African Mineral Standards
REFERENCE MATERIALS FOR AFRICAN ORES

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AMIS0140

Certified Reference Material

Tantalum, ore grade

Certificate of Analysis

Recommended Concentrations and Limits[1.]
(at two Standard Deviations)

Provisional Concentrations[2.3.]

Ta XRF	511	±	63	ppm
Nb XRF	114	±	33	ppm

Informational Mean

Nb M/ICP	104	ppm

1. *Manufacturers recommended limits for use of the material as control samples, based on two standard deviations, calculated using "Between Laboratory" statistics for treatment of the data for trivial, non-trivial and technically invalid results. See sections 1, 9 and 12.*
2. *The slightly high limits set for Ta and Nb (above) reflect a "high" between lab variance for results returned by different laboratories. This is normally due to slight differences in instrument calibrations or analytical methods between labs. The within lab variance for this material is actually very low. Users are therefore advised to read Section 19 (p6 of this certificate) and to set their own (lower) limits; before using this material to check the accuracy of a single analysis from a single laboratory.*
3. *There is additional certified major and trace element data presented on p2.*

1. Intended Use: AMIS0140 was prepared to check analysis of samples of tantalum bearing rock with a similar grade.

It is a Certified Reference Material, fit for use as control samples in routine assay laboratory quality control when inserted within runs of samples and measured in parallel to the unknown. Its purpose is to monitor inter-laboratory or instrument bias and within lab precision. It can be used, indirectly, to establish the traceability of results to an SI system of units.

The recommended concentrations and limits for this material are property values based on a measurement campaign (round robin) and reflect consensus results from the laboratories that participated in the round robin.

Slight variations in analytical procedures between laboratories will reflect as slight biases to the recommended concentrations (see 19). Good laboratories will report results within the two standard deviation levels with a failure rate of <10 %.

The material can also be used for method development and for the calibration of equipment.

2. Origin of Material: AMIS0140 was made from tantalum concentrate, blended down with quartz.

3. Chemical Composition: (Other elements)

Uncertified data received from some of the laboratories is presented below. This data is for informational purposes only.

Analyte	method	unit	mean	2SD	RSD%	n
Al_2O_3	XRF	%	1.43	0.03	1.1	16
CaO	XRF	%	0.21	0.00	0.8	8
Fe_2O_3	XRF	%	2.18	0.06	1.4	16
K_2O	XRF	%	0.33	0.02	2.5	16
MgO	XRF	%	0.12	0.16	64.8	16
MnO	XRF	%	0.16	0.01	4.6	16
Na_2O	XRF	%	0.20	0.01	3.2	14
P_2O_5	XRF	%	0.02	0.00	10.8	16
SiO_2	XRF	%	95.52	1.56	0.8	16
TiO_2	XRF	%	0.08	0.01	3.5	15
LOI	XRF	%	0.42	0.09	10.7	8

Analyte	method	unit	mean	2SD	RSD%	n
Al	M/ICP	%	0.7	0.11	7.3	31
As	M/ICP	ppm	1.4	0.6	23.1	8
Ba	M/ICP	ppm	53	16	15.4	32
Be	M/ICP	ppm	0.2	0.13	30.2	8
Bi	M/ICP	ppm	153	13	4.4	32
Ca	M/ICP	%	0.2	0.03	8.6	31
Ce	M/ICP	ppm	16	1	4.3	16
Co	M/ICP	ppm	7.9	1.2	7.4	32
Cr	M/ICP	ppm	316	128	20.2	32
Cs	M/ICP	ppm	0.4	0.04	5.0	8
Cu	M/ICP	ppm	230	37	8.0	31
Dy	M/ICP	ppm	0.8	0.00	0.0	7
Er	M/ICP	ppm	0.5	0.10	11.2	8
Eu	M/ICP	ppm	0.2	0.02	5.3	8
Fe	M/ICP	%	1.6	0.2	4.9	32
Ga	M/ICP	ppm	2.1	0.1	2.4	7
Gd	M/ICP	ppm	1.1	0.1	5.8	8
Hf	M/ICP	ppm	1.1	1.2	57.1	16
Ho	M/ICP	ppm	0.2	0.02	5.8	8
K	M/ICP	%	0.3	0.03	5.1	31
La	M/ICP	ppm	8.2	0.5	3.1	22
Li	M/ICP	ppm	2.1	0.6	15.0	21
Mg	M/ICP	%	0.1	0.02	6.9	31
Mn	M/ICP	ppm	1316	83	3.1	30
Mo	M/ICP	ppm	3.7	1.4	18.7	32

