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CERTIFICATE OF ANALYSIS FOR

Au-Cu-Mo-S ORE REFERENCE MATERIAL

OREAS 52c

SUMMARY STATISTICS

Constituent	Certified Value	1SD	95% Confidence Interval	
			Low	High
Gold, Au (ppb)	346	17	338	353
Copper, Cu (wt.%)	0.344	0.009	0.339	0.348
Molybdenum, Mo (ppm)	267	15	260	275
Sulphur, S (wt.%)	0.471	0.015	0.463	0.479

Prepared by:
ORE Research & Exploration Pty Ltd
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REPORT 09-828-52c

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

Reference material OREAS 52c is a porphyry Au-Cu-Mo-S standard prepared from Au-Cu ore and waste rock samples from a porphyry copper-gold deposit located in central western New South Wales, Australia with the addition of minor Mo concentrate.

Mineralisation in the region is hosted by a sequence of late Ordovician to Early Silurian volcanics, intrusives and sediments that occur within the Bogan Gate Synclinorial Zone of the Lachlan Fold Belt. The western portion of this zone is dominated by volcanics and host to the Goonumbla porphyry copper-gold deposits. The Late Ordovician Goonumbla Volcanics host the Northparkes deposits and are interpreted to have erupted from shallow water to partly emergent volcanic centres. They exhibit a broad range in composition from shoshonite through to latite to trachyte.

Coeval sub-volcanic quartz monzonite porphyries (and attendant mineralisation) have intruded the volcanics. They are generally small, sub-vertical, pipe-like intrusives. Typically the mineralised porphyries contain plagioclase and quartz phenocrysts in a matrix of fine-grained potassium feldspar and quartz with minor biotite and hornblende.

Copper-gold mineralisation occurs as stockwork quartz veins and disseminations associated with potassic alteration. This alteration is intimately associated spatially and temporally with the small finger-like quartz monzonite porphyries that intrude the Goonumbla Volcanics. Sulphides are zoned laterally from the centres of mineralisation. The central portions are bornite-rich with minor chalcopyrite, zoning outward through equal concentrations of bornite and chalcopyrite, to an outermost chalcopyrite-rich zone. Pyrite increases outward at the expense of bornite.

The approximate major and trace element composition of OREAS 52c is given in Table 1. The constituents SiO_2 to Total are the means of duplicate XRF analyses determined using a borate fusion method, S and C are means of duplicate IR combustion furnace analyses, while the remaining constituents, Ag to Zr, are means of duplicate 4-acid digestion ICP-MS analyses.

COMMINUTION AND HOMOGENISATION PROCEDURES

OREAS 52c was prepared in the following manner:

a) *jaw crushing to minus 3mm;*
b) *drying to constant mass at 105°C;*
c) *multi-stage milling of ore and waste components to 100% minus 30 microns;*
d) *combining in appropriate proportions to achieve target grades;*
e) *homogenisation and bagging into 25kg sublots;*
f) *packaging into 60g units in laminated foil pouches and 1kg units in plastic jars*

ANALYSIS OF OREAS 52c

Twenty laboratories participated in the analytical program to characterise gold, eighteen for copper and molybdenum and sixteen for sulphur. They are listed in the section headed 'Participating Laboratories'. To maintain anonymity these laboratories have been randomly designated the letter codes A through T. Each laboratory received six samples comprising two scoop-split 100 gram subsamples from each of three 1kg test units A total of twenty 1kg test units were taken at regular intervals during the bagging stage. For each sample laboratories were requested to carry out one 25-40 gram fire assay determination for gold (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).

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 52c; wt.% - weight percent; ppm - parts per million.

