 15.20 | 69.30 | 42.70 | 0.12 | 3.78 | 22.80 | 6.90 | 7.79 | 7.60 | 1.60 | 0.14 | 73.00 | 26.80 |
| 15.20 | 68.60 | 45.80 | 0.12 | 3.41 | 24.90 | 6.60 | 7.81 | 8.20 | 1.60 | 0.14 | 71.00 | 28.40 |
| 15.00 | 70.60 | 44.20 | 0.12 | 3.94 | 23.90 | 7.20 | 7.76 | 8.00 | 1.70 | 0.15 | 70.00 | 28.50 |
| 13.50 | 74.70 | 44.60 | 0.13 | 4.46 | 23.90 | 8.70 | 7.77 | 8.20 | 1.80 | 0.15 | 71.00 | 27.50 |
| 14.90 | 72.60 | 47.90 | 0.12 | 4.54 | 25.00 | 8.50 | 7.71 | 8.60 | 1.90 | 0.16 | 70.00 | 31.50 |
| 13.60 | 74.70 | 47.20 | 0.12 | 2.41 | 25.10 | 8.24 | 7.77 | 8.40 | 1.20 | 0.15 | 71.00 | 30.10 |
| 15.10 | 62.50 | 46.70 | 0.13 | 3.71 | 24.20 | 8.29 | 7.00 | 8.50 | 1.79 | 0.10 | 71.00 | 27.00 |
| 16.50 | 78.04 | 44.30 | 0.12 | 3.74 | 23.20 | 8.10 | 7.00 | 7.60 | 1.79 | 0.10 | 81.01 | 28.64 |
| 16.00 | 78.66 | 45.90 | 0.08 | 3.77 | 27.08 | 8.24 | 7.00 | 9.30 | 1.74 | 0.10 | 81.47 | 28.36 |
| 15.80 | 78.66 | 46.90 | 0.08 | 3.74 | 26.60 | 8.22 | 7.00 | 9.24 | 1.72 |  | 81.55 | 29.45 |
| 16.90 | 78.13 | 45.60 | 0.08 | 3.68 | 27.92 | 8.10 | 7.00 | 9.16 | 1.85 |  | 80.57 | 28.59 |
| 16.90 | 78.14 | 47.60 | 0.08 | 3.70 | 27.91 | 8.25 | 6.00 | 9.02 | 1.78 |  | 81.67 | 28.32 |
| 17.10 | 78.81 | 47.80 | 0.08 | 3.75 | 28.12 | 8.22 | 7.00 | 9.20 | 1.79 |  | 81.56 | 28.97 |
| 15.30 | 78.44 | 48.00 | 0.07 | 3.68 | 26.88 | 7.07 | 7.00 | 9.25 | 1.77 |  | 81.06 | 28.64 |
| 15.60 | 78.15 | 47.30 | 0.08 | 3.20 | 27.39 | 7.03 | 7.77 | 9.36 | 1.30 |  | 80.93 | 28.30 |
| 16.30 | 73.10 | 46.80 | 0.08 | 3.30 | 27.67 | 7.00 | 7.76 | 9.14 | 1.40 |  | 72.00 | 22.60 |
| 15.80 | 70.50 | 60.40 |  | 3.30 | 23.00 | 7.39 | 7.90 | 7.50 | 1.40 |  | 71.00 | 24.20 |
| 16.20 | 69.60 | 34.90 |  | 3.50 | 24.00 | 7.06 | 7.60 | 8.00 | 1.40 |  | 66.00 | 24.50 |
| 15.70 | 70.20 | 32.90 |  | 3.30 | 23.20 | 7.10 | 7.88 | 7.60 | 1.30 |  | 69.00 | 24.90 |
| 16.30 | 70.70 | 20.50 |  | 3.50 | 23.50 | 7.31 | 8.02 | 7.50 | 1.40 |  | 69.00 | 24.40 |
| 16.50 | 73.10 | 47.05 |  | 3.60 | 23.10 | 7.09 | 7.85 | 7.50 | 1.30 |  | 67.00 | 23.70 |
| 16.60 | 72.80 | 46.74 |  | 3.30 | 23.00 | 7.22 | 7.81 | 7.00 | 1.40 |  | 67.00 | 24.40 |
| 13.37 | 72.70 | 45.13 |  | 3.34 | 22.70 | 7.44 | 8.00 | 7.10 | 1.29 |  | 66.00 | 25.20 |
| 12.13 | 69.00 | 48.04 |  | 3.37 | 24.10 | 7.21 | 9.00 | 7.10 | 1.26 |  | 88.90 | 20.80 |
| 11.92 | 66.90 | 46.23 |  | 3.44 | 21.05 | 7.32 | 9.00 | 5.55 | 1.24 |  | 74.10 | 20.50 |
| 11.83 | 65.80 | 46.86 |  | 3.53 | 25.65 | 7.36 | 8.00 | 6.28 | 1.25 |  | 88.30 | 20.00 |
| 16.06 | 65.50 | 47.79 |  | 3.53 | 24.14 | 7.41 | 9.00 | 5.72 | 1.72 |  | 87.70 | 20.00 |
| 14.14 | 71.10 | 46.34 |  | 3.67 | 23.25 | 7.41 | 9.00 | 5.42 | 1.47 |  | 89.20 | 28.20 |
| 14.22 | 75.60 |  |  | 3.66 | 20.64 | 7.22 | 8.00 | 7.60 | 1.40 |  | 80.80 | 23.30 |
| 11.78 | 73.80 |  |  | 3.54 | 25.08 |  | 8.00 | 7.40 | 1.34 |  | 75.30 | 22.00 |
| 15.40 | 67.70 |  |  |  | 25.07 |  |  | 7.10 | 1.50 |  | 73.40 | 20.30 |
| 15.10 | 63.80 |  |  |  | 24.61 |  |  | 7.50 | 1.50 |  | 71.30 | 26.40 |
| 15.00 | 64.50 |  |  |  | 25.06 |  |  | 7.30 | 1.50 |  | 73.50 | 26.50 |
| 15.30 | 65.30 |  |  |  | 24.57 |  |  | 7.30 | 1.50 |  | 73.50 | 26.80 |
| 15.60 | 67.10 |  |  |  | 24.71 |  |  | 7.30 | 1.60 |  | 74.30 | 25.90 |
| 15.20 | 67.90 |  |  |  | 25.12 |  |  | 7.20 | 1.50 |  | 72.50 | 26.10 |
| 15.20 | 66.10 |  |  |  | 24.48 |  |  |  | 1.50 |  | 74.80 | 26.00 |
| 15.40 | 66.00 |  |  |  |  |  |  |  | 1.50 |  |  | 29.30 |
|  | 68.00 |  |  |  |  |  |  |  |  |  |  | 26.10 |
|  |  |  |  |  |  |  |  |  |  |  |  | 24.80 |
|  |  |  |  |  |  |  |  |  |  |  |  | 24.58 |
|  |  |  |  |  |  |  |  |  |  |  |  | 25.04 |
|  |  |  |  |  |  |  |  |  |  |  |  | 25.14 |
|  |  |  |  |  |  |  |  |  |  |  |  | 25.04 |
|  |  |  |  |  |  |  |  |  |  |  |  | 23.59 |
|  |  |  |  |  |  |  |  |  |  |  |  | 24.42 |
|  |  |  |  |  |  |  |  |  |  |  |  | 25.88 |

