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**CERTIFICATE OF ANALYSIS FOR**

**Manganese Ore**

**(Postmasburg Manganese Field, Northern Cape, South Africa)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 174**

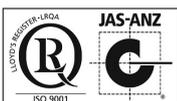
**Summary Statistics for Key\* Analytes.**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Borate Fusion XRF</b>						
Fe, Iron (wt.%)	20.32	0.115	20.27	20.36	20.24	20.39
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	29.05	0.165	28.98	29.12	28.94	29.15
Mn, Manganese (wt.%)	33.96	0.278	33.77	34.14	33.83	34.08
MnO, Manganese oxide (wt.%)	43.84	0.359	43.61	44.08	43.68	44.00
P, Phosphorus (ppm)	451	14	445	457	441	460
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.103	0.003	0.102	0.105	0.101	0.105

\*See Table 1 below for the full list of certified values.

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.



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**Table 1. Certified Values and Performance Gates for OREAS 174.**

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>Borate Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	7.36	0.082	7.20	7.53	7.11	7.61	1.12%	2.24%	3.35%	6.99	7.73
BaO, wt. %	1.06	0.029	1.00	1.12	0.97	1.15	2.76%	5.53%	8.29%	1.01	1.11
CaO, wt. %	0.320	0.010	0.300	0.341	0.289	0.352	3.25%	6.50%	9.76%	0.304	0.336
Cu, ppm	111	14	84	138	70	152	12.30%	24.60%	36.90%	105	116
Fe, wt. %	20.32	0.115	20.08	20.55	19.97	20.66	0.57%	1.14%	1.70%	19.30	21.33
Fe <sub>2</sub> O <sub>3</sub> , wt. %	29.05	0.165	28.72	29.37	28.55	29.54	0.57%	1.14%	1.70%	27.59	30.50
K <sub>2</sub> O, wt. %	0.820	0.019	0.783	0.858	0.764	0.876	2.28%	4.56%	6.84%	0.779	0.861
MgO, wt. %	0.354	0.015	0.324	0.383	0.310	0.397	4.11%	8.23%	12.34%	0.336	0.371
Mn, wt. %	33.96	0.278	33.40	34.51	33.12	34.79	0.82%	1.64%	2.46%	32.26	35.65
MnO, wt. %	43.84	0.359	43.13	44.56	42.77	44.92	0.82%	1.64%	2.46%	41.65	46.04
Na <sub>2</sub> O, wt. %	0.431	0.030	0.372	0.491	0.342	0.520	6.88%	13.77%	20.65%	0.410	0.453
P, ppm	451	14	424	478	410	492	3.01%	6.02%	9.03%	428	473
P <sub>2</sub> O <sub>5</sub> , wt. %	0.103	0.003	0.097	0.110	0.094	0.113	3.01%	6.02%	9.03%	0.098	0.108
SiO <sub>2</sub> , wt. %	6.06	0.092	5.87	6.24	5.78	6.33	1.51%	3.03%	4.54%	5.75	6.36
SO <sub>3</sub> , wt. %	0.039	0.007	0.025	0.053	0.019	0.060	17.42%	34.83%	52.25%	0.037	0.041
TiO <sub>2</sub> , wt. %	0.379	0.009	0.362	0.397	0.353	0.405	2.29%	4.58%	6.87%	0.360	0.398
V <sub>2</sub> O <sub>5</sub> , ppm	238	44	150	325	107	369	18.39%	36.78%	55.17%	226	250
<b>Thermogravimetry</b>											
LOI <sup>1000</sup> , wt. %	7.32	0.368	6.58	8.05	6.21	8.42	5.03%	10.06%	15.09%	6.95	7.68
<b>4-Acid Digestion</b>											
Al, wt. %	3.29	0.272	2.75	3.83	2.47	4.11	8.27%	16.55%	24.82%	3.13	3.45
As, ppm	28.3	1.84	24.6	31.9	22.8	33.8	6.49%	12.99%	19.48%	26.9	29.7
Ba, wt. %	0.937	0.040	0.856	1.017	0.816	1.058	4.31%	8.61%	12.92%	0.890	0.984
Be, ppm	4.26	0.46	3.34	5.19	2.87	5.66	10.88%	21.75%	32.63%	4.05	4.48
Bi, ppm	0.36	0.016	0.33	0.40	0.32	0.41	4.38%	8.76%	13.14%	0.35	0.38
Ca, wt. %	0.231	0.010	0.211	0.250	0.201	0.260	4.23%	8.46%	12.69%	0.219	0.242
Cd, ppm	0.33	0.05	0.24	0.43	0.20	0.47	13.66%	27.32%	40.98%	0.32	0.35
Ce, ppm	59	2.8	53	64	50	67	4.80%	9.60%	14.41%	56	62
Co, ppm	70	2.0	66	74	64	76	2.83%	5.65%	8.48%	67	74
Cr, ppm	84	10	65	104	55	113	11.50%	23.00%	34.49%	80	88
Cs, ppm	0.65	0.07	0.52	0.79	0.45	0.86	10.52%	21.04%	31.56%	0.62	0.69
Cu, ppm	97	6.6	84	110	77	117	6.75%	13.51%	20.26%	92	102
Dy, ppm	9.67	0.960	7.75	11.59	6.79	12.55	9.93%	19.86%	29.79%	9.19	10.15
Er, ppm	5.78	0.59	4.60	6.96	4.01	7.55	10.21%	20.42%	30.62%	5.49	6.07
Fe, wt. %	20.06	0.742	18.58	21.54	17.84	22.29	3.70%	7.39%	11.09%	19.06	21.06
Ga, ppm	17.7	3.4	10.9	24.5	7.5	27.9	19.28%	38.56%	57.84%	16.8	18.6
Gd, ppm	10.6	0.88	8.8	12.3	7.9	13.2	8.35%	16.70%	25.04%	10.0	11.1
Hf, ppm	2.07	0.174	1.72	2.42	1.55	2.59	8.40%	16.80%	25.20%	1.96	2.17
Ho, ppm	2.04	0.24	1.55	2.52	1.31	2.77	11.92%	23.84%	35.77%	1.94	2.14
In, ppm	0.077	0.006	0.064	0.090	0.058	0.096	8.30%	16.59%	24.89%	0.073	0.081
K, wt. %	0.678	0.023	0.632	0.725	0.608	0.749	3.44%	6.88%	10.33%	0.645	0.712

