



African Mineral Standards

MATRIX REFERENCE MATERIALS

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## AMIS0424

### ***Certified Reference Material***

**Copper ore, carbonatite,  
Palabora Mine, South Africa**

### ***Certificate of Analysis***

#### **Recommended Concentrations and Limits<sup>1</sup> (at two Standard Deviations)**

##### ***Certified Concentrations<sup>2</sup>***

Cu Fus	1.145	±	0.053	%
Cu M/ICP	1.145	±	0.058	%
Cu P	1.135	±	0.044	%
Specific Gravity	3.07	±	0.08	

##### ***Provisional Concentrations***

Au Pb Collection	0.100	±	0.012	g/t
Co M/ICP	78	±	16	ppm
Co P	75	±	9	ppm

##### ***Indicated Mean***

Cu Soluble 1191 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. There is additional certified major element data presented on p2 and uncertified trace element data presented as an appendix.

# Major Element Recommended Concentrations and Limits (at two Standard Deviations)

## ***Certified Concentrations***

Al <sub>2</sub> O <sub>3</sub>	0.49	±	0.03	%
CaO	31.68	±	0.44	%
Fe <sub>2</sub> O <sub>3</sub>	17.70	±	0.30	%
K <sub>2</sub> O	0.22	±	0.01	%
MgO	7.27	±	0.08	%
MnO	0.13	±	0.01	%
SiO <sub>2</sub>	11.08	±	0.14	%
TiO <sub>2</sub>	0.35	±	0.01	%
LOI	23.58	±	0.14	%

## ***Provisional Concentration***

S Comb/LECO	0.83	±	0.12	%
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## ***Indicated Means***

Cr <sub>2</sub> O <sub>3</sub>	0.01	%
Na <sub>2</sub> O	0.03	%

**1. Intended Use:** AMIS0424 is a certified reference material which may be used to demonstrate the validity of measurement results of a single analysis of carbonatite hosted copper ores with a similar grade and matrix.

It is a matrix matched 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:** This standard was made using ore sourced from Palabora Mining Company Limited. Palabora Mine is located next to the town of Phalaborwa in Mpumalanga Province, 380 kilometres north east of Johannesburg, South Africa. The Palabora alkali carbonatitic massif is an ovoid bicentered alkaline ring complex with a carbonatite core, intruded as a vertical pipe into Archean granite prior to 2.06Ga. The Palabora complex is unique in that it hosts the only economic copper sulphide carbonatite orebody in the world.

**3. Mineral and Chemical Composition:** The host rocks are carbonatite and foskorite. Chalcopyrite is the most abundant ore mineral followed by bornite. Chalcocite is a subsidiary sulphide in the foskorite. Other sulphides are valleriite, cubanite and pyrrhotite (minor). Other important mineral by-products are titaniferous magnetite and apatite. Uranothorite is also recovered.

**4. Appearance:** The material is a very fine powder. It is colored Medium Dark Grey.

**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 <54µm. Wet sieve particle size analysis of random samples confirmed the material was 98.5% <54µm. 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 the consensus test results were carried out by independent statisticians.

**7. Methods of Analysis requested:**

1. Au – Pb collection, ICP-OES/ICP-MS.
2. Cu. Acid Soluble AAS or ICP-OES.
3. Cu. Fusion AAS or ICP-OES.
4. Multi-acid digest multi-element scan - ( to include Co, Cu ). ICP-OES or ICP-MS.
5. Aqua regia digest – Co, Cu. ICP-OES or ICP-MS.
6. Majors ( Al<sub>2</sub>O<sub>3</sub>, CaO, Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, MgO, MnO, Na<sub>2</sub>O, SiO<sub>2</sub>, TiO<sub>2</sub>. LOI. ) XRF fusion.
7. SG, gas pycnometer.

**8. Information requested:**

1. Aliquots used for all determinations.
2. Results for individual PGM's reported in ppb.
3. Results for base metals reported in ppm.
4. QC data, to include replicates blanks and certified reference materials used.
5. Analytical techniques used.

**9. Method of Certification:** Twenty three laboratories were each given eight randomly selected packages of sample. Nineteen of the laboratories submitted results.

Final limits were calculated after first determining if all data was compatible within a spread normally expected for similar analytical methods done by reputable laboratories. 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”.

**10. Participating Laboratories:** The 19 out of 23 laboratories that provided results timeously were (not in same order as in the table of assays):

1. Activation Laboratorios Ltda (Chile)
2. ALS Chemex Laboratory Group Brisbane Australia
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. Bureau Veritas (Namibia)
8. Genalysis Laboratory Services (W Australia P)
9. Genalysis Zambia
10. Intertek Utama Services (Indonesia)
11. Set Point Laboratories (Isando) SA
12. SGS Australia Pty Ltd (Newburn) WA
13. SGS Geosol Laboratories Ltda (Brazil)
14. SGS Mineral Services Callao (Peru)
15. SGS Mineral Services Lakefield (Canada)
16. SGS South Africa (Pty) Ltd - Booyens JHB
17. SGS Vancouver (Canada)
18. Skyline Assayers and Labs (USA)
19. Ultra Trace (Pty) Ltd WA

**11. Assay Data:** Data as received from the laboratories for the important certified elements listed on p1 are set out below.

