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CERTIFICATE OF ANALYSIS FOR

# CERTIFIED REFERENCE MATERIAL

# OREAS 750b

## (Pegmatite Li Ore, Western Australia)

**Table 1. Certified Values, Uncertainty & Tolerance Intervals for multi-elements by 4-acid digestion in OREAS 750b.**

| Constituent | Certified Value† | 95% Expanded Uncertainty | | 95% Tolerance Limits | |
|---|---|---|---|---|---|
| | | Low | High | Low | High |
| **4-Acid Digestion** | | | | | |
| Ag, Silver (ppm) | 0.094 | 0.071 | 0.117 | IND | IND |
| Al, Aluminium (wt.%) | 3.91 | 3.81 | 4.01 | 3.84 | 3.98 |
| As, Arsenic (ppm) | 2.88 | 2.40 | 3.36 | 2.55 | 3.21 |
| Ba, Barium (ppm) | 370 | 361 | 379 | 364 | 376 |
| Be, Beryllium (ppm) | 3.53 | 3.38 | 3.67 | 3.42 | 3.63 |
| Bi, Bismuth (ppm) | 0.44 | 0.38 | 0.50 | 0.39 | 0.48 |
| Ca, Calcium (wt.%) | 0.609 | 0.589 | 0.628 | 0.598 | 0.619 |
| Cd, Cadmium (ppm) | 0.090 | 0.061 | 0.119 | IND | IND |
| Ce, Cerium (ppm) | 29.8 | 27.6 | 31.9 | 28.8 | 30.7 |
| Co, Cobalt (ppm) | 2.99 | 2.87 | 3.12 | 2.87 | 3.11 |
| Cr, Chromium (ppm) | 17.9 | 15.6 | 20.3 | 16.9 | 19.0 |
| Cs, Caesium (ppm) | 39.2 | 38.0 | 40.5 | 38.4 | 40.0 |
| Cu, Copper (ppm) | 10.7 | 10.0 | 11.5 | 10.2 | 11.3 |
| Dy, Dysprosium (ppm) | 1.52 | 1.37 | 1.67 | 1.42 | 1.62 |
| Er, Erbium (ppm) | 0.59 | 0.53 | 0.65 | 0.55 | 0.63 |
| Eu, Europium (ppm) | 0.52 | 0.45 | 0.58 | 0.49 | 0.54 |
| Fe, Iron (wt.%) | 1.07 | 1.04 | 1.10 | 1.05 | 1.09 |
| Ga, Gallium (ppm) | 16.8 | 16.2 | 17.5 | 16.4 | 17.3 |
| Gd, Gadolinium (ppm) | 2.28 | 2.09 | 2.47 | 2.13 | 2.43 |
| Hf, Hafnium (ppm) | 1.21 | 1.12 | 1.30 | 1.16 | 1.26 |
| Ho, Holmium (ppm) | 0.24 | 0.21 | 0.27 | 0.22 | 0.26 |
| In, Indium (ppm) | 0.021 | 0.017 | 0.025 | 0.018 | 0.024 |
| K, Potassium (wt.%) | 1.15 | 1.12 | 1.18 | 1.13 | 1.17 |
| La, Lanthanum (ppm) | 14.4 | 13.5 | 15.4 | 14.0 | 14.8 |
| Li, Lithium (wt.%) | 0.245 | 0.238 | 0.251 | 0.241 | 0.249 |
| $Li_2O$, Lithium oxide (wt.%) | 0.527 | 0.513 | 0.541 | 0.519 | 0.535 |
| Lu, Lutetium (ppm) | 0.065 | 0.054 | 0.077 | IND | IND |
| Mg, Magnesium (wt.%) | 0.267 | 0.257 | 0.276 | 0.260 | 0.273 |
| Mn, Manganese (wt.%) | 0.039 | 0.038 | 0.040 | 0.038 | 0.040 |
| Mo, Molybdenum (ppm) | 1.60 | 1.47 | 1.73 | 1.51 | 1.69 |
| Na, Sodium (wt.%) | 0.755 | 0.734 | 0.775 | 0.743 | 0.767 |
| Nb, Niobium (ppm) | 28.4 | 27.3 | 29.6 | 27.4 | 29.4 |
| Nd, Neodymium (ppm) | 13.5 | 12.4 | 14.6 | 13.1 | 13.9 |
| Ni, Nickel (ppm) | 7.66 | 7.25 | 8.07 | 7.39 | 7.94 |
| P, Phosphorus (wt.%) | 0.039 | 0.038 | 0.041 | 0.038 | 0.041 |

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

†The operationally defined measurands meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

Note: intervals may appear asymmetric due to rounding. IND = indeterminate (due to limited reading resolution of the methods employed).

**Table 1 continued.**

| Constituent | Certified Value† | 95% Expanded Uncertainty | | 95% Tolerance Limits | |
|---|---|---|---|---|---|
| | | Low | High | Low | High |
| **4-Acid Digestion continued** | | | | | |
| Pb, Lead (ppm) | 9.55 | 9.17 | 9.93 | 9.24 | 9.86 |
| Pr, Praseodymium (ppm) | 3.65 | 3.34 | 3.96 | 3.41 | 3.89 |
| Rb, Rubidium (ppm) | 288 | 276 | 299 | 282 | 293 |
| Re, Rhenium (ppm) | < 0.002 | IND | IND | IND | IND |
| S, Sulphur (wt.%) | 0.045 | 0.041 | 0.049 | 0.043 | 0.047 |
| Sb, Antimony (ppm) | 0.39 | 0.35 | 0.42 | 0.35 | 0.42 |
| Sc, Scandium (ppm) | 3.20 | 3.00 | 3.40 | 3.07 | 3.33 |
| Sm, Samarium (ppm) | 2.69 | 2.47 | 2.91 | 2.57 | 2.81 |
| Sn, Tin (ppm) | 25.8 | 24.3 | 27.2 | 24.8 | 26.7 |
| Sr, Strontium (ppm) | 60 | 58 | 63 | 59 | 62 |
| Ta, Tantalum (ppm) | 64 | 61 | 67 | 62 | 66 |
| Tb, Terbium (ppm) | 0.31 | 0.28 | 0.33 | 0.29 | 0.32 |
| Te, Tellurium (ppm) | < 0.05 | IND | IND | IND | IND |
| Th, Thorium (ppm) | 5.94 | 5.61 | 6.27 | 5.71 | 6.17 |
| Ti, Titanium (wt.%) | 0.151 | 0.145 | 0.156 | 0.147 | 0.154 |
| Tl, Thallium (ppm) | 1.93 | 1.86 | 2.00 | 1.89 | 1.98 |
| Tm, Thulium (ppm) | 0.071 | 0.054 | 0.088 | IND | IND |
| U, Uranium (ppm) | 1.91 | 1.70 | 2.13 | 1.77 | 2.06 |
| V, Vanadium (ppm) | 22.8 | 21.6 | 23.9 | 22.0 | 23.6 |
| W, Tungsten (ppm) | 5.08 | 4.52 | 5.64 | 4.72 | 5.44 |
| Y, Yttrium (ppm) | 6.13 | 5.81 | 6.45 | 5.93 | 6.33 |
| Yb, Ytterbium (ppm) | 0.46 | 0.40 | 0.53 | 0.43 | 0.50 |
| Zn, Zinc (ppm) | 60 | 57 | 62 | 58 | 61 |
| Zr, Zirconium (ppm) | 27.8 | 26.0 | 29.7 | 26.5 | 29.1 |

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.

