

**CERTIFICATE OF ANALYSIS FOR**

**CARBONATITE SUPERGENE REE-Nb ORE (TREO 9.88%)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 465**

**Summary Statistics for Key Analytes** (additional certified values are available in Table 1).

| Constituent  | Certified Value | 1SD   | 95% Confidence Limits |      | 95% Tolerance Limits |      |
|--|-----------------|-------|-----------------------|------|----------------------|------|
|  |                 |       | Low                   | High | Low                  | High |
| <b>Borate / Peroxide Fusion ICP</b>                                |                 |       |                       |      |                      |      |
| CeO <sub>2</sub> , Cerium(IV) oxide (wt.%)                         | 4.86            | 0.166 | 4.75                  | 4.96 | 4.76                 | 4.95 |
| Dy <sub>2</sub> O <sub>3</sub> , Dysprosium(III) oxide (ppm)       | 249             | 14    | 240                   | 257  | 241                  | 256  |
| Er <sub>2</sub> O <sub>3</sub> , Erbium(III) oxide (ppm)           | 58              | 3.6   | 56                    | 60   | 56                   | 60   |
| Eu <sub>2</sub> O <sub>3</sub> , Europium(III) oxide (ppm)         | 331             | 13    | 324                   | 338  | 321                  | 341  |
| Gd <sub>2</sub> O <sub>3</sub> , Gadolinium(III) oxide (ppm)       | 674             | 35    | 654                   | 693  | 656                  | 691  |
| Ho <sub>2</sub> O <sub>3</sub> , Holmium(III) oxide (ppm)          | 31.7            | 2.44  | 30.4                  | 33.1 | 30.6                 | 32.8 |
| La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)       | 2.83            | 0.097 | 2.78                  | 2.88 | 2.77                 | 2.89 |
| Lu <sub>2</sub> O <sub>3</sub> , Lutetium(III) oxide (ppm)         | 2.39            | 0.138 | 2.34                  | 2.45 | 2.27                 | 2.52 |
| Nb <sub>2</sub> O <sub>5</sub> , Niobium(V) oxide (ppm)            | 6695            | 431   | 6383                  | 7007 | 6341                 | 7049 |
| Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)       | 1.37            | 0.059 | 1.34                  | 1.41 | 1.34                 | 1.40 |
| Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm) | 4557            | 198   | 4443                  | 4672 | 4435                 | 4679 |
| Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)         | 1578            | 42    | 1560                  | 1597 | 1527                 | 1630 |
| Tb <sub>4</sub> O <sub>7</sub> , Terbium(III,IV) oxide (ppm)       | 67              | 3.7   | 65                    | 69   | 64                   | 69   |
| ThO <sub>2</sub> , Thorium dioxide (ppm)                           | 985             | 49    | 956                   | 1013 | 961                  | 1008 |
| Tm <sub>2</sub> O <sub>3</sub> , Thulium(III) oxide (ppm)          | 5.16            | 0.299 | 5.02                  | 5.31 | 4.90                 | 5.43 |
| U <sub>3</sub> O <sub>8</sub> , Uranium(V,VI) oxide (ppm)          | 16.0            | 0.47  | 15.8                  | 16.3 | 15.4                 | 16.7 |
| Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)           | 665             | 42    | 640                   | 691  | 639                  | 691  |
| Yb <sub>2</sub> O <sub>3</sub> , Ytterbium(III) oxide (ppm)        | 21.6            | 0.83  | 21.2                  | 22.0 | 20.4                 | 22.8 |
| ZrO <sub>2</sub> , Zirconium dioxide (ppm)                         | 2539            | 274   | 2280                  | 2798 | 2382                 | 2696 |

Note: intervals may appear asymmetric due to rounding.



**Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 465.**

| Constituent   | Certified Value | 1SD   | 95% Confidence Limits |       | 95% Tolerance Limits |       |
|---|-----------------|-------|-----------------------|-------|----------------------|-------|
|   |                 |       | Low                   | High  | Low                  | High  |
| <b>Borate Fusion XRF</b>  |                 |       |                       |       |                      |       |
| CeO <sub>2</sub> , Cerium(IV) oxide (wt.%)  | 4.88            | 0.045 | 4.84                  | 4.92  | 4.84                 | 4.91  |
| Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)   | 49.96           | 1.309 | 48.22                 | 51.70 | 49.62                | 50.29 |
| La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)                                    | 2.84            | 0.033 | 2.81                  | 2.87  | 2.82                 | 2.86  |
| Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)                                    | 1.38            | 0.102 | 1.28                  | 1.48  | 1.35                 | 1.40  |
| Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm)                              | 4534            | 143.9 | 4362                  | 4705  | 4353                 | 4714  |
| Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)                                      | 1640            | 135.7 | 1500                  | 1780  | IND                  | IND   |
| ThO <sub>2</sub> , Thorium dioxide (ppm)  | 901             | 128   | 757                   | 1045  | IND                  | IND   |
| Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)  | 637             | 45.0  | 614                   | 661   | IND                  | IND   |
| <b>Thermogravimetry</b>   |                 |       |                       |       |                      |       |
| LOI, Loss On Ignition @1000°C (wt.%)  | 0.824           | 0.133 | 0.697                 | 0.950 | 0.774                | 0.874 |
| <b>Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)</b> |                 |       |                       |       |                      |       |
| Al, Aluminium (wt.%)  | 6.60            | 0.261 | 6.39                  | 6.80  | 6.47                 | 6.72  |
| Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)                                    | 12.47           | 0.494 | 12.08                 | 12.85 | 12.23                | 12.70 |
| Ba, Barium (ppm)  | 4397            | 464   | 4127                  | 4667  | 4278                 | 4516  |
| BaO, Barium oxide (ppm)   | 4909            | 518   | 4608                  | 5211  | 4776                 | 5042  |
| Be, Beryllium (ppm)   | 13.2            | 0.89  | 12.5                  | 14.0  | IND                  | IND   |
| Bi, Bismuth (ppm)   | 16.9            | 1.04  | 16.0                  | 17.9  | 16.1                 | 17.7  |
| Ca, Calcium (wt.%)  | 0.900           | 0.060 | 0.861                 | 0.938 | 0.871                | 0.929 |
| CaO, Calcium oxide (wt.%)   | 1.26            | 0.084 | 1.21                  | 1.31  | 1.22                 | 1.30  |
| Ce, Cerium (wt.%)   | 3.95            | 0.135 | 3.87                  | 4.03  | 3.88                 | 4.03  |
| CeO <sub>2</sub> , Cerium(IV) oxide (wt.%)  | 4.86            | 0.166 | 4.75                  | 4.96  | 4.76                 | 4.95  |
| Cr, Chromium (ppm)  | 544             | 42    | 516                   | 572   | 530                  | 558   |
| Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)                                      | 795             | 61    | 755                   | 836   | 775                  | 815   |
| Cs, Cesium (ppm)  | < 0.1           | IND   | IND                   | IND   | IND                  | IND   |
| Dy, Dysprosium (ppm)  | 217             | 13    | 209                   | 224   | 210                  | 223   |
| Dy <sub>2</sub> O <sub>3</sub> , Dysprosium(III) oxide (ppm)                                    | 249             | 14    | 240                   | 257   | 241                  | 256   |
| Er, Erbium (ppm)  | 50              | 3.1   | 49                    | 52    | 49                   | 52    |
| Er <sub>2</sub> O <sub>3</sub> , Erbium(III) oxide (ppm)  | 58              | 3.6   | 56                    | 60    | 56                   | 60    |
| Eu, Europium (ppm)  | 286             | 11    | 280                   | 292   | 277                  | 294   |
| Eu <sub>2</sub> O <sub>3</sub> , Europium(III) oxide (ppm)                                      | 331             | 13    | 324                   | 338   | 321                  | 341   |
| Fe, Iron (wt.%)   | 34.71           | 0.634 | 34.16                 | 35.26 | 33.82                | 35.60 |
| Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)   | 49.63           | 0.906 | 48.84                 | 50.41 | 48.36                | 50.90 |
| Ga, Gallium (ppm)   | 214             | 43    | 166                   | 261   | 208                  | 220   |
| Gd, Gadolinium (ppm)  | 584             | 31    | 568                   | 601   | 569                  | 600   |
| Gd <sub>2</sub> O <sub>3</sub> , Gadolinium(III) oxide (ppm)                                    | 674             | 35    | 654                   | 693   | 656                  | 691   |
| Hf, Hafnium (ppm)   | 41.4            | 7.2   | 35.8                  | 47.0  | 38.5                 | 44.3  |
| HfO <sub>2</sub> , Hafnium dioxide (ppm)  | 48.8            | 8.5   | 42.3                  | 55.4  | 45.4                 | 52.2  |
| Ho, Holmium (ppm)   | 27.7            | 2.13  | 26.5                  | 28.9  | 26.7                 | 28.7  |
| Ho <sub>2</sub> O <sub>3</sub> , Holmium(III) oxide (ppm)                                       | 31.7            | 2.44  | 30.4                  | 33.1  | 30.6                 | 32.8  |
| In, Indium (ppm)  | 3.47            | 0.227 | 3.32                  | 3.62  | 2.94                 | 4.00  |
| La, Lanthanum (wt.%)  | 2.41            | 0.082 | 2.37                  | 2.46  | 2.36                 | 2.47  |
| La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)                                    | 2.83            | 0.097 | 2.78                  | 2.88  | 2.77                 | 2.89  |

