## Base Metal Sulphide Ore (Gamsberg Zinc Mine, Sth Africa) CERTIFIED REFERENCE MATERIAL OREAS 37

Table 1. Certified Values, SDs, 95\% Confidence and Tolerance Limits for OREAS 37.

| Constituent | Certified Value | 1SD | 95\% Confidence Limits |  | 95\% Tolerance Limits |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Low | High | Low | High |
| Acid digest |  |  |  |  |  |  |
| Silver, Ag (ppm) | 5.19 | 0.63 | 4.77 | 5.62 | 4.94 | 5.44 |
| Arsenic, As (ppm) | 449 | 28 | 438 | 461 | 439 | 459 |
| Copper, Cu (ppm) | 125 | 5 | 120 | 131 | 122 | 129 |
| Iron, Fe (wt.\%) | 23.76 | 0.56 | 23.31 | 24.22 | 23.45 | 24.08 |
| Manganese, Mn (wt.\%) | 0.719 | 0.017 | 0.687 | 0.751 | 0.707 | 0.731 |
| Lead, Pb (wt.\%) | 0.615 | 0.017 | 0.599 | 0.631 | 0.601 | 0.629 |
| Thallium, Tl (ppm) | 63 | 5 | 32 | 93 | 60 | 65 |
| Zinc, Zn (wt.\%) | 6.26 | 0.15 | 6.17 | 6.35 | 6.16 | 6.36 |
| Peroxide Fusion |  |  |  |  |  |  |
| Silver, Ag (ppm) | 5.0 | IND | 4.2 | 5.8 | IND | IND |
| Arsenic, As (ppm) | 460 | 42 | 424 | 496 | 435 | 485 |
| Copper, Cu (ppm) | 129 | 6 | 123 | 135 | IND | IND |
| Iron, Fe (wt.\%) | 23.53 | 0.54 | 22.88 | 24.19 | 23.03 | 24.04 |
| Manganese, Mn (wt.\%) | 0.769 | 0.027 | 0.751 | 0.786 | 0.750 | 0.787 |
| Lead, Pb (wt.\%) | 0.597 | 0.02 | 0.589 | 0.605 | 0.583 | 0.611 |
| Thallium, Tl (ppm) | 156 | IND | 139 | 174 | 152 | 161 |
| Zinc, Zn (wt. \%) | 6.30 | 0.18 | 6.09 | 6.51 | 6.17 | 6.43 |
| Leco |  |  |  |  |  |  |
| Sulphur, S (wt.\%) | 26.79 | 0.71 | 26.14 | 27.45 | 26.50 | 27.08 |

SI unit equivalents: ppm, parts per million $\equiv \mathrm{mg} / \mathrm{kg} \equiv \mu \mathrm{g} / \mathrm{g} \equiv 0.0001 \mathrm{wt} . \% \equiv 1000 \mathrm{ppb}$, parts per billion.
Note: intervals may appear asymmetric due to rounding

Table 2. Indicative Values for OREAS 37.

| Constituent | Unit | Value | Constituent | Unit | Value | Constituent | Unit | Value |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Oxidising Fusion XRF |  |  |  |  |  |  |  |  |
| $\mathrm{Al}_{2} \mathrm{O}_{3}$ | wt.\% | 5.56 | $\mathrm{Fe}_{2} \mathrm{O}_{3}$ | wt.\% | 33.65 | $\mathrm{SnO}_{2}$ | ppm | 12.7 |
| As | ppm | 500 | $\mathrm{K}_{2} \mathrm{O}$ | wt.\% | 1.42 | $\mathrm{SO}_{3}$ | wt.\% | 66.03 |
| BaO | ppm | 234 | MgO | wt.\% | 0.692 | SrO | ppm | 23.7 |
| CaO | wt.\% | 0.377 | MnO | wt.\% | 0.968 | $\mathrm{TiO}_{2}$ | wt.\% | 0.355 |
| Cl | ppm | 25.0 | NiO | ppm | 51 | $\mathrm{V}_{2} \mathrm{O}_{5}$ | ppm | 71 |
| CoO | ppm | 70 | $\mathrm{P}_{2} \mathrm{O}_{5}$ | wt.\% | 0.180 | ZnO | ppm | 80595 |
| $\mathrm{Cr}_{2} \mathrm{O}_{3}$ | ppm | 51 | PbO | ppm | 6673 | $\mathrm{ZrO}_{2}$ | ppm | 149 |
| CuO | ppm | 169 | $\mathrm{SiO}_{2}$ | wt.\% | 31.86 |  |  |  |
| Thermogravimetry |  |  |  |  |  |  |  |  |
| LOI ${ }^{1000}$ | wt.\% | 17.91 |  |  |  |  |  |  |
| Laser Ablation ICP-MS |  |  |  |  |  |  |  |  |
| Ag | ppm | 4.85 | Hf | ppb | 3265 | Sn | ppm | 1.20 |
| As | ppm | 465 | Ho | ppb | 565 | Sr | ppm | 5.95 |
| Ba | ppm | 210 | In | ppm | 0.25 | Ta | ppb | 465 |
| Be | ppm | 3.10 | La | ppm | 18.2 | Tb | ppb | 525 |

[^0]Table 2. Indicative Values for OREAS 37 continued.

| Constituent | Unit | Value | Constituent | Unit | Value | Constituent | Unit | Value |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Laser Ablation ICP-MS |  |  |  |  |  |  |  |  |
| Bi | ppm | 1.01 | Lu | ppb | 255 | Te | ppb | 200 |
| Cd | ppm | 116 | Mo | ppm | 8.10 | Th | ppm | 5.64 |
| Ce | ppm | 35.1 | Nb | ppm | 5.76 | TI | ppm | 140 |
| Co | ppm | 56 | Nd | ppm | 16.5 | Tm | ppb | 265 |
| Cr | ppm | 40.5 | Ni | ppm | 42.0 | U | ppm | 2.06 |
| Cs | ppm | 6.48 | Pb | $\mathrm{wt} . \%$ | 0.608 | V | ppm | 47.2 |
| Cu | ppm | 129 | Pr | ppm | 4.43 | W | ppm | 4.33 |
| Dy | ppm | 3.12 | Rb | ppm | 86 | Y | ppm | 17.3 |
| Er | ppm | 1.66 | Re | ppb | $<10$ | Yb | ppb | 1715 |
| Eu | ppb | 1225 | Sb | ppm | 16.5 | Zn | ppm | 61400 |
| Ga | ppm | 7.25 | Sc | ppm | 5.15 | Zr | ppm | 111 |
| Gd | ppm | 3.25 | Se | ppm | $<5$ |  |  |  |
| Ge | ppb | 3725 | Sm | ppm | 3.43 |  |  |  |

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

## INTRODUCTION

OREAS reference materials are intended to provide a low cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

## SOURCE MATERIAL

OREAS 37 is a medium grade zinc ore matrix-matched certified reference material (MMCRM) prepared by Ore Research and Exploration. It is one of 3 MMCRM's sourced from the Gamsberg Zn deposit located in the Northern Cape Province of South Africa, approximately 20 km west of the Black Mountain mine. Gamsberg is a stratiform base metal Broken Hill Style (BHS) deposit located in the mid-proterozoic Bushmanland Province of the Namaqualand Metamorphic Complex (NMC) of South Africa. The NMC is a highly deformed and metamorphosed supracrustal succession of dominantly pelitic schists and quartzites, deposited on a regionally extensive $\pm 2000 \mathrm{Ma}$ basement (Rozendal \& Stalder, 2001). The stratiform ores have a close spatial and genetic association with metamorphosed chemical sediments including manganiferous iron formations, quartz-garnet rocks (coticules), Ca-Mn marbles and barite (Rozendal \& Stalder, 2001).

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- Drying at $65^{\circ} \mathrm{C}$ to constant mass;
- Crushing and screening;
- Preliminary homogenisation;
- Milling to minus 30 microns;
- Final homogenisation;
- Packaging into 10 g units under nitrogen and sealed in laminated foil pouches.


## ANALYTICAL PROGRAM

Ten commercial laboratories participated in the analytical program to characterise Ag , As, $\mathrm{Cu}, \mathrm{Fe}, \mathrm{Mn}, \mathrm{Pb}, \mathrm{S}, \mathrm{Tl}$ and Zn in OREAS 37. The laboratories were requested to analyse all elements by three acid ore grade digest (preferred) or strong aqua regia digestion together with sodium peroxide fusion methods. To evaluate and compensate for the effects of batch-to-batch variation at individual laboratories, samples were submitted to six of the laboratories in three batches of four 10 g samples at weekly intervals. The remaining four laboratories completed one round only. Their data has been included in all statistical analysis excluding performance gates, where only the six labs incorporating batch to batch variation have been used (for further discussion see 'Performance Gates').

The approximate major and trace element composition of OREAS 37 is provided in Table 2. The non-certified values contained in this table are the means of duplicate assays from one laboratory.

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 ( $\mathrm{PDM}^{3}$ ) are presented in the Appendix (Tables A2 to A 18 ). The parameter $\mathrm{PDM}^{3}$ 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 and described in Table A1 of the Appendix.

