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CERTIFICATE OF ANALYSIS FOR

Matrix-Matched Certified Reference Material

OREAS 739

Smelter-grade Alumina (Australia)

In collaboration with
Alumina Quality Workshop Inc., Australia

AQW
ALUMINA QUALITY WORKSHOP INC

Table 1. Certified Values and Performance Gates for OREAS 739.

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
Borate Fusion XRF											
Al_2O_3, wt.%	98.60	0.326	97.95	99.26	97.62	99.58	0.33%	0.66%	0.99%	93.67	103.53
CaO, wt.%	0.031	0.003	0.025	0.038	0.022	0.041	10.0%	19.9%	29.9%	0.030	0.033
Cr_2O_3, ppm	2.58	1.51	0.00	5.61	0.00	7.12	58.6%	117%	176%	2.45	2.71
Fe_2O_3, wt.%	0.009	0.001	0.007	0.012	0.006	0.013	12.6%	25.2%	37.8%	0.009	0.010
K_2O, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
MgO, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
MnO, ppm	3.6	3.2	0.0	10.0	0.0	13.2	88.7%	177%	266%	3.4	3.8
Na_2O, wt.%	0.395	0.012	0.371	0.419	0.358	0.432	3.09%	6.19%	9.28%	0.375	0.415
P_2O_5, wt.%	0.0010	0.0002	0.0006	0.0013	0.0005	0.0014	16.8%	33.6%	50.3%	0.001	0.001
SiO_2, wt.%	0.012	0.001	0.009	0.014	0.008	0.016	11.7%	23.4%	35.1%	0.011	0.012
SrO, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
TiO_2, wt.%	0.003	0.001	0.002	0.004	0.002	0.005	16.0%	32.0%	48.1%	0.003	0.004
V_2O_5, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
ZrO_2, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Borate Fusion ICP											
BaO, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
BeO, ppm	2.77	0.51	1.75	3.79	1.24	4.30	18.36%	36.71%	55.07%	2.63	2.91
Dy, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Er, ppm	< 0.03	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Eu, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ga_2O_3, ppm	68	3.0	62	74	59	77	4.41%	8.83%	13.24%	65	71
Gd, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Lu, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Nd, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pr, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Rb, ppm	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sn, ppm	1.03	0.35	0.34	1.73	0.00	2.07	33.66%	67.33%	101.0%	0.98	1.08
SrO, ppm	3.85	0.70	2.44	5.26	1.73	5.96	18.32%	36.65%	54.97%	3.65	4.04
Ta, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tm, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
U, ppm	0.30	0.05	0.20	0.40	0.15	0.46	16.62%	33.24%	49.85%	0.29	0.32
V_2O_5, ppm	12.8	3.9	5.0	20.5	1.1	24.4	30.35%	60.69%	91.04%	12.1	13.4
W, ppm	0.84	0.30	0.24	1.43	0.00	1.73	35.81%	71.63%	107.4%	0.79	0.88
Y, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Yb, ppm	< 0.03	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
ZnO, ppm	69	4.8	59	78	54	83	6.94%	13.87%	20.81%	65	72
ZrO_2, ppm	1.82	0.81	0.21	3.44	0.00	4.24	44.25%	88.49%	132.7%	1.73	1.91
Thermogravimetry											
$LOI^{300-1000}$, wt.%	1.03	0.056	0.92	1.14	0.86	1.19	5.41%	10.81%	16.22%	0.98	1.08
MOI^{300}, wt.%	4.65	1.11	2.42	6.87	1.30	7.99	24.00%	47.99%	71.99%	4.41	4.88

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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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SOURCE MATERIAL

OREAS 739 is a smelter-grade alumina (SGA), certified reference material (CRM) prepared from three alumina products blended in equal proportions. The materials were sourced from Alcoa and Worsley Alumina in Western Australia, and Rio Tinto in Queensland (Australia). OREAS 739 was developed in collaboration with Alumina Quality Workshop Inc. and is typical of SGA produced in Australia and similar to globally produced SGA.

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). 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) ± 10%.

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

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 739 were prepared in the following manner:

- Drying to constant mass at 105°C;
- Milling to 100% minus 30 microns;
- Combining and blending equal proportions of each of the three source materials;
- Homogenisation using OREAS' novel processing technologies;
- Packaging into 1020 x 500g plastic jars.

ANALYTICAL PROGRAM

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

1. Al_2O_3, CaO, Cr_2O_3, Fe_2O_3, K_2O, MgO, MnO, Na_2O, P_2O_5, SiO_2, SrO, TiO_2, V_2O_5, and ZrO_2 by lithium borate fusion with XRF;
2. BaO, BeO, Ga_2O_3, Sn, SrO, U, V_2O_5, W, ZnO and ZrO_2 by borate fusion with ICP-OES and or ICP-MS finish (except for the BV Perth laboratory who performed laser ablation with ICP-MS on the fused discs);

3. MOI (moisture) at 300°C and LOI (loss on ignition) at 1000°C on the 'as received' sample by TGA as per AS2879.1-2000.

For the fusion with XRF analysis, the laboratories were provided strict pre-assay sample instructions as follows:

Please report XRF results on a dry sample basis via correction for moisture content or drying by either:

a) equilibration of XRF sample in laboratory atmosphere with all XRF analytes corrected to dry basis using the result of a moisture analysis undertaken on a separate subsample at 300°C held for a period of 2 hours or,
b) removal of H_2O- by drying at 300°C for 2 hours, then place sample in a desiccator with fresh desiccant until weighed for analysis.