†The operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

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 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 750b.**

| Constituent | Certified Value | 95 % Expanded Uncertainty | | 95 % Tolerance Limits | |
|---|---|---|---|---|---|
| | | Low | High | Low | High |
| **Borate / Peroxide Fusion ICP** | | | | | |
| Al, Aluminium (wt.%) | 4.02 | 3.92 | 4.11 | 3.95 | 4.08 |
| Ba, Barium (ppm) | 371 | 358 | 384 | 363 | 378 |
| Be, Beryllium (ppm) | 3.88 | 3.31 | 4.45 | IND | IND |
| Bi, Bismuth (ppm) | 0.46 | 0.24 | 0.67 | IND | IND |
| Ca, Calcium (wt.%) | 0.601 | 0.551 | 0.650 | 0.581 | 0.620 |
| Ce, Cerium (ppm) | 29.6 | 28.0 | 31.1 | 28.3 | 30.8 |
| Co, Cobalt (ppm) | 3.13 | 2.90 | 3.37 | 2.86 | 3.41 |
| Cs, Caesium (ppm) | 39.1 | 37.7 | 40.4 | 38.2 | 39.9 |
| Dy, Dysprosium (ppm) | 2.21 | 1.99 | 2.43 | 2.02 | 2.40 |
| Er, Erbium (ppm) | 1.13 | 0.98 | 1.28 | 1.03 | 1.24 |
| Eu, Europium (ppm) | 0.52 | 0.45 | 0.58 | 0.47 | 0.56 |
| Fe, Iron (wt.%) | 1.09 | 1.05 | 1.13 | 1.06 | 1.11 |
| Ga, Gallium (ppm) | 16.9 | 15.9 | 18.0 | 16.4 | 17.5 |
| Gd, Gadolinium (ppm) | 2.64 | 2.34 | 2.95 | 2.53 | 2.76 |
| Ge, Germanium (ppm) | 1.49 | 0.51 | 2.47 | IND | IND |
| Ho, Holmium (ppm) | 0.40 | 0.35 | 0.44 | 0.37 | 0.43 |
| In, Indium (ppm) | < 0.2 | IND | IND | IND | IND |
| K, Potassium (wt.%) | 1.18 | 1.14 | 1.23 | 1.16 | 1.21 |
| La, Lanthanum (ppm) | 14.6 | 13.4 | 15.8 | 14.1 | 15.1 |
| Li, Lithium (wt.%) | 0.246 | 0.237 | 0.255 | 0.241 | 0.251 |
| $Li_2O$, Lithium oxide (wt.%) | 0.529 | 0.510 | 0.548 | 0.518 | 0.540 |
| Lu, Lutetium (ppm) | 0.16 | 0.12 | 0.19 | IND | IND |
| Mg, Magnesium (wt.%) | 0.266 | 0.254 | 0.278 | 0.256 | 0.276 |
| Mn, Manganese (wt.%) | 0.039 | 0.038 | 0.040 | 0.038 | 0.040 |
| Nb, Niobium (ppm) | 29.3 | 27.5 | 31.1 | 27.9 | 30.7 |
| Nd, Neodymium (ppm) | 13.2 | 12.2 | 14.2 | 12.7 | 13.7 |
| P, Phosphorus (wt.%) | 0.039 | 0.038 | 0.040 | 0.038 | 0.040 |
| Pb, Lead (ppm) | 9.64 | 8.14 | 11.14 | IND | IND |
| Pr, Praseodymium (ppm) | 3.49 | 3.27 | 3.70 | 3.28 | 3.69 |
| Rb, Rubidium (ppm) | 287 | 273 | 300 | 280 | 293 |
| Re, Rhenium (ppm) | < 0.1 | IND | IND | IND | IND |
| Si, Silicon (wt.%) | 40.13 | 38.92 | 41.34 | 39.53 | 40.73 |
| Sm, Samarium (ppm) | 2.79 | 2.52 | 3.06 | 2.64 | 2.94 |
| Sn, Tin (ppm) | 38.7 | 36.6 | 40.9 | 36.6 | 40.9 |
| Sr, Strontium (ppm) | 62 | 57 | 67 | 60 | 64 |
| Ta, Tantalum (ppm) | 69 | 64 | 73 | 66 | 71 |
| Tb, Terbium (ppm) | 0.40 | 0.36 | 0.44 | 0.37 | 0.43 |

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 2 continued.**

| Constituent | Certified Value | 95% Expanded Uncertainty | | 95% Tolerance Limits | |
|---|---|---|---|---|---|
| | | Low | High | Low | High |
| **Borate / Peroxide Fusion ICP continued** | | | | | |
| Th, Thorium (ppm) | 5.96 | 5.58 | 6.33 | 5.67 | 6.24 |
| Ti, Titanium (wt.%) | 0.154 | 0.145 | 0.163 | 0.147 | 0.161 |
| Tl, Thallium (ppm) | 1.96 | 1.84 | 2.09 | 1.77 | 2.16 |
| Tm, Thulium (ppm) | 0.16 | 0.14 | 0.18 | IND | IND |
| U, Uranium (ppm) | 2.28 | 1.90 | 2.65 | 2.03 | 2.52 |
| V, Vanadium (ppm) | 22.8 | 20.7 | 24.8 | 19.8 | 25.7 |
| W, Tungsten (ppm) | 5.54 | 4.37 | 6.71 | 4.49 | 6.59 |
| Y, Yttrium (ppm) | 11.4 | 10.4 | 12.3 | 10.4 | 12.3 |
| Yb, Ytterbium (ppm) | 1.04 | 0.87 | 1.21 | IND | IND |
| Zn, Zinc (ppm) | 57 | 49 | 64 | 54 | 59 |
| **Borate Fusion XRF** | | | | | |
| $Al_2O_3$, Aluminium(III) oxide (wt.%) | 7.63 | 7.56 | 7.70 | 7.57 | 7.70 |
| BaO, Barium oxide (ppm) | 407 | 337 | 478 | 379 | 436 |
| CaO, Calcium oxide (wt.%) | 0.851 | 0.836 | 0.867 | 0.838 | 0.865 |
| $Cr_2O_3$, Chromium(III) oxide (ppm) | < 100 | IND | IND | IND | IND |
| $Fe_2O_3$, Iron(III) oxide (wt.%) | 1.56 | 1.54 | 1.59 | 1.54 | 1.58 |
| $K_2O$, Potassium oxide (wt.%) | 1.39 | 1.38 | 1.41 | 1.38 | 1.41 |
| MgO, Magnesium oxide (wt.%) | 0.452 | 0.431 | 0.473 | 0.443 | 0.462 |
| MnO, Manganese oxide (wt.%) | 0.050 | 0.050 | 0.051 | 0.049 | 0.052 |
| $Na_2O$, Sodium oxide (wt.%) | 1.01 | 0.98 | 1.04 | 0.99 | 1.03 |
| Nb, Niobium (ppm) | < 50 | IND | IND | IND | IND |
| $P_2O_5$, Phosphorus(V) oxide (wt.%) | 0.088 | 0.083 | 0.093 | 0.086 | 0.090 |
| Rb, Rubidium (ppm) | 259 | 229 | 290 | 222 | 296 |
| $SiO_2$, Silicon dioxide (wt.%) | 85.46 | 84.85 | 86.08 | 84.85 | 86.08 |
| $SO_3$, Sulphur trioxide (wt.%) | 0.105 | 0.093 | 0.117 | 0.096 | 0.114 |
| SrO, Strontium oxide (ppm) | 96 | 80 | 112 | IND | IND |
| $TiO_2$, Titanium dioxide (wt.%) | 0.257 | 0.248 | 0.265 | 0.249 | 0.264 |
| **Thermogravimetry** | | | | | |
| $LOI^{1000}$, Loss On Ignition @1000°C (wt.%) | 0.570 | 0.500 | 0.640 | 0.538 | 0.602 |