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: 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.

**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>											
La, ppm	37.1	3.65	29.8	44.4	26.2	48.0	9.83%	19.66%	29.50%	35.2	38.9
Li, ppm	843	49	745	941	695	991	5.84%	11.67%	17.51%	801	885
Lu, ppm	0.82	0.063	0.69	0.94	0.63	1.01	7.77%	15.54%	23.31%	0.78	0.86
Mg, wt. %	0.198	0.017	0.164	0.233	0.147	0.250	8.65%	17.31%	25.96%	0.188	0.208
Mn, wt. %	34.10	0.550	33.00	35.20	32.45	35.75	1.61%	3.22%	4.84%	32.40	35.81
Mo, ppm	1.93	0.31	1.31	2.56	1.00	2.87	16.16%	32.31%	48.47%	1.84	2.03
Na, wt. %	0.326	0.025	0.276	0.375	0.251	0.400	7.60%	15.21%	22.81%	0.309	0.342
Nb, ppm	5.57	0.483	4.61	6.54	4.13	7.02	8.67%	17.33%	26.00%	5.30	5.85
Nd, ppm	43.5	2.99	37.5	49.5	34.5	52.5	6.88%	13.77%	20.65%	41.3	45.7
Ni, ppm	145	11	123	167	112	178	7.62%	15.24%	22.86%	138	152
P, wt. %	0.044	0.003	0.038	0.051	0.035	0.054	7.31%	14.63%	21.94%	0.042	0.046
Pb, ppm	285	27	231	339	204	366	9.46%	18.92%	28.38%	271	299
Pr, ppm	9.83	1.25	7.32	12.34	6.07	13.59	12.76%	25.52%	38.28%	9.34	10.32
Rb, ppm	8.37	0.729	6.91	9.83	6.18	10.56	8.71%	17.42%	26.14%	7.95	8.79
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sb, ppm	1.80	0.099	1.61	2.00	1.51	2.10	5.48%	10.95%	16.43%	1.71	1.90
Sc, ppm	16.8	1.52	13.7	19.8	12.2	21.3	9.04%	18.07%	27.11%	15.9	17.6
Se, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	9.73	2.04	5.65	13.81	3.61	15.85	20.97%	41.95%	62.92%	9.24	10.21
Sn, ppm	1.84	0.113	1.62	2.07	1.51	2.18	6.11%	12.22%	18.33%	1.75	1.94
Sr, ppm	528	39	450	606	411	644	7.38%	14.76%	22.14%	501	554
Ta, ppm	0.40	0.037	0.32	0.47	0.29	0.51	9.25%	18.50%	27.76%	0.38	0.42
Tb, ppm	1.60	0.146	1.31	1.90	1.17	2.04	9.12%	18.24%	27.36%	1.52	1.68
Th, ppm	4.37	0.205	3.96	4.78	3.75	4.98	4.70%	9.40%	14.10%	4.15	4.59
Ti, wt. %	0.223	0.010	0.204	0.242	0.194	0.251	4.27%	8.54%	12.81%	0.211	0.234
Tl, ppm	0.29	0.03	0.22	0.35	0.19	0.38	10.72%	21.45%	32.17%	0.27	0.30
Tm, ppm	0.79	0.10	0.59	0.99	0.48	1.10	12.93%	25.87%	38.80%	0.75	0.83
U, ppm	1.36	0.061	1.24	1.49	1.18	1.55	4.46%	8.91%	13.37%	1.30	1.43
V, ppm	124	6	111	137	105	143	5.18%	10.35%	15.53%	118	130
W, ppm	3.96	0.271	3.42	4.50	3.15	4.77	6.84%	13.67%	20.51%	3.76	4.16
Y, ppm	69	5.5	58	80	53	86	7.98%	15.95%	23.93%	66	73
Yb, ppm	5.14	0.489	4.16	6.12	3.68	6.61	9.51%	19.02%	28.53%	4.88	5.40
Zn, ppm	89	5.6	78	100	72	106	6.28%	12.57%	18.85%	84	93
<b>Gas / Liquid Pycnometry</b>											
SG, Unity	4.36	0.182	3.99	4.72	3.81	4.90	4.17%	8.34%	12.51%	4.14	4.57