Samples for the interlaboratory (round robin) program were taken at twelve predetermined sampling intervals during packaging and are considered representative of the entire prepared batch. Five 30g samples were dispatched to each laboratory and these were taken from staggered sampling (lot) intervals to maximise representation (e.g., the five samples a laboratory may have received could be from lots 7, 9, 11, 2 and 4).

Table 1 above presents the certified values and performance gate intervals based on their pooled 1SD's. Table 2 provides shows indicative values and Table 3 presents 95% confidence and tolerance limits. Tabulated results of all elements together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations, per cent deviation of lab means from the corrected mean of means (PDM^3) and z-scores for corrected laboratory means are presented in an Excel-compatible file for this CRM (**OREAS 739-DataPack.1.1.230803_150814.xlsx**).

Results for Al_2O_3, CaO, Na_2O, Fe_2O_3 and SiO_2 by lithium borate fusion with XRF finish are also presented in scatter plots (Figures 1 to 5) 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.

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.

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 3) 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) a laboratory reported analytes beyond those requested; ii) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; iii) inter-laboratory consensus is poor; or iv) a significant proportion of results are outlying.

Table 2. Indicative Values for OREAS 739.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
BaO	ppm	*< 100*	SO_3	wt.%	*0.005*			
Ga_2O_3	ppm	*84*	ZnO	ppm	*< 12*			
Borate Fusion ICP								
Ag	ppm	*< 0.5*	Hf	ppm	*0.13*	P_2O_5	wt.%	*0.008*
As	ppm	*< 5*	Ho	ppm	*< 0.01*	Pb	ppm	*< 5*
Bi	ppm	*< 0.4*	In	ppm	*< 0.2*	Re	ppm	*< 0.1*
CaO	wt.%	*0.056*	K_2O	wt.%	*0.044*	Sb	ppm	*< 0.5*
Ce	ppm	*0.085*	La	ppm	*0.14*	Sc	ppm	*< 1*
Co	ppm	*2.62*	MgO	wt.%	*0.014*	SiO_2	wt.%	*0.040*
Cr_2O_3	ppm	*6.44*	MnO	ppm	*22.6*	Te	ppm	*< 5*
Cs	ppm	*< 0.5*	Mo	ppm	*< 2*	Th	ppm	*< 0.05*
Cu	ppm	*< 10*	Na_2O	wt.%	*0.393*	TiO_2	wt.%	*0.003*
Fe_2O_3	wt.%	*0.024*	Nb	ppm	*< 0.1*	Tl	ppm	*< 0.1*
Ge	ppm	*< 1*	Ni	ppm	*4.63*			
Pb Fire Assay								
Pd	ppb	*< 5*						
Infrared Combustion								
S	wt.%	*0.002*						

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

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.