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. Indicative Values for OREAS 750b.**

| Constituent | Unit | Value | Constituent | Unit | Value | Constituent | Unit | Value |
|---|---|---|---|---|---|---|---|---|
| **4-Acid Digestion** | | | | | | | | |
| B | ppm | *< 10* | Hg | ppm | *< 1* | | | |
| Ge | ppm | *0.14* | Se | ppm | *1.34* | | | |
| **Peroxide Fusion ICP** | | | | | | | | |
| Ag | ppm | *< 10* | Hf | ppm | *3.67* | Sb | ppm | *0.48* |
| As | ppm | *24.3* | Hg | ppm | *< 5* | Sc | ppm | *< 5* |
| B | ppm | *17.6* | Mo | ppm | *2.11* | Se | ppm | *< 10* |
| Cd | ppm | *< 10* | Na | wt.% | *0.740* | Te | ppm | *< 1* |
| Cr | ppm | *42.2* | Ni | ppm | *18.8* | Zr | ppm | *114* |
| Cu | ppm | *16.1* | S | wt.% | *0.042* | | | |
| **Borate Fusion XRF** | | | | | | | | |
| Ag | ppm | *0.097* | Ho | ppm | *0.42* | Ta | ppm | *55* |
| As | ppm | *37.2* | In | ppm | *< 0.1* | Tb | ppm | *0.38* |
| Be | ppm | *3.50* | La | ppm | *33.4* | Te | ppm | *< 0.1* |
| Bi | ppm | *< 100* | Lu | ppm | *0.15* | Th | ppm | *17.0* |
| Ce | ppm | *31.2* | Mo | ppm | *< 50* | Tl | ppm | *1.97* |
| Co | ppm | *< 100* | Nd | ppm | *14.0* | Tm | ppm | *0.17* |
| Cs | ppm | *40.9* | NiO | ppm | *28.7* | U | ppm | *5.33* |
| CuO | ppm | *43.0* | Pb | ppm | *< 50* | $V_2O_5$ | ppm | *50* |
| Dy | ppm | *2.12* | Pr | ppm | *3.59* | W | ppm | *6.28* |
| Er | ppm | *1.11* | Re | ppm | *< 0.1* | Y | ppm | *23.3* |
| Eu | ppm | *0.51* | Sb | ppm | *< 50* | Yb | ppm | *1.03* |
| Ga | ppm | *16.6* | Sc | ppm | *4.17* | Zn | ppm | *69* |
| Gd | ppm | *2.65* | Se | ppm | *1.25* | Zr | ppm | *128* |
| $H_2O$- | wt.% | *0.107* | Sm | ppm | *2.88* | | | |
| Hf | ppm | *< 80* | Sn | ppm | *27.4* | | | |
| **Aqua Regia Digestion** | | | | | | | | |
| Cs | ppm | *40.3* | | | | | | |

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.

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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 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. In evaluating laboratory performance with this CRM, the section headed 'Instructions for correct use' should be read carefully.

Table 1 (generated from data supplied by laboratories all accredited to ISO 17025 for 4-acid digestion) and Table 2 (generated from data supplied by laboratories mostly accredited to ISO 17025) provide the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties and Table 5 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 750b-DataPack.1.3.241028_143941.xlsx**). Results are also presented in scatter plots for $Li_2O$ (wt.%) by 4-acid digestion and borate/peroxide fusion with ICP in Figures 1 and 2 respectively, together with ±3 SD (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.

# SOURCE MATERIAL

OREAS 750b was prepared from a blend of spodumene concentrate derived from the processing of lithium pegmatite ores sourced from the Greenbushes area of southwest Western Australia, Londonderry lithium-pegmatite ore (containing elevated levels of lithium, rubidium, caesium, tin and tantalum), barren granodiorite and quartz. The barren granodiorite was sourced from the mafic, S-Type, Late Devonian Bulla Granodiorite complex located in northern Melbourne, Australia.

# COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 750b was prepared in the following manner:

- Drying of ore and barren materials to constant mass at 105 °C;
- Crushing and milling of the barren materials to > 98 % minus 75 µm;
- Multi-stage milling of ore and spodumene concentrate materials to 100 % minus 30 µm;
- Check analysis of ore and spodumene concentrate for contained Li concentration;
- Blending the ore, concentrate and barren materials in appropriate proportions to achieve the desired Li grade;

- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10 g units in laminated foil pouches and 1 kg units in plastic wide-mouth jars.

## PHYSICAL PROPERTIES

OREAS 750b was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

**Table 4. Physical properties of OREAS 750b.**

| Bulk Density (kg/m$^3$) | Moisture (wt.%) | Munsell Notation‡ | Munsell Color‡ |
|---|---|---|---|
| 841 | 0.43 | N8 | Very Light Gray |

‡The Munsell Rock Color 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

Twenty-eight commercial analytical laboratories participated in the program to certify the elements reported in Table 1 and 2. The following methods were employed:

- Sodium peroxide fusion with full suite ICP-OES and ICP-MS elemental packages (up to 24 laboratories depending on the element) except for one laboratory that used lithium borate fusion with ICP-MS for Nb, Sn and Ta only;
- 4-acid ($HNO_3$-HF-$HClO_4$-HCl) digestion with full suite ICP-OES and ICP-MS elemental packages (up to 26 laboratories depending on the element);
- Lithium borate fusion whole rock analysis package by X-ray fluorescence (up to 20 laboratories depending on the element);
- Thermogravimetry: Loss on Ignition (LOI) at 1000 °C (8 laboratories used a thermogravimetric analyser, 8 laboratories included LOI with their fusion package and 5 laboratories used a conventional muffle furnace).

For the round robin program twelve 1 kg 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 15 g scoop splits from each of three separate 1 kg test units. This format enabled a 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).

## STATISTICAL ANALYSIS

**Certified Values and their uncertainty intervals** (Tables 1 and 2) 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. 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. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

**95% Expanded Uncertainty** provides a 95 % probability that the true value of the analyte under consideration lies between the upper and lower limits and is calculated according to the method outlined in [5] and [15]. All known or suspected sources of bias have been investigated or taken into account.

**Indicative (uncertified) values** (Table 3) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor. This data is intended for 'informational purposes' only.

