AMIS0945

Certified Reference Material

Orogenic Gold, Ghana

Certificate of Analysis

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Summary Statistics

Recommended Concentrations and Limits (at two Standard Deviations) Certified Concentrations

Analyte	Method	Certified Value (μ)	[11]Two standard deviations (2s) ±	Unit
Au	[1] Pb Collection	0.250	0.03	g/t
Au	[2] 2A_MICP	0.243	0.03	g/t
Au	[3] CL	0.244	0.06	g/t
Ag	[4] 4A_MICP	0.1	0.04	ppm
Cu	4A_MICP	50	2	ppm
C	[5] Combustion/LECO	1.35	0.092	%
S	Combustion/LECO	0.18	0.03	%
S	4A_MICP	0.19	0.03	%
LOI	[6] LOI	7.14	0.61	%
SG	[7] SG	2.71	0.16	Dimensionless
As	4A_MICP	22	9	ppm
Ba	4A_MICP	712	41	ppm
Be	4A_MICP	2	0.1	ppm
Bi	4A_MICP	0.06	0.02	ppm
Ca	4A_MICP	3.34	0.11	%
Ce	4A_MICP	67	12	ppm
Co	4A_MICP	16	3	ppm
Cr	4A_MICP	87	22	ppm
Cs	4A_MICP	4	0.2	ppm
Dy	4A_MICP	2	0.4	ppm
Er	4A_MICP	1	0.2	ppm
Eu	4A_MICP	2	0.5	ppm
Fe	4A_MICP	3.48	0.10	%
Ga	4A_MICP	19	3	ppm
Gd	4A_MICP	4	2	ppm
Hf	4A_MICP	2	0.2	ppm
K	4A_MICP	1.89	0.13	%
La	4A_MICP	34	5	ppm
Li	4A_MICP	24	6	ppm
Lu	4A_MICP	0.1	0.02	ppm
Mg	4A_MICP	1.62	0.14	%
Mn	4A_MICP	589	60	ppm
Mo	4A_MICP	1	0.3	ppm
Na	4A_MICP	3.09	0.14	%
Nb	4A_MICP	12	1	ppm
Nd	4A_MICP	30	5	ppm
Ni	4A_MICP	47	5	ppm
P	4A_MICP	932	64	ppm
Pb	4A_MICP	7	3	ppm
Pr	4A_MICP	8	2	ppm
Rb	4A_MICP	58	10	ppm
Sb	4A_MICP	4	0.8	ppm
Sc	4A_MICP	10	1	ppm
Sm	4A_MICP	5	1	ppm
Sr	4A_MICP	741	39	ppm
Ta	4A_MICP	7	1	ppm
Tb	4A_MICP	0.5	0.1	ppm
Th	4A_MICP	5	2	ppm
Tl	4A_MICP	0.3	0.04	ppm
Tm	4A_MICP	0.1	0.03	ppm
U	4A_MICP	2	0.4	ppm
V	4A_MICP	96	7	ppm
W	4A_MICP	12	3	ppm
Y	4A_MICP	10	1	ppm
Yb	4A_MICP	0.9	0.2	ppm
Zn	4A_MICP	65	5	ppm

Major Oxides
Certified Concentrations (at two Standard Deviations)

Analyte	Method	Certified Value (μ)	[11]Two standard deviations (2s) ±	Unit
Al_2O_3	[8] XRF	14.43	0.28	%
CaO	XRF	4.75	0.093	%
Fe_2O_3	XRF	5.11	0.20	%
K_2O	XRF	2.27	0.063	%
MgO	XRF	2.83	0.087	%
MnO	XRF	0.078	0.01	%
Na_2O	XRF	4.14	0.42	%
P_2O_5	XRF	0.22	0.02	%
SiO_2	XRF	58.23	0.88	%
TiO_2	XRF	0.57	0.02	%

Uncertified Concentrations (at two Standard Deviations) on Table 3

Analyte	Method	(Mean)	[11]Two standard deviations (2s) ±	Unit
Au	[16] AL_AAS	0.2	0.018	g/t
Au	[17] FA_GRAV	0.3	0.02	g/t

1. Certified Concentrations and Uncertainties

AMIS0945 is a new standard material, developed and certified in April 2025. Table 1 gives the certified concentrations, confidence interval, combined and expanded uncertainty for the certified reference material. Table 2 shows the certified major oxides concentrations, two standard deviations, confidence interval, combined and expanded uncertainty.

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

Analyte	Method	[9]Certified Value (μ)	Number of labs (N)	Number of results (n)	k	% Relative Standard Deviation (RSD)	[10]Combined Uncertainty (u_c) ±	[11]Two standard deviations (2s) ±	[12]Confidence interval (CI) ±	[13]Expanded uncertainty (U) ±	Unit
Au	[1] Pb Collection	0.250	17	132	2.120	6	0.02	0.03	0.01	0.03	g/t
Au	[2] 2A_MICP	0.243	4	30	3.182	6	0.01	0.03	0.02	0.04	g/t
Au	[3] CL	0.244	2	15	12.706	13	0.03	0.06	0.05	[14] #0.41	g/t
Ag	[4] 4A_MICP	0.1	4	32	3.182	19	0.02	0.04	0.03	0.07	ppm
Cu	4A_MICP	50	10	79	2.262	2	1	2	0.6	3	ppm
C	[5] Combustion/LECO	1.35	8	64	2.365	3	0.046	0.092	0.038	0.11	%
S	Combustion/LECO	0.18	8	62	2.365	9	0.02	0.03	0.01	0.04	%
S	4A_MICP	0.19	8	60	2.365	8	0.02	0.03	0.01	0.04	%
LOI	[6] LOI	7.14	6	48	2.571	4	0.31	0.61	0.32	0.79	%
SG	[7] SG	2.71	4	32	3.182	3	0.081	0.16	0.13	0.26	Dimensionless
As	4A_MICP	22	9	72	2.306	20	4	9	3	10	ppm
Ba	4A_MICP	712	9	72	2.306	3	21	41	15	47	ppm
Be	4A_MICP	2	5	40	2.776	4	0.07	0.1	0.08	0.2	ppm
Bi	4A_MICP	0.06	4	32	3.182	14	0.009	0.02	0.01	0.03	ppm
Ca	4A_MICP	3.34	9	68	2.306	2	0.055	0.11	0.041	0.13	%
Ce	4A_MICP	67	5	37	2.776	9	6	12	8	17	ppm
Co	4A_MICP	16	11	88	2.228	8	1	3	0.9	3	ppm
Cr	4A_MICP	87	11	84	2.228	13	11	22	8	25	ppm
Cs	4A_MICP	4	5	40	2.776	3	0.1	0.2	0.1	0.3	ppm
Dy	4A_MICP	2	2	16	12.706	10	0.2	0.4	2	#3	ppm
Er	4A_MICP	1	3	24	4.303	11	0.1	0.2	0.3	0.5	ppm
Eu	4A_MICP	2	3	24	4.303	15	0.2	0.5	0.6	#1	ppm
Fe	4A_MICP	3.48	9	72	2.306	1	0.051	0.10	0.032	0.12	%
Ga	4A_MICP	19	7	53	2.447	8	1	3	1	3	ppm
Gd	4A_MICP	4	2	16	12.706	23	1	2	#9	#12	ppm
Hf	4A_MICP	2	4	31	3.182	5	0.1	0.2	0.2	0.4	ppm
K	4A_MICP	1.89	9	69	2.306	3	0.064	0.13	0.049	0.15	%
La	4A_MICP	34	9	72	2.306	7	2	5	2	6	ppm
Li	4A_MICP	24	10	79	2.262	13	3	6	2	7	ppm
Lu	4A_MICP	0.1	3	23	4.303	7	0.009	0.02	0.02	0.04	ppm

Table 1. Certified concentrations, two standard deviations, combined and expanded uncertainty. (Continued)