Constituent	wt.%	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO_2	59.7	Ag	1.3	Gd	4.1	Sb	1.1
TiO_2	0.64	As	14	Hf	3.4	Sc	15
Al_2O_3	15.3	Ba	832	Ho	0.77	Sm	4.5
Fe_2O_3	7.4	Be	1.9	In	0.23	Sn	5
MnO	0.07	Bi	1.5	La	24.4	Sr	452
MgO	2.71	Cd	<0.5	Li	22.8	Ta	0.75
CaO	3.45	Ce	46.6	Lu	0.32	Tb	0.61
Na_2O	3.47	Co	18.5	Mo	292	Te	<0.2
K_2O	3.89	Cs	5	Nb	16.5	Th	12.7
P_2O_5	0.259	Cu	3690	Nd	21.2	U	3.6
LOI	2.43	Dy	3.5	Ni	29	W	4.0
Total	100.63	Er	2.1	Pb	24	Y	20.0
C	0.32	Eu	1.08	Pr	5.77	Yb	2.13
S	0.44	Ga	17.5	Rb	126	Zn	100
						Zr	108

For the determination of a statistical tolerance interval, a 10 gram scoop split was taken from each of the twenty test units and submitted to 'Lab A' for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 1.5 gram.

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[3]). The analytical methods employed by each laboratory are given in the table captions. For gold, interlaboratory agreement of the fire assay means is good with 16 of the 20 labs lying within 6% relative of the certified value. For copper, interlaboratory agreement of the lab means is very good with all labs but one within 4.5% relative of the certified value. For molybdenum all labs fall within 11.2% and for sulphur, excluding the 3 outlying labs, the remaining labs fall within 6.2% relative of the certified values.

Table 2. Analytical results for gold in OREAS 52c (FA - fire assay; AAS - flame atomic absorption spectrometry; SXAAS - solvent extraction 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[3] – 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	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
	FA*AAS	FA*AAS	FA*OES	FA*SXAAS	FA*AAS	FA*MS	FA*OES	FA*AAS	FA*OES	FA*OES
	30g	30g	30g	25g	30g	30g	30g	30g	40g	30g
1	349	360	335	351	347	410	331	348	348	364
2	350	362	338	346	346	339	327	335	345	363
3	348	**351**	342	358	347	411	334	347	345	370
4	351	370	336	356	347	383	334	339	346	363
5	352	366	338	349	348	386	329	350	345	377
6	346	365	334	351	345	360	326	341	**351**	372
Mean	349	362	337	352	347	**382**	330	343	347	368
Median	350	364	337	351	347	385	330	344	346	367
Std.Dev.	2	7	3	4	1	28	3	6	2	6
Rel.Std.Dev	0.62%	1.80%	0.85%	1.26%	0.30%	7.39%	1.04%	1.71%	0.70%	1.57%
PDM[3]	1.08%	4.84%	-2.44%	1.81%	0.31%	10.39%	-4.46%	-0.65%	0.31%	6.53%

Table 2. Continued

Replicate No.	Lab K	Lab L	Lab M	Lab N	Lab O	Lab P	Lab Q	Lab R	Lab S	Lab T
	FA*AAS	FA*OES	FA*AAS	FA*AAS	FA*OES	FA*MS	FA*OES	FA*AAS	FA*AAS	FA*AAS
	30g	30g	30g	30g	40g	30g	30g	30g	30g	30g
1	373	356	366	340	335	326	306	320	342	339
2	374	345	362	350	333	364	302	284	319	342
3	376	361	366	**290**	337	346	302	296	319	342
4	374	363	360	350	334	356	320	313	299	336
5	369	357	361	340	338	334	318	310	328	339
6	374	346	353	340	335	324	321	286	334	332
Mean	373	355	361	335	335	342	311	**302**	324	338
Median	374	357	362	340	335	340	312	303	324	339
Std.Dev.	2	8	5	23	2	16	9	15	15	4
Rel.Std.Dev	0.63%	2.13%	1.33%	6.74%	0.56%	4.79%	2.95%	4.97%	4.61%	1.13%
PDM[3]	8.03%	2.63%	4.55%	-3.07%	-2.97%	-1.14%	-9.88%	-12.76%	-6.39%	-2.10%

Table 3. Analytical results for gold in OREAS 52c by INAA (instrumental neutron activation analysis on 1.5 gram analytical subsample weights; other abbreviations as for Table 2).