All ten commercial labs participated in the acid digest work and employed flame AAS, ICP-OES or ICP-MS instrumental finishes. Up to eight of these labs (depending on the analyte) also carried out sodium peroxide fusion ICP-OES/MS analysis to evaluate the presence of an acid insoluble component. Sulphur was determined via Leco by nine labs with the remaining lab employing aqua regia digest with an ICP-OES finish. Each of the four samples submitted to each laboratory were taken at regular intervals during packaging of the standard in order to maximise their representation. Comparisons of interlaboratory bias and precision are graphically presented in scatter plots for acid digest Pb and Zn (Figures 1 and 2) together with $\pm 3$ SD (magenta) and $\pm 5 \%$ (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified by red and violet, respectively.

ORE RESEARCH \& EXPIORATION

## STATISTICAL EVALUATION

## Certified Value and Confidence Interval

Each batch of results is treated as a separate data set in testing for outliers. The certified value is determined from the mean of lab means after filtering of individual and batch outliers. It is computed according to the formulae

$$
\bar{x}_{i}=\frac{1}{n_{i}} \sum_{j=1}^{n_{i}} x_{i j} \quad \ddot{x}=\frac{1}{p} \sum_{i=1}^{p} \bar{x}_{i}
$$

where,

$$
\begin{aligned}
& x_{i j} \text { is the } j \text { th result reported by laboratory } i \text {; } \\
& p \text { is the number of participating laboratories; } \\
& n_{i} \text { is the number of results reported by laboratory } i \text {; } \\
& \bar{x}_{i} \text { is the mean for laboratory } i \text {; } \\
& \ddot{x} \text { is the mean of means. }
\end{aligned}
$$

The confidence intervals are obtained by calculation of the variance ( $\hat{V}$ ) of the consensus value


$$
\begin{gathered}
\hat{V}(\ddot{x})=\frac{1}{p(p-1)} \sum_{i=1}^{p}\left(\bar{x}_{i}-\ddot{x}\right)^{2} \\
\text { Confidence Interval }=\ddot{x} \pm t_{1-x / 2}(p-1)(\hat{V}(\ddot{x}))^{1 / 2}
\end{gathered}
$$

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 interval. The test for rejection of individual outliers from each laboratory data set is based on $z$ scores (rejected if $\left|z_{i}\right|>2.5$ ) computed from the robust estimators of location and scale, $T$ and $S$, respectively, according to the formulae

$$
\begin{gathered}
S=1.483 \underset{j=1 \ldots . . n}{\text { median }} / x_{j}-\underset{i=1 \ldots \ldots n}{\operatorname{median}}\left(x_{i}\right) / \\
z_{i}=\frac{x_{i}-T}{S}
\end{gathered}
$$

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.

Following identification of z-score outliers a 3SD filter is applied, with those values lying outside this window relegated to outlying status also. In certain instances statistician's prerogative has been employed in discriminating outliers. The test for outlying laboratory batches is also based on z-score discrimination (rejected if $\left|z_{i}\right|>2.5$ ) and these batches are deleted from the respective lab mean before calculation of the mean of lab means
(Certified Value). All outliers are shown in bold and aligned left in the tabulated data of the Appendix and to reiterate, have been omitted in the determination of the certified value.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and inter-laboratory 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.

## Indicative (uncertified) values

The indicative (uncertified) values (Table 2) are provided for the major and trace elements determined by oxidising fusion XRF $\left(\mathrm{Al}_{2} \mathrm{O}_{3}\right.$ to $\left.\mathrm{ZrO}_{2}\right)$, LOl at $1000^{\circ} \mathrm{C}$ and laser ablation with ICP-MS (Ag to Zr ) and are the means of duplicate assays from Bureau Veritas, Perth. Additional indicative values by other analytical methods are present where the number of laboratories reporting a particular analyte is insufficient (<5) to support certification or where inter-laboratory consensus is poor.

## 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 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

$$
x_{i j}^{\prime}=x_{i j}-\bar{x}_{i}+\frac{\sum_{i=1}^{p} \sum_{j=1}^{n_{i}} x_{i j}}{\sum_{i=1}^{p} n_{i}}
$$

where,
$x_{i j}$ is the $j$ th raw result reported by laboratory $i ;$
$x_{i j}^{\prime}$ is the $j$ th transformed result reported by laboratory $i$;
$n_{i}$ is the number of results reported by laboratory $i ;$
$p$ is the number of participating laboratories;
$\bar{x}_{i}$ is the raw mean for laboratory $i$.

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$
\begin{aligned}
& \text { Lower limit is } \ddot{x}-k_{2}^{\prime}(n, p, l-\alpha) s_{g}^{\prime \prime} \\
& \text { Upper limit is } \ddot{x}+k_{2}^{\prime}(n, p, l-\alpha) s_{g}^{\prime \prime}
\end{aligned}
$$

where,

$$
n \text { is the number of results; }
$$

$1-\alpha$ is the confidence level;
p is the proportion of results expected within the tolerance limits;
$k_{2}^{\prime}$ is the factor for two - sided tolerance limits ( $m, \alpha$ unknown);
$s_{g}^{\prime \prime}$ is the corrected grand standard deviation.

The meaning of these tolerance limits may be illustrated for zinc by acid digest, where 99\% of the time at least $95 \%$ of subsamples will have concentrations lying between 4.13 and 4.24 wt.\%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, $99 \%$ of the tolerance intervals so constructed would cover at least $95 \%$ of the total population, and $1 \%$ of the tolerance intervals would cover less than $95 \%$ of the total population (ISO Guide 35).

The corrected grand standard deviation, $s_{g}{ }^{\prime \prime}$, 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

$$
s_{g}^{\prime \prime}=\frac{\sum_{i=1}^{p}\left(s_{i}\left(1-\frac{s_{i}}{s_{g}^{\prime}}\right)\right)}{\sum_{i=1}^{p}\left(1-\frac{s_{i}}{s_{g}^{\prime}}\right)}
$$

where,

$$
1-\left(\frac{s_{i}}{2 s_{g}^{\prime}}\right) \text { is the weighting factor for laboratory } i \text {; }
$$

$s_{g}^{\prime}$ is the grand standard deviation computed from the transformed (i.e. means -adjusted) results
according to the formula

$$
s_{g}^{\prime}=\left[\frac{\sum_{i=j}^{p} \sum_{j=i}^{n_{i}}\left(x_{i j}^{\prime}-\bar{x}_{i}^{\prime}\right)^{2}}{\sum_{i=1}^{p} n_{i}-1}\right]^{1 / 2}
$$

where $\bar{x}_{i}^{\prime}$ is the transforme $d$ 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. Individual outliers (shown in bold in Tables A2 to A18) were removed prior to the calculation of tolerance intervals and a weighting factor of zero was applied to those data sets where $s_{l} / 2 s_{g}{ }^{\prime}>1$ (i.e. where the weighting factor $1-s_{l} / 2 s_{g}{ }^{\prime}<0$ ). Data sets displaying poor resolution (i.e. where the ratio of the reading increment divided by the measured value is $<1 / 20$ ) were also omitted.

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. Despite the limitations of this method, the tolerance intervals presented in Table 1 are considered to confirm a high level of homogeneity for this CRM.

Fig. 1. Mixed Acid Digest (no HF) results for $\mathbf{Z n}$ in OREAS 37


Fig. 2. Mixed Acid Digest (no HF) results for Pb in OREAS 37


## ANOVA Study

The sampling format for OREAS 37 was structured to enable nested ANOVA treatment of the round robin results. During the bagging stage immediately following final homogenization, samples were taken at 10 intervals representative of the entire batch of OREAS 37. Each lab received 4 samples per batch made up of paired samples from two different (non-adjacent) intervals. For example, the four samples that Lab A received consisted of:

- Sample 1 (from sampling interval 1 )
- Sample 2 (from sampling interval 6)
- Sample 3 (from sampling interval 1)
- Sample 4 (from sampling interval 6)

The acid digest zinc results were used as the test data for the ANOVA investigation comparing within- and between-unit variance. This approach permitted an assessment of homogeneity across the entire batch of OREAS 37. The test was performed using the following parameters:

- $\quad$ Significance Level $\alpha=P$ (type I error) $=0.05$
- Null Hypothesis, $\mathrm{H}_{0}$ : Between-unit variance is no greater than within-unit variance (reject $\mathrm{H}_{0}$ if p -value < 0.05 )
- Alternative Hypothesis, $\mathrm{H}_{1}$ : 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 same filtered dataset used to calculate the certified value for zinc via acid digest was used yielding a total of 76 samples from nine labs. The derived p-value of 0.692 indicates that there is no significant evidence that suggests between-unit variance is greater than within-unit variance. Conclusion: do not reject $\mathrm{H}_{0}$. Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that zinc is uniformly distributed throughout OREAS 37 and that the variance between two aliquots from the same unit is identical to the variance from two aliquots 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 standard 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).

Performance gates have been calculated from the same filtered data set used to determine the certified value, i.e. after removal of all individual and batch 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 zscore discrimination (rejected if $\left|z_{i}\right|>2.5$ ). The 1SD used to calculate performance gates is the mean of the remaining (accepted) lab standard deviations. Because batch to batch bias is an important component of performance gates, only results from the six labs that received 3 submissions of samples have been used in the calculations.