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  |
| <b>Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)</b> |                 |       |                       |       |                      |       |
| Lu, Lutetium (ppm)  | 2.10            | 0.122 | 2.06                  | 2.15  | 1.99                 | 2.21  |
| Lu <sub>2</sub> O <sub>3</sub> , Lutetium(III) oxide (ppm)  | 2.39            | 0.138 | 2.34                  | 2.45  | 2.27                 | 2.52  |
| Mg, Magnesium (wt.%)  | 0.392           | 0.021 | 0.377                 | 0.406 | 0.380                | 0.404 |
| MgO, Magnesium oxide (wt.%)   | 0.650           | 0.034 | 0.625                 | 0.674 | 0.629                | 0.670 |
| Mn, Manganese (wt.%)  | 0.263           | 0.023 | 0.247                 | 0.279 | 0.254                | 0.271 |
| MnO, Manganese oxide (wt.%)   | 0.339           | 0.030 | 0.318                 | 0.360 | 0.328                | 0.350 |
| Mo, Molybdenum (ppm)  | 114             | 6     | 109                   | 119   | 108                  | 120   |
| Nb, Niobium (ppm)   | 4680            | 301   | 4462                  | 4898  | 4433                 | 4928  |
| Nb <sub>2</sub> O <sub>5</sub> , Niobium(V) oxide (ppm)   | 6695            | 431   | 6383                  | 7007  | 6341                 | 7049  |
| Nd, Neodymium (wt.%)  | 1.18            | 0.050 | 1.15                  | 1.20  | 1.15                 | 1.20  |
| Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)  | 1.37            | 0.059 | 1.34                  | 1.41  | 1.34                 | 1.40  |
| P, Phosphorus (wt.%)  | 3.81            | 0.122 | 3.70                  | 3.92  | 3.69                 | 3.94  |
| P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)  | 8.74            | 0.280 | 8.49                  | 8.98  | 8.45                 | 9.02  |
| Pb, Lead (ppm)  | 506             | 19    | 496                   | 516   | 480                  | 532   |
| PbO, Lead oxide (ppm)   | 545             | 20    | 534                   | 556   | 517                  | 573   |
| Pr, Praseodymium (ppm)  | 3772            | 164   | 3677                  | 3867  | 3671                 | 3873  |
| Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm)  | 4557            | 198   | 4443                  | 4672  | 4435                 | 4679  |
| S, Sulphur (ppm)  | 1941            | 132   | 1787                  | 2095  | IND                  | IND   |
| Si, Silicon (wt.%)  | 1.53            | 0.043 | 1.48                  | 1.58  | 1.49                 | 1.58  |
| SiO <sub>2</sub> , Silicon dioxide (wt.%)   | 3.28            | 0.093 | 3.18                  | 3.39  | 3.19                 | 3.37  |
| Sm, Samarium (ppm)  | 1361            | 36    | 1345                  | 1377  | 1317                 | 1405  |
| Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)  | 1578            | 42    | 1560                  | 1597  | 1527                 | 1630  |
| Sn, Tin (ppm)   | 136             | 20    | 124                   | 148   | 129                  | 143   |
| SnO <sub>2</sub> , Tin dioxide (ppm)  | 172             | 26    | 157                   | 188   | 164                  | 181   |
| Sr, Strontium (ppm)   | 5204            | 182   | 5085                  | 5322  | 5102                 | 5305  |
| SrO, Strontium oxide (ppm)  | 6154            | 215   | 6014                  | 6294  | 6034                 | 6274  |
| Ta, Tantalum (ppm)  | 79              | 5.0   | 75                    | 82    | 74                   | 83    |
| Ta <sub>2</sub> O <sub>5</sub> , Tantalum(V) oxide (ppm)  | 96              | 6.2   | 91                    | 101   | 91                   | 102   |
| Tb, Terbium (ppm)   | 57              | 3.1   | 55                    | 58    | 54                   | 59    |
| Tb <sub>4</sub> O <sub>7</sub> , Terbium(III,IV) oxide (ppm)  | 67              | 3.7   | 65                    | 69    | 64                   | 69    |
| Th, Thorium (ppm)   | 866             | 43    | 840                   | 891   | 845                  | 886   |
| ThO <sub>2</sub> , Thorium dioxide (ppm)  | 985             | 49    | 956                   | 1013  | 961                  | 1008  |
| Ti, Titanium (wt.%)   | 6.30            | 0.184 | 6.16                  | 6.44  | 6.14                 | 6.46  |
| TiO <sub>2</sub> , Titanium dioxide (wt.%)  | 10.51           | 0.307 | 10.28                 | 10.74 | 10.25                | 10.78 |
| Tm, Thulium (ppm)   | 4.52            | 0.262 | 4.40                  | 4.65  | 4.29                 | 4.75  |
| Tm <sub>2</sub> O <sub>3</sub> , Thulium(III) oxide (ppm)   | 5.16            | 0.299 | 5.02                  | 5.31  | 4.90                 | 5.43  |
| U, Uranium (ppm)  | 13.6            | 0.40  | 13.4                  | 13.8  | 13.0                 | 14.2  |
| U <sub>3</sub> O <sub>8</sub> , Uranium(V,VI) oxide (ppm)   | 16.0            | 0.47  | 15.8                  | 16.3  | 15.4                 | 16.7  |
| V, Vanadium (ppm)   | 534             | 36    | 512                   | 557   | 518                  | 551   |
| V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)   | 953             | 64    | 913                   | 994   | 924                  | 983   |
| W, Tungsten (ppm)   | 7.52            | 1.24  | 6.51                  | 8.53  | IND                  | IND   |
| WO <sub>3</sub> , Tungsten trioxide (ppm)   | 9.48            | 1.56  | 8.20                  | 10.76 | IND                  | IND   |