Analyte	Method	[9]Certified Value (μ)	Number of labs (N)	Number of results (n)	k	% Relative Standard Deviation (RSD)	[10]Combined Uncertainty (u_c) ±	[11]Two standard deviations (2s) ±	[12]Confidence interval (CI) ±	[13]Expanded uncertainty (U) ±	Unit
Mg	4A_MICP	1.62	9	72	2.306	4	0.072	0.14	0.054	0.165	%
Mn	4A_MICP	589	10	78	2.262	5	30	60	22	68	ppm
Mo	4A_MICP	1	5	37	2.776	16	0.2	0.3	0.2	0.5	ppm
Na	4A_MICP	3.09	7	56	2.447	2	0.071	0.14	0.061	0.17	%
Nb	4A_MICP	12	5	38	2.776	5	0.6	1	0.6	2	ppm
Nd	4A_MICP	30	3	24	4.303	9	3	5	6	11	ppm
Ni	4A_MICP	47	13	98	2.179	5	3	5	2	6	ppm
P	4A_MICP	932	7	56	2.447	3	32	64	29	78	ppm
Pb	4A_MICP	7	6	48	2.571	21	1	3	1	4	ppm
Pr	4A_MICP	8	3	24	4.303	10	0.8	2	2	3	ppm
Rb	4A_MICP	58	5	36	2.776	8	5	10	8	14	ppm
Sb	4A_MICP	4	6	48	2.571	9	0.4	0.8	0.4	1	ppm
Sc	4A_MICP	10	7	54	2.447	6	0.5	1	0.5	1	ppm
Sm	4A_MICP	5	3	24	4.303	9	0.5	1	1	2	ppm
Sr	4A_MICP	741	9	71	2.306	3	19	39	14	45	ppm
Ta	4A_MICP	7	5	39	2.776	8	0.6	1	0.7	2	ppm
Tb	4A_MICP	0.5	4	32	3.182	11	0.05	0.1	0.08	0.2	ppm
Th	4A_MICP	5	5	39	2.776	16	0.8	2	1	2	ppm
Tl	4A_MICP	0.3	6	47	2.571	6	0.02	0.04	0.02	0.05	ppm
Tm	4A_MICP	0.1	2	16	12.706	10	0.01	0.03	0.1	#0.2	ppm
U	4A_MICP	2	5	40	2.776	9	0.2	0.4	0.2	0.5	ppm
V	4A_MICP	96	9	72	2.306	4	4	7	3	8	ppm
W	4A_MICP	12	8	64	2.365	12	1	3	1	3	ppm
Y	4A_MICP	10	7	51	2.447	6	0.6	1	0.6	1	ppm
Yb	4A_MICP	0.9	2	15	12.706	9	0.08	0.2	0.7	#1	ppm
Zn	4A_MICP	65	12	91	2.201	4	3	5	2	6	ppm

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

Analyte	Method	[9]Certified Value (μ)	Number of labs (N)	Number of results (n)	k	% Relative Standard Deviation (RSD)	[10]Combined Uncertainty (u_c) ±	[11]Two standard deviations (2s) ±	[12]Confidence interval (CI) ±	[13]Expanded uncertainty (U) ±	Unit
Al_2O_3	[8] XRF	14.43	6	48	2.571	1	0.14	0.28	0.13	0.36	%
CaO	XRF	4.75	6	48	2.571	1	0.046	0.093	0.043	0.12	%
Fe_2O_3	XRF	5.11	6	48	2.571	2	0.10	0.20	0.10	0.26	%
K_2O	XRF	2.27	6	48	2.571	1	0.031	0.063	0.031	0.081	%
MgO	XRF	2.83	6	48	2.571	2	0.043	0.087	0.043	0.11	%
MnO	XRF	0.078	5	39	2.776	8	0.007	0.01	0.007	0.02	%
Na_2O	XRF	4.14	6	45	2.571	5	0.21	0.42	0.24	0.54	%
P_2O_5	XRF	0.22	5	40	2.776	4	0.009	0.02	0.009	0.03	%
SiO_2	XRF	58.23	8	61	2.365	0.8	0.44	0.88	0.37	1.04	%
TiO_2	XRF	0.57	5	40	2.776	1	0.008	0.02	0.007	0.02	%

1. *Pb collection is Fire Assay Pb Collection with either ICPOES/ICPMS/AAS finish.*
2. *2A_MICP is a Two-acid digestion with either ICPOES/ICPMS/AAS finish.*
3. *CL is Cyanide Leach.*
4. *4A_MICP is a Four-acid digestion with either ICPOES/ICPMS/AAS finish.*
5. *Combustion/LECO.*
6. *LOI is Loss on Ignition.*
7. *SG is Specific Gravity.*
8. *XRF is X-ray Fluorescence.*
9. *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.*
10. *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. (u_c)*
11. *Two standard deviations (2s) is u_c x 2*
12. *Confidence interval at 95% level of confidence.*
13. *Expanded uncertainty (U) at a confidence level of 95% is determined by multiplication of the combined uncertainty (u_c) 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=0.5%*
14. *# denotes U> μ or CI> μ. AMIS advises that U and CI should not be used in this instance.*

2. Uncertified element statistics

Uncertified element statistics are shown in Table 3.

Table 3. Uncertified element concentrations statistics (Before the removal of outliers).

Element	Generic Method	Number of results (n)	Mean	Standard Deviation (SD) ±	Relative Standard Deviation (RSD) %	Unit
Ag	[15] 3A_MICP	8	1.2	0.05	4	ppm
Al	4A_MICP	72	69568.5	5615.5	8	ppm
Al_2O_3	4A_MICP	8	14.2	0.3	2	%
Au	[16] AL_AAS	8	0.2	0.009	4	g/t
Au	[17] FA_GRAV	8	0.3	0.01	4	g/t
Ba	[18] 4A_ICPES	8	714.9	2.7	0.4	ppm
BaO	XRF	16	0.09	0.02	21	%
CaO	4A_MICP	8	4.6	0.02	0.5	%
Cd	4A_MICP	32	0.3	0.3	96	ppm
Co	4A_ICPES	8	16.7	0.1	0.7	ppm
Cr_2O_3	XRF	32	0.02	0.004	25	%
Fe	XRF	8	36262.5	302.1	0.8	ppm
Fe_2O_3	4A_MICP	8	5.1	0.02	0.4	%
Ga	4A_ICPES	8	14.7	0.6	4	ppm
Ge	4A_MICP	16	0.7	0.4	53	ppm
Ho	4A_MICP	16	0.4	0.03	7	ppm
In	4A_MICP	15	0.03	0.007	20	ppm
K2O	4A_MICP	8	2.2	0.01	0.4	%
La	4A_ICPES	8	28.2	0.2	0.9	ppm
Li	4A_ICPES	8	13.1	0.07	0.5	ppm
LOI	XRF	24	6.9	0.3	4	%
MgO	4A_MICP	8	2.8	0.009	0.3	%
Mn_3O_4	XRF	8	0.08	0.005	6	%

Table 3 (Continued)

Element	Generic Method	Number of results (n)	Mean	Standard Deviation (SD) ±	Relative Standard Deviation (RSD) %	Unit
MnO	4A_MICP	8	0.07	0.001	1	%
Mo	4A_ICPES	8	1.1	0.06	5	ppm
Moisture	LOI	7	0.6	0.008	1	%
Moisture	[19] Moisture	40	0.5	0.3	65	%
Na_2O	4A_MICP	8	3.8	0.02	0.4	%
Nb	4A_ICPES	8	7.5	0.04	0.5	ppm
P	XRF	8	950.0	18.5	2	ppm
P_2O_5	4A_MICP	8	0.2	0.001	0.5	%
Re	4A_MICP	1	0.002	*	*	ppm
S	XRF	8	0.2	0.002	1	%
Sc	4A_ICPES	8	9.9	0.04	0.4	ppm
Se	4A_MICP	1	0.6	*	*	ppm
Sn	4A_MICP	32	2.6	1.9	74	ppm
SO_3	XRF	40	0.4	0.07	17	%
SrO	XRF	8	0.08	*	*	%
Te	4A_MICP	15	6.1	5.8	96	ppm
Ti	4A_MICP	72	2841.8	267.9	9	ppm
TiO_2	4A_MICP	8	0.5	0.01	2	%
Tl	4A_ICPES	8	10.3	0.1	1	ppm
V_2O_5	XRF	24	0.02	0.004	17	%
W	4A_ICPES	8	6.7	0.2	3	ppm
Zn	[20] 4A_AAS	8	64.9	0.6	1	ppm
Zr	4A_MICP	56	82.8	17.4	21	ppm

* denotes that the results were exactly the same and SD and RSD% could not be calculated
# denotes that SD>Mean

15. *3A_MICP is a Multi-acid digestion with either ICPOES/ICPMS/AAS finish*
16. *AL_AAS is Acid leach with AAS finish.*
17. *FA_GRAV is Fire Assay Pb Collection with Gravimetry finish.*
18. *4A_ICPES is a Multi-acid digestion with ICPOES finish.*
19. *Moisture is residual moisture content.*
20. *4A_AAS is a Multi-acid digestion with AAS finish.*

3. Statistical Comparison of Means

A comparison of means for replicate data for the same element concentration determined by different analytical methods is 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 (Table 4 and 5). This gives the analyst confidence in the certified values reported by different analytical methods on the same analyte.

