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CERTIFICATE OF ANALYSIS FOR

ZINC TAILINGS CERTIFIED REFERENCE MATERIAL

OREAS 34i

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 34i.

Constituent	Certified Value	Within-Lab SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Acid Digestions (no HF)						
Ag, Silver (ppm)	9.09	0.562	8.72	9.47	IND	IND
As, Arsenic (ppm)	2236	71	2179	2293	2198	2274
Ca, Calcium (wt.%)	10.40	0.421	8.80	12.00	10.18	10.61
Cd, Cadmium (ppm)	24.4	2.25	22.8	26.1	23.5	25.4
Co, Cobalt (ppm)	148	4	144	152	146	150
Cu, Copper (ppm)	237	8	228	246	231	243
Fe, Iron (wt.%)	19.86	0.436	19.00	20.71	19.57	20.15
Mg, Magnesium (wt.%)	4.89	0.098	4.79	4.98	4.82	4.96
Mn, Manganese (ppm)	658	16	603	713	645	671
Ni, Nickel (ppm)	411	14	399	422	403	418
P, Phosphorus (ppm)	446	37	397	496	418	475
Pb, Lead (wt.%)	0.292	0.011	0.280	0.303	0.285	0.298
S, Sulphur (wt.%)	22.58	0.487	21.13	24.04	22.25	22.92
Tl, Thallium (ppm)	234	22	219	250	222	246
Zn, Zinc (wt.%)	1.34	0.041	1.32	1.36	1.31	1.37
Peroxide Fusion ICP*						
Ag, Silver (ppm)	9.45	0.793	8.80	10.09	IND	IND
As, Arsenic (ppm)	2325	76	2179	2471	2291	2359
Co, Cobalt (ppm)	152	24	140	163	IND	IND
Cu, Copper (ppm)	230	21	215	246	IND	IND
Fe, Iron (wt.%)	19.99	0.476	19.43	20.55	19.55	20.43
Mg, Magnesium (wt.%)	5.13	0.240	4.87	5.40	5.02	5.24
Ni, Nickel (ppm)	413	34	380	446	IND	IND
Pb, Lead (wt.%)	0.297	0.011	0.290	0.304	0.292	0.302
Tl, Thallium (ppm)	235	7	187	282	228	241
Zn, Zinc (wt.%)	1.33	0.029	1.30	1.36	1.31	1.35

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.

Please note: intervals may appear asymmetric due to rounding.

*With the exception of some analytes where up to two laboratories used a 4-acid digestion and one laboratory used INAA (see ‘OREAS 34i DataPack-1.0.190508_162701.xlsx’ for details).

Table 2. Indicative Values for OREAS 34i.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Acid Digestions (no HF)								
Al	wt.%	*0.305*	Hg	ppm	*1.16*	Sr	ppm	*88*
B	ppm	*< 10*	K	wt.%	*0.106*	Th	ppm	*< 100*
Ba	ppm	*2463*	La	ppm	*< 50*	Ti	wt.%	*< 0.05*
Be	ppm	*< 5*	Mo	ppm	*14.2*	U	ppm	*< 50*
Bi	ppm	*5.14*	Na	wt.%	*0.071*	V	ppm	*9.91*
Ce	ppm	*10.0*	Sb	ppm	*3.72*	W	ppm	*< 50*
Cr	ppm	*18.6*	Sc	ppm	*< 5*			
Ga	ppm	*< 50*	Sn	ppm	*< 100*			
Peroxide Fusion ICP*								
Al	wt.%	*1.02*	Cr	ppm	*51*	S	wt.%	*25.48*
Ba	ppm	*2396*	K	wt.%	*0.425*	Sb	ppm	*3.27*
Ca	wt.%	*11.58*	Mn	wt.%	*0.080*	Si	wt.%	*3.46*
Cd	ppm	*24.1*	P	ppm	*273*	Ti	wt.%	*0.079*

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.

*With the exception of some analytes where up to two laboratories used a 4-acid digestion and one laboratory used INAA (see 'OREAS 34i DataPack-1.0.190508_162701.xlsx' for details).