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  |
| <b>Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)</b> |                 |       |                       |       |                      |       |
| Y, Yttrium (ppm)  | 524             | 33    | 504                   | 544   | 503                  | 544   |
| Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)  | 665             | 42    | 640                   | 691   | 639                  | 691   |
| Yb, Ytterbium (ppm)   | 19.0            | 0.72  | 18.6                  | 19.4  | 17.9                 | 20.1  |
| Yb <sub>2</sub> O <sub>3</sub> , Ytterbium(III) oxide (ppm)   | 21.6            | 0.83  | 21.2                  | 22.0  | 20.4                 | 22.8  |
| Zr, Zirconium (ppm)   | 1880            | 203   | 1688                  | 2071  | 1764                 | 1996  |
| ZrO <sub>2</sub> , Zirconium dioxide (ppm)  | 2539            | 274   | 2280                  | 2798  | 2382                 | 2696  |
| <b>4-Acid Digestion</b>   |                 |       |                       |       |                      |       |
| Ag, Silver (ppm)  | 5.5             | 0.35  | 5.3                   | 5.7   | 5.1                  | 5.9   |
| Al, Aluminium (wt.%)  | 6.21            | 0.399 | 6.00                  | 6.42  | 6.02                 | 6.40  |
| Ba, Barium (ppm)  | 4359            | 158.0 | 4270                  | 4448  | 4291                 | 4427  |
| Be, Beryllium (ppm)   | 11.6            | 0.76  | 11.2                  | 12.1  | 11.2                 | 12.1  |
| Bi, Bismuth (ppm)   | 17.3            | 1.03  | 16.7                  | 17.9  | 16.7                 | 17.8  |
| Ca, Calcium (wt.%)  | 0.872           | 0.062 | 0.830                 | 0.914 | 0.850                | 0.894 |
| Cd, Cadmium (ppm)   | 1.20            | 0.14  | 1.12                  | 1.28  | 1.08                 | 1.32  |
| Ce, Cerium (wt.%)   | 3.91            | 0.149 | 3.76                  | 4.05  | 3.81                 | 4.00  |
| Co, Cobalt (ppm)  | 18.7            | 1.26  | 18.1                  | 19.3  | 18.1                 | 19.3  |
| Cs, Cesium (ppm)  | < 0.1           | IND   | IND                   | IND   | IND                  | IND   |
| Cu, Copper (ppm)  | 128             | 5.3   | 125                   | 130   | 123                  | 132   |
| Dy, Dysprosium (ppm)  | 215             | 9.3   | 210                   | 220   | 210                  | 221   |
| Er, Erbium (ppm)  | 47.3            | 2.51  | 45.8                  | 48.7  | 45.5                 | 49.0  |
| Eu, Europium (ppm)  | 282             | 11.8  | 275                   | 290   | 275                  | 289   |
| Fe, Iron (wt.%)   | 29.55           | 2.025 | 28.38                 | 30.73 | 28.92                | 30.19 |
| Ga, Gallium (ppm)   | 188             | 31    | 157                   | 220   | 175                  | 201   |
| Gd, Gadolinium (ppm)  | 581             | 35.8  | 556                   | 606   | 570                  | 592   |
| Hf, Hafnium (ppm)   | 14.4            | 1.9   | 13.7                  | 15.1  | 13.0                 | 15.8  |
| Ho, Holmium (ppm)   | 26.8            | 0.94  | 26.3                  | 27.2  | 25.9                 | 27.7  |
| In, Indium (ppm)  | 3.18            | 0.208 | 3.06                  | 3.31  | 3.05                 | 3.32  |
| La, Lanthanum (wt.%)  | 2.27            | 0.093 | 2.15                  | 2.39  | 2.22                 | 2.33  |
| Li, Lithium (ppm)   | 3.04            | 0.33  | 2.80                  | 3.28  | 2.86                 | 3.22  |
| Lu, Lutetium (ppm)  | 1.72            | 0.28  | 1.54                  | 1.90  | 1.62                 | 1.82  |
| Mg, Magnesium (wt.%)  | 0.374           | 0.026 | 0.356                 | 0.391 | 0.362                | 0.386 |
| Mn, Manganese (wt.%)  | 0.198           | 0.022 | 0.181                 | 0.215 | 0.191                | 0.204 |
| Mo, Molybdenum (ppm)  | 98              | 10    | 92                    | 105   | 95                   | 101   |
| Na, Sodium (wt.%)   | < 0.2           | IND   | IND                   | IND   | IND                  | IND   |
| Nd, Neodymium (wt.%)  | 1.10            | 0.043 | 1.05                  | 1.14  | 1.04                 | 1.15  |
| Ni, Nickel (ppm)  | 106             | 14    | 95                    | 116   | 95                   | 116   |
| P, Phosphorus (wt.%)  | 3.15            | 0.38  | 2.83                  | 3.47  | 3.06                 | 3.24  |
| Pb, Lead (ppm)  | 573             | 16.1  | 562                   | 583   | 561                  | 584   |
| Pr, Praseodymium (ppm)  | 3670            | 192.8 | 3488                  | 3852  | 3561                 | 3778  |
| Rb, Rubidium (ppm)  | 0.43            | 0.05  | 0.40                  | 0.45  | 0.35                 | 0.50  |