Lab Code	Au Pb Coll g/t	Co M/ICP ppm	Co P ppm	Cu Fus ppm	Cu M/ICP ppm	Cu P ppm	Cu Soluble ppm	Al <sub>2</sub> O <sub>3</sub> XRF %	CaO XRF %	Cr <sub>2</sub> O <sub>3</sub> XRF %	Fe <sub>2</sub> O <sub>3</sub> XRF %	K <sub>2</sub> O XRF %	MgO XRF %	MnO XRF %	Na <sub>2</sub> O XRF %	SiO <sub>2</sub> XRF %	TiO <sub>2</sub> XRF %	LOI %	S Comb LECO %	SG pyc	
B	0.11	80.0	50.0		11750		840												23.6		3.24
B	0.10	80.0	60.0		11550	11600	800												23.6		3.26
B	0.10	80.0	60.0		11650	10900	790												23.6		3.26
B	0.11	90.0	60.0		11950	11000	800												23.6		3.21
B	0.09	90.0	70.0		11600	11450	830												23.6		3.21
B	0.09	80.0	60.0		11750	11150	820												23.6		3.20
B	0.10	80.0	50.0		11750	11300	750												23.6		3.27
B	0.11	80.0	60.0		11750	11550	800												23.6		3.20
C	0.10				12000		1300													0.84	
C	0.10				11900		1300													0.81	
C	0.10				11700		1300													0.82	
C	0.10				11800		1200													0.83	
C	0.11				12000		1300													0.83	
C	0.10				12000		1300													0.83	
C	0.10				11900		1200													0.82	
C	0.11				11900		1200													0.80	
D		76.0	80.0	11200	10900	10800															3.11
D		74.0	82.0	11300	10900	11100															3.09
D		74.0	82.0	11200	11100	11300															3.09
D		74.0	81.0	11500	11100	10900															3.11
D		76.0	81.0	11500	11000	11100															3.09
D		76.0	80.0	11400	11100	11200															3.10
D		76.0	80.0	11200	11100	11200															3.10
D		76.0	82.0	11300	11100	11100															3.09
E	0.09	72.0		11400				0.47	31.7	0.02	17.9	0.22	7.31	0.13	0.04	11.2	0.36	23.5	0.04		
E	0.09	74.0		11000				0.47	31.7	0.02	17.7	0.22	7.28	0.13	0.04	11.2	0.36	23.5	0.05		
E	0.10	70.0		11000				0.47	31.7	0.02	17.8	0.22	7.31	0.13	0.05	11.2	0.34	23.5	0.03		
E	0.09	73.0		11200				0.47	31.7	0.02	17.7	0.23	7.30	0.13	0.04	11.2	0.35	23.4	0.02		
E	0.09	74.0		11000				0.49	31.7	0.02	17.7	0.23	7.28	0.13	0.04	11.2	0.35	23.5	0.02		
E	0.09	73.0		11300				0.47	31.7	0.02	17.7	0.23	7.32	0.13	0.04	11.2	0.35	23.5	0.02		
E	0.09	71.0		11200				0.48	31.7	0.02	17.7	0.23	7.28	0.13	0.05	11.2	0.35	23.5	0.02		
E	0.08	70.0		11500				0.47	31.7	0.02	17.8	0.22	7.30	0.13	0.04	11.2	0.35	23.5	0.03		
F	0.09				11100		3200														
F	0.09				11100		3200														
F	0.10				11100		3200														
F	0.10				11100		3200														
F	0.09				11100		3100														
F	0.10				11000		3200														
F	0.09				11100		3200														
F	0.10				11100		3200														



