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Certificate

AMIS0391

Certified Reference Material

Chrome, LG6 Ore Tails,
Kroondal Mine, Bushveld, South Africa

Certificate of Analysis

AMIS

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Summary Statistics

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

Analyte	Method	Certified (μ) ⁶	(2s) ⁸ \pm	Unit
Cr	FUS ¹	10.12	1.0	%
Cr	XRF ²	10.28	0.30	%
S	4A_MICP ³	0.010	0.002	%
SG	SG ⁴	3.59	0.16	Dimensionless
Au	Pb Collection ⁵	0.004	0.0037	g/t
Pd	Pb Collection	0.048	0.013	g/t
Pt	Pb Collection	0.105	0.020	g/t
Al	FUS	3.87	0.29	%
Ba	4A_MICP	28	5	ppm
Be	4A_MICP	0.1	0.1	ppm
Ca	4A_MICP	1.29	0.15	%
Co	4A_MICP	145	37	ppm
Cs	4A_MICP	0.1	0.04	ppm
Cu	4A_MICP	19	4	ppm
Fe	FUS	11.66	0.48	%
Hf	4A_MICP	0.3	0.2	ppm
K	4A_MICP	467	96	ppm
La	4A_MICP	2	0.9	ppm
Li	4A_MICP	3	0.8	ppm
Mg	FUS	12.61	0.76	%
Mn	4A_MICP	1679	147	ppm
Mo	4A_MICP	3	0.8	ppm
Na	4A_MICP	1584	123	ppm
Nb	4A_MICP	0.4	0.2	ppm
Ni	4A_MICP	641	90	ppm
Rb	4A_MICP	2	0.5	ppm
Sb	4A_MICP	0.3	0.2	ppm
Sc	4A_MICP	22	3	ppm
Si	FUS	17.12	0.90	%
Sr	4A_MICP	24	3	ppm
Ti	4A_MICP	1785	339	ppm
Ti	FUS	1978	192	ppm
U	4A_MICP	0.1	0.1	ppm
V	4A_MICP	677	152	ppm
V	FUS	723	115	ppm
Y	4A_MICP	3	0.3	ppm
Zn	4A_MICP	265	19	ppm
Zr	4A_MICP	7	3	ppm

Major Oxides
Certified Concentrations (at two Standard Deviations)

Analyte	Method	Certified (μ) ⁶	(2s) ⁸ \pm	Unit
Cr ₂ O ₃	XRF	15.03	0.44	%
Al ₂ O ₃	XRF	7.49	0.19	%
CaO	XRF	1.85	0.040	%
Fe ₂ O ₃	XRF	16.90	0.27	%
K ₂ O	XRF	0.055	0.01	%
MgO	XRF	21.25	0.59	%
MnO	XRF	0.22	0.02	%
P ₂ O ₅	XRF	0.010	0.003	%
SiO ₂	XRF	36.97	0.82	%
TiO ₂	XRF	0.32	0.02	%

1. Certified Concentrations and Uncertainties

AMIS0391 recertified in April 2019. 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	Certified (μ) ⁶	N	n	k	% RSD	(u_c) ⁷	(2s) ⁸ \pm	(U) ⁹ \pm	Unit
Cr	FUS ¹	10.12	5	38	2.776	5	0.51	1.0	1	%
Cr	XRF ²	10.28	9	71	2.306	1	0.15	0.30	0.3	%
S	4A_MICP ³	0.010	6	46	2.571	10	0.001	0.002	0.003	%
SG	SG ⁴	3.59	11	83	2.228	2	0.079	0.16	0.2	Dimensionless
Au	Pb Collection ⁵	0.004	7	49	2.447	44	0.0019	0.0037	0.005	g/t
Pd	Pb Collection	0.048	14	106	2.160	13	0.0063	0.013	0.01	g/t
Pt	Pb Collection	0.105	14	108	2.160	9	0.010	0.020	0.02	g/t
Al	FUS	3.87	5	38	2.776	4	0.14	0.29	0.4	%
Ba	4A_MICP	28	5	40	2.776	9	2	5	7	ppm
Be	4A_MICP	0.1	4	31	3.182	37	0.05	0.1	0.2	ppm
Ca	4A_MICP	1.29	6	47	2.571	6	0.074	0.15	0.2	%
Co	4A_MICP	145	8	64	2.365	13	18	37	43	ppm
Cs	4A_MICP	0.1	4	26	3.182	17	0.02	0.04	0.06	ppm
Cu	4A_MICP	19	8	57	2.365	11	2	4	5	ppm
Fe	FUS	11.66	5	40	2.776	2	0.24	0.48	0.7	%
Hf	4A_MICP	0.3	5	40	2.776	35	0.09	0.2	0.2	ppm
K	4A_MICP	467	6	48	2.571	10	48	96	124	ppm
La	4A_MICP	2	4	30	3.182	25	0.5	0.9	1	ppm
Li	4A_MICP	3	5	40	2.776	12	0.4	0.8	1	ppm
Mg	FUS	12.61	5	38	2.776	3	0.38	0.76	1	%
Mn	4A_MICP	1679	7	54	2.447	4	74	147	180	ppm
Mo	4A_MICP	3	5	40	2.776	14	0.4	0.8	1	ppm
Na	4A_MICP	1584	6	46	2.571	4	61	123	158	ppm
Nb	4A_MICP	0.4	5	35	2.776	26	0.1	0.2	0.3	ppm
Ni	4A_MICP	641	8	64	2.365	7	45	90	106	ppm
Rb	4A_MICP	2	4	32	3.182	14	0.2	0.5	0.7	ppm
Sb	4A_MICP	0.3	5	40	2.776	28	0.09	0.2	0.3	ppm
Sc	4A_MICP	22	7	52	2.447	8	2	3	4	ppm
Si	FUS	17.12	4	31	3.182	3	0.45	0.90	1	%
Sr	4A_MICP	24	5	39	2.776	7	2	3	4	ppm
Ti	4A_MICP	1785	5	39	2.776	9	169	339	470	ppm
Ti	FUS	1978	5	40	2.776	5	96	192	267	ppm
U	4A_MICP	0.1	4	30	3.182	38	0.05	0.1	0.2	ppm
V	4A_MICP	677	6	45	2.571	11	76	152	195	ppm
V	FUS	723	5	40	2.776	8	58	115	160	ppm
Y	4A_MICP	3	6	45	2.571	5	0.1	0.3	0.4	ppm
Zn	4A_MICP	265	7	55	2.447	4	9	19	23	ppm
Zr	4A_MICP	7	4	32	3.182	18	1	3	4	ppm