Analyte	method	unit	mean	2SD	RSD%	n
Na	M/ICP	%	0.1	0.02	7.1	30
Nd	M/ICP	ppm	6.4	0.5	3.7	8
Ni	M/ICP	ppm	34	8	11.8	32
P	M/ICP	%	0.0	0.00	11.2	8
Pb	M/ICP	ppm	8	6	37.6	32
Pr	M/ICP	ppm	1.8	0.09	2.6	8
Rb	M/ICP	ppm	11	1	6.7	23
S	ICP	ppm	270.0	23.9	4.4	8
Sb	M/ICP	ppm	5	1	13.4	29
Sc	M/ICP	ppm	2.2	0.75	17.4	19
Sm	M/ICP	ppm	1	0	5.3	8
Sn	M/ICP	ppm	2.2	0.48	10.8	16
Sr	M/ICP	ppm	14.3	2.14	7.5	30
Tb	M/ICP	ppm	0.2	0.02	5.5	8
Th	M/ICP	ppm	4.3	0.9	10.8	32
Ti	M/ICP	%	0.0	0.0	10.5	31
Tm	M/ICP	ppm	0.1	0.0	0.0	7
U	M/ICP	ppm	9.3	1.5	8.2	31
V	M/ICP	ppm	16.1	2.25	7.0	26
W	M/ICP	ppm	3.8	0.82	10.8	15
Y	M/ICP	ppm	3.7	0.5	7.1	23
Yb	M/ICP	ppm	0.4	0.1	10.9	8
Zn	M/ICP	ppm	25.0	16.43	32.9	30
Zr	M/ICP	ppm	36	8	11.6	30

4. Appearance: The material is a very fine white coloured powder (Corstor 8N).

5. Handling instructions: The material is packaged in Laboratory Packs and Explorer Packs that must be shaken or otherwise agitated before use. Normal safety precautions for handling fine particulate matter are suggested, such as the use of safety glasses, breathing protection, gloves and a laboratory coat.

6. Method of Preparation: The material was crushed, dry-milled and air-classified to <54um. Wet sieve partic**l**e size analysis of random samples confirmed the material was 98.5% <54um. It was then blended in a bi-conical mixer, systematically divided and then sealed into 1kg Laboratory Packs. Explorer Packs are subdivided from the Laboratory packs as required. Samples were randomly selected for homogeneity testing and third party analysis. Statistical analysis of both homogeneity and consensus test results were carried out by an independent statistician.

7. Methods of Analysis requested:

1. Ta & Nb - Multi-acid digest, including HF, ICP- OES or ICP-MS.
2. Ta & Nb - XRF.

8. Information requested:

1. State and provide brief description of analytical techniques used.
2. State aliquots used for all determinations.
3. All results to be reported in ppm.
4. Report all QC data, to include replicates, blanks and certified reference materials used.

9. Method of Certification: Fifteen laboratories were each given eight packages, comprising eight samples scientifically selected from throughout the batch. Final limits were calculated after first determining if all data was compatible within a spread normally expected for similar analytical methods done by reputable l**a**boratories. Data from any one laboratory was then removed from further calculations when the mean of all analyses from that laboratory failed a “t test” of the global means of the other laboratories. The means and standard deviations were then re-calculated using all remaining data. Any analysis that fell outside of the new two standard deviations was removed from the ensuing data base. The mean and standard deviations were again calculated using the remaining data.

The “between-laboratory” standard deviation is used in the calculation to eliminate technically and statistically invalid data. Upper and lower limits are based on the standard deviation of the remaining data, which reflect individual analyses and can be used to monitor accuracy in routine laboratory quality control. This is different to limits based on standard deviations derived from grouped set of analyses (see 12), which provide important measures for precision and trueness, but which are less useful for routine QC.

Standards with an RSD of near or less than 5 % are termed “Certified”, RSD’s of between near 5 % and 15 % are termed “Provisional”, and RSD’s over 15 % are termed “Informational”.

