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**CERTIFICATE OF ANALYSIS FOR**

**Lead Concentrate**

**(Cannington Mine, North West Queensland, Australia)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 353**

**Table 1. Certified Values and Performance Gates for OREAS 353.**

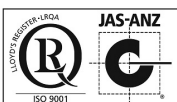
Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Umpire Labs (dry sample basis)</b>											
<b>Acid Digestion Titration</b>											
Pb, wt.%	61.26	0.117	61.03	61.50	60.91	61.61	0.19%	0.38%	0.57%	58.20	64.33
<b>Fire Assay (Gravimetric)</b>											
Ag, ppm	2184	17	2151	2218	2134	2234	0.77%	1.53%	2.30%	2075	2293

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

Note 1: SD's are based on the pooled Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

Note 3: 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.



Document: COA-1470-OREAS353-R0 (Template: BUP-70-10-01 Rev:2.0)	28-May-2020
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**Table 1 continued.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Geoanalytical Labs ('as received' sample basis)</b>											
<b>4-Acid Digestion</b>											
Al, wt. %	0.204	0.010	0.184	0.224	0.174	0.234	4.84%	9.69%	14.53%	0.194	0.214
As, ppm	490	53	384	595	332	647	10.73%	21.47%	32.20%	465	514
Ba, ppm	25.0	3.2	18.6	31.4	15.4	34.7	12.85%	25.71%	38.56%	23.8	26.3
Be, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	32.1	1.94	28.2	36.0	26.3	37.9	6.04%	12.07%	18.11%	30.5	33.7
Ca, wt. %	0.236	0.010	0.215	0.257	0.205	0.267	4.39%	8.78%	13.16%	0.224	0.248
Cd, ppm	205	7	192	218	185	225	3.26%	6.51%	9.77%	195	215
Ce, ppm	14.0	1.21	11.5	16.4	10.3	17.6	8.69%	17.39%	26.08%	13.3	14.7
Co, ppm	48.8	4.10	40.6	57.0	36.5	61.1	8.40%	16.79%	25.19%	46.4	51.3
Cr, ppm	136	7	122	149	115	156	5.07%	10.15%	15.22%	129	142
Cs, ppm	0.19	0.010	0.17	0.21	0.16	0.22	4.93%	9.87%	14.80%	0.18	0.20
Cu, wt. %	0.312	0.009	0.294	0.331	0.284	0.340	2.98%	5.97%	8.95%	0.297	0.328
Dy, ppm	0.77	0.13	0.51	1.04	0.37	1.17	17.18%	34.36%	51.55%	0.73	0.81
Eu, ppm	1.63	0.21	1.21	2.06	0.99	2.28	13.06%	26.12%	39.18%	1.55	1.72
Fe, wt. %	5.35	0.174	5.00	5.70	4.83	5.87	3.25%	6.50%	9.74%	5.08	5.62
Ga, ppm	1.35	0.19	0.98	1.72	0.79	1.91	13.82%	27.64%	41.45%	1.28	1.42
Gd, ppm	1.01	0.13	0.75	1.26	0.62	1.39	12.73%	25.46%	38.19%	0.96	1.06
In, ppm	2.01	0.22	1.56	2.46	1.34	2.68	11.14%	22.29%	33.43%	1.91	2.11
K, wt. %	0.045	0.005	0.034	0.055	0.029	0.061	11.79%	23.57%	35.36%	0.043	0.047
La, ppm	7.91	0.746	6.42	9.40	5.67	10.15	9.44%	18.87%	28.31%	7.51	8.30
Li, ppm	4.27	0.76	2.75	5.80	1.98	6.57	17.88%	35.76%	53.63%	4.06	4.49
Mg, wt. %	0.685	0.029	0.628	0.742	0.599	0.771	4.19%	8.38%	12.57%	0.651	0.719
Mn, wt. %	0.184	0.006	0.172	0.195	0.166	0.201	3.15%	6.31%	9.46%	0.175	0.193
Mo, ppm	18.4	1.25	15.9	20.9	14.6	22.1	6.82%	13.64%	20.46%	17.5	19.3
Na, wt. %	0.028	0.004	0.020	0.035	0.016	0.039	14.03%	28.05%	42.08%	0.026	0.029
Nb, ppm	0.80	0.13	0.54	1.06	0.41	1.19	16.38%	32.75%	49.13%	0.76	0.84
Nd, ppm	5.29	0.483	4.32	6.25	3.84	6.74	9.15%	18.29%	27.44%	5.02	5.55
Ni, ppm	38.4	3.12	32.2	44.7	29.1	47.8	8.13%	16.25%	24.38%	36.5	40.4
P, wt. %	0.024	0.001	0.022	0.027	0.020	0.029	5.56%	11.11%	16.67%	0.023	0.026
Pr, ppm	1.43	0.18	1.07	1.79	0.89	1.97	12.58%	25.16%	37.74%	1.36	1.50
Rb, ppm	2.06	0.139	1.78	2.34	1.65	2.48	6.74%	13.49%	20.23%	1.96	2.17
S, wt. %	15.13	0.834	13.46	16.80	12.63	17.63	5.51%	11.02%	16.53%	14.37	15.88
Sb, ppm	1746	133	1480	2012	1347	2145	7.61%	15.22%	22.83%	1659	1833
Se, ppm	4.95	0.480	3.99	5.91	3.51	6.39	9.69%	19.38%	29.08%	4.70	5.20
Sm, ppm	1.10	0.23	0.65	1.56	0.42	1.79	20.77%	41.55%	62.32%	1.05	1.16
Sn, ppm	7.67	0.98	5.72	9.63	4.75	10.60	12.72%	25.44%	38.17%	7.29	8.06
Sr, ppm	10.8	1.1	8.6	12.9	7.5	14.0	10.11%	20.22%	30.33%	10.2	11.3
Th, ppm	0.85	0.09	0.67	1.03	0.58	1.12	10.57%	21.14%	31.70%	0.81	0.89

