ORE RESEARCH & EXPLORATION P/L ABN 28 006 859 856
37A Hosie Street · Bayswater North · VIC 3153 · AUSTRALIA
61 3 9729 0333 Fax 61 3 9729 8338
info@ore.com.au www.ore.com.au

**CERTIFICATE OF ANALYSIS FOR**

# SEDEX TYPE Zn-Pb-Ag ORE

# CERTIFIED REFERENCE MATERIAL

# OREAS 139

**Summary Statistics for Key Analytes.**

| Constituent | Certified Value | 1SD | 95% Confidence Limits | | 95% Tolerance Limits | |
|---|---|---|---|---|---|---|
| | | | Low | High | Low | High |
| **4-Acid Digestion** | | | | | | |
| Ag, Silver (ppm) | 76.7 | 3.91 | 75.0 | 78.3 | 74.9 | 78.5 |
| Pb, Lead (wt.%) | 2.21 | 0.095 | 2.17 | 2.25 | 2.16 | 2.26 |
| Zn, Zinc (wt.%) | 13.63 | 0.305 | 13.49 | 13.77 | 13.41 | 13.85 |

Note: intervals may appear asymmetric due to rounding.

**Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 139.**

| Constituent | Certified Value | 1SD | 95% Confidence Limits | | 95% Tolerance Limits | |
|---|---|---|---|---|---|---|
| | | | Low | High | Low | High |
| **4-Acid Digestion** | | | | | | |
| Ag, Silver (ppm) | 76.7 | 3.91 | 75.0 | 78.3 | 74.9 | 78.5 |
| Al, Aluminium (wt.%) | 3.62 | 0.270 | 3.49 | 3.75 | 3.53 | 3.71 |
| As, Arsenic (ppm) | 315 | 13 | 309 | 321 | 302 | 328 |
| Be, Beryllium (ppm) | 2.87 | 0.197 | 2.76 | 2.98 | 2.72 | 3.01 |
| Bi, Bismuth (ppm) | 6.83 | 0.542 | 6.55 | 7.12 | 6.64 | 7.03 |
| Ca, Calcium (wt.%) | 1.15 | 0.055 | 1.13 | 1.18 | 1.13 | 1.18 |
| Cd, Cadmium (ppm) | 278 | 25 | 264 | 292 | 270 | 286 |
| Ce, Cerium (ppm) | 33.8 | 7.3 | 29.3 | 38.3 | 32.3 | 35.3 |
| Co, Cobalt (ppm) | 25.6 | 1.86 | 24.6 | 26.6 | 24.7 | 26.5 |
| Cr, Chromium (ppm) | 29.4 | 4.0 | 26.7 | 32.0 | 27.7 | 31.0 |
| Cs, Cesium (ppm) | 3.22 | 0.286 | 3.04 | 3.40 | 3.10 | 3.34 |
| Cu, Copper (ppm) | 271 | 8 | 267 | 276 | 264 | 279 |
| Dy, Dysprosium (ppm) | 2.81 | 0.093 | 2.76 | 2.87 | 2.70 | 2.92 |
| Er, Erbium (ppm) | 1.51 | 0.082 | 1.43 | 1.59 | 1.43 | 1.59 |
| Eu, Europium (ppm) | 0.71 | 0.13 | 0.59 | 0.83 | 0.65 | 0.77 |
| Fe, Iron (wt.%) | 11.79 | 0.663 | 11.48 | 12.10 | 11.48 | 12.10 |
| Ga, Gallium (ppm) | 10.2 | 0.81 | 9.6 | 10.8 | 9.7 | 10.7 |
| Gd, Gadolinium (ppm) | 3.63 | 0.345 | 3.33 | 3.92 | 3.46 | 3.80 |
| Hf, Hafnium (ppm) | 2.29 | 0.24 | 2.13 | 2.44 | 2.20 | 2.37 |
| Ho, Holmium (ppm) | 0.53 | 0.027 | 0.50 | 0.56 | 0.50 | 0.56 |
| In, Indium (ppm) | 0.73 | 0.071 | 0.69 | 0.77 | 0.70 | 0.76 |
| K, Potassium (wt.%) | 3.19 | 0.202 | 3.10 | 3.28 | 3.10 | 3.28 |
| La, Lanthanum (ppm) | 13.7 | 2.7 | 12.0 | 15.4 | 13.0 | 14.4 |
| Li, Lithium (ppm) | 38.9 | 3.56 | 37.1 | 40.7 | 37.1 | 40.6 |
| Lu, Lutetium (ppm) | 0.22 | 0.03 | 0.20 | 0.25 | 0.21 | 0.24 |
| Mg, Magnesium (wt.%) | 0.491 | 0.054 | 0.467 | 0.515 | 0.479 | 0.502 |
| Mn, Manganese (wt.%) | 0.663 | 0.027 | 0.650 | 0.676 | 0.653 | 0.672 |
| Mo, Molybdenum (ppm) | 9.82 | 0.752 | 9.34 | 10.31 | 9.32 | 10.33 |
| Na, Sodium (wt.%) | 0.108 | 0.010 | 0.103 | 0.114 | 0.103 | 0.113 |
| Nb, Niobium (ppm) | 5.76 | 0.85 | 5.22 | 6.30 | 5.56 | 5.96 |
| Nd, Neodymium (ppm) | 17.8 | 2.2 | 16.2 | 19.5 | 17.1 | 18.6 |
| Ni, Nickel (ppm) | 35.9 | 3.05 | 34.3 | 37.5 | 34.7 | 37.2 |
| P, Phosphorus (wt.%) | 0.089 | 0.005 | 0.087 | 0.092 | 0.087 | 0.092 |
| Pb, Lead (wt.%) | 2.21 | 0.095 | 2.17 | 2.25 | 2.16 | 2.26 |
| Pr, Praseodymium (ppm) | 4.72 | 0.323 | 4.39 | 5.04 | 4.46 | 4.97 |
| Rb, Rubidium (ppm) | 142 | 11 | 135 | 149 | 138 | 146 |
| S, Sulphur (wt.%) | 16.02 | 0.481 | 15.79 | 16.25 | 15.61 | 16.44 |
| Sb, Antimony (ppm) | 58 | 5.2 | 55 | 61 | 56 | 60 |
| Sc, Scandium (ppm) | 5.04 | 0.462 | 4.67 | 5.40 | 4.82 | 5.26 |
| Se, Selenium (ppm) | 2.78 | 0.44 | 2.40 | 3.16 | 2.55 | 3.00 |
| Sm, Samarium (ppm) | 4.01 | 0.222 | 3.81 | 4.20 | 3.83 | 4.19 |
| Sn, Tin (ppm) | 2.27 | 0.24 | 2.10 | 2.43 | 2.06 | 2.48 |