Table 4. The results of a two-sample equal or unequal variance t-test (two-tailed) data sets in which different analytical methods /instrumentation were used.

Method	Certified value	Method	Certified Value	F-test Outcome	p-value (t-test)	t-test Outcome
S Combustion/ LECO	0.178 %	S 4A_MICP	0.193 %	p=0.480. Accept H_0. Variances are Equal	0.087	p>0.05. Accept H_0; certified values are equal

Table 5. Comparison of the mean values for which three different analytical methods are used to determine their concentration.

Method	Certified Value (g/t)	Method	Certified Value	Method	Certified Value	ANOVA outcome
Au Pb Collection	0.250 g/t	Au 2A_MICP	0.243 g/t	Au CL	0.244 g/t	p=0.513. Accept H_0. Means are equal.

4. Intended Use

AMIS0945 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 2). 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 5). 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 took part in the exercise.

5. Abbreviations and Symbols

Abbreviations and symbols used in this document are shown in Table 6.

Table 6. 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
Bq	The becquerel is the SI derived unit of radioactivity.
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
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

Abbreviation/Symbol	Description
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 1×10^{-6} of a meter

6. Uncertified Concentration Values

Table 3 gives uncertified concentrations for other elements present in the CRM.

7. Units

All results certified for major oxides are reported as oxides in percentages. All results certified for major elements analyses reported in percentages or ppm. Results for Au is reported in g/t. 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.

8. 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

9. Origin of Material

The Akyem Gold Mine is a prominent gold mining operation located in the Eastern Region of Ghana, near the town of New Abirem. It is owned and operated by Newmont Corporation, one of the largest gold producers in the world.

10. Approximate Mineral and Chemical Composition

The Gold mineralization is hosted within a northeast-trending belt of folded, metamorphosed volcanic and sedimentary rocks and is controlled by a series of anastomosing brittle fracture zones. The brittle fractures acted as plumbing, allowing gold-rich fluids access to host lithologies with favorable fracture-enhanced permeability. The key minerals include Chlorite, Dolomite/Ankerite, Kaolinite, Muscovite/Sericite, Plagioclase, Pyrite, Quartz and Rutile.

11. 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 recognized and accounted for. X-ray diffraction is effective in such a way that it allows the identification of different phases of compounds that are identical in chemistry, but have distinctly different 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.

Sample preparation for X-Ray Diffraction

The material submitted was prepared for XRD analysis using a backloading preparation method.
It was analysed with a PANalytical Aeris diffractometer with PIXcel detector and fixed slits with Fe filtered Co-Kα radiation. The phases were identified using X'Pert Highscore plus software and PAN-ICSD and ICDD PDF4 2016 databases. The relative phase amounts (weight %) were estimated using the Rietveld method. The amorphous phases such as glass do not produce a pattern and are excluded in the quantification, but the amount of amorphous component could be quantified by an internal or external standard method.

Comment

- Mineral names may not reflect the actual compositions of minerals identified, but rather the mineral group.
- Smectite, lizardite (serpentine), vermiculite, chlorite and kaolinite peaks overlap and further test would be necessary to distinguish. Identification is largely based on peak shapes and positions.
- Due to preferred orientation and crystallite size effects, results may not be as accurate as shown.
- Traces of additional phases may be present. Quantities below 0.5% may be unreliable.
- Amorphous phases, if present, were not taken into consideration during quantification.

Table 7. Results of XRD analysis.

Mineral	Unit	Composite
Quartz	wt%	28.2
Plagioclase	wt%	50.9
Dolomite	wt%	8
Chlorite	wt%	1.7
Muscovite	wt%	10
Calcite	wt%	1.2
Total		100%

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12. Health and Safety

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

13. Method of Preparation

The particle size distribution for this material was shown to have a nominal top size of 53µm (99.07% passing 53µm). The procedure of preparation in brief is as follows: the material was crushed, dry-milled and air-classified to <53µm. 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 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.

14. Particle Size Determination

The sample has been analysed using sieve analysis on a wet sample. The particles are passed through multiple sieves of different sizes. The results for this standard are presented in Table 8.

Table 8. Particle Size Determination.

Size (µm)	Vol. Under %
<38µm	97.43%
<53µm	99.07%
<63µm	99.88%
<75µm	99.93%
<90µm	99.96%
<106µm	99.97%
<150µm	99.98%

For informational purposes only

15. 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 to convert any obtained assay values to an air-dry basis (see Appendix 6 for an example calculation).

16. 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.

17. Methods of Analysis Requested

The following methods of analysis were requested:

a) Au-Pb collection finished with either ICP-OES or ICP-MS or AAS.
b) Au-Pb Collection with gravimetric finish.
c) Au-2 acid digestion with any finish.
d) Multi element scan to include all elements especially Ag and Cu- 4-acid total digestion including HF finished with either ICP-OES or ICP-MS or AAS.
e) LOI and all major oxides with XRF finish (Please specify the temperature for LOI).
f) SG – gas pycnometer.
g) S and C Combustion/LECO.
h) Moisture.

18. Information Requested of Participating Laboratories

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

a) All aliquots used for all determinations to be stated.
b) All results for major elements to be reported as oxides in percentage (%).
c) All results for multi-element scans and fusion to be reported in parts per million (ppm).
d) All results for Au to be reported in grams per ton (g/t).
e) All QC data to include replicates, blanks and any certified reference material used.
f) All Round robin samples must be treated the same as routine test samples.
g) All results must be reported to maximum decimal places i.e., dependent on laboratories capabilities.
h) Moisture correction factor to be used to calculate and determine the moisture content. The moisture correction factor is to be stipulated when reporting results.
i) Results to be recorded onto the PT Reporting template provided (Include uncertainty of measurements) together with a copy of the PDF.
j) Ensure correct PPE is used i.e., gloves, dust masks and protective clothing when handling and analyzing the samples.
k) Storage and analysis shall be conducted under controlled environmental conditions.
l) Ensure all safety requirements as per your laboratory requirements are adhered to.
m) Samples shall be retained for 60 days after completion of analysis, prior to being discarded.

19. 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 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 Harmonized 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 are re-calculated using all remaining data (Thompson, 2008; Carr, 2011) (see Appendix 2).

20. Two Standard Deviations

AMIS reports two-standard deviations (2s) with all certified values. Two -standard deviations are calculated using the expression:

$$Two\ standard\ deviations = 2\ (u_c)$$

Where u_c is the standard combined uncertainty (see Appendix, equation [14]).

21. Confidence Interval

AMIS reports a confidence interval (*CI*) with all certified values. Confidence interval as used by AMIS is:

$$Confidence\ Interval\ (CI) = \frac{(t_{critical})s}{\sqrt{N}}$$

Where, N is the number of laboratories (accepted laboratory data), $t_{critical}$ is a two-tailed value for $N - 1$ degrees of freedom *(df)* and *s*, is the standard deviation of the accepted laboratory means. A two-tailed critical value is found for *N* -1 degrees of freedom from either a *t*-distribution table (Appendix 8) or MS Excel as =TINV (5%, df).

22. Expanded Uncertainty

ANOVA gives an estimate of the repeatability and the reproducibility of the data accepted for certification of the candidate reference material (see equations, [15] and [16], in the Appendix). Therefore, random variables (e.g., subsampling, instrument effects, interferences, operators and measurement conditions) that occur during the analysis of the candidate reference material by the various laboratories is considered. This approach does not necessarily quantify each individual source of uncertainty; however, the combined effect of random uncertainties is assessed (Ramsey & Ellison, 2007). A combined standard uncertainty is calculated from equation [14], which when multiplied by the t-critical value for *N*-1 laboratories, gives an *expanded uncertainty* at a 95% level of confidence. The expanded uncertainty is a measure of the doubt around the certified value at a level of 95% confidence. The expanded uncertainty is used in the validation of accuracy (see equation [18]).

23. Confidence Interval and Expanded Uncertainty

A combined standard uncertainty will be greater than a combined *CI*. This is because ANOVA considers the within-lab repeatability (that is repeatability within each lab group) as well as the repeatability between each lab data set. This attends to random variables that contribute to the measurement of uncertainty, during the analysis of the test sample at the participating laboratories. The within-lab repeatability and the between lab repeatability is combined as the square root of the sum of squares of these two values giving a combined standard uncertainty, at a 68% confidence level. Multiplying the combined standard uncertainty by the t-critical value for *N*-1, gives the expanded uncertainty at 95% level of confidence. It is recommended that the procedure described in Appendix 5, *"Using the CRM in Quality Contro*l" be used, in setting the limits of the CRM. Table 9 below shows mean gold values obtained by fire assay lead collection, for nine different laboratories, the confidence interval, two-standard deviations and expanded uncertainty.