Table 3 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. Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate comparison with a $5 \%$ window calculated directly from the certified value. 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 3. Performance Gates for OREAS 37.

| Constituent | Certified Value | Absolute Standard Deviations |  |  |  |  | Relative Standard Deviations |  |  | 5\% window |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 1SD | $\begin{aligned} & 2 \mathrm{SD} \\ & \text { Low } \\ & \hline \end{aligned}$ | $\begin{aligned} & \text { 2SD } \\ & \text { High } \\ & \hline \end{aligned}$ | $\begin{aligned} & \text { 3SD } \\ & \text { Low } \end{aligned}$ | $\begin{aligned} & \text { 3SD } \\ & \text { High } \\ & \hline \end{aligned}$ | 1RSD | 2RSD | 3RSD | Low | High |
| Acid Digest |  |  |  |  |  |  |  |  |  |  |  |
| Ag (ppm) | 5.19 | 0.63 | 3.93 | 6.45 | 3.30 | 7.08 | 12.13\% | 24.26\% | 36.38\% | 4.93 | 5.45 |
| As (ppm) | 449 | 28 | 393 | 505 | 366 | 533 | 6.20\% | 12.41\% | 18.61\% | 427 | 472 |
| $\mathrm{Cu}(\mathrm{ppm})$ | 125 | 5 | 115 | 136 | 109 | 142 | 4.27\% | 8.54\% | 12.81\% | 119 | 132 |
| Fe (wt.\%) | 23.76 | 0.56 | 22.65 | 24.88 | 22.10 | 25.43 | 2.34\% | 4.68\% | 7.02\% | 22.58 | 24.95 |
| Mn (wt.\%) | 0.719 | 0.017 | 0.685 | 0.753 | 0.668 | 0.770 | 2.37\% | 4.73\% | 7.10\% | 0.683 | 0.755 |
| Pb (wt.\%) | 0.615 | 0.017 | 0.580 | 0.650 | 0.563 | 0.667 | 2.81\% | 5.63\% | 8.44\% | 0.584 | 0.646 |
| TI (ppm) | 63 | 5 | 52 | 73 | 47 | 78 | 8.18\% | 16.36\% | 24.54\% | 59 | 66 |
| Zn (wt.\%) | 6.26 | 0.15 | 5.97 | 6.55 | 5.82 | 6.70 | 2.35\% | 4.71\% | 7.06\% | 5.95 | 6.57 |
| Peroxide Fusion |  |  |  |  |  |  |  |  |  |  |  |
| Ag (ppm) | $\sim 5$ | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| As (ppm) | 460 | 42 | 375 | 544 | 42 | 375 | 9.19\% | 18.37\% | 27.56\% | 437 | 483 |
| $\mathrm{Cu}(\mathrm{ppm})$ | 129 | 6 | 118 | 141 | 6 | 118 | 4.47\% | 8.94\% | 13.41\% | 123 | 136 |
| Fe (wt.\%) | 23.53 | 0.54 | 22.46 | 24.61 | 0.54 | 22.46 | 2.29\% | 4.58\% | 6.87\% | 22.36 | 24.71 |
| Mn (wt.\%) | 0.769 | 0.027 | 0.715 | 0.822 | 0.027 | 0.715 | 3.50\% | 7.00\% | 10.50\% | 0.730 | 0.807 |
| Pb (wt.\%) | 0.597 | 0.02 | 0.566 | 0.628 | 0.02 | 0.566 | 2.57\% | 5.14\% | 7.70\% | 0.567 | 0.627 |
| TI (ppm) | 156 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| Zn (wt.\%) | 6.30 | 0.18 | 5.94 | 6.67 | 0.18 | 5.94 | 2.90\% | 5.80\% | 8.70\% | 5.99 | 6.62 |
| S (wt.\%) | 26.79 | 0.71 | 25.37 | 28.21 | 0.71 | 25.37 | 2.65\% | 5.31\% | 7.96\% | 25.45 | 28.13 |

SI unit equivalents: ppm, parts per million $\equiv \mathrm{mg} / \mathrm{kg} \equiv \mu \mathrm{g} / \mathrm{g} \equiv 0.0001 \mathrm{wt} . \% \equiv 1000 \mathrm{ppb}$, parts per billion.
Note: intervals may appear asymmetric due to rounding

## PARTICIPATING LABORATORIES

1. Acme Analytical Laboratories, Vancouver, BC, Canada
2. Activation Laboratories, Ancaster, Ontario, Canada
3. ALS Chemex, Johannesburg, Australia
4. ALS Chemex, Stafford, QLD, Australia
5. ALS Chemex, North Vancouver, BC, Canada
6. Amdel Laboratories, Perth, WA, Australia
7. Bureau Veritas (Ultra Trace) Geoanalytical, Perth, WA, Australia
8. Genalysis, Maddington, WA, Australia
9. SGS Analabs, Welshpool, Perth, WA, Australia
10. OMAC, Loughrea, Ireland

## PREPARER AND SUPPLIER

Certified reference material OREAS 37 is prepared, certified and supplied by:

| ORE Research \& Exploration Pty Ltd | Tel: | $+613-97290333$ |
| :--- | :--- | :--- |
| 37A Hosie Street | Fax: | $+613-97298338$ |
| Bayswater North VIC 3153 | Web: | www.ore.com.au |
| AUSTRALIA | Email: | info@ore.com.au |

OREAS 37 has been packaged under nitrogen in laminated foil pouches in 10 g units.

## INTENDED USE

OREAS 37 is a reference material intended for the following:
i) For the calibration of instruments used in the determination of the concentration of $\mathrm{Ag}, \mathrm{As}, \mathrm{Cu}, \mathrm{Fe}, \mathrm{Mn}, \mathrm{Pb}, \mathrm{S}, \mathrm{Tl}$ and Zn ;
ii) For the verification of analytical methods for $\mathrm{Ag}, \mathrm{As}, \mathrm{Cu}, \mathrm{Fe}, \mathrm{Mn}, \mathrm{Pb}, \mathrm{S}, \mathrm{Tl}$ and Zn ;
iii) For the monitoring of laboratory performance in the analysis of $\mathrm{Ag}, \mathrm{As}, \mathrm{Cu}, \mathrm{Fe}, \mathrm{Mn}$, $\mathrm{Pb}, \mathrm{S}, \mathrm{Tl}$ and Zn in geological samples.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 37 is sourced from medium grade zinc sulphide ore and has been packaged under dry nitrogen in robust laminated foil pouches. In its unopened state and under normal conditions of storage it has a shelf life beyond five years.

## INSTRUCTIONS FOR THE CORRECT USE

The certified values for CRM OREAS 37 refer to the concentration level of Ag, As, Cu, Fe, $\mathrm{Mn}, \mathrm{Pb}, \mathrm{S}, \mathrm{Tl}$ and Zn in its packaged state. Therefore it should not be dried prior to weighing and analysis.

## HANDLING INSTRUCTIONS

Fine powders pose a risk to eyes and lungs and therefore standard precautions such as the use of safety glasses and dust masks are advised.

## METROLOGICAL TRACEABILITY

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, "Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results (see ISO Guide 35:2006, Clause 10)."

## COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their field samples.

## 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.

## DOCUMENT HISTORY

| Revision <br> No | Date | Changes applied |
| :---: | :---: | :--- |
| 1 | $3^{\text {rd }}$ Sep, 2018 | Added major and trace element characterisation |
| 0 | $7^{\text {th }}$ Aug, 2012 | First publication |

## QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2015 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.


## CERTIFYING OFFICER

$3^{\text {rd }}$ Sep, 2018
Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager - ORE P/L

## REFERENCES

Rozendaal, A. \& Stalder, M. 2001. REE geochemistry of garnet associated with the Gamsberg $\mathrm{Zn}-\mathrm{Pb}$ deposit, South Africa. Mineral Deposits at the Beginning of the 21st Century, pp. 325.
ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.
ISO Guide 31 (2015), Reference materials - Contents of certificates and labels.
ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.
ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.