## 21. Reported Values

The certified values listed in this certificate fulfil the AMIS statistical criteria (see section 18) regarding agreement for certification and have been independently validated by Allan Fraser.

## 22. Validation of Accuracy (Trueness)

This CRM can be used to validate accuracy (trueness) as required in method validation as stated in the ISO17025:2005 standard, clause 5.4. See Appendix 3 for an example on the validation of accuracy using replicate data derived from the analysis of a CRM.

## 23. Metrological Traceability

The values quoted herein are based on the consensus values derived from statistical analysis of the data from an inter-laboratory measurement program. Traceability to SI units is via the standards used by the individual laboratories the majority of which are accredited to the ISO17025:2005 general requirements for the competence of testing and calibration laboratories and who have maintained measurement traceability during the analytical process.

## 24. Period of Validity

The certified values are valid for this product, while still sealed in its original packaging, until notification to the contrary. The stability of the material will be subject to continuous testing for the duration of the inventory. Should product stability become an issue, all customers will be notified and notification to that effect will be placed on the www.amis.co.za website.

## 25. Minimum Sample Size

The majority of laboratories reporting used a 0.5g sample size for the ICP-OES and a 30g sample size for the fire assay. These are the recommended minimum sample sizes for the use of this material.

## 26. Availability

This product is available in Laboratory Packs containing 1kg of material and Explorer Packs containing custom weights (from 50 to 250g) of material. The Laboratory Packs are sealed bottles delivered in sealed foil pouches. The Explorer Packs contain material in standard geochem envelopes, nitrogen flushed, and vacuum sealed in foil pouches.

## 27. Recommended use in Quality Control

Users should set their own limits *i.e.* 1, 2 and 3 standard deviations from an obtained mean value based on at least 10 replicate analyses using this CRM (see Appendix 4 for detail on the use of this CRM in quality control).

## 28. Legal Notice

This certificate and the reference material described in it have been prepared with due care and attention. However, AMIS, a division of Torre Analytical Services (Pty) Ltd, Thivhafuni Matodzi, and Allan Fraser; accept no liability for any decisions or actions taken following the use of the reference material.

