

## CERTIFICATE OF ANALYSIS FOR

## ZINC CONCENTRATE

(Dugald River Zinc-Lead-Silver Mine, Cloncurry, Northern Queensland, Australia)

# CERTIFIED REFERENCE MATERIAL OREAS 354

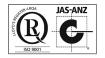
Constituent	Constituent		Absolute Standard Deviations				Relative Standard Deviations			5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Umpire Labs	(dry sample	basis)									
Classical Wet	Chemistry - \	Volumetric	: Titration								
Zn, wt.%	49.30	0.161	48.98	49.63	48.82	49.79	0.33%	0.65%	0.98%	46.84	51.77
Geoanalytical	Geoanalytical Labs ('as received' sample basis)										
Oxidising Fusio	on XRF										
Al <sub>2</sub> O <sub>3</sub> , wt.%	0.743	0.055	0.633	0.854	0.577	0.910	7.45%	14.90%	22.35%	0.706	0.781
Ba, ppm	1443	171	1101	1785	930	1956	11.86%	23.71%	35.57%	1371	1515
CaO, wt.%	0.232	0.011	0.210	0.254	0.199	0.265	4.73%	9.46%	14.18%	0.220	0.244
Cu, ppm	1380	93	1193	1567	1100	1661	6.77%	13.54%	20.31%	1311	1449
Fe, wt.%	9.65	0.110	9.43	9.87	9.32	9.98	1.14%	2.28%	3.42%	9.16	10.13
K <sub>2</sub> O, wt.%	0.440	0.012	0.416	0.465	0.404	0.477	2.75%	5.50%	8.25%	0.418	0.462
Mn, wt.%	1.52	0.029	1.46	1.58	1.44	1.61	1.88%	3.76%	5.64%	1.45	1.60
Pb, wt.%	1.55	0.091	1.37	1.73	1.28	1.82	5.86%	11.71%	17.57%	1.47	1.63
S, wt.%	31.36	0.519	30.32	32.39	29.80	32.91	1.65%	3.31%	4.96%	29.79	32.92

#### Table 1. Certified Values and Performance Gates for OREAS 354.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.