## APPENDIX

## Analytical Data for OREAS 37

Table A1. Key to abbreviations used in Tables A2 - A18.

| Abbreviation | Explanation |
| :--- | :--- |
| 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 |
| PF | sodium peroxide fusion |
| AR | aqua regia digest $\left(\mathrm{HNO}_{3}-\mathrm{HCl}\right)$ |
| 3A | three acid digest $\left(\mathrm{HNO}_{3}-\mathrm{HCl}-\mathrm{HClO}_{4}\right)$ |
| MA | mixed acid digest $\left(\mathrm{KClO}_{4}-\mathrm{HNO} 3-\mathrm{HBr}-\mathrm{HCl}\right)$ |
| OES | inductively coupled plasma optical emission spectrometry |
| MS | inductively coupled plasma mass spectrometry |
| AAS | atomic absorption spectrometry |
| Leco | IR combustion furnace |

Table A2. Mixed acid digest (no HF) results for Ag in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab A 3A*MS | $\begin{gathered} \hline \text { Lab } \\ \text { B } \\ 3 A^{*} \mathrm{OES} \end{gathered}$ | Lab C AR*MS | ```Lab D AR*OES``` | Lab E AR*OES | Lab F MA*MS | Lab G AR*OES | Lab H 3A*MS | $\begin{gathered} \text { Lab } \\ \text { I } \\ 3 A^{*} \mathrm{MS} \\ \hline \end{gathered}$ | ```Lab``` |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 5.20 | 5.00 | 4.90 | 6.00 | 4.00 | 5.02 | <2 | 6.00 | 5.00 | 6.00 |
| 2 | 5.10 | 5.00 | 4.80 | 4.00 | 5.00 | 4.82 | <2 | 6.00 | 5.00 | 6.00 |
| 3 | 5.10 | 4.00 | 4.70 | 5.00 | 6.00 | 4.50 | <2 | 6.00 | 5.00 | 6.00 |
| 4 | 5.20 | 3.00 | 4.80 | 6.00 | 5.00 | 5.01 | 2.00 | 8.00 | 5.00 | 7.00 |
| 5 | 6.00 | 5.00 | 4.07 | 5.00 | 5.00 | 4.94 |  |  |  |  |
| 6 | 6.00 | 6.00 | 4.14 | 6.00 | 4.00 | 4.91 |  |  |  |  |
| 7 | 6.00 | 6.00 | 4.60 | 5.00 | 3.00 | 5.13 |  |  |  |  |
| 8 | 6.00 | 5.00 | 4.50 | 6.00 | 5.00 | 5.15 |  |  |  |  |
| 9 | 4.70 | 5.00 | 5.00 | 6.00 | 2.00 | 5.07 |  |  |  |  |
| 10 | 5.00 | 7.00 | 4.90 | 4.00 | 2.00 | 5.22 |  |  |  |  |
| 11 | 4.70 | 6.00 | 4.80 | 5.00 | 3.00 | 5.08 |  |  |  |  |
| 12 | 4.60 | 6.00 | 4.80 | 6.00 | 2.00 | 5.41 |  |  |  |  |
| Mean | 5.30 | 5.25 | 4.67 | 5.33 | 3.83 | 5.02 | <2 | 6.50 | 5.00 | 6.25 |
| Median | 5.15 | 5.00 | 4.80 | 5.50 | 4.00 | 5.04 | <2 | 6.00 | 5.00 | 6.00 |
| Std.Dev. | 0.55 | 1.06 | 0.30 | 0.78 | 1.40 | 0.23 | - | 1.00 | 0.00 | 0.50 |
| Rel.Std.Dev. | 10.4\% | 20.1\% | 6.32\% | 14.6\% | 36.6\% | 4.48\% | - | 15.4\% | 0.00\% | 8.00\% |
| PDM ${ }^{3}$ | 2.11\% | 1.15\% | -10.1\% | 2.75\% | -26.1\% | -3.24\% | - | 25.2\% | -3.67\% | 20.4\% |

Table A3. Mixed acid digest (no HF) results for As in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab A 3A*MS | Lab B 3A*OES | Lab C AR*MS | Lab D AR*OES | Lab E AR*OES | Lab F MA*MS | Lab G AR*OES | Lab H 3A*MS | Lab I 3A*MS | Lab $J$ AR*OES $^{*}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 480 | 400 | NR | 430 | 460 | 468 | 380 | 454 | 445 | 450 |
| 2 | 472 | 400 | NR | 430 | 460 | 465 | 390 | 460 | 459 | 460 |
| 3 | 468 | 400 | NR | 440 | 490 | 457 | 400 | 459 | 426 | 450 |
| 4 | 479 | 400 | NR | 450 | 470 | 458 | 360 | 472 | 420 | 470 |
| 5 | 434 | 400 | 491 | 440 | 440 | 466 |  |  |  |  |
| 6 | 445 | 500 | 408 | 430 | 500 | 471 |  |  |  |  |
| 7 | 439 | 400 | 525 | 460 | 480 | 465 |  |  |  |  |
| 8 | 435 | 500 | 507 | 450 | 450 | 475 |  |  |  |  |
| 9 | 419 | 400 | 436 | 480 | 440 | 454 |  |  |  |  |
| 10 | 435 | 400 | 427 | 480 | 380 | 456 |  |  |  |  |
| 11 | 435 | 400 | 428 | 460 | 410 | 448 |  |  |  |  |
| 12 | 411 | 400 | 428 | 460 | 440 | 444 |  |  |  |  |
| Mean | 446 | 417 | 456 | 451 | 452 | 460 | 383 | 461 | 438 | 458 |
| Median | 437 | 400 | 432 | 450 | 455 | 462 | 385 | 460 | 436 | 455 |
| Std.Dev. | 23 | 39 | 44 | 18 | 34 | 9 | 17 | 8 | 18 | 10 |
| Rel.Std.Dev. | 5.20\% | 9.34\% | 9.70\% | 3.95\% | 7.42\% | 2.01\% | 4.46\% | 1.65\% | 4.08\% | 2.09\% |
| PDM ${ }^{3}$ | -0.70\% | -7.23\% | 1.58\% | 0.37\% | 0.56\% | 2.52\% | -14.8\% | 2.69\% | -2.60\% | 1.86\% |

Table A4. Mixed acid digest (no HF) results for Cu in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab A 3A*MS | ```Lab B 3A*OES``` | Lab C AR*OES | Lab $D$ AR*OES | Lab E AR*OES | Lab <br> F <br> MA*MS | Lab G AR*OES |  |  | Lab J AR*OES |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 130 | 120 | 110 | 117 | 104 | 125 | 100 | 143 | 140 | 123 |
| 2 | 129 | 120 | 100 | 118 | 102 | 124 | 100 | 143 | 130 | 123 |
| 3 | 128 | 120 | 110 | 132 | 112 | 123 | 100 | 135 | 135 | 121 |
| 4 | 131 | 120 | 110 | 118 | 108 | 122 | 100 | 138 | 125 | 122 |
| 5 | 137 | 120 | 136 | 121 | 128 | 126 |  |  |  |  |
| 6 | 135 | 120 | 108 | 118 | 121 | 124 |  |  |  |  |
| 7 | 137 | 120 | 134 | 123 | 123 | 124 |  |  |  |  |
| 8 | 133 | 120 | 124 | 127 | 119 | 124 |  |  |  |  |
| 9 | 121 | 120 | 132 | 125 | 123 | 134 |  |  |  |  |
| 10 | 122 | 120 | 131 | 122 | 120 | 129 |  |  |  |  |
| 11 | 123 | 110 | 136 | 124 | 119 | 132 |  |  |  |  |
| 12 | 118 | 110 | 134 | 122 | 120 | 127 |  |  |  |  |
| Mean | 129 | 118 | 122 | 122 | 117 | 126 | 100 | 140 | 133 | 122 |
| Median | 130 | 120 | 128 | 122 | 120 | 124 | 100 | 141 | 133 | 123 |
| Std.Dev. | 6 | 4 | 13 | 4 | 8 | 4 | 0 | 4 | 6 | 1 |
| Rel.Std.Dev. | 5.00\% | 3.29\% | 11.0\% | 3.59\% | 7.00\% | 3.00\% | 0.00\% | 2.82\% | 4.87\% | 0.78\% |
| PDM ${ }^{3}$ | 2.54\% | -5.70\% | -2.71\% | -2.57\% | -7.09\% | 0.58\% | -20.3\% | 11.4\% | 5.59\% | -2.57\% |

Table A5. Mixed acid digest (no HF) results for Fe in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | ```Lab A 3A*OES``` | $\begin{gathered} \text { Lab } \\ \text { B } \\ 3 A^{*} \text { OES } \end{gathered}$ | Lab <br> C <br> AR*OES | Lab D AR*OES | Lab <br> E <br> AR*OES | Lab F MA*MS | Lab G AR*OES | ```Lab H 3A*OES``` | $\begin{gathered} \text { Lab } \\ \text { I } \\ 3 A^{*} \mathrm{OES} \end{gathered}$ | Lab J AR*OES $^{*}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 23.70 | 22.43 | 18.50 | 23.30 | 23.00 | 23.09 | 23.98 | 24.76 | 24.80 | 23.30 |
| 2 | 23.50 | 23.39 | 18.00 | 23.00 | 22.90 | 22.95 | 23.90 | 24.80 | 24.70 | 22.90 |
| 3 | 23.40 | 22.88 | 19.50 | 23.20 | 24.60 | 23.11 | 24.38 | 24.34 | 24.90 | 23.00 |
| 4 | 23.50 | 23.36 | 18.50 | 22.80 | 23.50 | 23.03 | 24.10 | 24.49 | 24.40 | 23.40 |
| 5 | 24.20 | 23.70 | 20.60 | 24.20 | 24.20 | 22.94 |  |  |  |  |
| 6 | 24.40 | 24.05 | 21.10 | 24.00 | 23.40 | 22.99 |  |  |  |  |
| 7 | 24.70 | 23.80 | 21.30 | 24.50 | 24.20 | 23.00 |  |  |  |  |
| 8 | 24.20 | 24.00 | 20.40 | 24.40 | 23.20 | 23.03 |  |  |  |  |
| 9 | 24.70 | 23.41 | 23.70 | 24.90 | 24.00 | 23.22 |  |  |  |  |
| 10 | 24.60 | 23.78 | 23.00 | 24.30 | 23.40 | 22.97 |  |  |  |  |
| 11 | 24.90 | 22.81 | 23.00 | 24.80 | 23.50 | 23.14 |  |  |  |  |
| 12 | 24.80 | 23.23 | 22.30 | 24.50 | 23.30 | 22.89 |  |  |  |  |
| Mean | 24.22 | 23.40 | 20.83 | 23.99 | 23.60 | 23.03 | 24.09 | 24.60 | 24.70 | 23.15 |
| Median | 24.30 | 23.40 | 20.85 | 24.25 | 23.45 | 23.01 | 24.04 | 24.63 | 24.75 | 23.15 |
| Std.Dev. | 0.56 | 0.50 | 1.93 | 0.73 | 0.53 | 0.09 | 0.21 | 0.22 | 0.22 | 0.24 |
| Rel.Std.Dev. | 2.30\% | 2.14\% | 9.27\% | 3.03\% | 2.24\% | 0.40\% | 0.87\% | 0.89\% | 0.87\% | 1.03\% |
| PDM ${ }^{3}$ | 1.91\% | -1.52\% | -12.4\% | 0.96\% | -0.69\% | -3.09\% | 1.37\% | 3.51\% | 3.94\% | -2.58\% |