**Standard Deviation** intervals (see Table 5, 'Performance Gates') 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.***

**Homogeneity Evaluation**

The tolerance limits (ISO 16269:2014) shown in Tables 1 and 2 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 lithium ($Li_2O$) by 4-acid digestion with ICP, where 99 % of the time ($1-\alpha=0.99$) at least 95 % of subsamples ($\rho=0.95$) will have concentrations lying between 0.519 and 0.535 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. ***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 interlaboratory certification program, it can be concluded that OREAS 750b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

# PERFORMANCE GATES

Table 5 below 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* [1].

**Table 5. Performance Gates for OREAS 750b.**

| 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 |
| **4-Acid Digestion** | | | | | | | | | | | |
| Ag, ppm | 0.094 | 0.019 | 0.055 | 0.133 | 0.036 | 0.152 | 20.60% | 41.20% | 61.80% | 0.090 | 0.099 |
| Al, wt.% | 3.91 | 0.150 | 3.61 | 4.21 | 3.46 | 4.36 | 3.84% | 7.67% | 11.51% | 3.71 | 4.10 |
| As, ppm | 2.88 | 0.51 | 1.87 | 3.90 | 1.36 | 4.40 | 17.61% | 35.23% | 52.84% | 2.74 | 3.03 |
| Ba, ppm | 370 | 10 | 349 | 391 | 339 | 401 | 2.81% | 5.62% | 8.43% | 351 | 388 |
| Be, ppm | 3.53 | 0.211 | 3.11 | 3.95 | 2.90 | 4.16 | 5.97% | 11.94% | 17.91% | 3.35 | 3.70 |
| Bi, ppm | 0.44 | 0.06 | 0.32 | 0.55 | 0.27 | 0.61 | 13.11% | 26.23% | 39.34% | 0.42 | 0.46 |
| Ca, wt.% | 0.609 | 0.023 | 0.563 | 0.654 | 0.541 | 0.676 | 3.71% | 7.41% | 11.12% | 0.578 | 0.639 |
| Cd, ppm | 0.090 | 0.032 | 0.025 | 0.155 | 0.000 | 0.187 | 35.96% | 71.93% | 107.9% | 0.086 | 0.095 |
| Ce, ppm | 29.8 | 2.12 | 25.5 | 34.0 | 23.4 | 36.1 | 7.12% | 14.23% | 21.35% | 28.3 | 31.2 |
| Co, ppm | 2.99 | 0.147 | 2.70 | 3.29 | 2.55 | 3.44 | 4.92% | 9.85% | 14.77% | 2.84 | 3.14 |
| Cr, ppm | 17.9 | 3.3 | 11.4 | 24.5 | 8.2 | 27.7 | 18.14% | 36.27% | 54.41% | 17.1 | 18.8 |
| Cs, ppm | 39.2 | 1.34 | 36.5 | 41.9 | 35.2 | 43.2 | 3.42% | 6.83% | 10.25% | 37.2 | 41.2 |
| Cu, ppm | 10.7 | 0.93 | 8.9 | 12.6 | 7.9 | 13.5 | 8.70% | 17.39% | 26.09% | 10.2 | 11.3 |
| Dy, ppm | 1.52 | 0.145 | 1.23 | 1.81 | 1.09 | 1.96 | 9.51% | 19.01% | 28.52% | 1.45 | 1.60 |
| Er, ppm | 0.59 | 0.047 | 0.50 | 0.68 | 0.45 | 0.73 | 7.87% | 15.73% | 23.60% | 0.56 | 0.62 |
| Eu, ppm | 0.52 | 0.06 | 0.40 | 0.63 | 0.35 | 0.69 | 11.02% | 22.04% | 33.06% | 0.49 | 0.54 |
| Fe wt.% | 1.07 | 0.038 | 1.00 | 1.15 | 0.96 | 1.19 | 3.50% | 6.99% | 10.49% | 1.02 | 1.13 |
| Ga, ppm | 16.8 | 0.93 | 15.0 | 18.7 | 14.1 | 19.6 | 5.52% | 11.04% | 16.55% | 16.0 | 17.7 |
| Gd, ppm | 2.28 | 0.121 | 2.04 | 2.52 | 1.92 | 2.64 | 5.29% | 10.58% | 15.88% | 2.17 | 2.40 |
| Hf, ppm | 1.21 | 0.091 | 1.03 | 1.39 | 0.94 | 1.48 | 7.49% | 14.97% | 22.46% | 1.15 | 1.27 |
| Ho, ppm | 0.24 | 0.013 | 0.21 | 0.27 | 0.20 | 0.28 | 5.48% | 10.97% | 16.45% | 0.23 | 0.25 |
| In, ppm | 0.021 | 0.004 | 0.013 | 0.029 | 0.009 | 0.033 | 18.66% | 37.32% | 55.99% | 0.020 | 0.022 |

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.