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

Note 1: SD's are based on the pooled Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

Note 3: 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.

**Table 1 continued.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>4-Acid Digestion continued</b>											
Ti, wt. %	0.013	0.002	0.008	0.017	0.006	0.019	17.93%	35.86%	53.79%	0.012	0.013
Tl, ppm	0.83	0.09	0.65	1.00	0.56	1.09	10.60%	21.20%	31.80%	0.79	0.87
U, ppm	1.79	0.125	1.54	2.05	1.42	2.17	7.00%	13.99%	20.99%	1.70	1.88
V, ppm	10.6	0.68	9.2	11.9	8.6	12.6	6.39%	12.77%	19.16%	10.1	11.1
W, ppm	1.81	0.125	1.56	2.06	1.43	2.19	6.93%	13.86%	20.79%	1.72	1.90
Y, ppm	4.32	0.53	3.25	5.38	2.72	5.91	12.32%	24.65%	36.97%	4.10	4.53
Zn, wt. %	4.13	0.130	3.87	4.39	3.74	4.52	3.14%	6.28%	9.42%	3.93	4.34
Zr, ppm	3.91	0.97	1.96	5.86	0.99	6.83	24.89%	49.77%	74.66%	3.72	4.11
<b>Peroxide Fusion ICP</b>											
Al, wt. %	0.206	0.013	0.180	0.232	0.167	0.245	6.32%	12.65%	18.97%	0.196	0.217
As, ppm	538	33	472	604	440	637	6.10%	12.21%	18.31%	511	565
Ca, wt. %	0.255	0.052	0.150	0.359	0.098	0.412	20.51%	41.02%	61.54%	0.242	0.268
Co, ppm	52	4.4	44	61	39	66	8.47%	16.94%	25.41%	50	55
Cr, ppm	169	33	103	235	70	267	19.49%	38.99%	58.48%	160	177
Fe, wt. %	5.44	0.100	5.24	5.64	5.14	5.74	1.84%	3.67%	5.51%	5.17	5.71
Mg, wt. %	0.704	0.017	0.670	0.737	0.654	0.753	2.36%	4.72%	7.07%	0.668	0.739
Mn, wt. %	0.186	0.004	0.177	0.195	0.173	0.199	2.39%	4.77%	7.16%	0.177	0.195
S, wt. %	16.08	0.542	15.00	17.16	14.45	17.71	3.37%	6.74%	10.11%	15.28	16.88
Sb, ppm	1827	67	1693	1962	1626	2029	3.68%	7.36%	11.04%	1736	1919
SiO <sub>2</sub> , wt. %	5.14	0.219	4.70	5.57	4.48	5.79	4.26%	8.52%	12.78%	4.88	5.39
Zn, wt. %	4.14	0.101	3.93	4.34	3.83	4.44	2.44%	4.87%	7.31%	3.93	4.34
<b>Infrared Combustion</b>											
C, wt. %	0.766	0.047	0.673	0.860	0.626	0.906	6.09%	12.17%	18.26%	0.728	0.805
S, wt. %	15.13	0.719	13.69	16.57	12.97	17.29	4.76%	9.51%	14.27%	14.37	15.88