**22 June 2018**

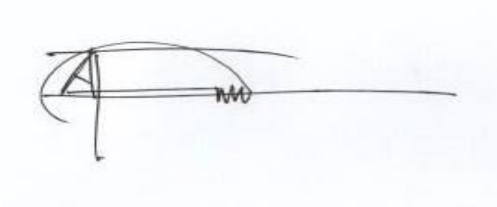
***Revision:000***

**Certifying Officers:**

T. Matodzi

**African Mineral Standards:** _____

**Thivhafuni Matodzi**



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# APPENDICES

**Appendix 1:** Uncertified Element Statistics.

**Table 8.** Uncertified element concentrations statistics.

| Element | Generic Method | n | Mean | s | % RSD | Unit |
|---|---|---|---|---|---|---|
| Al | 4A_MICP | 47 | 70242.271 | 6392.696 | 9.101 | ppm |
| As | 4A_ICPES | 7 | 8.649 | 0.660 | 7.631 | ppm |
| As | FUS | 4 | 5.750 | 0.500 | 8.696 | ppm |
| Ba | 4A_ICPES | 8 | 35.988 | 0.779 | 2.165 | ppm |
| Ba | FUS | 16 | 31.313 | 1.778 | 5.679 | ppm |
| BaO | XRF | 2 | 0.010 | <0.001 | <0.001 | % |
| Be | 4A_ICPES | 8 | 330.375 | 8.314 | 2.517 | ppm |
| Be | FUS | 15 | 416.667 | 9.649 | 2.316 | ppm |
| Bi | 4A_ICPES | 8 | 37.725 | 0.587 | 1.557 | ppm |
| Bi | FUS | 8 | 36.325 | 0.618 | 1.702 | ppm |
| Ca | FUS | 24 | 17602.965 | 1453.241 | 8.256 | ppm |
| CaO | FUS | 24 | 2.462 | 0.203 | 8.249 | % |
| Cd | 4A_MICP | 36 | 0.121 | 0.074 | 61.006 | ppm |
| Ce | 4A_MICP | 45 | 2.581 | 0.385 | 14.931 | ppm |
| Ce | FUS | 16 | 2.379 | 0.181 | 7.611 | ppm |
| Co | FUS | 7 | 1.629 | 0.049 | 2.996 | ppm |
| Cr | 4A_ICPES | 7 | 95.114 | 9.453 | 9.938 | ppm |
| Cr | 4A_MICP | 56 | 91.455 | 49.817 | 54.471 | ppm |
| Cr | FUS | 16 | 178.750 | 5.994 | 3.354 | ppm |
| $Cr_2O_3$ | FUS | 16 | 0.019 | 0.007 | 37.579 | % |
| $Cr_2O_3$ | XRF | 39 | 0.028 | 0.005 | 17.332 | % |
| Cs | FUS | 16 | 601.688 | 14.351 | 2.385 | ppm |
| Cs | XRF | 8 | 815.000 | 18.516 | 2.272 | ppm |
| Cu | 2A_MICP | 8 | 15.347 | 0.443 | 2.884 | ppm |
| Cu | 4A_ICPES | 15 | 11.760 | 1.709 | 14.533 | ppm |
| Cu | FUS | 11 | 10.091 | 0.302 | 2.988 | ppm |
| Dy | FUS | 15 | 0.406 | 0.011 | 2.715 | ppm |
| Er | FUS | 15 | 0.149 | 0.016 | 10.553 | ppm |
| Eu | 4A_MICP | 13 | 0.067 | 0.006 | 8.924 | ppm |
| Eu | FUS | 1 | 0.050 | <0.001 | <0.001 | ppm |
| $Fe_2O_3$ | FUS | 32 | 0.572 | 0.156 | 27.222 | % |
| Ga | FUS | 16 | 70.125 | 24.391 | 34.782 | ppm |
| Gd | FUS | 16 | 0.321 | 0.016 | 5.044 | ppm |
| Ge | 4A_MICP | 16 | 4.064 | 4.054 | 99.756 | ppm |
| Ge | FUS | 8 | 8.000 | <0.001 | <0.001 | ppm |
| Hf | FUS | 16 | 6.434 | 1.515 | 23.552 | ppm |
| Ho | 4A_MICP | 17 | 0.050 | 0.010 | 20.716 | ppm |
| Ho | FUS | 11 | 0.054 | 0.006 | 11.793 | ppm |
| Ind | 4A_MICP | 3 | 0.008 | 0.003 | 32.825 | ppm |
| K | FUS | 24 | 32378.724 | 2568.603 | 7.933 | ppm |
| $K_2O$ | FUS | 24 | 3.901 | 0.310 | 7.941 | % |
| La | FUS | 16 | 1.296 | 0.123 | 9.498 | ppm |
| Li | 4A_ICPES | 16 | 6189.819 | 264.887 | 4.279 | ppm |
| Li | 4A_MICP | 64 | 5332.658 | 324.281 | 6.081 | ppm |
| Li | FUS | 40 | 5348.075 | 289.618 | 5.415 | ppm |
| $Li_2O$ | FUS | 8 | 1.279 | 0.099 | 7.713 | % |
| Lu | 4A_MICP | 15 | 0.015 | 0.005 | 33.678 | ppm |
| Mg | 4A_MICP | 55 | 2485.193 | 277.488 | 11.166 | ppm |
| Mn | 4A_ICPES | 7 | 1683.857 | 14.323 | 0.851 | ppm |
| Mn | FUS | 24 | 1868.323 | 401.494 | 21.490 | ppm |
| MnO | FUS | 24 | 0.241 | 0.052 | 21.477 | % |
| Mo | 4A_ICPES | 8 | 0.826 | 0.128 | 15.512 | ppm |
| Moisture | Moisture | 15 | 0.154 | 0.068 | 44.436 | % |
| Na | FUS | 16 | 15496.846 | 1121.541 | 7.237 | ppm |