## Assay data (cont)

Lab Code	Au Pb Coll g/t	Co M/ICP ppm	Co P ppm	Cu Fus ppm	Cu M/ICP ppm	Cu P ppm	Cu Soluble ppm	Al <sub>2</sub> O <sub>3</sub> XRF %	CaO XRF %	Cr <sub>2</sub> O <sub>3</sub> XRF %	Fe <sub>2</sub> O <sub>3</sub> XRF %	K <sub>2</sub> O XRF %	MgO XRF %	MnO XRF %	Na <sub>2</sub> O XRF %	SiO <sub>2</sub> XRF %	TiO <sub>2</sub> XRF %	LOI %	S Comb LECO %	SG pyc
U		75.0			11100		1530	0.51	31.8		17.8	0.22	7.33	0.13	0.02	11.3	0.35	23.4		
U		79.0			11000		1470	0.50	31.9		17.8	0.22	7.33	0.14	0.01	11.3	0.35	23.4		
U		75.0			11500		1460	0.50	31.8		17.7	0.22	7.31	0.13	0.01	11.3	0.35	23.4		
U		79.0			11600		1510	0.51	31.7		17.7	0.22	7.30	0.13	0.02	11.3	0.35	23.5		
U		75.0			11300		1480	0.52	31.9		17.8	0.22	7.36	0.13	0.02	11.4	0.35	23.5		
U		76.0			11200		1470	0.51	32.0		17.9	0.22	7.33	0.14	0.03	11.4	0.35	23.5		
U		76.0			11100		1400	0.51	31.8		17.8	0.22	7.34	0.13	0.03	11.4	0.35	23.5		
U		79.0			11000		1550	0.51	31.8		17.8	0.22	7.30	0.13	0.02	11.3	0.35	23.5		
V	0.13	73.0	68.0	11300	11000	10600	1579	0.30	31.7		18.0	0.18	7.50	0.12		11.0	0.32	23.7	0.99	3.08
V	0.10	74.0	77.0	11300	11200	11200	1617	0.20	32.1		18.4	0.17	7.50	0.12		11.1	0.33	23.6	0.99	3.09
V	0.09	68.0	81.0	10900	11000	11400	1607	0.20	32.0		18.3	0.15	7.50	0.12		11.0	0.33	23.6	0.97	3.08
V	0.10	73.0	72.0	11400	10800	11000	1636	0.20	32.1		18.3	0.16	7.50	0.12		11.0	0.33	23.7	0.96	3.08
V	0.10	71.0	78.0	10900	10800	10900	1590	0.20	32.0		18.3	0.17	7.50	0.12		11.0	0.33	23.7	0.97	3.08
V	0.11	70.0	75.0	11400	11200	11200	1520	0.20	32.0		18.3	0.18	7.50	0.12		11.0	0.32	23.7	0.97	3.08
V	0.10	73.0	70.0	11300	11100	11300	1568	0.30	32.1		18.3	0.16	7.50	0.12		11.0	0.33	23.6	0.96	3.08
V	0.10	70.0	74.0	11600	10900	10800	1449	0.30	31.6		18.0	0.16	7.40	0.12		11.0	0.32	23.6	0.97	3.08
W	0.10	64.0	70.0	11800																0.83
W	0.10	66.3	76.0	11900																0.80
W	0.10	64.1	77.0	11900																0.81
W	0.09	67.0	76.0	11800																0.86
W	0.10	65.5	77.0	11800																0.84
W	0.10	65.5	78.0	11700																0.81
W	0.11	64.8	78.0	11700																0.85
W	0.10	63.9	77.0	11700																0.83

### 12. Measurement of Uncertainty: (ref Dr Hugh Bartlett, Hugh Bartlett Consulting CC.)

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 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:

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

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 <sup>1</sup>	$\sigma_L$ <sup>2</sup>	SW <sup>3</sup>	CSU <sup>4</sup>
Au	Pb Coll	g/t	0.006	0.003	0.005	0.001
Co	M/ICP	ppm	8.20	5.77	2.91	1.51
Co	P	ppm	4.67	3.61	2.61	1.18
Cu	Fus	ppm	263	209	186.8	82.8
Cu	M/ICP	ppm	287	178	159.8	48.2
Cu	P	ppm	220	143	154	48.6
Cu	Soluble	ppm	224	206	45.0	65.4
Al <sub>2</sub> O <sub>3</sub>	XRF	%	0.015	0.013	0.008	0.005
CaO	XRF	%	0.217	0.189	0.120	0.068
Cr <sub>2</sub> O <sub>3</sub>	XRF	%	0.004	0.004	0.002	0.002
Fe <sub>2</sub> O <sub>3</sub>	XRF	%	0.151	0.143	0.081	0.055
K <sub>2</sub> O	XRF	%	0.005	0.004	0.003	0.002
LOI		%	0.065	0.052	0.031	0.017
MgO	XRF	%	0.037	0.027	0.026	0.010
MnO	XRF	%	0.005	0.005	0.002	0.002
Na <sub>2</sub> O	XRF	%	0.014	0.017	0.006	0.008
SiO <sub>2</sub>	XRF	%	0.070	0.066	0.040	0.025
TiO <sub>2</sub>	XRF	%	0.005	0.003	0.004	0.001
S Comb	LECO	%	0.062	0.062	0.025	0.024
SG	pyc		0.044	0.034	0.028	0.012

1. S - Std Dev for use on control charts.
2.  $\sigma_L$  - 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. Certified values:** The Certified, Provisional and Indicated values listed on p1 and p2 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 and who have maintained measurement traceability during the analytical process.