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	Certified Value		Absolute	Standard	Deviation	s	Relative Standard Deviations			5% window	
Constituent		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Geoanalytical	Labs ('as re	eceived' s	ample ba	nsis)							
Oxidising Fusio	Oxidising Fusion XRF continued										
SiO <sub>2</sub> , wt.%	3.36	0.074	3.21	3.51	3.14	3.58	2.20%	4.41%	6.61%	3.19	3.53
TiO <sub>2</sub> , wt.%	0.057	0.019	0.019	0.095	0.000	0.114	33.57%	67.15%	100.72	0.054	0.060
Zn, wt.%	49.46	0.567	48.32	50.59	47.76	51.16	1.15%	2.29%	3.44%	46.98	51.93
Infrared Combi	ustion										
C, wt.%	0.526	0.026	0.475	0.578	0.449	0.604	4.90%	9.80%	14.69%	0.500	0.553
C-(Graphite), wt.%	0.414	0.035	0.344	0.485	0.308	0.520	8.53%	17.05%	25.58%	0.394	0.435
S, wt.%	31.49	0.808	29.88	33.11	29.07	33.92	2.56%	5.13%	7.69%	29.92	33.07
4-Acid Digestic	on										
Ag, ppm	98.0	4.24	89.5	106.5	85.3	110.7	4.33%	8.66%	12.98%	93.1	102.9
Al, wt.%	0.411	0.021	0.369	0.454	0.348	0.475	5.17%	10.34%	15.51%	0.391	0.432
Be, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	5.60	0.499	4.60	6.60	4.10	7.10	8.92%	17.83%	26.75%	5.32	5.88
Ca, wt.%	0.165	0.007	0.150	0.180	0.143	0.187	4.48%	8.97%	13.45%	0.157	0.173
Cd, ppm	1157	35	1086	1228	1050	1263	3.07%	6.14%	9.20%	1099	1214
Co, ppm	9.56	0.622	8.31	10.80	7.69	11.42	6.51%	13.02%	19.52%	9.08	10.03
Cr, ppm	6.74	1.41	3.93	9.55	2.52	10.95	20.85%	41.71%	62.56%	6.40	7.08
Cu, ppm	1387	49	1289	1486	1240	1535	3.55%	7.11%	10.66%	1318	1457
Fe, wt.%	9.82	0.330	9.16	10.48	8.83	10.81	3.36%	6.72%	10.08%	9.33	10.31
Ga, ppm	9.85	0.351	9.14	10.55	8.79	10.90	3.56%	7.13%	10.69%	9.35	10.34
K, wt.%	0.354	0.017	0.319	0.389	0.302	0.406	4.91%	9.82%	14.74%	0.336	0.371
Mg, wt.%	0.054	0.006	0.043	0.065	0.037	0.070	10.28%	20.55%	30.83%	0.051	0.056
Mn, wt.%	1.54	0.074	1.39	1.69	1.32	1.76	4.83%	9.66%	14.48%	1.46	1.62
Mo, ppm	2.37	0.24	1.90	2.85	1.66	3.09	10.06%	20.13%	30.19%	2.26	2.49
Na, wt.%	0.019	0.003	0.013	0.025	0.010	0.028	15.66%	31.32%	46.99%	0.018	0.020
Nb, ppm	1.06	0.31	0.44	1.69	0.13	2.00	29.25%	58.49%	87.74%	1.01	1.12
Ni, ppm	10.9	1.4	8.2	13.6	6.8	15.0	12.48%	24.97%	37.45%	10.3	11.4
P, wt.%	0.006	0.001	0.005	0.007	0.004	0.008	10.72%	21.43%	32.15%	0.006	0.006
Pb, wt.%	1.58	0.071	1.43	1.72	1.36	1.79	4.53%	9.06%	13.58%	1.50	1.65
S, wt.%	26.63	4.09	18.45	34.80	14.37	38.89	15.35%	30.70%	46.05%	25.30	27.96
Sb, ppm	51	8	34	67	26	76	16.36%	32.71%	49.07%	48	53
Sc, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sn, ppm	3.83	0.74	2.36	5.30	1.63	6.04	19.19%	38.38%	57.57%	3.64	4.03
Sr, ppm	42.6	4.6	33.5	51.8	28.9	56.3	10.74%	21.48%	32.21%	40.5	44.7
Ti, wt.%	0.025	0.004	0.018	0.032	0.014	0.036	14.24%	28.47%	42.71%	0.024	0.026
U, ppm	4.42	0.411	3.59	5.24	3.18	5.65	9.31%	18.62%	27.93%	4.20	4.64
V, ppm	9.12	1.51	6.11	12.13	4.60	13.64	16.52%	33.03%	49.55%	8.67	9.58
Y, ppm	2.99	0.230	2.53	3.45	2.30	3.68	7.68%	15.36%	23.04%	2.84	3.14
Zn, wt.%	49.77	2.271	45.23	54.31	42.96	56.58	4.56%	9.12%	13.69%	47.28	52.26
Zr, ppm	9.95	1.17	7.62	12.29	6.45	13.45	11.73%	23.47%	35.20%	9.45	10.45

#### Table 1 continued.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.



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## INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological and metallurgical 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 354 is a zinc concentrate certified reference material (CRM) prepared and certified by Ore Research & Exploration Pty Ltd. The material constituting OREAS 354 was sourced from zinc sulphide concentrate samples taken from the Dugald River metallurgical plant. The Dugald River deposit is located in the Mt Isa Inlier, ~65km northwest of Cloncurry in north-west Queensland, Australia. The mineralisation style is dominated by sphalerite and galena with a gangue of graphitic slate, pyrrhotite and pyrite. The deposit is hosted within a sequence of upper greenschist to amphibolite facies metamorphic rocks consisting quartzite, schists, slates and dolomite.

