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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD-SULPHUR
REFERENCE MATERIAL OREAS 55P

SUMMARY STATISTICS

Recommended Values, 95% Confidence and Tolerance Intervals

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppm)	0.927	0.904	0.950	0.920	0.934
Copper, Cu (ppm)	1397	1367	1427	1373	1421
Sulphur, S (%)	2.89	2.78	3.00	2.85	2.93

Prepared by:
Ore Research & Exploration Pty Ltd
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SOURCE MATERIAL

Reference material OREAS 55P is a copper-gold-sulphur standard prepared from low grade material from a porphyry Cu-Au deposit, central New South Wales, Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

a) drying for 24 hours at 105^0 C;
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 55P

Ten Australian commercial laboratories participated in the analytical program for gold, copper and sulphur. To evaluate the effects of batch to batch variation, samples were submitted to the laboratories in four batches, dispatched at weekly intervals. All 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 and 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 Lab A each laboratory received a batch of five 60g samples for analysis. Gold (Table 1) was determined at these laboratories using a fire assay technique (40-50g charge with new pots) with AAS or ICPOES finish. Lab A determined gold in twenty replicates via instrumental neutron activation analysis (INAA) using reduced analytical subsample weights of 0.5g. Copper and sulphur were determined by aqua regia digest with ICPOES finish (Table 3). Lab F determined sulphur by Leco high temperature combustion.
Batch means for each laboratory and standard deviations (95% confidence) of these means are given in Tables 2, 4 and 6, the latter parameter providing a measure of inter-batch bias at each laboratory.
The five subsamples comprising each batch were taken at widely spaced 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 similarly considered representative of the entire batch.

Table 1. Analytical results for gold in standard OREAS 55P (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 left-justified bold; outlying batches in bold; values in parts per million).

Replicate No.	Lab A INAA (0.5g)	Lab B FA-AAS (50g)	Lab C FA-AAS (50g)	Lab D FA-AAS (50g)	Lab E FA-OES (40g)	Lab F FA-AAS (50g)	Lab G FA-AAS (50g)	Lab H FA-AAS (50g)	Lab I FA-AAS (50g)	Lab J FA-AAS (40g)
1	0.8651	0.92	0.94	0.925	0.929	**0.83**	0.96	0.83	0.88	0.965
2	0.8661	0.96	0.93	0.915	0.911	**0.81**	0.93	0.86	0.92	0.970
3	**0.8231**	0.96	0.96	**0.995**	0.908	**0.80**	0.92	0.87	0.91	0.970
4	0.8953	0.95	0.97	0.925	0.921	**0.80**	0.96	0.87	0.89	0.965
5	0.8802	0.95	0.96	**0.865**	0.931	**0.83**	0.95	0.85	0.90	0.965
6	0.8800	0.87	0.91	0.965	0.950	0.97	0.92	**0.99**	0.91	0.995
7	0.8488	0.89	0.89	0.975	0.956	0.96	0.98	0.94	0.90	0.970
8	0.8848	0.90	0.88	0.950	0.943	0.94	0.94	0.94	0.88	0.980
9	0.8466	0.89	0.88	0.965	0.940	0.94	0.96	0.95	0.87	0.965
10	0.8462	**0.83**	0.90	0.950	0.969	0.95	0.99	0.98	0.91	0.960
11	0.9021	0.89	**0.81**	0.916	0.930	0.94	0.95	1.00	0.89	0.945
12	0.8546	0.90	**0.70**	0.947	0.945	0.95	0.99	0.98	0.90	**0.910**
13	0.8807	0.90	0.90	0.929	0.930	0.93	0.98	1.00	0.90	0.940
14	0.8586	0.90	0.93	0.923	0.930	0.96	0.98	0.98	0.91	0.940
15	0.8777	0.90	0.91	0.939	0.912	0.95	0.97	1.00	0.92	0.945
16	0.8930	0.94	**0.99**	0.87	0.968	0.90	0.93	0.94	0.89	0.975
17	0.8712	0.96	0.96	0.94	0.936	0.91	0.95	0.96	0.93	0.995
18	0.8762	0.94	0.95	0.93	0.940	0.89	0.93	**0.91**	0.89	1.010
19	0.8691	0.93	0.94	0.89	0.879	0.90	0.95	**0.99**	0.95	0.985
20	0.8712	0.96	0.95	0.89	0.893	0.91	0.93	0.95	0.92	0.965
Mean	0.870	0.917	0.913	0.930	0.931	0.904	0.954	0.940	0.904	0.966
Median	0.871	0.910	0.930	0.930	0.931	0.920	0.950	0.950	0.900	0.965
Std.Dev.	0.019	0.036	0.065	0.034	0.023	0.058	0.023	0.055	0.019	0.023
Rel.Std.Dev.	2.21%	3.89%	7.09%	3.62%	2.45%	6.39%	2.39%	5.91%	2.10%	2.34%
PDM[3]	-6.24%	-1.13%	-1.56%	0.30%	0.39%	-2.58%	2.81%	1.30%	-2.58%	4.13%