**15. Certification:** AMIS0424 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](http://www.amis.co.za) website.

**17. Minimum sample size:** The majority of laboratories reporting used a 0.5g sample size for the ICP and a 30g sample size for the fire assay. 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 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.

**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 20 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.

**23 October 2013**

**Certifying Officers:**



**African Mineral Standards:** \_\_\_\_\_

**Mike McWha**  
**BSc (Hons), FGSSA, MAusIMM, Pr.Sci.Nat**



**Geochemist:** \_\_\_\_\_

**Barry W. Smee**  
**BSc, PhD, P.Geo, (B.C.)**



### Appendix – uncertified trace element statistics

Analyte	Method	Unit	Mean	2SD	RSD%	n
Ag	M/ICP	ppm	1.2	0.80	32.4	70
Al	M/ICP	%	0.26	0.05	9.2	104
As	M/ICP	ppm	24.1	16.9	35.0	92
Ba	M/ICP	ppm	408	36.3	4.4	75
Be	M/ICP	ppm	0.23	0.10	20.5	31
Bi	M/ICP	ppm	1.3	0.14	5.1	38
Ca	M/ICP	%	22.4	1.6	3.5	67
Cd	M/ICP	ppm	0.30	0.67	111	36
Ce	M/ICP	ppm	538	49.9	4.6	32
Co	Fusion	ppm	85.8	12.6	7.3	16
Cr	M/ICP	ppm	69.3	18.0	13.0	77
Cs	M/ICP	ppm	0.25	0.13	26.3	24
Dy	M/ICP	ppm	15.1	0.60	2.0	16
Er	M/ICP	ppm	3.7	0.26	3.4	16
Eu	M/ICP	ppm	12.1	0.38	1.6	16
Fe	M/ICP	%	12.7	0.91	3.6	96
Ga	M/ICP	ppm	6.9	2.3	16.8	40
Gd	M/ICP	ppm	32.6	15.6	23.9	24
Ge	M/ICP	ppm	0.76	0.79	52.4	16
Hf	M/ICP	ppm	6.8	22.1	162	44
Ho	M/ICP	ppm	1.4	1.7	63.2	24
In	M/ICP	ppm	0.11	0.07	29.8	40
K	M/ICP	%	0.20	0.03	7.6	96
La	M/ICP	ppm	232	48.9	10.5	55
Li	M/ICP	ppm	2.2	1.1	26.4	48
Lu	M/ICP	ppm	0.21	0.06	13.5	32
Mg	M/ICP	%	4.1	0.39	4.7	99
Mn	M/ICP	ppm	979	87.3	4.5	99
Mo	M/ICP	ppm	0.85	0.28	16.8	40
Na	M/ICP	%	0.03	0.02	24.7	87
Nb	M/ICP	ppm	6.5	2.8	21.3	46
Nd	M/ICP	ppm	325	18.3	2.8	16
Ni	M/ICP	ppm	135	24.2	9.0	102
P	M/ICP	ppm	14541	1391	4.8	92
Pb	M/ICP	ppm	38.3	20.8	27.1	88
Pr	M/ICP	ppm	76.4	4.1	2.7	16
Rb	M/ICP	ppm	13.2	1.2	4.6	37
S	M/ICP	%	0.78	0.12	7.6	93
Sb	M/ICP	ppm	2.1	6.0	142	45
Sc	M/ICP	ppm	11.4	2.1	9.4	95
Se	M/ICP	ppm	30.0	56.6	94.3	16
Si	M/ICP	%	5.2	0.05	0.50	8
Sm	M/ICP	ppm	31.1	56.2	90.4	16
Sn	M/ICP	ppm	5.2	1.2	11.9	46
Sr	M/ICP	ppm	3460	340	4.9	100
Ta	M/ICP	ppm	0.72	1.2	86.1	38
Tb	M/ICP	ppm	4.0	1.3	16.0	32
Te	M/ICP	ppm	0.43	0.09	10.1	28
Th	M/ICP	ppm	161	23.3	7.2	62
Ti	M/ICP	%	0.19	0.05	12.7	73
Tl	M/ICP	ppm	0.04	0.01	14.2	23
Tm	M/ICP	ppm	0.29	0.20	33.5	16
U	M/ICP	ppm	38.7	7.9	10.2	44
V	M/ICP	ppm	97.9	14.7	7.5	99
W	M/ICP	ppm	0.23	0.09	19.7	24
Y	M/ICP	ppm	50.8	9.5	9.4	60
Yb	M/ICP	ppm	1.6	0.46	14.1	32
Zn	M/ICP	ppm	65.5	20.8	15.9	101
Zr	M/ICP	ppm	149	223	74.7	67