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CERTIFICATE OF ANALYSIS FOR

GOLD-COPPER-SULPHUR CERTIFIED REFERENCE MATERIAL, OREAS 58P

SUMMARY STATISTICS

Constituent	Recommended value	95% Confidence Interval		Tolerance limits 1-a=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppb)	523	512	535	520	527
Copper, Cu (ppm)	5110	5024	5196	5025	5194
Sulphur, S (ppm)	6220	5744	6696	6045	6394

Note: intervals may appear asymmetric due to rounding

Prepared by:
Ore Research & Exploration Pty Ltd
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SOURCE MATERIAL

OREAS 58P is a gold-copper-sulphur certified reference material (CRM) prepared from samples sourced from a porphyry copper-gold deposit located in central western New South Wales, Australia.

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 Late Ordovician Goonumbla porphyry copper-gold deposits. These volcanics are interpreted to have erupted from shallow water to partly emergent volcanic centres and show 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.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

a) drying at 105°C to constant mass;
b) crushing and screening;
b) preliminary homogenisation;
c) milling to minus 20 microns;
d) final homogenisation;
e) packaging into 60g lots sealed in laminated foil pouches.

ANALYSIS OF OREAS 58P

Ten commercial laboratories participated in the analytical program to characterise gold, copper and sulphur concentrations in OREAS 58P. To evaluate the magnitude of batch to batch variation at individual laboratories, samples were submitted in four batches to five of the participating laboratories at weekly intervals. The results, together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM^3) are presented in Tables 1, 3 & 5. The parameter PDM^3 is a measure of laboratory

accuracy while the relative standard deviation is an effective measure of analytical precision where homogeneity of the test material has been confirmed. The analytical methods employed by each laboratory are given in the table captions. With the exception of the INAA data, a batch of five 70g samples were submitted at a time to each laboratory for analysis.

Gold (Table 1) was determined in replicate assays using a fire assay technique (40-50g charge with new pots) together with flame furnace AAS, ICPOES or ICPMS finish at nine laboratories, while one lab determined gold in twenty replicates via instrumental neutron activation analysis (INAA) using 0.5g analytical subsample weights. Each of the five samples submitted to each laboratory were taken at regular intervals during packaging of the standard in order to maximise their representation. The twenty INAA subsamples, on which much of the homogeneity evaluation is based, were also taken at regular intervals during packaging and are considered representative of the entire batch.

Copper and sulphur were determined by aqua regia digest with ICPOES or AAS finish (Tables 3 & 5). Batch means and standard deviations (95% confidence) of these means are given in Tables 2, 4 & 6, the latter parameter providing a measure of inter-batch bias at each laboratory. Comparisons of interlaboratory bias and precision, expressed as PDM[3] and coefficient of variation, respectively, for the participating laboratories are presented in Figures 1 – 6.

Table 1. Analytical results for gold in OREAS 58P (INAA - instrumental neutron activation analysis; FA*AAS - fire assay / atomic absorption spectrometry; FA*OES - fire assay / inductively coupled optical emission spectrometry; Std.Dev. and Rel.Std.Dev. are one sigma values; PDM[3] - percent deviation of lab mean from corrected mean of means; individual outliers in bold italics; outlying batches in bold; values in ppb).

Replicate No.	Lab 1 INAA (0.5g)	Lab 2 FA*AAS (50g)	Lab 3 FA*MS (40g)	Lab 4 FA*AAS (50g)	Lab 5 FA*AAS (50g)	Lab 6 FA*AAS (50g)	Lab 7 FA*AAS (50g)	Lab 8 FA*AAS (50g)	Lab 9 FA*OES (50g)	Lab 10 FA*AAS (50g)
1	510.2	520	527	**580**	510	497	532	***430***	540	505
2	521.1	510	524	**530**	510	499	533	498	584	506
3	516.9	530	523	**580**	520	494	526	509	566	520
4	518.1	520	532	**570**	530	499	533	503	539	519
5	527.8	520	**542**	**620**	510	497	500	470	543	520
6	518.9	520	555	560	540	499				
7	526.7	520	535	560	540	491				
8	530.3	530	550	530	530	495				
9	494.4	520	546	540	530	501				
10	519.4	520	**589**	570	540	501				
11	528.2	520	540	540	520	504				
12	513.2	540	535	560	520	510				
13	533.8	530	512	530	510	506				
14	529.3	530	510	540	530	513				
15	531.4	510	539	550	530	506				
16	514.3	550	530	500	520	**469**				
17	534.7	540	539	530	530	**486**				
18	505.2	530	533	520	520	**460**				
19	544.1	530	533	520	530	**490**				
20	519.6	560	**554**	510	510	**479**				
Mean	522	528	537	547	524	495	525	482	554	514
Median	520	525	535	540	525	498	532	498	543	519
Std.Dev.	11	13	17	28	10	13	14	33	20	8
Rel.Std.Dev.	2.19%	2.37%	3.18%	5.21%	2.00%	2.66%	2.70%	6.78%	3.59%	1.51%
PDM[3]	-0.28%	0.80%	2.69%	4.52%	0.13%	-5.45%	0.28%	-7.90%	5.94%	-1.78%

Table 2. Batch means for gold in OREAS 58P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in ppb).

