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**CERTIFICATE OF ANALYSIS FOR**

**Au-Cu-Mo-S ORE REFERENCE MATERIAL**

**OREAS 151a**

**SUMMARY STATISTICS**

Constituent	Certified Value	1SD	95% Confidence Interval	
			Low	High
Gold, Au (ppb)	43	2	42	44
Copper, Cu (wt.%)	0.166	0.005	0.162	0.169
Molybdenum, Mo (ppm)	40	3	39	41
Sulphur, S (wt.%)	0.856	0.035	0.840	0.873

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 151a 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.

## COMMINUTION AND HOMOGENISATION PROCEDURES

OREAS 151a 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 151a

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 151a is given in Table 1. The constituents SiO<sub>2</sub> 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 (PDM<sup>3</sup>). 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 19 of the 21 labs lying within 9.1% relative of the certified value. For copper, interlaboratory agreement of the lab means is also very good with all labs but two within 5.3% relative of the certified value. For molybdenum, all labs but three fall within 9% relative of the certified value and for sulphur, all labs fall within 7.8% relative of the certified value.

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 151a; wt.% - weight percent; ppm - parts per million.

Constituent	wt. %	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO <sub>2</sub>	63.16	Ag	<0.5	Hf	0.3	Sc	22
TiO <sub>2</sub>	0.63	As	35	Ho	0.43	Sm	1.98
Al <sub>2</sub> O <sub>3</sub>	15.22	Ba	81	In	<0.02	Sn	1
Fe <sub>2</sub> O <sub>3</sub>	6.25	Be	0.4	La	4.3	Sr	120
MgO	3.44	Bi	<0.1	Li	6.3	Ta	<0.1
MnO	0.057	Cd	<0.5	Lu	0.14	Tb	0.34
CaO	2.78	Ce	10.0	Mo	41	Te	0.4
Na <sub>2</sub> O	2.35	Co	15	Nb	1	Th	0.9
K <sub>2</sub> O	1.31	Cs	0.5	Nd	7.13	U	0.4
P <sub>2</sub> O <sub>5</sub>	0.132	Cu	1725	Ni	12	W	2.8
LOI	3.97	Dy	2.15	Pb	12	Y	12.7
Total	101.4	Er	1.2	Pr	1.52	Yb	1.1
C	0.29	Eu	0.68	Rb	19.3	Zn	81
S	0.85	Ga	16.6	Re	0.1	Zr	9
		Gd	2.2	Sb	0.8		

Table 2. Analytical results for gold in OREAS 151a (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; PDM<sup>3</sup> - 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	39	45	41	47	48	42	43	45	41	42
2	39	46	42	43	51	42	44	42	41	41
3	40	47	42	45	50	42	41	41	42	38
4	40	42	41	43	48	44	44	42	39	39
5	38	44	42	44	49	44	44	43	42	41
6	39	46	41	43	50	44	45	42	41	37
Mean	39	45	42	44	<b>49</b>	43	44	43	41	40
Median	39	46	42	44	50	43	44	42	41	40
Std.Dev.	1	2	1	2	1	1	1	1	1	2
Rel.Std.Dev	1.92%	3.98%	1.32%	3.63%	2.45%	2.55%	3.17%	3.24%	2.67%	4.96%
PDM <sup>3</sup>	-9.11%	4.42%	-3.70%	2.49%	14.48%	-0.22%	0.94%	-1.38%	-4.86%	-7.95%

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
42	40	45	45	43	44	43	61	43	44	43
44	41	47	46	43	56	45	46	43	42	42
42	40	46	46	43	45	43	45	43	42	43
41	39	46	41	44	45	44	48	43	43	42
41	40	47	47	45	46	44	46	43	44	44
41	39	47	46	44	45	45	48	43	43	44
42	40	46	45	44	47	44	49	43	43	43
42	40	47	46	44	45	44	47	43	43	43
1	1	1	2	1	5	1	6	0	1	1
2.79%	1.89%	1.76%	4.73%	1.86%	9.68%	2.34%	12.24%	0.00%	2.08%	2.08%
-2.92%	-7.57%	7.52%	4.81%	1.21%	8.68%	2.47%	13.71%	-0.22%	-0.22%	-0.22%

Table 3. Analytical results for gold in OREAS 151a 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	43
2	42
3	41
4	42
5	41
6	40
7	39
8	42
9	43
10	43
11	44
12	42
13	42
14	46
15	43
16	43
17	41
18	42
19	39
20	42
Mean	42
Median	42
Std.Dev.	1.6
Rel.Std.Dev.	3.86%
PDM <sup>3</sup>	-2.54%