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

Analyte	Method	Certified (μ) ⁶	N	n	k	% RSD	(u_c) ⁷	(2s) ⁸ \pm	(U) ⁹ \pm	Unit
Cr ₂ O ₃	XRF	15.03	9	71	2.306	1	0.22	0.44	0.5	%
Al ₂ O ₃	XRF	7.49	8	64	2.365	1	0.095	0.19	0.2	%
CaO	XRF	1.85	10	76	2.262	1	0.020	0.040	0.04	%
Fe ₂ O ₃	XRF	16.90	9	72	2.306	1	0.14	0.27	0.3	%
K ₂ O	XRF	0.055	8	63	2.365	12	0.007	0.01	0.02	%
MgO	XRF	21.25	10	78	2.262	1	0.29	0.59	0.7	%
MnO	XRF	0.22	9	71	2.306	4	0.009	0.02	0.02	%
P ₂ O ₅	XRF	0.010	7	46	2.447	15	0.001	0.003	0.004	%
SiO ₂	XRF	36.97	10	77	2.262	1	0.41	0.82	0.9	%
TiO ₂	XRF	0.32	9	65	2.306	4	0.01	0.02	0.03	%

1. FUS is Fusion digestion with ICP finish XRF is X-ray Fluorescence
2. 4A_MICP is a Multi-acid digestion with either ICPOES/ICPMS/AAS finish
3. SG is Specific Gravity
4. Pb Collection
5. 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.
6. 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)
7. Two standard deviations (2s)
8. 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: $U = 2.36 \times 0.23 = 0.5\%$

2. 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 3). 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 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
Al 4A_MICP	7.63 %	Al Fus	7.96 %	Unequal Variance ($p=0.021$)	0.041	Reject H_0 ; certified values are <i>not</i> equal
Co 4A_MICP	271 ppm	Co Fus	279 ppm	Equal Variance ($p=0.29$)	0.67	Accept H_0 ; certified values are equal
Mg 4A_MICP	5.32 %	Mg Fus	5.79 %	Unequal Variance ($p=0.003$)	0.001	Reject H_0 ; certified values are not equal
Mn 4A_MICP	0.167 %	Mn Fus	0.186 %	Equal Variance ($p=0.49$)	0.008	Reject H_0 ; certified values are <i>not</i> equal
Ni 4A_MICP	0.084 %	Ni Fus	0.094 %	Equal Variance ($p=0.48$)	0.052	Accept H_0 ; certified values are equal
Cr Fus	30.98 %	Cr XRF	31.31 %	Unequal Variance ($p<0.001$)	0.45	Accept H_0 ; certified values are equal
Al ₂ O ₃ Fus	14.60 %	Al ₂ O ₃ XRF	15.04 %	Unequal Variance ($p=0.002$)	0.16	Accept H_0 ; certified values are equal
Fe ₂ O ₃ Fus	28.17 %	Fe ₂ O ₃ XRF	28.90 %	Unequal Variance ($p<0.001$)	0.033	Reject H_0 ; certified values are <i>not</i> equal
MgO Fus	9.68 %	MgO XRF	9.50%	Equal Variance ($p=0.26$)	0.33	Accept H_0 ; certified values are equal

3. Intended Use

AMIS0391 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 6). 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

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
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

Abbreviation/Symbol	Description
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 1×10 ⁻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

This standard was made using LG6 foundry grade material supplied by Glencore Xstrata plc from the Kroondal Chrome Mine. The mine is situated on the Western Limb of the Bushveld Complex 12km south east of Rustenburg in the North West Province of South Africa. The standard comprises of LG6 foundry grade. The foundry is a concentrated product derived from mining the LG6 chromitite layer, the LG6 middling pyroxenite and the LG6A chromitite layer which occurs in the lower portion of the Critical Zone.

9. Approximate Mineral and Chemical Composition

The LG6 foundry consists of chromite concentrate derived from the mining of the LG6 and LG6A chromitite layers. The LG6 foundry consist mainly of fine to medium size liberated chromite grains with minor liberated pyroxene and plagioclase grains.

10. 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 7 for an example calculation).

11. 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.

12. Methods of Analysis Requested

The following methods of analysis were requested:

- a) Pt, Pd and Au. ICP-OES or ICP-MS, Pb collection with Ag as a co-collector.
- b) Au, Pt, Pd, Rh, Ru and Ir. ICP-MS, nickel sulphide collection.
- c) Co, Cu and Ni. Multi-acid total digestion, including HF, with ICP-OES finish.
- d) Co, Cu and Ni. Aqua regia digestion with ICP-OES finish.
- e) Cr, Co, Cu and Ni. Pressed pellet XRF.
- f) S by LECO
- g) Specific Gravity. Gas pycnometer.
- h) XRF (major elements).
- i) Multi acid digest ICP scan – trace elements.

13. Information Requested of Participating Laboratories

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

- a) Aliquots used for all determinations.
- b) Results for individual PGM's reported in ppb.
- c) Results for base metals reported in ppm.
- d) QC data, to include replicates, blanks and certified reference materials used.
- e) Analytical techniques used.

14. 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 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)

15. Two Standard Deviations

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

$$\text{Two standard deviations} = 2 (u_c)$$

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

16. Confidence Interval

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

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

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 9) or MS Excel as =TINV (5%, df).

17. 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]).