Replicate No.	Lab T INAA 1.5g
1	334
2	354
3	304
4	327
5	327
6	321
7	340
8	327
9	339
10	350
11	325
12	355
13	357
14	344
15	343
16	353
17	339
18	340
19	343
20	351
Mean	339
Median	340
Std.Dev.	14
Rel.Std.Dev.	4.02%
PDM[3]	-2.01%

Table 4. Analytical results for copper in OREAS 52c (4A - four acid digest (HNO_3-$HClO_4$-HCl-HF); AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; other abbreviations as for Table 2; values in wt.%).

Replicate No.	Lab A 4A*OES	Lab B 4A*OES	Lab C 4A*OES/AAS	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	0.335	0.343	0.350	0.362	0.357	0.351	0.341	0.346	0.334	0.346
2	0.331	0.337	0.357	0.348	0.366	0.352	0.338	0.344	0.331	0.347
3	0.332	0.328	0.350	0.354	0.355	0.354	0.339	0.341	0.342	**0.325**
4	0.326	**0.323**	0.348	0.351	0.355	0.347	0.342	0.340	0.348	0.356
5	0.331	0.337	0.350	0.350	0.360	0.345	0.340	0.338	0.352	0.338
6	0.330	0.337	0.350	0.341	0.361	0.362	0.336	0.348	0.352	0.342
Mean	0.331	0.334	0.351	0.351	0.359	0.352	0.339	0.343	0.343	0.342
Median	0.331	0.337	0.350	0.350	0.359	0.352	0.340	0.343	0.345	0.344
Std.Dev.	0.003	0.007	0.003	0.007	0.004	0.006	0.002	0.004	0.009	0.010
Rel.Std.Dev	0.88%	2.23%	0.89%	1.97%	1.18%	1.70%	0.64%	1.09%	2.65%	3.04%
PDM[3]	-3.70%	-2.70%	2.12%	2.12%	4.50%	2.42%	-1.22%	-0.19%	-0.11%	-0.35%

Table 4. Continued

Replicate No.	Lab K -	Lab L 4A*OES	Lab M -	Lab N 4A*OES	Lab O 4A*OES	Lab P 4A*AAS	Lab Q 4A*OES	Lab R 4A*AAS	Lab S 4A*OES	Lab T 4A*OES
1	NR	0.328	NR	0.325	0.348	0.335	0.358	0.318	0.349	0.333
2	NR	0.343	NR	0.329	0.341	0.349	0.356	0.294	0.345	0.335
3	NR	0.336	NR	0.335	0.348	0.334	0.348	0.311	0.352	0.336
4	NR	0.335	NR	0.329	0.346	0.333	0.359	0.336	0.348	0.336
5	NR	0.340	NR	0.330	0.345	0.349	0.358	0.308	0.338	0.339
6	NR	0.343	NR	0.325	0.347	0.344	0.349	0.300	0.347	0.335
Mean		0.338		0.329	0.346	0.341	0.355	**0.311**	0.347	0.335
Median		0.338		0.329	0.346	0.340	0.357	0.309	0.348	0.335
Std.Dev.		0.006		0.004	0.003	0.008	0.005	0.015	0.005	0.002
Rel.Std.Dev		1.70%		1.13%	0.78%	2.22%	1.35%	4.80%	1.38%	0.59%
PDM^3		-1.76%		-4.28%	0.62%	-0.84%	3.21%	-9.50%	0.86%	-2.38%

Table 5. Analytical results for molybdenum in OREAS 52c (4A - four acid digest (HNO_3-$HClO_4$-HCl-HF); AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; other abbreviations as for Table 2; values in ppm).