Note: the Ta multi acid digest results received were unsuitable for certification, reflecting diverse methodologies between the different laboratories. The raw data is however quoted in Section 11.

10. Participating Laboratories: (Not in same order as in the table of assays):

1. ACME Analytical Laboratories Ltd CA
2. Activation Laboratories Pty Ltd (ActLabs) CA
3. ALS Chemex Laboratory Group Perth WA
4. ALS Chemex Laboratory Group Vancouver CA
5. Assayers Canada

6. Genalysis Laboratory Services WA
7. Intertek Testing Services Ltd Shanghai (ITS Beijing)
8. Intertek Utama Services (Indonesia)
9. Labtium Inc Finland
10. OMAC Laboratories Limited (Ireland)
11. Set Point Laboratories (Isando) SA
12. SGS Australia Pty Ltd (Newburn) WA
13. SGS Lakefield Research Africa Pty Ltd (Booysens) SA
14. SGS Mineral Services Lakefield (Canada)
15. Ultra Trace (Pty) Ltd WA

11. Assay Data: Data as received from the laboratories for the important certified elements listed on p1 is set out below. A proficiency report has been sent to the managers of the participating laboratories. Additional digital data from this round robin is available on request.

Lab Code	Ta M/ICP ppm	Ta XRF ppm	Nb M/ICP ppm	Nb XRF ppm
A	513.97	527.73	114.24	121.00
A	510.88	556.99	107.52	113.00
A	494.40	535.04	108.64	118.00
A	490.28	527.73	112.00	119.00
A	487.19	554.90	107.52	120.00
A	501.61	549.67	112.00	116.00
A	495.43	568.48	110.88	117.00
A	507.79	531.91	112.00	122.00
B	345.00	500.00	65.50	100.00
B	337.00	500.00	61.10	100.00
B	333.00	500.00	60.60	200.00
B	347.00	500.00	63.40	100.00
B	328.00	500.00	58.90	200.00
B	341.00	500.00	65.10	200.00
B	323.00	500.00	68.20	200.00
B	332.00	500.00	66.80	100.00
C		460.00		110.00
C		460.00		110.00
C		460.00		110.00
C		440.00		110.00
C		450.00		120.00
C		460.00		110.00
C		450.00		110.00
C		460.00		120.00
D	500.00	600.00	100.00	70.00
D	500.00	500.00	100.00	80.00
D	500.00	600.00	100.00	70.00
D	500.00	600.00	100.00	70.00
D	500.00	500.00	200.00	80.00
D	500.00	600.00	200.00	80.00
D	500.00	600.00	100.00	80.00
D	500.00	500.00	100.00	70.00
E	363.61	674.00	84.72	122.00
E	366.93	666.00	88.41	129.00
E	319.62	672.00	78.90	124.00
E	414.72	646.00	88.81	128.00
E	363.57	666.00	87.17	132.00
E	372.26	660.00	84.04	121.00
E	419.28	673.00	93.75	124.00
E	387.54	651.00	88.33	125.00
F	314.00		82.00	
F	345.00		90.00	
F	329.00		83.00	
F	331.00		86.00	
F	329.00		88.00	
F	299.00		81.00	
F	364.00		89.00	
F	352.00		90.00	

Lab Code	Ta M/ICP ppm	Ta XRF ppm	Nb M/ICP ppm	Nb XRF ppm
G	638.00		136.00	
G	632.00		136.00	
G	619.00		136.00	
G	635.00		135.00	
G	642.00		134.00	
G	587.00		125.00	
G	601.00		125.00	
G	608.00		135.00	
H	348.00		61.00	
H	369.00		80.00	
H	389.00		71.00	
H	345.00		87.00	
H	322.00		73.00	
H	381.00		83.00	
H	337.00		75.00	
H	386.00		84.00	
I	217.90		82.70	
I	190.90		87.60	
I	275.20		131.40	
I	214.30		107.90	
I	378.50		91.90	
I	299.00		107.80	
I	224.30		115.90	
I	220.80		103.60	
J		508.00		86.00
J		525.00		82.00
J		511.00		84.00
J		522.00		82.00
J		541.00		82.00
J		519.00		85.00
J		512.00		80.00
J		528.00		82.00
K	455.00	485.00	105.00	129.00
K	393.00	484.00	97.80	125.00
K	313.00	489.00	88.10	134.00
K	437.00	487.00	104.00	125.00
K	406.00	485.00	96.60	132.00
K	341.00	500.00	98.40	137.00
K	537.00	506.00	121.00	136.00
K	333.00	507.00	88.60	132.00
L	610.72		141.74	
L	631.99		139.20	
L	578.94		136.10	
L	640.54		137.84	
L	626.46		136.88	
L	662.01		140.34	
L	665.51		141.70	
L	622.25		133.56	