### **PERFORMANCE GATES**

The standard deviations (SD's) reported in Table 1 provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors (see 'Homogeneity Evaluation' for verification of OREAS 354's high level of homogeneity).

Table 1 above shows intervals 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 (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit <u>www.westgard.com/mltirule.htm</u>). A second method utilises a 5% window calculated directly from the certified value. For information on the calculation of standard deviations see the 'Statistical Analysis' section below.

Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL)  $\pm$  10%.

*i.e.* Certified Value ± 10% ± 2DL (adapted from Govett, 1983).



## **COMMINUTION AND HOMOGENISATION PROCEDURES**

The material constituting OREAS 354 was prepared in the following manner:

- Drying of ore materials to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging into 10g units sealed under nitrogen in laminated foil pouches.

## ANALYTICAL PROGRAM

For the interlaboratory 'round robin' certification program, samples were taken at 10 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire prepared batch of OREAS 354.

Thirteen 'umpire' laboratories employing classical wet chemistry methods and twelve other mineral laboratories employing instrumental methods participated in the round robin certification program to certify the analytes reported in Table 1.

#### Umpire Laboratories

Thirteen umpire laboratories each received a 30g sample and employed the following methods:

- Zinc was determined in 3 trials by classical wet chemistry with titration finish (13 laboratories);
- Moisture (H<sub>2</sub>O-) at 105°C by thermogravimetry (16 laboratories).

The umpire laboratories were given strict pre-assay sample instructions relating to moisture correction. These instructions included:

- Equilibration of sample material to lab atmosphere for a minimum of 2 hours;
- Hygroscopic moisture analysis at 105°C determined on a separate subsample <u>and</u> weighed for analysis at the same time as the sample aliquots for Zn as per ISO <u>9599.</u>

The umpire laboratories were also requested to report metal concentrations on both a dry and moisture-bearing basis and include all results for moisture determinations. A value for moisture ( $H_2O_-$ ) is provided in the Indicative Values table for informational purposes only (see Table 2). **The certified value for zinc is on a dry sample basis**.

#### Geoanalytical Laboratories

Twelve geoanalytical laboratories also participated in the analytical program for OREAS 354. Each laboratory was sent 6 x 20g samples to undertake the following:

- Al<sub>2</sub>O<sub>3</sub>, Ba, CaO, Cu, Fe, K<sub>2</sub>O, MgO, Mn, P<sub>2</sub>O<sub>5</sub>, Pb, S, SiO<sub>2</sub>, TiO<sub>2</sub> & Zn by oxidising fusion with XRF finish (up to 12 laboratories depending on the element);
- Full elemental suites by 4-acid digestion with ICP-OES and MS finish (up to 11 laboratories depending on the element);
- Total C and S by IR combustion furnace (10 laboratories);



• Graphitic Carbon by HCI leach, with IR induction furnace finish (9 laboratories).

Table 1 provides performance gate intervals for the certified values and Table 2 shows indicative values. Table 3 provides some indicative physical properties and Table 4 presents the 95% confidence and tolerance limits for all certified values. 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<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 354-DataPack.1.0.201108\_193609.xlsx**).