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  |
| <b>4-Acid Digestion continued</b> |                 |       |                       |       |                      |       |
| Re, Rhenium (ppm)                 | < 0.05          | IND   | IND                   | IND   | IND                  | IND   |
| Sc, Scandium (ppm)                | 149             | 9.1   | 143                   | 154   | 143                  | 154   |
| Sm, Samarium (ppm)                | 1307            | 80.8  | 1230                  | 1383  | 1241                 | 1373  |
| Sr, Strontium (wt.%)              | 0.505           | 0.026 | 0.487                 | 0.523 | 0.490                | 0.520 |
| Tb, Terbium (ppm)                 | 57              | 3.3   | 55                    | 59    | 55                   | 59    |
| Te, Tellurium (ppm)               | < 1             | IND   | IND                   | IND   | IND                  | IND   |
| Th, Thorium (ppm)                 | 805             | 150   | 693                   | 916   | 773                  | 837   |
| Tl, Thallium (ppm)                | 0.087           | 0.009 | 0.082                 | 0.093 | IND                  | IND   |
| Tm, Thulium (ppm)                 | 3.82            | 0.274 | 3.65                  | 4.00  | 3.70                 | 3.95  |
| U, Uranium (ppm)                  | 12.6            | 0.68  | 12.2                  | 13.0  | 12.1                 | 13.1  |
| V, Vanadium (ppm)                 | 427             | 69    | 380                   | 473   | 410                  | 443   |
| Y, Yttrium (ppm)                  | 478             | 32.4  | 455                   | 500   | 464                  | 492   |
| Yb, Ytterbium (ppm)               | 14.9            | 0.95  | 14.3                  | 15.5  | 14.3                 | 15.5  |
| Zn, Zinc (ppm)                    | 921             | 133   | 841                   | 1001  | 894                  | 949   |

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 465.

| Constituent                         | Unit | Value | Constituent                    | Unit | Value | Constituent                    | Unit | Value |
|-------------------------------------|------|-------|--------------------------------|------|-------|--------------------------------|------|-------|
| <b>Borate Fusion XRF</b>            |      |       |                                |      |       |                                |      |       |
| Al <sub>2</sub> O <sub>3</sub>      | wt.% | 12.62 | Lu <sub>2</sub> O <sub>3</sub> | ppm  | < 20  | SrO                            | wt.% | 0.603 |
| BaO                                 | ppm  | 5342  | MgO                            | wt.% | 0.664 | Ta <sub>2</sub> O <sub>5</sub> | ppm  | < 100 |
| CaO                                 | wt.% | 1.31  | MnO                            | wt.% | 0.369 | Tb <sub>4</sub> O <sub>7</sub> | ppm  | 67    |
| Cr <sub>2</sub> O <sub>3</sub>      | ppm  | 735   | Na <sub>2</sub> O              | wt.% | 0.172 | TiO <sub>2</sub>               | wt.% | 10.59 |
| Dy <sub>2</sub> O <sub>3</sub>      | ppm  | 329   | Nb <sub>2</sub> O <sub>5</sub> | ppm  | 6651  | Tm <sub>2</sub> O <sub>3</sub> | ppm  | < 10  |
| Er <sub>2</sub> O <sub>3</sub>      | ppm  | 62    | NiO                            | ppm  | 200   | U <sub>3</sub> O <sub>8</sub>  | ppm  | < 100 |
| Eu <sub>2</sub> O <sub>3</sub>      | ppm  | 350   | P <sub>2</sub> O <sub>5</sub>  | wt.% | 8.77  | V <sub>2</sub> O <sub>5</sub>  | ppm  | 1088  |
| Gd <sub>2</sub> O <sub>3</sub>      | ppm  | 870   | PbO                            | ppm  | 650   | WO <sub>3</sub>                | ppm  | < 100 |
| HfO <sub>2</sub>                    | ppm  | < 100 | SiO <sub>2</sub>               | wt.% | 3.20  | Yb <sub>2</sub> O <sub>3</sub> | ppm  | 80    |
| Ho <sub>2</sub> O <sub>3</sub>      | ppm  | 30.0  | SnO <sub>2</sub>               | ppm  | 217   | ZnO                            | ppm  | 1500  |
| K <sub>2</sub> O                    | wt.% | 0.015 | SO <sub>3</sub>                | wt.% | 0.485 | ZrO <sub>2</sub>               | ppm  | 2617  |
| <b>Thermogravimetry</b>             |      |       |                                |      |       |                                |      |       |
| H <sub>2</sub> O-                   | wt.% | 0.312 |                                |      |       |                                |      |       |
| <b>Borate / Peroxide Fusion ICP</b> |      |       |                                |      |       |                                |      |       |
| Ag                                  | ppm  | 6.5   | K                              | wt.% | 0.099 | Sc                             | ppm  | 171   |
| As                                  | ppm  | 420   | Li                             | ppm  | 3.33  | Se                             | ppm  | 33.6  |
| B                                   | ppm  | 23.3  | Na                             | wt.% | 0.078 | Te                             | ppm  | < 1   |
| Cd                                  | ppm  | 1.04  | Ni                             | ppm  | 101   | Tl                             | ppm  | < 0.5 |
| Co                                  | ppm  | 23.0  | Rb                             | ppm  | 0.98  | Zn                             | ppm  | 1097  |
| Cu                                  | ppm  | 128   | Re                             | ppm  | < 0.1 |                                |      |       |
| Ge                                  | ppm  | 60    | Sb                             | ppm  | 0.98  |                                |      |       |
| <b>4-Acid Digestion</b>             |      |       |                                |      |       |                                |      |       |
| As                                  | ppm  | 31.7  | S                              | ppm  | 1103  | Ti                             | wt.% | 1.30  |
| Cr                                  | ppm  | 357   | Sb                             | ppm  | 0.57  | W                              | ppm  | 2.40  |
| Ge                                  | ppm  | 23.6  | Se                             | ppm  | 35.4  | Zr                             | ppm  | 367   |
| K                                   | wt.% | 0.023 | Sn                             | ppm  | 48.8  |                                |      |       |
| Nb                                  | ppm  | 399   | Ta                             | ppm  | 50    |                                |      |       |

## 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 465 is a high grade ore, rare earth element (TREO = 9.88%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 465 were sourced from Lynas Corporation's Mount Weld Project (the 'Central Lanthanide Deposit') which is located 35 kilometres south of Laverton in Western Australia. The Mount Weld source materials (waste, low and medium grade REE ores) were found to be highly hygroscopic to the extent that significant analytical errors would likely result during analysis unless strict moisture handling procedures were adhered. To avoid this complication, the hygroscopic property was destroyed by roasting the materials at 900°C for 2 hours. Following re-equilibration of the materials to laboratory atmosphere the hygroscopic moisture content was deemed acceptable (~0.5% H<sub>2</sub>O-).