Batch Number	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
1	520	527	**576**	516	497
2	522	547	552	536	497
3	526	527	544	522	508
4	542	534	516	522	**477**
Overall Mean	528	533	547	524	495
Std.Dev.	20	19	49	17	26
Rel.Std.Dev.	3.8%	3.5%	9.0%	3.2%	5.2%

Table 3. Analytical results for copper in OREAS 58P (AR – aqua regia digest; OES -inductively coupled plasma optical emission spectrometry; AAS - atomic absorption spectrometry; abbreviations as in Table 1; values in ppm).

Replicate No.	Lab 2 AR*OES	Lab 3 AR*OES	Lab 4 AR*OES	Lab 5 AR*OES	Lab 6 AR*AAS	Lab 7 AR*AAS	Lab 8 AR*OES	Lab 9 AR*AAS	Lab 10 AR*OES
1	4827	5380	5350	5090	5150	4980	5060	5180	5107
2	5064	5340	5350	5020	5160	5000	5250	5160	5061
3	5162	5260	5300	4880	5170	5000	5120	5140	5043
4	5089	5230	5350	5070	***5320***	4980	5110	5200	5079
5	5179	5300	5350	4960	5230	5000	5170	5110	5063
6	4841	5080	5300	5170	**5780**				
7	4898	5040	5400	5120	**5720**				
8	4928	5030	5250	***4840***	**5820**				
9	4863	5100	5300	5070	**5710**				
10	***5106***	5010	5250	5090	**5680**				
11	5905	5060	5050	5340	5120				
12	4730	5030	5050	5410	4950				
13	4686	5070	5050	4970	4990				
14	4635	5190	4950	5460	5030				
15	5447	***5270***	5050	5160	5290				
16	**4271**	5160	5350	**5270**	**4580**				
17	**4154**	4910	5300	**5390**	**4920**				
18	**4360**	5060	5300	**5400**	**4830**				
19	**4273**	4980	5300	**5530**	**4670**				
20	**4467**	5050	5250	**5410**	**4720**				
Mean	4844	5128	5243	5183	5192	4992	5142	5158	5071
Median	4852	5075	5300	5140	5155	5000	5120	5160	5063
Std.Dev.	428	130	133	205	381	11	72	35	24
Rel.Std.Dev.	8.83%	2.54%	2.54%	3.96%	7.34%	0.22%	1.40%	0.68%	0.47%
PDM[3]	-5.20%	0.35%	2.60%	1.42%	1.61%	0.63%	-2.30%	-0.77%	0.94%

Table 4. Batch means for copper in OREAS 58P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in ppm).

Batch Number	Lab 5	Lab 4	Lab 2	Lab 3	Lab 6
1	5004	5340	5064	5302	5206
2	5058	5300	4927	5052	**5742**
3	5268	5030	5081	5124	5076
4	**5400**	5300	**4305**	5032	**4744**
Overall Mean	5183	5243	4844	5128	5192
Std.Dev.	369	286	732	246	830
Rel.Std.Dev.	7.1%	5.5%	15.1%	4.8%	16.0%

Table 5. Analytical results for sulphur in OREAS 58P (AR – aqua regia digest; ICPOES -inductively coupled plasma optical emission spectrometry; AAS - atomic absorption spectrometry; abbreviations as in Table 1; values in parts per million).