| Element | Generic Method | n | Mean | s | % RSD | Unit |
|---|---|---|---|---|---|---|
| $Na_2O$ | FUS | 16 | 2.094 | 0.152 | 7.237 | % |
| Nb | FUS | 31 | 51.513 | 8.142 | 15.806 | ppm |
| $Nb_2O_5$ | XRF | 3 | 0.010 | <0.001 | <0.001 | % |
| Nd | FUS | 16 | 1.151 | 0.094 | 8.150 | ppm |
| Ni | 4A_ICPES | 16 | 5.187 | 0.694 | 13.382 | ppm |
| Ni | FUS | 12 | 9.750 | 1.215 | 12.466 | ppm |
| P | FUS | 24 | 1833.302 | 199.766 | 10.897 | ppm |
| $P_2O_5$ | FUS | 24 | 0.420 | 0.046 | 10.853 | % |
| Pb | 2A_MICP | 8 | 25.383 | 0.488 | 1.922 | ppm |
| Pb | 4A_ICPES | 7 | 32.143 | 0.640 | 1.991 | ppm |
| Pb | FUS | 8 | 32.750 | 0.707 | 2.159 | ppm |
| Pr | FUS | 15 | 0.338 | 0.012 | 3.564 | ppm |
| Rb | 4A_MICP | 48 | 4010.105 | 421.616 | 10.514 | ppm |
| Rb | XRF | 8 | 4318.375 | 79.022 | 1.830 | ppm |
| Re | 4A_MICP | 1 | 0.002 | <0.001 | <0.001 | ppm |
| S | 4A_MICP | 32 | 0.011 | 0.002 | 17.548 | % |
| S | Combustion/LECO | 37 | 0.011 | 0.002 | 15.862 | % |
| Sb | 4A_ICPES | 8 | 4.125 | 1.979 | 47.979 | ppm |
| Sb | FUS | 8 | 3.700 | 0.200 | 5.405 | ppm |
| Se | 4A_MICP | 7 | 2.000 | 1.000 | 50.000 | ppm |
| Si | FUS | 16 | 32.528 | 0.310 | 0.952 | % |
| $SiO_2$ | FUS | 16 | 69.209 | 0.659 | 0.952 | % |
| Sm | FUS | 16 | 0.242 | 0.041 | 17.054 | ppm |
| Sn | 4A_ICPES | 8 | 15.400 | 1.322 | 8.587 | ppm |
| Sn | FUS | 24 | 17.125 | 3.275 | 19.122 | ppm |
| $SO_3$ | XRF | 16 | 0.021 | 0.010 | 48.384 | % |
| Sr | 4A_ICPES | 16 | 67.381 | 5.114 | 7.589 | ppm |
| Sr | FUS | 8 | 72.375 | 1.923 | 2.656 | ppm |
| SrO | XRF | 14 | 0.011 | 0.001 | 6.743 | % |
| Ta | FUS | 32 | 57.197 | 13.695 | 23.944 | ppm |
| Tb | FUS | 15 | 0.097 | 0.009 | 8.870 | ppm |
| Te | 4A_MICP | 21 | 0.113 | 0.080 | 71.281 | ppm |
| Th | FUS | 15 | 3.529 | 0.212 | 6.006 | ppm |
| $ThO_2$ | XRF | 2 | 0.010 | <0.001 | <0.001 | % |
| Ti | 4A_MICP | 48 | 0.015 | 0.003 | 21.242 | % |
| Ti | FUS | 24 | 0.014 | 0.004 | 26.193 | % |
| $TiO_2$ | FUS | 24 | 0.024 | 0.006 | 26.166 | % |
| $TiO_2$ | XRF | 55 | 0.025 | 0.006 | 21.773 | % |
| Tl | FUS | 8 | 24.738 | 0.207 | 0.835 | ppm |
| U | FUS | 15 | 7.445 | 0.310 | 4.162 | ppm |
| V | 2A_MICP | 7 | 6.944 | 0.059 | 0.849 | ppm |
| V | 4A_ICPES | 7 | 8.729 | 0.239 | 2.742 | ppm |
| V | FUS | 15 | 8.533 | 1.598 | 18.722 | ppm |
| W | FUS | 8 | 7.750 | 0.463 | 5.973 | ppm |
| Y | FUS | 16 | 1.678 | 0.134 | 8.010 | ppm |
| Yb | FUS | 15 | 0.112 | 0.015 | 13.157 | ppm |
| Zn | 4A_ICPES | 8 | 73.125 | 1.246 | 1.705 | ppm |
| Zn | FUS | 14 | 70.571 | 1.697 | 2.404 | ppm |
| Zr | 4A_ICPES | 8 | 27.596 | 0.605 | 2.191 | ppm |
| Zr | FUS | 16 | 26.413 | 6.452 | 24.429 | ppm |
| $ZrO_2$ | XRF | 8 | 0.009 | 0.001 | 7.508 | % |