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.***

Table 3. 95% Confidence & Tolerance Limits for OREAS 739.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	98.60	98.37	98.84	98.48	98.73
CaO, Calcium oxide (wt.%)	0.031	0.029	0.033	0.030	0.032
Cr_2O_3, Chromium(III) oxide (ppm)	2.58	1.32	3.84	IND	IND
Fe_2O_3, Iron(III) oxide (wt.%)	0.009	0.008	0.010	0.008	0.010
K_2O, Potassium oxide (wt.%)	< 0.01	IND	IND	IND	IND
MgO, Magnesium oxide (wt.%)	< 0.01	IND	IND	IND	IND
MnO, Manganese oxide (ppm)	3.6	0.0	11.8	IND	IND
Na_2O, Sodium oxide (wt.%)	0.395	0.386	0.404	0.389	0.401
P_2O_5, Phosphorus(V) oxide (wt.%)	0.001	0.001	0.001	IND	IND
SiO_2, Silicon dioxide (wt.%)	0.012	0.010	0.013	0.010	0.014
SrO, Strontium oxide (ppm)	< 100	IND	IND	IND	IND
TiO_2, Titanium dioxide (wt.%)	0.003	0.003	0.004	0.003	0.004
V_2O_5, Vanadium(V) oxide (ppm)	< 10	IND	IND	IND	IND
ZrO_2, Zirconium dioxide (ppm)	< 100	IND	IND	IND	IND
Borate Fusion ICP					
BaO, Barium oxide (ppm)	< 1	IND	IND	IND	IND
BeO, Beryllium oxide (ppm)	2.77	2.21	3.33	IND	IND
Dy, Dysprosium (ppm)	< 0.05	IND	IND	IND	IND
Er, Erbium (ppm)	< 0.03	IND	IND	IND	IND
Eu, Europium (ppm)	< 0.02	IND	IND	IND	IND
Ga_2O_3, Gallium(III) oxide (ppm)	68	65	71	66	70
Gd, Gadolinium (ppm)	< 0.05	IND	IND	IND	IND
Lu, Lutetium (ppm)	< 0.01	IND	IND	IND	IND
Nd, Neodymium (ppm)	< 0.1	IND	IND	IND	IND
Pr, Praseodymium (ppm)	< 0.02	IND	IND	IND	IND
Rb, Rubidium (ppm)	< 0.2	IND	IND	IND	IND
Sm, Samarium (ppm)	< 0.05	IND	IND	IND	IND
Sn, Tin (ppm)	1.03	0.79	1.27	IND	IND
SrO, Strontium oxide (ppm)	3.85	3.04	4.65	3.33	4.36
Ta, Tantalum (ppm)	< 0.1	IND	IND	IND	IND
Tb, Terbium (ppm)	< 0.01	IND	IND	IND	IND
Tm, Thulium (ppm)	< 0.01	IND	IND	IND	IND
U, Uranium (ppm)	0.30	0.27	0.34	IND	IND
V_2O_5, Vanadium(V) oxide (ppm)	12.8	9.7	15.8	IND	IND
W, Tungsten (ppm)	0.84	0.57	1.10	IND	IND
Y, Yttrium (ppm)	< 0.1	IND	IND	IND	IND
Yb, Ytterbium (ppm)	< 0.03	IND	IND	IND	IND
ZnO, Zinc oxide (ppm)	69	55	82	IND	IND
ZrO_2, Zirconium dioxide (ppm)	1.82	0.92	2.73	IND	IND

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 3 continued.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
Thermogravimetry					
$LOI^{300-1000}$, Loss On Ignition 300-1000°C (wt.%)	1.03	0.99	1.07	1.00	1.05
MOI^{300}, Moisture @300°C (wt.%)	4.65	3.66	5.63	4.56	4.73

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
Note: intervals may appear asymmetric due to rounding.

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Table 3 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 Al_2O_3 by lithium borate fusion with XRF, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 98.48 and 98.73 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 739 displays a high level of homogeneity and is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALCOA of Australia, Kwinana, WA, Australia
3. ALS, Brisbane, QLD, Australia
4. ALS, Vancouver, BC, Canada
5. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
6. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
7. Bureau Veritas Geoanalytical, Cardiff, NSW, Australia
8. Bureau Veritas Geoanalytical, Perth, WA, Australia
9. Inspectorate (BV), Lima, Peru
10. Intertek Genalysis, Perth, WA, Australia
11. Queensland Alumina Ltd, Gladstone, QLD, Australia
12. Rio Tinto Yarwun Operations, Gladstone, QLD, Australia
13. RUSAL, Saint Petersburg, Russian Federation
14. SGS Australia Mineral Services, Perth, WA, Australia
15. SGS Mineral Services, Townsville, QLD, Australia
16. South32 Worsley Alumina, Collie, WA, Australia