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 5 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 |
| **4-Acid Digestion continued** | | | | | | | | | | | |
| K, wt.% | 1.15 | 0.041 | 1.07 | 1.23 | 1.03 | 1.27 | 3.56% | 7.12% | 10.68% | 1.09 | 1.21 |
| La, ppm | 14.4 | 1.14 | 12.1 | 16.7 | 11.0 | 17.8 | 7.91% | 15.81% | 23.72% | 13.7 | 15.1 |
| Li, wt.% | 0.245 | 0.007 | 0.231 | 0.259 | 0.224 | 0.266 | 2.83% | 5.67% | 8.50% | 0.233 | 0.257 |
| $Li_2O$, wt.% | 0.527 | 0.015 | 0.497 | 0.557 | 0.482 | 0.572 | 2.83% | 5.67% | 8.50% | 0.501 | 0.553 |
| Lu, ppm | 0.065 | 0.009 | 0.048 | 0.083 | 0.039 | 0.092 | 13.58% | 27.16% | 40.73% | 0.062 | 0.069 |
| Mg, wt.% | 0.267 | 0.012 | 0.243 | 0.290 | 0.231 | 0.302 | 4.44% | 8.87% | 13.31% | 0.253 | 0.280 |
| Mn, wt.% | 0.039 | 0.001 | 0.036 | 0.041 | 0.035 | 0.043 | 3.35% | 6.70% | 10.04% | 0.037 | 0.041 |
| Mo, ppm | 1.60 | 0.088 | 1.42 | 1.78 | 1.34 | 1.86 | 5.49% | 10.97% | 16.46% | 1.52 | 1.68 |
| Na, wt.% | 0.755 | 0.034 | 0.686 | 0.823 | 0.652 | 0.857 | 4.54% | 9.07% | 13.61% | 0.717 | 0.792 |
| Nb, ppm | 28.4 | 1.51 | 25.4 | 31.5 | 23.9 | 33.0 | 5.31% | 10.61% | 15.92% | 27.0 | 29.9 |
| Nd, ppm | 13.5 | 0.99 | 11.5 | 15.5 | 10.5 | 16.5 | 7.31% | 14.62% | 21.93% | 12.8 | 14.2 |
| Ni, ppm | 7.66 | 0.329 | 7.00 | 8.32 | 6.67 | 8.65 | 4.30% | 8.60% | 12.89% | 7.28 | 8.05 |
| P, wt.% | 0.039 | 0.002 | 0.035 | 0.044 | 0.033 | 0.046 | 5.67% | 11.33% | 17.00% | 0.037 | 0.041 |
| Pb, ppm | 9.55 | 0.436 | 8.68 | 10.42 | 8.24 | 10.86 | 4.57% | 9.14% | 13.71% | 9.07 | 10.03 |
| Pr, ppm | 3.65 | 0.229 | 3.19 | 4.11 | 2.96 | 4.34 | 6.28% | 12.55% | 18.83% | 3.47 | 3.83 |
| Rb, ppm | 288 | 15 | 258 | 317 | 243 | 332 | 5.17% | 10.34% | 15.51% | 273 | 302 |
| Re, ppm | < 0.002 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| S, wt.% | 0.045 | 0.004 | 0.037 | 0.053 | 0.032 | 0.058 | 9.42% | 18.83% | 28.25% | 0.043 | 0.047 |
| Sb, ppm | 0.39 | 0.04 | 0.30 | 0.47 | 0.26 | 0.51 | 10.77% | 21.54% | 32.31% | 0.37 | 0.41 |
| Sc, ppm | 3.20 | 0.244 | 2.71 | 3.69 | 2.47 | 3.93 | 7.61% | 15.23% | 22.84% | 3.04 | 3.36 |
| Sm, ppm | 2.69 | 0.180 | 2.33 | 3.05 | 2.15 | 3.23 | 6.69% | 13.39% | 20.08% | 2.55 | 2.82 |
| Sn, ppm | 25.8 | 1.81 | 22.1 | 29.4 | 20.3 | 31.2 | 7.03% | 14.06% | 21.09% | 24.5 | 27.1 |
| Sr, ppm | 60 | 3.0 | 54 | 66 | 51 | 69 | 4.97% | 9.94% | 14.91% | 57 | 63 |
| Ta, ppm | 64 | 5.5 | 53 | 75 | 48 | 81 | 8.48% | 16.95% | 25.43% | 61 | 68 |
| Tb, ppm | 0.31 | 0.023 | 0.26 | 0.35 | 0.24 | 0.38 | 7.60% | 15.20% | 22.80% | 0.29 | 0.32 |
| Te, ppm | < 0.05 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| Th, ppm | 5.94 | 0.445 | 5.05 | 6.83 | 4.60 | 7.28 | 7.50% | 14.99% | 22.49% | 5.64 | 6.24 |
| Ti, wt.% | 0.151 | 0.006 | 0.139 | 0.162 | 0.133 | 0.168 | 3.92% | 7.85% | 11.77% | 0.143 | 0.158 |
| Tl, ppm | 1.93 | 0.105 | 1.72 | 2.14 | 1.62 | 2.25 | 5.43% | 10.86% | 16.30% | 1.83 | 2.03 |
| Tm, ppm | 0.071 | 0.009 | 0.053 | 0.089 | 0.044 | 0.098 | 12.46% | 24.92% | 37.38% | 0.067 | 0.075 |
| U, ppm | 1.91 | 0.161 | 1.59 | 2.24 | 1.43 | 2.40 | 8.43% | 16.85% | 25.28% | 1.82 | 2.01 |
| V, ppm | 22.8 | 0.94 | 20.9 | 24.7 | 20.0 | 25.6 | 4.13% | 8.26% | 12.39% | 21.6 | 23.9 |
| W, ppm | 5.08 | 0.471 | 4.14 | 6.02 | 3.67 | 6.49 | 9.27% | 18.54% | 27.81% | 4.83 | 5.34 |
| Y, ppm | 6.13 | 0.316 | 5.50 | 6.76 | 5.18 | 7.08 | 5.15% | 10.31% | 15.46% | 5.83 | 6.44 |
| Yb, ppm | 0.46 | 0.05 | 0.36 | 0.57 | 0.31 | 0.62 | 11.17% | 22.34% | 33.51% | 0.44 | 0.49 |
| Zn, ppm | 60 | 3.5 | 53 | 67 | 49 | 70 | 5.84% | 11.69% | 17.53% | 57 | 63 |
| Zr, ppm | 27.8 | 2.28 | 23.3 | 32.4 | 21.0 | 34.7 | 8.19% | 16.39% | 24.58% | 26.4 | 29.2 |
| **Borate / Peroxide Fusion ICP** | | | | | | | | | | | |
| Al, wt.% | 4.02 | 0.139 | 3.74 | 4.29 | 3.60 | 4.43 | 3.45% | 6.90% | 10.35% | 3.82 | 4.22 |
| Ba, ppm | 371 | 12 | 348 | 394 | 336 | 406 | 3.12% | 6.24% | 9.37% | 352 | 389 |
| Be, ppm | 3.88 | 0.62 | 2.63 | 5.13 | 2.01 | 5.75 | 16.06% | 32.12% | 48.18% | 3.69 | 4.08 |
| Bi, ppm | 0.46 | 0.12 | 0.22 | 0.70 | 0.10 | 0.81 | 25.82% | 51.65% | 77.47% | 0.44 | 0.48 |
| Ca, wt.% | 0.601 | 0.036 | 0.529 | 0.672 | 0.494 | 0.708 | 5.94% | 11.87% | 17.81% | 0.571 | 0.631 |

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.