Table A6. Mixed acid digest (no HF) results for Mn in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | Lab A $3 A^{*} A A S$ | ```Lab B 3A*OES``` | Lab <br> C <br> AR*OES | Lab D AR*OES | Lab E AR*OES | Lab F MA*MS | Lab G AR*OES | Lab H 3A*OES | $\begin{gathered} \text { Lab } \\ \text { I } \\ 3 A^{*} \mathrm{OES} \end{gathered}$ | Lab J AR*OES |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 0.750 | 0.710 | 0.618 | 0.655 | 0.631 | 0.739 | 1.040 | 0.789 | 0.750 | 0.721 |
| 2 | 0.748 | 0.730 | 0.591 | 0.645 | 0.630 | 0.735 | 1.025 | 0.796 | 0.744 | 0.704 |
| 3 | 0.764 | 0.700 | 0.652 | 0.649 | 0.676 | 0.745 | 1.060 | 0.780 | 0.758 | 0.716 |
| 4 | 0.764 | 0.720 | 0.610 | 0.634 | 0.644 | 0.735 | 1.060 | 0.775 | 0.747 | 0.721 |
| 5 | <0.01 | 0.740 | 0.694 | 0.667 | 0.710 | 0.735 |  |  |  |  |
| 6 | <0.01 | 0.740 | 0.641 | 0.648 | 0.680 | 0.734 |  |  |  |  |
| 7 | <0.01 | 0.730 | 0.701 | 0.668 | 0.706 | 0.735 |  |  |  |  |
| 8 | <0.01 | 0.750 | 0.616 | 0.667 | 0.672 | 0.732 |  |  |  |  |
| 9 | 0.732 | 0.720 | 0.762 | 0.696 | 0.668 | 0.738 |  |  |  |  |
| 10 | 0.724 | 0.740 | 0.763 | 0.674 | 0.645 | 0.735 |  |  |  |  |
| 11 | 0.739 | 0.710 | 0.770 | 0.693 | 0.651 | 0.753 |  |  |  |  |
| 12 | 0.727 | 0.720 | 0.780 | 0.678 | 0.645 | 0.743 |  |  |  |  |
| Mean | 0.744 | 0.726 | 0.683 | 0.665 | 0.663 | 0.738 | 1.046 | 0.785 | 0.750 | 0.716 |
| Median | 0.744 | 0.725 | 0.673 | 0.667 | 0.660 | 0.735 | 1.050 | 0.784 | 0.749 | 0.719 |
| Std.Dev. | 0.016 | 0.015 | 0.071 | 0.019 | 0.027 | 0.006 | 0.017 | 0.009 | 0.006 | 0.008 |
| Rel.Std.Dev. | 2.10\% | 2.07\% | 10.38\% | 2.88\% | 4.04\% | 0.80\% | 1.63\% | 1.17\% | 0.80\% | 1.12\% |
| PDM ${ }^{3}$ | 3.41\% | 0.95\% | -4.98\% | -7.58\% | -7.76\% | 2.67\% | 45.5\% | 9.17\% | 4.28\% | -0.48\% |

Table A7. Mixed acid digest (no HF) results for Pb in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | $\begin{gathered} \text { Lab } \\ \text { A } \\ 3 A^{*} A A S \end{gathered}$ | $\begin{gathered} \hline \text { Lab } \\ \text { B } \\ 3 A^{*} \mathrm{OES} \\ \hline \end{gathered}$ | Lab C AR*OES | Lab D AR*OES | Lab E AR*OES | Lab F MA*MS | Lab G AR*OES | Lab <br> H <br> 3A*MS | Lab I 3A*MS | Lab <br> J <br> AR*OES |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 0.553 | 0.620 | 0.636 | 0.586 | 0.580 | 0.639 | 0.615 | 0.585 | 0.593 | 0.617 |
| 2 | 0.577 | 0.630 | 0.661 | 0.603 | 0.610 | 0.657 | 0.625 | 0.595 | 0.606 | 0.633 |
| 3 | 0.575 | 0.610 | 0.625 | 0.584 | 0.590 | 0.638 | 0.630 | 0.577 | 0.603 | 0.612 |
| 4 | 0.567 | 0.630 | 0.632 | 0.596 | 0.630 | 0.657 | 0.625 | 0.599 | 0.596 | 0.646 |
| 5 | 0.600 | 0.640 | 0.631 | 0.594 | 0.619 | 0.650 |  |  |  |  |
| 6 | 0.610 | 0.670 | 0.646 | 0.610 | 0.619 | 0.650 |  |  |  |  |
| 7 | $\mathbf{0 . 5 8 0}$ | 0.630 | 0.655 | 0.604 | 0.612 | 0.650 |  |  |  |  |
| 8 | 0.610 | 0.670 | 0.633 | 0.623 | 0.613 | 0.655 |  |  |  |  |
| 9 | 0.557 | 0.660 | 0.624 | 0.627 | 0.566 | 0.640 |  |  |  |  |
| 10 | 0.622 | 0.640 | 0.629 | 0.632 | 0.583 | 0.651 |  |  |  |  |
| 11 | 0.567 | 0.650 | 0.616 | 0.622 | 0.561 | 0.632 |  |  |  |  |
| 12 | 0.603 | 0.640 | 0.616 | 0.636 | 0.579 | 0.642 |  |  |  |  |
| Mean | 0.585 | 0.641 | 0.634 | 0.610 | 0.597 | 0.647 | 0.624 | 0.589 | 0.600 | 0.627 |
| Median | 0.579 | 0.640 | 0.632 | 0.607 | 0.600 | 0.650 | 0.625 | 0.590 | 0.600 | 0.625 |
| Std.Dev. | 0.023 | 0.019 | 0.014 | 0.018 | 0.023 | 0.008 | 0.006 | 0.010 | 0.006 | 0.016 |
| Rel.Std.Dev. | 3.93\% | 2.94\% | 2.2\% | 2.95\% | 3.85\% | 1.28\% | 1.01\% | 1.68\% | 1.01\% | 2.47\% |
| PDM ${ }^{3}$ | -4.86\% | 4.20\% | 3.03\% | -0.85\% | -2.95\% | 5.17\% | 1.42\% | -4.26\% | -2.52\% | 1.95\% |

Table A8. Mixed acid digest (no HF) results for TI in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | ```Lab A 3A*MS``` | $\begin{gathered} \text { Lab } \\ \text { B } \\ 3 A^{*} \mathrm{OES} \end{gathered}$ | Lab C AR*OES | $\begin{gathered} \text { Lab } \\ \mathrm{D} \\ \mathrm{AR}^{*} \mathrm{OES} \\ \hline \end{gathered}$ | Lab E AR*OES | Lab F MA*MS | $\begin{gathered} \text { Lab } \\ \text { G } \\ \text { AR*OES }^{*} \end{gathered}$ | $\begin{gathered} \text { Lab } \\ \mathrm{H} \\ 3 \mathrm{~A}^{*} \mathrm{MS} \end{gathered}$ | $\begin{gathered} \text { Lab } \\ \text { I } \\ 3 A^{*} \mathrm{MS} \end{gathered}$ | $\begin{gathered} \text { Lab } \\ \mathrm{J} \\ \text { AR*OES }^{*} \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 140 | 100 | 77 | 50 | <50 | 33 | <50 | 158 | 57 | 60 |
| 2 | 140 | 100 | 74 | 50 | 50 | 38 | <50 | 156 | 58 | 60 |
| 3 | 140 | 100 | 77 | 50 | <50 | 39 | <50 | 153 | 61 | 60 |
| 4 | 141 | 100 | 75 | 50 | <50 | 41 | <50 | 152 | 69 | 60 |
| 5 | 144 | 100 | 69 | 60 | 50 | 33 |  |  |  |  |
| 6 | 147 | 100 | 56 | 50 | 60 | 34 |  |  |  |  |
| 7 | 155 | 100 | 64 | 50 | 50 | 36 |  |  |  |  |
| 8 | 153 | 100 | 62 | 50 | 60 | 36 |  |  |  |  |
| 9 | 142 | NR | 81 | 50 | <50 | 28 |  |  |  |  |
| 10 | 147 | NR | 82 | 50 | <50 | 32 |  |  |  |  |
| 11 | 138 | NR | 82 | 50 | <50 | 33 |  |  |  |  |
| 12 | 135 | NR | 77 | 50 | <50 | 38 |  |  |  |  |
| Mean | 144 | 100 | 73 | 51 | 54 | 35 | <50 | 155 | 61 | 60 |
| Median | 142 | 100 | 76 | 50 | 50 | 35 | <50 | 155 | 60 | 60 |
| Std.Dev. | 6 | 0 | 8 | 3 | 5 | 3 | - | 3 | 5 | 0 |
| Rel.Std.Dev. | 4.18\% | 0.00\% | 11.3\% | 5.68\% | 10.1\% | 9.87\% | - | 1.76\% | 8.88\% | 0.00\% |
| PDM ${ }^{3}$ | 129\% | 59.9\% | 16.6\% | -18.7\% | -13.7\% | -43.8\% | - | 147\% | -2.06\% | -4.06\% |