OREAS 465 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 99 certified values (and 66 indicative values) including REE's, majors and traces by fusion XRF, fusion ICP and 4-acid digestion.

The following summary of the mineralogy and supergene enrichment processes that operated in the host lateritic rocks is from Duncan and Willett (1990), Lottermoser (1990) and Lawrence (2006) as cited by S. Jaireth *et al* in 'Ore Geology Reviews 62 (2014) 72-128'.

The Mt Weld carbonatite has a thick weathering/regolith layer (10 to >70 m) of laterite overlying the unweathered carbonatite that contains high-grade REO deposits and concentrations of niobium, zirconium, and other 'rare' metals. A zone of supergene-enrichment contains abundant insoluble phosphates, aluminophosphates, clays, crandallite group minerals, iron and manganese-bearing oxides that contain elevated concentrations of REE, Y, U, Th, Nb, Ta, Zr, Ti, V, Cr, Ba and Sr, including economic accumulations of REE, niobium-tantalum and phosphatic minerals. Extreme lateritic weathering prevailed in the supergene zone over a protracted period of time and resulted in the degradation of the residual magmatic REE-bearing minerals. The majority of the REOs are contained within secondary, low Th phosphate minerals with low levels of deleterious elements (e.g. F and Ca). The Central lanthanide deposit contains an indicative mix of predominantly LREE and shows the following proportions when summed to 100%: CeO<sub>2</sub> (46.7%), La<sub>2</sub>O<sub>3</sub> (25.5%), Nd<sub>2</sub>O<sub>3</sub> (18.5%), Pr<sub>6</sub>O<sub>11</sub> (5.32%), Sm<sub>2</sub>O<sub>3</sub> (2.27%) and Eu<sub>2</sub>O<sub>3</sub> (0.443%), together with minor components of HREE: Dy<sub>2</sub>O<sub>3</sub> (0.124%) and Tb<sub>4</sub>O<sub>7</sub> (0.068%).

## COMMINUTION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 465 were prepared in the following manner:

- drying of materials to constant mass at 105°C;
- destruction of the hygroscopic property of the Mount Weld materials by roasting at 900°C for 2 hours;
- crushing and milling of materials to >99.5% minus 75 microns;
- preliminary homogenisation and check assaying of each material;
- blending in appropriate proportions to achieve the desired grades;
- packaging into 10g units sealed in laminated foil pouches and into 1kg units sealed in plastic jars.

## ANALYTICAL PROGRAM

Twenty one commercial analytical laboratories participated in the program to certify the 99 elements reported in Table 1. The following methods were employed:

- REE Suite XRF package (up to 7 laboratories depending on the element);
- Thermogravimetry for Loss On Ignition (LOI) at 1000°C (7 laboratories);
- Borate/peroxide fusion for full elemental suite ICP-OES and ICP-MS (up to 15 laboratories depending on the element);
- 4-acid digestion (HF-HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) for full elemental suite ICP-OES and ICP-MS finish (up to 14 laboratories depending on the element).

Samples for the round robin program were taken at nine predetermined sampling intervals immediately following final homogenisation and are considered representative of the entire batch of OREAS 465. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate sampling lots. 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 99 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 66 indicative values. Table 3 provides performance gate intervals for the certified values of each method group based on their pooled 1SD's. Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 465 Datapack.xlsx**).

## 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 a statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if  $>2.5$ . After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

**Certified Values** are the means of accepted laboratory means after outlier filtering. Indicative (uncertified) values (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient ( $< 5$ ) to support certification; ii) inter-laboratory consensus is poor; or iii) a significant proportion of results are outlying.

**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 values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS 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 per cent 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.