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

Note 1: SD's are based on the pooled Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

Note 3: 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 sulphide concentrate 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 353 is a certified reference material (CRM) prepared from a Pb-Ag concentrate sample sourced from South32 Ltd's Cannington mine. The stratabound, metasediment hosted (Broken Hill Type) deposit is located ~200 kms south-east of Mount Isa in north-west Queensland, Australia. The major sulphides occur as argentiferous galena and sphalerite with subordinate magnetite-pyrrhotite and minor marcasite and arsenopyrite-lollingite-chalcopyrite.

## 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 353'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 [www.westgard.com/mltirule.htm](http://www.westgard.com/mltirule.htm)). 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  $\pm$  10%  $\pm$  2DL (adapted from Govett, 1983).*

## COMMINATION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 353 was prepared in the following manner:

- Drying of material to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging under nitrogen in 10g and 50g units in laminated foil pouches.

## ANALYTICAL PROGRAM

For the interlaboratory 'round robin' certification program, six 800g test units were taken at predetermined sampling intervals immediately following homogenisation and are considered representative of the entire prepared batch of OREAS 353.

### Umpire Laboratories

Twelve 'umpire' laboratories were used to determine the key elements: Pb and Ag. The term 'umpire' here refers to the routine analysis of these laboratories using classical methodologies for precious and base metals.

The umpire laboratories each received a single 110g sample and employed the following methods:

- Lead was determined in 3 trials by ethylenediaminetetraacetic acid (EDTA) with titration finish (12 laboratories);
- Silver by reduced charge, fire assay with gravimetric finish (12 laboratories);
- Moisture (H<sub>2</sub>O-) at 105°C by thermogravimetry (11 laboratories).

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

- Equilibration of sample material to laboratory atmosphere for a minimum of 2 hours;
- Hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Pb and Ag as per ISO 9599.

The umpire laboratories were requested to report analyte concentrations on both a dry (using the moisture value to correct the sample to dry basis) and moisture-bearing basis and include all results for moisture determinations. **The 'Umpire Lab' certified values shown in Table 1 are on a dry sample basis (see 'Instructions for correct use' section).**

### Geoanalytical Laboratories

Thirteen geoanalytical laboratories also participated in the analytical program. Each laboratory was sent 6 x 30g samples to undertake the following:

- 4-acid digestion (HF-HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) with ICP-OES, ICP-MS and/or AAS finish (up to 19 laboratories depending on the element);
- Peroxide fusion with ICP-OES and/or ICP-MS finish (up to 12 laboratories depending on the element);
- Infrared combustion furnace for C (12 laboratories) and S (13 laboratories).