Note: intervals may appear asymmetric due to rounding

**Table 1 continued.**

| Constituent | Certified Value | 1SD | 95% Confidence Limits | | 95% Tolerance Limits | |
|---|---|---|---|---|---|---|
| | | | Low | High | Low | High |
| **4-Acid Digestion continued** | | | | | | |
| Sr, Strontium (ppm) | 331 | 65 | 297 | 364 | 312 | 349 |
| Tb, Terbium (ppm) | 0.47 | 0.028 | 0.45 | 0.50 | 0.45 | 0.49 |
| Ti, Titanium (wt.%) | 0.117 | 0.013 | 0.107 | 0.126 | 0.113 | 0.121 |
| Tl, Thallium (ppm) | 37.6 | 1.40 | 36.7 | 38.5 | 36.5 | 38.6 |
| Tm, Thulium (ppm) | 0.21 | 0.015 | 0.19 | 0.22 | 0.19 | 0.23 |
| U, Uranium (ppm) | 12.2 | 0.92 | 11.7 | 12.7 | 11.8 | 12.6 |
| V, Vanadium (ppm) | 69 | 4.2 | 67 | 72 | 67 | 71 |
| W, Tungsten (ppm) | 2.21 | 0.24 | 2.07 | 2.34 | 2.07 | 2.34 |
| Y, Yttrium (ppm) | 15.4 | 0.86 | 14.8 | 15.9 | 14.9 | 15.9 |
| Yb, Ytterbium (ppm) | 1.46 | 0.050 | 1.42 | 1.49 | 1.39 | 1.52 |
| Zn, Zinc (wt.%) | 13.63 | 0.305 | 13.49 | 13.77 | 13.41 | 13.85 |
| Zr, Zirconium (ppm) | 80 | 8 | 75 | 85 | 77 | 83 |
| **Peroxide Fusion ICP** | | | | | | |
| Al, Aluminium (wt.%) | 3.70 | 0.204 | 3.59 | 3.81 | 3.60 | 3.79 |
| As, Arsenic (ppm) | 332 | 24 | 317 | 347 | 312 | 353 |
| Be, Beryllium (ppm) | 3.17 | 0.51 | 2.81 | 3.54 | IND | IND |
| Bi, Bismuth (ppm) | 6.64 | 0.642 | 6.19 | 7.08 | 6.30 | 6.97 |
| Ca, Calcium (wt.%) | 1.20 | 0.093 | 1.15 | 1.25 | 1.14 | 1.26 |
| Cd, Cadmium (ppm) | 296 | 16 | 285 | 307 | 282 | 311 |
| Ce, Cerium (ppm) | 49.4 | 4.70 | 43.3 | 55.5 | 46.9 | 51.9 |
| Co, Cobalt (ppm) | 26.0 | 0.90 | 25.4 | 26.6 | 24.0 | 28.0 |
| Cs, Cesium (ppm) | 3.21 | 0.264 | 2.88 | 3.54 | 3.03 | 3.39 |
| Cu, Copper (ppm) | 274 | 25 | 263 | 285 | 258 | 290 |
| Er, Erbium (ppm) | 1.69 | 0.20 | 1.51 | 1.86 | IND | IND |
| Fe, Iron (wt.%) | 11.90 | 0.400 | 11.71 | 12.09 | 11.60 | 12.21 |
| Ga, Gallium (ppm) | 10.2 | 0.98 | 9.1 | 11.4 | IND | IND |
| In, Indium (ppm) | 0.69 | 0.10 | 0.62 | 0.76 | IND | IND |
| K, Potassium (wt.%) | 3.30 | 0.122 | 3.24 | 3.36 | 3.17 | 3.42 |
| La, Lanthanum (ppm) | 23.1 | 3.1 | 19.6 | 26.5 | 21.8 | 24.3 |
| Li, Lithium (ppm) | 40.4 | 3.23 | 37.9 | 42.8 | 37.7 | 43.1 |
| Mg, Magnesium (wt.%) | 0.501 | 0.022 | 0.491 | 0.511 | 0.483 | 0.519 |
| Mn, Manganese (wt.%) | 0.657 | 0.029 | 0.644 | 0.670 | 0.636 | 0.678 |
| Mo, Molybdenum (ppm) | 11.1 | 0.87 | 10.4 | 11.8 | IND | IND |
| Pb, Lead (wt.%) | 2.20 | 0.108 | 2.15 | 2.26 | 2.13 | 2.27 |
| Rb, Rubidium (ppm) | 145 | 8 | 138 | 152 | 141 | 149 |
| S, Sulphur (wt.%) | 16.04 | 0.418 | 15.82 | 16.26 | 15.64 | 16.43 |
| Sb, Antimony (ppm) | 63 | 6.1 | 58 | 67 | 59 | 66 |
| Si, Silicon (wt.%) | 16.34 | 0.383 | 16.13 | 16.55 | 16.07 | 16.61 |
| Sr, Strontium (ppm) | 479 | 54 | 442 | 516 | 463 | 494 |
| Tb, Terbium (ppm) | 0.50 | 0.09 | 0.38 | 0.61 | IND | IND |
| Th, Thorium (ppm) | 7.54 | 1.01 | 6.71 | 8.38 | 7.03 | 8.06 |
| Ti, Titanium (wt.%) | 0.157 | 0.009 | 0.152 | 0.161 | 0.150 | 0.163 |