**Table 3. Performance Gates for OREAS 465.**

| Constituent   | Certified Value | Absolute Standard Deviations |         |          |         |          | Relative Standard Deviations |        |        | 5% window |       |
|---|-----------------|------------------------------|---------|----------|---------|----------|------------------------------|--------|--------|-----------|-------|
|   |                 | 1SD                          | 2SD Low | 2SD High | 3SD Low | 3SD High | 1RSD                         | 2RSD   | 3RSD   | Low       | High  |
| <b>Borate Fusion XRF</b>  |                 |                              |         |          |         |          |                              |        |        |           |       |
| CeO <sub>2</sub> , wt.%   | 4.88            | 0.045                        | 4.79    | 4.97     | 4.74    | 5.01     | 0.93%                        | 1.85%  | 2.78%  | 4.63      | 5.12  |
| Fe <sub>2</sub> O <sub>3</sub> , wt.%   | 49.96           | 1.309                        | 47.34   | 52.58    | 46.03   | 53.89    | 2.62%                        | 5.24%  | 7.86%  | 47.46     | 52.46 |
| La <sub>2</sub> O <sub>3</sub> , wt.%   | 2.84            | 0.033                        | 2.77    | 2.90     | 2.74    | 2.94     | 1.16%                        | 2.33%  | 3.49%  | 2.70      | 2.98  |
| Nd <sub>2</sub> O <sub>3</sub> , wt.%   | 1.38            | 0.102                        | 1.17    | 1.58     | 1.07    | 1.68     | 7.43%                        | 14.86% | 22.29% | 1.31      | 1.45  |
| Pr <sub>6</sub> O <sub>11</sub> , ppm   | 4534            | 144                          | 4246    | 4821     | 4102    | 4965     | 3.17%                        | 6.35%  | 9.52%  | 4307      | 4760  |
| Sm <sub>2</sub> O <sub>3</sub> , ppm  | 1640            | 136                          | 1368    | 1911     | 1233    | 2047     | 8.27%                        | 16.55% | 24.82% | 1558      | 1722  |
| ThO <sub>2</sub> , ppm  | 901             | 128                          | 645     | 1158     | 517     | 1286     | 14.23%                       | 28.46% | 42.68% | 856       | 946   |
| Y <sub>2</sub> O <sub>3</sub> , ppm   | 637             | 45                           | 547     | 728      | 502     | 773      | 7.06%                        | 14.13% | 21.19% | 606       | 669   |
| <b>Thermogravimetry</b>   |                 |                              |         |          |         |          |                              |        |        |           |       |
| LOI, wt. %  | 0.824           | 0.133                        | 0.558   | 1.089    | 0.425   | 1.222    | 16.12%                       | 32.23% | 48.35% | 0.783     | 0.865 |
| <b>Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)</b> |                 |                              |         |          |         |          |                              |        |        |           |       |
| Al, wt. %   | 6.60            | 0.261                        | 6.08    | 7.12     | 5.81    | 7.38     | 3.96%                        | 7.92%  | 11.88% | 6.27      | 6.93  |
| Al <sub>2</sub> O <sub>3</sub> , wt. %  | 12.47           | 0.494                        | 11.48   | 13.45    | 10.99   | 13.95    | 3.96%                        | 7.92%  | 11.88% | 11.84     | 13.09 |
| Ba, ppm   | 4397            | 464                          | 3469    | 5325     | 3005    | 5789     | 10.56%                       | 21.11% | 31.67% | 4177      | 4617  |
| BaO, ppm  | 4909            | 518                          | 3873    | 5946     | 3355    | 6464     | 10.56%                       | 21.11% | 31.67% | 4664      | 5155  |
| Be, ppm   | 13.2            | 0.89                         | 11.5    | 15.0     | 10.6    | 15.9     | 6.74%                        | 13.49% | 20.23% | 12.6      | 13.9  |
| Bi, ppm   | 16.9            | 1.04                         | 14.8    | 19.0     | 13.8    | 20.0     | 6.16%                        | 12.31% | 18.47% | 16.1      | 17.8  |
| Ca, wt. %   | 0.900           | 0.060                        | 0.780   | 1.020    | 0.720   | 1.080    | 6.67%                        | 13.33% | 20.00% | 0.855     | 0.945 |
| CaO, wt. %  | 1.26            | 0.084                        | 1.09    | 1.43     | 1.01    | 1.51     | 6.67%                        | 13.33% | 20.00% | 1.20      | 1.32  |
| Ce, wt. %   | 3.95            | 0.135                        | 3.68    | 4.22     | 3.55    | 4.36     | 3.42%                        | 6.83%  | 10.25% | 3.75      | 4.15  |
| CeO <sub>2</sub> , wt. %  | 4.86            | 0.166                        | 4.52    | 5.19     | 4.36    | 5.35     | 3.42%                        | 6.83%  | 10.25% | 4.61      | 5.10  |
| Cr, ppm   | 544             | 42                           | 460     | 628      | 418     | 670      | 7.70%                        | 15.40% | 23.10% | 517       | 571   |
| Cr <sub>2</sub> O <sub>3</sub> , ppm  | 795             | 61                           | 673     | 918      | 612     | 979      | 7.70%                        | 15.40% | 23.10% | 756       | 835   |
| Cs, ppm   | < 0.1           | IND                          | IND     | IND      | IND     | IND      | IND                          | IND    | IND    | IND       | IND   |
| Dy, ppm   | 217             | 13                           | 192     | 242      | 179     | 254      | 5.80%                        | 11.61% | 17.41% | 206       | 228   |
| Dy <sub>2</sub> O <sub>3</sub> , ppm  | 249             | 14                           | 220     | 278      | 205     | 292      | 5.80%                        | 11.61% | 17.41% | 236       | 261   |
| Er, ppm   | 50              | 3.1                          | 44      | 57       | 41      | 60       | 6.22%                        | 12.44% | 18.66% | 48        | 53    |
| Er <sub>2</sub> O <sub>3</sub> , ppm  | 58              | 3.6                          | 51      | 65       | 47      | 68       | 6.22%                        | 12.44% | 18.66% | 55        | 61    |
| Eu, ppm   | 286             | 11                           | 263     | 309      | 251     | 320      | 4.02%                        | 8.03%  | 12.05% | 271       | 300   |