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: 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.

**Table 2. Indicative Values for OREAS 174.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Borate Fusion XRF</b>								
As <sub>2</sub> O <sub>3</sub>	ppm	50	NiO	ppm	206	Zn	ppm	108
Cl	ppm	23.3	Pb	ppm	282	ZrO <sub>2</sub>	ppm	103
CoO	ppm	224	Sb <sub>2</sub> O <sub>3</sub>	ppm	15.8			
Cr <sub>2</sub> O <sub>3</sub>	ppm	137	SrO	ppm	625			
<b>4-Acid Digestion</b>								
Ag	ppm	0.040	Ge	ppm	0.21	Te	ppm	0.090
Eu	ppm	3.47	S	wt.%	0.076			
<b>Infrared Combustion</b>								
C	wt.%	0.018	S	wt.%	0.015			
<b>Peroxide Fusion ICP</b>								
Al	wt.%	3.11	K	wt.%	0.646	Si	wt.%	2.72
Ca	wt.%	0.189	Mg	wt.%	0.168	Ti	wt.%	0.211
Fe	wt.%	22.11	Mn	wt.%	33.39			

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

## INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

## SOURCE MATERIAL

OREAS 174 has been prepared from manganese ores sourced from the Glosam Mine situated within Postmasburg Manganese Field located in the Northern Cape Province of South Africa. The ores are composed mainly of braunite group minerals including braunite (3Mn<sub>2</sub>O<sub>3</sub>.MnSiO<sub>3</sub>), partridgeite (Mn<sub>2</sub>O<sub>3</sub>) and bixbyite (Mn,Fe)<sub>2</sub>O<sub>3</sub>.

## PERFORMANCE GATES

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.

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).*

## COMMUNITION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 174 were prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and multi-stage milling to 100 minus 30 microns;
- Homogenisation;
- Packaging in 10g units sealed in laminated foil pouches and 1kg units in plastic jars.