Table 9. Example of replicate assay data in which the *CI*, 2*s* and *U* are shown.

Lab No.	Mean Au (g/t)
1	0.268
2	0.273
3	0.270
4	0.288
5	0.274
6	0.256
7	0.263
8	0.258
9	0.288

CI	0.0088
2s	0.031
U	0.04

24. Participating Laboratories

The laboratories provided timeous results are:

1. Actlabs Skyline Peru SAC
2. AL AMRI LABS
3. ALS Kansanshi Mine, Zambia
4. Argetest Mineral Processing, R&D and Analysis Services
5. Bureau Veritas Adelaide GeoAnalytical Laboratory
6. Couer Mexicana S.A de C.VLaboratorio Químico
7. Freda Rebecca Gold Mine
8. Geoangol
9. Intertek Minerals (Orezone Lab)
10. Intertek Minerals Ltd Somisa SA Minesite Lab (Burkina Faso)
11. Intertek Perth
12. Labomine
13. Laboratory of Analytical Chemistry-INGEMMET
14. Misenge Environmental and Technical Service
15. Ready Lead Assay Laboratory
16. SGS Mineral Services Lakefield (Canada)
17. SGS Philippines, Inc. - Masbate Laboratory
18. SGS Vancouver (Canada)
19. Shiva Analyticals India
20. Stewart Assay and Environmental Laboratories LLC
21. TEA Metallurgical Laboratories (PTY) LTD.

25. Accepted Assay Data

Data from the 21 laboratories used for certification are set out in Table 10.

Table 10. Data used to calculate the certified values after removal of outliers.

Pb Collection	Pb Collection	Pb Collection	Pb Collection	2A_MICP	CL
Au	Au	Au	Au	Au	Au
g/t	g/t	g/t	g/t	g/t	g/t
0.26	0.24	0.28	0.26	0.24	0.19
0.26	0.23	0.25	0.26	0.26	0.30
0.27	0.26	0.25	0.26	0.24	0.24
0.24	0.24	0.25	0.27	0.25	0.26
0.24	0.25	0.25	0.27	0.24	0.25
0.25	0.24	0.28	0.27	0.26	0.29
0.24	0.22	0.26	0.26	0.25	0.22
0.26	0.24	0.28	0.26	0.25	0.23
0.25	0.24	0.28	0.26	0.21	0.24
0.26	0.24	0.27	0.24	0.24	0.22
0.25	0.25	0.25	0.23	0.23	0.23
0.27	0.24	0.23	0.24	0.24	0.23
0.26	0.24	0.23	0.24	0.23	0.27
0.25	0.25	0.24	0.23	0.23	0.23
0.26	0.23	0.23	0.25	0.24	0.28
0.24	0.25	0.22	0.24	0.25	
0.28	0.24	0.23	0.23	0.24	
0.28	0.24	0.22	0.22	0.23	
0.26	0.24	0.23	0.22	0.26	
0.26	0.24	0.22	0.27	0.26	
0.26	0.25	0.22	0.25	0.24	
0.22	0.25	0.26	0.26	0.24	
0.26	0.26	0.22	0.25	0.24	
0.24	0.25	0.22	0.24	0.26	
0.24	0.25	0.24	0.23	0.27	
0.25	0.25	0.25	0.26	0.27	
0.22	0.25	0.28	0.25	0.23	
0.27	0.25	0.27	0.24	0.25	
0.24	0.26	0.26	0.25	0.25	
0.25	0.26	0.28	0.26	0.24	
0.23	0.25	0.28	0.26		
0.26	0.25	0.27	0.25		
0.25	0.24	0.27	0.25		

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	Combustion /LECO	Combustion/LECO	Combustion/ LECO	Combustion/ LECO
Ag	Cu	Cu	C	C	S	S
ppm	ppm	ppm	ppm	ppm	%	%
0.13	48.37	49.07	14120.00	13200.00	0.17	0.18
0.11	48.82	50.05	14060.00	13300.00	0.17	0.21
0.10	49.22	49.94	14160.00	13200.00	0.17	0.21
0.15	49.49	49.59	14180.00	13200.00	0.17	0.21
0.11	48.93	49.37	14210.00	13300.00	0.17	0.21
0.13	49.67	49.97	14090.00	13200.00	0.17	0.21
0.11	48.74	49.64	14280.00	13300.00	0.17	0.21
0.10	48.80	49.00	14100.00	13300.00	0.17	0.17
0.10	50.00	48.00	14100.00	13400.00	0.17	0.17
0.10	51.30	49.00	13900.00	13200.00	0.17	0.18
0.12	50.30	48.00	14000.00	13600.00	0.17	0.17
0.10	49.90	48.00	13900.00	13400.00	0.17	0.18
0.09	50.50	48.00	14100.00	13400.00	0.17	0.18
0.10	50.30	48.00	13900.00	13200.00	0.17	0.18
0.10	51.20	51.00	14100.00	13400.00	0.17	0.18
0.10	50.10	52.00	14000.00	13400.00	0.17	0.15
0.10	49.73	50.00	13600.00	13400.00	0.17	0.15
0.09	49.70	52.00	13400.00	13300.00	0.17	0.16
0.10	49.51	50.00	13700.00	13300.00	0.18	0.16
0.10	49.82	52.00	13600.00	13200.00	0.18	0.16
0.09	49.80	50.00	13500.00	13200.00	0.17	0.16
0.12	49.72	50.00	13400.00	13300.00	0.18	0.15
0.10	49.87	52.00	13600.00	13200.00	0.17	0.16
0.10	49.60	52.00	13600.00	13200.00	0.17	0.19
0.15	50.00	51.00	13704.00	12700.00	0.18	0.18
0.13	51.00	52.00	13709.00	12700.00	0.18	0.19
0.14	50.00	51.00	13697.00	12700.00	0.19	0.19
0.14	50.00	51.00	13716.00	12800.00	0.18	0.19
0.14	50.00	52.00	13702.67	12800.00	0.18	0.18
0.15	51.00	52.00	13771.00	12800.00	0.18	0.19
0.14	50.00	51.00	13708.67	12700.00	0.18	0.18
0.14	50.60	49.80	13684.00	12700.00		
	50.90	48.20				
	49.70	51.10				
	51.10	49.10				
	50.70	50.40				
	51.60	48.10				
	51.50	51.20				
	50.70	51.50				
	48.83					

Assay Data (Continued)