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**Appendix 2.** Certification of Reference Material and Estimation of Measurement Uncertainty
(Prepared by Allan Fraser)

In the establishment of a consensus value for the CRM, outlier tests are carried out followed by performance statistics and the estimation of the measurement uncertainty. In practice, it is highly likely that data generated by multiple laboratories as an inter-laboratory comparison of material for certification, will contain erroneous as well as extreme measurements (outliers). The influence of outliers on summary statistics needs to be minimised by the application of procedures for outlier identification on raw data. The application of *z*-scoring, Cochran test for suspect repeatability variances, along with Grubbs test as single and paired tests for suspect measurement values allows for the detection of outliers (IUPAC, 1995). Method performance in terms of precision as relative standard deviation is judged by the application of the Horwitz ratio, which gives an indication of whether the observed relative standard deviation at the concentration levels of analyte determined are acceptable (Horwitz & Albert, 2006).

In the absence of an extensive uncertainty budget, measurement uncertainty is estimated from the reproducibility standard deviation from inter-laboratory data and reported as an expanded uncertainty at typically a level of confidence of 95% (Miller & Miller, 2010).

The steps below give detail on the establishment of a consensus value through the elimination of outliers, method performance and estimation of measurement uncertainty using standard uncertainties and the analysis of variance.

**Outlier Removal**

An initial purge of outliers in inter-laboratory analyses of a candidate reference material is done using *z*-scoring followed by the exclusion of further outliers as defined by the IUPAC Harmonised Protocol of 1995. Here both Cochran and Grubbs tests are applied until all outliers are identified.

***Z-Score***

A z-score is calculated using equation [1]:

$$z = \frac{x - x_a}{s_p} \qquad [1]$$

Where, $x$ is the result of a submitted sample, $x_a$ is the mean and $s_p$is the standard deviation of the submitted results from all of the participating laboratories. Z-Scores are interpreted as follows:

$|z|\leq 2$ satisfactory performance
$2<|z|\leq 3$ questionable performance
$|z|>2$ unsatisfactory performance

(Thompson & Lowthian, 2011)

Data with z-scores exceeding 2 are discarded and are not included for further assessment.

***Cochran Test***

The test of Cochran (1950) as shown in equation [2] is applied to any suspect repeatability variances:

$$C_{calc} = \frac{s_{max}^2}{\sum_{i=1}^{l} s_i^2} \qquad [2]$$

Where, $C_{calc}$ , $s_{max}^2$ and $\sum_{i=1}^{l} s_i^2$, are the calculated values for Cochran's test, data set with the maximum variance and the sum of the variances of all of the participating $l$ laboratory datasets. The $C_{calc}$ value is

compared with a critical value, $C_{crit}$ at a level of confidence of 95% and an alpha of 0.05% (see Ellison, *et al.,* 2009, Appendix A, Table A.3a, page 209 for a table of critical values for the test of Cochran at LOC 95%).

According to ISO 5725-2 (1999), results from a laboratory with a suspect repeatability variance can be excluded if it is shown by the Cochran test to be an outlier. Therefore, if $C_{calc} > C_{crit}$ , the laboratory with the maximum variance is removed. The data found to be excluded should not be >2/9, or 22% of the total data.

***Grubbs Test***

The test of Grubbs (1969) calculates a test statistic, G. In the detection of a single outlier, $G_1$ is found by using

$$G_{1\,calc} = \frac{|Suspect\ value - \bar{x}|}{s} \quad [3]$$

where the sample mean and standard deviation, $\bar{x}$ and s, are calculated with the suspect value included. The $G_{1\,calc}$ statistic is compared to a critical value for *N* measurements. See Ellison, *et al.,* 2009, Appendix A, Table A.2, page 208 for a table of critical values for the test of Cochran at LOC 95%.

**Method Performance**

The Horwitz function is used to assess the performance of the data under consideration, with respect to precision (Horwitz & Albert, 2006). A calculated %RSD is found using the Horwitz expression

$$\%RSD = \pm 2^{(1-0.5logC)} \quad [4]$$

where, *C* is the analyte concentration in percent divided by 100 and log is the natural logarithm. The observed %RSD is calculated as

$$Observed\ \%RSD = \frac{s}{Mean} x100 \quad [5]$$

where *s* is the standard deviation of n replicates.

The ratio of the observed %RSD and the calculated %RSD gives the Horwitz ratio (HorRat):

$$HorRat = \frac{\%RSD\ Observed}{\%RSD\ Calculated} \quad [6]$$

A HorRat ≤2 indicates that the method is of adequate precision. Should the HorRat be >2 the overall data are discarded and the candidate material considered not suitable for certification as the precision is excessive for the concentration of the analyte being determined (Nelsen & Wehling, 2008).