18. 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 6, “Using the CRM in Quality Control” be used, in setting the limits of the CRM. Table 5 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 5. Example of replicate assay data in which the *CI*, *2s* and *U* are shown.

Lab No.	Mean Au (g/t)	<i>CI</i>	0.0088
1	0.268	<i>2s</i>	0.031
2	0.273	<i>U</i>	0.04
3	0.270		
4	0.288		
5	0.274		
6	0.256		
7	0.263		
8	0.258		
9	0.288		

19. Participating Laboratories

The laboratories that are accredited with ISO17025 and provided timeous results are:

1. Activation Laboratories Pty Ltd (ActLabs) CA
2. Alfred H Knight (SA)
3. ALS Chemex Laboratory Group Johannesburg SA
4. ALS Chemex Laboratory Group Perth WA
5. ALS Chemex Laboratory Group Vancouver CA
6. ALS OMAC (Ireland)
7. Genalysis Laboratory Services (South Africa) Pty
8. Genalysis Laboratory Services (W Australia P)
9. GNK Laboratories (Pvt) Ltd
10. Intertek Utama Services (Indonesia)
11. Labtium Inc Finland
12. Lanxess Chrome Mine
13. Samancor Western Chrome Mine
14. Set Point Laboratories (Isando) SA
15. SGS Australia Pty Ltd (Newburn) WA
16. SGS Geosol Laboratories Ltda (Brazil)
17. SGS Mineral Services Lakefield (Canada)
18. SGS South Africa (Pty) Ltd - Booyens JHB
19. Ultra-Trace (Pty) Ltd WA
20. Xstrata Alloys Lydenburg

20. Accepted Assay Data

Data from the 20 laboratories used for certification are set out in Table 6.

Table 6. Data used to calculate the certified values after removal of outliers.

XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF	XRF
Cr ₂ O ₃	Cr ₂ O ₃	Al ₂ O ₃	Al ₂ O ₃	CaO	CaO	Fe ₂ O ₃	Fe ₂ O ₃	K ₂ O	K ₂ O	MgO	MgO	MnO	MnO	P ₂ O ₅	SiO ₂	SiO ₂	TiO ₂
%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%
15.28	15.10	7.65	7.38	1.87	1.85	17.31	16.93	0.06	0.05	21.84	21.35	0.23	0.21	0.01	37.63	37.16	0.31
15.16	15.09	7.65	7.34	1.86	1.83	17.24	16.97	0.05	0.06	21.69	21.26	0.23	0.22	0.01	37.55	37.05	0.31
15.26	14.94	7.58	7.38	1.89	1.85	17.13	16.90	0.06	0.05	21.44	21.30	0.23	0.22	0.01	37.26	37.23	0.30
15.33	14.70	7.48	7.50	1.86	1.88	16.86	16.88	0.05	0.05	21.53	21.32	0.22	0.22	0.01	37.43	36.90	0.30
15.38	14.82	7.53	7.53	1.82	1.89	17.05	16.77	0.05	0.05	21.59	21.26	0.22	0.22	0.01	37.71	37.19	0.30
15.43	14.64	7.56	7.55	1.83	1.89	16.76	16.94	0.05	0.05	21.54	21.10	0.22	0.22	0.01	36.80	37.44	0.33
15.37	14.72	7.50	7.48	1.85	1.86	16.82	17.05	0.05	0.05	21.70	21.36	0.22	0.22	0.01	36.95	37.57	0.34
15.40	14.88	7.60	7.54	1.84	1.87	17.06	16.73	0.05	0.05	21.30	21.81	0.23	0.22	0.01	36.90	37.59	0.33
15.19	15.00	7.47	7.57	1.85	1.85	16.90	16.80	0.05	0.05	21.30	21.79	0.21	0.21	0.01	36.90	37.44	0.33
15.21	15.10	7.44	7.47	1.85	1.84	16.90	17.00	0.05	0.05	21.40	21.84	0.21	0.23	0.01	37.10	37.38	0.33
15.36	15.00	7.49	7.50	1.84	1.83	16.90	16.86	0.05	0.05	21.30	21.77	0.21	0.23	0.01	37.00	37.47	0.33
15.01	15.00	7.45	7.51	1.85	1.85	16.90	16.86	0.05	0.05	21.30	21.82	0.21	0.23	0.01	37.00	37.45	0.33
14.95	15.00	7.46	7.67	1.86	1.86	17.00	16.58	0.05	0.05	21.30	21.72	0.21	0.23	0.01	37.00	37.56	0.32
14.98	15.10	7.46	7.72	1.83	1.83	16.90	16.87	0.05	0.05	21.30	21.78	0.21	0.23	0.01	37.00	36.26	0.32
15.02	15.00	7.46	7.44	1.85	1.82	16.90	16.90	0.05	0.05	21.30	21.76	0.21	0.23	0.01	37.00	36.44	0.32
14.88	15.00	7.47	7.54	1.83	1.84	17.00	16.93	0.05	0.05	21.27	20.75	0.21	0.23	0.01	37.23	36.46	0.32
14.96	14.71	7.48	7.63	1.84	1.84	16.98	17.04	0.05	0.06	21.21	20.79	0.22	0.22	0.01	37.32	36.32	0.32
14.88	14.69	7.47	7.74	1.85	1.84	17.00	16.84	0.06	0.07	21.29	20.88	0.22	0.21	0.01	37.23	36.28	0.32
15.03	14.73	7.43	7.78	1.84	1.84	16.98	16.98	0.06	0.07	21.32	20.81	0.22	0.21	0.01	37.23	36.20	0.32
15.00	14.69	7.38		1.84	1.82	17.00	16.97	0.05	0.07	21.24	20.69	0.22	0.21	0.01	37.26	36.53	0.32
14.80	14.69	7.47		1.85	1.84	16.95	16.81	0.06	0.07	21.32	20.78	0.22	0.21	0.01	37.23	36.60	0.32
14.90	14.72	7.46		1.83	1.86	16.97	16.86	0.05	0.06	21.32	20.88	0.22	0.21	0.01	37.32	36.80	0.31
14.90	14.72	7.38		1.83	1.82	16.94	16.90	0.05	0.06	21.21	21.10	0.22	0.21	0.01	37.23	36.70	0.32
14.90	14.73	7.48		1.85	1.83	16.92	16.80	0.05		21.16	21.20	0.22	0.21	0.01	37.31	36.50	0.31
14.80	15.06	7.50		1.82	1.81	17.01	16.70	0.06		20.95	21.10	0.23	0.21	0.01	36.94	36.80	0.31
14.80	14.92	7.51		1.88	1.82	16.96	16.60	0.06		21.25	21.10	0.22	0.24	0.01	37.22	36.80	0.31
	15.20	7.52		1.81	1.85	17.18	16.70	0.05		20.80	21.10	0.23	0.24	0.01	37.13	36.40	0.31
	14.95	7.51		1.82	1.82	16.95	16.80	0.06		21.31	21.20	0.22	0.22	0.01	37.33	36.60	0.31
	15.20	7.50		1.80	1.81	17.02	16.60	0.07		21.18	21.00	0.23	0.24	0.01	37.00	36.10	0.33
	14.98	7.50		1.84	1.84	16.77	16.60	0.06		20.90	21.10	0.22	0.24	0.01	37.07	36.30	0.33
	15.07	7.50		1.84	1.82	16.86		0.06		20.92	20.70	0.23	0.23	0.01	37.06	36.60	0.33
	14.95	7.51		1.86	1.86	16.96		0.06		21.20	21.30	0.22	0.24	0.01	36.90	36.40	0.33
	15.20	7.48		1.86		16.90		0.06		21.20	21.10	0.21		0.01	36.90	37.50	0.34
	15.20	7.48		1.85		16.80		0.05		21.10	21.00	0.21		0.01	36.80	36.20	0.33
	15.10	7.44		1.86		16.90		0.06		21.20	21.30	0.22		0.01	36.90	36.30	0.34