XRF Al_2O_3 %	XRF CaO %	XRF Fe_2O_3 %	XRF K_2O %	XRF MgO %	XRF MnO %	XRF Na_2O %	XRF P_2O_5 %	XRF SiO_2 %	XRF SiO_2 %	XRF TiO_2 %
14.54	4.73	5.13	2.27	2.82	0.080	4.18	0.22	58.70	57.96	0.59
14.41	4.69	5.12	2.25	2.77	0.080	4.15	0.21	58.43	57.81	0.58
14.36	4.67	5.11	2.25	2.79	0.080	4.14	0.21	58.18	57.63	0.58
14.31	4.69	5.09	2.24	2.78	0.080	4.14	0.21	58.00	57.80	0.57
14.36	4.71	5.12	2.23	2.80	0.080	4.13	0.21	58.55	58.23	0.57
14.33	4.65	5.08	2.26	2.79	0.080	4.13	0.21	58.08	58.15	0.58
14.30	4.68	5.10	2.24	2.79	0.080	4.13	0.21	58.10	58.03	0.57
14.35	4.70	5.10	2.22	2.78	0.080	4.11	0.22	58.16	57.96	0.58
14.50	4.75	5.10	2.25	2.81	0.080	3.75	0.22	59.10	58.21	0.57
14.50	4.75	5.14	2.23	2.81	0.080	3.78	0.23	59.10	57.83	0.57
14.50	4.74	5.19	2.24	2.80	0.080	3.74	0.22	59.10	58.23	0.56
14.50	4.75	5.16	2.23	2.81	0.080	3.76	0.22	58.70	58.33	0.57
14.50	4.75	5.10	2.24	2.82	0.080	3.73	0.21	59.10	57.89	0.57
14.50	4.78	5.14	2.23	2.83	0.080	4.13	0.22	58.60		0.58
14.50	4.76	5.16	2.25	2.82	0.080	4.16	0.21	58.70		0.57
14.50	4.74	5.10	2.22	2.80	0.070	4.17	0.21	58.60		0.57
14.46	4.78	5.10	2.24	2.82	0.070	4.14	0.21	58.60		0.58
14.46	4.79	5.09	2.26	2.82	0.070	4.15	0.21	58.40		0.57
14.48	4.78	5.09	2.25	2.83	0.080	4.17	0.21	58.60		0.58
14.41	4.72	5.07	2.23	2.81	0.070	4.15	0.21	58.40		0.57
14.45	4.78	5.09	2.24	2.81	0.070	4.15	0.21	58.50		0.57
14.48	4.77	5.10	2.25	2.83	0.070	4.25	0.21	58.54		0.57
14.48	4.79	5.07	2.26	2.82	0.070	4.24	0.21	58.50		0.57
14.41	4.73	5.06	2.24	2.82	0.080	4.26	0.22	58.46		0.57
14.29	4.75	5.12	2.27	2.85	0.090	4.24	0.23	58.40		0.56
14.22	4.76	5.20	2.27	2.84	0.090	4.22	0.22	58.45		0.55
14.28	4.75	5.11	2.28	2.85	0.080	4.24	0.23	58.54		0.57
14.24	4.74	5.08	2.27	2.84	0.090	4.24	0.23	58.50		0.57
14.24	4.76	5.09	2.27	2.84	0.080	4.25	0.22	58.36		0.56
14.28	4.74	5.10	2.27	2.86	0.090	4.10	0.23	58.03		0.57
14.23	4.74	5.12	2.28	2.83	0.080	4.12	0.23	57.98		0.56
14.20	4.74	5.07	2.27	2.85	0.070	4.10	0.23	57.98		0.57
14.70	4.81	4.94	2.31	2.90	0.070	4.10	0.20	58.00		0.59
14.70	4.81	4.99	2.32	2.87	0.070	4.12	0.21	58.04		0.58
14.60	4.81	4.95	2.31	2.89	0.070	4.11	0.20	57.92		0.57
14.60	4.82	4.94	2.30	2.91	0.070	4.10	0.22	57.99		0.58
14.50	4.83	4.95	2.31	2.92	0.080	4.10	0.22	58.00		0.58
14.60	4.81	4.96	2.30	2.93	0.080	4.45	0.20	57.71		0.56
14.60	4.81	4.93	2.32	2.91	0.080	4.45	0.22	57.52		0.58
14.70	4.83	4.93	2.33	2.92		4.45	0.21	57.54		0.58
14.60	4.77	5.18	2.30	2.79		4.46		57.58		
14.47	4.78	5.22	2.29	2.84		4.46		57.51		
14.29	4.76	5.25	2.28	2.78		4.47		57.72		
14.45	4.76	5.30	2.29	2.82		4.46		57.56		
14.39	4.76	5.21	2.30	2.80		4.46		57.57		
14.30	4.65	5.29	2.26	2.83				57.89		
14.42	4.74	5.28	2.29	2.83				57.69		
14.38	4.78	5.25	2.29	2.81				57.96		

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
As	As	Ba	Ba	Be	Bi	Ca	Ca	Ce
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
23.00	30.77	691.00	688.30	1.58	0.050	32980.00	33000.00	69.91
24.00	27.95	695.00	681.90	1.63	0.050	33170.00	33000.00	69.36
24.00	29.26	685.00	686.80	1.60	0.050	32770.00	33200.00	68.49
25.00	30.79	684.00	683.30	1.62	0.050	32540.00	32800.00	70.95
24.00	28.14	688.00	685.10	1.59	0.050	32800.00	33700.00	69.15
24.00	30.76	691.00	684.50	1.66	0.050	33010.00	33400.00	70.34
24.00	28.23	694.00	686.70	1.67	0.050	33060.00	33800.00	68.39
25.00	29.47	688.00	688.30	1.63	0.050	32910.00	33600.00	68.54
11.30	23.00	724.17	708.66	1.50	0.060	33705.61	32500.00	69.77
12.61	22.00	725.63	707.88	1.51	0.070	33087.16	32300.00	68.96
13.59	22.00	727.77	704.82	1.52	0.060	33858.38	32400.00	67.60
14.90	22.00	726.38	707.38	1.51	0.070	33873.26	32300.00	67.69
14.12	22.00	724.83	705.73	1.51	0.060	33723.53	33900.00	69.17
14.52	23.00	723.52	700.56	1.51	0.060	33723.53	34000.00	68.21
13.16	24.00	727.10	706.55	1.50	0.070	33507.18	34200.00	68.09
12.00	23.00	721.64	705.96	1.51	0.060	33760.73	33900.00	68.75
23.10	17.98	714.00	713.00	1.63	0.067	33200.00	34000.00	72.62
24.10	17.36	716.00	713.00	1.57	0.068	33200.00	33700.00	71.26
23.00	16.63	723.00	709.00	1.51	0.072	33200.00	34000.00	70.49
23.10	18.42	713.00	709.00	1.52	0.068	33000.00	34500.00	71.55
23.00	18.97	718.00	710.00	1.50	0.071	33400.00	34000.00	73.03
23.10	20.89	719.00	711.00	1.56	0.072	32900.00	34100.00	72.52
23.80	18.79	716.00	712.00	1.53	0.065	33200.00	33900.00	72.19
23.40	19.98	716.00	711.00	1.46	0.070	33300.00	33800.00	72.42
23.00	23.12	750.00	704.00	1.54	0.059	33754.00	33900.00	55.64
23.55	24.51	750.80	695.00	1.53	0.056	32763.00	34000.00	54.02
23.98	24.27	727.00	696.00	1.57	0.061	33671.00	33800.00	56.44
23.67	23.94	743.30	694.00	1.56	0.058	32748.00	34000.00	54.42
23.22	22.40	750.00	698.00	1.55	0.055	33437.00		55.13
23.60	23.12	753.00	696.00	1.57	0.057	33138.00		68.30
22.86	22.93	755.00	702.00	1.56	0.059	32757.00		70.20
23.50	21.94	741.60	697.00	1.51	0.059	32781.00		70.10
22.00		742.00		1.45		33451.52		71.00
22.00		740.00		1.45		33191.06		70.50
21.00		736.00		1.45		34222.98		69.90
21.00		723.00		1.45		34253.07		72.70
21.00		733.00		1.45		33700.40		71.80
22.00		726.00		1.45		33707.88		
22.00		732.00		1.45		33570.61		
24.00		728.00		1.44		33887.36		

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Co	Co	Cr	Cr	Cs	Dy	Er	Eu	Fe
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
17.11	13.00	74.00	98.52	3.60	2.34	1.17	1.72	34600.00
16.97	13.00	72.00	100.00	3.71	2.35	1.17	1.60	34700.00
17.47	13.00	68.00	98.71	3.53	2.36	1.16	1.58	34400.00
17.06	13.00	74.00	99.13	3.67	2.34	1.16	1.59	34200.00
16.81	13.00	72.00	100.00	3.63	2.37	1.13	1.63	34400.00
17.67	14.00	64.00	90.00	3.64	2.33	1.13	1.57	34700.00
17.16	14.00	70.00	90.00	3.74	2.35	1.13	1.66	34800.00
17.46	14.00	68.00	80.00	3.56	2.39	1.10	1.59	34700.00
16.70	16.75	77.18	80.00	3.34	2.09	0.93	1.24	34374.49
16.80	17.16	77.76	90.00	3.36	2.01	0.91	1.23	34764.36
16.90	16.88	76.51	90.00	3.59	2.09	0.94	1.29	34684.82
17.50	16.16	77.64	90.00	3.41	2.06	0.90	1.23	34394.79
17.00	16.88	76.96	100.22	3.42	2.03	0.93	1.29	34511.19
17.20	17.06	76.58	99.25	3.34	2.06	0.94	1.25	34297.79
17.20	16.74	75.96	97.56	3.39	2.04	0.90	1.21	34791.52
17.20	16.04	77.66	101.23	3.35	2.05	0.95	1.27	34424.86
15.72	16.00	64.57	98.37	3.68		1.05	1.60	35000.00
15.61	16.00	63.63	104.74	3.67		1.05	1.65	35100.00
15.59	16.00	62.56	101.43	3.60		1.05	1.65	34900.00
15.52	16.00	64.09	100.45	3.65		1.05	1.70	34700.00
15.10	17.00	65.00	91.00	3.64		1.10	1.70	35200.00
15.13	16.00	94.00	92.00	3.74		1.10	1.70	34800.00
15.67	17.00	87.00	91.00	3.66		1.10	1.70	34900.00
15.78	17.00	83.00	91.00	3.69		1.05	1.65	35100.00
16.80	15.75	91.00	91.00	3.48				35700.00
16.80	15.78	97.00	90.00	3.48				34900.00
16.30	16.07	93.00	91.00	3.56				35500.00
16.40	15.57	94.00	91.00	3.54				34500.00
16.80	15.77	93.00	89.00	3.53				35200.00
16.60	15.65	92.71	89.00	3.49				34900.00
16.80	15.71	92.65	91.00	3.49				34500.00
16.70	15.52	93.00	88.00	3.53				34600.00
16.79	17.00	93.05	89.00	3.50				34465.58
17.07	17.00	92.53	89.00	3.50				34620.40
17.37	16.00	93.82	88.00	3.60				35382.75
17.13	17.00	92.11	90.00	3.60				35357.99
17.14	17.00	93.44		3.60				34861.36
16.86	17.00	98.00		3.60				34801.08
16.63	17.00	97.00		3.70				34806.12
17.15	17.00	90.00		3.60				35158.62
	19.00	91.00						34200.00
	19.00	95.00						34600.00
	18.00	94.00						33600.00
	18.00	94.00						33800.00
	19.00	90.00						33400.00
	19.00	98.93						34100.00
	19.00	98.01						33600.00
	18.00	97.13						

