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CERTIFICATE OF ANALYSIS FOR

ZINC ORE
CERTIFIED REFERENCE MATERIAL
OREAS 34h

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

Constituent	Certified Value	Within-Lab SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Acid Digestions (no HF)						
Ag, Silver (ppm)	21.8	0.89	21.1	22.5	21.1	22.4
As, Arsenic (ppm)	2415	71	2359	2472	2373	2458
Ca, Calcium (wt.%)	8.08	0.272	6.87	9.30	7.98	8.18
Cd, Cadmium (ppm)	196	5	188	204	193	199
Co, Cobalt (ppm)	168	6	161	174	162	174
Cu, Copper (ppm)	628	16	606	650	616	640
Fe, Iron (wt.%)	18.89	0.486	18.39	19.38	18.63	19.15
Mg, Magnesium (wt.%)	3.75	0.088	3.63	3.87	3.69	3.81
Mn, Manganese (ppm)	405	11	364	447	398	413
Ni, Nickel (ppm)	494	16	478	511	486	502
P, Phosphorus (ppm)	336	26	292	379	314	357
Pb, Lead (wt.%)	1.46	0.042	1.44	1.49	1.44	1.49
S, Sulphur (wt.%)	26.32	0.880	25.39	27.26	25.72	26.93
Sb, Antimony (ppm)	4.08	0.55	3.23	4.92	IND	IND
Tl, Thallium (ppm)	246	16	228	265	234	258
Zn, Zinc (wt.%)	12.25	0.215	11.99	12.52	12.07	12.43
Peroxide Fusion ICP*						
Ag, Silver (ppm)	22.4	0.69	19.5	25.3	IND	IND
As, Arsenic (ppm)	2550	89	2409	2691	2462	2638
Co, Cobalt (ppm)	169	11	161	177	IND	IND
Cu, Copper (ppm)	610	16	595	626	IND	IND
Fe, Iron (wt.%)	18.73	0.546	17.94	19.52	18.20	19.26
Mg, Magnesium (wt.%)	3.87	0.178	3.62	4.11	3.81	3.93
Ni, Nickel (ppm)	483	5	447	519	IND	IND
Pb, Lead (wt.%)	1.44	0.031	1.39	1.48	1.42	1.46
Tl, Thallium (ppm)	241	22	199	283	236	247
Zn, Zinc (wt.%)	12.18	0.253	12.00	12.37	11.94	12.43

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 34h DataPack-1.0.190508_162256.xlsx' for details).

Table 2. Indicative Values for OREAS 34h.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Acid Digestions (no HF)								
Al	wt.%	*0.179*	Ga	ppm	*< 50*	Sn	ppm	*< 100*
B	ppm	*< 10*	Hg	ppm	*1.94*	Sr	ppm	*71*
Ba	ppm	*2528*	K	wt.%	*0.083*	Th	ppm	*< 100*
Be	ppm	*< 5*	La	ppm	*< 50*	Ti	wt.%	*< 0.05*
Bi	ppm	*6.82*	Mo	ppm	*18.0*	U	ppm	*< 50*
Ce	ppm	*10.0*	Na	wt.%	*0.055*	V	ppm	*5.69*
Cr	ppm	*15.9*	Sc	ppm	*< 5*	W	ppm	*< 50*
Peroxide Fusion ICP*								
Al	wt.%	*0.615*	Cr	ppm	*< 50*	S	wt.%	*30.55*
Ba	ppm	*2370*	K	wt.%	*0.313*	Sb	ppm	*4.07*
Ca	wt.%	*9.01*	Mn	ppm	*467*	Si	wt.%	*2.08*
Cd	ppm	*197*	P	ppm	*231*	Ti	wt.%	*0.036*

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 34h DataPack-1.0.190508_162256.xlsx' for details).

SOURCE MATERIAL

OREAS 34h is a zinc ore 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 34h. 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 34h DataPack-1.0.190508_162256.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

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

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

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 (see OREAS 34h DataPack.xlsx) 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* j*th raw result reported by laboratory* i;

x'_{ij} *is the* j*th transformed result reported by laboratory* i;

n_i *is the number of results reported by laboratory* i;

p *is the number of participating laboratories*;

$\bar{x}_i$ *is the raw mean for laboratory* i.

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 12.07 and 12.43 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 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. Within-Lab Performance Gates for OREAS 34h.

Constituent	Certified Value	1SD	Within-Lab Performance Gates					
			2SD		3SD		5% Interval	
			Low	High	Low	High	Low	High
Acid Digests (no HF)								
Silver, Ag (ppm)	21.8	0.9	20.0	23.6	19.1	24.5	20.7	22.9
Arsenic, As (wt.%)	0.241	0.005	0.231	0.252	0.226	0.257	0.229	0.254
Barium, Ba (ppm)	<3000	IND	IND	IND	IND	IND	IND	IND
Cadmium, Cd (ppm)	194	4	186	202	183	206	185	204
Cobalt, Co (ppm)	168	5	157	178	152	183	159	176
Copper, Cu (ppm)	623	14	596	651	583	664	592	655
Iron, Fe (wt.%)	19.1	0.3	18.5	19.7	18.2	20.0	18.2	20.1
Magnesium, Mg (wt.%)	3.74	0.08	3.59	3.90	3.51	3.98	3.56	3.93
Nickel, Ni (ppm)	494	12	469	519	456	531	469	518
Lead, Pb (wt.%)	1.45	0.04	1.38	1.52	1.35	1.56	1.38	1.53
Antimony, Sb (ppm)	4.1	0.7	2.7	5.5	2.0	6.2	3.9	4.3
Thallium, Tl (ppm)	239	9	221	258	211	267	227	251
Zinc, Zn (wt.%)	12.2	0.2	11.7	12.7	11.5	12.9	11.6	12.8
Peroxide Fusion ICP*								
Silver, Ag (ppm)	21.0	0.6	19.9	22.2	19.3	22.8	20.0	22.1
Arsenic, As (wt.%)	0.254	0.015	0.224	0.283	0.210	0.298	0.241	0.266
Barium, Ba (ppm)	2397	292	1813	2982	1521	3274	2278	2517
Cadmium, Cd (ppm)	198	2	193	203	191	205	188	208
Cobalt, Co (ppm)	174	7	160	188	153	195	165	183
Copper, Cu (ppm)	610	17	576	643	560	660	579	640
Iron, Fe (wt.%)	18.7	0.4	17.8	19.6	17.4	20.1	17.8	19.7
Magnesium, Mg (wt.%)	3.89	0.12	3.65	4.14	3.52	4.26	3.70	4.09
Nickel, Ni (ppm)	474	11	453	496	442	506	451	498
Lead, Pb (wt.%)	1.45	0.03	1.40	1.51	1.37	1.53	1.38	1.52
Antimony, Sb (ppm)	3.9	0.6	2.6	5.2	2.0	5.8	3.7	4.1
Thallium, Tl (ppm)	221	8	205	237	197	245	210	232
Zinc, Zn (wt.%)	12.18	0.20	11.78	12.58	11.58	12.78	11.57	12.79

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 34h DataPack-1.0.190508_162256.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 34h has been prepared and certified and is supplied by:

ORE Research & Exploration Pty Ltd	Tel: +613-9729 0333
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OREAS 34h 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 34h 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 34h is sourced from 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 ten years.

INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 34h 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.