Assay Data (Cont.)

FUS	XRF	XRF	4A_MICP	SG	SG	Pb Collection	Pb Collection	Pb Collection	Pb Collection
Cr	Cr	Cr	S	SG	SG	Au	Au	Pd	Pd
ppm	ppm	ppm	%	Dimensionless	Dimensionless	g/t	g/t	g/t	g/t
108000	103314	103725	0.01	3.74	3.58	0.00	0.01	0.05	0.06
107000	103246	104409	0.01	3.73	3.57	0.00	0.00	0.05	0.05
106000	102219	104888	0.01	3.71	3.58	0.00	0.00	0.04	0.05
104000	100577	105230	0.01	3.69	3.58	0.00	0.00	0.04	0.05
103000	101398	105572	0.01	3.68	3.58	0.00	0.01	0.05	0.05
105000	100167	105162	0.01	3.49	3.62	0.00	0.00	0.05	0.05
105000	100714	105367	0.01	3.49	3.62	0.01	0.00	0.05	0.05
105000	101809	103930	0.01	3.52	3.68	0.00	0.00	0.05	0.05
103000	102630	104067	0.01	3.50	3.69	0.01		0.05	0.05
102000	103314	105093	0.01	3.51	3.61	0.01		0.05	0.06
103000	102630	102705	0.01	3.55	3.63	0.01		0.05	0.05
105000	102630	102267	0.01	3.50	3.64	0.01		0.05	0.05
102000	102630	102486	0.01	3.48	3.68	0.01		0.05	0.06
103000	103314	102736	0.01	3.57	3.55	0.01		0.05	0.06
104000	102630	101829	0.01	3.56	3.57	0.01		0.05	0.04
104000	102630	102380	0.01	3.54	3.59	0.00		0.05	0.04
97700	100660	101819	0.01	3.56	3.55	0.00		0.04	0.06
100900	100523	102866	0.01	3.53	3.58	0.00		0.04	0.06
106900	100755	102630	0.01	3.55	3.55	0.01		0.04	0.04
86300	100509	101262	0.01	3.56	3.58	0.01		0.05	0.06
90800	100516	101946	0.01	3.55	3.57	0.01		0.04	0.05
91300	100680	101946	0.01	3.49	3.70	0.00		0.04	0.04
93200	100735	101946	0.01	3.45	3.71	0.00		0.04	0.05
95800	100755	101262	0.01	3.48	3.72	0.01		0.04	0.05
103000	103047	101262	0.01	3.52	3.73	0.00		0.05	0.05
102000	102103		0.02	3.53	3.72	0.00		0.05	0.04
102000	103971		0.01	3.51	3.68	0.00		0.05	0.05
103000	102261		0.01	3.49	3.64	0.00		0.05	0.05
103000	103985		0.01	3.63	3.70	0.00		0.05	0.04
104000	102466		0.01	3.68	3.56	0.00		0.05	0.04
106000	103136		0.01	3.63	3.53	0.00		0.05	0.05
103000	102315		0.02	3.62	3.56	0.00		0.05	0.05
98097	103998		0.01	3.61	3.55	0.00		0.04	0.04
97599	103998		0.01	3.64	3.54	0.00		0.04	0.05
99409	103314		0.01	3.67	3.58	0.00		0.04	0.04
99432	103998		0.01	3.63	3.56	0.00		0.04	0.04
97288	103998		0.01	3.52	3.58	0.00		0.04	0.05
99362	103998		0.01	3.51		0.00		0.04	0.05
	103314		0.01	3.51		0.01		0.04	0.04
	103998		0.01	3.47		0.01		0.04	0.05

Assay Data (Cont.)