Table 4. Analytical results for copper in OREAS 151a (4A - four acid digest (HNO<sub>3</sub>-HClO<sub>4</sub>-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	0.172	0.162	0.166	0.162	0.172	0.175	0.164	0.174	0.170	0.166
2	0.174	<b>0.176</b>	0.166	0.156	0.170	0.169	0.166	0.178	0.164	0.160
3	0.172	0.163	0.166	0.158	0.171	0.170	0.165	0.174	0.166	0.171
4	0.169	0.160	0.164	0.157	0.171	<b>0.180</b>	0.166	0.174	0.168	0.165
5	0.175	0.159	0.162	0.158	0.173	0.167	0.169	0.174	0.166	0.163
6	<b>0.162</b>	0.164	0.163	0.157	0.170	0.171	0.163	0.174	0.170	0.166
Mean	0.171	0.164	0.164	0.158	0.171	0.172	0.165	0.175	0.167	0.165
Median	0.172	0.162	0.165	0.157	0.171	0.171	0.165	0.174	0.167	0.166
Std.Dev.	0.005	0.006	0.002	0.002	0.001	0.005	0.002	0.002	0.002	0.004
Rel.Std.Dev	2.76%	3.74%	1.12%	1.22%	0.68%	2.75%	1.29%	0.94%	1.45%	2.21%
PDM <sup>3</sup>	3.02%	-1.16%	-0.90%	-4.67%	3.32%	3.83%	-0.13%	5.34%	1.01%	-0.30%

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	0.174	NR	0.159	0.169	0.167	0.164	0.151	0.160	0.169	0.145
NR	0.170	NR	<b>0.169</b>	0.170	0.168	0.165	0.155	0.170	0.171	0.146
NR	0.167	NR	0.158	0.175	0.168	0.160	0.154	0.170	0.178	0.146
NR	0.167	NR	0.161	0.172	0.164	0.165	0.144	0.170	0.165	0.146
NR	0.167	NR	0.161	0.169	0.170	0.160	0.147	0.160	0.169	0.147
NR	0.176	NR	0.160	0.169	0.168	0.161	0.144	0.170	0.161	0.146
	0.170		0.161	0.170	0.168	0.162	<b>0.149</b>	0.167	0.169	<b>0.146</b>
	0.169		0.161	0.169	0.168	0.163	0.149	0.170	0.169	0.146
	0.004		0.004	0.002	0.002	0.002	0.005	0.005	0.006	0.001
	2.33%		2.44%	1.45%	1.18%	1.44%	3.24%	3.10%	3.37%	0.38%
	2.72%		-2.61%	2.87%	1.11%	-1.92%	-10.06%	0.61%	2.07%	-11.9%

Table 5. Analytical results for molybdenum in OREAS 151a (4A - four acid digest (HNO<sub>3</sub>-HClO<sub>4</sub>-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	37.0	37.0	38.0	40.4	37.0	38.0	40.0	42.9	42.0	41.0
2	37.0	41.0	38.0	39.9	37.0	36.0	41.0	41.6	40.0	40.0
3	38.0	39.0	38.0	40.5	38.0	36.0	40.0	42.7	40.0	42.0
4	36.0	38.0	38.0	40.1	37.0	39.0	40.0	42.7	40.0	40.0
5	38.0	36.0	36.0	40.6	35.0	37.0	41.0	43.0	42.0	42.0
6	37.0	39.0	37.0	41.2	35.0	38.0	39.0	42.9	42.0	43.0
Mean	37.2	38.3	37.5	40.5	36.5	37.3	40.2	42.6	41.0	41.3
Median	37.0	38.5	38.0	40.5	37.0	37.5	40.0	42.8	41.0	41.5
Std.Dev.	0.8	1.8	0.8	0.5	1.2	1.2	0.8	0.5	1.1	1.2
Rel.Std.Dev	2.03%	4.57%	2.23%	1.11%	3.36%	3.24%	1.87%	1.20%	2.67%	2.93%
PDM <sup>3</sup>	-7.10%	-4.18%	-6.26%	1.11%	-8.76%	-6.68%	0.40%	6.60%	2.49%	3.32%

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	41.0	NR	35.0	41.2	41.0	38.4	46.0	46.0	43.0	38.5
NR	41.0	NR	37.0	41.8	42.0	38.0	46.0	48.0	48.0	38.5
NR	42.0	NR	34.0	43.0	43.0	38.2	44.0	47.0	46.0	38.5
NR	41.0	NR	35.0	42.6	42.0	39.0	45.0	48.0	45.0	38.4
NR	40.0	NR	36.0	41.6	43.0	38.5	45.0	48.0	44.0	38.2
NR	41.0	NR	36.0	41.0	43.0	38.5	43.0	48.0	46.0	38.1
	41.0		35.5	41.9	42.3	38.4	44.8	<b>47.5</b>	45.3	38.4
	41.0		35.5	41.7	42.5	38.4	45.0	48.0	45.5	38.5
	0.6		1.0	0.8	0.8	0.3	1.2	0.8	1.8	0.2
	1.54%		2.95%	1.89%	1.93%	0.84%	2.61%	1.76%	3.86%	0.46%
	2.49%		-11.3%	4.66%	5.82%	-3.92%	12.1%	18.7%	13.3%	-4.10%