**Table 5 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 |
| **Borate / Peroxide Fusion ICP continued** | | | | | | | | | | | |
| Ce, ppm | 29.6 | 1.75 | 26.1 | 33.1 | 24.3 | 34.8 | 5.91% | 11.83% | 17.74% | 28.1 | 31.0 |
| Co, ppm | 3.13 | 0.296 | 2.54 | 3.73 | 2.25 | 4.02 | 9.45% | 18.89% | 28.34% | 2.98 | 3.29 |
| Cs, ppm | 39.1 | 1.61 | 35.8 | 42.3 | 34.2 | 43.9 | 4.13% | 8.26% | 12.38% | 37.1 | 41.0 |
| Dy, ppm | 2.21 | 0.122 | 1.97 | 2.46 | 1.85 | 2.58 | 5.52% | 11.04% | 16.56% | 2.10 | 2.32 |
| Er, ppm | 1.13 | 0.084 | 0.97 | 1.30 | 0.88 | 1.38 | 7.40% | 14.80% | 22.20% | 1.08 | 1.19 |
| Eu, ppm | 0.52 | 0.046 | 0.43 | 0.61 | 0.38 | 0.65 | 8.81% | 17.62% | 26.43% | 0.49 | 0.54 |
| Fe, wt.% | 1.09 | 0.030 | 1.03 | 1.15 | 1.00 | 1.18 | 2.74% | 5.47% | 8.21% | 1.03 | 1.14 |
| Ga, ppm | 16.9 | 0.83 | 15.3 | 18.6 | 14.4 | 19.4 | 4.91% | 9.82% | 14.73% | 16.1 | 17.8 |
| Gd, ppm | 2.64 | 0.256 | 2.13 | 3.16 | 1.88 | 3.41 | 9.69% | 19.39% | 29.08% | 2.51 | 2.78 |
| Ge, ppm | 1.49 | 0.43 | 0.62 | 2.36 | 0.18 | 2.79 | 29.22% | 58.43% | 87.65% | 1.41 | 1.56 |
| Ho, ppm | 0.40 | 0.029 | 0.34 | 0.46 | 0.31 | 0.49 | 7.36% | 14.72% | 22.08% | 0.38 | 0.42 |
| In, ppm | < 0.2 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| K, wt.% | 1.18 | 0.052 | 1.08 | 1.29 | 1.03 | 1.34 | 4.39% | 8.79% | 13.18% | 1.12 | 1.24 |
| La, ppm | 14.6 | 0.94 | 12.7 | 16.5 | 11.8 | 17.5 | 6.46% | 12.92% | 19.39% | 13.9 | 15.4 |
| Li, wt.% | 0.246 | 0.009 | 0.227 | 0.264 | 0.218 | 0.273 | 3.77% | 7.53% | 11.30% | 0.233 | 0.258 |
| $Li_2O$, wt.% | 0.529 | 0.020 | 0.489 | 0.569 | 0.469 | 0.589 | 3.77% | 7.53% | 11.30% | 0.502 | 0.555 |
| Lu, ppm | 0.16 | 0.02 | 0.11 | 0.20 | 0.09 | 0.22 | 13.67% | 27.34% | 41.02% | 0.15 | 0.17 |
| Mg, wt.% | 0.266 | 0.013 | 0.239 | 0.293 | 0.226 | 0.306 | 5.01% | 10.03% | 15.04% | 0.253 | 0.279 |
| Mn, wt.% | 0.039 | 0.001 | 0.037 | 0.041 | 0.036 | 0.042 | 2.28% | 4.55% | 6.83% | 0.037 | 0.041 |
| Nb, ppm | 29.3 | 1.23 | 26.9 | 31.8 | 25.6 | 33.0 | 4.18% | 8.36% | 12.54% | 27.8 | 30.8 |
| Nd, ppm | 13.2 | 0.76 | 11.7 | 14.8 | 10.9 | 15.5 | 5.76% | 11.52% | 17.28% | 12.6 | 13.9 |
| P, wt.% | 0.039 | 0.003 | 0.034 | 0.044 | 0.031 | 0.047 | 6.94% | 13.89% | 20.83% | 0.037 | 0.041 |
| Pb, ppm | 9.64 | 0.925 | 7.79 | 11.49 | 6.87 | 12.42 | 9.60% | 19.19% | 28.79% | 9.16 | 10.12 |
| Pr, ppm | 3.49 | 0.242 | 3.00 | 3.97 | 2.76 | 4.21 | 6.93% | 13.86% | 20.79% | 3.31 | 3.66 |
| Rb, ppm | 287 | 12 | 263 | 310 | 252 | 321 | 4.05% | 8.09% | 12.14% | 272 | 301 |
| Re, ppm | < 0.1 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| Si, wt.% | 40.13 | 1.355 | 37.42 | 42.84 | 36.06 | 44.19 | 3.38% | 6.75% | 10.13% | 38.12 | 42.13 |
| Sm, ppm | 2.79 | 0.176 | 2.44 | 3.14 | 2.26 | 3.32 | 6.30% | 12.61% | 18.91% | 2.65 | 2.93 |
| Sn, ppm | 38.7 | 2.18 | 34.4 | 43.1 | 32.2 | 45.3 | 5.62% | 11.24% | 16.87% | 36.8 | 40.7 |
| Sr, ppm | 62 | 8 | 47 | 77 | 39 | 85 | 12.39% | 24.77% | 37.16% | 59 | 65 |
| Ta, ppm | 69 | 3.9 | 61 | 76 | 57 | 80 | 5.65% | 11.30% | 16.96% | 65 | 72 |
| Tb, ppm | 0.40 | 0.021 | 0.35 | 0.44 | 0.33 | 0.46 | 5.36% | 10.71% | 16.07% | 0.38 | 0.42 |
| Th, ppm | 5.96 | 0.245 | 5.47 | 6.45 | 5.22 | 6.69 | 4.11% | 8.22% | 12.32% | 5.66 | 6.25 |
| Ti, wt.% | 0.154 | 0.008 | 0.138 | 0.170 | 0.131 | 0.177 | 5.07% | 10.14% | 15.21% | 0.146 | 0.162 |
| Tl, ppm | 1.96 | 0.096 | 1.77 | 2.16 | 1.67 | 2.25 | 4.90% | 9.80% | 14.70% | 1.87 | 2.06 |
| Tm, ppm | 0.16 | 0.013 | 0.14 | 0.19 | 0.12 | 0.20 | 7.88% | 15.76% | 23.65% | 0.15 | 0.17 |
| U, ppm | 2.28 | 0.31 | 1.65 | 2.90 | 1.33 | 3.22 | 13.80% | 27.61% | 41.41% | 2.16 | 2.39 |
| V, ppm | 22.8 | 2.6 | 17.6 | 28.0 | 15.0 | 30.6 | 11.42% | 22.83% | 34.25% | 21.6 | 23.9 |
| W, ppm | 5.54 | 0.76 | 4.02 | 7.06 | 3.26 | 7.82 | 13.72% | 27.44% | 41.16% | 5.26 | 5.82 |
| Y, ppm | 11.4 | 0.82 | 9.7 | 13.0 | 8.9 | 13.8 | 7.24% | 14.49% | 21.73% | 10.8 | 11.9 |
| Yb, ppm | 1.04 | 0.13 | 0.78 | 1.29 | 0.66 | 1.42 | 12.19% | 24.38% | 36.57% | 0.99 | 1.09 |
| Zn, ppm | 57 | 10 | 37 | 76 | 27 | 86 | 17.20% | 34.39% | 51.59% | 54 | 59 |

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.

**Table 5 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 |
| **Borate Fusion XRF** | | | | | | | | | | | |
| $Al_2O_3$, wt.% | 7.63 | 0.117 | 7.40 | 7.87 | 7.28 | 7.98 | 1.54% | 3.08% | 4.62% | 7.25 | 8.01 |
| BaO, ppm | 407 | 43 | 322 | 493 | 279 | 536 | 10.49% | 20.98% | 31.47% | 387 | 428 |
| CaO, wt.% | 0.851 | 0.016 | 0.819 | 0.884 | 0.803 | 0.900 | 1.91% | 3.83% | 5.74% | 0.809 | 0.894 |
| $Cr_2O_3$, ppm | < 100 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| $Fe_2O_3$, wt.% | 1.56 | 0.027 | 1.51 | 1.62 | 1.48 | 1.64 | 1.76% | 3.52% | 5.28% | 1.48 | 1.64 |
| $K_2O$, wt.% | 1.39 | 0.016 | 1.36 | 1.42 | 1.34 | 1.44 | 1.13% | 2.26% | 3.38% | 1.32 | 1.46 |
| MgO, wt.% | 0.452 | 0.023 | 0.406 | 0.499 | 0.382 | 0.522 | 5.15% | 10.30% | 15.44% | 0.430 | 0.475 |
| MnO, wt.% | 0.050 | 0.002 | 0.047 | 0.054 | 0.045 | 0.055 | 3.20% | 6.40% | 9.61% | 0.048 | 0.053 |
| $Na_2O$, wt.% | 1.01 | 0.037 | 0.94 | 1.09 | 0.90 | 1.12 | 3.67% | 7.34% | 11.00% | 0.96 | 1.06 |
| Nb, ppm | < 50 | IND | IND | IND | IND | IND | IND | IND | IND | IND | IND |
| $P_2O_5$, wt.% | 0.088 | 0.007 | 0.075 | 0.101 | 0.068 | 0.108 | 7.50% | 14.99% | 22.49% | 0.084 | 0.092 |
| Rb, ppm | 259 | 21 | 218 | 300 | 198 | 321 | 7.92% | 15.83% | 23.75% | 246 | 272 |
| $SiO_2$, wt.% | 85.46 | 0.785 | 83.89 | 87.03 | 83.11 | 87.82 | 0.92% | 1.84% | 2.75% | 81.19 | 89.74 |
| $SO_3$, wt.% | 0.105 | 0.014 | 0.077 | 0.133 | 0.063 | 0.148 | 13.48% | 26.96% | 40.44% | 0.100 | 0.110 |
| SrO, ppm | 96 | 19 | 57 | 134 | 38 | 153 | 19.97% | 39.93% | 59.90% | 91 | 100 |
| $TiO_2$, wt.% | 0.257 | 0.009 | 0.239 | 0.274 | 0.231 | 0.283 | 3.39% | 6.78% | 10.16% | 0.244 | 0.269 |
| **Thermogravimetry** | | | | | | | | | | | |
| $LOI^{1000}$, wt.% | 0.570 | 0.106 | 0.358 | 0.782 | 0.252 | 0.887 | 18.57% | 37.14% | 55.71% | 0.541 | 0.598 |