Table 2. Batch means for gold in standard OREAS 55P (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 B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	0.948	0.952	0.925	0.920	**0.814**	0.944	0.856	0.900	0.967
2	0.876	0.892	0.961	0.952	0.952	0.958	0.960	0.894	0.974
3	0.898	0.850	0.931	0.929	0.946	0.974	0.992	0.904	0.936
4	0.946	0.958	0.904	0.923	0.902	0.938	0.950	0.916	0.986
Overall Mean	0.917	0.913	0.930	0.931	0.904	0.954	0.940	0.904	0.966
Std.Dev.	0.072	0.103	0.047	0.028	0.127	0.032	0.117	0.019	0.043
Rel.Std.Dev.	7.8%	11.3%	5.1%	3.1%	14.1%	3.4%	12.4%	2.1%	4.4%

Table 3. Analytical results for copper in standard OREAS 55P (AR-OES – aqua regia digest / inductively coupled plasma optical emission spectrometry; abbreviations as in Table 1; values in ppm).

Replicate No.	Lab B AR-OES	Lab C AR-OES	Lab D AR-OES	Lab E AR-OES	Lab F AR-OES	Lab G AR-OES	Lab H AR-OES	Lab I AR-OES	Lab J AR-OES
1	1447	**1500**	1374	1410	1340	1420	1400	1370	1450
2	1444	**1500**	1380	1420	1350	1390	1410	1335	1450
3	1421	**1520**	1381	1450	1360	1390	1380	1330	1600
4	1416	**1520**	1379	1390	1360	1400	1350	1370	**1700**
5	1439	**1510**	**1361**	1470	1350	1390	1430	1330	1500
6	1367	**1420**	1347	**1500**	1340	1440	1420	**1550**	1400
7	1419	1380	1385	**1500**	1390	1440	1490	1370	1450
8	1395	1380	1346	**1530**	1350	1410	1460	1390	1450
9	1371	1390	1368	**1520**	1380	1400	1540	1365	1450
10	1386	1360	1323	**1500**	1400	1410	1450	1335	1400
11	1414	1380	1419	**1560**	**1320**	1410	**1300**	**1265**	1380
12	1428	1380	1405	**1520**	1360	1400	1340	1305	**1440**
13	1432	1390	1414	**1530**	1370	1440	1340	1360	1390
14	1413	1410	1400	**1530**	1360	1350	1380	1365	1390
15	1432	1390	1384	**1510**	1380	1380	1330	1345	1400
16	1338	1410	1359	**1560**	1380	1420	1490	1500	1300
17	1371	1410	1410	**1540**	1350	1410	1430	1410	1340
18	1389	1400	1449	**1550**	1360	1390	1440	1445	1290
19	1404	1400	1437	**1560**	**1270**	1390	1480	1360	1360
20	1387	1380	1404	**1530**	1380	1410	1420	1375	1290
Mean	1406	1422	1386	1504	1358	1405	1414	1374	1422
Median	1414	1400	1383	1520	1360	1405	1420	1365	1400
Std.Dev.	29	54	32	51	28	22	62	64	99
Rel.Std.Dev.	2.09%	3.8%	2.28%	3.38%	2.07%	1.56%	4.42%	4.68%	6.9%
PDM[3]	0.61%	1.74%	-0.79%	7.65%	-2.84%	0.53%	1.21%	-1.67%	1.74%