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Fe	Ga	Ga	Gd	Hf	K	K	La	La
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
34300.00	18.09	17.80	4.92	2.07	19500.00	18400.00	33.44	33.00
35000.00	18.38	18.00	4.95	2.20	19500.00	18700.00	32.86	33.80
35100.00	18.06	18.00	4.90	2.08	19300.00	18800.00	32.46	33.50
34300.00	18.59	18.00	4.92	2.16	19300.00	18300.00	34.44	34.40
34800.00	18.27	18.20	4.83	2.16	19400.00	18300.00	33.94	34.20
35300.00	18.42	17.80	4.88	2.16	19400.00	18900.00	34.25	33.50
34400.00	18.58	19.20	4.80	2.13	19500.00	18800.00	34.02	35.20
34600.00	18.36	18.80	4.87	2.04	19400.00	18900.00	33.43	34.60
34400.00	16.37	22.00	3.43	2.17	19222.00	17800.00	31.96	35.31
35400.00	16.33	21.00	3.38	2.22	19060.00	17800.00	31.95	34.54
35200.00	16.73	21.00	3.53	1.98	19993.00	17700.00	31.65	34.43
35200.00	16.78	21.00	3.61	2.13	19001.00	18000.00	31.52	34.02
35600.00	16.75	22.00	3.62	2.03	19196.00	17600.00	31.77	33.86
35900.00	16.72		3.52	2.13	19954.00	17600.00	31.87	34.85
35300.00	16.84		3.41	2.08	19235.00	19000.00	31.86	34.37
36100.00	16.76		3.58	2.09	19145.00	19000.00	31.67	33.55
35200.00	18.58			2.10	18600.00	19100.00	35.54	36.00
34800.00	18.19			2.12	18500.00	19400.00	35.34	36.00
34800.00	17.19			2.19	18500.00	18600.00	34.33	36.00
35100.00	17.63			2.16	18600.00	18700.00	35.39	36.00
34800.00	17.68			2.10	18400.00	19300.00	36.20	37.00
35200.00	17.94			2.22	18300.00	19500.00	35.79	35.00
34900.00	18.69			2.12	18200.00	19800.00	35.73	36.00
34700.00	17.53			2.19	18400.00	19800.00	35.52	36.00
35000.00	18.27			2.20	19225.00	20200.00	35.00	38.00
	17.98			2.40	18601.00	19700.00	36.00	37.50
	18.97			2.40	19053.00	20200.00	35.00	37.40
	18.74			2.40	18555.00	20000.00	34.00	38.20
	18.17			2.20	19005.00	19600.00	34.00	37.30
	17.79			2.20	18742.00		34.00	37.70
	17.74			2.40	18526.00		35.00	39.40
	18.52				18548.00		33.00	39.10
	19.00				18384.59		29.98	
	20.00				18441.04		29.97	
	19.00				18849.10		29.79	
	21.00				18819.91		30.29	
	19.00				18565.67		29.89	
	19.00				18583.77		30.62	
	20.00				18590.67		29.96	
	19.00				18650.06		29.90	

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Li	Li	Lu	Mg	Mg	Mn	Mn	Mo	Na
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
24.80	24.43	0.13	16140.00	16200.00	533.29	611.00	0.89	31560.00
25.00	25.34	0.13	16200.00	16900.00	527.85	594.00	0.91	31750.00
24.70	24.78	0.13	16040.00	16500.00	529.18	607.00	0.88	31390.00
24.50	24.66	0.13	15900.00	16600.00	531.10	602.00	0.91	31220.00
24.70	25.46	0.13	16040.00	16700.00	531.77	596.00	0.91	31500.00
24.90	24.49	0.13	16090.00	16900.00	532.02	595.00	0.93	31600.00
25.10	25.05	0.12	16160.00	16900.00	531.07	591.21	0.90	31710.00
24.60	22.00	0.13	16100.00	16800.00	533.55	597.79	0.86	31570.00
18.13	22.00	0.13	15892.10	16300.00	606.00	609.38	1.21	30176.27
18.22	22.00	0.11	15567.20	16300.00	613.00	606.16	1.11	30144.34
18.17	22.00	0.13	15451.30	16300.00	601.00	594.43	1.17	30891.99
18.15	22.00	0.13	15389.55	16100.00	599.00	600.85	1.11	30204.26
18.19	22.00	0.12	15600.45	16400.00	601.00	596.27	1.18	30272.06
18.18	22.00	0.13	15387.65	16300.00	606.00	603.65	1.15	30455.85
18.16	22.00	0.12	15817.05	16300.00	610.00	544.10	1.22	30303.99
18.11	23.93	0.12	15548.20	16300.00	604.00	546.00	1.20	30333.86
18.33	24.34	0.14	16200.00	16800.00	591.77	544.80	0.90	31900.00
17.73	23.35	0.14	16200.00	16900.00	589.71	541.70	0.90	31900.00
17.91	23.00	0.14	16200.00	17000.00	589.25	551.20	0.80	31800.00
19.51	23.57	0.14	16300.00	17300.00	591.28	548.80	1.00	31600.00
19.43	24.09	0.14	16200.00	16700.00	591.64	543.50	0.90	32000.00
18.55	24.39	0.14	16100.00	16600.00	587.46	547.10	1.00	31800.00
19.03	23.31	0.14	16100.00	17300.00	588.45	598.00	1.00	32000.00
26.00	27.00		16200.00	17200.00	593.07	594.00	0.90	31900.00
26.00	27.00		16891.00	14700.00	640.11	604.00	1.39	31345.00
26.00	26.00		16378.00	14600.00	614.33	610.00	1.19	30176.00
26.00	27.00		16824.00	14700.00	636.38	594.00	1.22	30916.00
26.00	27.00		16379.00	14500.00	645.34	592.00	1.30	30121.00
26.00	26.00		16756.00	14600.00	635.55	610.00	1.31	30807.00
26.00	27.00		16567.00	14600.00	647.07	610.00	1.00	30242.00
26.00	26.00		16388.00	14500.00	578.00	595.00	0.97	29936.00
23.50	28.58		16419.00	14800.00	579.00	598.00	1.00	29949.00
23.80	27.63		16144.33		584.00	602.00	0.98	30035.42
22.70	26.90		16212.88		579.00	597.00	0.95	30100.53
23.00	27.83		16588.42		583.00	595.00	0.90	30974.15
23.40	28.54		16473.12		578.00	599.00	0.94	30914.75
23.40	27.44		16323.64		581.00	596.00	0.91	30445.90
23.70	26.58		16711.84		580.00	600.00		30477.69
23.30	27.87		16359.24		613.00			30491.09
24.39			16527.20		594.00			30452.78