The six samples received by each geoanalytical laboratory were obtained by taking two 30g scoop splits from each of three separate 800g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance (see 'Homogeneity Evaluation' section below).

**Table 2. Indicative Values for OREAS 353.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Thermogravimetry</b>								
H <sub>2</sub> O-	wt.%	0.380						
<b>Borate Fusion XRF</b>								
Pb	wt.%	61.40						
<b>4-Acid Digestion</b>								
Ag	ppm	2146	Ho	ppm	0.14	Ta	ppm	< 0.5
Er	ppm	0.40	Lu	ppm	0.029	Tb	ppm	0.13
Ge	ppm	0.28	Pb	wt.%	58.82	Te	ppm	0.49
Hf	ppm	0.11	Re	ppm	0.003	Tm	ppm	0.057
Hg	ppm	9.03	Sc	ppm	< 1	Yb	ppm	0.30
<b>Peroxide Fusion ICP</b>								
Ag	ppm	2167	In	ppm	2.21	Se	ppm	12.3
B	ppm	< 50	K	wt.%	0.086	Sm	ppm	1.17
Ba	ppm	25.9	La	ppm	8.17	Sn	ppm	< 100
Be	ppm	< 1	Li	ppm	4.67	Sr	ppm	< 20
Bi	ppm	32.0	Lu	ppm	0.13	Ta	ppm	0.17
Cd	ppm	210	Mo	ppm	22.3	Tb	ppm	0.20
Ce	ppm	11.8	Na	wt.%	0.073	Te	ppm	< 1
Cs	ppm	0.34	Nb	ppm	12.5	Th	ppm	0.89
Cu	wt.%	0.320	Nd	ppm	5.50	Ti	wt.%	0.016
Dy	ppm	1.00	Ni	ppm	46.6	Tl	ppm	0.86
Er	ppm	0.43	P	wt.%	0.032	Tm	ppm	< 0.1
Eu	ppm	1.75	Pb	wt.%	60.90	U	ppm	1.86
Ga	ppm	1.63	Pr	ppm	1.50	V	ppm	< 100
Gd	ppm	1.00	Rb	ppm	2.11	W	ppm	2.92
Ge	ppm	4.75	Re	ppm	< 0.1	Y	ppm	4.52
Ho	ppm	0.092	Sc	ppm	< 5	Yb	ppm	0.30

SI unit equivalents: ppm (parts per million) ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 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.

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 (PDM3) are presented in the detailed certification data for this CRM (**OREAS 353-DataPack.1.0.200527\_192527.xlsx**).

Results are also presented in scatter plots for Pb by EDTA titration and Ag by fire assay with gravimetric finish in Figures 1 and 2 respectively, together with ±3SD (magenta) and ±5% (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

## PHYSICAL PROPERTIES

OREAS 353 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.

**Table 3. Physical properties of OREAS 353.**

CRM Name	Bulk Density (g/L)	Moisture%	Munsell Notation‡	Munsell Color‡
OREAS 353	955.8	0.74	N3	Dark Gray

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

## STATISTICAL ANALYSIS

**Standard Deviation** intervals (see 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. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

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 (see Intended Use section for more detail).

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 all 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.***

**Certified Values, Confidence Limits and Tolerance Limits** (Table 4) 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.

**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.**

**Indicative (uncertified) values** (Table 2) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where inter-laboratory consensus is poor.

