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CERTIFICATE OF ANALYSIS FOR

Au-Cu-Mo-S ORE REFERENCE MATERIAL

OREAS 152a

SUMMARY STATISTICS

Constituent	Certified Value	1SD	95% Confidence Interval	
			Low	High
Gold, Au (ppb)	116	5	114	118
Copper, Cu (wt.%)	0.385	0.009	0.379	0.391
Molybdenum, Mo (ppm)	80	5	77	82
Sulphur, S (wt.%)	0.921	0.046	0.900	0.941

Prepared by:

ORE Research & Exploration Pty Ltd

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INTRODUCTION

OREAS reference materials (RM) are intended to provide a low cost method of evaluating and improving the quality of precious and base metal analysis of geological samples. To the explorationist, they provide an important control in analytical data sets related to exploration from the grass roots level through to resource definition. To the mine geologist, they provide a tool for grade control in routine mining operations. To the analyst, they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

SOURCE MATERIALS

OREAS 152a is one of three porphyry Au-Cu-Mo-S certified reference materials prepared from copper ore from the Waisoi district, Viti Levu, Fiji. The two deposits in the area are the Waisoi East deposit (quartz porphyry) and the Waisoi West deposit (diorite porphyry). Copper mineralisation in the region is accompanied by stockwork quartz veinlets and is characterised by bornite-chalcopyrite-pyrite assemblages formed under a high sulphidation environment.

COMMUNITION AND HOMOGENISATION PROCEDURES

OREAS 152a was prepared in the following manner:

- a) *jaw crushing to minus 3mm;*
- b) *drying to constant mass at 105°C;*
- c) *multi-stage milling to 100% minus 30 microns;*
- d) *homogenisation;*
- e) *blending and bagging into 25kg sublots;*
- f) *packaging into 60g (laminated foil pouches) and 1kg (plastic jars) units.*

ANALYSIS OF OREAS 152a

Twenty-one laboratories participated in the analytical program to characterise gold (21 labs), copper and molybdenum (19 labs) and sulphur (17 labs). They are listed in the section headed 'Participating Laboratories'. To maintain anonymity these laboratories have been randomly designated the letter codes A through U. Each laboratory received two scoop-split 110 gram subsamples from each of three 800g test units (6 samples in total). A total of 20 of these 800g test units were taken at regular intervals during the bagging stage and are considered representative of the entire batch. For each sample laboratories were requested to carry out one 30-50 gram fire assay determination for gold, employing an appropriate low level method with new pots, and one 4-acid digest determination for copper, molybdenum and sulphur using their preferred finish (Lab S used infra red combustion furnace to determine sulphur). The nested design of the interlaboratory programme is amenable to analysis of variance (ANOVA) and enables a comparative assessment of within- and between-unit homogeneity (see 'ANOVA study' section).

For the determination of a statistical tolerance interval for gold a 10 gram scoop split was taken from each of the 20 test units and submitted to 'Lab A' for analysis via instrumental neutron activation analysis on a reduced analytical subsample weight of ~1.3 gram.

The approximate major and trace element composition of OREAS 152a is given in Table 1. The constituents SiO₂ to LOI are the means of duplicate XRF analyses determined using a lithium borate fusion method, C and S are means of duplicate IR combustion furnace analyses, while the remaining constituents, Ag to Zr, are means of duplicate analyses determined by 4-acid digestion with ICP-MS finish (except for Cu, Zn, Co, Ni and Sc via ICP-OES finish). Individual assay results for gold via fire assay and INAA are presented in Tables 2 and 3, respectively, and results for copper, molybdenum and sulphur are presented in Tables 4 to 6, respectively. These results are shown together with the mean, median, standard deviations (absolute and relative) and percent deviation of the lab mean from the corrected mean of means for each data set (PDM³). The analytical methods employed by each laboratory are given in the table captions. For gold, interlaboratory agreement of the fire assay means is very good with all labs lying within 7.6% relative of the certified value. For copper, interlaboratory agreement of the lab means is also very good with all labs but two within 5.7% relative of the certified value. For molybdenum, all labs but one fall within 8.4% relative of the certified value and for sulphur, all labs but one fall within 5.7% relative of the certified value.

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 152a; wt.% - weight percent; ppm - parts per million.