Replicate No.	Lab 2 AR*OES	Lab 3 AR*OES	Lab 4 AR*OES	Lab 5 AR*OES	Lab 6 AR*AAS	Lab 7 LECO	Lab 8 AR*OES	Lab 9 AR*OES	Lab 10 AR*OES
1	6483	6900	7100	6300	**8250**	5900	5000	***7000***	5100
2	6747	6600	7000	6100	**8310**	5900	5200	7200	5000
3	6854	6900	7100	5900	**8340**	5900	5400	7400	5000
4	6784	6600	7500	6200	**8530**	5900	5300	7500	5100
5	6880	6800	7100	6000	**8200**	5800	5100	7500	5000
6	5611	6100	7100	6400	7830				
7	5826	6300	7300	6200	7690				
8	5706	6100	7300	***5800***	7640				
9	5673	6400	7300	6300	7730				
10	5959	6200	7100	6200	7710				
11	7199	6220	7000	6200	6970				
12	5576	6260	6900	6300	6640				
13	5451	6320	7000	5900	6630				
14	5324	6340	6900	6500	6810				
15	6739	6220	7300	6100	7190				
16	5649	6300	6400	6200	6130				
17	5432	6200	6300	6400	6640				
18	5781	5060	6400	***7500***	6450				
19	5660	4980	6600	6700	6220				
20	5978	5050	6600	6400	6270				
Mean	6066	6193	6965	6280	7309	5880	5200	7320	5040
Median	5804	6280	7050	6200	7415	5900	5200	7400	5000
Std.Dev.	595	557	339	359	802	45	158	217	55
Rel.Std.Dev.	9.81%	8.99%	4.87%	5.72%	10.98%	0.76%	3.04%	2.96%	1.09%
PDM[3]	-2.48%	-0.44%	11.98%	0.97%	17.51%	-16.40%	-5.47%	-18.97%	17.69%

Table 6. Batch means for sulphur in OREAS 58P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in weight percent).

Batch Number	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
1	6100	7160	6750	6760	**8326**
2	6180	7220	5755	6220	7720
3	6200	7020	6058	6272	6848
4	6640	6460	5700	5518	6342
Overall Mean	6280	6965	6066	6193	7309
Std.Dev.	488	694	965	1022	1770
Rel.Std.Dev.	7.8%	10.0%	15.9%	16.5%	24.2%

STATISTICAL EVALUATION OF OREAS 58P

Recommended Value and Confidence Limits

Each batch of results is treated as a separate data set in testing for outliers and in determining the consensus mean. A weighting is applied to each batch mean to ensure equal representation for all laboratories irrespective of the number of batches analysed. The certified value is the mean of means of accepted replicate values of accepted laboratory batches computed 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 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 are 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).

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

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

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 are 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 is 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

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

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

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.

Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in bold and have been omitted in the determination of recommended values.

Statement of Homogeneity

The standard deviation of each laboratory data set includes error due to both the imprecision of the analytical method employed and to possible inhomogeneity of the material analysed. 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 analysed and, in particular, to deficiencies in accuracy of each analytical method. In determining tolerance intervals for elements other than gold 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.

Table 7. Recommended values and 95% confidence intervals for OREAS 58P.

Constituent	Recommended value	95% Confidence Interval	
		Low	High
Gold, Au (ppb)	523	512	535
Copper, Cu (ppm)	5110	5024	5196
Sulphur, S (ppm)	6220	5744	6696

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 *is the number of results*;

$1-\alpha$ *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, α *unknown*);

s_g'' *is the corrected grand standard deviation*.

The meaning of these tolerance limits may be illustrated for copper, where 99% of the time at least 95% of subsamples will have concentrations lying between 5006 and 5214 ppm. 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).

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

$1 - \left(\frac{s_i}{2s_g'}\right)$ *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

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

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. 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.
For gold a more simplified procedure was used in the determination of homogeneity. This entailed using the high precision INAA data alone, obtained on an analytical subsample weight of 0.5g (compared to 40-50g for the fire assay method). By employing a sufficiently reduced subsample weight in a series of determinations by the same method, analytical error becomes negligible in comparison to subsampling error. The corresponding standard deviation at a 50g subsample weight can then be determined from the observed standard deviation of the 0.5g data using the known relationship between the two parameters (Kleeman, 1967). The homogeneity of gold was then determined from tables of factors for two-sided tolerance limits for normal distributions. The high level of repeatability indicated by the low coefficients of variation in Table 1 (particularly the 0.5 g INAA data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 58P.

For elements other than gold, outliers (shown in bold in Tables 1, 3 & 5) 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 $1- s_l / 2s_g' < 0$).

Table 8. Recommended values and tolerance limits for OREAS 58P.

Constituent	Recommended value	Tolerance limits 1-a=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	523	520	527
Copper, Cu (ppm)	5110	5025	5194
Sulphur, S (ppm)	6220	6045	6394

PARTICIPATING LABORATORIES

Acme Analytical Laboratories, Vancouver, BC, Canada
Actlabs Pacific Pty Ltd, Kalgoorlie, WA, Australia
ALS Chemex, Garbutt, QLD, Australia
ALS Chemex, Vancouver, BC, Canada
Amdel Laboratories, Wangara, WA, Australia
Becquerel Laboratories, Lucas Heights, NSW, Australia
Genalysis, Maddington, WA, Australia
Intertek Testing Laboratories, Jakarta, Indonesia
OMAC Laboratories, Loughrea, Ireland
Standard & Reference Laboratories, Malaga, WA, Australia
Ultra Trace, Canning Vale, WA, Australia

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