**Grand Mean**

The grand mean ($\bar{\bar{x}}$) *i.e.* the certified value of a dataset is the total of all the data values divided by the total sample size (*n*):

$$\bar{\bar{x}} = \sum \frac{x}{n} \quad [7]$$

**Certified Value**

From ANOVA as per the description in section 18, an 'appropriate precision' as shown in [8] is calculated for sufficient homogeneity (Thompson, 2008):

$$s_r \leq 0.3u_c \quad [8]$$

Where, $s_r$ is the within laboratory repeatability, as determined from [14]. Once [8] is satisfied, a grand mean [7] is calculated and this is taken to be the certified value.

**Total Variation (SST)**

The total variation (not the variance) comprises the sum of the squares of the differences of each mean with the grand mean.

$$\mathrm{SST} = \sum (x - \bar{\bar{x}})^2 \quad [9]$$

**Between Group Variation (SSB)**

The *variation* due to the interaction between the laboratories is denoted SSB or Sum of Squares Between laboratories and given by [10]. If the laboratory means are close to each other (and therefore the Grand Mean) SSB will be a small value. There are P samples involved with one datum value for each sample (the sample mean), so there are P-1 degrees of freedom.

$$\mathrm{SSB} = \sum n(\bar{x} - \bar{\bar{x}})^2 \quad [10]$$

The *variance* due to the interaction between the laboratories is denoted MSB for Mean Square Between groups and is the SSB divided by its degrees of freedom.

$$MS = \frac{SSB}{n-1} \quad [11]$$

**Within Group Variation (SSW)**

The variation due to differences within individual samples is denoted SSW for Sum of Squares Within laboratories. The degrees of freedom are equal to the sum of the individual degrees of freedom for each sample. Since each sample has degrees of freedom *(df)* equal to one less than their sample sizes, and there are *k* samples, the total degrees of freedom is P less than the total sample size: *df* = *n* - P.

$$SSW = \sum df \cdot s^2 \quad [12]$$

The variance due to the differences within individual samples is denoted MSW for Mean Square Within groups. This is the within group variation divided by its degrees of freedom:

$$MSW = \frac{SSW}{\mathrm{P} - n} \quad [13]$$

From equations [9] through [13], the ANOVA table as shown in Table 9 is developed.

**Table 9.** A single-factor ANOVA table showing key elements. Where P is the total number of groups, or laboratories. P-1 is 1 less than number of laboratories, P (n-1) is the number of data values minus number of groups (equals degrees of freedom for each group added together), and P-1 + P(n-1) is 1 less than number of data points. MS is the mean squares of between laboratories and within laboratories. After Ellison *et al.,* (2009), Table 6.2, page 61.

| **Source** | **Sum of Squares** | ***df*** | **Mean Sum of Squares** | ***F*** | ***p*** | $F_{crit}$ |
|---|---|---|---|---|---|---|
| Between Laboratories | SSB | P-1 | MSB=SSB/df | MSB/MSW | =FDIST(x,*df,df*) | F-table |
| Within Laboratories | SSW | P(n-1) | MSW=SSW/df | – | – | – |
| Total | SSB+SSW | P-1 + P(n-1) | – | – | – | – |

**Combined Standard Uncertainty**

The combined standard uncertainty ($u_c$) represents the effects of random events such as days, instruments, and analysts on the precision of the analytical procedures of all accepted data of the participating laboratories. Using the output from ANOVA, the combined standard uncertainty ($u_c$) is determined from the square root of the sum of squares of the variances of the within laboratory repeatability, $s_r$ and the between laboratory precision, $s_s$:

$$u_c = \sqrt{{s_r}^2 + {s_s}^2} \quad [14]$$

Within laboratory repeatability is determined as

$$s_r = \sqrt{MSB} \quad [15]$$

and, the between laboratory precision as

$$s_s = \sqrt{\frac{(MSW - MSB)}{n}} \quad [16]$$

where $MSW$ is the mean squares of the within laboratory variance, $MSB$ is the mean squares for the between laboratories and $n$ in this case, is the number of replicates in a group of the accepted data (Thompson & Lowthian, 2011).

**Expanded Uncertainty**

The expanded uncertainty (*U*) at a confidence level of 95% is determined by multiplication of the combined uncertainty ($u_c$) by a coverage factor (*k*) found from *N*-1 degrees of freedom (*df*), where *N* is the number of laboratory means accepted in the establishment of the certified value. The t-critical value for 5% significance can be found in a t-critical table (see Appendix 6, or from MS Excel as =TINV (5%, *df*).

**Uncertainty Statement**

Typically, an uncertainty statement is presented as follows: Au =0.77±0.04 g/t, where the number following the symbol ± is the numerical value of an expanded uncertainty, $U = ku_c$, with *U* determined from a combined standard uncertainty multiplied by a coverage factor $k = 2$ or, a t-critical value for *N*-1 accepted laboratories. Since it can be assumed that the possible estimated values of the standard are approximately normally distributed with standard uncertainty, $u_c$, the certified value of the CRM is believed to lie in the interval defined by *U* with a level of confidence of approximately 95 %, *e.g.* a mean value of 0.77±0.04g/t will have intervals of: 0.73<0.77<0.81 g/t.