Pb Collection	Pb Collection	Pb Collection	Pb Collection	FUS	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Pd	Pt	Pt	Pt	Al	Ba	Be	Ca	Ca
g/t	g/t	g/t	g/t	ppm	ppm	ppm	ppm	ppm
0.04	0.11	0.10	0.10	39700	30.00	0.12	11900	13500
0.04	0.10	0.11	0.11	39300	30.00	0.12	12800	13500
0.05	0.10	0.10	0.11	39200	30.00	0.10	12400	13300
0.04	0.09	0.11	0.10	38300	30.00	0.15	12400	13500
0.05	0.10	0.11	0.10	37800	30.00	0.10	12000	13300
0.04	0.11	0.10	0.09	38500	30.00	0.09	12000	13400
0.06	0.10	0.13	0.12	38900	30.00	0.12	11900	13300
0.06	0.10	0.10	0.11	38800	30.00	0.12	12400	
0.06	0.11	0.10	0.09	39800	28.00	0.07	13989	
0.05	0.11	0.10	0.10	39200	29.00	0.11	13904	
0.06	0.11	0.11	0.10	39800	27.00	0.10	13828	
0.06	0.11	0.10	0.10	39600	28.00	0.11	13736	
0.06	0.11	0.10	0.10	39700	28.00	0.18	13748	
0.06	0.11	0.11	0.11	39100	27.00	0.13	13810	
0.05	0.11	0.11	0.11	39700	29.00	0.09	13762	
0.05	0.09	0.10	0.11	39400	28.00	0.08	13893	
0.05	0.10	0.11	0.11	38500	30.00	0.10	11800	
0.05	0.09	0.12	0.12	38100	30.00	0.10	12300	
0.05	0.10	0.13	0.11	38800	30.00	0.10	12500	
0.04	0.10	0.12	0.11	39000	30.00	0.20	11600	
0.05	0.10	0.10	0.11	39100	30.00	0.10	12000	
0.05	0.09	0.12	0.11	38700	30.00	0.10	12000	
	0.09	0.11	0.11	38800	30.00	0.10	12300	
	0.11	0.10	0.11	38400	30.00	0.20	13300	
	0.11	0.11	0.11	36300	28.00	0.20	13400	
	0.11	0.10	0.09	35700	27.00	0.20	13200	
	0.11	0.11	0.11	35900	27.00	0.20	13500	
	0.11	0.11	0.11	36200	28.00	0.20	13400	
	0.11	0.11		36700	27.00	0.20	13500	
	0.11	0.12		36600	26.00	0.10	13300	
	0.11	0.10		39700	28.00	0.20	13400	
	0.09	0.10		39700	27.00		12500	
	0.09	0.10		41000	24.00		12500	
	0.08	0.11		39300	24.00		12400	
	0.09	0.12		40400	24.00		12900	
	0.08	0.10		40500	25.00		12400	
	0.09	0.10		40000	24.00		13000	
	0.08	0.10		40100	25.00		12500	
	0.09	0.10			24.00		12600	
	0.12	0.10			24.00		13300	

Assay Data (Cont.)

4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	FUS	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Co	Co	Cs	Cu	Cu	Fe	Hf	K	K	La	Li
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
143	150	0.11	17.30	19.00	120000	0.10	400	500	1.50	3.20
152	160	0.12	18.70	20.00	120000	0.20	500	500	1.70	3.70
147	150	0.11	18.30	20.00	119000	0.10	400	500	1.60	3.30
151	150	0.11	18.60	20.00	116000	0.20	500	400	1.70	3.50
149	150	0.11	17.10	20.00	115000	0.20	400	500	1.60	3.30
146	150	0.11	16.80	20.00	117000	0.10	400	500	1.50	3.20
149	150	0.11	18.20	20.00	118000	0.10	400	500	1.60	3.30
148	106	0.11	18.10	17.00	118000	0.20	400	500	1.60	3.20
147	100	0.10	20.00	17.00	115000	0.30	491		1.50	3.10
152	105	0.10	20.00	17.00	115000	0.30	499		1.60	3.00
145	101	0.10	20.00	17.00	118000	0.30	493		1.60	3.10
141	101	0.10	19.00	17.00	120000	0.30	497		1.50	3.10
153	101	0.10	19.00	17.00	117000	0.30	489		1.60	3.00
140	100	0.10	19.00	17.00	117000	0.30	491		1.40	3.00
146	102	0.10	20.00	17.00	120000	0.30	483		1.50	3.10
144	140	0.12	20.00	20.00	119000	0.30	487		1.40	3.00
155	140	0.10	18.50	20.00	117000	0.20	500		1.80	3.30
158	140	0.10	18.50	20.00	117000	0.20	500		1.60	3.80
160	140	0.11	17.50	20.00	118000	0.20	500		1.70	3.80
162	140	0.10	21.60	20.00	117000	0.30	500		1.60	3.60
164	140	0.10	21.40		117000	0.20	500		1.60	3.10
158	150	0.10	23.50		118000	0.20	400		1.70	3.50
164	150	0.10	22.00		120000	0.20	500		1.60	3.70
158		0.10	20.30		118000	0.20	500		1.60	3.70
155		0.10	15.00		113000	0.40	500		2.60	3.50
155		0.10	15.00		111000	0.40	500		2.50	4.00
150		0.10	15.00		111000	0.40	500		2.60	4.00
155		0.10	15.00		114000	0.20	500		2.60	3.50
155		0.10	15.00		114000	0.40	500		2.50	4.00
155		0.15	15.00		117000	0.20	500		2.50	4.00
155		0.11	20.00		115000	0.20	500			4.00
155		0.15	18.00		114000	0.40	500			4.00
151		0.14	19.00		117000	0.26	400			3.00
151		0.14	19.00		114000	0.28	400			3.00
152			19.00		114000	0.29	400			3.00
157			20.00		116000	0.24	400			3.00
152			19.00		117000	0.23	400			3.00
157					115000	0.26	500			3.00
153					117000	0.28	400			3.00
154					117000	0.26	400			3.00

Assay Data (Cont.)