Comparisons of inter-laboratory bias and precision are graphically presented in scatter plots for Zinc by classical titration method by the umpire laboratories and Zinc by oxidising fusion XRF by geoanalytical laboratories (Figures 1 and 2, respectively) together with ±3SD (magenta) and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Oxidising Fus	ion XRF	-						
Со	ppm	< 100	MgO	wt.%	0.090			
Cr <sub>2</sub> O <sub>3</sub>	ppm	< 100	P <sub>2</sub> O <sub>5</sub>	wt.%	0.031			
Thermogravimetry								
LOI <sup>1000</sup>	wt.%	15.06	H <sub>2</sub> O-	wt.%	0.379			
4-Acid Digest	ion							
As	ppm	14.9	Но	ppm	0.12	Sm	ppm	0.74
Ва	ppm	490	In	ppm	2.29	Та	ppm	0.27
Ce	ppm	6.03	La	ppm	2.69	Tb	ppm	0.11
Cs	ppm	0.31	Li	ppm	3.47	Те	ppm	0.93
Dy	ppm	0.54	Lu	ppm	0.040	Th	ppm	1.73
Er	ppm	0.30	Nd	ppm	3.31	TI	ppm	6.21
Eu	ppm	0.17	Pr	ppm	0.92	Tm	ppm	0.065
Gd	ppm	0.63	Rb	ppm	13.8	W	ppm	1.27
Ge	ppm	< 5	Re	ppm	< 0.1	Yb	ppm	0.34
Hf	ppm	0.40	Se	ppm	22.7			

Table 2. Indicative Values for OREAS 354.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 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.

## PHYSICAL PROPERTIES

OREAS 354 was tested at ORE Research & Exploration Pty Ltd's onsite laboratory for various physical properties. Table 3 presents these findings which should be used for informational purposes only.

CRM Name	Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
OREAS 354	911	0.77	10YR 4/2	Dark Yellowish Brown

<sup>‡</sup>The Munsell Rock Color Chart helps geologists and archeologists communicate with color more effectively by crossreferencing ISCC-NBS color names with unique Munsell alpha-numeric color notations for rock color samples.



## STATISTICAL ANALYSIS

**Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits** (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances, statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if >2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

**Certified Values** are the means of accepted laboratory means after outlier filtering. Indicative (uncertified) values (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) inter-laboratory consensus is poor; or iii) a significant proportion of results are outlying.

**95% Confidence Limits** are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. *95% Confidence Limits should not be used as control limits for laboratory performance.* 

**Standard Deviation** values (1SDs) are reported in Table 1 and provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. The SD values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e. after removal of any individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-laboratory



bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

Constituent	Certified	95% Confid	lence Limits	95% Tolerance Limits		
Constituent	Value	Low	High	Low	High	
Umpire Labs (dry sample basis)						
Classical Wet Chemistry - Volumetric Titration						
Zn, Zinc (wt.%)	49.30	49.19	49.42	49.26	49.35	
Geoanalytical Labs ('as received' sar	nple basis)					
Oxidising Fusion XRF						
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	0.743	0.700	0.787	0.717	0.769	
Ba, Barium (ppm)	1443	1321	1565	IND	IND	
CaO, Calcium oxide (wt.%)	0.232	0.225	0.239	0.220	0.244	
Cu, Copper (ppm)	1380	1316	1444	IND	IND	
Fe, Iron (wt.%)	9.65	9.57	9.72	9.55	9.74	
K <sub>2</sub> O, Potassium oxide (wt.%)	0.440	0.432	0.449	0.430	0.451	
Mn, Manganese (wt.%)	1.52	1.50	1.54	1.51	1.54	
Pb, Lead (wt.%)	1.55	1.49	1.61	1.47	1.63	
S, Sulphur (wt.%)	31.36	30.83	31.88	30.80	31.92	
SiO <sub>2</sub> , Silicon dioxide (wt.%)	3.36	3.30	3.42	3.26	3.46	
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.057	0.043	0.071	IND	IND	
Zn, Zinc (wt.%)	49.46	49.09	49.82	49.09	49.82	
Infrared Combustion						
C, Carbon (wt.%)	0.526	0.518	0.535	0.499	0.554	
C-(Graphite), Carbon, graphitic (wt.%)	0.414	0.388	0.440	0.366	0.462	
S, Sulphur (wt.%)	31.49	31.05	31.94	30.97	32.01	
4-Acid Digestion						
Ag, Silver (ppm)	98.0	95.4	100.6	96.0	100.0	
Al, Aluminium (wt.%)	0.411	0.396	0.426	0.401	0.422	
Be, Beryllium (ppm)	< 0.5	IND	IND	IND	IND	
Bi, Bismuth (ppm)	5.60	5.08	6.12	5.36	5.84	
Ca, Calcium (wt.%)	0.165	0.160	0.169	0.160	0.170	
Cd, Cadmium (ppm)	1157	1130	1183	1139	1174	
Co, Cobalt (ppm)	9.56	8.98	10.13	IND	IND	
Cr, Chromium (ppm)	6.74	5.27	8.20	IND	IND	
Cu, Copper (ppm)	1387	1356	1419	1357	1418	
Fe, Iron (wt.%)	9.82	9.60	10.03	9.65	9.98	
Ga, Gallium (ppm)	9.85	9.52	10.17	IND	IND	
K, Potassium (wt.%)	0.354	0.341	0.366	0.345	0.362	
Mg, Magnesium (wt.%)	0.054	0.050	0.057	0.052	0.056	
Mn, Manganese (wt.%)	1.54	1.49	1.59	1.50	1.58	
Mo, Molybdenum (ppm)	2.37	2.15	2.60	2.03	2.71	
Na, Sodium (wt.%)	0.019	0.017	0.021	IND	IND	
Nb, Niobium (ppm)	1.06	0.66	1.47	IND	IND	