Note: intervals may appear asymmetric due to rounding

**Table 1 continued.**

| Constituent | Certified Value | 1SD | 95% Confidence Limits | | 95% Tolerance Limits | |
|---|---|---|---|---|---|---|
| | | | Low | High | Low | High |
| **Peroxide Fusion ICP continued** | | | | | | |
| Tl, Thallium (ppm) | 35.4 | 2.43 | 33.4 | 37.5 | 33.9 | 36.9 |
| U, Uranium (ppm) | 12.2 | 0.63 | 11.8 | 12.6 | 11.3 | 13.1 |
| Y, Yttrium (ppm) | 17.1 | 1.62 | 15.3 | 18.8 | 16.2 | 18.0 |
| Zn, Zinc (wt.%) | 13.36 | 0.396 | 13.15 | 13.58 | 13.04 | 13.68 |
| **Aqua Regia Digestion** | | | | | | |
| Ag, Silver (ppm) | 78.2 | 2.35 | 77.2 | 79.3 | 75.7 | 80.8 |
| Al, Aluminium (wt.%) | 0.709 | 0.054 | 0.684 | 0.734 | 0.686 | 0.732 |
| As, Arsenic (ppm) | 322 | 24 | 311 | 333 | 311 | 333 |
| Be, Beryllium (ppm) | 1.45 | 0.27 | 1.21 | 1.68 | 1.37 | 1.52 |
| Bi, Bismuth (ppm) | 7.18 | 0.93 | 6.53 | 7.83 | 7.00 | 7.36 |
| Ca, Calcium (wt.%) | 1.14 | 0.091 | 1.11 | 1.18 | 1.11 | 1.18 |
| Cd, Cadmium (ppm) | 280 | 34 | 264 | 297 | 273 | 288 |
| Ce, Cerium (ppm) | 41.1 | 4.2 | 38.0 | 44.2 | 39.5 | 42.6 |
| Co, Cobalt (ppm) | 22.3 | 3.5 | 20.7 | 23.8 | 21.4 | 23.2 |
| Cr, Chromium (ppm) | 16.1 | 2.3 | 15.0 | 17.2 | 14.4 | 17.8 |
| Cs, Cesium (ppm) | 1.52 | 0.095 | 1.46 | 1.59 | 1.47 | 1.57 |
| Cu, Copper (ppm) | 269 | 10 | 265 | 274 | 261 | 278 |
| Fe, Iron (wt.%) | 11.65 | 0.470 | 11.42 | 11.88 | 11.35 | 11.95 |
| Ga, Gallium (ppm) | 4.78 | 0.432 | 4.44 | 5.11 | 4.60 | 4.96 |
| Gd, Gadolinium (ppm) | 3.21 | 0.50 | 2.52 | 3.90 | 3.10 | 3.32 |
| Hf, Hafnium (ppm) | 0.65 | 0.08 | 0.56 | 0.73 | 0.61 | 0.69 |
| Hg, Mercury (ppm) | 4.18 | 0.408 | 3.83 | 4.53 | 4.01 | 4.35 |
| In, Indium (ppm) | 0.66 | 0.07 | 0.61 | 0.72 | 0.61 | 0.72 |
| K, Potassium (wt.%) | 0.360 | 0.036 | 0.340 | 0.380 | 0.343 | 0.377 |
| La, Lanthanum (ppm) | 16.5 | 2.3 | 14.7 | 18.3 | 15.8 | 17.2 |
| Li, Lithium (ppm) | 23.8 | 3.8 | 21.1 | 26.5 | 22.7 | 24.9 |
| Lu, Lutetium (ppm) | 0.13 | 0.02 | 0.11 | 0.16 | IND | IND |
| Mg, Magnesium (wt.%) | 0.399 | 0.025 | 0.389 | 0.410 | 0.383 | 0.415 |
| Mn, Manganese (wt.%) | 0.618 | 0.022 | 0.607 | 0.629 | 0.598 | 0.638 |
| Mo, Molybdenum (ppm) | 9.89 | 0.663 | 9.47 | 10.32 | 9.63 | 10.16 |
| Nb, Niobium (ppm) | 0.66 | 0.08 | 0.55 | 0.77 | 0.61 | 0.70 |
| Nd, Neodymium (ppm) | 18.5 | 3.0 | 15.0 | 21.9 | 17.1 | 19.8 |
| Ni, Nickel (ppm) | 34.9 | 3.7 | 33.1 | 36.7 | 33.1 | 36.7 |
| P, Phosphorus (wt.%) | 0.090 | 0.006 | 0.088 | 0.093 | 0.087 | 0.093 |
| Pb, Lead (wt.%) | 2.22 | 0.067 | 2.19 | 2.26 | 2.16 | 2.29 |
| Rb, Rubidium (ppm) | 26.7 | 3.0 | 24.6 | 28.9 | 25.6 | 27.9 |
| S, Sulphur (wt.%) | 15.98 | 0.724 | 15.48 | 16.48 | 15.48 | 16.49 |
| Sb, Antimony (ppm) | 49.1 | 9.5 | 43.2 | 54.9 | 47.6 | 50.6 |
| Sc, Scandium (ppm) | 2.53 | 0.49 | 2.18 | 2.88 | 2.36 | 2.70 |
| Te, Tellurium (ppm) | 0.57 | 0.08 | 0.50 | 0.64 | 0.51 | 0.62 |
| Th, Thorium (ppm) | 6.53 | 0.91 | 5.92 | 7.15 | 6.29 | 6.78 |
| Ti, Titanium (wt.%) | 0.016 | 0.002 | 0.015 | 0.017 | 0.015 | 0.018 |

Note: intervals may appear asymmetric due to rounding

**Table 1 continued.**

| Constituent | Certified Value | 1SD | 95% Confidence Limits | | 95% Tolerance Limits | |
|---|---|---|---|---|---|---|
| | | | Low | High | Low | High |
| **Aqua Regia Digestion continued** | | | | | | |
| Tl, Thallium (ppm) | 8.91 | 0.835 | 8.32 | 9.50 | 8.65 | 9.17 |
| U, Uranium (ppm) | 11.4 | 0.38 | 11.2 | 11.6 | 10.9 | 12.0 |
| V, Vanadium (ppm) | 36.2 | 3.01 | 34.8 | 37.6 | 34.6 | 37.8 |
| W, Tungsten (ppm) | 1.77 | 0.20 | 1.64 | 1.90 | 1.68 | 1.85 |
| Y, Yttrium (ppm) | 12.0 | 0.75 | 11.4 | 12.5 | 11.6 | 12.4 |
| Yb, Ytterbium (ppm) | 0.95 | 0.12 | 0.81 | 1.08 | IND | IND |
| Zn, Zinc (wt.%) | 13.59 | 0.492 | 13.41 | 13.76 | 13.36 | 13.82 |
| Zr, Zirconium (ppm) | 20.4 | 3.7 | 17.2 | 23.6 | 19.4 | 21.4 |
| **Infrared Combustion** | | | | | | |
| C, Carbon (wt.%) | 3.83 | 0.100 | 3.78 | 3.88 | 3.79 | 3.87 |
| S, Sulphur (wt.%) | 16.52 | 0.271 | 16.41 | 16.63 | 16.36 | 16.68 |

Note: intervals may appear asymmetric due to rounding

## 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.

## SOURCE MATERIALS

OREAS 139 has been prepared from a blend of barren and ore grade SEDEX Type Zn-Pb-Ag materials sourced from the Dugald River deposit located in the Mt Isa Inlier, ~65km north-west of Cloncurry in north-west Queensland, Australia. The mineralisation style is dominated by sphalerite and galena with a gangue of graphitic slate, pyrrhotite and pyrite. The deposit is hosted within a sequence of upper greenschist to amphibolite facies metamorphic rocks consisting quartzite, schists, slates and dolomite.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 139 was prepared in the following manner:

- Drying to constant mass at 85°C;
- Crushing and milling to 98% minus 75 microns;
- Preliminary homogenisation and check assaying of barren, low, medium and high grade source materials;
- Final homogenisation by blending the source materials in specific ratios to achieve target grades;
- Packaging in 10g units sealed under nitrogen in laminated foil pouches.