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Na	Nb	Nd	Ni	Ni	Ni	P	P	Pb
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
31200.00	12.23	31.19	48.05	46.00	47.70	930.00	950.00	7.00
31100.00	12.20	31.12	47.97	49.00	47.14	920.00	950.00	7.50
31300.00	12.28	31.33	48.92	48.80	47.29	930.00	950.00	7.20
31600.00	12.22	31.81	48.07	46.90	47.57	910.00	950.00	7.10
31200.00	12.16	31.33	47.18	48.30	47.00	930.00	950.00	7.00
32000.00	12.12	31.90	47.30	47.50	47.32	930.00	950.00	7.10
31600.00	12.40	31.81	48.40	48.20	46.00	940.00	950.00	6.90
32000.00	12.19	31.62	47.42	48.50	46.00	930.00	950.00	6.90
30300.00	12.45	26.13	50.00	48.20	46.00	916.60	989.00	4.35
30300.00	11.61	26.04	50.00	48.37	46.00	918.52	991.00	4.54
30600.00	11.12	27.38	50.00	49.53	46.00	915.40	993.00	4.56
30500.00	11.12	26.96	50.00	50.35	46.00	917.40	988.00	4.40
30400.00	11.37	27.84	50.00	49.95	46.00	917.32	993.00	4.22
30400.00	11.18	27.47	50.00	49.91	46.00	914.76	990.00	4.45
30500.00	11.64	28.60	50.00	49.24	47.00	911.32	984.00	4.24
30400.00	11.31	27.36	50.00	48.40	47.00	918.12	987.00	4.36
	11.87	30.80	46.46	49.90	48.00	901.00		7.80
	12.10	31.50	46.21	47.00	47.00	904.00		7.60
	12.16	31.60	46.32	48.00	47.00	909.00		7.30
	12.09	31.90	46.47	47.00	50.00	900.00		7.40
	11.91	31.50	46.06	49.00	48.00	898.00		7.50
	12.30	31.20	46.34	49.00	50.00	895.00		7.90
	11.98	32.70	47.19	49.00		907.00		7.50
	12.32	32.50	47.15	50.00		904.00		7.40
	11.00		41.11	50.00		963.00		8.56
	11.00		42.11	40.88		931.00		8.67
	11.50		42.03	40.84		956.00		8.90
	11.50		41.00	40.77		927.00		8.68
	11.50		42.31	42.32		956.00		8.25
	11.50		45.00	41.17		941.00		8.09
	12.50		43.00	48.00		926.00		8.40
	12.00		45.00	46.00		925.00		8.48
	11.00		46.00	48.00		900.00		9.00
	11.00		45.00	50.00		900.00		7.00
	11.00		44.00	48.00		900.00		7.00
	11.00		44.00	48.00		900.00		7.00
	11.00			50.00		900.00		7.00
	11.00			50.00		900.00		7.00
				47.61		900.00		7.00
				47.64		900.00		7.00
								6.37
								6.07
								5.98
								6.89
								6.36
								6.36
								6.47
								6.86

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Pr	Rb	Sb	Sc	Sc	Sm	Sr	Sr	Ta
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
8.24	60.20	4.22	10.10	10.00	5.51	743.00	737.00	7.37
8.14	60.60	4.47	10.10	10.00	5.78	748.00	772.00	7.56
8.13	57.30	4.20	10.00	10.00	5.61	743.00	732.00	7.48
8.16	61.10	4.33	10.40	10.00	5.40	735.00	754.00	8.43
8.19	61.50	4.37	10.20	10.00	5.48	743.00	749.00	7.83
8.13	61.50	4.27	10.30	10.00	5.70	747.00	733.00	8.05
8.23	63.10	4.28	10.40	9.10	5.57	748.00	732.00	8.04
8.11	59.90	4.09	10.30	9.20	5.36	745.00	704.30	7.77
6.77	62.50	4.70	10.44	9.40	4.70	757.05	707.20	6.91
6.73	62.77	4.73	10.28	9.50	4.72	758.79	702.30	6.82
6.97	62.68	4.72	10.34	9.40	4.89	759.74	704.30	6.80
6.96	62.94	4.62	10.73	9.20	4.89	757.09	705.60	6.91
7.08	62.66	4.81	10.64	9.10	4.96	757.73	701.40	6.75
6.99	62.36	4.69	10.24	9.40	4.78	756.81	701.90	6.66
6.73	62.41	4.68	10.40		4.61	758.58	703.80	6.83
7.10	62.32	4.65	10.34		4.84	763.51	757.00	6.91
8.00	60.61	4.12	10.30		5.65	739.00	757.00	8.19
8.20	59.59	4.46	9.90		5.70	703.00	759.00	8.21
8.20	58.38	3.93	9.80		5.70	731.00	753.00	7.49
8.35	59.93	4.11	9.70		5.75	727.00	755.00	8.17
8.30	59.51	4.47	10.20		5.75	729.00	754.00	7.75
8.15	59.92	4.25	10.10		5.65	733.00	757.00	8.60
8.55	60.10	3.99	9.80		5.85	717.00	753.00	7.98
8.45	59.21	3.99	10.10		5.70	719.00	731.30	7.26
	46.85	4.53	9.56			753.02	729.20	7.54
	47.73	4.56	9.24			749.42	733.80	7.55
	47.49	4.82	9.45			732.12	730.50	7.46
	48.84	4.69	9.63			746.29	734.00	7.23
	59.80	4.72	9.60			737.75	733.50	7.95
	60.80	4.40	9.72			746.13	731.90	7.58
	60.40	4.40	9.24			762.79	730.90	7.63
	60.60	4.51	9.43			744.11		6.40
	61.40	4.60	9.00			749.26		6.80
	61.60	4.50	9.00			751.82		7.50
	63.80	5.10	9.00			773.03		7.30
	63.60	4.70	9.00			764.01		7.50
		4.40	9.00			761.93		6.30
		4.60	9.00			742.70		6.70
		5.10	9.00			749.96		7.40
		4.80	9.00			759.69		
		3.99						
		3.88						
		3.87						
		3.99						
		3.64						
		3.49						
		3.73						
		3.62						

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Tb	Th	Tl	Tm	U	V	V	W	W
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
0.47	4.84	0.32	0.13	1.86	97.00	101.00	12.20	11.50
0.46	5.00	0.32	0.12	1.91	97.00	99.00	12.90	12.00
0.45	4.71	0.32	0.13	1.88	96.00	100.00	12.30	12.50
0.48	4.98	0.32	0.13	1.90	96.00	98.00	12.20	13.00
0.47	4.83	0.31	0.13	1.89	97.00	99.00	12.40	13.00
0.48	5.18	0.33	0.13	2.03	98.00	101.00	12.70	12.50
0.46	4.90	0.31	0.13	1.89	94.00	102.00	11.80	14.00
0.46	5.19	0.31	0.13	1.93	95.00	102.00	11.30	13.50
0.55	6.41	0.35	0.15	2.15	93.72	95.91	14.67	10.56
0.52	6.22	0.35	0.15	2.19	93.80	96.35	14.48	10.64
0.54	6.35	0.35	0.15	2.27	93.48	95.64	14.27	10.01
0.53	6.69	0.36	0.15	2.16	93.45	95.74	14.62	10.79
0.55	6.61	0.34	0.15	2.21	93.44	96.00	14.19	10.18
0.55	6.38	0.34	0.15	2.24	93.86	95.84	14.65	9.75
0.53	6.68	0.33	0.15	2.16	93.76	96.24	14.20	11.37
0.53	5.15	0.35	0.15	2.20	93.75	95.22	14.47	10.68
0.40	5.12	0.31		2.00	98.00	90.00	12.80	11.00
0.40	5.03	0.31		1.95	98.00	89.00	11.80	10.00
0.41	5.06	0.30		1.95	98.00	89.00	11.10	10.00
0.42	5.15	0.33		1.94	97.00	89.00	11.80	11.00
0.42	5.22	0.31		2.02	97.00	89.00	11.90	11.00
0.41	5.19	0.34		1.98	97.00	90.00	12.00	10.00
0.40	5.16	0.33		1.95	98.00	89.00	12.30	11.00
0.42	4.00	0.32		2.00	98.00	90.00	12.80	11.00
0.48	3.93	0.33		1.70	92.00	100.00	12.94	
0.50	4.06	0.32		1.70	89.00	98.00	12.88	
0.48	4.13	0.33		1.70	94.00	102.00	13.42	
0.50	4.10	0.32		1.70	94.00	99.00	13.36	
0.52	4.31	0.31		1.80	93.00	98.00	13.10	
0.50	4.14	0.35		1.70	89.00	98.00	13.58	
0.52	4.27	0.34		1.70	90.00	100.00	13.40	
0.50	4.80	0.30		1.70	94.00	101.00	14.04	
	5.00	0.30		2.00	95.99		12.00	
	5.00	0.30		2.09	95.77		12.00	
	5.00	0.30		2.00	97.42		11.00	
	5.00	0.30		2.06	97.16		11.00	
	4.90	0.30		2.03	96.36		11.00	
	5.00	0.30		2.06	96.30		12.00	
	5.00	0.30		2.06	96.37		13.00	
		0.30		2.11	97.67		11.00	
		0.31						
		0.31						
		0.28						
		0.31						
		0.31						
		0.31						
		0.31						