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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1. Actlabs, Ancaster, Ontario, Canada
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3. AGAT Laboratories, Calgary, Alberta, Canada
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5. ALS, Lima, Peru
6. ALS, Loughrea, Galway, Ireland
7. ALS, Malaga, WA, Australia
8. ALS, Vancouver, BC, Canada
9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
11. CERTIMIN, Lima, Peru
12. CRS Laboratories Oy, Kempele, Northern Ostrobothnia, Finland
13. Inspectorate (BV), Lima, Peru
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15. Intertek, Perth, WA, Australia
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***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.***

## PREPARER AND SUPPLIER

Certified reference material OREAS 750b is prepared, certified and supplied by:

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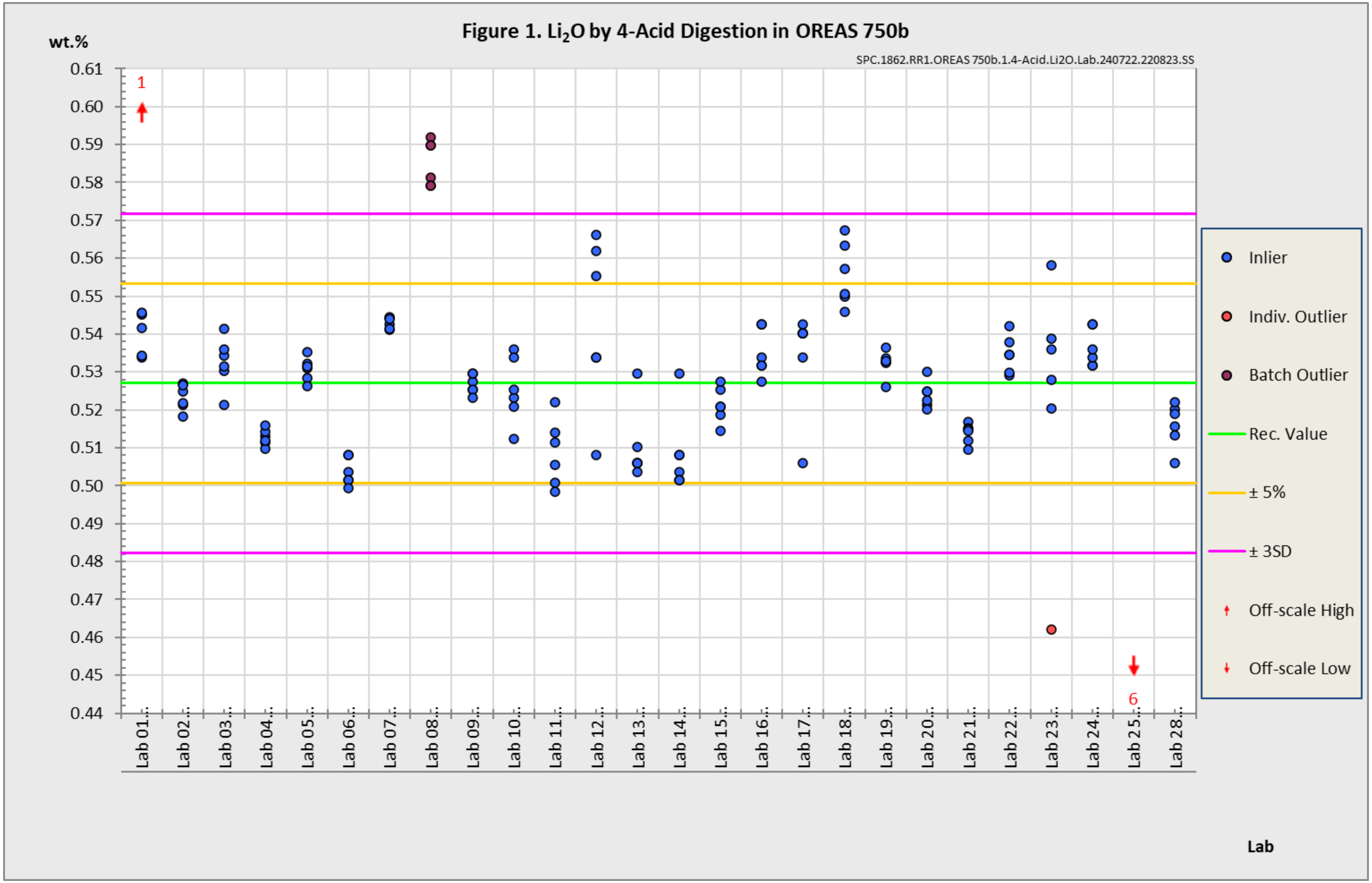

Figure 1. Li2O by 4-Acid Digestion in OREAS 750b
wt.%
SPC.1862.RR1.OREAS 750b.1.4-Acid.Li2O.Lab.240722.220823.SS
0.61
0.60
0.59
0.58
0.57
0.56
0.55
0.54
0.53
0.52
0.51
0.50
0.49
0.48
0.47
0.46
0.45
0.44
1
6
Lab 01...
Lab 02...
Lab 03...
Lab 04...
Lab 05...
Lab 06...
Lab 07...
Lab 08...
Lab 09...
Lab 10...
Lab 11...
Lab 12...
Lab 13...
Lab 14...
Lab 15...
Lab 16...
Lab 17...
Lab 18...
Lab 19...
Lab 20...
Lab 21...
Lab 22...
Lab 23...
Lab 24...
Lab 25...
Lab 28...
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale High
Off-scale Low
Lab