**Table 4. 95% Confidence & Tolerance Limits for OREAS 353.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Umpire Labs (dry sample basis)</b>					
<b>Classical Wet Chemistry</b>					
Pb, Lead (wt.%)	61.26	61.19	61.34	61.22	61.31
<b>Fire Assay (Gravimetric)</b>					
Ag, Silver (ppm)	2184	2173	2196	2176	2192
<b>Geoanalytical Labs ('as received' sample basis)</b>					
<b>4-Acid Digestion</b>					
Al, Aluminium (wt.%)	0.204	0.197	0.211	0.198	0.210
As, Arsenic (ppm)	490	455	524	468	511
Ba, Barium (ppm)	25.0	22.3	27.7	23.6	26.4
Be, Beryllium (ppm)	< 0.5	IND	IND	IND	IND
Bi, Bismuth (ppm)	32.1	30.9	33.3	31.4	32.8
Ca, Calcium (wt.%)	0.236	0.230	0.242	0.226	0.245
Cd, Cadmium (ppm)	205	201	209	199	211
Ce, Cerium (ppm)	14.0	13.0	15.0	13.5	14.4
Co, Cobalt (ppm)	48.8	46.4	51.3	46.2	51.5
Cr, Chromium (ppm)	136	131	140	131	140
Cs, Caesium (ppm)	0.19	0.18	0.20	IND	IND
Cu, Copper (wt.%)	0.312	0.307	0.318	0.305	0.320
Dy, Dysprosium (ppm)	0.77	0.63	0.91	IND	IND
Eu, Europium (ppm)	1.63	1.42	1.85	IND	IND
Fe, Iron (wt.%)	5.35	5.25	5.45	5.24	5.46
Ga, Gallium (ppm)	1.35	1.13	1.57	1.23	1.47
Gd, Gadolinium (ppm)	1.01	0.90	1.11	IND	IND
In, Indium (ppm)	2.01	1.85	2.17	1.92	2.10
K, Potassium (wt.%)	0.045	0.042	0.048	0.041	0.048
La, Lanthanum (ppm)	7.91	7.38	8.44	7.61	8.20
Li, Lithium (ppm)	4.27	3.43	5.12	4.05	4.50
Mg, Magnesium (wt.%)	0.685	0.667	0.703	0.669	0.701
Mn, Manganese (wt.%)	0.184	0.180	0.187	0.181	0.187

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

Note: intervals may appear asymmetric due to rounding.

Table 4 continued.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
Mo, Molybdenum (ppm)	18.4	17.4	19.3	17.8	19.0
Na, Sodium (wt.%)	0.028	0.025	0.030	IND	IND
Nb, Niobium (ppm)	0.80	0.69	0.91	0.76	0.83
Nd, Neodymium (ppm)	5.29	4.72	5.85	4.94	5.63
Ni, Nickel (ppm)	38.4	36.6	40.2	37.0	39.9
P, Phosphorus (wt.%)	0.024	0.024	0.025	0.023	0.026
Pr, Praseodymium (ppm)	1.43	1.24	1.61	IND	IND
Rb, Rubidium (ppm)	2.06	1.93	2.19	1.88	2.24
S, Sulphur (wt.%)	15.13	14.45	15.81	14.74	15.52
Sb, Antimony (ppm)	1746	1664	1828	1684	1808
Se, Selenium (ppm)	4.95	4.46	5.44	4.36	5.54
Sm, Samarium (ppm)	1.10	0.86	1.35	IND	IND
Sn, Tin (ppm)	7.67	6.81	8.54	7.09	8.26
Sr, Strontium (ppm)	10.8	10.1	11.4	10.1	11.5
Th, Thorium (ppm)	0.85	0.79	0.90	0.81	0.89
Ti, Titanium (wt.%)	0.013	0.011	0.014	0.012	0.013
Tl, Thallium (ppm)	0.83	0.74	0.92	0.80	0.85
U, Uranium (ppm)	1.79	1.70	1.89	1.74	1.85
V, Vanadium (ppm)	10.6	10.2	10.9	IND	IND
W, Tungsten (ppm)	1.81	1.68	1.93	1.72	1.90
Y, Yttrium (ppm)	4.32	3.86	4.77	4.15	4.49
Zn, Zinc (wt.%)	4.13	4.06	4.20	4.04	4.23
Zr, Zirconium (ppm)	3.91	2.60	5.22	3.52	4.31
<b>Peroxide Fusion ICP</b>					
Al, Aluminium (wt.%)	0.206	0.198	0.215	0.194	0.219
As, Arsenic (ppm)	538	494	582	525	552
Ca, Calcium (wt.%)	0.255	0.211	0.299	0.242	0.268
Co, Cobalt (ppm)	52	50	55	50	55
Cr, Chromium (ppm)	169	148	189	IND	IND
Fe, Iron (wt.%)	5.44	5.38	5.49	5.33	5.55
Mg, Magnesium (wt.%)	0.704	0.692	0.715	0.688	0.719
Mn, Manganese (wt.%)	0.186	0.184	0.189	0.183	0.190
S, Sulphur (wt.%)	16.08	15.65	16.51	15.81	16.35
Sb, Antimony (ppm)	1827	1778	1877	1753	1902
SiO <sub>2</sub> , Silicon dioxide (wt.%)	5.14	5.01	5.27	4.95	5.32
Zn, Zinc (wt.%)	4.14	4.06	4.21	4.06	4.21
<b>Infrared Combustion</b>					
C, Carbon (wt.%)	0.766	0.741	0.791	0.748	0.785
S, Sulphur (wt.%)	15.13	14.69	15.56	14.88	15.37