Table 6. Analytical results for sulphur in OREAS 151a (4A - four acid digest (HNO<sub>3</sub>-HClO<sub>4</sub>-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	0.810	NR	0.870	0.856	0.829	0.860	0.800	0.869	0.840	0.932
2	0.820	NR	0.870	0.803	0.832	0.830	0.800	0.897	0.850	0.880
3	0.820	NR	0.870	0.830	0.836	0.830	0.800	0.882	0.840	0.908
4	0.790	NR	0.860	0.818	0.843	<b>0.880</b>	0.800	0.884	0.845	0.877
5	0.810	NR	0.840	0.809	0.835	0.830	0.800	0.888	0.860	0.891
6	0.790	NR	0.850	0.832	0.833	0.840	0.800	0.890	0.850	0.903
Mean	0.807		0.860	0.825	0.835	0.845	0.800	0.885	0.848	0.899
Median	0.810		0.865	0.824	0.834	0.835	0.800	0.886	0.848	0.897
Std.Dev.	0.014		0.013	0.019	0.005	0.021	0.000	0.009	0.008	0.020
Rel.Std.Dev	1.69%		1.47%	2.31%	0.57%	2.45%	0.00%	1.07%	0.89%	2.28%
PDM <sup>3</sup>	-5.81%		0.42%	-3.71%	-2.54%	-1.33%	-6.58%	3.33%	-1.04%	4.92%

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	0.900	NR	0.820	0.835	0.909	NR	0.850	0.840	0.870	0.868
NR	0.870	NR	0.870	0.851	0.929	NR	0.860	0.900	0.890	0.869
NR	0.880	NR	0.820	0.859	0.918	NR	0.910	0.850	0.850	0.865
NR	0.870	NR	0.830	0.841	0.910	NR	0.910	0.830	0.880	0.871
NR	0.870	NR	0.840	0.844	0.940	NR	0.840	0.870	0.900	0.866
NR	0.890	NR	0.840	0.845	0.932	NR	0.830	0.920	0.860	0.870
	0.880		0.837	0.846	0.923		0.867	0.868	0.875	0.868
	0.875		0.835	0.844	0.924		0.855	0.860	0.875	0.868
	0.013		0.019	0.008	0.013		0.035	0.035	0.019	0.002
	1.44%		2.23%	0.98%	1.37%		4.04%	4.08%	2.14%	0.24%
	2.76%		-2.30%	-1.24%	7.78%		1.20%	1.40%	2.17%	1.38%

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 151a

### 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

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

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

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  $i$ ;

$\bar{\dot{x}}$  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

$$S = 1.483 \text{ median } / x_j - \text{median } (x_i) /$$

$j=1 \dots n$                        $i=1 \dots n$

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

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)	43	42	44
Copper, Cu (wt.%)	0.166	0.162	0.169
Molybdenum, Mo (ppm)	40	39	41
Sulphur, S (wt.%)	0.856	0.840	0.873

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'<sub>2</sub>* 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- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Gold, Au (ppb)	43	42	44
Copper, Cu (wt.%)	0.166	0.161	0.170
Molybdenum, Mo (ppm)	40	39	41
Sulphur, S (wt.%)	0.856	0.837	0.876

Note: intervals may appear asymmetric due to rounding

From the INAA data set an estimated tolerance interval of  $\pm 1$  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 42 and 44 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;

$k'_{\alpha}$  is the factor for two-sided tolerance limits ( $m, \alpha$  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 151a. 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 151a. 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 0.93 for gold, 0.98 for copper, 0.96 for molybdenum and 0.80 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 151a 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 151a

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)	43	2	38	48	36	50	5.39%	10.79%	16.18%	41	45
Cu (wt.%)	0.166	0.005	0.155	0.176	0.150	0.181	3.14%	6.28%	9.42%	0.157	0.174
Mo (ppm)	40	3	34	46	31	49	7.22%	14.4%	21.7%	38	42
S (wt.%)	0.856	0.035	0.786	0.927	0.751	0.962	4.09%	8.19%	12.28%	0.814	0.899

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  
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 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 151a has been prepared and certified, and is supplied by:

Ore Research & Exploration Pty Ltd  
 6-8 Gatwick Road  
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It is available in unit sizes of 60g foil packets and 1kg plastic jars.

## **INTENDED USE**

OREAS 151a 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 151a 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 151a 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

## **REFERENCES**

Ingamells, C. O. and Switzer, P. (1973), *Talanta* 20, 547-568.

ISO Guide 3207 (1975), *Statistical interpretation of data - Determination of a statistical tolerance interval.*

ISO Guide 35 (1985), *Certification of reference materials - General and statistical principals.*

ISO Guide 35 (2006), *Reference materials- General and statistical principals for certification.*