FUS	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP	4A_MICP
Mg	Mn	Mn	Mo	Na	Nb	Ni	Ni	Rb	Sb	Sc
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
132000	1540	1700	2.62	1500	0.40	653	670	1.60	0.27	21.40
132000	1660	1700	3.00	1600	0.40	690	670	1.70	0.18	23.00
131000	1610	1700	3.19	1600	0.30	668	640	1.70	0.34	22.40
129000	1600	1700	3.34	1600	0.40	680	650	1.70	0.19	22.90
129000	1550	1700	3.61	1600	0.30	663	650	1.60	0.29	21.90
130000	1540	1700	3.02	1600	0.30	655	640	1.60	0.27	21.60
131000	1530	1700	2.97	1600	0.30	654	660	1.60	0.24	22.20
131000	1600	1700	3.06	1500	0.30	659	561	1.70	0.22	22.10
125000	1743	1700	3.00	1610	0.40	660	549	2.00	0.40	23.00
125000	1725	1700	2.90	1601	0.40	641	547	1.90	0.40	23.00
128000	1721	1700	3.30	1597	0.40	653	529	2.00	0.30	22.00
129000	1719	1700	2.90	1605	0.40	640	531	1.80	0.40	23.00
127000	1726	1700	3.00	1599	0.40	652	533	1.90	0.40	22.00
128000	1732	1700	2.90	1591	0.40	657	523	1.80	0.40	23.00
129000	1731		3.10	1599	0.40	661	521	1.90	0.30	22.00
129000	1710		3.20	1598	0.40	654	660	1.80	0.30	22.00
127000	1530		3.18	1700	0.60	633	670	1.70	0.30	20.80
125000	1600		2.64	1600	0.30	665	680	1.80	0.36	24.80
128000	1620		2.94	1700	0.30	674	670	1.70	0.37	25.10
127000	1560		2.55	1600	0.30	631	660	1.50	0.35	23.00
128000	1550		2.92	1700	0.60	633	680	1.60	0.38	19.40
126000	1600		2.40	1700	0.30	599	680	1.50	0.42	21.70
127000	1720		2.96	1600	0.30	632	660	1.70	0.36	23.70
126000	1710		2.48	1500	0.30	653		1.50	0.37	22.20
118000	1700		3.50	1500	0.50	665		1.70	0.40	23.00
120000	1760		3.50	1600	0.50	650		1.70	0.40	23.00
123000	1700		3.00	1600	0.50	655		1.10	0.40	23.00
128000	1750		3.50	1600	0.50	665		1.00	0.40	23.00
121000	1720		3.00	1600	0.60	655		1.50	0.40	23.00
122000	1720		3.00	1500	0.50	665		1.50	0.60	23.00
121000	1770		3.00	1500	0.60	645		1.60	0.40	23.00
123000	1690		3.00	1500	0.50	665		1.60	0.40	23.00
125000	1760		2.47	1500	0.60	644			0.26	23.00
124000	1760		2.22	1500	0.60	636			0.27	23.00
126000	1680		2.42	1500	0.50	644			0.23	23.00
126000	1760		2.68	1500		652			0.26	24.00
120000	1760		2.14	1500		641			0.24	23.00
124000	1800		2.21	1500		672			0.23	24.00
	1700		2.38	1600		643			0.25	23.00
	1700		2.39	1600		652			0.22	23.00

Assay Data (Cont.)

4A MICP	FUS	4A MICP	4A MICP	FUS	4A MICP	4A MICP	FUS	4A MICP	4A MICP	4A MICP	4A MICP
Sc	Si	Sr	Ti	Ti	U	V	V	Y	Zn	Zn	Zr
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
	172000	22.90	1730	2100	0.10	698	770	2.60	261	280	5.30
18.10	173000	24.70	1850	2000	0.10	749	750	2.90	283	270	5.30
21.00	172000	23.70	1780	2000	0.10	723	750	2.80	271	280	5.10
21.00	171000	24.20	1800	2000	0.10	726	740	2.80	272	270	5.10
21.00	169000	23.70	1770	1900	0.10	702	730	2.70	263	270	5.20
21.00	169000	22.90	1750	2000	0.10	694	740	2.60	260	270	5.50
21.00	171000	24.10	1740	2000	0.10	689	760	2.70	260	260	5.30
21.00	168000	23.80	1780	2000	0.10	721	750	2.70	270	270	7.90
21.00	171000	24.70	1977	2000	0.10	771	750	2.80	262	270	9.20
21.00	171000	25.80	1965	2000	0.14	760	750	2.80	251	270	8.50
	173000	25.40	1962	2100	0.13	763	750	2.80	262	260	8.30
	174000	24.70	1996	2100	0.11	764	750	2.70	251		7.80
	174000	25.60	1973	2100	0.10	763	750	2.80	259		8.10
	174000	25.10	1966	2000	0.11	762	750	2.70	261		7.90
	175000	25.50	1967	2100	0.10	758	750	2.80	262		7.90
	176000	25.70	1938	2100	0.11	750	750	2.80	262		7.90
	174000	22.80	1710	2000	0.10	704	700	2.60	259		7.80
	177000	24.20	1780	2000	0.10	735	700	2.90	268		7.70
	177000	23.20	1800	2000	0.10	744	700	3.00	271		7.90
	174000	21.20	1670	2100	0.10	689	700	2.70	252		7.10
	174000	21.30	1730	2100	0.10	717	700	2.80	263		7.80
	175000	23.00	1650	2100	0.10	673	700	2.70	262		7.10
	174000	20.90	1730	2100	0.10	716	700	3.10	267		7.80
	178000	25.50	1790	2000	0.10	737	700	2.90	255		7.30
	163000	25.00	1900	1825	0.10	655	637	3.00	250		8.00
	164000	25.00	1900	1818	0.10	652	630	2.90	250		9.00
	165000	25.00	1900	1824	0.10	650	632	3.00	255		8.00
	166000	25.50	2000	1870	0.10	666	633	2.80	250		8.00
	167000	24.50	1900	1955	0.10	646	634	2.90	260		8.00
	166000	25.00	1900	1916	0.10	681	640	2.80	250		8.00
	167000	24.50	1900	1887	0.20	660	628	3.00	255		8.00
		22.00	1900	1915	0.20	674	643	3.00	264		8.00
		22.00	1599	1900	0.20	520	783	3.00	267		
		22.00	1595	1900	0.20	515	776	3.00	270		
		23.00	1520	1900	0.18	512	787	3.00	278		
		22.00	1506	1900	0.19	516	783	3.00	268		
		23.00	1480	1900	0.20	514	781	3.00	277		
		22.00	1532	1900	0.21	700	782	3.00	267		
		22.00	1523	1900		700	779	2.90	274		
				1900		700	780	2.90	280		

21. Reported Values

The certified values listed in this certificate fulfil the AMIS statistical criteria (see section 14) 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 standard. 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 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

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.