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

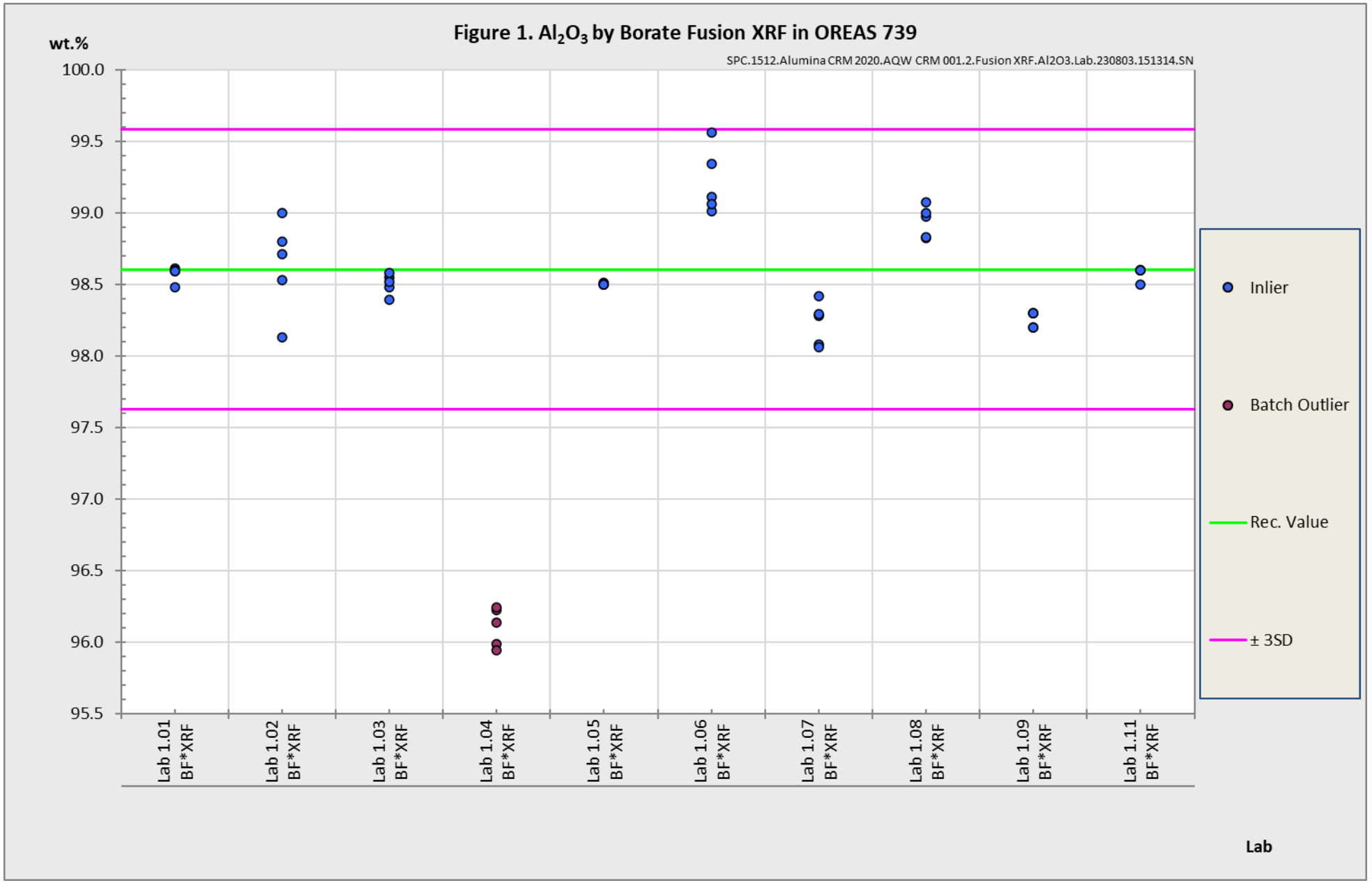
Figure 1. Al2O3 by Borate Fusion XRF in OREAS 739
wt.%
SPC.1512.Alumina CRM 2020.AQW CRM 001.2.Fusion XRF.Al2O3.Lab.230803.151314.SN
100.0
99.5
99.0
98.5
98.0
97.5
97.0
96.5
96.0
95.5
Lab 1.01 BF*XRF
Lab 1.02 BF*XRF
Lab 1.03 BF*XRF
Lab 1.04 BF*XRF
Lab 1.05 BF*XRF
Lab 1.06 BF*XRF
Lab 1.07 BF*XRF
Lab 1.08 BF*XRF
Lab 1.09 BF*XRF
Lab 1.11 BF*XRF
Inlier
Batch Outlier
Rec. Value
± 3SD
Lab

