



African Mineral Standards

MATRIX REFERENCE MATERIALS

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## AMIS0247

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

**Copper cobalt oxide ore  
Kinsevere, DRC**

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

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

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

Co F	1064	±	47	ppm
Co M/ICP	1034	±	73	ppm
Co P	1061	±	112	ppm
Cu F	4.123	±	0.325	%
Cu M/ICP	4.134	±	0.225	%
Cu P	4.094	±	0.117	%
Specific Gravity	2.86	±	0.10	

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>	13.58	±	0.26	%
Fe <sub>2</sub> O <sub>3</sub>	3.39	±	0.18	%
K <sub>2</sub> O	2.69	±	0.12	%
MgO	4.73	±	0.19	%
SiO <sub>2</sub>	62.65	±	0.98	%
TiO <sub>2</sub>	0.85	±	0.04	%
LOI	6.45	±	0.74	%

## ***Provisional Concentrations***

CaO	0.07	±	0.02	%
MnO	0.030	±	0.004	%
Na <sub>2</sub> O	0.07	±	0.01	%

## ***Indicated Mean***

Cr <sub>2</sub> O <sub>3</sub>	0.03	%
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**1. Intended Use:** AMIS0247 can be used to check analysis of samples of copper cobalt ores with a similar grade and matrix.

It is a matrix matched Certified Reference Material (CRM) 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:** AMIS0247 is a commissioned CRM made from “DSH” material supplied by SGS Minerals Services from the Kinsevere Copper Mine, 27 km north on Lubumbashi, in the Katanga Province of the Democratic Republic of Congo (DRC). Kinsevere, formerly owned by Anvil Mining Limited, is now 95% owned by Minmetals Resources.

The Kinsevere Project comprises three known structurally modified stratiform copper deposits; Tshifufia, Tshifufiamashi and Kinsevere Hill. These are hosted within interbedded calcareous siltstones and silty dolomites of the Mines Group, overturned to a steep eastward dip during the Lufilian Orogeny (approximately 550 Ma). Deep weathering, extending to approximately 100 m below surface, has led to the development of a large oxide resource hosted by highly altered dolomitic and argillaceous sediments. Underlying sulphide mineralisation comprises chalcopyrite in crosscutting veins and stringers.

**3. Mineral and Chemical Composition:** Oxide ore mineralogy at Tshifufia, Tshifufiamashi and Kinsevere Hill is composed predominantly of malachite and pseudomalachite, with minor chrysocolla and rare intergrown heterogenite. These occur as disseminations and/or in veins and veinlets, which sometimes coalesce into prominent "clots". Underlying sulphide mineralisation comprises chalcopyrite, chalcocite, and bornite; typically occurring as finely disseminated, bedding parallel layers, stratiform veins or as replacement of pyrite nodules. Lesser cross cutting quartz-carbonate- sulphide veins are also observed. Sulphide remobilisation during faulting has also led to the development of variably mineralised breccias.

Within the oxide zone, cobalt mineralogy is composed predominantly of rare intergrown heterogenite (CoO(OH)). Heterogenite is generally limited to vuggy infills in well-developed malachite veins along with manganese oxides.

Within the sulfide zone, carrollite is the dominant cobalt mineral. It appears to be an early formed mineral since it is rimmed or replaced by chalcopyrite or bornite. It is more resistant to weathering than Cu sulfides and often survives into the supergene and mixed zones. The presence of very coarse and disseminated carrollite plus pyrite in late stage siliceous and dolomitic veins appears to denote another phase of mineralization that may postdate the principal copper mineralizing event.

**4. Appearance:** The material is a very fine Pale Yellow powder (Corstor 5Y 7/4).

**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 homogenized in a double cone blender, systematically divided and 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. Co, Cu. Fusion AAS or ICP-OES (F).
2. Multi-acid digest multi-element scan - ( to include Co, Cu ). ICP-OES or ICP-MS (M/ICP).
3. Aqua regia digest – Co, Cu. ICP-OES or ICP-MS (P).
4. Pressed pellet multi-element scan - (to include Co, Cu ) (XRF).
5. 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.
6. SG. Gas pycnometer.

**8. Information requested:**

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

**9. Method of Certification:** Twenty two laboratories were each given eight packages, comprising eight samples scientifically selected from throughout the batch. Seventeen laboratories reported results in time for certification

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 17 out of 22 laboratories that provided results timeously were (not in same order as in the table of assays):

1. Activation Laboratories Pty Ltd (ActLabs) CA
2. Alex Stewart International Corporation Zambia
3. ALS Chemex Laboratory Group Brisbane Australia
4. ALS Chemex Laboratory Group Johannesburg SA
5. ALS Chemex Laboratory Group Vancouver CA
6. Genalysis Laboratory Services (South Africa) Pty
7. Genalysis Laboratory Services W Australia
8. Intertek Utama Services (Indonesia)
9. Set Point Laboratories (Isando) SA
10. SGS Australia Pty Ltd (Newburn) WA
11. SGS Durango (Mexico)
12. SGS Geosol Laboratories Ltda (Brazil)
13. SGS Mineral Services Callao (Peru)
14. SGS Mineral Services Lakefield (Canada)
15. SGS South Africa (Pty) Ltd - Booyens JHB
16. SGS Toronto (Canada)
17. 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.