Replicate No.	Lab A 4A*OES	Lab B 4A*OES	Lab C 4A*OES/AAS	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	283	255	250	282	272	260	260	283	295	265
2	273	260	260	274	271	260	260	281	295	271
3	283	286	250	280	270	260	260	280	295	258
4	274	274	250	281	272	260	260	280	300	268
5	279	274	250	278	273	250	260	**286**	300	264
6	278	286	250	270	271	270	260	279	300	**248**
Mean	278	273	252	278	272	260	260	282	298	262
Median	279	274	250	279	272	260	260	280	298	265
Std.Dev.	4	13	4	5	1	6	0	3	3	8
Rel.Std.Dev	1.54%	4.73%	1.62%	1.67%	0.39%	2.43%	0.00%	0.96%	0.92%	3.15%
PDM^3	4.06%	1.88%	-5.91%	3.75%	1.51%	-2.79%	-2.79%	5.25%	11.23%	-1.92%

Table 5. Continued

Replicate No.	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
1	NR	276	NR	250	259	268	268	241	258	287
2	NR	278	NR	248	253	271	269	226	261	291
3	NR	274	NR	253	245	262	269	241	258	288
4	NR	**282**	NR	251	259	261	270	255	257	288
5	NR	276	NR	250	251	271	267	236	257	285
6	NR	276	NR	**243**	260	264	**257**	230	**251**	287
Mean		277		249	255	266	267	238	257	288
Median		276		250	256	266	268	239	258	288
Std.Dev.		3		3	6	4	5	10	3	2
Rel.Std.Dev		1.00%		1.38%	2.33%	1.67%	1.77%	4.28%	1.28%	0.68%
PDM^3		3.56%		-6.84%	-4.85%	-0.49%	-0.26%	-10.96%	-3.91%	7.55%

Table 6. Analytical results for sulphur in OREAS 52c (4A - four acid digest (HNO_3-$HClO_4$-HCl-HF); AAS - flame atomic absorption spectrometry; 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/AAS	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	0.470	NR	**0.490**	0.464	0.471	0.470	0.450	0.478	0.485	0.586
2	0.470	NR	0.480	0.449	0.479	0.470	0.450	0.475	0.485	0.546
3	0.470	NR	0.470	0.468	0.463	0.470	0.450	0.472	0.485	0.498
4	0.470	NR	0.470	0.462	0.466	0.450	0.450	0.480	0.480	0.542
5	0.470	NR	0.460	0.457	0.470	0.450	0.440	0.483	**0.500**	0.535
6	0.470	NR	0.470	0.441	0.474	0.490	0.450	0.473	**0.500**	0.519
Mean	0.470		0.473	0.457	0.471	0.467	0.448	0.477	0.489	**0.538**
Median	0.470		0.470	0.460	0.471	0.470	0.450	0.477	0.485	0.539
Std.Dev.	0.000		0.010	0.010	0.006	0.015	0.004	0.004	0.009	0.030
Rel.Std.Dev	0.00%		2.18%	2.23%	1.21%	3.23%	0.91%	0.87%	1.76%	5.49%
PDM^3	-0.25%		0.46%	-3.02%	-0.14%	-0.96%	-4.85%	1.20%	3.82%	14.11%

Table 6. Continued

Replicate No.	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
1	NR	0.510	NR	0.480	0.505	0.520	NR	0.457	0.480	0.460
2	NR	0.510	NR	0.480	0.493	0.524	NR	0.452	0.480	0.470
3	NR	0.510	NR	0.490	0.499	0.510	NR	0.483	0.480	0.460
4	NR	0.520	NR	0.480	0.501	0.513	NR	**0.495**	0.480	0.450
5	NR	0.510	NR	0.480	0.496	0.519	NR	0.458	0.470	0.460
6	NR	0.510	NR	0.470	0.508	0.509	NR	0.468	0.480	0.460
Mean		**0.512**		0.480	0.500	**0.516**		0.469	0.478	0.460
Median		0.510		0.480	0.500	0.516		0.463	0.480	0.460
Std.Dev.		0.004		0.006	0.006	0.006		0.017	0.004	0.006
Rel.Std.Dev		0.80%		1.32%	1.17%	1.17%		3.60%	0.85%	1.37%
PDM^3		8.59%		1.87%	6.19%	9.48%		-0.50%	1.52%	-2.37%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 52c

