CERTIFICATE OF ANALYSIS FOR

# Au-Ag-Cu ORE REFERENCE MATERIAL

# OREAS 66a

SUMMARY STATISTICS

| Constituent (ppm) | Certified Value | 1SD | 95% Confidence Interval | |
|---|---|---|---|---|
| | | | Low | High |
| Gold, Au | 1.237 | 0.054 | 1.211 | 1.263 |
| Silver, Ag | 18.9 | 1.2 | 18.4 | 19.4 |
| Copper, Cu | 121 | 7 | 117 | 124 |

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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 66a was prepared from gold-silver ore from Martabe, North Sumatra, Indonesia blended with fresh alkali olivine basalt from the Newer Volcanics Province, Victoria, Australia. Martabe is an epithermal high sulphidation gold deposit hosted by a sequence of tertiary volcanic and sedimentary rocks near a fault splay of the Great Sumatran Fault complex. The approximate major and trace element composition of OREAS 66a 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 (except for Pb where ICP-OES was used).
Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty 0.5 gram sample portions and by a nested ANOVA program on the fire assay data. The tolerance interval for Au is determined from the INAA data while the certified value and confidence interval are based on the fire assay results of a round robin program incorporating a total of 134 analyses at 19 laboratories.

# COMMINUTION AND HOMOGENISATION PROCEDURES

OREAS 66a 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 the ore component to 100% minus 30 microns;*
   d)   *multi-stage milling of the basalt component to 100% minus 75 microns;*
   e)   *blending and bagging into 25kg sublots;*
   f)   *packaging into 60g units in laminated foil pouches and 1kg units in plastic jars*

# ANALYSIS OF OREAS 66a

Nineteen laboratories participated in the analytical program to characterise gold and seventeen for silver and copper. They are listed in the section headed 'Participating Laboratories'. To maintain anonymity these laboratories have been randomly designated the letter codes A through T. With the exception of Laboratory T each laboratory received two scoop-split 100 gram subsamples from each of three 1kg test units (6 samples total) taken at regular intervals during the bagging stage (20 test units in total). For each sample labs were requested to carry out one 30-50 gram fire assay determination for gold (with new pots) and one 4-acid digest determination for silver and copper using their preferred finish. 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 66a; wt.% - weight percent; ppm - parts per million.

| Constituent | wt.% | Constituent | ppm | Constituent | ppm | Constituent | ppm |
|---|---|---|---|---|---|---|---|
| $SiO_2$ | 73.6 | Ag | 18.8 | Gd | 2.9 | Sb | 64 |
| $TiO_2$ | 0.83 | As | 282 | Hf | 1.9 | Sc | 9 |
| $Al_2O_3$ | 8.49 | Ba | 808.5 | Ho | 0.4 | Sm | 3.35 |
| $Fe_2O_3$ | 6.745 | Be | 0.6 | In | 0.48 | Sn | 3.50 |
| MnO | 0.04 | Bi | 10.2 | La | 17.6 | Sr | 488 |
| MgO | 1.82 | Cd | 1 | Li | 14.8 | Ta | 0.4 |
| CaO | 2.32 | Ce | 33.6 | Lu | 0.21 | Tb | 0.40 |
| $Na_2O$ | 0.85 | Co | 18 | Mo | 5.5 | Te | 9.6 |
| $K_2O$ | 0.355 | Cs | 0.5 | Nb | 6.8 | Th | 4.1 |
| $P_2O_5$ | 0.1635 | Cu | 119 | Nd | 16.0 | U | 1.4 |
| LOI | 4.16 | Dy | 2.15 | Ni | 59 | W | 10.5 |
| Total | 102.25 | Er | 1.23 | Pb | 260 | Y | 10.3 |
| C | 0.03 | Eu | 0.98 | Pr | 4 | Yb | 1.3 |
| S | 1.075 | Ga | 17.4 | Rb | 8 | Zn | 91 |
| | | | | | | Zr | 75 |

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 T' for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 0.5 gram.

Individual assay results for gold via fire assay and INAA are presented in Tables 2 and 3, respectively, and results for silver and copper are presented in Table 4 and 5, 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 all labs lying within 8.6% relative of the corrected mean of means (certified value) of 1.237 ppm Au. For silver, interlaboratory agreement of the lab means is good with all labs lying within 9.4% relative of the certified value of 18.9 ppm Ag. For copper interlaboratory agreement of the lab means is good with 14 labs within 7.8% relative of the certified value of 121 ppm Cu and 3 labs at +12.09%, +12.78% and -16.66% relative of the certified value.