Table A9. Mixed acid digest (no HF) results for Zn in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | Lab A 3A*AAS | Lab <br> B <br> 3A*OES | Lab C AR*OES | Lab D AR*OES | Lab <br> E <br> AR*OES | Lab F MA*MS | Lab <br> G <br> AR*OES | Lab <br> H 3A*OES | Lab I $3 A^{*} O E S$ | Lab J AR*OES |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 6.18 | 6.22 | 6.02 | 5.98 | 5.96 | 6.10 | 6.41 | 6.28 | 6.42 | 6.09 |
| 2 | 6.30 | 6.41 | 6.19 | 6.11 | 6.17 | 6.15 | 6.40 | 6.29 | 6.55 | 6.27 |
| 3 | 6.35 | 6.12 | 6.03 | 5.44 | 6.11 | 6.03 | 6.48 | 6.15 | 6.41 | 6.13 |
| 4 | 6.18 | 6.35 | 6.07 | 6.18 | 6.47 | 6.16 | 6.43 | 6.21 | 6.39 | 6.34 |
| 5 | 6.25 | 6.38 | 6.03 | 6.06 | 6.27 | 6.20 |  |  |  |  |
| 6 | 6.39 | 6.63 | 6.17 | 6.10 | 6.42 | 6.23 |  |  |  |  |
| 7 | 6.02 | 6.29 | 6.20 | 6.07 | 6.30 | 6.23 |  |  |  |  |
| 8 | 6.24 | 6.77 | 6.12 | 6.18 | 6.10 | 6.27 |  |  |  |  |
| 9 | 5.98 | 6.79 | 6.01 | 6.18 | 6.43 | 6.18 |  |  |  |  |
| 10 | 6.15 | 6.75 | 6.07 | 6.34 | 6.59 | 6.20 |  |  |  |  |
| 11 | 6.28 | 6.64 | 5.94 | 6.51 | 6.29 | 6.22 |  |  |  |  |
| 12 | 6.43 | 6.61 | 5.78 | 6.45 | 6.30 | 6.14 |  |  |  |  |
| Mean | 6.23 | 6.50 | 6.05 | 6.13 | 6.28 | 6.18 | 6.43 | 6.23 | 6.44 | 6.21 |
| Median | 6.25 | 6.51 | 6.05 | 6.15 | 6.30 | 6.19 | 6.42 | 6.25 | 6.42 | 6.20 |
| Std.Dev. | 0.14 | 0.23 | 0.12 | 0.27 | 0.18 | 0.07 | 0.04 | 0.07 | 0.07 | 0.12 |
| Rel.Std.Dev. | 2.20\% | 3.53\% | 1.93\% | 4.42\% | 2.84\% | 1.05\% | 0.55\% | 1.09\% | 1.13\% | 1.89\% |
| PDM ${ }^{3}$ | -0.49\% | 3.78\% | -3.32\% | -2.03\% | 0.38\% | -1.35\% | 2.71\% | -0.42\% | 2.91\% | -0.84\% |

Table A10. Peroxide fusion results for Ag in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab <br> A <br> PF*MS | Lab <br> B | $\begin{gathered} \text { Lab } \\ \mathrm{C} \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab D | Lab E | Lab F | $\begin{gathered} \text { Lab } \\ \text { G } \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab H PF*MS | Lab I PF*MS | Lab J - |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 5.00 | NR | < 60 | NR | NR | NR | <20 | 5.00 | <5 | NR |
| 2 | 5.00 | NR | < 60 | NR | NR | NR | <20 | 5.00 | <5 | NR |
| 3 | 5.00 | NR | < 60 | NR | NR | NR | <20 | 5.00 | < | NR |
| 4 | 5.00 | NR | < 60 | NR | NR | NR | <20 | 5.00 | <5 | NR |
| 5 | 6.00 | NR | 4.70 | NR | NR | NR |  |  |  |  |
| 6 | 6.00 | NR | 4.80 | NR | NR | NR |  |  |  |  |
| 7 | 6.00 | NR | 4.60 | NR | NR | NR |  |  |  |  |
| 8 | 6.00 | NR | 4.70 | NR | NR | NR |  |  |  |  |
| 9 | 5.00 | NR | $<50$ | NR | NR | NR |  |  |  |  |
| 10 | 5.00 | NR | $<50$ | NR | NR | NR |  |  |  |  |
| 11 | 5.00 | NR | $<50$ | NR | NR | NR |  |  |  |  |
| 12 | 5.00 | NR | $<50$ | NR | NR | NR |  |  |  |  |
| Mean | 5.33 |  | 4.70 |  |  |  | <20 | 5.00 | < |  |
| Median | 5.00 |  | 4.70 |  |  |  | <20 | 5.00 | < |  |
| Std.Dev. | 0.49 |  | 0.08 |  |  |  | - | 0.00 | - |  |
| Rel.Std.Dev. | 9.23\% |  | 1.74\% |  |  |  | - | 0.00\% | - |  |
| $\mathrm{PDM}^{3}$ | 6.43\% |  | -6.21\% |  |  |  | - | -0.22\% | - |  |

Table A11. Peroxide fusion results for As in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab A PF*MS | $\begin{gathered} \text { Lab } \\ \text { B } \\ \text { PF*OES }^{\star} \end{gathered}$ | $\begin{gathered} \text { Lab } \\ \mathrm{C} \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | $\begin{gathered} \hline \text { Lab } \\ \text { D } \\ \mathrm{PF}^{\star} \mathrm{OES} \\ \hline \end{gathered}$ | Lab | Lab | $\begin{gathered} \text { Lab } \\ \mathrm{G} \\ \mathrm{PF}^{\star} \mathrm{OES} \end{gathered}$ | Lab H PF*MS | $\begin{gathered} \text { Lab } \\ \text { I } \\ \mathrm{PF}^{*} \mathrm{MS} \end{gathered}$ | Lab J $\mathrm{PF}^{*} \mathrm{OES}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 510 | 400 | 400 | 400 | NR | NR | 360 | 487 | 465 | 500 |
| 2 | 490 | 400 | 400 | 400 | NR | NR | 435 | 511 | 478 | 500 |
| 3 | 490 | 400 | 400 | 400 | NR | NR | 415 | 510 | 465 | 500 |
| 4 | 450 | 400 | 400 | 500 | NR | NR | 400 | 506 | 480 | 600 |
| 5 | 430 | 400 | 500 | 400 | NR | NR |  |  |  |  |
| 6 | 470 | 500 | 400 | 500 | NR | NR |  |  |  |  |
| 7 | 420 | 400 | 500 | 400 | NR | NR |  |  |  |  |
| 8 | 430 | 400 | 500 | 400 | NR | NR |  |  |  |  |
| 9 | 470 | NR | 400 | 500 | NR | NR |  |  |  |  |
| 10 | 470 | NR | 500 | 500 | NR | NR |  |  |  |  |
| 11 | 470 | NR | 500 | 400 | NR | NR |  |  |  |  |
| 12 | 480 | NR | 500 | 500 | NR | NR |  |  |  |  |
| Mean | 465 | 413 | 450 | 442 |  |  | 403 | 504 | 472 | 525 |
| Median | 470 | 400 | 450 | 400 |  |  | 408 | 508 | 471 | 500 |
| Std.Dev. | 27 | 35 | 52 | 51 |  |  | 32 | 11 | 8 | 50 |
| Rel.Std.Dev. | 5.91\% | 8.57\% | 11.6\% | 11.7\% |  |  | 7.89\% | 2.23\% | 1.70\% | 9.52\% |
| PDM ${ }^{3}$ | 1.13\% | -10.3\% | -2.14\% | -3.95\% |  |  | -12.5\% | 9.50\% | 2.62\% | 14.2\% |