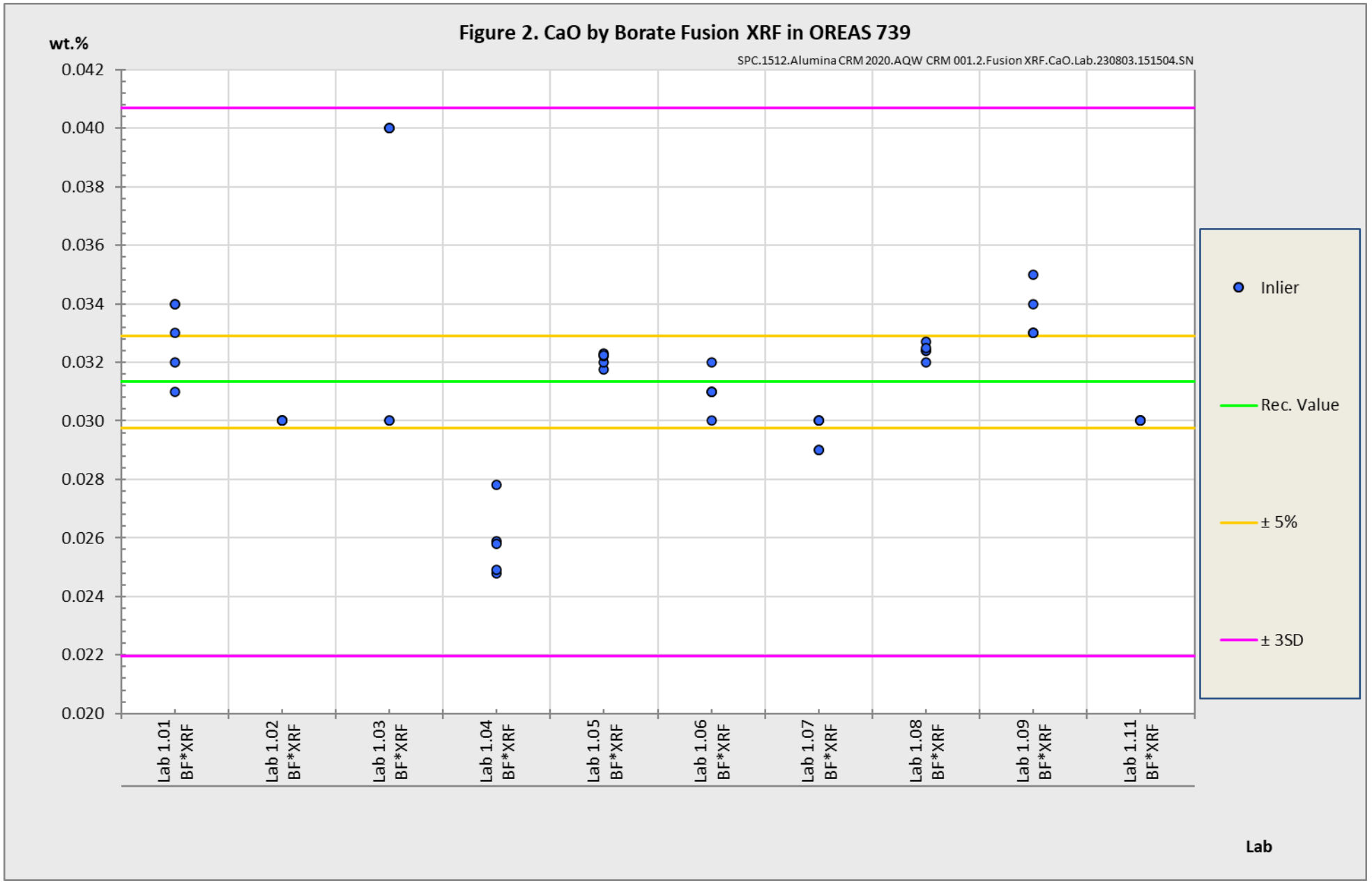
Figure 2. CaO by Borate Fusion XRF in OREAS 739
wt.%
SPC.1512.Alumina CRM 2020.AQW CRM 001.2.Fusion XRF.CaO.Lab.230803.151504.SN
0.042
0.040
0.038
0.036
0.034
0.032
0.030
0.028
0.026
0.024
0.022
0.020
Lab 1.01 BF*XRF
Lab 1.02 BF*XRF
Lab 1.03 BF*XRF
Lab 1.04 BF*XRF
Lab 1.05 BF*XRF
Lab 1.06 BF*XRF
Lab 1.07 BF*XRF
Lab 1.08 BF*XRF
Lab 1.09 BF*XRF
Lab 1.11 BF*XRF
Lab
Inlier
Rec. Value
± 5%
± 3SD