Table 2. Analytical results for gold in OREAS 66a (FA - fire assay; AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; GRAV - gravimetry; 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 ppm).

| Replicate No. | Lab A FA*AAS 30g | Lab B FA*AAS 30g | Lab C FA*AAS 30g | Lab D FA*AAS 25g | Lab E FA*AAS 50g | Lab F FA*AAS 30g | Lab G FA*OES 30g | Lab H FA*AAS 50g | Lab I FA*OES 40g |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 1.18 | 1.18 | 1.25 | 1.27 | 1.25 | 1.28 | 1.19 | 1.17 | 1.25 |
| 2 | 1.21 | 1.19 | 1.31 | 1.23 | 1.24 | 1.31 | 1.19 | 1.20 | 1.24 |
| 3 | 1.17 | 1.20 | 1.31 | 1.27 | 1.27 | 1.31 | 1.24 | 1.18 | 1.28 |
| 4 | 1.16 | 1.19 | 1.31 | 1.27 | 1.24 | 1.32 | 1.23 | 1.23 | 1.26 |
| 5 | 1.16 | 1.18 | 1.32 | 1.26 | 1.24 | 1.29 | 1.19 | 1.20 | 1.25 |
| 6 | 1.20 | 1.18 | 1.36 | 1.24 | 1.26 | 1.29 | 1.19 | 1.18 | 1.28 |
| Mean | 1.18 | 1.19 | 1.31 | 1.26 | 1.25 | 1.30 | 1.21 | 1.19 | 1.26 |
| Median | 1.18 | 1.19 | 1.31 | 1.27 | 1.25 | 1.30 | 1.19 | 1.19 | 1.26 |
| Std.Dev. | 0.02 | 0.01 | 0.04 | 0.02 | 0.01 | 0.02 | 0.02 | 0.02 | 0.02 |
| Rel.Std.Dev. | 1.78% | 0.85% | 2.83% | 1.39% | 1.01% | 1.19% | 1.95% | 1.81% | 1.33% |
| PDM[3] | -4.62% | -4.04% | 5.76% | 1.58% | 1.04% | 5.08% | -2.60% | -3.54% | 1.85% |

Table 2. Continued

| Replicate No. | Lab J FA*AAS 30g | Lab K FA*AAS 30g | Lab L FA*AAS 30g | Lab M FA*AAS 30g | Lab N FA*AAS 30g | Lab O FA*AAS 30g | Lab P FA*AAS 40g | Lab Q FA*AAS 30g | Lab R FA*OES 25g | Lab S FA*AAS 30g |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1.19 | 1.27 | 1.25 | 1.26 | 1.21 | 1.38 | 1.26 | 1.29 | 1.19 | 1.11 |
| 2 | 1.17 | 1.27 | 1.27 | 1.26 | 1.22 | 1.20 | 1.27 | 1.29 | 1.21 | 1.21 |
| 3 | 1.13 | 1.24 | 1.28 | 1.25 | 1.22 | 1.30 | 1.24 | 1.27 | 1.19 | 1.18 |
| 4 | 1.12 | 1.26 | 1.26 | 1.28 | 1.24 | 1.24 | 1.23 | 1.25 | 1.22 | 1.09 |
| 5 | 1.13 | 1.24 | 1.24 | 1.27 | 1.22 | 1.34 | 1.22 | 1.19 | 1.23 | 1.11 |
| 6 | 1.13 | 1.23 | 1.25 | 1.27 | 1.21 | 1.24 | 1.23 | 1.21 | 1.21 | 1.09 |
| Mean | 1.15 | 1.25 | 1.25 | 1.26 | 1.22 | 1.28 | 1.24 | 1.25 | 1.21 | 1.13 |
| Median | 1.13 | 1.25 | 1.25 | 1.26 | 1.22 | 1.27 | 1.24 | 1.26 | 1.21 | 1.11 |
| Std.Dev. | 0.03 | 0.02 | 0.01 | 0.01 | 0.01 | 0.07 | 0.02 | 0.04 | 0.02 | 0.05 |
| Rel.Std.Dev | 2.45% | 1.46% | 1.19% | 0.89% | 0.90% | 5.35% | 1.56% | 3.36% | 1.33% | 4.50% |
| PDM[3] | -7.45% | 0.97% | 1.38% | 2.05% | -1.38% | 3.74% | 0.37% | 1.04% | -2.33% | -8.60% |