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 6 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, Makhosi Khoza, and Allan Fraser; accept no liability for any decisions or actions taken following the use of the reference material.

Date of Version v1.00: 08 April 2019

Version: v1.00

Reason for Version v0.01: Rectification of all elements.

Version v1.00 replaces the original report of AMIS0391 Certification

Date of Version 000: 26 February 2014

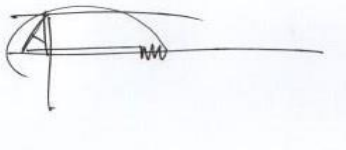
Version: 000

Approving Officer:

African Mineral Standards: _____

Makhosi Khoza (Quality Specialist)

Certifying Officer:

A handwritten signature in black ink, appearing to be 'AF', written over a horizontal line.

Geochemist: _____

Allan Fraser

M.Sc. (Geology), N.D. (Analytical Chem.),
Pr.Sci.Nat. Pr.Chem.SA

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Appendices

Appendix 1: Uncertified Element Statistics

Uncertified element statistics are shown in **Table 7**.

Table 7. Uncertified element concentrations statistics.

Element	Generic Method	n	Mean	SD	RSD %	Unit
Ag	2A_MICP	14	0.9	1.2	133	ppm
Ag	4A_MICP	20	0.4	0.5	106	ppm
Al	2A_MICP	8	8237.5	106.1	1	ppm
Al	4A_MICP	47	37266.6	4197.9	11	ppm
Al ₂ O ₃	FUS	8	7.3	0.1	1	%
As	2A_MICP	8	0.4	0.05	11	ppm
As	4A_MICP	32	3.5	4.1	116	ppm
As	FUS	1	34.0	*	*	ppm
B	2A_MICP	8	317.5	82.2	26	ppm
B	FUS	6	20.0	*	*	ppm
Ba	2A_MICP	8	20.0	*	*	ppm
Ba	FUS	8	28.8	1.2	4	ppm
BaO	XRF	9	0.02	0.01	34	%
Be	2A_MICP	6	0.1	0.01	15	ppm
Bi	2A_MICP	8	0.01	*	*	ppm
Bi	4A_MICP	30	4.0	8.0	201	ppm
Ca	2A_MICP	8	5812.5	83.5	1	ppm
Ca	FUS	8	11000.0	456.7	4	ppm
CaO	FUS	8	1.9	0.04	2	%
Cd	2A_MICP	8	0.01	*	*	ppm
Cd	4A_MICP	26	0.1	0.1	70	ppm
Ce	2A_MICP	7	2.1	0.02	1	ppm
Ce	4A_MICP	32	3.7	1.0	28	ppm
Co	FUS	8	122.9	4.6	4	ppm
Cr	2A_MICP	8	737.8	10.8	1	ppm
Cr	4A_MICP	9	88968.2	5566.8	6	ppm
Cr ₂ O ₃	FUS	8	14.8	0.1	1	%
Cs	2A_MICP	8	0.1	0.01	6	ppm
Cu	4A_ICPES	8	17.5	4.6	26	ppm
Cu	FUS	8	74.2	1.3	2	ppm
Dy	4A_MICP	15	0.5	0.02	5	ppm
Er	4A_MICP	14	0.3	0.02	6	ppm
Eu	4A_MICP	16	0.1	0.03	21	ppm
Fe	2A_MICP	16	10462.5	1903.3	18	ppm
Fe	4A_ICPES	8	115437.5	1935.3	2	ppm
Fe	4A_MICP	46	117487.5	7069.3	6	ppm
Fe ₂ O ₃	FUS	8	16.4	0.1	1	%
Ga	2A_MICP	8	1.5	0.03	2	ppm
Ga	4A_MICP	40	17.3	6.1	35	ppm
Gd	4A_MICP	16	0.4	0.03	7	ppm
Ge	2A_MICP	8	0.1	0.005	7	ppm
Ge	4A_MICP	25	0.4	0.6	154	ppm
GOI	GOI	62	0.6	0.1	20	%
Hf	2A_MICP	8	0.03	*	*	ppm
Hg	4A_MICP	4	1.3	0.5	40	ppm
Ho	4A_MICP	16	0.1	0.02	17	ppm
In	2A_MICP	1	0.01	*	*	ppm
In	4A_MICP	29	0.04	0.02	59	ppm
K	2A_MICP	8	250.0	53.5	21	ppm
K	FUS	8	600.0	*	*	ppm
K ₂ O	FUS	8	0.1	0.02	26	%
La	2A_MICP	8	1.1	0.05	4	ppm
Li	2A_MICP	8	0.9	0.05	5	ppm
Mg	4A_MICP	45	120473.1	7156.4	6	ppm