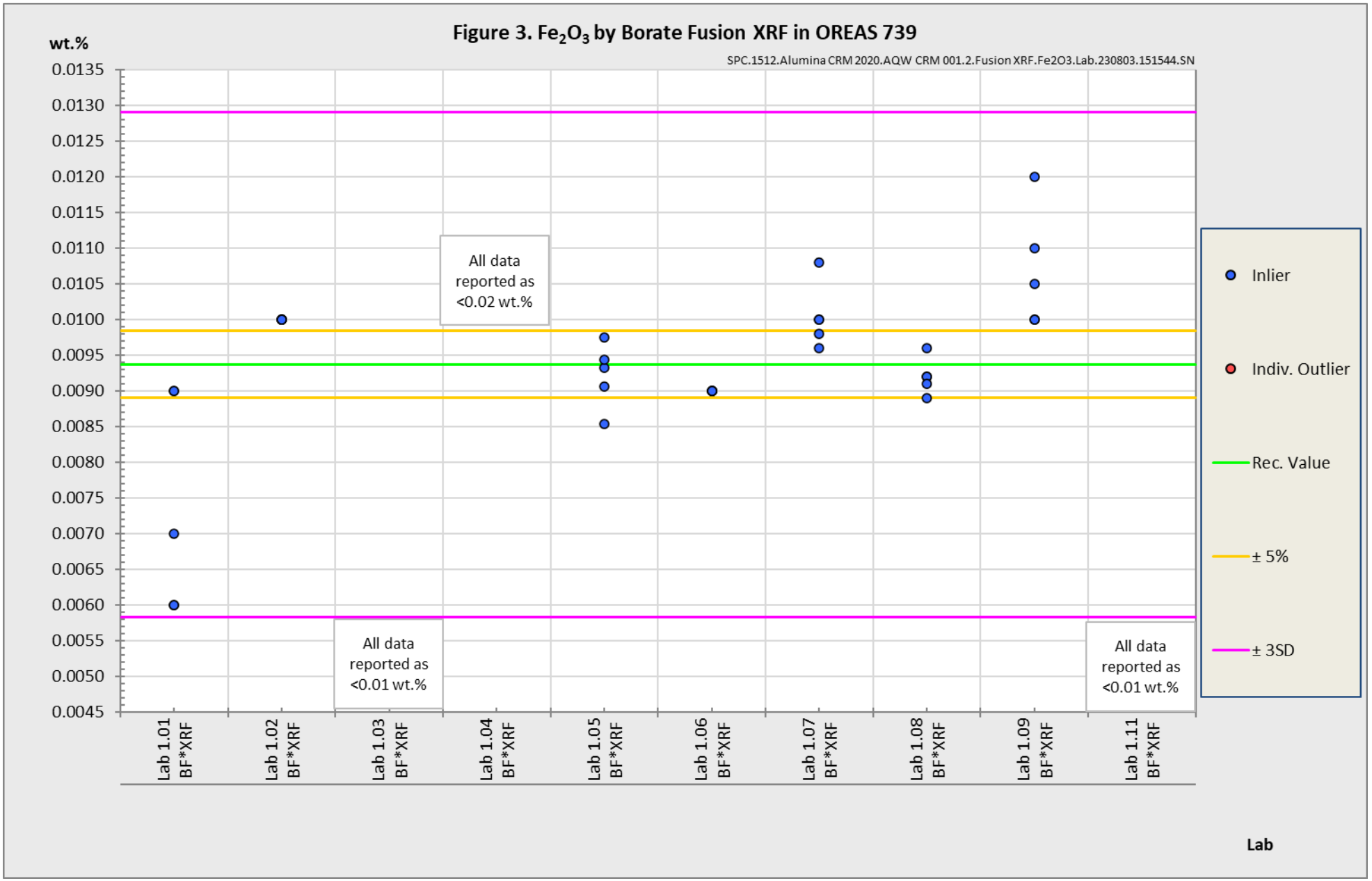
Figure 3. Fe2O3 by Borate Fusion XRF in OREAS 739
wt.%
SPC.1512.Alumina CRM 2020.AQW CRM 001.2.Fusion XRF.Fe2O3.Lab.230803.151544.SN
0.0135
0.0130
0.0125
0.0120
0.0115
0.0110
0.0105
0.0100
0.0095
0.0090
0.0085
0.0080
0.0075
0.0070
0.0065
0.0060
0.0055
0.0050
0.0045
All data reported as <0.02 wt.%
All data reported as <0.01 wt.%
All data reported as <0.01 wt.%
Lab 1.01 BF*XRF
Lab 1.02 BF*XRF
Lab 1.03 BF*XRF
Lab 1.04 BF*XRF
Lab 1.05 BF*XRF
Lab 1.06 BF*XRF
Lab 1.07 BF*XRF
Lab 1.08 BF*XRF
Lab 1.09 BF*XRF
Lab 1.11 BF*XRF
Inlier
Indiv. Outlier
Rec. Value
± 5%
± 3SD
Lab