Table 3. Analytical results for gold in OREAS 66a by INAA (instrumental neutron activation analysis on 0.5 gram analytical subsample weights; other abbreviations as for Table 2).

| Replicate No. | Lab T INAA 0.5g |
|---|---|
| 1 | 1.36 |
| 2 | 1.32 |
| 3 | 1.32 |
| 4 | 1.36 |
| 5 | 1.39 |
| 6 | 1.36 |
| 7 | 1.38 |
| 8 | 1.38 |
| 9 | 1.35 |
| 10 | 1.35 |
| 11 | 1.36 |
| 12 | 1.37 |
| 13 | 1.39 |
| 14 | 1.37 |
| 15 | 1.35 |
| 16 | 1.35 |
| 17 | 1.35 |
| 18 | 1.34 |
| 19 | 1.34 |
| 20 | 1.35 |
| Mean | 1.36 |
| Median | 1.36 |
| Std.Dev. | 0.02 |
| Rel.Std.Dev. | 1.44% |
| PDM[3] | 9.69% |

Table 4. Analytical results for silver in OREAS 66a (4A - four acid digest ($HNO_3$-$HClO_4$-$HCl$-$HF$); 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 ppm).

| Replicate No. | Lab A 4A*OES | Lab B 4A*OES | Lab C 4A*MS | Lab D 4A*AAS | Lab E 4A*OES | Lab F 4A*OES | Lab G 4A*OES | Lab H 4A*OES | Lab I 4A*MS |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 20.5 | 19.0 | 19.2 | 17.0 | 20.5 | 17.4 | 19.0 | 18.8 | 18.5 |
| 2 | 20.2 | 21.0 | **19.8** | 18.0 | 20.0 | 18.1 | 20.0 | 19.0 | 19.0 |
| 3 | **19.5** | **22.0** | 19.4 | 18.0 | 20.3 | 17.2 | 18.0 | 19.0 | 18.0 |
| 4 | 20.3 | 20.0 | 19.3 | 18.0 | 20.6 | 17.3 | 17.0 | 18.9 | 18.0 |
| 5 | 20.6 | 19.0 | 19.1 | 18.0 | 20.4 | 17.5 | 19.0 | 18.8 | 18.0 |
| 6 | 20.4 | 19.0 | 19.1 | 18.0 | 20.0 | 17.8 | 18.0 | 19.2 | 19.0 |
| Mean | 20.3 | 20.0 | 19.3 | 17.8 | 20.3 | 17.6 | 18.5 | 19.0 | 18.4 |
| Median | 20.4 | 19.5 | 19.2 | 18.0 | 20.4 | 17.5 | 18.5 | 19.0 | 18.3 |
| Std.Dev. | 0.4 | 1.3 | 0.3 | 0.4 | 0.3 | 0.3 | 1.0 | 0.2 | 0.5 |
| Rel.Std.Dev. | 1.94% | 6.32% | 1.40% | 2.29% | 1.25% | 1.93% | 5.67% | 0.80% | 2.67% |
| PDM[3] | 7.20% | 5.87% | 2.12% | -5.60% | 7.46% | -7.10% | -2.07% | 0.32% | -2.51% |

Table 4. Continued

| Replicate No. | Lab J 4A*OES | Lab L 4A*AAS | Lab N 4A*AAS | Lab O 4A*OES | Lab P 4A*OES | Lab Q 4A*OES | Lab R 4A*OES | Lab S 4A*AAS |
|---|---|---|---|---|---|---|---|---|
| 1 | 18.1 | 18.2 | 16.3 | 18.7 | 21.0 | 18.1 | 20.6 | 19.6 |
| 2 | **22.9** | 17.9 | 16.7 | 18.6 | 21.0 | 18.4 | 20.2 | 20.5 |
| 3 | 18.6 | 19.1 | 16.4 | 18.8 | 21.0 | 18.8 | 20.3 | 19.5 |
| 4 | 17.6 | 18.7 | 16.8 | 19.3 | 20.0 | 19.4 | 20.6 | 15.6 |
| 5 | 17.6 | 18.7 | 18.5 | 18.3 | 21.0 | 19.2 | 20.3 | 16.7 |
| 6 | 18.4 | 18.5 | 19.0 | 18.9 | 20.0 | 18.9 | 20.0 | 16.0 |
| Mean | 18.9 | 18.5 | 17.3 | 18.8 | 20.7 | 18.8 | 20.3 | 18.0 |
| Median | 18.3 | 18.6 | 16.8 | 18.8 | 21.0 | 18.9 | 20.3 | 18.1 |
| Std.Dev. | 2.0 | 0.4 | 1.2 | 0.3 | 0.5 | 0.5 | 0.2 | 2.1 |
| Rel.Std.Dev. | 10.69% | 2.28% | 6.72% | 1.77% | 2.50% | 2.58% | 1.15% | 11.79% |
| PDM[3] | -0.13% | -1.98% | -8.51% | -0.66% | 9.40% | -0.48% | 7.64% | -4.86% |