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

Note: intervals may appear asymmetric due to rounding.

## Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) in Table 4 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 Pb by the umpire laboratories employing EDTA titration, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 61.22 and 61.31 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.**

The homogeneity of OREAS 353 has also been evaluated in an ANOVA study for all certified analytes present in concentrations that are at least 20 times the lower limit of detection. No significant  $p$ -values were found indicating that no evidence exists that between-unit variance is greater than within-unit variance.

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 353 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 353 is sufficiently homogenous and is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PREPARER AND SUPPLIER

Certified reference material OREAS 353 was prepared, certified and supplied by:



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4. \*ALS, Loughrea, Galway, Ireland
5. \*ALS, Perth, WA, Australia
6. \*ALS, Vancouver, BC, Canada
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♦ = Umpire laboratory (classical methods); \* = Geoanalytical laboratory (instrumental methods).

***Please note: Above numbered alphabetical list of participating laboratories does not reflect the Lab ID numbering on the scatter plots below.***

Figure 1. Pb by Classical in OREAS 353

SPC.1470.Pb-Ag Conc 2019.OREAS 353.2.Classical.Pb.Lab.200528.141057.SS

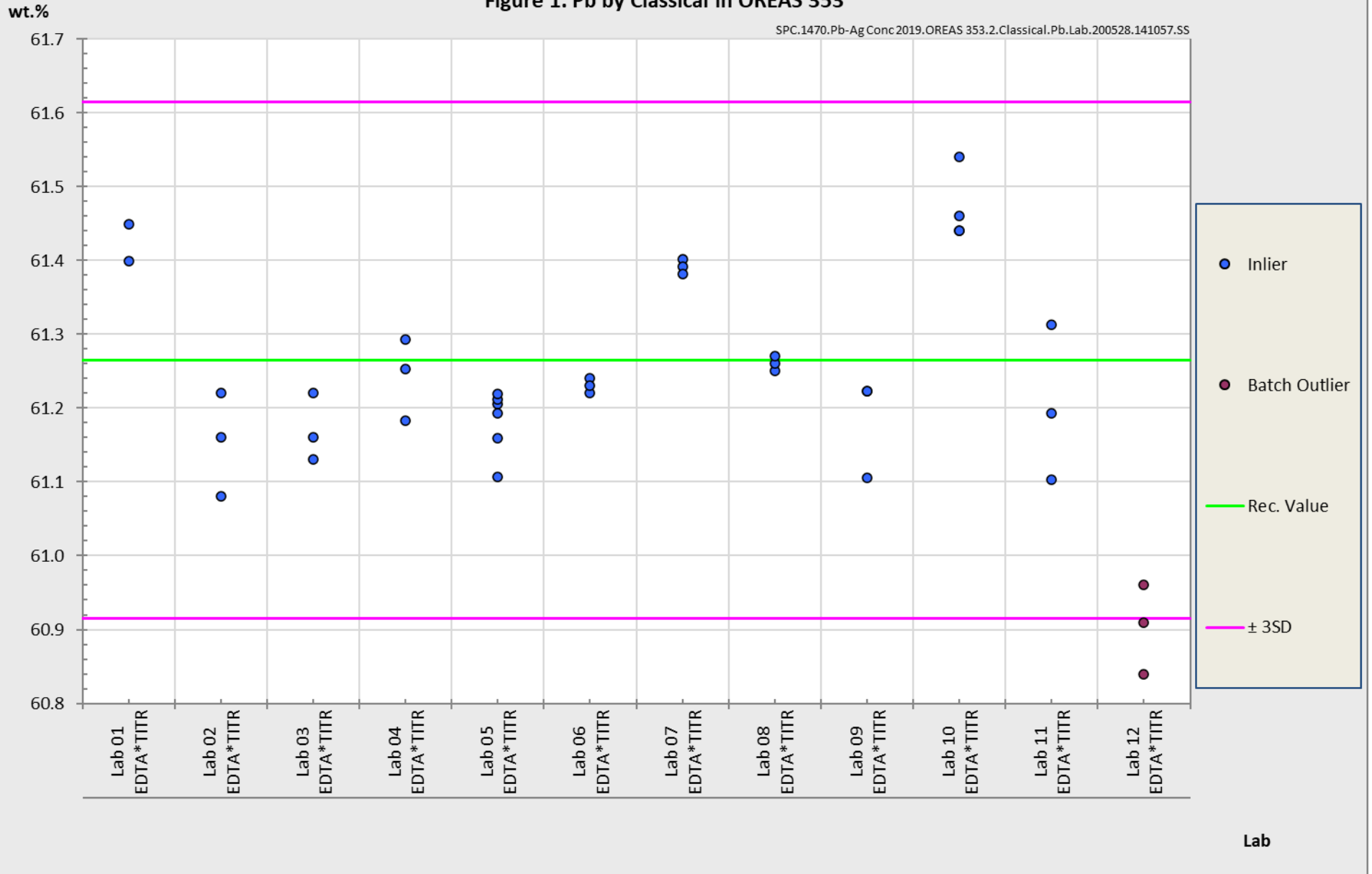
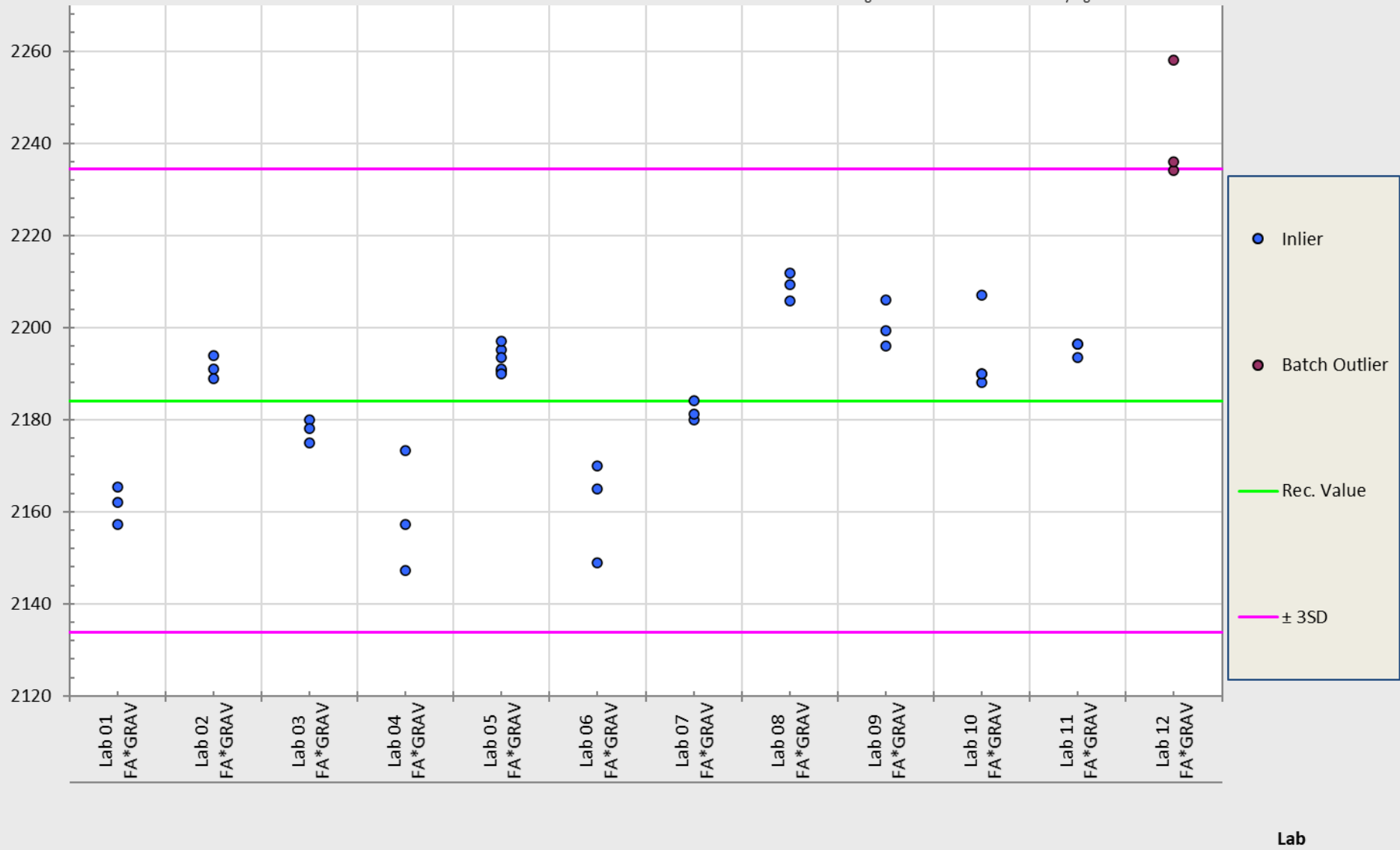