# ANALYTICAL PROGRAM

Twenty four commercial analytical laboratories participated in the program to certify the analytes reported in Table 1. The following methods were employed:

- Four acid digestion for full ICP-OES and ICP-MS elemental suites except for one laboratory who used an AAS finish for Ag, five laboratories who used an AAS finish for Pb, two laboratories who used an AAS finish for S and nine laboratories who used an AAS finish for Zn (up to 23 laboratories depending on the element);
- Peroxide fusion for full ICP-OES and ICP-MS elemental suites except for one laboratory who used borate fusion with an ICP-OES finish for Si only (up to 18 laboratories depending on the element);
- Aqua regia digestion for full ICP-OES and ICP-MS elemental suites except for one laboratory who used an AAS finish for Ag, two laboratories who used an AAS finish for Pb and six laboratories who used an AAS finish for Zn (up to 20 laboratories depending on the element);
- C and S by IR combustion furnace (19 laboratories for C; 20 laboratories for S);

For the round robin program ten 300g test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire 265kg batch. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate 300g 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. Table 1 presents the 134 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 below shows 62 indicative values. Table 3 provides performance gate intervals for the certified values based on their associated pooled standard deviations. Tabulated results of all elements together with analytical method codes, 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 139 DataPack.xlsx**).

**Table 2. Indicative Values for OREAS 139.**

| Constituent | Unit | Value | Constituent | Unit | Value | Constituent | Unit | Value |
|---|---|---|---|---|---|---|---|---|
| **4-Acid Digestion** | | | | | | | | |
| B | ppm | *1.31* | Ir | ppm | *0.010* | Ta | ppm | *0.33* |
| Ba | ppm | *1710* | Pt | ppm | *0.005* | Te | ppm | *0.55* |
| Ge | ppm | *3.04* | Re | ppm | *0.013* | Th | ppm | *4.48* |
| Hg | ppm | *1.49* | Si | wt.% | *8.09* | | | |
| **Peroxide Fusion ICP** | | | | | | | | |
| Ag | ppm | *70.0* | Lu | ppm | *0.26* | Sn | ppm | *34.9* |
| B | ppm | *30.8* | Nb | ppm | *5.36* | Ta | ppm | *0.41* |
| Ba | ppm | *12460* | Nd | ppm | *21.8* | Te | ppm | *< 1* |
| Cr | ppm | *54* | Ni | ppm | *31.2* | Tm | ppm | *0.23* |
| Dy | ppm | *2.84* | P | wt.% | *0.098* | V | ppm | *79* |
| Eu | ppm | *0.68* | Pr | ppm | *5.70* | W | ppm | *1.96* |
| Gd | ppm | *3.85* | Re | ppm | *< 0.1* | Yb | ppm | *1.69* |

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

**Table 2 continued.**

| Constituent | Unit | Value | Constituent | Unit | Value | Constituent | Unit | Value |
|---|---|---|---|---|---|---|---|---|
| **Peroxide Fusion ICP continued** | | | | | | | | |
| Ge | ppm | *6.55* | Sc | ppm | *< 5* | Zr | ppm | *109* |
| Hf | ppm | *3.67* | Se | ppm | *25.8* | | | |
| Ho | ppm | *0.57* | Sm | ppm | *4.00* | | | |
| **Aqua Regia Digestion** | | | | | | | | |
| Au | ppm | *0.003* | Ir | ppm | *0.004* | Sm | ppm | *3.14* |
| B | ppm | *14.0* | Na | wt.% | *0.016* | Sn | ppm | *1.87* |
| Ba | ppm | *69* | Pd | ppm | *< 0.01* | Sr | ppm | *57* |
| Dy | ppm | *2.28* | Pr | ppm | *4.31* | Ta | ppm | *< 0.01* |
| Er | ppm | *1.08* | Pt | ppm | *4.82* | Tb | ppm | *0.41* |
| Eu | ppm | *0.54* | Re | ppm | *0.014* | Tm | ppm | *0.13* |
| Ge | ppm | *0.60* | Se | ppm | *3.44* | | | |
| Ho | ppm | *0.41* | Si | wt.% | *0.077* | | | |
| **Thermogravimetry** | | | | | | | | |
| $LOI^{1000}$ | wt.% | *12.55* | | | | | | |

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.

## STATISTICAL ANALYSIS

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

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

**Standard Deviation** values (1SDs) are reported in Table 1 and provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. The SD's 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 SD values thus include all sources of measurement uncertainty: between-lab variance, within-run variance

(precision errors) and CRM variability. OREAS prepared reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

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

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

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

Table 3 shows **Performance Gates** 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. 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.

**Tolerance Limits** (ISO Guide 3207) 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 zinc (Zn) by 4-acid digestion, where 99% of the time ($1-\alpha=0.99$) at least 95% of subsamples ($\rho=0.95$) will have concentrations lying between 13.41 and 13.85 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 139 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty four round robin laboratories received six samples per CRM and these samples were made up of paired samples from three different, non-adjacent sampling intervals. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between-units to that of the

variance within-units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 139. The test was performed using the following parameters:

- Null Hypothesis, $H_0$: Between-unit variance is no greater than within-unit variance (reject $H_0$ if *p*-value < 0.05);
- Alternative Hypothesis, $H_1$: Between-unit variance is greater than within-unit variance.

*P*-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of *p*-values. This process derived no significant *p*-values across the entire 134 certified values.