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  |
| <b>Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)</b> |                 |                              |         |          |         |          |                              |        |        |           |       |
| Eu <sub>2</sub> O <sub>3</sub> , ppm  | 331             | 13                           | 304     | 357      | 291     | 371      | 4.02%                        | 8.03%  | 12.05% | 314       | 347   |
| Fe, wt.%  | 34.71           | 0.634                        | 33.45   | 35.98    | 32.81   | 36.61    | 1.83%                        | 3.65%  | 5.48%  | 32.98     | 36.45 |
| Fe <sub>2</sub> O <sub>3</sub> , wt.%   | 49.63           | 0.906                        | 47.82   | 51.44    | 46.91   | 52.35    | 1.83%                        | 3.65%  | 5.48%  | 47.15     | 52.11 |
| Ga, ppm   | 214             | 43                           | 128     | 299      | 85      | 342      | 20.03%                       | 40.06% | 60.08% | 203       | 224   |
| Gd, ppm   | 584             | 31                           | 523     | 646      | 492     | 676      | 5.25%                        | 10.50% | 15.76% | 555       | 614   |
| Gd <sub>2</sub> O <sub>3</sub> , ppm  | 674             | 35                           | 603     | 744      | 567     | 780      | 5.25%                        | 10.50% | 15.76% | 640       | 707   |
| Hf, ppm   | 41.4            | 7.2                          | 27.0    | 55.8     | 19.8    | 63.0     | 17.41%                       | 34.82% | 52.22% | 39.3      | 43.5  |
| HfO <sub>2</sub> , ppm  | 48.8            | 8.5                          | 31.8    | 65.8     | 23.3    | 74.3     | 17.41%                       | 34.82% | 52.22% | 46.4      | 51.3  |
| Ho, ppm   | 27.7            | 2.13                         | 23.4    | 32.0     | 21.3    | 34.1     | 7.70%                        | 15.39% | 23.09% | 26.3      | 29.1  |
| Ho <sub>2</sub> O <sub>3</sub> , ppm  | 31.7            | 2.44                         | 26.8    | 36.6     | 24.4    | 39.1     | 7.70%                        | 15.39% | 23.09% | 30.1      | 33.3  |
| In, ppm   | 3.47            | 0.227                        | 3.02    | 3.92     | 2.79    | 4.15     | 6.53%                        | 13.07% | 19.60% | 3.30      | 3.64  |
| La, wt.%  | 2.41            | 0.082                        | 2.25    | 2.58     | 2.17    | 2.66     | 3.41%                        | 6.83%  | 10.24% | 2.29      | 2.53  |
| La <sub>2</sub> O <sub>3</sub> , wt.%   | 2.83            | 0.097                        | 2.64    | 3.02     | 2.54    | 3.12     | 3.41%                        | 6.83%  | 10.24% | 2.69      | 2.97  |
| Lu, ppm   | 2.10            | 0.122                        | 1.86    | 2.35     | 1.74    | 2.47     | 5.78%                        | 11.56% | 17.34% | 2.00      | 2.21  |
| Lu <sub>2</sub> O <sub>3</sub> , ppm  | 2.39            | 0.138                        | 2.12    | 2.67     | 1.98    | 2.81     | 5.78%                        | 11.56% | 17.34% | 2.27      | 2.51  |
| Mg, wt.%  | 0.392           | 0.021                        | 0.351   | 0.433    | 0.330   | 0.453    | 5.23%                        | 10.47% | 15.70% | 0.372     | 0.411 |
| MgO, wt.%   | 0.650           | 0.034                        | 0.582   | 0.718    | 0.548   | 0.752    | 5.23%                        | 10.47% | 15.70% | 0.617     | 0.682 |
| Mn, wt.%  | 0.263           | 0.023                        | 0.217   | 0.308    | 0.194   | 0.331    | 8.72%                        | 17.43% | 26.15% | 0.249     | 0.276 |
| MnO, wt.%   | 0.339           | 0.030                        | 0.280   | 0.398    | 0.250   | 0.428    | 8.72%                        | 17.43% | 26.15% | 0.322     | 0.356 |
| Mo, ppm   | 114             | 6                            | 103     | 125      | 97      | 131      | 4.97%                        | 9.95%  | 14.92% | 108       | 120   |
| Nb, ppm   | 4680            | 301                          | 4078    | 5283     | 3776    | 5584     | 6.44%                        | 12.87% | 19.31% | 4446      | 4914  |
| Nb <sub>2</sub> O <sub>5</sub> , ppm  | 6695            | 431                          | 5833    | 7557     | 5402    | 7988     | 6.44%                        | 12.87% | 19.31% | 6360      | 7030  |
| Nd, wt.%  | 1.18            | 0.050                        | 1.08    | 1.28     | 1.03    | 1.33     | 4.29%                        | 8.57%  | 12.86% | 1.12      | 1.24  |
| Nd <sub>2</sub> O <sub>3</sub> , wt.%   | 1.37            | 0.059                        | 1.25    | 1.49     | 1.20    | 1.55     | 4.29%                        | 8.57%  | 12.86% | 1.30      | 1.44  |
| P, wt.%   | 3.81            | 0.122                        | 3.57    | 4.06     | 3.45    | 4.18     | 3.21%                        | 6.42%  | 9.63%  | 3.62      | 4.00  |
| P <sub>2</sub> O <sub>5</sub> , wt.%  | 8.74            | 0.280                        | 8.18    | 9.30     | 7.89    | 9.58     | 3.21%                        | 6.42%  | 9.63%  | 8.30      | 9.17  |
| Pb, ppm   | 506             | 19                           | 469     | 543      | 450     | 562      | 3.68%                        | 7.36%  | 11.04% | 481       | 531   |
| PbO, ppm  | 545             | 20                           | 505     | 585      | 485     | 605      | 3.68%                        | 7.36%  | 11.04% | 518       | 572   |
| Pr, ppm   | 3772            | 164                          | 3444    | 4100     | 3280    | 4264     | 4.35%                        | 8.70%  | 13.05% | 3583      | 3960  |
| Pr <sub>6</sub> O <sub>11</sub> , ppm   | 4557            | 198                          | 4161    | 4953     | 3963    | 5152     | 4.35%                        | 8.70%  | 13.05% | 4329      | 4785  |
| S, ppm  | 1941            | 132                          | 1676    | 2206     | 1544    | 2338     | 6.82%                        | 13.65% | 20.47% | 1844      | 2038  |
| Si, wt.%  | 1.53            | 0.043                        | 1.45    | 1.62     | 1.40    | 1.66     | 2.83%                        | 5.65%  | 8.48%  | 1.46      | 1.61  |