Table 5. Analytical results for copper in OREAS 66a (4A - four acid digest ($HNO_3$-$HClO_4$-$HCl$-$HF$); 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 ppm).

| Replicate No. | Lab A 4A*OES | Lab B 4A*OES | Lab C 4A*MS | Lab D 4A*AAS | Lab E 4A*OES | Lab F 4A*OES | Lab G 4A*OES | Lab H 4A*OES | Lab I 4A*OES |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 139 | 113 | 115 | **111** | 124 | 118 | 110 | 121 | 122 |
| 2 | 135 | 110 | 115 | 114 | 125 | 116 | 110 | 122 | 126 |
| 3 | **129** | 111 | 116 | 115 | 126 | 116 | 110 | 123 | 126 |
| 4 | 136 | 110 | 114 | 116 | 128 | 115 | 120 | 122 | 124 |
| 5 | 137 | 112 | 113 | 115 | 126 | 115 | 110 | 122 | 124 |
| 6 | 135 | 111 | 114 | 116 | 124 | 119 | 110 | 123 | 120 |
| Mean | 135 | 111 | 114 | 115 | 126 | 117 | 112 | 122 | 124 |
| Median | 136 | 111 | 114 | 115 | 126 | 116 | 110 | 122 | 124 |
| Std.Dev. | 3 | 1 | 1 | 2 | 2 | 2 | 4 | 1 | 2 |
| Rel.Std.Dev. | 2.49% | 1.05% | 1.02% | 1.63% | 1.21% | 1.41% | 3.66% | 0.62% | 1.89% |
| PDM[3] | 12.09% | -7.82% | -5.33% | -5.05% | 4.07% | -3.39% | -7.40% | 1.31% | 2.55% |

Table 5. Continued

| Replicate No. | Lab J 4A*OES | Lab L 4A*AAS | Lab N 4A*AAS | Lab O 4A*OES | Lab P 4A*OES | Lab Q 4A*OES | Lab R 4A*OES | Lab S 4A*AAS |
|---|---|---|---|---|---|---|---|---|
| 1 | 130 | NR | 121 | NR | 120 | 122 | 121 | 128 |
| 2 | **157** | NR | 120 | NR | 120 | 121 | 120 | 127 |
| 3 | 133 | NR | 123 | NR | 120 | 123 | 122 | 127 |
| 4 | 127 | NR | 125 | NR | 120 | 122 | 118 | 127 |
| 5 | 135 | NR | 123 | NR | 120 | 121 | 119 | 127 |
| 6 | 134 | NR | 122 | NR | 120 | 121 | 120 | 126 |
| Mean | 136 | | 122 | | 120 | 122 | 120 | 127 |
| Median | 134 | | 123 | | 120 | 122 | 120 | 127 |
| Std.Dev. | 11 | | 2 | | 0 | 1 | 1 | 1 |
| Rel.Std.Dev. | 7.86% | | 1.43% | | 0.00% | 0.67% | 1.18% | 0.50% |
| PDM[3] | 12.78% | | 1.44% | | -0.49% | 0.89% | -0.49% | 5.31% |

# STATISTICAL EVALUATION OF ANALYTICAL DATA
# FOR OREAS 66a

**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 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;*
      *$\overline{x}_i$ 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):

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

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

*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

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

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

*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 > 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 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 5) 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 6).