 Table 4. 95% Confidence & Tolerance Limits for OREAS 354.

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note 1: intervals may appear asymmetric due to rounding.



lable 4 continued.							
Constituent	Certified	95% Confid	lence Limits	95% Tolera	ance Limits		
Constituent	Value	Low	High	Low	High		
4-Acid Digestion continued							
Ni, Nickel (ppm)	10.9	10.1	11.7	IND	IND		
P, Phosphorus (wt.%)	0.006	0.005	0.007	IND	IND		
Pb, Lead (wt.%)	1.58	1.54	1.61	1.51	1.64		
S, Sulphur (wt.%)	26.63	23.36	29.90	26.14	27.12		
Sb, Antimony (ppm)	51	44	57	47	55		
Sc, Scandium (ppm)	< 1	IND	IND	IND	IND		
Sn, Tin (ppm)	3.83	3.03	4.64	IND	IND		
Sr, Strontium (ppm)	42.6	39.4	45.8	41.2	44.1		
Ti, Titanium (wt.%)	0.025	0.022	0.027	0.024	0.026		
U, Uranium (ppm)	4.42	3.88	4.95	4.25	4.58		
V, Vanadium (ppm)	9.12	8.00	10.24	IND	IND		
Y, Yttrium (ppm)	2.99	2.71	3.27	2.81	3.17		
Zn, Zinc (wt.%)	49.77	46.72	52.82	49.23	50.31		
Zr, Zirconium (ppm)	9.95	8.44	11.46	IND	IND		

Table 4 continued.

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note 1: intervals may appear asymmetric due to rounding.

#### **Homogeneity Evaluation**

The tolerance limits (ISO 16269:2014) shown in Table 1 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for Zn by the umpire laboratories employing classical wet chemistry with titration finish, where 99% of the time  $(1-\alpha=0.99)$  at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 49.26 and 49.35 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). *Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.* 

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 354 is fit-for-purpose as a certified reference material (see 'Intended Use' below).