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 139 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 139 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

**Table 3. Pooled-Lab Performance Gates for OREAS 139.**

| 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 | 76.7 | 3.91 | 68.8 | 84.5 | 64.9 | 88.4 | 5.10% | 10.20% | 15.30% | 72.8 | 80.5 |
| Al, wt.% | 3.62 | 0.270 | 3.08 | 4.16 | 2.81 | 4.43 | 7.47% | 14.93% | 22.40% | 3.44 | 3.80 |
| As, ppm | 315 | 13 | 289 | 341 | 276 | 354 | 4.12% | 8.24% | 12.36% | 299 | 331 |
| Be, ppm | 2.87 | 0.197 | 2.47 | 3.26 | 2.28 | 3.46 | 6.86% | 13.73% | 20.59% | 2.72 | 3.01 |
| Bi, ppm | 6.83 | 0.542 | 5.75 | 7.92 | 5.21 | 8.46 | 7.93% | 15.86% | 23.80% | 6.49 | 7.18 |
| Ca, wt.% | 1.15 | 0.055 | 1.04 | 1.26 | 0.99 | 1.32 | 4.79% | 9.58% | 14.37% | 1.09 | 1.21 |
| Cd, ppm | 278 | 25 | 228 | 328 | 203 | 353 | 8.95% | 17.91% | 26.86% | 264 | 292 |
| Ce, ppm | 33.8 | 7.3 | 19.2 | 48.4 | 11.9 | 55.7 | 21.63% | 43.26% | 64.89% | 32.1 | 35.5 |
| Co, ppm | 25.6 | 1.86 | 21.9 | 29.3 | 20.0 | 31.2 | 7.26% | 14.52% | 21.78% | 24.3 | 26.9 |
| Cr, ppm | 29.4 | 4.0 | 21.4 | 37.3 | 17.4 | 41.3 | 13.57% | 27.14% | 40.71% | 27.9 | 30.8 |
| Cs, ppm | 3.22 | 0.286 | 2.65 | 3.79 | 2.37 | 4.08 | 8.86% | 17.73% | 26.59% | 3.06 | 3.38 |
| Cu, ppm | 271 | 8 | 255 | 288 | 246 | 296 | 3.09% | 6.18% | 9.27% | 258 | 285 |
| Dy, ppm | 2.81 | 0.093 | 2.63 | 3.00 | 2.53 | 3.09 | 3.30% | 6.61% | 9.91% | 2.67 | 2.95 |
| Er, ppm | 1.51 | 0.082 | 1.35 | 1.68 | 1.27 | 1.76 | 5.43% | 10.86% | 16.28% | 1.44 | 1.59 |
| Eu, ppm | 0.71 | 0.13 | 0.44 | 0.98 | 0.31 | 1.11 | 18.86% | 37.73% | 56.59% | 0.67 | 0.75 |
| Fe, wt.% | 11.79 | 0.663 | 10.46 | 13.12 | 9.80 | 13.78 | 5.62% | 11.25% | 16.87% | 11.20 | 12.38 |
| Ga, ppm | 10.2 | 0.81 | 8.6 | 11.8 | 7.8 | 12.6 | 7.92% | 15.85% | 23.77% | 9.7 | 10.7 |
| Gd, ppm | 3.63 | 0.345 | 2.94 | 4.32 | 2.59 | 4.66 | 9.53% | 19.05% | 28.58% | 3.45 | 3.81 |
| Hf, ppm | 2.29 | 0.24 | 1.80 | 2.78 | 1.55 | 3.02 | 10.71% | 21.42% | 32.12% | 2.17 | 2.40 |

Note: intervals may appear asymmetric due to rounding.