Constituent	wt. %	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO ₂	61.70	Ag	1	Hf	0.2	Sc	23
TiO ₂	0.70	As	32	Ho	0.48	Sm	2.2
Al ₂ O ₃	16.13	Ba	86	In	<0.02	Sn	2
Fe ₂ O ₃	5.22	Be	0.4	La	4.4	Sr	113
MgO	3.59	Bi	0.1	Li	6.3	Ta	<0.1
MnO	0.039	Cd	<0.5	Lu	0.14	Tb	0.36
CaO	2.21	Ce	11.1	Mo	77.8	Te	0.4
Na ₂ O	3.14	Co	15	Nb	1	Th	0.5
K ₂ O	1.75	Cs	0.7	Nd	7.85	U	0.1
P ₂ O ₅	0.150	Cu	3875	Ni	14	W	4.0
LOI	3.96	Dy	2.30	Pb	7	Y	13.1
Total	100.8	Er	1.23	Pr	1.62	Yb	1.1
C	0.28	Eu	0.73	Rb	28.1	Zn	81
S	0.90	Ga	17.8	Re	0.3	Zr	10
		Gd	2.4	Sb	1.4		

Table 2. Analytical results for gold in OREAS 152a (FA - fire assay; AAS - flame atomic absorption spectrometry; SXAAS - solvent extraction atomic absorption spectrometry; GFAAS - graphite furnace atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; MS - inductively coupled plasma mass spectrometry; Std.Dev. - one sigma standard deviation; Rel.Std.Dev. - one sigma relative standard deviation; PDM³ - percent deviation of lab mean from corrected mean of means; outliers in bold and left justified; sample charge weights shown in row 3; values in ppb).

Replicate No.	Lab A FA*AAS 30g	Lab B FA*OES 30g	Lab C FA*SXAAS 30g	Lab D FA*SXAAS 25g	Lab E FA*GFAAS 50g	Lab F FA*OES 30g	Lab G FA*OES 30g	Lab H FA*AAS 25g	Lab I FA*OES 40g	Lab J FA*OES 30g
1	113	116	110	121	125	120	118	109	112	110
2	108	117	112	122	124	117	119	116	110	113
3	107	122	111	121	125	119	126	108	112	114
4	111	111	111	121	122	119	129	110	109	115
5	109	115	111	122	124	120	123	108	113	113
6	112	117	107	123	128	117	120	110	111	111
Mean	110	116	110	122	125	119	123	110	111	113
Median	110	117	111	122	125	119	122	110	112	113
Std.Dev.	2	4	2	1	2	1	4	3	1	2
Rel.Std.Dev	2.15%	3.06%	1.59%	0.67%	1.58%	1.15%	3.53%	2.72%	1.32%	1.65%
PDM ³	-5.05%	0.42%	-4.76%	5.03%	7.62%	2.44%	5.74%	-4.90%	-4.04%	-2.74%

Table 2 continued

Lab K FA*AAS 30g	Lab L FA*OES 30g	Lab M FA*AAS 30g	Lab N FA*SXAAS 30g	Lab O FA*OES 30g	Lab P FA*MS 30g	Lab Q FA*OES 30g	Lab R FA*AAS 30g	Lab S FA*AAS 30g	Lab T FA*AAS 30g	Lab U FA*OES 30g
110	110	120	120	118	118	115	110	116	116	112
112	110	118	125	116	118	114	106	120	114	111
112	111	111	113	118	120	119	120	110	118	112
117	112	121	122	119	120	118	116	116	116	113
117	113	118	126	122	116	117	132	117	116	111
114	113	118	122	118	117	119	110	120	116	110
114	112	118	121	118	118	117	116	117	116	112
113	112	118	122	118	118	118	113	117	116	112
3	1	4	5	2	2	2	9	4	1	1
2.53%	1.24%	2.98%	3.82%	1.66%	1.36%	1.76%	8.14%	3.15%	1.09%	0.94%
-1.88%	-3.75%	1.57%	4.74%	2.27%	2.00%	1.30%	-0.15%	0.57%	0.13%	-3.75%

Table 3. Analytical results for gold in OREAS 152a by INAA (instrumental neutron activation analysis on ~1.3 gram analytical subsample weights; other abbreviations as for Table 2).