**Appendix 3.** Example: Comparison of Mean and Certified Value for Validation of Accuracy
(Prepared by Allan Fraser)

According to ERM (2005); Eurolab (2007); Abzalov (2011) and Carr (2011), the validation of accuracy for a given mean and certified value requires the inclusion of the measurement uncertainty of the CRM in a t-test for statistical significance. The classical Student's t-test as shown in [17], does not take into account the measurement uncertainty of the CRM. To compensate for this, Eurolab Technical Report No.1/2007 recommends equation [18] for the validation of CRMs with stated measurement uncertainties.

$$t_{calc} = \frac{|\bar{x} - \mu|}{\frac{s}{\sqrt{n}}} \qquad [17]$$

$$t_{calc} = \frac{|\bar{x} - \mu|}{\sqrt{(u_\mu)^2 + \frac{s^2}{n}}} \qquad [18]$$

Where, $t_{calc}$ is the calculated t-statistic, $\bar{x}$ the mean of n replicates with a standard deviation of *s* for a CRM of μ certified value. The standard uncertainty *u* is the stated expanded uncertainty (*U*) of the CRM divided by the coverage factor (*k*) as stated on the certificate of analysis. Note that the | | bars indicate that the absolute value between the mean and the certified value is to be used, *i.e.* ignore the sign.

An example in which [18] is used for validation of accuracy is given below.

**Example**

A CRM is independently replicated nine times for $Al_2O_3$ concentration by XRF analysis, *i.e.* 9 individual fused glass beads were prepared. The observed mean and standard deviation of the replicate data are shown with the certified value and expanded uncertainty in Table 10. In validation of accuracy, the hypothesis question is: Is the difference between the observed mean and the certified value statistically significant at a level of confidence of 95%? Alternatively put, is there sufficient evidence to conclude that the data *i.e.* replicates generated, are inaccurate?

The relevant hypotheses are:

**Null hypothesis:** $H_0$: Mean = Certified value of CRM with stated measurement uncertainty. The acceptance of $H_0$ means that accuracy is demonstrated; *i.e.* insufficient evidence to reject $H_0$;
**Alternate hypothesis:** $H_1$: Mean ≠ Certified value of CRM with stated measurement uncertainty. The acceptance of $H_1$ means that accuracy is not demonstrated, *i.e.* there is sufficient evidence to accept $H_1$;

**Table 10.** CRM certified value quoted expanded uncertainty *U*, the coverage factor for the CRM, *k*=2.25 and mean for *n*=9 replicates and corresponding standard deviation for the replicate data.

| CRM Certified Value | Expanded Uncertainty (*U*) | Coverage Factor (*k*) | Mean (*n*=9) | *n* | Standard Deviation (*s*) |
|---|---|---|---|---|---|
| 4.62% | 0.08% | 2.25 | 4.59 | 9 | 0.01015 |

The standard uncertainty (*u*) is found by dividing the expanded uncertainty by the coverage factor:

$$u = \frac{0.08}{2.25} = 0.0356\ \%$$

Using the observed mean for the replicate data (*n*=9) obtained for the CRM and substituting into [18]:

$$t_{calc} = \frac{|\bar{x} - \mu|}{\sqrt{0.0356^2 + \frac{0.01015^2}{9}}} = \frac{|4.59 - 4.62|}{\sqrt{0.00126 + 0.00001145}} = 0.84$$

Therefore, $t_{calc}$= 0.84 and $t_{crit}(5\%, 8) = 2.31$ (*df* is 8, therefore, $t_{crit}$=2.31, see Appendix 7, page 31) which is >0.84. Similarly, the *p*-value=0.43 which is >0.05. This is strong evidence in favour of accepting the null hypothesis that there is no significant statistical difference between the certified value and the observed mean. Therefore, under the conditions that the uncertainty associated with the certified value is known the accuracy is validated for the CRM tested. If the null hypothesis is accepted that the mean obtained is not statistically different from the certified value, then the principle of traceability has been conformed to.

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**Appendix 4.** Using the CRM in Quality Control
(Prepared by Allan Fraser)

QC chart control limits should not be determined by the certified value and stated measurement uncertainty of the certified reference material used. These parameters although "certified" will never be known; it is only the corresponding statistical estimates, *i.e.* standard deviation and the mean calculated from replicated results that are known and these should be used in quality control charts. However, should the laboratory choose to use the certified value as the mean then the quoted 2s value for the CRM can be used in the quality control chart.

It is recommended that a Shewhart chart be developed for the use if this CRM is to be used as a control sample in laboratory quality control. A Shewhart chart is a plot of sequential assay results obtained from quality control material such as an AMIS CRM. The warning and control limits are based on the standard deviation obtained from the mean of the replicates of a CRM (Ellison, *et al.,* 2009; Thompson, 2010). The procedure in preparing a Shewhart chart is as follows:

1. Analyse 10 to15 replicates or more of the AMIS CRM;
2. Apply the Grubbs test for outliers;
3. Determine the mean of the replicates after application of the Grubbs test;
4. Determine the standard deviation, using equation [19], of the replicates;
5. Calculate the standard deviation, *s* from:

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n - 1}} \qquad [19]$$

where, $x_i$ is an individual measurement in the data set, $\bar{x}$ is the mean of the data set at n-*1* degrees of freedom (*df*) and *n* is the number of replicates. The sample standard deviation can be found using the MS Excel formula "=stdev.s (number1;)".