Lab Code	Co F ppm	Co M/ICP ppm	Co P ppm	Co XRF ppm	Cu F ppm	Cu M/ICP ppm	Cu P ppm	Cu XRF ppm	Al2O3 XRF %	CaO XRF %	Cr2O3 XRF %	Fe2O3 XRF %	K2O XRF %	MgO XRF %	MnO XRF %	Na2O XRF %	SiO2 XRF %	TiO2 XRF %	LOI %	S Comb LECO %	SG pyc
A		1050	1010	1100		40300	41300	42400	13.5	0.06	0.01	3.41	2.65	4.72	0.03	0.08	62.20	0.81	6.21		
A		1060	1020	1100		40700	41600	42400	13.5	0.06	0.02	3.46	2.66	4.73	0.03	0.08	62.10	0.81	6.17		
A		1090	1000	1100		41500	40600	42700	13.6	0.07	0.01	3.46	2.66	4.77	0.03	0.08	62.60	0.83	6.37		
A		1070	990	1100		40900	40400	42600	13.7	0.06	0.02	3.45	2.67	4.75	0.03	0.08	62.60	0.82	6.33		
A		1080	1010	1100		41600	41400	42600	13.6	0.07	0.02	3.43	2.65	4.75	0.03	0.08	62.50	0.82	6.19		
A		1060	1000	1100		41100	40900	42400	13.5	0.06	0.02	3.43	2.66	4.74	0.03	0.08	62.30	0.82	6.30		
A		1080	1000	1100		40700	40900	42400	13.5	0.06	0.02	3.40	2.66	4.72	0.03	0.08	62.10	0.81	6.38		
A		1080	1020	1100		40500	41400	42400	13.7	0.06	0.01	3.39	2.66	4.74	0.03	0.08	62.50	0.82	6.21		
C	999	1048	1141			42702				0.07							0.08				
C	1074	1118	1139			45313				0.07							0.08				
C	998	1020	1140			37951				0.07							0.08				
C	965	1018	1136			44680				0.07							0.07				
C	1040	1022	1117			42295				0.07							0.08				
C	1051	1094	1107			42561				0.07							0.07				
C	953	1058	1090			40782				0.07							0.08				
C	944	1064	1100			39696				0.07							0.08				
D		956	1060				40600								0.03	0.07					
D		982	1050				40400								0.03	0.07					
D		972	1010				40900								0.03	0.07					
D		966	1000				41500								0.03	0.07					
D		971	1020				40800								0.03	0.07					
D		987	966				37500								0.03	0.07					
D		1010	1100				40600								0.03	0.07					
D		996	1060				40700								0.03	0.07					
E		1010	913			42300	42400		13.5	0.07	0.03	3.35	2.67	4.67	0.04		62.72	0.84			
E		1040	908			42500	43000		13.6	0.07	0.02	3.36	2.65	4.68	0.04		62.97	0.84			
E		1030	911			42300	42300		13.5	0.07	0.02	3.34	2.64	4.64	0.04		62.57	0.83			
E		1000	921			40900	42200		13.6	0.07	0.03	3.36	2.65	4.68	0.03		62.80	0.83			
E		998	901			40600	42300		13.6	0.07	0.02	3.36	2.65	4.69	0.04		62.83	0.84			
E		1010	908			41500	42600		13.6	0.07	0.02	3.36	2.66	4.70	0.04		62.99	0.84			
E		1010	902			41600	42200		13.6	0.07	0.03	3.35	2.67	4.69	0.04		62.78	0.84			
E		1010	940			42400	42300		13.5	0.07	0.03	3.35	2.66	4.67	0.04		62.60	0.84			

**Assay data (cont)**



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:

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

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>
Co	F	ppm	23.2	15.2	18.8	6.31
Co	M/ICP	ppm	36.6	29.6	17.9	9.57
Co	P	ppm	56.0	42.5	23.3	12.5
Cu	F	ppm	1625	1331	890	456
Cu	M/ICP	ppm	1126	818	525	243
Cu	P	ppm	1194	1141	644	440
Al <sub>2</sub> O <sub>3</sub>	XRF	%	0.18	0.13	0.12	0.05
CaO	XRF	%	0.01	0.01	0.01	0.00
Cr <sub>2</sub> O <sub>3</sub>	XRF	%	0.01	0.01	0.00	0.00
Fe <sub>2</sub> O <sub>3</sub>	XRF	%	0.11	0.10	0.06	0.03
K <sub>2</sub> O	XRF	%	0.06	0.05	0.03	0.01
LOI		%	0.37	0.30	0.28	0.13
MgO	XRF	%	0.10	0.07	0.05	0.02
MnO	XRF	%	0.002	0.001	0.001	0.000
Na <sub>2</sub> O	XRF	%	0.006	0.004	0.004	0.001
SiO <sub>2</sub>	XRF	%	0.49	0.38	0.32	0.14
TiO <sub>2</sub>	XRF	%	0.02	0.02	0.01	0.01
SG	pyc		0.05	0.04	0.03	0.02

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

**15. Certification:** AMIS0247 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. This is the recommended minimum sample size 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 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.

20 October 2012

**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	0.20	0.19	47.1	19
Al	M/ICP	%	6.8	0.94	6.9	80
As	M/ICP	ppm	29.4	14.7	24.9	88
Ba	M/ICP	ppm	129	24.4	9.5	82
Be	M/ICP	ppm	6.8	1.7	12.6	75
Bi	M/ICP	ppm	4.4	14.2	163	53
Ca	M/ICP	%	0.05	0.01	12.0	89
Cd	M/ICP	ppm	14.9	82.8	278	36
Ce	M/ICP	ppm	229	95.3	20.8	39
Co	XRF	ppm	1002	267.2	13.3	32
Cr	M/ICP	ppm	130	65.6	25.3	72
Cs	M/ICP	ppm	1.3	0.11	4.1	31
Cu	XRF	ppm	42402	805	0.95	30
Dy	M/ICP	ppm	6.3	0.47	3.8	31
Er	M/ICP	ppm	3.5	1.4	19.3	40
Eu	M/ICP	ppm	1.0	0.1	5.2	31
Fe	M/ICP	%	2.3	0.21	4.6	86
Ga	M/ICP	ppm	19.1	5.4	14.2	46
Gd	M/ICP	ppm	7.2	1.3	9.2	31
Ge	M/ICP	ppm	1.1	0.26	12.3	8
Hf	M/ICP	ppm	6.8	2.6	19.2	39
Ho	M/ICP	ppm	1.3	0.11	4.4	30
In	M/ICP	ppm	0.57	0.06	5.6	38
K	M/ICP	%	2.2	0.21	4.9	91
La	M/ICP	ppm	128	35.9	14.0	78
Li	M/ICP	ppm	98.8	11.0	5.6	90
Lu	M/ICP	ppm	0.53	0.08	7.8	39
Mg	M/ICP	%	2.8	0.25	4.4	92
Mn	M/ICP	ppm	224	32.3	7.2	94
Mo	M/ICP	ppm	2.5	1.4	28.2	75
Na	M/ICP	%	0.06	0.06	43.7	88
Nb	M/ICP	ppm	15.9	17.7	55.4	55
Nd	M/ICP	ppm	96.3	12.5	6.5	31
Ni	M/ICP	ppm	119	20.1	8.4	92
P	M/ICP	ppm	808	131	8.1	72
Pb	M/ICP	ppm	12.1	11.5	47.7	72
Pr	M/ICP	ppm	29.3	2.4	4.0	31
Rb	M/ICP	ppm	70.8	14.4	10.2	38
S	M/ICP	%	0.08	0.01	8.2	63
Sb	M/ICP	ppm	3.2	4.0	62.7	57
Sc	M/ICP	ppm	13.0	2.2	8.5	84
Se	M/ICP	ppm	4.2	1.5	18.0	37
Si	M/ICP	%	29.8	0.76	1.3	8
Sm	M/ICP	ppm	11.5	0.93	4.0	31
Sn	M/ICP	ppm	4.2	8.2	97.4	38
Sr	M/ICP	ppm	101	12.0	6.0	80
Ta	M/ICP	ppm	1.5	1.3	41.7	32
Tb	M/ICP	ppm	1.0	0.44	21.4	40
Te	M/ICP	ppm	0.07	0.03	22.9	12
Th	M/ICP	ppm	17.4	2.7	7.8	39
Ti	M/ICP	%	0.25	0.27	54.1	79
Tl	M/ICP	ppm	0.20	0.03	6.5	37
Tm	M/ICP	ppm	0.57	0.06	5.4	31
U	M/ICP	ppm	15.1	1.3	4.3	39
V	M/ICP	ppm	177	33.9	9.6	94
W	M/ICP	ppm	1.5	1.1	35.8	37
Y	M/ICP	ppm	29.8	9.3	15.6	84
Yb	M/ICP	ppm	3.6	0.60	8.4	39
Zn	M/ICP	ppm	30.6	14.6	23.9	80
Zr	M/ICP	ppm	216	48.0	11.1	78