Table 4. Batch means for copper in standard OREAS 55P (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 B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	1433	**1510**	1378	1428	1352	1398	1394	1347	1500
2	1388	1378	1354	**1510**	1372	1420	1472	1365	1430
3	1424	1390	1404	**1530**	1368	1396	1337	1344	1390
4	1378	1400	1412	**1548**	1368	1404	1452	1418	1316
Overall Mean	1406	1419	1387	1504	1365	1405	1414	1368	1409
Std.Dev.	54	122	53	106	18	22	122	69	154
Rel.Std.Dev.	3.9%	8.6%	3.8%	7.0%	1.3%	1.5%	8.6%	5.0%	10.9%

Table 5. Analytical results for sulphur in standard OREAS 55P (AR-OES – aqua regia digest / inductively coupled plasma optical emission spectrometry; Leco – high temperature combustion; abbreviations as in Table 1; values in percent; NR – not reported).

Replicate No.	Lab B AR-OES	Lab C AR-OES	Lab D AR-OES	Lab E AR-OES	Lab F Leco	Lab G AR-OES	Lab H AR-OES	Lab I AR-OES	Lab J AR-OES
1	2.902	**3.03**	**2.454**	2.88	**2.75**	2.82	3.00	2.80	2.89
2	2.870	2.92	**2.411**	2.83	2.83	2.75	3.05	2.70	2.99
3	2.815	2.98	**2.447**	2.87	2.80	2.77	2.95	2.65	2.92
4	2.795	2.92	**2.458**	**2.56**	2.79	2.75	2.95	2.75	2.87
5	2.876	2.93	**2.469**	2.86	2.81	2.72	3.05	2.70	2.92
6	**2.702**	2.97	**2.419**	3.07	2.87	2.83	3.10	2.85	3.05
7	2.825	2.95	**2.585**	2.95	2.91	2.82	3.15	2.80	3.14
8	2.788	2.91	**2.493**	3.14	2.89	2.74	3.15	2.80	3.15
9	2.777	2.99	**2.465**	2.93	2.88	2.74	**3.30**	2.80	3.14
10	2.798	2.92	**2.391**	2.86	2.89	2.74	3.15	2.70	3.05
11	2.845	**3.29**	**2.757**	**3.19**	2.84	2.75	2.75	NR	3.18
12	2.838	**3.33**	2.632	3.11	2.96	2.75	2.85	NR	3.15
13	2.858	**3.40**	2.656	3.13	2.95	2.84	2.80	NR	3.09
14	2.812	**3.41**	2.651	3.08	2.96	2.70	2.85	NR	3.09
15	2.835	**3.37**	2.627	3.12	2.84	2.72	2.80	NR	3.11
16	**2.681**	2.91	2.706	3.27	3.02	2.80	2.85	**3.17**	2.97
17	2.750	2.90	2.715	3.18	2.94	2.79	2.95	3.01	3.03
18	2.777	2.88	2.822	3.20	3.02	2.77	3.00	3.05	2.92
19	2.769	2.88	2.843	3.20	2.95	2.78	3.05	2.98	3.04
20	2.776	2.86	2.783	**3.12**	3.09	2.79	2.95	2.95	2.9
Mean	2.804	3.038	2.589	3.028	2.900	2.769	2.985	2.847	3.030
Median	2.805	2.940	2.606	3.095	2.890	2.760	2.975	2.800	3.045
Std.Dev.	0.056	0.196	0.149	0.177	0.087	0.039	0.143	0.152	0.101
Rel.Std.Dev.	1.99%	6.47%	5.74%	5.85%	3.01%	1.42%	4.80%	5.33%	3.33%
PDM[3]	-3.05%	5.01%	-10.5%	4.66%	0.24%	-4.29%	3.19%	-1.56%	4.75%