Replicate No.	Lab A INAA ~1.3g
1	107
2	108
3	110
4	105
5	106
6	109
7	110
8	106
9	106
10	110
11	109
12	114
13	105
14	108
15	109
16	113
17	111
18	109
19	105
20	111
Mean	109
Median	109
Std.Dev.	2.6
Rel.Std.Dev.	2.40%
PDM ³	-6.30%

Table 4. Analytical results for copper in OREAS 152a (4A - four acid digest (HNO₃-HClO₄-HCl-HF); AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; MS - inductively coupled plasma mass spectrometry; other abbreviations as for Table 2; values in wt.%).

Replicate No.	Lab A 4A*OES	Lab B 4A*MS	Lab C 4A*OES	Lab D 4A*OES	Lab E 4A*OES	Lab F 4A*OES	Lab G 4A*OES	Lab H 4A*OES	Lab I 4A*OES	Lab J 4A*OES
1	0.400	0.373	0.380	0.355	0.386	0.385	0.375	0.396	0.398	0.389
2	0.392	0.379	0.371	0.362	0.384	0.392	0.382	0.401	0.396	0.398
3	0.402	0.380	0.383	0.364	0.389	0.396	0.387	0.401	0.395	0.379
4	0.397	0.376	0.398	0.367	0.397	0.434	0.378	0.393	0.390	0.398
5	0.380	0.371	0.394	0.370	0.397	0.385	0.390	0.391	0.387	0.403
6	0.399	0.375	0.380	0.360	0.399	0.400	0.381	0.399	0.388	0.379
Mean	0.395	0.375	0.384	0.363	0.392	0.399	0.382	0.397	0.392	0.391
Median	0.398	0.376	0.382	0.363	0.393	0.394	0.381	0.398	0.393	0.394
Std.Dev.	0.008	0.004	0.010	0.005	0.006	0.018	0.005	0.004	0.005	0.010
Rel.Std.Dev	2.05%	0.96%	2.59%	1.40%	1.65%	4.59%	1.42%	1.07%	1.17%	2.64%
PDM ³	2.69%	-2.39%	-0.08%	-5.68%	1.91%	3.65%	-0.64%	3.19%	2.00%	1.65%

Table 4 continued

Lab K -	Lab L 4A*OES	Lab M -	Lab N 4A*OES	Lab O 4A*OES	Lab P 4A*OES	Lab Q 4A*OES	Lab R 4A*OES	Lab S 4A*OES	Lab T 4A*OES	Lab U 4A*OES
NR	0.390	NR	0.377	0.391	0.387	0.360	0.343	0.380	0.372	0.353
NR	0.378	NR	0.389	0.386	0.386	0.370	0.352	0.380	0.376	0.345
NR	0.376	NR	0.388	0.387	0.392	0.379	0.339	0.380	0.383	0.349
NR	0.422	NR	0.366	0.391	0.382	0.374	0.336	0.380	0.393	0.350
NR	0.422	NR	0.390	0.386	0.392	0.368	0.315	0.400	0.390	0.347
NR	0.405	NR	0.387	0.386	0.385	0.378	0.352	0.370	0.393	0.349
	0.399		0.383	0.388	0.387	0.372	0.339	0.382	0.385	0.349
	0.398		0.388	0.387	0.387	0.372	0.341	0.380	0.387	0.349
	0.021		0.009	0.003	0.004	0.007	0.014	0.010	0.009	0.003
	5.19%		2.48%	0.68%	1.03%	1.99%	4.09%	2.58%	2.30%	0.76%
	3.69%		-0.47%	0.82%	0.70%	-3.39%	-11.7%	-0.77%	-0.03%	-9.33%

Table 5. Analytical results for molybdenum in OREAS 152a (4A - four acid digest (HNO₃-HClO₄-HCl-HF); OES - inductively coupled plasma optical emission spectrometry; MS - inductively coupled plasma mass spectrometry; other abbreviations as for Table 2; values in ppm).