Assay Data (Continued)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Y	Y	Yb	Zn	Zn	Zn
ppm	ppm	ppm	ppm	ppm	ppm
9.90	11.20	0.82	68.18	64.08	64.33
10.00	11.00	0.83	68.27	64.65	68.74
9.80	11.10	0.81	68.09	65.45	65.03
10.10	9.90	0.83	68.32	64.96	63.89
10.00	9.80	0.83	67.94	64.58	62.82
10.20	10.20	0.83	68.29	65.10	64.98
10.00	9.90	0.81	68.80	65.52	63.18
9.80	9.80	0.83	68.79	65.67	69.60
9.99	10.00	0.95	64.00	61.00	70.60
9.73	9.80	0.90	64.00	65.00	68.40
9.86	10.30	0.95	64.00	65.00	69.70
9.73		0.90	63.00	65.00	
9.70		0.95	63.00	65.00	
9.80		0.95	65.00	65.00	
9.72		0.95	64.00	66.00	
9.75			64.00	68.00	
9.70			62.34	70.42	
9.50			62.36	68.39	
9.70			62.23	67.39	
9.80			62.38	69.12	
9.80			62.24	69.95	
9.60			62.25	68.58	
9.70			62.18	70.70	
9.90			62.30	64.00	
10.15			63.00	62.00	
10.09			65.00	70.00	
10.35			64.00	64.00	
10.89			67.00	62.00	
10.09			63.00	64.00	
10.08			61.00	64.00	
10.17			63.00	66.00	
8.79			64.00	63.97	
8.76			65.00	63.18	
8.76			65.00	61.16	
8.79			63.00	61.07	
8.73			65.00	61.39	
10.70			64.00	62.60	
10.70			65.00	60.89	
10.40			65.00	60.34	
10.60			64.00	65.12	

26. Reported Values

The certified values listed in this certificate fulfil the AMIS statistical criteria (see section 19) regarding agreement for certification and have been independently validated by Allan Fraser.

27. Validation of Accuracy (Trueness)

This CRM can be used to validate accuracy (trueness) as required in method validation as stated in the ISO17025 standard. See Appendix 2 for an example on the validation of accuracy using replicate data derived from the analysis of a CRM.

28. Limit of Detection and Limit of Quantitation in Gravimetric Fire Assay

In the determination of limit of detection (LOD) and limit of quantitation (LOQ) in gravimetric fire assay (*i.e.* lead collection and weighing of resulting gold prill), the minimum mass that an assay microbalance is capable of weighing and the original test sample mass determines the LOD and the LOQ in the assay (Fraser, 2015), (see Appendix 7 for an example of the calculation LOD and LOQ and Table 14 for a recommend reporting scheme for LOD and LOQ values).

29. 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 general requirements for the competence of testing and calibration laboratories and who have maintained measurement traceability during the analytical process.

30. 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.

31. Minimum Sample Size

Most of the 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.

32. Availability

This product is available in Laboratory Packs containing 1kg of material and Explorer Packs containing custom weights (from 30g 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.

33. 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 5 for detail on the use of this CRM in quality control).

34. Legal Notice

This certificate and the reference material described in it have been prepared with due care and attention. However, AMIS, Muvhuso Ramasunga, Melesha Gopi Mungaroo and Allan Fraser; accept no liability for any decisions or actions taken following the use of the reference material.

Date of Version 0.00: 17 April 2025

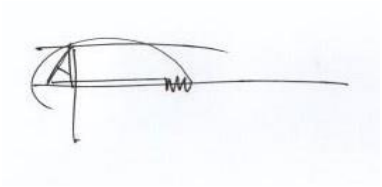
Version: 0.00

Approving Officer:

African Mineral Standards: _____

Melesha Gopi Mungaroo (Technical Supervisor)

Certifying Officer:



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Appendices

Appendix 1 through 8, prepared by Allan Fraser.

Appendix 1. Certification of Reference Material and Estimation of Measurement Uncertainty

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 use of *z*-scoring, Cochran's test for suspect repeatability variances, along with Grubbs test for suspect measurement values allows for the detection of outliers (IUPAC, 2006). 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 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.

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 the participating laboratories. Z-Scores are interpreted as follows:

$|z|$≤2 satisfactory performance
2<$|z|$≤3 questionable performance
$|z|$>2 unsatisfactory performance

(Thompson & Lowthian, 2011)

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

Cochran's 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_{calc} and 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 logarithm to base 10. 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 19, 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 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 11 is developed.

Table 11. 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 the 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

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

where MSW is the mean squares of the within laboratory variance, MSB is the mean squares for the between laboratories and $\bar{n}$ in this case, is the mean of the 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 8, 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 2. Example: Comparison of Mean and Certified Value for Validation of Accuracy

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 consider 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 12. 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 12. 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 % (*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 8, page 41) 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.

Appendix 3. Two-standard Deviations

Two-standard deviations are calculated using the expression:

$$Two\ standard\ deviations = 2\ (u_c) \qquad [19]$$

Where, u_c is the standard combined uncertainty (equation [14]).

Appendix 4. Confidence Interval

Confidence interval is calculated as:

$$Confidence\ Interval\ (CI) = \frac{(t_{critical})s}{\sqrt{N}} \qquad [20]$$

Where, *N* is the number of laboratories (accepted laboratory data), $t_{critical}$ is a two-tailed value for $N - 1$ degrees of freedom *(df)* and *s*, is the standard deviation of the accepted laboratory means. A two-tailed critical value is found for *N* -1 degrees of freedom from either a *t*-distribution table (Appendix 8) or MS Excel as =TINV (5%, df).

Appendix 5. Using the CRM in Quality Control

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, or *CI* 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 [21], of the replicates following Grubbs test.
5. Calculate the standard deviation, *s* from:

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}} \qquad [21]$$

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 [21].
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 the warning limits analyse another sample and if it is 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].

Appendix 6. 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:

$$Moisture\ correction\ factor\ (MCF) = \frac{100 - \%Moisture\ at\ 105^{O}C}{100} \qquad [22]$$

$$Air\ dry\ basis\ concentration = MCF\ x\ certified\ value\ on\ a\ dry\ basis \qquad [23]$$

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 [22] 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 [23]) 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 [22] and [23], *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.

Appendix 7. Example of Determination of LOD and LOQ in Fire Assay

The limit of detection (LOD) is the minimum detectable quantity of the analyte of interest (Skoog & West, 1985). To determine the LOD in fire assay by lead collection, the minimum mass that an assay microbalance is capable of weighing (*m* in micrograms, and the original test sample mass, $Mass_{assay}$ in grams) determines the LOD. The smallest prill mass most assay microbalances can measure is 1µg or 0.001mg. Even with a microscope it may be difficult to locate and pick up a prill weighing ten times that amount (*i.e.* 0.01mg or 10µg) and weigh it. If an analyst can weigh a prill of 1µg then the LOD becomes 1µg. However, the concentration factor would be 50 times for a 50-gram assay sample and therefore the LOD in g/t becomes 1µg divided by the original mass of the sample in grams taken for fire assay [24]. Therefore, the LOD in fire assay is computed as:

$$LOD = \frac{m\ (\mu g)}{Mass_{assay}\ (g)}\ \text{(g/t)} \qquad [24]$$

The limit of quantitation (LOQ), is simply the LOD multiplied by 10 (Long & Winefordner, 1983):

$$LOQ = 10 \cdot \frac{m\ (\mu g)}{Mass_{assay}\ (g)}\ \text{(g/t)} \qquad [25]$$

Therefore, with a sample mass of 50g taken for fire assay, the limit of detection would be 0.02g/t. *i.e.* 1µg = 1g/t, therefore 1µg/50g = 0.02g/t. If no prill was found, then the LOD result would be <0.02 g/t or "not detected". Using a larger assay sample mass improves the LOD and LOQ (Table 12). Table 14 gives a recommended reporting scheme for LOD and LOQ.

Table 13. 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 14. Recommended reporting scheme for LOD and LOQ in fire assay.

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

Appendix 8. T-distribution table

Table 15. 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		

End of certificate