## PHYSICAL PROPERTIES

OREAS 174 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

**Table 3. Physical properties of OREAS 174.**

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
870.6	1.00	5YR 3/2	Grayish Brown

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

## ANALYTICAL PROGRAM

Fifteen commercial analytical laboratories participated in the program to characterise the elements reported in Table 1. The following methods were employed:

- Mn ore package for Al<sub>2</sub>O<sub>3</sub>, BaO, CaO, Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, MgO, MnO, Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, S, SiO<sub>2</sub>, TiO<sub>2</sub> & V<sub>2</sub>O<sub>5</sub> by borate fusion with XRF finish;
- Thermogravimetry: Loss on Ignition (LOI) at 1000°C (6 laboratories used a thermogravimetric analyser, 6 laboratories included LOI with their fusion package and 3 laboratories used conventional muffle furnace);
- 4-acid digestion for full suite elemental package by ICP-OES and MS finish (up to 10 laboratories depending on the element);
- Specific gravity by gas pycnometry (10 laboratories), liquid pycnometry (1 laboratory) or volumetric flask (1 laboratory).

For the round robin program twelve 200g test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate 200g 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 1 provides performance gate intervals for the certified values. 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 174-DataPack.2.0.200825\_100143.xlsx**).

Results are also presented in scatter plots for Mn by borate fusion with XRF finish and 4-acid digestion in Figures 1 and 2 respectively, together with  $\pm 3SD$  (magenta) and  $\pm 5\%$  (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

## 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, Standard Deviations, 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.

**Certified Values** are the means of accepted laboratory means after outlier filtering.

**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 174.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Borate Fusion XRF</b>					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	7.36	7.31	7.41	7.31	7.41
BaO, Barium oxide (wt.%)	1.06	1.04	1.08	1.05	1.07
CaO, Calcium oxide (wt.%)	0.320	0.315	0.326	0.315	0.326
Cu, Copper (ppm)	111	95	127	IND	IND
Fe, Iron (wt.%)	20.32	20.27	20.36	20.24	20.39
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	29.05	28.98	29.12	28.94	29.15
K <sub>2</sub> O, Potassium oxide (wt.%)	0.820	0.810	0.831	0.813	0.828
MgO, Magnesium oxide (wt.%)	0.354	0.346	0.361	0.343	0.364
Mn, Manganese (wt.%)	33.96	33.77	34.14	33.83	34.08
MnO, Manganese oxide (wt.%)	43.84	43.61	44.08	43.68	44.00
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.431	0.410	0.452	0.411	0.452
P, Phosphorus (ppm)	451	445	457	441	460
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.103	0.102	0.105	0.101	0.105
SiO <sub>2</sub> , Silicon dioxide (wt.%)	6.06	6.01	6.10	6.01	6.10
SO <sub>3</sub> , Sulphur trioxide (wt.%)	0.039	0.035	0.043	IND	IND
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.379	0.375	0.384	0.374	0.385
V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)	238	210	266	207	269

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>Thermogravimetry</b>					
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	7.32	7.09	7.54	7.20	7.43
<b>4-Acid Digestion</b>					
Al, Aluminium (wt.%)	3.29	3.03	3.55	3.16	3.42
As, Arsenic (ppm)	28.3	26.5	30.0	26.9	29.6
Ba, Barium (wt.%)	0.937	0.908	0.966	0.921	0.952
Be, Beryllium (ppm)	4.26	3.93	4.60	4.16	4.37
Bi, Bismuth (ppm)	0.36	0.35	0.38	0.35	0.38
Ca, Calcium (wt.%)	0.231	0.224	0.237	0.225	0.236
Cd, Cadmium (ppm)	0.33	0.29	0.37	0.31	0.36
Ce, Cerium (ppm)	59	57	61	57	61
Co, Cobalt (ppm)	70	68	72	68	72
Cr, Chromium (ppm)	84	76	92	80	88
Cs, Caesium (ppm)	0.65	0.59	0.72	0.63	0.68
Cu, Copper (ppm)	97	92	102	94	100
Dy, Dysprosium (ppm)	9.67	8.39	10.95	9.47	9.87
Er, Erbium (ppm)	5.78	4.99	6.56	5.53	6.02
Fe, Iron (wt.%)	20.06	19.47	20.65	19.53	20.59
Ga, Gallium (ppm)	17.7	14.9	20.5	16.5	18.9
Gd, Gadolinium (ppm)	10.6	9.4	11.8	10.2	10.9
Hf, Hafnium (ppm)	2.07	1.96	2.18	1.98	2.15
Ho, Holmium (ppm)	2.04	1.77	2.31	1.98	2.10
In, Indium (ppm)	0.077	0.071	0.083	0.049	0.104
K, Potassium (wt.%)	0.678	0.665	0.692	0.663	0.694
La, Lanthanum (ppm)	37.1	34.2	39.9	36.2	38.0
Li, Lithium (ppm)	843	809	877	824	862
Lu, Lutetium (ppm)	0.82	0.74	0.90	0.78	0.85
Mg, Magnesium (wt.%)	0.198	0.186	0.211	0.195	0.202
Mn, Manganese (wt.%)	34.10	33.58	34.63	33.54	34.66
Mo, Molybdenum (ppm)	1.93	1.63	2.23	1.83	2.04
Na, Sodium (wt.%)	0.326	0.308	0.343	0.315	0.336
Nb, Niobium (ppm)	5.57	5.20	5.95	5.23	5.92
Nd, Neodymium (ppm)	43.5	39.5	47.5	42.9	44.1
Ni, Nickel (ppm)	145	137	153	140	150
P, Phosphorus (wt.%)	0.044	0.042	0.047	0.042	0.046
Pb, Lead (ppm)	285	266	304	278	292
Pr, Praseodymium (ppm)	9.83	8.08	11.58	9.17	10.48
Rb, Rubidium (ppm)	8.37	7.85	8.89	8.04	8.70
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND

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>					
Sb, Antimony (ppm)	1.80	1.72	1.89	1.70	1.91
Sc, Scandium (ppm)	16.8	15.9	17.6	16.0	17.6
Se, Selenium (ppm)	< 1	IND	IND	IND	IND
Sm, Samarium (ppm)	9.73	6.86	12.59	9.40	10.05
Sn, Tin (ppm)	1.84	1.76	1.93	IND	IND
Sr, Strontium (ppm)	528	498	558	515	541
Ta, Tantalum (ppm)	0.40	0.38	0.41	0.37	0.42
Tb, Terbium (ppm)	1.60	1.41	1.80	1.56	1.64
Th, Thorium (ppm)	4.37	4.24	4.50	4.22	4.52
Ti, Titanium (wt.%)	0.223	0.216	0.230	0.216	0.229
Tl, Thallium (ppm)	0.29	0.26	0.31	0.27	0.30
Tm, Thulium (ppm)	0.79	0.65	0.93	IND	IND
U, Uranium (ppm)	1.36	1.32	1.40	1.33	1.40
V, Vanadium (ppm)	124	119	129	120	128
W, Tungsten (ppm)	3.96	3.77	4.14	3.77	4.14
Y, Yttrium (ppm)	69	65	73	68	71
Yb, Ytterbium (ppm)	5.14	4.49	5.80	5.01	5.27
Zn, Zinc (ppm)	89	84	94	85	93
<b>Gas / Liquid Pycnometry</b>					
SG, Specific Gravity (Unity)	4.36	4.22	4.49	4.31	4.40

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 Manganese by borate fusion XRF finish, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 33.83 and 34.08 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 174 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 174 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 174 is sufficiently homogenous and is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PARTICIPATING LABORATORIES

1. ALS, Brisbane, QLD, Australia
2. ALS, Johannesburg, South Africa
3. ALS, Vancouver, BC, Canada
4. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
5. Bureau Veritas Geoanalytical, Perth, WA, Australia
6. Carsurin, Kendari, Sulawesi, Indonesia
7. Intertek Genalysis, Johannesburg, South Africa
8. Intertek Genalysis, Perth, WA, Australia
9. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
10. Nagrom, Perth, WA, Australia
11. SGS, Randfontein, Gauteng, South Africa
12. SGS Australia Mineral Services, Perth, WA, Australia
13. SGS Canada Inc., Vancouver, BC, Canada
14. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
15. UIS Analytical Services, Centurion, South Africa

***Please note: To preserve anonymity, the above numbered alphabetical list of participating laboratories does not correspond with the Lab ID numbering on the scatter plots below.***

Figure 1. Mn by Fusion XRF in OREAS 174

SPC.1503.Postmasburg Mn-Fe ore CRMs. OREAS 174.1.Fusion XRF.Mn.Lab.200806.115903.SN