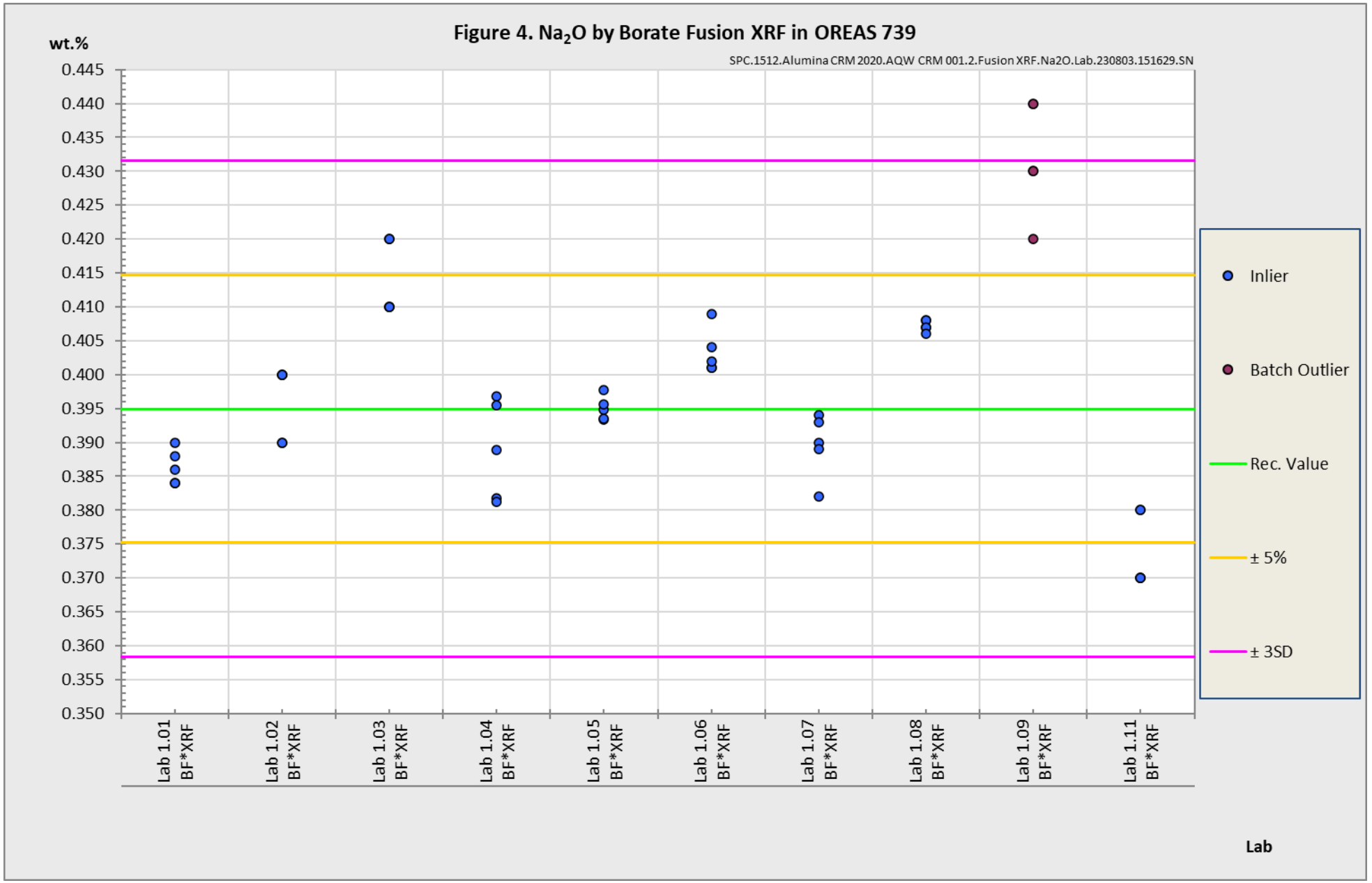
Figure 4. Na2O by Borate Fusion XRF in OREAS 739
wt.%
SPC.1512.Alumina CRM 2020.AQW CRM 001.2.Fusion XRF.Na2O.Lab.230803.151629.SN
0.445
0.440
0.435
0.430
0.425
0.420
0.415
0.410
0.405
0.400
0.395
0.390
0.385
0.380
0.375
0.370
0.365
0.360
0.355
0.350
Lab 1.01 BF*XRF
Lab 1.02 BF*XRF
Lab 1.03 BF*XRF
Lab 1.04 BF*XRF
Lab 1.05 BF*XRF
Lab 1.06 BF*XRF
Lab 1.07 BF*XRF
Lab 1.08 BF*XRF
Lab 1.09 BF*XRF
Lab 1.11 BF*XRF
Lab
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD

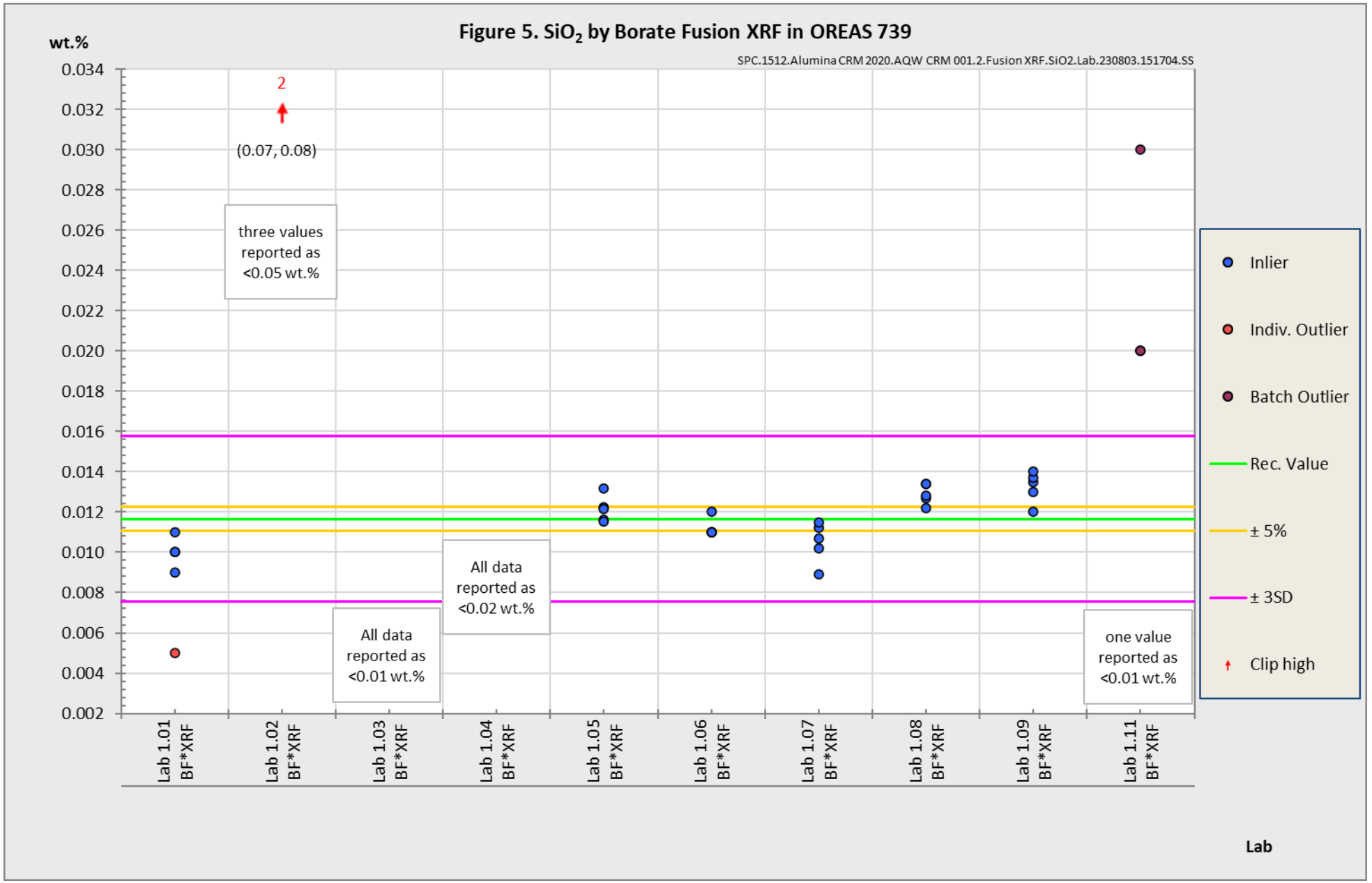
Figure 5. SiO2 by Borate Fusion XRF in OREAS 739
wt.%
SPC.1512.Alumina CRM 2020.AQW CRM 001.2.Fusion XRF.SiO2.Lab.230803.151704.SS
0.034
0.032
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
2
(0.07, 0.08)
three values reported as <0.05 wt.%
All data reported as <0.01 wt.%
All data reported as <0.02 wt.%
one value reported as <0.01 wt.%
Lab 1.01 BF*XRF
Lab 1.02 BF*XRF
Lab 1.03 BF*XRF
Lab 1.04 BF*XRF
Lab 1.05 BF*XRF
Lab 1.06 BF*XRF
Lab 1.07 BF*XRF
Lab 1.08 BF*XRF
Lab 1.09 BF*XRF
Lab 1.11 BF*XRF
Lab
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Clip high

PREPARER AND SUPPLIER

Certified Reference Material OREAS 739 is prepared, certified and supplied by:

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METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified values are metrologically traceable to the international measurement scale (SI) of mass (either as a % mass fraction or as milligrams per kilogram (mg/kg)). In line with popular use, all data within tables in this certificate are expressed as the mass fraction in either weight percent (wt.%) or parts per million (ppm).

The analytical samples sent to participating laboratories were selected in a manner to be representative of the entire prepared batch of 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. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [10], each analytical data set received from the participating laboratories has been validated by its assayer through the inclusion of internal reference materials and QC checks during and post 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. Being matrix-matched, OREAS 739 will display similar behaviour in the relevant measurement process to the routine samples for which OREAS 739 is designated to monitor. To maintain commutability, care should be taken to always ensure 'matrix matching' as close as practically achievable. The chemical composition of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their routine samples.

INTENDED USE

OREAS 739 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 739 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution. OREAS 739 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.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 739 is naturally stable. In its unopened packaged state and under normal conditions of storage it has a shelf life beyond ten years.

**Normal storage conditions: means storage not in direct sunlight in a dry, clean, well-ventilated area at temperatures between -5° and 50°C.*

INSTRUCTIONS FOR CORRECT USE

The certified values by lithium borate fusion with XRF and for LOI (300-1000°C) are on a '300°C dry sample' basis while the certified values by other methods (fusion with ICP-OES/MS, and MOI at 300°C are reported on a 'sample as received' basis.

Minimum sample size

As a practical guide, the minimum mass of sample used should match the typical mass that the laboratories used in the inter-laboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Borate fusion with XRF finish: ≥0.2g;
- MOI at 300°C and LOI between 300-1000°C: ≥1g;
- Borate fusion with ICP-OES and/or ICP-MS finish: ≥0.2g.

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.

The performance gates shown in Table 1 are intended only to be used as a preliminary guide as to what a laboratory may be able to achieve. Over a period of time monitoring your own laboratory's data for this CRM, SD's should be calculated directly from your own laboratory's process. This will enable you to establish more specific performance gates that are fit for purpose for your application as well as the ability to monitor bias. If your long-term trend analysis shows an average value that is within the 95% confidence interval then generally there is no cause for concern in regard to bias.

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 strongly advised.

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034.

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.

LEGAL NOTICE

Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the use of this material and information.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	19th October, 2023	First publication.

CERTIFYING OFFICER

19th October, 2023

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

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