Table A12. Peroxide fusion results for Cu in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate No. | Lab <br> A <br> PF*MS | Lab B $\mathrm{PF}^{*} \mathrm{OES}$ | Lab C PF*OES | Lab D $\mathrm{PF}{ }^{*} \mathrm{OES}$ | Lab E | Lab F | $\begin{gathered} \hline \text { Lab } \\ \mathrm{G} \\ \mathrm{PF}^{*} \mathrm{OES} \\ \hline \end{gathered}$ | $\begin{gathered} \hline \hline \text { Lab } \\ \mathrm{H} \\ \mathrm{PF}^{*} \mathrm{OES} \\ \hline \end{gathered}$ | $\begin{gathered} \hline \text { Lab } \\ \mathrm{I} \\ \mathrm{PF}^{*} \mathrm{OES} \\ \hline \end{gathered}$ | Lab J $\mathrm{PF}^{*} \mathrm{OES}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 150 | 120 | 120 | 120 | NR | NR | 140 | NR | 140 | 100 |
| 2 | 150 | 120 | 130 | 150 | NR | NR | 120 | NR | 130 | 100 |
| 3 | 150 | 110 | 120 | 130 | NR | NR | 130 | NR | 140 | 100 |
| 4 | 140 | 120 | 120 | 130 | NR | NR | 125 | NR | 140 | 200 |
| 5 | 130 | 130 | 130 | 120 | NR | NR |  |  |  |  |
| 6 | 130 | 130 | 120 | 130 | NR | NR |  |  |  |  |
| 7 | 140 | 120 | 130 | 130 | NR | NR |  |  |  |  |
| 8 | 130 | 130 | 130 | 120 | NR | NR |  |  |  |  |
| 9 | 130 | 100 | 110 | 130 | NR | NR |  |  |  |  |
| 10 | 130 | 100 | 110 | 130 | NR | NR |  |  |  |  |
| 11 | 130 | 100 | 120 | 130 | NR | NR |  |  |  |  |
| 12 | 140 | 100 | 100 | 140 | NR | NR |  |  |  |  |
| Mean | 138 | 115 | 120 | 130 |  |  | 129 |  | 138 | 125 |
| Median | 135 | 120 | 120 | 130 |  |  | 128 |  | 140 | 100 |
| Std.Dev. | 9 | 12 | 10 | 9 |  |  | 9 |  | 5 | 50 |
| Rel.Std.Dev. | 6.30\% | 10.8\% | 7.95\% | 6.56\% |  |  | 6.63\% |  | 3.64\% | 40.0\% |
| PDM ${ }^{3}$ | 6.53\% | -10.9\% | -7.03\% | 0.72\% |  |  | -0.25\% |  | 6.53\% | -3.15\% |

Table A13. Peroxide fusion results for Fe in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | Lab <br> A <br> PF*OES | Lab B $\mathrm{PF}^{*} \mathrm{OES}$ | Lab <br> C <br> PF*OES | $\begin{gathered} \text { Lab } \\ \text { D } \\ \text { PF*OES }^{*} \end{gathered}$ | Lab E | Lab F - | $\begin{gathered} \text { Lab } \\ \mathrm{G} \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab <br> H <br> PF*OES | $\begin{gathered} \text { Lab } \\ \text { I } \\ \text { PF*OES }^{*} \text { OES } \end{gathered}$ | Lab J $\mathrm{PF}^{*} \mathrm{OES}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 21.50 | 23.06 | 23.60 | 22.70 | NR | NR | 22.60 | 23.64 | 24.90 | 31.70 |
| 2 | 21.80 | 23.40 | 23.20 | 23.60 | NR | NR | 22.70 | 23.86 | 24.60 | 33.40 |
| 3 | 23.10 | 22.46 | 23.50 | 22.40 | NR | NR | 22.60 | 23.87 | 24.90 | 35.10 |
| 4 | 22.70 | 23.01 | 23.10 | 23.00 | NR | NR | 22.80 | 24.49 | 25.20 | 34.80 |
| 5 | 25.00 | 24.37 | 24.80 | 23.50 | NR | NR |  |  |  |  |
| 6 | 23.80 | 23.86 | 23.90 | 23.10 | NR | NR |  |  |  |  |
| 7 | 25.10 | 24.13 | 24.10 | 23.50 | NR | NR |  |  |  |  |
| 8 | 24.00 | 24.09 | 23.80 | 24.20 | NR | NR |  |  |  |  |
| 9 | 22.70 | 23.43 | 22.90 | 23.10 | NR | NR |  |  |  |  |
| 10 | 23.10 | 23.51 | 23.00 | 22.40 | NR | NR |  |  |  |  |
| 11 | 24.20 | 23.35 | 23.30 | 22.70 | NR | NR |  |  |  |  |
| 12 | 24.20 | 22.81 | 23.40 | 21.70 | NR | NR |  |  |  |  |
| Mean | 23.43 | 23.46 | 23.55 | 22.99 |  |  | 22.68 | 23.97 | 24.90 | 33.75 |
| Median | 23.45 | 23.42 | 23.45 | 23.05 |  |  | 22.65 | 23.87 | 24.90 | 34.10 |
| Std.Dev. | 1.15 | 0.58 | 0.54 | 0.67 |  |  | 0.10 | 0.37 | 0.24 | 1.55 |
| Rel.Std.Dev. | 4.91\% | 2.45\% | 2.29\% | 2.91\% |  |  | 0.42\% | 1.53\% | 0.98\% | 4.61\% |
| PDM ${ }^{3}$ | -0.43\% | -0.33\% | 0.07\% | -2.31\% |  |  | -3.65\% | 1.83\% | 5.80\% | 43.4\% |

Table A14. Analytical results fusion Mn in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | Lab <br> A PF*OES | $\begin{gathered} \text { Lab } \\ \mathrm{B} \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab C $\mathrm{PF}^{*} \mathrm{OES}$ | Lab <br> D PF*OES | Lab E | Lab F | Lab G PF*OES | Lab H $\mathrm{PF}^{*} \mathrm{OES}$ | Lab I $\mathrm{PF}^{*} \mathrm{OES}$ | Lab J PF*OES |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 0.721 | 0.790 | 0.770 | 0.805 | NR | NR | 1.050 | 0.800 | 0.752 | 0.751 |
| 2 | 0.715 | 0.790 | 0.740 | 0.805 | NR | NR | 1.050 | 0.800 | 0.734 | 0.743 |
| 3 | 0.740 | 0.760 | 0.770 | 0.790 | NR | NR | 1.040 | 0.800 | 0.752 | 0.805 |
| 4 | 0.730 | 0.790 | 0.750 | 0.790 | NR | NR | 1.060 | 0.900 | 0.736 | 0.798 |
| 5 | 0.965 | 0.770 | 0.800 | 0.790 | NR | NR |  |  |  |  |
| 6 | 0.894 | 0.750 | 0.750 | 0.751 | NR | NR |  |  |  |  |
| 7 | 0.965 | 0.780 | 0.780 | 0.790 | NR | NR |  |  |  |  |
| 8 | 0.899 | 0.750 | 0.740 | 0.782 | NR | NR |  |  |  |  |
| 9 | 0.773 | NR | 0.720 | 0.790 | NR | NR |  |  |  |  |
| 10 | 0.763 | NR | 0.720 | 0.743 | NR | NR |  |  |  |  |
| 11 | 0.823 | NR | 0.730 | 0.782 | NR | NR |  |  |  |  |
| 12 | 0.801 | NR | 0.730 | 0.720 | NR | NR |  |  |  |  |
| Mean | 0.816 | 0.773 | 0.750 | 0.778 |  |  | 1.050 | 0.825 | 0.744 | 0.774 |
| Median | 0.787 | 0.775 | 0.745 | 0.790 |  |  | 1.050 | 0.800 | 0.744 | 0.774 |
| Std.Dev. | 0.093 | 0.018 | 0.025 | 0.026 |  |  | 0.008 | 0.050 | 0.010 | 0.032 |
| Rel.Std.Dev. | 11.4\% | 2.27\% | 3.36\% | 3.35\% |  |  | 0.78\% | 6.06\% | 1.32\% | 4.08\% |
| PDM ${ }^{3}$ | 6.15\% | 0.52\% | -2.41\% | 1.28\% |  |  | 36.6\% | 7.35\% | -3.25\% | 0.78\% |


| Replicate No. | Lab <br> A PF*MS | Lab <br> B <br> PF*OES | $\begin{gathered} \text { Lab } \\ \mathrm{C} \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | $\begin{gathered} \hline \text { Lab } \\ \text { D } \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab E | Lab F | Lab G - | Lab <br> H PF*MS |  | Lab J $\mathrm{PF}^{*} \mathrm{OES}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 0.594 | 0.590 | 0.600 | 0.570 | NR | NR | NR | 0.583 | 0.605 | 0.510 |
| 2 | 0.599 | 0.610 | 0.610 | 0.610 | NR | NR | NR | 0.614 | 0.629 | 0.570 |
| 3 | 0.611 | 0.580 | 0.630 | 0.560 | NR | NR | NR | 0.582 | 0.604 | 0.580 |
| 4 | 0.553 | 0.610 | 0.620 | 0.610 | NR | NR | NR | 0.603 | 0.605 | 0.590 |
| 5 | 0.566 | 0.660 | 0.620 | 0.590 | NR | NR |  |  |  |  |
| 6 | 0.593 | 0.660 | 0.620 | 0.610 | NR | NR |  |  |  |  |
| 7 | 0.589 | 0.650 | 0.600 | 0.590 | NR | NR |  |  |  |  |
| 8 | 0.596 | 0.670 | 0.620 | 0.630 | NR | NR |  |  |  |  |
| 9 | 0.585 | NR | 0.580 | 0.600 | NR | NR |  |  |  |  |
| 10 | 0.605 | NR | 0.590 | 0.610 | NR | NR |  |  |  |  |
| 11 | 0.593 | NR | 0.590 | 0.590 | NR | NR |  |  |  |  |
| 12 | 0.621 | NR | 0.590 | 0.590 | NR | NR |  |  |  |  |
| Mean | 0.592 | 0.629 | 0.606 | 0.597 |  |  |  | 0.596 | 0.611 | 0.563 |
| Median | 0.594 | 0.630 | 0.605 | 0.595 |  |  |  | 0.593 | 0.605 | 0.575 |
| Std.Dev. | 0.018 | 0.035 | 0.016 | 0.019 |  |  |  | 0.016 | 0.012 | 0.036 |
| Rel.Std.Dev. | 3.10\% | 5.60\% | 2.68\% | 3.22\% |  |  |  | 2.63\% | 1.99\% | 6.39\% |
| $\mathrm{PDM}^{3}$ | -0.82\% | 5.32\% | 1.48\% | -0.05\% |  |  |  | -0.22\% | 2.31\% | -5.78\% |