Table 6. Certified Value and 95% Confidence Interval

| Constituent | Certified | 95% Confidence Interval | |
|---|---|---|---|
| | Value | Low | High |
| Gold, Au (ppm) | 1.237 | 1.211 | 1.263 |
| Silver, Ag (ppm) | 18.9 | 18.4 | 19.4 |
| Copper, Cu (ppm) | 121 | 117 | 124 |

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 0.5 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

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

*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, $\sigma$ unknown);*

and *s* is computed according to the formula

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

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

Table 7.  Certified Value and Tolerance Interval.

| Constituent | Certified Value | Tolerance Interval 1-α=0.99, ρ=0.95 | |
|---|---|---|---|
| | | Low | High |
| Gold, Au (ppm) | 1.237 | 1.230 | 1.244 |
| Silver, Ag (ppm) | 18.9 | 18.2 | 19.6 |
| Copper, Cu (ppm) | 121 | 118 | 124 |

Note: intervals may appear asymmetric due to rounding

From the INAA data set an estimated tolerance interval of ±0.007 ppm 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 7), where 99% of the time at least 95% of 30g-sized subsamples will have concentrations lying between 1.230 and 1.244 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).

A different approach was used in estimating tolerance for silver and copper. 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 silver and copper 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

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

*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

<div align="center">

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

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

</div>

*where*

        *n the number of results*
        *$1-\alpha$ is the confidence level;*
        *p is the proportion of results expected within tolerance limits;*
        *$k'_2$ is the factor for two-sided tolerance limits (m, $\alpha$ 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

$$s''_g = \frac{\sum\limits_{i=1}^{p}\left(s_i\left(1 - \frac{s_i}{s'_g}\right)\right)}{\sum\limits_{i=1}^{p}\left(1 - \frac{s_i}{s'_g}\right)}$$

*where*

<div align="center">

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

</div>

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

according to the formula

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

<div align="center">

*where $\overline{x}'_i$ is the transformed mean for laboratorty i*

</div>

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 and 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 $1 - s_l / 2s_g' < 0$). 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 66a was structured to enable nested ANOVA treatment of the round robin results. During the bagging stage, immediately following final homogenization, twenty 1kg samples were taken at regular intervals representative of the entire batch of OREAS 66a. Each laboratory received paired samples from three different, non-adjacent 1kg samples. For example, the six samples that any one of the nineteen participating labs could have received are:

- Sample 1 (from sampling interval 4)
- Sample 2 (from sampling interval 11)
- Sample 3 (from sampling interval 17)
- Sample 4 (from sampling interval 4)
- Sample 5 (from sampling interval 11)
- 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 66a. 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 1.00 for gold, 0.99 for silver and 0.95 for copper 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 are distributed in a similar manner throughout OREAS 66a 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 8 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 8. Performance Gates for OREAS 66a

| 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) | 1.237 | 0.054 | 1.129 | 1.345 | 1.075 | 1.399 | 4.37% | 8.74% | 13.11% | 1.175 | 1.299 |
| Ag (ppm) | 18.9 | 1.2 | 16.4 | 21.4 | 15.2 | 22.6 | 6.55% | 13.10% | 19.65% | 17.9 | 19.8 |
| Cu (ppm) | 121 | 7 | 107 | 134 | 100 | 141 | 5.68% | 11.36% | 17.05% | 115 | 127 |

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
OMAC Laboratories, Loughrea, County Galway, Ireland
SGS Lakefield Research, Lakefield, ON, Canada
SGS, Townsville, QLD, Australia
SGS Australia, Perth, WA, Australia
Ultra Trace, Perth, WA, Australia
Zarazma Mineral Studies Company, Tehran, Iran

# PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Gold-silver-copper ore reference material OREAS 66a has been prepared and certified, and is supplied by:

> Ore Research & Exploration Pty Ltd
> 6-8 Gatwick Road
> Bayswater North, VIC  3153
> AUSTRALIA
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It is available in unit sizes of 60g foil packets and 1kg plastic jars.


# INTENDED USE

OREAS 66a is a reference material intended for the following:

i)      for the monitoring of laboratory performance in the analysis of gold, silver and copper in geological samples;

ii)     for the calibration of instruments used in the determination of the concentration of gold, silver and copper;

iii)    for the verification of analytical methods for gold, silver and copper;

iv)    for the preparation of secondary  reference materials of similar composition.


# STABILITY AND STORAGE INSTRUCTIONS

OREAS 66a has been prepared from epithermal high sulphidation Au-Ag-Cu ore blended with barren basalt. The robust foil laminate film used in the 60g unit packaging is an effective barrier to oxygen and moisture and the sealed CRM is considered to have long-term stability under normal storage conditions.


# INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The certified values for OREAS 66a refer to the concentration level of gold, silver and copper in their packaged state. Therefore it should not be dried prior to weighing and analysis.


# 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.