Assay Data (cont):

Lab Code	Ta M/ICP ppm	Ta XRF ppm	Nb M/ICP ppm	Nb XRF ppm
M	526.00	550.00	125.00	130.00
M	527.00	550.00	135.00	120.00
M	541.00	550.00	130.00	130.00
M	526.00	560.00	130.00	130.00
M	539.00	560.00	130.00	130.00
M	526.00	550.00	135.00	130.00
M	530.00	550.00	125.00	130.00
M	527.00	560.00	125.00	130.00
N		520.00	101.50	99.00
N		506.00	114.50	98.00
N		497.00	98.00	98.00
N		481.00	108.50	97.00
N		530.00	109.50	98.00
N		482.00	107.50	95.00
N		518.00	108.50	98.00
N		505.00	110.50	100.00

Lab Code	Ta M/ICP ppm	Ta XRF ppm	Nb M/ICP ppm	Nb XRF ppm
O	360.00		81.00	
O	330.00		76.00	
O	370.00		86.00	
O	380.00		84.00	
O	300.00		66.00	
O	310.00		72.00	
O	350.00		80.00	
O	360.00		80.00	

12. Measurement of Uncertainty: The samples used in the certification process were selected in such a way as to represent the entire batch of material and were taken from the final packaged units; therefore all possible sources of uncertainty (sample uncertainty and measurement uncertainty) are included in the final combined standard uncertainty determination.

The uncertainty measurement takes into consideration the between lab and the within lab variances and is calculated from the square roots of the variances of these components using the formula:

Combined standard uncertainty=sqrt((between lab.var/no of labs) + (mean square within lab.var /no of assays))

These uncertainty measurements may be used, by laboratories, as a component for calculating the total uncertainty for method validation according to the relevant ISO guidelines.

Analyte	Method	Unit	S [1]	σL [2]	Sw [3]	CSU [4]
Ta	XRF	%	31.62	33.66	10.89	12.81
Nb	XRF	%	16.45	18.23	3.46	6.91
Nb	M/ICP	%	21.33	16.87	7.13	4.93

1. S - Std Dev for use on control charts.
2. σL - Betw Lab Std Dev, for use to calculate a measure of accuracy.
3. SW - Within Lab Stc Dev, for use to calculate a measure of precision.
4. CSU - Combined Standard Uncertainty, a component for use to calculate the total uncertainty in method validation.

13. Uncertified values: The Provisional and Informational values listed on p1 of this certificate fulfill the AMIS statistical criteria regarding agreement for certification and have been independently validated by Dr Barry Smee.

14. 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, who have maintained measurement traceability during the analytical process.

15. Certification: AMIS0140 is a new material.

16. 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.

17. Minimum sample size: The majority of laboratories reporting used a 0.5g sample size for the ICP and 10g for the XRF. These are the recommended minimum sample sizes for the use of this material.

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

19. Recommended use: The data used to characterize this CRM has been scrutinized using outlier treatment techniques. This, together with the number of participating laboratories, should overcome any "inter-laboratory issues" and should lead to a very accurate measure for the given methods, notwithstanding the underlying assumption that what the good inter-laboratory labs reported was accurate. However an amount of bad data might have had an effect, resulting in limits which in some situations might be too broad for the effective monitoring of a single analytical method, laboratory or production process. Users should set their own limits based on their own data quality objectives and control measurements, after determining the performance characteristics of their own particular method, using a minimum of 40 analyses using this CRM. User set limits should normally be within the limits recommended on p1 and 2 of this certificate.

20. Legal Notice: This certificate and the reference material described in it have been prepared with due care and attention. However AMIS, Set Point Technology (Pty) Ltd, Mike McWha, Dr Barry Smee and Smee and Associates Ltd; accept no liability for any decisions or actions taken following the use of the reference material.

20 October 2009

Certifying Officers:

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