SOURCE MATERIAL

OREAS 34i is a zinc tailings matrix-matched certified reference material (MMCRM) prepared by Ore Research and Exploration. The material was sourced from the Lisheen carbonate hosted zinc-lead deposit located in south-central Ireland. It is prepared from a blend of tailings, ore, intermediate and concentrate samples.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- drying at 65°C to constant mass;
- crushing and screening;
- preliminary homogenisation;
- milling to minus 30 microns;
- final homogenisation;
- packaging into 10g units under nitrogen and sealed in laminated foil pouches.

ANALYTICAL PROGRAM

Ten commercial laboratories participated in the analytical program to characterise Ag, As, Ba, Cd, Co, Cu, Fe, Mg, Ni, Pb, Sb, Tl and Zn in OREAS 34i. The laboratories were requested to analyse all elements by three acid (preferred) or strong aqua regia digestion and by sodium peroxide fusion. To evaluate and compensate for the effects of batch-to-batch variation at individual laboratories, samples were submitted in two batches of four

20g samples to each of the participating laboratories at weekly intervals. The Mine Laboratory received one batch of samples for analysis to enable a comparison of their 3-acid digest method with that of the commercial labs. It is important to note, however, that their data has not been included in the statistical analysis.

Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 34i DataPack-1.0.190508_162701.xlsx**).

All ten commercial labs and the mine lab participated in the acid digest work and employed flame AAS, ICP-OES and ICP-MS instrumental finishes. Seven of these commercial labs also carried out total method determination of the analytes including sodium peroxide fusion ICP-OES/MS analysis (6 labs), four acid digest with ICP-OES/MS (1 lab) and at Actlabs INAA was used (in addition to sodium peroxide fusion) to determine some analytes. 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.

STATISTICAL ANALYSIS

Certified Value and Confidence Interval

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

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

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

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 intervals are obtained by calculation of the variance of the consensus value (mean of means) and reference to Student's-*t* distribution with degrees of freedom (*p*-1).

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

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

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 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 $|z_i| > 2.5$) 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.

The test for outlying laboratory batches is also based on z-score discrimination (rejected if $|z_i| > 2.5$) and these batches are deleted from the respective lab mean before calculation of the mean of lab means (Certified Value). Following identification of z-score outliers a 3SD filter is applied, with those values lying outside this window relegated to outlying status. In certain instances statistician's prerogative has been employed in discriminating outliers.

Individual outliers and, more rarely, laboratory batches deemed to be outlying, are shown in bold and left justified in the tablulated data of the Appendix and 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 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.

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:

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

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:

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

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

where

n is the number of results;

$1-\alpha$ is the confidence level;

p is the proportion of results expected within the tolerance limits;

k'_2 is the factor for two – sided tolerance limits (m, α unknown);

s''_g is the corrected grand standard deviation.

The meaning of these tolerance limits may be illustrated for zinc by acid digest (no HF), where 99% of the time at least 95% of subsamples will have concentrations lying between 1.31 and 1.37 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'', 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:*

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

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

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. Individual outliers (shown in bold in Tables A2 to A27) 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. Despite the limitations of this method, the tolerance intervals presented in Table 2 are considered to confirm a high level of homogeneity for this CRM.

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 (analytical bias and precision) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. There are three main sources of measurement error:

i) inter-lab bias
ii) analytical precision (repeatability), and
iii) 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 z-score discrimination (rejected if $|z_i| > 2.5$). The one sigma standard deviation used for performance gates is the mean of the remaining (accepted) lab standard deviations. Performance gates have been calculated for two and three standard deviations and are given in Table 3.

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 is included which uses a ±5% error bar on the certified value as the window of acceptability (see Table 3). 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 3. Performance Gates for OREAS 34i.