6. Verify accuracy of the mean value using equation [18];
7. Once accuracy is verified, calculate ±2s and ±3s, where *s* is the standard deviation calculated from [19].
8. Construct the Shewhart control chart around the mean of *n* replicates;
9. Use ±2s as the warning limits;
10. Use ±3s as the control limits;

11. It is recommended that if 2 to 3 points are outside warning the limits analyse another sample and if it then within warning limits, continue. If it is outside the warning limits, stop and troubleshoot;
12. It is recommended that if any point is outside control limits, analyse another portion (sample) of the CRM. If it is within control limits, continue. If it is outside control limits, stop and troubleshoot;
13. For reference purposes, the CRM certified value can be plotted on the Shewhart chart alongside the mean value.

On a regular basis the accuracy of the replicates of the CRM should be assessed in terms of the certified value of the CRM using equation [18].

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**Appendix 5.** Conversion to Air-dry Basis
(Prepared by Allan Fraser)

Since AMIS certified analyte values are reported on a dry-basis, the user laboratory is required to dry a portion (accurately weigh out 1.0 grams in duplicate) of the CRM material in air at 105°C in a drying oven to constant mass to determine the moisture content. Use a crucible with a flat inner surface with a surface area not smaller than 10 $cm^2$ with the CRM material spread evenly over same; this represents a 0.1 gram spread per $cm^2$. In correcting the certified value for moisture content, a moisture correction factor is calculated:

$$Moisture\ correction\ factor\ (MCF) = \frac{100 - \%Moisture\ at\ 105^{O}C}{100} \qquad [20]$$

$$Air\ dry\ basis\ concentration = MCF\ x\ certified\ value\ on\ a\ dry\ basis \qquad [21]$$

**Example**

The moisture content determined at 105°C on a CRM is 0.500%. The certified analyte concentration for the CRM is 12.62±0.52% (dry basis). Calculating the moisture correction factor using [21] gives:

$$Moisture\ correction\ factor = \frac{100 - 0.500}{100} = 0.995$$

Multiplying the factor of 0.995 by the certified value as stated on the certificate of analysis on a dry basis (as in [21]) gives the analyte concentration on an air-dry basis:

$$0.995\ x\ 12.62\% = 12.56\%$$

The stated measurement uncertainty also needs to be corrected using [20] and [21], *e.g.* 0.995 x 0.52 = $0.51_{(7)}$, rounded to 0.52%. The air-dry basis concentration *i.e.* 12.56±0.52% is to be used as the certified value with its corresponding measurement of uncertainty.

**Table 11.** Mass of assay sample and corresponding limit of detection and limit of quantitation for an assay microbalance capability of smallest prill mass of 1µg or 0.001mg.

| Mass Assay Sample (g) | LOD (g/t) | LOQ (g/t) |
|---|---|---|
| 30 | 0.03 | 0.3 |
| 50 | 0.02 | 0.2 |
| 100 | 0.01 | 0.1 |

**Table 12.** Recommended reporting scheme for LOD and LOQ in fire assay.

| Data | Report as |
|---|---|
| <LOD | Not detected |
| <LOQ | Detected |
| ≥LOQ | Report assay result |

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**Appendix 6.** T-distribution table

Table 13. T-distribution table for t-critical values (t crit.) for a two-tailed t-test at a 95% level of confidence.

| *df* | Two-tailed | *df* | Two-tailed |
|---|---|---|---|
| 1 | 12.71 | 23 | 2.06 |
| 2 | 4.30 | 24 | 2.06 |
| 3 | 3.18 | 25 | 2.06 |
| 4 | 2.78 | 26 | 2.05 |
| 5 | 2.57 | 27 | 2.05 |
| 6 | 2.44 | 28 | 2.04 |
| 7 | 2.36 | 29 | 2.04 |
| 8 | 2.30 | 30 | 2.04 |
| 9 | 2.26 | 35 | 2.03 |
| 10 | 2.22 | 40 | 2.02 |
| 11 | 2.20 | 45 | 2.01 |
| 12 | 2.17 | 50 | 2.00 |
| 13 | 2.16 | 55 | 2.00 |
| 14 | 2.14 | 60 | 2.00 |
| 15 | 2.13 | 70 | 1.99 |
| 16 | 2.12 | 80 | 1.98 |
| 17 | 2.11 | 90 | 1.98 |
| 18 | 2.10 | 100 | 1.98 |
| 19 | 2.09 | 120 | 1.98 |
| 20 | 2.08 | Infinity | 1.96 |
| 21 | 2.08 | | |
| 22 | 2.07 | | |