Table A16. Peroxide fusion results for Tl in OREAS 37 (abbreviations as in Table A1; values in ppm)

| Replicate | Lab | Lab | Lab | Lab | Lab | Lab | Lab | Lab | Lab | Lab |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| No. | A | B | C | D | E | F | G | H | I | $J$ |
|  | PF*MS | PF*OES | PF*OES | - | - | - | - | PF*MS | PF*MS | - |
| 1 | 143 | 100 | 187 | NR | NR | NR | NR | 156 | 135 | NR |
| 2 | 144 | 200 | 194 | NR | NR | NR | NR | 163 | 140 | NR |
| 3 | 143 | 200 | 190 | NR | NR | NR | NR | 157 | 152 | NR |
| 4 | 144 | 100 | 186 | NR | NR | NR | NR | 156 | 149 | NR |
| 5 | 142 | 200 | 162 | NR | NR | NR |  |  |  |  |
| 6 | 142 | 200 | 175 | NR | NR | NR |  |  |  |  |
| 7 | 146 | 100 | 173 | NR | NR | NR |  |  |  |  |
| 8 | 147 | 200 | 166 | NR | NR | NR |  |  |  |  |
| 9 | 140 | NR | 171 | NR | NR | NR |  |  |  |  |
| 10 | 142 | NR | 167 | NR | NR | NR |  |  |  |  |
| 11 | 141 | NR | 170 | NR | NR | NR |  |  |  |  |
| 12 | 145 | NR | 178 | NR | NR | NR |  |  |  |  |
| Mean | 143 | 163 | 177 |  |  |  |  | 158 | 144 |  |
| Median | 143 | 200 | 174 |  |  |  |  | 156 | 144 |  |
| Std.Dev. | 2 | 52 | 10 |  |  |  |  | 4 | 8 |  |
| Rel.Std.Dev. | 1.43\% | 31.8\% | 5.89\% |  |  |  |  | 2.26\% | 5.53\% |  |
| PDM ${ }^{3}$ | -8.45\% | 3.85\% | 12.9\% |  |  |  |  | 1.01\% | -8.05\% |  |

Table A17. Peroxide fusion results for Zn in OREAS 37 (abbreviations as in Table A1; values in wt.\%)

| Replicate No. | Lab <br> A PF*OES | $\begin{gathered} \text { Lab } \\ \text { B } \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | $\begin{gathered} \text { Lab } \\ \text { C } \\ \text { PF*OES }^{*} \end{gathered}$ | $\begin{gathered} \hline \text { Lab } \\ \text { D } \\ \mathrm{PF}^{*} \mathrm{OES} \end{gathered}$ | Lab E | Lab F | Lab G PF*OES | Lab <br> H <br> PF*OES | Lab । PF*OES | Lab J $\mathrm{PF}^{*} \mathrm{OES}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 6.19 | 6.52 | 6.07 | 5.98 | NR | NR | 4.23 | 6.03 | 6.55 | 5.85 |
| 2 | 6.00 | 6.57 | 6.16 | 6.24 | NR | NR | 4.27 | 6.09 | 6.63 | 6.17 |
| 3 | 5.89 | 6.25 | 6.10 | 6.23 | NR | NR | 4.25 | 6.07 | 6.57 | 6.01 |
| 4 | 5.85 | 6.45 | 6.23 | 6.38 | NR | NR | 4.29 | 6.27 | 6.55 | 6.32 |
| 5 | 6.55 | 6.78 | 6.31 | 5.97 | NR | NR |  |  |  |  |
| 6 | 6.49 | 6.93 | 6.34 | 6.22 | NR | NR |  |  |  |  |
| 7 | 6.58 | 6.76 | 6.14 | 6.18 | NR | NR |  |  |  |  |
| 8 | 6.48 | 7.00 | 6.12 | 6.45 | NR | NR |  |  |  |  |
| 9 | 6.14 | 6.68 | 6.39 | 6.18 | NR | NR |  |  |  |  |
| 10 | 6.32 | 6.57 | 6.34 | 6.34 | NR | NR |  |  |  |  |
| 11 | 6.31 | 6.54 | 6.12 | 6.51 | NR | NR |  |  |  |  |
| 12 | 6.47 | 6.44 | 6.27 | 6.45 | NR | NR |  |  |  |  |
| Mean | 6.27 | 6.62 | 6.22 | 6.26 |  |  | 4.26 | 6.12 | 6.58 | 6.09 |
| Median | 6.32 | 6.57 | 6.20 | 6.24 |  |  | 4.26 | 6.08 | 6.56 | 6.09 |
| Std.Dev. | 0.26 | 0.22 | 0.11 | 0.17 |  |  | 0.03 | 0.11 | 0.04 | 0.20 |
| Rel.Std.Dev. | 4.10\% | 3.25\% | 1.78\% | 2.78\% |  |  | 0.61\% | 1.74\% | 0.58\% | 3.33\% |
| PDM ${ }^{3}$ | -0.47\% | 5.11\% | -1.37\% | -0.66\% |  |  | -32.4\% | -2.97\% | 4.33\% | -3.41\% |

Table A18. Analytical results for S in OREAS 37 (abbreviations as in Table A1; values in wt. \%)

| Replicate No. | Lab <br> A <br> Leco | Lab <br> B <br> Leco | Lab C Leco | Lab D Leco | Lab E Leco | Lab F Leco | Lab G AR*OES | Lab H Leco | Lab <br> I Leco | Lab <br> J Leco |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 25.40 | 26.12 | 26.00 | 25.20 | 27.00 | 27.47 | 26.65 | 27.24 | 26.50 | 28.60 |
| 2 | 26.20 | 26.82 | 26.20 | 26.00 | 26.20 | 27.19 | 27.10 | 26.91 | 25.70 | 28.60 |
| 3 | 26.50 | 26.36 | 26.90 | 25.60 | 26.90 | 27.88 | 27.15 | 27.03 | 26.20 | 28.40 |
| 4 | 26.70 | 26.04 | 26.10 | 25.90 | 26.20 | 27.29 | 27.00 | 27.19 | 26.20 | 28.40 |
| 5 | 27.30 | 26.46 | 28.60 | 26.20 | 25.80 | 27.43 |  |  |  |  |
| 6 | 27.40 | 15.55 | 27.80 | 26.30 | 25.00 | 27.51 |  |  |  |  |
| 7 | 27.50 | 26.22 | 28.70 | 26.30 | 25.10 | 27.98 |  |  |  |  |
| 8 | 27.20 | 25.30 | 28.40 | 26.50 | 25.00 | 28.04 |  |  |  |  |
| 9 | 27.70 | NR | 26.20 | 25.40 | 25.40 | 28.19 |  |  |  |  |
| 10 | 27.40 | NR | 25.80 | 23.80 | 25.90 | 27.50 |  |  |  |  |
| 11 | 27.90 | NR | 26.20 | 23.50 | 26.00 | 27.79 |  |  |  |  |
| 12 | 27.50 | NR | 25.70 | 22.60 | 25.60 | 27.46 |  |  |  |  |
| Mean | 27.06 | 24.86 | 26.88 | 25.28 | 25.84 | 27.64 | 26.98 | 27.09 | 26.15 | 28.50 |
| Median | 27.35 | 26.17 | 26.20 | 25.75 | 25.85 | 27.50 | 27.05 | 27.11 | 26.20 | 28.50 |
| Std.Dev. | 0.72 | 3.79 | 1.16 | 1.28 | 0.67 | 0.32 | 0.23 | 0.15 | 0.33 | 0.12 |
| Rel.Std.Dev. | 2.67\% | 15.2\% | 4.31\% | 5.06\% | 2.60\% | 1.16\% | 0.84\% | 0.55\% | 1.27\% | 0.41\% |
| PDM ${ }^{3}$ | 0.99\% | -7.22\% | 0.34\% | -5.66\% | -3.55\% | 3.18\% | 0.68\% | 1.12\% | -2.40\% | 6.37\% |


[^0]:    SI unit equivalents: ppm, parts per million $\equiv \mathrm{mg} / \mathrm{kg} \equiv \mu \mathrm{g} / \mathrm{g} \equiv 0.0001 \mathrm{wt} . \% \equiv 1000 \mathrm{ppb}$, parts per billion.
    Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