Constituent	**Certified Value**	**1SD**	**Within-Lab Performance Gates**					
			2SD		**3SD**		**5% Interval**	
			Low	**High**	**Low**	**High**	**Low**	**High**
Acid Digests (no HF)								
Ag, Silver (ppm)	9.09	0.562	7.97	10.22	7.41	10.78	8.64	9.55
As, Arsenic (ppm)	2236	71	2094	2378	2023	2449	2124	2348
Ca, Calcium (wt.%)	10.40	0.421	9.56	11.24	9.13	11.66	9.88	10.92
Cd, Cadmium (ppm)	24.4	2.25	19.9	28.9	17.7	31.2	23.2	25.6
Co, Cobalt (ppm)	148	4	140	156	136	160	141	156
Cu, Copper (ppm)	237	8	222	253	214	261	225	249
Fe, Iron (wt.%)	19.86	0.436	18.99	20.73	18.55	21.16	18.86	20.85
Mg, Magnesium (wt.%)	4.89	0.098	4.69	5.08	4.59	5.18	4.64	5.13
Mn, Manganese (ppm)	658	16	625	690	609	707	625	691
Ni, Nickel (ppm)	411	14	382	439	368	454	390	431
P, Phosphorus (ppm)	446	37	372	521	334	559	424	469
Pb, Lead (wt.%)	0.292	0.011	0.269	0.315	0.257	0.326	0.277	0.306
S, Sulphur (wt.%)	22.58	0.487	21.61	23.56	21.12	24.05	21.46	23.71
Tl, Thallium (ppm)	234	22	191	278	169	299	222	246
Zn, Zinc (wt.%)	1.34	0.041	1.26	1.42	1.22	1.46	1.27	1.41
Peroxide Fusion ICP*								
Ag, Silver (ppm)	9.45	0.793	7.86	11.03	7.07	11.82	8.97	9.92
As, Arsenic (ppm)	2325	76	2174	2476	2098	2552	2209	2441
Co, Cobalt (ppm)	152	24	103	200	78	225	144	159
Cu, Copper (ppm)	230	21	187	273	166	295	219	242
Fe, Iron (wt.%)	19.99	0.476	19.04	20.94	18.56	21.42	18.99	20.99
Mg, Magnesium (wt.%)	5.13	0.240	4.65	5.61	4.42	5.85	4.88	5.39
Ni, Nickel (ppm)	413	34	344	482	309	516	392	434
Pb, Lead (wt.%)	0.297	0.011	0.274	0.320	0.263	0.331	0.282	0.312
Tl, Thallium (ppm)	235	7	221	248	215	254	223	246
Zn, Zinc (wt.%)	1.33	0.029	1.27	1.39	1.24	1.42	1.26	1.40

Note - values may appear asymmetric due to rounding.

*With the exception of some analytes where up to two laboratories used a 4-acid digestion and one laboratory used INAA (see ‘OREAS 34i DataPack-1.0.190508_162701.xlsx’ for details).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Johannesburg, South Africa
4. ALS, Loughrea, Galway, Ireland
5. ALS, Perth, WA, Australia
6. ALS, Vancouver, BC, Canada
7. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
8. Bureau Veritas Geoanalytical, Perth, WA, Australia
9. Intertek Genalysis, Perth, WA, Australia
10. SGS Australia Mineral Services, Perth, WA, Australia

PREPARER AND SUPPLIER

The reference material OREAS 34i has been prepared and certified and is supplied by:

ORE Research & Exploration Pty Ltd		Tel:	+613-9729 0333
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Bayswater North VIC 3153		Web:	www.ore.com.au
AUSTRALIA		Email:	info@ore.com.au

OREAS 34i has been packaged under nitrogen in unit sizes of 10g.

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.

INTENDED USE

OREAS 34i is a reference material intended for the following:

- for the calibration of instruments used in the determination of the concentration of Ag, As, Ba, Cd, Co, Cu, Fe, Mg, Ni, Pb, Sb, Tl and Zn;
- for the verification of analytical methods for Ag, As, Ba, Cd, Co, Cu, Fe, Mg, Ni, Pb, Sb, Tl and Zn;
- for the monitoring of laboratory performance in the analysis of Ag, As, Ba, Cd, Co, Cu, Fe, Mg, Ni, Pb, Sb, Tl and Zn in geological samples.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 34i is sourced from zinc sulphide ore tailings 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 ten years.

INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 34i refer to the concentration level of Ag, As, Ba, Cd, Co, Cu, Fe, Mg, Ni, Pb, Sb, 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.

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 No	Date	Changes applied
0	7th May, 2019	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

7th May, 2019

Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager - ORE P/L

REFERENCES

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.