**Table 3 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** | | | | | | | | | | | |
| Ho, ppm | 0.53 | 0.027 | 0.48 | 0.59 | 0.45 | 0.62 | 5.10% | 10.20% | 15.31% | 0.51 | 0.56 |
| In, ppm | 0.73 | 0.071 | 0.59 | 0.87 | 0.52 | 0.94 | 9.71% | 19.43% | 29.14% | 0.69 | 0.77 |
| K, wt.% | 3.19 | 0.202 | 2.79 | 3.59 | 2.58 | 3.80 | 6.34% | 12.67% | 19.01% | 3.03 | 3.35 |
| La, ppm | 13.7 | 2.7 | 8.2 | 19.1 | 5.5 | 21.9 | 19.98% | 39.96% | 59.95% | 13.0 | 14.4 |
| Li, ppm | 38.9 | 3.56 | 31.8 | 46.0 | 28.2 | 49.6 | 9.16% | 18.32% | 27.48% | 36.9 | 40.8 |
| Lu, ppm | 0.22 | 0.03 | 0.17 | 0.28 | 0.15 | 0.30 | 11.69% | 23.38% | 35.07% | 0.21 | 0.24 |
| Mg, wt.% | 0.491 | 0.054 | 0.383 | 0.599 | 0.329 | 0.653 | 10.99% | 21.98% | 32.97% | 0.466 | 0.515 |
| Mn, wt.% | 0.663 | 0.027 | 0.608 | 0.718 | 0.580 | 0.745 | 4.14% | 8.28% | 12.42% | 0.630 | 0.696 |
| Mo, ppm | 9.82 | 0.752 | 8.32 | 11.33 | 7.57 | 12.08 | 7.66% | 15.32% | 22.97% | 9.33 | 10.32 |
| Na, wt.% | 0.108 | 0.010 | 0.088 | 0.129 | 0.078 | 0.139 | 9.48% | 18.95% | 28.43% | 0.103 | 0.114 |
| Nb, ppm | 5.76 | 0.85 | 4.07 | 7.46 | 3.22 | 8.31 | 14.71% | 29.42% | 44.13% | 5.48 | 6.05 |
| Nd, ppm | 17.8 | 2.2 | 13.4 | 22.3 | 11.1 | 24.6 | 12.59% | 25.17% | 37.76% | 17.0 | 18.7 |
| Ni, ppm | 35.9 | 3.05 | 29.8 | 42.0 | 26.8 | 45.1 | 8.49% | 16.97% | 25.46% | 34.1 | 37.7 |
| P, wt.% | 0.089 | 0.005 | 0.079 | 0.099 | 0.074 | 0.104 | 5.73% | 11.45% | 17.18% | 0.085 | 0.094 |
| Pb, wt.% | 2.21 | 0.095 | 2.02 | 2.40 | 1.93 | 2.50 | 4.28% | 8.56% | 12.83% | 2.10 | 2.32 |
| Pr, ppm | 4.72 | 0.323 | 4.07 | 5.36 | 3.75 | 5.68 | 6.85% | 13.70% | 20.54% | 4.48 | 4.95 |
| Rb, ppm | 142 | 11 | 121 | 163 | 110 | 174 | 7.50% | 15.01% | 22.51% | 135 | 149 |
| S, wt.% | 16.02 | 0.481 | 15.06 | 16.99 | 14.58 | 17.47 | 3.00% | 6.00% | 9.00% | 15.22 | 16.83 |
| Sb, ppm | 58 | 5.2 | 48 | 69 | 42 | 74 | 9.02% | 18.03% | 27.05% | 55 | 61 |
| Sc, ppm | 5.04 | 0.462 | 4.11 | 5.96 | 3.65 | 6.42 | 9.17% | 18.33% | 27.50% | 4.79 | 5.29 |
| Se, ppm | 2.78 | 0.44 | 1.90 | 3.65 | 1.46 | 4.09 | 15.76% | 31.52% | 47.29% | 2.64 | 2.92 |
| Sm, ppm | 4.01 | 0.222 | 3.57 | 4.45 | 3.34 | 4.68 | 5.54% | 11.07% | 16.61% | 3.81 | 4.21 |
| Sn, ppm | 2.27 | 0.24 | 1.78 | 2.75 | 1.54 | 2.99 | 10.63% | 21.26% | 31.89% | 2.15 | 2.38 |
| Sr, ppm | 331 | 65 | 201 | 460 | 136 | 525 | 19.62% | 39.24% | 58.86% | 314 | 347 |
| Tb, ppm | 0.47 | 0.028 | 0.42 | 0.53 | 0.39 | 0.56 | 5.93% | 11.87% | 17.80% | 0.45 | 0.50 |
| Ti, wt.% | 0.117 | 0.013 | 0.091 | 0.142 | 0.078 | 0.155 | 10.97% | 21.93% | 32.90% | 0.111 | 0.123 |
| Tl, ppm | 37.6 | 1.40 | 34.8 | 40.4 | 33.4 | 41.8 | 3.72% | 7.45% | 11.17% | 35.7 | 39.5 |
| Tm, ppm | 0.21 | 0.015 | 0.18 | 0.24 | 0.16 | 0.25 | 7.34% | 14.68% | 22.02% | 0.20 | 0.22 |
| U, ppm | 12.2 | 0.92 | 10.3 | 14.0 | 9.4 | 14.9 | 7.54% | 15.09% | 22.63% | 11.6 | 12.8 |
| V, ppm | 69 | 4.2 | 61 | 77 | 56 | 82 | 6.11% | 12.21% | 18.32% | 66 | 72 |
| W, ppm | 2.21 | 0.24 | 1.74 | 2.68 | 1.50 | 2.91 | 10.66% | 21.33% | 31.99% | 2.10 | 2.32 |
| Y, ppm | 15.4 | 0.86 | 13.6 | 17.1 | 12.8 | 18.0 | 5.63% | 11.25% | 16.88% | 14.6 | 16.1 |
| Yb, ppm | 1.46 | 0.050 | 1.36 | 1.56 | 1.31 | 1.61 | 3.45% | 6.90% | 10.35% | 1.38 | 1.53 |
| Zn, wt.% | 13.63 | 0.305 | 13.02 | 14.24 | 12.71 | 14.54 | 2.24% | 4.48% | 6.72% | 12.95 | 14.31 |
| Zr, ppm | 80 | 8 | 64 | 96 | 56 | 104 | 10.12% | 20.25% | 30.37% | 76 | 84 |
| **Peroxide Fusion ICP** | | | | | | | | | | | |
| Al, wt.% | 3.70 | 0.204 | 3.29 | 4.11 | 3.09 | 4.31 | 5.51% | 11.01% | 16.52% | 3.51 | 3.88 |
| As, ppm | 332 | 24 | 285 | 379 | 262 | 403 | 7.08% | 14.16% | 21.24% | 316 | 349 |
| Be, ppm | 3.17 | 0.51 | 2.16 | 4.19 | 1.66 | 4.69 | 15.93% | 31.87% | 47.80% | 3.02 | 3.33 |
| Bi, ppm | 6.64 | 0.642 | 5.35 | 7.92 | 4.71 | 8.56 | 9.67% | 19.35% | 29.02% | 6.30 | 6.97 |
| Ca, wt.% | 1.20 | 0.093 | 1.02 | 1.39 | 0.92 | 1.48 | 7.71% | 15.42% | 23.12% | 1.14 | 1.26 |
| Cd, ppm | 296 | 16 | 265 | 328 | 249 | 343 | 5.29% | 10.58% | 15.88% | 281 | 311 |
| Ce, ppm | 49.4 | 4.70 | 40.0 | 58.8 | 35.3 | 63.5 | 9.51% | 19.01% | 28.52% | 46.9 | 51.9 |

Note: intervals may appear asymmetric due to rounding.