Replicate No.	Lab A 4A*OES	Lab B 4A*MS	Lab C 4A*OES	Lab D 4A*MS	Lab E 4A*OES	Lab F 4A*OES	Lab G 4A*OES	Lab H 4A*OES	Lab I 4A*OES	Lab J 4A*OES
1	74.0	76.0	76.0	79.8	72.0	77.0	77.0	84.1	78.0	82.0
2	74.0	76.0	73.0	79.9	73.0	76.0	77.0	84.4	80.0	85.0
3	74.0	72.0	76.0	79.1	73.0	77.0	80.0	85.2	80.0	84.0
4	75.0	76.0	78.0	80.9	73.0	83.0	77.0	85.3	80.0	86.0
5	76.0	72.0	79.0	80.3	74.0	75.0	80.0	85.5	80.0	86.0
6	74.0	74.0	76.0	79.6	72.0	77.0	79.0	86.1	82.0	85.0
Mean	74.5	74.3	76.3	79.9	72.8	77.5	78.3	85.1	80.0	84.7
Median	74.0	75.0	76.0	79.9	73.0	77.0	78.0	85.3	80.0	85.0
Std.Dev.	0.8	2.0	2.1	0.6	0.8	2.8	1.5	0.7	1.3	1.5
Rel.Std.Dev	1.12%	2.65%	2.71%	0.77%	1.03%	3.63%	1.92%	0.88%	1.58%	1.78%
PDM ³	-6.34%	-6.55%	-4.03%	0.49%	-8.43%	-2.57%	-1.52%	6.98%	0.58%	6.44%

Table 5 continued

Lab K -	Lab L 4A*OES	Lab M -	Lab N 4A*OES	Lab O 4A*OES	Lab P 4A*OES	Lab Q 4A*OES	Lab R 4A*OES	Lab S 4A*OES	Lab T 4A*OES	Lab U 4A*OES
NR	81.0	NR	72.0	81.6	82.0	75.3	87.0	82.0	87.0	76.2
NR	80.0	NR	74.0	79.6	83.0	75.7	89.0	81.0	85.0	74.7
NR	79.0	NR	74.0	83.6	82.0	76.3	83.0	83.0	92.0	76.1
NR	87.0	NR	73.0	84.2	82.0	75.7	87.0	88.0	89.0	75.6
NR	85.0	NR	75.0	82.1	83.0	75.0	80.0	82.0	89.0	75.8
NR	84.0	NR	76.0	80.3	83.0	76.6	87.0	77.0	85.0	75.0
	82.7		74.0	81.9	82.5	75.8	85.5	82.2	87.8	75.6
	82.5		74.0	81.8	82.5	75.7	87.0	82.0	88.0	75.7
	3.1		1.4	1.8	0.5	0.6	3.3	3.5	2.7	0.6
	3.80%		1.91%	2.16%	0.66%	0.82%	3.90%	4.31%	3.09%	0.80%
	3.93%		-6.97%	2.97%	3.72%	-4.74%	7.49%	3.30%	10.4%	-5.00%

Table 6. Analytical results for sulphur in OREAS 152a (4A - four acid digest (HNO₃-HClO₄-HCl-HF); OES - inductively coupled plasma optical emission spectrometry; IRC - infra red combustion furnace; other abbreviations as for Table 2; values in wt.%).

Replicate No.	Lab A 4A*OES	Lab B -	Lab C 4A*OES	Lab D 4A*OES	Lab E 4A*OES	Lab F 4A*OES	Lab G 4A*OES	Lab H 4A*OES	Lab I 4A*OES	Lab J 4A*OES
1	0.900	NR	0.930	0.850	0.863	0.910	0.800	0.966	0.910	0.978
2	0.870	NR	0.900	0.895	0.875	0.940	0.800	0.966	0.920	0.956
3	0.900	NR	0.930	0.871	0.870	0.930	0.800	0.967	0.940	0.902
4	0.860	NR	0.960	0.899	0.875	0.990	0.800	0.959	0.915	1.010
5	0.880	NR	0.960	0.881	0.863	0.900	0.900	0.959	0.930	1.020
6	0.870	NR	0.930	0.883	0.861	0.920	0.900	0.973	0.925	0.925
Mean	0.880		0.935	0.880	0.868	0.932	0.833	0.965	0.923	0.965
Median	0.875		0.930	0.882	0.867	0.925	0.800	0.966	0.923	0.967
Std.Dev.	0.017		0.023	0.018	0.006	0.032	0.052	0.005	0.011	0.047
Rel.Std.Dev	1.90%		2.42%	2.00%	0.73%	3.42%	6.20%	0.55%	1.17%	4.83%
PDM ³	-4.40%		1.57%	-4.42%	-5.72%	1.21%	-9.47%	4.85%	0.31%	4.85%