Figure 2. Ag by Fire Assay in OREAS 353

SPC.1470.Pb-Ag Conc 2019.OREAS 353.2.Fire Assay.Ag.Lab.200528.140816.SN

ppm



## 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 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 353 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 353 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 353 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.

### **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.

## **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.

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 353 is a sulphide-rich reference material (S ~15.1%) and is reactive under normal atmospheric conditions. To inhibit oxidation and prolong its shelf life it has been sealed under nitrogen in robust laminated foil pouches. In its unopened state under normal conditions of storage it has a shelf life beyond five years.

## **INSTRUCTIONS FOR CORRECT USE**

The 'umpire lab' Pb and Ag certified values for OREAS 353 refer to the concentration levels on a dry sample basis. 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 aliquots for Pb and Ag as per ISO 9599. The results were then corrected to dry basis using the moisture value. Moisture content varied amongst the laboratories from 0.26 -0.56% with an average of 0.38% (excluding two labs which reported a mean of 0.1 and 0.9%).

The 'geoanalytical lab' certified values for OREAS 353 are on a 'sample as received' basis. The CRM should not be dried prior to analysis.

## **DOCUMENT HISTORY**

<b>Revision No.</b>	<b>Date</b>	<b>Changes applied</b>
0	28 <sup>th</sup> May, 2020	First publication.



## 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.

## 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

A handwritten signature in blue ink, appearing to read 'Craig Hamlyn'.

28<sup>th</sup> May, 2020

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

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