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 T (excluding the INAA data) according to the formulae

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

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

where

x_{ij} is the jth 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):

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

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

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

$$S = 1.483 \underset{j=1.....n}{median} / x_j - \underset{i=1.....n}{median} (x_i) /$$

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

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 > 1.5%. 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 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)	346	338	353
Copper, Cu (wt.%)	0.344	0.339	0.348
Molybdenum, Mo (ppm)	267	260	275
Sulphur, S (wt.%)	0.471	0.463	0.479

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 1.5 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

$$\text{Lower limit is } \ddot{x} - k_2'(n, p, 1-\alpha)s$$
$$\text{Upper limit is } \ddot{x} + k_2'(n, p, 1-\alpha)s$$

where

n is the number of results reported by laboratory Q;
$1-\alpha$ 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

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

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)	346	336	356
Copper, Cu (wt.%)	0.344	0.336	0.351
Molybdenum, Mo (ppm)	267	264	271
Sulphur, S (wt.%)	0.471	0.461	0.481

Note: intervals may appear asymmetric due to rounding

From the INAA data set an estimated tolerance interval of ±10 ppb at an analytical subsample weight of 30 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 30g-sized subsamples will have concentrations lying between 336 and 356 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 (IS0 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

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

where

x_{ij} *is the jth raw result reported by laboratory i;*

x'_{ij} *is the jth transformed result reported by laboratory i;*

n_i *is the number of results reported by laboratory i;*

p *is the number of participating laboratories;*

$\overline{x}_i$ *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

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

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

where

n the number of results
1-α 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

$$s_g'' = \frac{\sum_{i=1}^{p} \left(s_i \left(1 - \frac{s_i}{s_g'} \right) \right)}{\sum_{i=1}^{p} \left(1 - \frac{s_i}{s_g'} \right)}$$

where

$$1 - \left(\frac{s_i}{2s_g'} \right) \text{ is the weighting factor for laboratory } i\,;$$

$$s_g' \text{ is the grand standard deviation computed from the transformed (i.e. means} - \text{adjusted) results.}$$

according to the formula

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

$$\text{where } \bar{x}_i' \text{ is the transformed mean for laboratorty } i$$

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. 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 $s_l / 2s_g' > 1$ (i.e. where the weighting factor 1- $s_l / 2s_g' < 0$). 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

All laboratories were included in the ANOVA study for gold, copper, molybdenum and sulphur. The sampling format for OREAS 52c was structured to enable nested ANOVA treatment of the round robin results. During the bagging stage, immediately following homogenization, twenty 1kg samples were taken at regular intervals representative of the entire batch of OREAS 52c. Each laboratory received paired samples from three different, non-adjacent 1kg samples. For example, the six samples that any one of the twenty 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 52c. The test was performed using the following parameters:

- Significance Level α = 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 1.00 for gold, 0.54 for copper, 0.21 for molybdenum and 0.78 for sulphur and indicates no evidence that between-unit variance is greater than within-unit variance. Conclusion: do not reject H_o.

Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that the metals (and sulphur) are distributed in a similar manner throughout OREAS 52c 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 52c

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)	346	17	312	379	296	396	4.81%	9.63%	14.44%	328	363
Cu (wt.%)	0.344	0.009	0.325	0.362	0.315	0.372	2.75%	5.50%	8.25%	0.326	0.361
Mo (ppm)	267	15	237	298	222	313	5.67%	11.33%	17.00%	254	281
S (wt.%)	0.471	0.015	0.442	0.500	0.427	0.515	3.11%	6.22%	9.33%	0.448	0.495

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

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Gold-copper-molybdenum-sulphur ore reference material OREAS 52c 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 52c 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 52c has been prepared from sulphide-poor mineralised quartz monzonite porphyry samples. The robust foil laminate film used to package it 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 52c 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.