## PARTICIPATING LABORATORIES

- 1. \*AH Knight, St Helens, Merseyside, UK
- 2. •AH Knight, Tianjin, China
- 3. \*ALS, Brisbane, QLD, Australia
- 4. \*\*ALS, Burnie, TAS, Australia
- 5. \*ALS, Loughrea, Galway, Ireland
- 6. \*ALS, Vancouver, BC, Canada
- 7. \*ALS Inspection, Prescot, Merseyside, UK
- 8. \*Bachelet, Angleur, Liege, Belgium
- 9. \*Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 10. \*Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 11. \*Bureau Veritas Geoanalytical, Perth, WA, Australia
- 12. Independent, Perth, WA, Australia
- 13. Inspectorate (BV), Lima, Peru
- 14. Inspectorate (BV), Shanghai, Bao Shan District, China
- 15. Inspectorate (BV), Witham, Essex, UK
- 16. <sup>\*</sup>Intertek Genalysis, Perth, WA, Australia
- 17. \*Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
- 18. \*Nagrom, Perth, WA, Australia
- 19. \*SGS Australia Mineral Services, Perth, WA, Australia
- 20. \*\*SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 21. \*SGS Nederland B.V., Spijkenisse, Zuid-Holland, Netherlands
- 22. \*\*Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 23. \*SRL, Perth, WA, Australia

♦= Umpire laboratory (classical methods); \* = Geoanalytical laboratory (instrumental methods).

# Please note: Above numbered alphabetical list of participating laboratories <u>does not</u> reflect the Lab ID numbering on the scatter plots below.

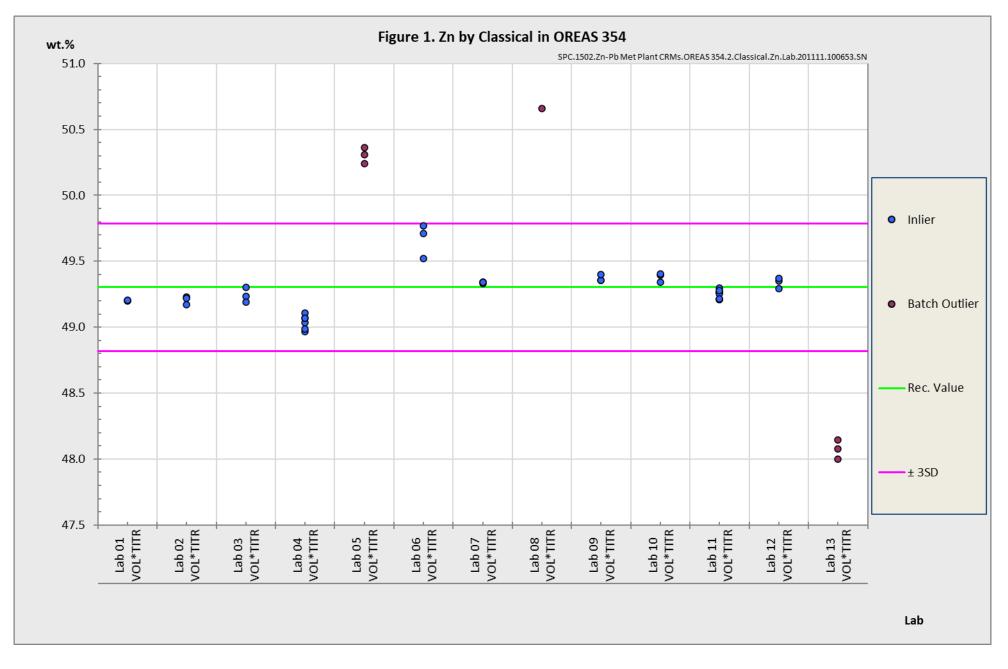
## PREPARER AND SUPPLIER

Certified reference material OREAS 354 was prepared, certified and supplied by:

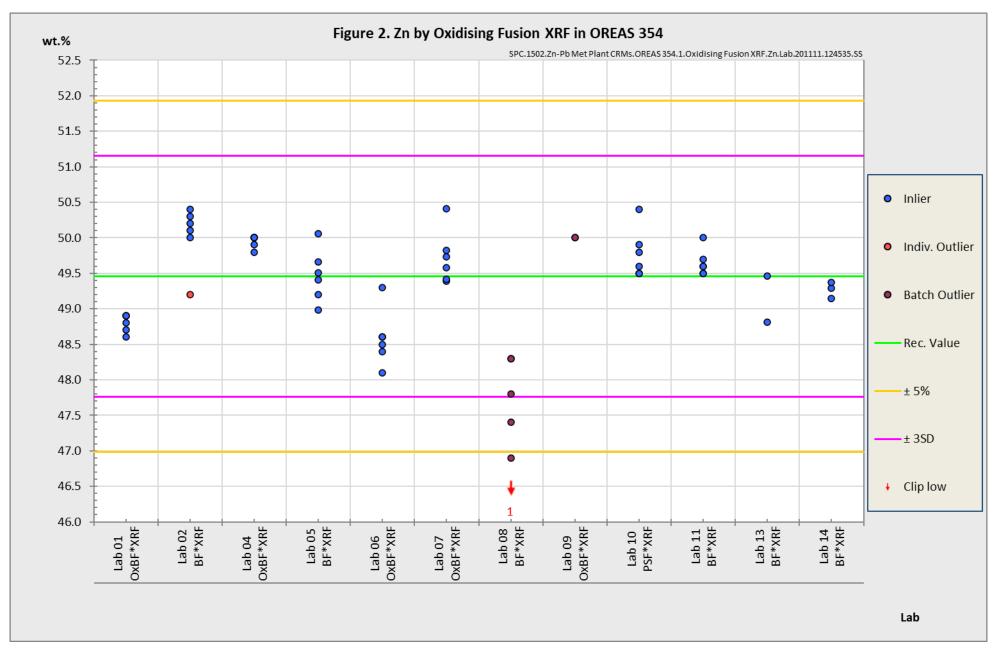


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## METROLOGICAL TRACEABILITY

The analytical samples were selected in a manner representative of the entire batch of the 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 procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 35:2017, 9.2.4c)." Certification takes place on the basis of agreement among operationally defined, independent measurement results.* 

## 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/concentrate samples 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 354 is intended to cover all activities needed to produce a measurement result. This includes extraction, possible separation steps and the actual measurement process (the signal producing step). OREAS 354 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 354 is intended for the following uses:



- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in sulphide concentrate samples;
- For the verification of analytical methods for analytes reported in Table 1;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Table 1.

## HANDLING INSTRUCTIONS

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised. Sulphur is a known transitory upper respiratory irritant. Close exposure may cause coughing or throat irritation.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 354 is a sulphide-rich reference material (S = 26.6%) and is reactive under normal atmospheric conditions. To inhibit oxidation and prolong its shelf life it has been sealed under nitrogen in single-use, robust laminated foil pouches. In its unopened state under normal conditions of storage it has a shelf life beyond five years

\*normal storage conditions: not in direct sunlight in a dry, clean, well ventilated area at temperatures between -10° and 50°C.

## INSTRUCTIONS FOR CORRECT USE

The certified value for Zn by volumetric titration is on a dry sample basis. There was considerable variation in moisture content reported by the laboratories (lab means varied from 0.14 to 0.58 wt.% H<sub>2</sub>O-) and this can be a significant source of error if not properly controlled. Therefore, all analyses were performed on the samples after equilibration with the laboratory atmosphere for a minimum of 2 hours and hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquot for Zn as per ISO 9599. If the reference material is not dried prior to analysis, the EDTA with titration certified value for Zn should be corrected to the moisture-bearing basis.

As per routine analysis at commercial laboratories, the certified values derived by oxidising fusion with XRF finish are on a dry sample basis.

All other methods (4-acid digestion with ICP-OES and MS finish and infrared combustion furnace for C and S) refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis for these methods.

#### QC monitoring using multiples of the Standard Deviation (SD)

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.



The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

## DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	19 <sup>th</sup> Nov, 2020	First publication.

## **QMS CERTIFICATION**

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



## **CERTIFYING OFFICER**

19<sup>th</sup> November, 2020

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

## REFERENCES

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