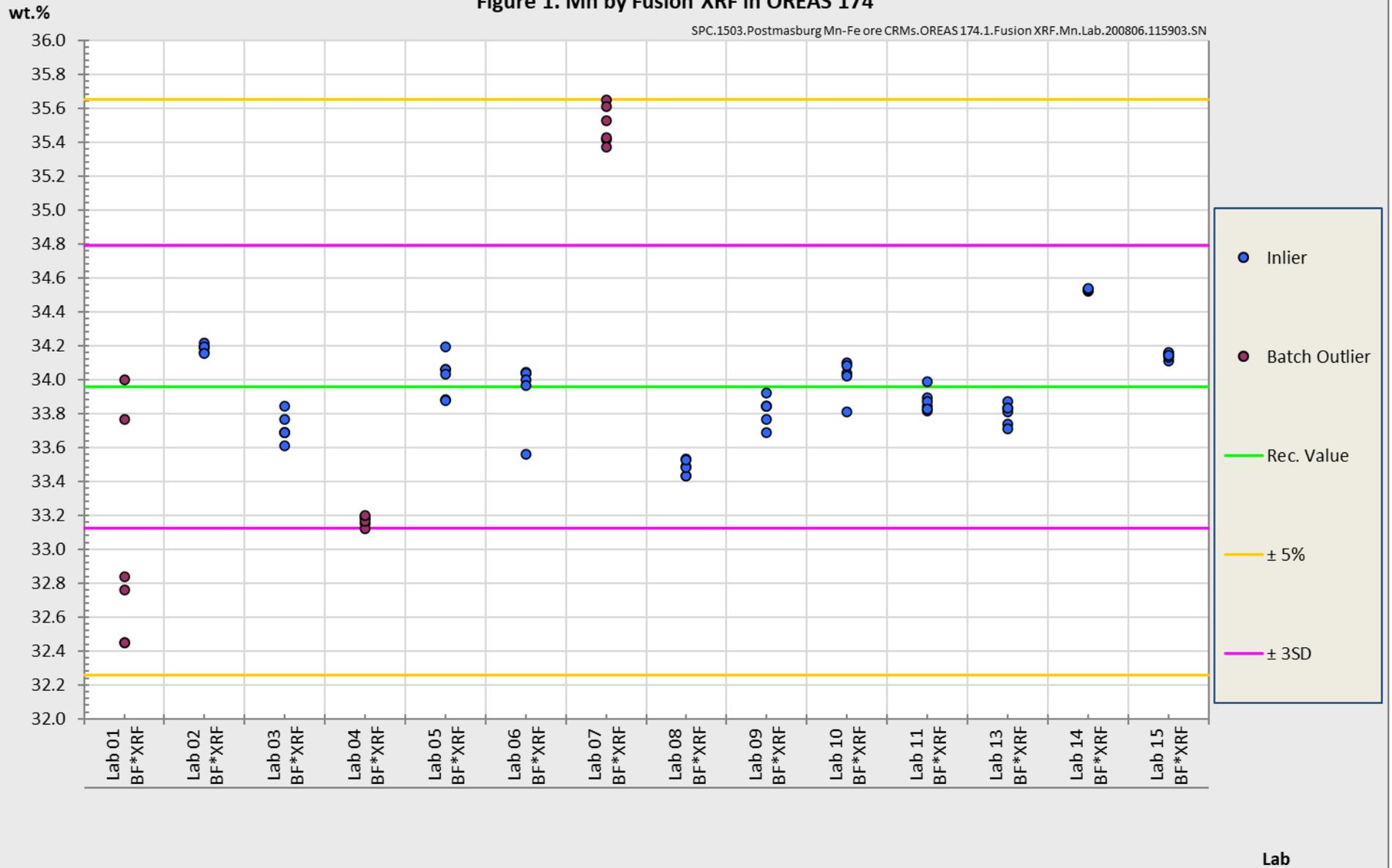
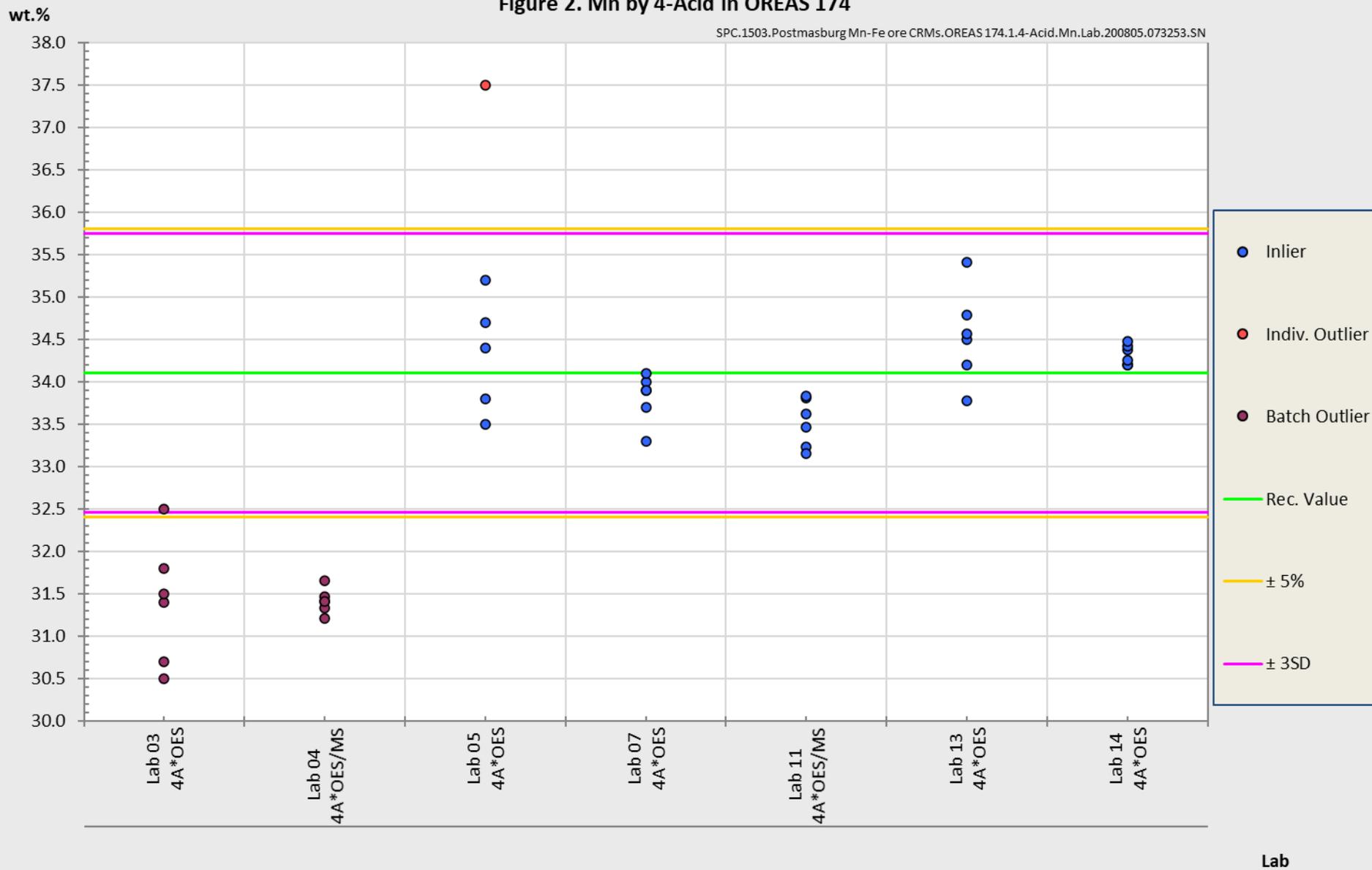


Figure 2. Mn by 4-Acid in OREAS 174

SPC.1503.Postmasburg Mn-Fe ore CRMs.OREAS 174.1.4-Acid.Mn.Lab.200805.073253.SN



## PREPARER AND SUPPLIER

Certified reference material OREAS 174 was prepared, certified and supplied by:



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## 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 174 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 174 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 174 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological 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. 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

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 174 is an oxidised reference material and is stable in the laminated foil pouches. Under normal conditions of storage it has a shelf life beyond ten years.

## INSTRUCTIONS FOR CORRECT USE

The certified values for lithium borate fusion XRF and for LOI are on a dry basis. This requires the removal of hygroscopic moisture by drying in air to constant mass at 105°C. If the reference material is not dried prior to analysis, the certified values should be corrected to the moisture-bearing basis.

The certified values by 4-acid digestion and Specific Gravity by pycnometry are reported on a 'sample as received' basis.

## 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.

## DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	6 <sup>th</sup> August, 2020	First publication.
1	25 <sup>th</sup> August, 2020	Changed LOI statistics.

## 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

25<sup>th</sup> August, 2020

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

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