Table 6 continued

Lab K -	Lab L 4A*OES	Lab M -	Lab N 4A*OES	Lab O 4A*OES	Lab P 4A*OES	Lab Q -	Lab R 4A*OES	Lab S IRC	Lab T 4A*OES	Lab U 4A*OES
NR	0.940	NR	0.930	0.896	0.976	NR	0.920	0.900	0.900	0.958
NR	0.920	NR	0.940	0.880	0.968	NR	0.850	0.920	0.910	0.952
NR	0.920	NR	0.950	0.899	0.962	NR	0.930	0.910	0.940	0.966
NR	1.030	NR	0.930	0.900	0.952	NR	0.870	0.920	0.950	0.962
NR	1.010	NR	0.950	0.900	0.985	NR	0.910	0.900	0.960	0.964
NR	0.990	NR	0.960	0.868	0.988	NR	0.890	0.910	0.940	0.969
	0.968		0.943	0.891	0.972		0.895	0.910	0.933	0.962
	0.965		0.945	0.898	0.972		0.900	0.910	0.940	0.963
	0.048		0.012	0.013	0.014		0.031	0.009	0.023	0.006
	4.95%		1.28%	1.51%	1.42%		3.44%	0.98%	2.51%	0.63%
	5.20%		2.48%	-3.23%	5.58%		-2.77%	-1.14%	1.39%	4.49%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 152a

Certified Value and Confidence Limits

The certified value was determined from the mean of means of accepted replicate values of accepted laboratory data sets A to U (excluding the INAA data) according to the formulae

$$\bar{x}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} x_{ij}$$

$$\ddot{x} = \frac{1}{p} \sum_{i=1}^p \bar{x}_i$$

where

x_{ij} is the j th result reported by laboratory i ;

p is the number of participating laboratories;

n_i is the number of results reported by laboratory i ;

\bar{x}_i is the mean for laboratory i ;

\ddot{x} is the mean of means.

The confidence limits were obtained by calculation of the variance of the consensus value (mean of means) and reference to Student's-*t* distribution with degrees of freedom (*p*-1):

$$\hat{V}(\bar{x}) = \frac{1}{p(p-1)} \sum_{i=1}^p (\bar{x}_i - \bar{x})^2$$

$$\text{Confidence limits} = \bar{x} \pm t_{1-x/2}(p-1) \left(\hat{V}(\bar{x}) \right)^{1/2}$$

where $t_{1-x/2}(p-1)$ is the 1-*x*/2 fractile of the *t*-distribution with (*p*-1) degrees of freedom.

The distribution of the values is assumed to be symmetrical about the mean in the calculation of the confidence limits.

The test for rejection of individual outliers from each laboratory data set was based on *z* scores (rejected if $|z_i| > 2.5$) computed from the robust estimators of location and scale, *T* and *S*, respectively, according to the formulae

$$S = 1.483 \frac{\text{median } |x_j - \text{median}(x_i)|}{j=1 \dots n \quad i=1 \dots n}$$

$$z_i = \frac{x_i - T}{S}$$

where

T is the median value in a data set;

S is the median of all absolute deviations from the sample median multiplied by 1.483, a correction factor to make the estimator consistent with the usual parameter of a normal distribution.

The *z*-score test is used in combination with a second method of individual outlier detection that determines the percent deviation of the individual value from the median. Outliers in general are selected on the basis of *z*-scores > 2.5 and with percent deviations $> 5\%$. In certain instances statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set is tested for outlying status based on *z*-score discrimination and rejected if $|z_i| > 2.5$. After individual and entire lab data set outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

Individual outliers and, more rarely, laboratory means deemed to be outlying are shown left justified and in bold in the tabulated results (Tables 2 to 6) and have been omitted in the determination of certified values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It is a measure of the reliability of the certified value, i.e. the narrower the confidence interval the greater the certainty in the certified value (Table 7).

Table 7. Certified Value and 95% Confidence Interval

Constituent	Certified Value	95% Confidence Interval	
		Low	High
Gold, Au (ppb)	116	114	118
Copper, Cu (wt.%)	0.385	0.379	0.391
Molybdenum, Mo (ppm)	80	77	82
Sulphur, S (wt.%)	0.921	0.900	0.941

Note: intervals may appear asymmetric due to rounding

Statement of Homogeneity

The variability of replicate assays from each laboratory is a result of both measurement and subsampling errors. In the determination of a statistical tolerance interval it is therefore necessary to eliminate, or at least substantially minimise, those errors attributable to measurement. One way of achieving this is by substantially reducing the analytical subsample weight to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible. This approach was adopted in the INAA gold data set (Table 3) where a ~1.3 gram subsample weight was employed.