Table 6. Batch means for sulphur in standard OREAS 55P (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 percent).

Batch Number	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	2.85	2.94	**2.45**	2.86	2.81	2.76	3.00	2.72	2.92
2	2.80	2.95	**2.47**	2.99	2.89	2.77	3.14	2.79	3.11
3	2.84	**3.36**	2.64	3.11	2.91	2.75	2.81	0.00	3.12
4	2.77	2.89	2.77	3.21	3.00	2.79	2.96	3.00	2.97
Overall Mean	2.814	3.033	2.583	3.043	2.902	2.769	2.977	2.127	3.030
Std.Dev.	0.076	0.440	0.307	0.304	0.162	0.029	0.269	2.846	0.202
Rel.Std.Dev.	2.7%	14.5%	11.9%	10.0%	5.6%	1.1%	9.1%	133.8%	6.7%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 55P

Recommended Value and Confidence Limits

The certified value is the mean of means of accepted replicate values of accepted participating laboratories 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 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).

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

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

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 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

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

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

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.

In certain instances statistician's prerogative has been employed in discriminating outliers. Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in left-justified bold and bold, respectively, and have been omitted in the determination of recommended 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 recommended value, i.e. the narrower the confidence interval the greater the certainty in the recommended value.

Table 7. Recommended values and 95% confidence intervals for OREAS 55P.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	0.927	0.904	0.950
Copper, Cu (ppm)	1397	1367	1427
Sulphur, S (%)	2.89	2.78	3.00

Note - intervals may appear asymmetric due to rounding

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 dataset. 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 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*;
$\bar{x}_i$ *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

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

where

n *is the number of results*;
$1-\alpha$ *is the confidence level*;
ρ *is the proportion of results expected within the tolerance limits*;
k_2' *is the factor for two-sided tolerance limits* (m, σ *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 1373 and 1421 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 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. 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 a reduced analytical subsample weight of 0.5g. By employing a sufficiently reduced subsample weight in a series of determinations by the same method, analytical error should become negligible in comparison to subsampling error. The standard deviation at a 50g subsample weight has been 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, in particular the 0.5g INAA data, is consistent with the narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 55P.
No outliers were removed from the INAA results prior to the calculation of tolerance intervals for gold, however for the other elements outliers were removed prior to the calculation of s_g' 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 55P.

Constituent	Recommended value	Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	0.927	0.920	0.934
Copper, Cu (ppm)	1397	1373	1421
Sulphur, S (%)	2.89	2.85	2.93

Note - intervals may appear asymmetric due to rounding

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, analytical precision (repeatability) and inter-batch bias (reproducibility).
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 deviations are then calculated for each lab's results and then each SD is tested for outlying status using z-score discrimination (rejected if $|z_i| > 2.5$). The 1SD used to calculate performance gates is the mean of the remaining (accepted) lab standard deviations. 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.
Both methods should be used with caution 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 Gate for OREAS 55P

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 (ppm)	0.927	0.025	0.878	0.976	0.853	1.001	2.65%	5.30%	7.94%	0.881	0.973
Cu (ppm)	1397	35	1327	1467	1292	1502	2.50%	4.99%	7.49%	1327	1467
S (wt.%)	2.89	0.09	2.71	3.07	2.62	3.16	3.13%	6.26%	9.38%	2.75	3.03

Note - intervals may appear asymmetric due to rounding

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