**Table 3 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 |
| **Peroxide Fusion ICP continued** | | | | | | | | | | | |
| Co, ppm | 26.0 | 0.90 | 24.2 | 27.8 | 23.3 | 28.7 | 3.47% | 6.93% | 10.40% | 24.7 | 27.3 |
| Cs, ppm | 3.21 | 0.264 | 2.68 | 3.74 | 2.42 | 4.00 | 8.21% | 16.42% | 24.64% | 3.05 | 3.37 |
| Cu, ppm | 274 | 25 | 224 | 324 | 199 | 349 | 9.10% | 18.20% | 27.30% | 260 | 288 |
| Er, ppm | 1.69 | 0.20 | 1.29 | 2.08 | 1.10 | 2.27 | 11.62% | 23.24% | 34.86% | 1.60 | 1.77 |
| Fe, wt.% | 11.90 | 0.400 | 11.10 | 12.70 | 10.70 | 13.10 | 3.36% | 6.72% | 10.08% | 11.31 | 12.50 |
| Ga, ppm | 10.2 | 0.98 | 8.2 | 12.2 | 7.3 | 13.1 | 9.60% | 19.20% | 28.80% | 9.7 | 10.7 |
| In, ppm | 0.69 | 0.10 | 0.49 | 0.89 | 0.39 | 0.99 | 14.64% | 29.28% | 43.91% | 0.65 | 0.72 |
| K, wt.% | 3.30 | 0.122 | 3.06 | 3.54 | 2.93 | 3.66 | 3.69% | 7.37% | 11.06% | 3.13 | 3.46 |
| La, ppm | 23.1 | 3.1 | 16.8 | 29.3 | 13.7 | 32.4 | 13.57% | 27.14% | 40.72% | 21.9 | 24.2 |
| Li, ppm | 40.4 | 3.23 | 33.9 | 46.8 | 30.7 | 50.1 | 8.00% | 15.99% | 23.99% | 38.4 | 42.4 |
| Mg, wt.% | 0.501 | 0.022 | 0.457 | 0.545 | 0.435 | 0.567 | 4.39% | 8.78% | 13.17% | 0.476 | 0.526 |
| Mn, wt.% | 0.657 | 0.029 | 0.598 | 0.716 | 0.569 | 0.745 | 4.49% | 8.97% | 13.46% | 0.624 | 0.690 |
| Mo, ppm | 11.1 | 0.87 | 9.4 | 12.8 | 8.5 | 13.7 | 7.84% | 15.68% | 23.51% | 10.5 | 11.7 |
| Pb, wt.% | 2.20 | 0.108 | 1.99 | 2.42 | 1.88 | 2.53 | 4.90% | 9.80% | 14.70% | 2.09 | 2.31 |
| Rb, ppm | 145 | 8 | 129 | 161 | 121 | 169 | 5.52% | 11.05% | 16.57% | 138 | 152 |
| S, wt.% | 16.04 | 0.418 | 15.20 | 16.87 | 14.78 | 17.29 | 2.61% | 5.21% | 7.82% | 15.24 | 16.84 |
| Sb, ppm | 63 | 6.1 | 50 | 75 | 44 | 81 | 9.76% | 19.53% | 29.29% | 60 | 66 |
| Si, wt.% | 16.34 | 0.383 | 15.57 | 17.11 | 15.19 | 17.49 | 2.34% | 4.68% | 7.03% | 15.52 | 17.16 |
| Sr, ppm | 479 | 54 | 370 | 587 | 316 | 642 | 11.35% | 22.71% | 34.06% | 455 | 503 |
| Tb, ppm | 0.50 | 0.09 | 0.32 | 0.68 | 0.23 | 0.77 | 18.13% | 36.26% | 54.39% | 0.47 | 0.52 |
| Th, ppm | 7.54 | 1.01 | 5.52 | 9.56 | 4.51 | 10.57 | 13.39% | 26.78% | 40.17% | 7.17 | 7.92 |
| Ti, wt.% | 0.157 | 0.009 | 0.138 | 0.175 | 0.129 | 0.184 | 5.88% | 11.76% | 17.64% | 0.149 | 0.165 |
| Tl, ppm | 35.4 | 2.43 | 30.5 | 40.3 | 28.1 | 42.7 | 6.87% | 13.75% | 20.62% | 33.6 | 37.2 |
| U, ppm | 12.2 | 0.63 | 10.9 | 13.4 | 10.3 | 14.1 | 5.13% | 10.26% | 15.39% | 11.6 | 12.8 |
| Y, ppm | 17.1 | 1.62 | 13.8 | 20.3 | 12.2 | 21.9 | 9.47% | 18.95% | 28.42% | 16.2 | 17.9 |
| Zn, wt.% | 13.36 | 0.396 | 12.57 | 14.16 | 12.18 | 14.55 | 2.97% | 5.93% | 8.90% | 12.70 | 14.03 |
| **Aqua Regia Digestion** | | | | | | | | | | | |
| Ag, ppm | 78.2 | 2.35 | 73.5 | 82.9 | 71.2 | 85.3 | 3.01% | 6.02% | 9.03% | 74.3 | 82.1 |
| Al, wt.% | 0.709 | 0.054 | 0.601 | 0.816 | 0.548 | 0.870 | 7.58% | 15.16% | 22.74% | 0.674 | 0.744 |
| As, ppm | 322 | 24 | 274 | 370 | 250 | 394 | 7.47% | 14.94% | 22.41% | 306 | 338 |
| Be, ppm | 1.45 | 0.27 | 0.90 | 1.99 | 0.63 | 2.26 | 18.74% | 37.48% | 56.22% | 1.37 | 1.52 |
| Bi, ppm | 7.18 | 0.93 | 5.31 | 9.05 | 4.38 | 9.98 | 13.01% | 26.01% | 39.02% | 6.82 | 7.54 |
| Ca, wt.% | 1.14 | 0.091 | 0.96 | 1.33 | 0.87 | 1.42 | 7.99% | 15.98% | 23.96% | 1.09 | 1.20 |
| Cd, ppm | 280 | 34 | 213 | 348 | 179 | 382 | 12.04% | 24.08% | 36.12% | 266 | 295 |
| Ce, ppm | 41.1 | 4.2 | 32.6 | 49.5 | 28.4 | 53.8 | 10.29% | 20.59% | 30.88% | 39.0 | 43.1 |
| Co, ppm | 22.3 | 3.5 | 15.3 | 29.2 | 11.9 | 32.7 | 15.57% | 31.13% | 46.70% | 21.2 | 23.4 |
| Cr, ppm | 16.1 | 2.3 | 11.4 | 20.8 | 9.0 | 23.1 | 14.58% | 29.17% | 43.75% | 15.3 | 16.9 |
| Cs, ppm | 1.52 | 0.095 | 1.33 | 1.71 | 1.24 | 1.81 | 6.24% | 12.49% | 18.73% | 1.45 | 1.60 |
| Cu, ppm | 269 | 10 | 249 | 290 | 238 | 300 | 3.83% | 7.67% | 11.50% | 256 | 283 |
| Fe, wt.% | 11.65 | 0.470 | 10.71 | 12.59 | 10.24 | 13.06 | 4.04% | 8.08% | 12.11% | 11.07 | 12.23 |
| Ga, ppm | 4.78 | 0.432 | 3.92 | 5.64 | 3.48 | 6.07 | 9.03% | 18.06% | 27.09% | 4.54 | 5.02 |
| Gd, ppm | 3.21 | 0.50 | 2.21 | 4.21 | 1.70 | 4.72 | 15.64% | 31.28% | 46.92% | 3.05 | 3.37 |
| Hf, ppm | 0.65 | 0.08 | 0.49 | 0.80 | 0.42 | 0.88 | 11.98% | 23.95% | 35.93% | 0.62 | 0.68 |

Note: intervals may appear asymmetric due to rounding.