Element	Generic Method	n	Mean	SD	RSD %	Unit
MgO	FUS	8	21.0	0.2	1	%
Mn	2A_MICP	24	181.8	31.6	17	ppm
Mn	FUS	8	1643.0	24.5	1	ppm
MnO	FUS	8	0.2	0.002	1	%
Mo	2A_MICP	8	2.8	0.2	7	ppm
Moisture	GOI	8	0.2	0.02	11	%
Na	2A_MICP	7	1100.0	*	*	ppm
Na ₂ O	FUS	8	0.2	0.002	1	%
Na ₂ O	XRF	54	0.2	0.1	34	%
Nd	4A_MICP	15	1.7	0.1	5	ppm
Ni	FUS	8	579.5	6.5	1	ppm
P	2A_MICP	8	27.5	4.6	17	ppm
P	4A_MICP	32	30.8	8.9	29	ppm
P	FUS	8	100.0	*	*	ppm
P ₂ O ₅	FUS	8	0.01	0.001	13	%
Pb	2A_MICP	10	3.5	3.5	98	ppm
Pb	4A_MICP	42	2.2	1.1	47	ppm
Pb	FUS	42	21.0	*	*	ppm
Pr	4A_MICP	42	0.4	0.1	18	ppm
Rb	2A_MICP	42	1.1	*	*	ppm
Re	4A_MICP	42	0.003	0.001	32	ppm
S	2A_MICP	42	0.1	0.1	81	%
S	Combustion/LECO	42	0.02	0.005	32	%
Sb	2A_MICP	42	0.3	0.04	14	ppm
Sb	FUS	42	283.1	40.8	14	ppm
Sc	2A_MICP	42	2.3	0.1	3	ppm
Sc	FUS	42	18.5	0.5	3	ppm
Se	4A_MICP	42	1.1	0.4	33	ppm
SiO ₂	FUS	42	37.6	0.3	1	%
Sm	4A_MICP	42	0.4	0.1	13	ppm
Sn	2A_MICP	42	0.4	*	*	ppm
Sn	4A_MICP	42	0.6	0.3	51	ppm
Sn	FUS	42	102.9	30.9	30	ppm
SO ₃	XRF	42	0.03	0.01	21	%
Sr	2A_MICP	42	20.2	0.2	1	ppm
Sr	FUS	42	30.9	0.7	2	ppm
Ta	4A_MICP	42	0.1	0.03	38	ppm
Tb	4A_MICP	42	0.1	0.02	25	ppm
Te	4A_MICP	42	1.5	2.0	136	ppm
Th	2A_MICP	42	0.2	*	*	ppm
Th	4A_MICP	42	0.5	0.2	45	ppm
Ti	2A_MICP	42	120.0	*	*	ppm
TiO ₂	FUS	42	0.3	0.003	1	%
Tl	4A_MICP	42	14.6	16.8	115	ppm
Tm	4A_MICP	42	0.1	0.02	26	ppm
V	2A_MICP	42	11.5	0.5	5	ppm
V ₂ O ₅	FUS	42	0.1	0.002	2	%
V ₂ O ₅	XRF	42	0.1	0.004	3	%
W	2A_MICP	42	0.1	0.004	4	ppm
W	4A_MICP	42	1.9	2.9	153	ppm
Y	2A_MICP	42	0.5	0.01	1	ppm
Yb	4A_MICP	42	0.4	0.03	7	ppm
Zn	FUS	42	65.1	6.6	10	ppm
Zr	2A_MICP	42	1.1	*	*	ppm

* denotes that the results were too similar and SD and RSD% could not be calculated

Appendix 2 through 9, prepared by Allan Fraser.

Appendix 2. 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, 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 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]:

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

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:

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

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

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

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

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

$$Observed\ \%RSD = \frac{s}{Mean} \times 100 \quad [5]$$

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 14, 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.

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

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.

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

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:

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

From equations [9] through [13], the ANOVA table as shown in Table 8 is developed.

Table 8. 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

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

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 9, 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

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.

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

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

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 $| \quad |$ 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 9. 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 \neq 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 9. 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 9, page 32) 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 4. Two-standard Deviations

Two-standard deviations are calculated using the expression:

$$\text{Two standard deviations} = 2 (u_c) \quad [19]$$

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

Appendix 5. Confidence Interval

Confidence interval is calculated as:

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

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 9) or MS Excel as =TINV (5%, df).

Appendix 6. 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 to 15 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:

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

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 $\pm 2s$ and $\pm 3s$, where s is the standard deviation calculated from [21].
8. Construct the Shewhart control chart around the mean of n replicates.
9. Use $\pm 2s$ as the warning limits.
10. Use $\pm 3s$ 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 7. 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 cm² with the CRM material spread evenly over same; this represents a 0.1 gram spread per cm². In correcting the certified value for moisture content, a moisture correction factor is calculated:

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

$$\text{Air dry basis concentration} = \text{MCF} \times \text{certified value on a dry basis} \quad [23]$$

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:

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

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:

$$0.995 \times 12.62\% = 12.56\%$$

The stated measurement uncertainty also needs to be corrected using [22] and [23], e.g. $0.995 \times 0.52 = 0.51_{(7)}$, rounded to 0.52%. The air-dry basis concentration *i.e.* $12.56 \pm 0.52\%$ is to be used as the certified value with its corresponding measurement of uncertainty.

Appendix 8. 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 $1\mu\text{g}$ 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 $10\mu\text{g}$) and weigh it. If an analyst can weigh a prill of $1\mu\text{g}$ then the LOD becomes $1\mu\text{g}$. However, the concentration factor would be 50 times for a 50-gram assay sample and therefore the LOD in g/t becomes $1\mu\text{g}$ divided by the original mass of the sample in grams taken for fire assay [24]. Therefore, the LOD in fire assay is computed as:

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

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

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

Therefore, with a sample mass of 50g taken for fire assay, the limit of detection would be 0.02g/t . *i.e.* $1\mu\text{g} = 1\text{g/t}$, therefore $1\mu\text{g}/50\text{g} = 0.02\text{g/t}$. If no prill was found, then the LOD result would be $<0.02\text{g/t}$ or “not detected”. Using a larger assay sample mass improves the LOD and LOQ (Table 10 **Table 9**). Table 11 gives a recommended reporting scheme for LOD and LOQ.

Table 10. Mass of assay sample and corresponding limit of detection and limit of quantitation for an assay microbalance capability of smallest prill mass of $1\mu\text{g}$ 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 11. Recommended reporting scheme for LOD and LOQ in fire assay.

Data	Report as
<LOD	Not detected
<LOQ	Detected
$\geq\text{LOQ}$	Report assay result

Appendix 9. T-distribution table

Table 12. 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