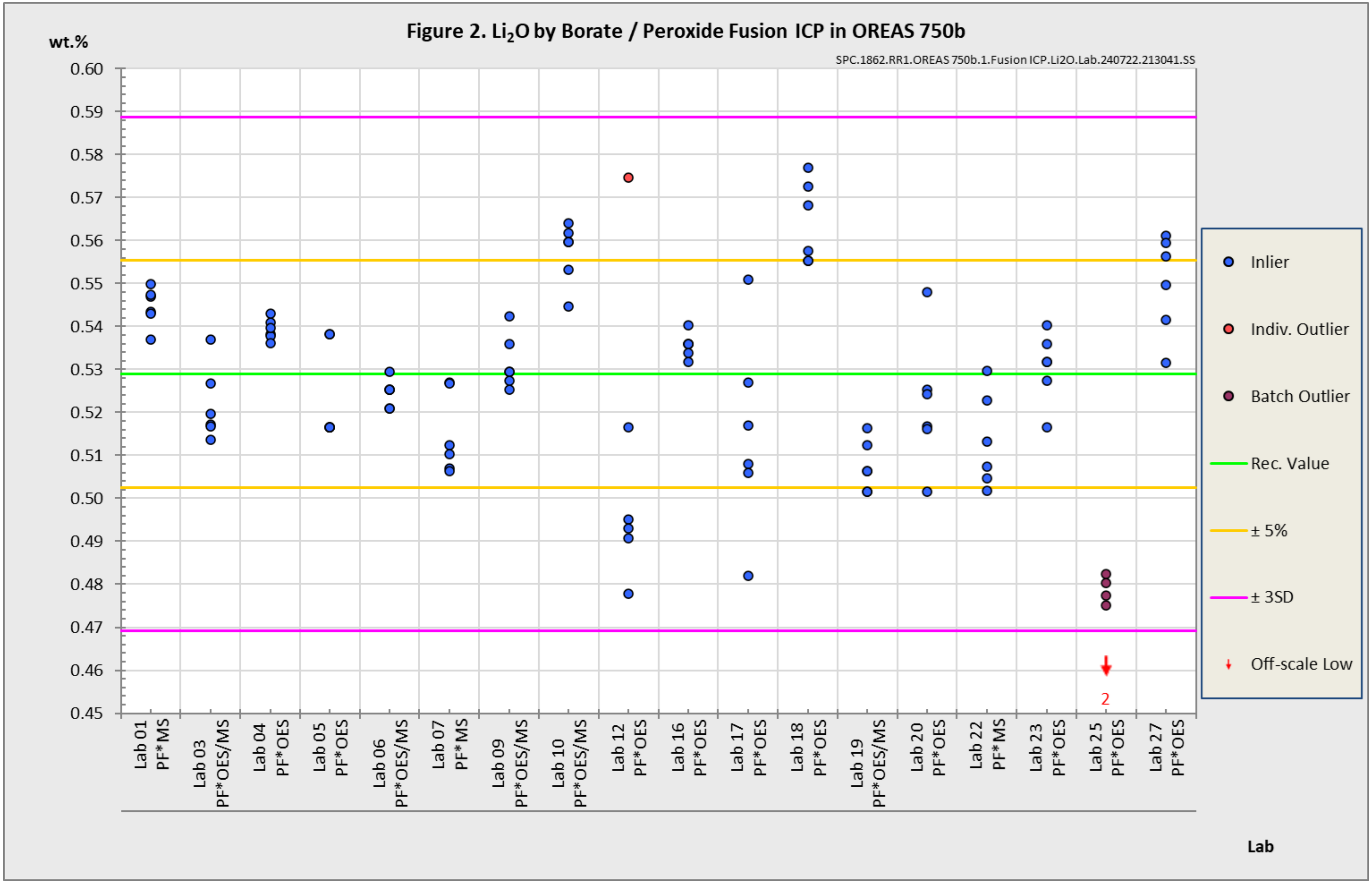
Figure 2. $Li_2O$ by Borate / Peroxide Fusion ICP in OREAS 750b
wt.%
SPC.1862.RR1.OREAS 750b.1.Fusion ICP.Li2O.Lab.240722.213041.SS
0.60
0.59
0.58
0.57
0.56
0.55
0.54
0.53
0.52
0.51
0.50
0.49
0.48
0.47
0.46
0.45
Lab 01 PF*MS
Lab 03 PF*OES/MS
Lab 04 PF*OES
Lab 05 PF*OES
Lab 06 PF*OES/MS
Lab 07 PF*MS
Lab 09 PF*OES/MS
Lab 10 PF*OES/MS
Lab 12 PF*OES
Lab 16 PF*OES
Lab 17 PF*OES
Lab 18 PF*OES
Lab 19 PF*OES/MS
Lab 20 PF*OES
Lab 22 PF*MS
Lab 23 PF*OES
Lab 25 PF*OES
Lab 27 PF*OES
2
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale Low
Lab

## 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)) [14]. 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 representativeness 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 [8], 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 participating laboratories were chosen on the basis of their competence (from past performance in interlaboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. These laboratories are accredited to ISO 17025 for 4-acid digestion (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016 [7], 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 33405:2024-05, 9.2.4c) [4]."* 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 (fusion/digestion) 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 the field samples being analysed.

# INTENDED USE

OREAS 750b 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 750b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 750b is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 and 2 in geological samples;
- For the verification of analytical methods for analytes reported in Tables 1 and 2;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 and 2. When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. Users can determine an approximation of the standard uncertainty by calculating one fourth of the width of the Expanded Uncertainty interval given in this certificate (Expanded Uncertainty intervals are provided in Tables 1 and 2).

# MINIMUM SAMPLE SIZE

To relate analytical determinations to the values in this certificate, the minimum mass of sample used should match the typical mass that the laboratories used in the interlaboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Lithium Borate / Sodium peroxide fusion with ICP-OES and/or MS finish: ≥0.2 g;
- Borate fusion with X-ray fluorescence finish: ≥0.2 g;
- Loss on Ignition (LOI) at 1000°C: ≥1 g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish: ≥0.25 g.

# PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 750b remains valid, within the specified measurement uncertainties, until at least February 2039, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

Store in a clean and cool dry place away from direct sunlight.

Long-term stability will be monitored at appropriate intervals and purchasers notified if any changes are observed. The period of validity may well be indefinite and will be reassessed prior to expiry with the aim of extending the validity if possible.

### Single-use sachets

OREAS 750b is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

### Repeat-use packaging (e.g., 1 kg plastic jars)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 750b contains a non-hygroscopic* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (~0.04 wt.% S).

*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorped moisture (weakly held $H_2O$- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2mm thick) layer and left exposed for a period of 2 hours.

## INSTRUCTIONS FOR HANDLING & CORRECT USE

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [12].

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised.

### 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 interlaboratory 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 5 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 % expanded uncertainty then generally there is no cause for concern in regard to bias.

## 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

## DOCUMENT HISTORY

| Revision No. | Date | Changes applied |
|---|---|---|
| 1 | 28th October, 2024 | Changed $Fe_2O_3$ to Fe for 4-Acid Digestion and Borate / Peroxide Fusion ICP data in Table 1, 2 & 5. |
| 0 | 9th September, 2024 | First publication. |

## CERTIFYING OFFICER

28th October, 2024

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

## QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016.

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.

## REFERENCES

[1] Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).

[2] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.

[3] ISO Guide 33401:2024-01. Reference materials – Contents of certificates, labels and accompanying documentation.

[4] ISO Guide 33405:2024-05. Reference materials – Approaches for characterization and assessment of homogeneity and stability.

[5] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[6] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.

[7] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.

[8] ISO 17025:2017, General requirements for the competence of testing and calibration laboratories.

[9] ISO 17034:2016. General requirements for the competence of reference material producers.

[10] Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), Minnesota (USA).

[11] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.

[12] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.

[13] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.

[14] Thompson, A.; Taylor, B.N. (2008); Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Nov 2021).

[15] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.