**Table 3 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 |
| **Aqua Regia Digestion continued** | | | | | | | | | | | |
| Hg, ppm | 4.18 | 0.408 | 3.36 | 5.00 | 2.95 | 5.40 | 9.77% | 19.54% | 29.30% | 3.97 | 4.39 |
| In, ppm | 0.66 | 0.07 | 0.52 | 0.80 | 0.45 | 0.87 | 10.56% | 21.11% | 31.67% | 0.63 | 0.70 |
| K, wt.% | 0.360 | 0.036 | 0.288 | 0.433 | 0.251 | 0.469 | 10.07% | 20.14% | 30.21% | 0.342 | 0.378 |
| La, ppm | 16.5 | 2.3 | 11.9 | 21.0 | 9.7 | 23.3 | 13.77% | 27.55% | 41.32% | 15.6 | 17.3 |
| Li, ppm | 23.8 | 3.8 | 16.2 | 31.3 | 12.4 | 35.1 | 15.89% | 31.78% | 47.66% | 22.6 | 25.0 |
| Lu, ppm | 0.13 | 0.02 | 0.09 | 0.17 | 0.07 | 0.20 | 15.51% | 31.02% | 46.53% | 0.13 | 0.14 |
| Mg, wt.% | 0.399 | 0.025 | 0.349 | 0.450 | 0.323 | 0.475 | 6.35% | 12.70% | 19.05% | 0.379 | 0.419 |
| Mn, wt.% | 0.618 | 0.022 | 0.575 | 0.661 | 0.553 | 0.683 | 3.51% | 7.01% | 10.52% | 0.587 | 0.649 |
| Mo, ppm | 9.89 | 0.663 | 8.57 | 11.22 | 7.90 | 11.88 | 6.70% | 13.40% | 20.10% | 9.40 | 10.39 |
| Nb, ppm | 0.66 | 0.08 | 0.49 | 0.83 | 0.40 | 0.91 | 12.81% | 25.62% | 38.43% | 0.62 | 0.69 |
| Nd, ppm | 18.5 | 3.0 | 12.5 | 24.5 | 9.5 | 27.5 | 16.26% | 32.51% | 48.77% | 17.5 | 19.4 |
| Ni, ppm | 34.9 | 3.7 | 27.5 | 42.3 | 23.8 | 46.1 | 10.63% | 21.26% | 31.89% | 33.2 | 36.7 |
| P, wt.% | 0.090 | 0.006 | 0.078 | 0.102 | 0.073 | 0.107 | 6.42% | 12.84% | 19.27% | 0.086 | 0.095 |
| Pb, wt.% | 2.22 | 0.067 | 2.09 | 2.36 | 2.02 | 2.42 | 3.00% | 6.00% | 9.00% | 2.11 | 2.34 |
| Rb, ppm | 26.7 | 3.0 | 20.8 | 32.7 | 17.8 | 35.7 | 11.10% | 22.20% | 33.31% | 25.4 | 28.1 |
| S, wt.% | 15.98 | 0.724 | 14.54 | 17.43 | 13.81 | 18.15 | 4.53% | 9.05% | 13.58% | 15.18 | 16.78 |
| Sb, ppm | 49.1 | 9.5 | 30.2 | 68.0 | 20.7 | 77.4 | 19.27% | 38.53% | 57.80% | 46.6 | 51.5 |
| Sc, ppm | 2.53 | 0.49 | 1.54 | 3.52 | 1.05 | 4.01 | 19.51% | 39.02% | 58.54% | 2.40 | 2.66 |
| Te, ppm | 0.57 | 0.08 | 0.41 | 0.72 | 0.34 | 0.80 | 13.62% | 27.24% | 40.86% | 0.54 | 0.60 |
| Th, ppm | 6.53 | 0.91 | 4.72 | 8.34 | 3.81 | 9.25 | 13.87% | 27.74% | 41.61% | 6.20 | 6.86 |
| Ti, wt.% | 0.016 | 0.002 | 0.013 | 0.020 | 0.011 | 0.021 | 10.34% | 20.67% | 31.01% | 0.015 | 0.017 |
| Tl, ppm | 8.91 | 0.835 | 7.24 | 10.58 | 6.41 | 11.42 | 9.37% | 18.74% | 28.11% | 8.47 | 9.36 |
| U, ppm | 11.4 | 0.38 | 10.7 | 12.2 | 10.3 | 12.6 | 3.36% | 6.71% | 10.07% | 10.9 | 12.0 |
| V, ppm | 36.2 | 3.01 | 30.2 | 42.2 | 27.2 | 45.2 | 8.30% | 16.61% | 24.91% | 34.4 | 38.0 |
| W, ppm | 1.77 | 0.20 | 1.37 | 2.17 | 1.17 | 2.37 | 11.34% | 22.68% | 34.02% | 1.68 | 1.86 |
| Y, ppm | 12.0 | 0.75 | 10.5 | 13.5 | 9.7 | 14.2 | 6.23% | 12.46% | 18.68% | 11.4 | 12.6 |
| Yb, ppm | 0.95 | 0.12 | 0.70 | 1.20 | 0.57 | 1.32 | 13.11% | 26.22% | 39.33% | 0.90 | 0.99 |
| Zn, wt.% | 13.59 | 0.492 | 12.60 | 14.57 | 12.11 | 15.06 | 3.62% | 7.24% | 10.86% | 12.91 | 14.27 |
| Zr, ppm | 20.4 | 3.7 | 12.9 | 27.9 | 9.1 | 31.6 | 18.38% | 36.76% | 55.15% | 19.4 | 21.4 |
| **Infrared Combustion** | | | | | | | | | | | |
| C, wt.% | 3.83 | 0.100 | 3.63 | 4.03 | 3.53 | 4.13 | 2.62% | 5.24% | 7.87% | 3.64 | 4.02 |
| S, wt.% | 16.52 | 0.271 | 15.98 | 17.06 | 15.71 | 17.33 | 1.64% | 3.28% | 4.92% | 15.69 | 17.35 |

Note: intervals may appear asymmetric due to rounding.

## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Lima, Peru
4. ALS, Loughrea, Galway, Ireland
5. ALS, Perth, WA, Australia
6. ALS, Vancouver, BC, Canada

7. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
8. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
9. Bureau Veritas Geoanalytical, Perth, WA, Australia
10. Inspectorate (BV), Lima, Peru
11. Intertek Genalysis, Perth, WA, Australia
12. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
13. Laboratorio Stewart-Blaitt LTDA, Santiago, Chile
14. LCT, Sao Paulo, Sao Paulo, Brazil
15. MinAnalytical Services, Perth, WA, Australia
16. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
17. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
18. SGS Australia Mineral Services, Perth, WA, Australia
19. SGS del Peru, Lima, Peru
20. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
21. SGS Mineral Services, Townsville, QLD, Australia
22. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
23. UIS Analytical Services, Centurion , South Africa
24. Zarazma Mineral Studies Company, Tehran, Iran

## PREPARER AND SUPPLIER

Certified reference material OREAS 139 is prepared, certified and supplied by:

ORE RESEARCH & EXPLORATION

ORE Research & Exploration Pty Ltd
37A Hosie Street
Bayswater North VIC 3153
AUSTRALIA

Tel: +613-9729 0333
Fax: +613-9729 8338
Web: www.ore.com.au
Email: info@ore.com.au

It is packaged under nitrogen in unit sizes of 10g (single-use laminated foil pouches).

## INTENDED USE

OREAS 139 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 139 has been prepared from primary sulphide bearing ores from the Dugald River deposit. It contains reactive sulphide (16.52% S) and has been packaged under a nitrogen

environment (single use laminated foil pouches). In its unopened state and under normal conditions of storage the CRM has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

## INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 139 refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

## 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.

## 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) 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.

## 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 ACCREDITED

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

## CERTIFYING OFFICER

8th August, 2017

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

## REFERENCES

ISO Guide 30 (1992), Terms and definitions used in connection with reference materials.
ISO Guide 31 (2000), Reference materials – Contents of certificates and labels.
ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.
ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.