The homogeneity was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO Guide 3207) in which

$$\text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha)s$$

$$\text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha)s$$

where

n is the number of results reported by laboratory *Q*;

1 - α is the confidence level;

p is the proportion of results expected within the tolerance limits;

k'₂ is the factor for two - sided tolerance limits (*m*, *σ* unknown);

and *s* is computed according to the formula

$$s = \left[\frac{\sum_{j=1}^n (x_j - \bar{x})^2}{n - 1} \right]^{1/2}$$

No individual outliers were removed from the results prior to the calculation of tolerance intervals.

Table 8. Certified Value and Tolerance Interval.

Constituent	Certified Value	Tolerance Interval 1- α =0.99, ρ =0.95	
		Low	High
Gold, Au (ppb)	115.8	114.4	117.2
Copper, Cu (wt.%)	0.385	0.374	0.396
Molybdenum, Mo (ppm)	79.5	77.7	81.4
Sulphur, S (wt.%)	0.921	0.897	0.944

Note: intervals may appear asymmetric due to rounding

From the INAA data set an estimated tolerance interval of ± 1.4 ppb at an analytical subsample weight of 50 gram was obtained (using the sampling constant relationship of Ingamells and Switzer, 1973) and is considered to reflect the actual homogeneity of the material under test. The meaning of this tolerance interval may be illustrated for gold (refer Table 8), where 99% of the time at least 95% of 50g-sized subsamples will have concentrations lying between 114.4 and 117.2 ppb. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

A different approach was used in estimating tolerance for copper, molybdenum and sulphur. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material under test and, in particular, to deficiencies in accuracy of each analytical method. In determining tolerance intervals for copper, molybdenum and sulphur that component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \bar{x}_i + \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^p n_i}$$

where

- x_{ij} is the j th raw result reported by laboratory i ;
- x'_{ij} is the j th transformed result reported by laboratory i ;
- n_i is the number of results reported by laboratory i ;
- p is the number of participating laboratories;
- \bar{x}_i is the raw mean for laboratory i .

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$\text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha) s_g''$$

$$\text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha) s_g''$$

where

n the number of results

$1-\alpha$ is the confidence level;

p is the proportion of results expected within tolerance limits;

k'_2 is the factor for two-sided tolerance limits (m, α unknown);

s'_g is the corrected grand standard deviation.

The corrected grand standard deviation, s'_g , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula

$$s'_g = \frac{\sum_{i=1}^p (s_i (1 - \frac{s_i}{s'_g}))}{\sum_{i=1}^p (1 - \frac{s_i}{s'_g})}$$

where

$1 - (\frac{s_i}{2s'_g})$ is the weighting factor for laboratory i ;

s'_g is the grand standard deviation computed from the transformed (i.e. means-adjusted) results.

according to the formula

$$s'_g = \left[\frac{\sum_{i=1}^p \sum_{j=1}^{n_i} (x'_{ij} - \bar{x}'_i)^2}{\sum_{i=1}^p n_i - 1} \right]^{1/2}$$

where \bar{x}'_i is the transformed mean for laboratory i

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. Individual outliers (shown in bold in Tables 4 to 6) were removed prior to the calculation of tolerance intervals and a weighting factor of zero was applied to those data sets where $s_i / 2s'_g > 1$ (i.e. where the weighting factor $1 - s_i / 2s'_g < 0$). It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error.

ANOVA Study

The sampling format for OREAS 151a was structured to enable nested ANOVA treatment of the round robin results. All laboratories were included in this treatment for gold, copper, molybdenum and sulphur. During the bagging stage, immediately following homogenization, twenty 800g samples were taken at regular intervals representative of the entire batch of OREAS 152a. Each laboratory received paired samples from three different, non-adjacent 800g samples. For example, the six samples that any one of the twenty-one participating labs could have received are:

- Sample 1 (from sampling interval 3)
- Sample 2 (from sampling interval 10)
- Sample 3 (from sampling interval 17)
- Sample 4 (from sampling interval 3)
- Sample 5 (from sampling interval 10)
- Sample 6 (from sampling interval 17)

The purpose of the ANOVA investigation was to compare the within-unit variance with that of the between-unit variance. This approach permitted an assessment of homogeneity across the entire batch of OREAS 152a. The test was performed using the following parameters:

- Significance Level $\alpha = P$ (type I error) = 0.05
- Null Hypothesis, H_0 : Between-unit variance is no greater than within-unit variance (reject H_0 if p-value < 0.05)
- Alternative Hypothesis, H_1 : Between-unit variance is greater than within-unit variance

P-values are a measure of probability whereby values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The dataset was filtered for both individual and laboratory outliers prior to the calculation of the p-value. This process derived p-values of 0.991 for gold, 0.84 for copper, 0.999 for molybdenum and 0.999 for sulphur and indicates no evidence that between-unit variance is greater than within-unit variance. Conclusion: do not reject H_0 .

Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that the analytes are distributed in a uniform manner throughout OREAS 152a and that the variance between two subsamples from the same unit is statistically indistinguishable to the variance from two subsamples taken from any two separate units.

Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. Sources of measurement error include inter-lab bias and analytical precision (repeatability). Two methods have been employed to calculate performance gates. The first method uses the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers. These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. The standard deviation is then calculated for each analyte from the pooled individual analyses generated from the certification program. Table 9 shows performance gates calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

A second method utilises a 5% window calculated directly from the certified value. Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow.

Table 9. Performance Gates for OREAS 152a

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Au (ppb)	116	5	106	126	101	131	4.31%	8.62%	12.93%	110	122
Cu (wt.%)	0.385	0.009	0.366	0.404	0.356	0.413	2.47%	4.94%	7.40%	0.365	0.404
Mo (ppm)	80	5	70	89	65	94	5.98%	11.96%	17.95%	76	84
S (wt.%)	0.921	0.046	0.828	1.013	0.782	1.059	5.03%	10.06%	15.08%	0.874	0.967

Note: intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

Accurassay Laboratories, Thunder Bay, ON, Canada
 Acme Analytical Laboratories, Vancouver, BC, Canada
 Activation Laboratories, Ancaster, ON, Canada
 Alaska Assay Laboratories, Fairbanks, AK, United States of America
 ALS Chemex, La Serena, Chile, South America
 ALS Chemex, Perth, WA, Australia
 ALS Chemex, Sparks, Nevada, USA
 ALS Chemex, Townsville, QLD, Australia
 ALS Chemex, Val-d'or, Quebec, Canada
 ALS Chemex, Vancouver, BC, Canada
 Amdel Laboratories, Adelaide, SA, Australia
 Genalysis Laboratory Services, Perth, WA, Australia
 Intertek Testing Services, Jakarta, Indonesia
 McPhar Laboratories, Legaspi Village, Makati, Philippines
 OMAC Laboratories, Loughrea, County Galway, Ireland
 SGS Australia, Perth, WA, Australia
 SGS Lakefield Research, Lakefield, ON, Canada
 SGS Mineral Services, Toronto, ON, Canada
 SGS Australia, Townsville, QLD, Australia
 Ultra Trace, Perth, WA, Australia
 Zarazma, Tehran, Iran

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Gold-copper-molybdenum-sulphur ore reference material OREAS 152a has been prepared and certified, and is supplied by:

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It is available in unit sizes of 60g foil packets and 1kg plastic jars.

INTENDED USE

OREAS 152a is a reference material intended for the following:

- i) for the monitoring of laboratory performance in the analysis of gold, copper, molybdenum and sulphur in geological samples;
- ii) for the calibration of instruments used in the determination of the concentration of gold, copper, molybdenum and sulphur;
- iii) for the verification of analytical methods for gold, copper, molybdenum and sulphur;
- iv) for the preparation of secondary reference materials of similar composition.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 152a has been prepared from sulphide-poor mineralised porphyry copper samples. The robust foil laminate packaging film is an effective barrier to oxygen and moisture and the sealed CRM is considered to have long-term stability (>5 years) under normal storage conditions.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The certified values for OREAS 152a refer to the concentration levels of gold, copper, molybdenum and sulphur after removal of hygroscopic moisture by drying in air to constant mass at 105° C. If the reference material is not dried by the user prior to analysis, the moisture content should be verified and the certified values corrected to the moisture-bearing basis.

LEGAL NOTICE

Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the use of this material and information.

CERTIFYING OFFICER

Craig Hamlyn (B.Sc. Hons.), Geology

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