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  |
| <b>Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)</b> |                 |                              |         |          |         |          |                              |        |        |           |       |
| SiO <sub>2</sub> , wt.%   | 3.28            | 0.093                        | 3.10    | 3.47     | 3.00    | 3.56     | 2.83%                        | 5.65%  | 8.48%  | 3.12      | 3.45  |
| Sm, ppm   | 1361            | 36                           | 1289    | 1433     | 1252    | 1470     | 2.66%                        | 5.32%  | 7.97%  | 1293      | 1429  |
| Sm <sub>2</sub> O <sub>3</sub> , ppm  | 1578            | 42                           | 1494    | 1662     | 1452    | 1704     | 2.66%                        | 5.32%  | 7.97%  | 1499      | 1657  |
| Sn, ppm   | 136             | 20                           | 95      | 176      | 75      | 196      | 14.89%                       | 29.78% | 44.68% | 129       | 143   |
| SnO <sub>2</sub> , ppm  | 172             | 26                           | 121     | 224      | 95      | 249      | 14.89%                       | 29.78% | 44.68% | 164       | 181   |
| Sr, ppm   | 5204            | 182                          | 4840    | 5568     | 4658    | 5750     | 3.50%                        | 7.00%  | 10.50% | 4944      | 5464  |
| SrO, ppm  | 6154            | 215                          | 5723    | 6585     | 5508    | 6800     | 3.50%                        | 7.00%  | 10.50% | 5846      | 6462  |
| Ta, ppm   | 79              | 5.0                          | 69      | 89       | 64      | 94       | 6.41%                        | 12.82% | 19.24% | 75        | 83    |
| Ta <sub>2</sub> O <sub>5</sub> , ppm  | 96              | 6.2                          | 84      | 108      | 78      | 115      | 6.41%                        | 12.82% | 19.24% | 91        | 101   |
| Tb, ppm   | 57              | 3.1                          | 50      | 63       | 47      | 66       | 5.52%                        | 11.03% | 16.55% | 54        | 59    |
| Tb <sub>4</sub> O <sub>7</sub> , ppm  | 67              | 3.7                          | 59      | 74       | 56      | 78       | 5.52%                        | 11.03% | 16.55% | 63        | 70    |
| Th, ppm   | 866             | 43                           | 780     | 951      | 737     | 994      | 4.95%                        | 9.89%  | 14.84% | 822       | 909   |
| ThO <sub>2</sub> , ppm  | 985             | 49                           | 887     | 1082     | 839     | 1131     | 4.95%                        | 9.89%  | 14.84% | 936       | 1034  |
| Ti, wt.%  | 6.30            | 0.184                        | 5.93    | 6.67     | 5.75    | 6.85     | 2.92%                        | 5.83%  | 8.75%  | 5.99      | 6.62  |
| TiO <sub>2</sub> , wt.%   | 10.51           | 0.307                        | 9.90    | 11.13    | 9.59    | 11.43    | 2.92%                        | 5.83%  | 8.75%  | 9.99      | 11.04 |
| Tm, ppm   | 4.52            | 0.262                        | 4.00    | 5.05     | 3.74    | 5.31     | 5.80%                        | 11.59% | 17.39% | 4.30      | 4.75  |
| Tm <sub>2</sub> O <sub>3</sub> , ppm  | 5.16            | 0.299                        | 4.57    | 5.76     | 4.27    | 6.06     | 5.80%                        | 11.59% | 17.39% | 4.91      | 5.42  |
| U, ppm  | 13.6            | 0.40                         | 12.8    | 14.4     | 12.4    | 14.8     | 2.95%                        | 5.89%  | 8.84%  | 12.9      | 14.3  |
| U <sub>3</sub> O <sub>8</sub> , ppm   | 16.0            | 0.47                         | 15.1    | 17.0     | 14.6    | 17.5     | 2.95%                        | 5.89%  | 8.84%  | 15.2      | 16.8  |
| V, ppm  | 534             | 36                           | 463     | 605      | 427     | 641      | 6.66%                        | 13.32% | 19.99% | 507       | 561   |
| V <sub>2</sub> O <sub>5</sub> , ppm   | 953             | 64                           | 826     | 1081     | 763     | 1144     | 6.66%                        | 13.32% | 19.99% | 906       | 1001  |
| W, ppm  | 7.52            | 1.24                         | 5.04    | 10.00    | 3.81    | 11.23    | 16.47%                       | 32.93% | 49.40% | 7.14      | 7.90  |
| WO <sub>3</sub> , ppm   | 9.48            | 1.56                         | 6.36    | 12.61    | 4.80    | 14.17    | 16.47%                       | 32.93% | 49.40% | 9.01      | 9.96  |
| Y, ppm  | 524             | 33                           | 457     | 590      | 424     | 623      | 6.34%                        | 12.67% | 19.01% | 498       | 550   |
| Y <sub>2</sub> O <sub>3</sub> , ppm   | 665             | 42                           | 581     | 749      | 539     | 792      | 6.34%                        | 12.67% | 19.01% | 632       | 698   |
| Yb, ppm   | 19.0            | 0.72                         | 17.5    | 20.4     | 16.8    | 21.2     | 3.82%                        | 7.63%  | 11.45% | 18.0      | 19.9  |
| Yb <sub>2</sub> O <sub>3</sub> , ppm  | 21.6            | 0.83                         | 20.0    | 23.3     | 19.1    | 24.1     | 3.82%                        | 7.63%  | 11.45% | 20.5      | 22.7  |
| Zr, ppm   | 1880            | 203                          | 1473    | 2286     | 1270    | 2489     | 10.81%                       | 21.62% | 32.42% | 1786      | 1974  |
| ZrO <sub>2</sub> , ppm  | 2539            | 274                          | 1990    | 3088     | 1716    | 3362     | 10.81%                       | 21.62% | 32.42% | 2412      | 2666  |
| <b>4-Acid Digestion</b>   |                 |                              |         |          |         |          |                              |        |        |           |       |
| Ag, ppm   | 5.48            | 0.353                        | 4.78    | 6.19     | 4.42    | 6.54     | 6.44%                        | 12.88% | 19.32% | 5.21      | 5.76  |
| Al, wt.%  | 6.21            | 0.399                        | 5.41    | 7.01     | 5.01    | 7.41     | 6.43%                        | 12.86% | 19.30% | 5.90      | 6.52  |

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  |
| <b>4-Acid Digestion continued</b> |                 |                              |         |          |         |          |                              |        |        |           |       |
| Ba, ppm                           | 4359            | 158                          | 4043    | 4675     | 3885    | 4833     | 3.62%                        | 7.25%  | 10.87% | 4141      | 4577  |
| Be, ppm                           | 11.6            | 0.76                         | 10.1    | 13.1     | 9.3     | 13.9     | 6.52%                        | 13.04% | 19.56% | 11.0      | 12.2  |
| Bi, ppm                           | 17.3            | 1.03                         | 15.2    | 19.3     | 14.2    | 20.4     | 5.96%                        | 11.92% | 17.89% | 16.4      | 18.1  |
| Ca, wt.%                          | 0.872           | 0.062                        | 0.748   | 0.995    | 0.687   | 1.057    | 7.08%                        | 14.15% | 21.23% | 0.828     | 0.915 |
| Cd, ppm                           | 1.20            | 0.14                         | 0.92    | 1.49     | 0.78    | 1.63     | 11.83%                       | 23.66% | 35.49% | 1.14      | 1.26  |
| Ce, wt.%                          | 3.91            | 0.149                        | 3.61    | 4.20     | 3.46    | 4.35     | 3.81%                        | 7.62%  | 11.43% | 3.71      | 4.10  |
| Co, ppm                           | 18.7            | 1.26                         | 16.2    | 21.2     | 14.9    | 22.5     | 6.74%                        | 13.49% | 20.23% | 17.8      | 19.6  |
| Cs, ppm                           | < 0.1           | IND                          | IND     | IND      | IND     | IND      | IND                          | IND    | IND    | IND       | IND   |
| Cu, ppm                           | 128             | 5                            | 117     | 138      | 112     | 144      | 4.17%                        | 8.34%  | 12.51% | 121       | 134   |
| Dy, ppm                           | 215             | 9                            | 197     | 234      | 187     | 243      | 4.30%                        | 8.61%  | 12.91% | 204       | 226   |
| Er, ppm                           | 47.3            | 2.51                         | 42.2    | 52.3     | 39.7    | 54.8     | 5.31%                        | 10.61% | 15.92% | 44.9      | 49.6  |
| Eu, ppm                           | 282             | 12                           | 259     | 306      | 247     | 318      | 4.18%                        | 8.37%  | 12.55% | 268       | 296   |
| Fe, wt.%                          | 29.55           | 2.025                        | 25.50   | 33.60    | 23.48   | 35.63    | 6.85%                        | 13.70% | 20.55% | 28.08     | 31.03 |
| Ga, ppm                           | 188             | 31                           | 127     | 250      | 96      | 281      | 16.34%                       | 32.69% | 49.03% | 179       | 198   |
| Gd, ppm                           | 581             | 36                           | 509     | 653      | 474     | 689      | 6.16%                        | 12.33% | 18.49% | 552       | 610   |
| Hf, ppm                           | 14.4            | 1.9                          | 10.5    | 18.3     | 8.6     | 20.2     | 13.42%                       | 26.84% | 40.26% | 13.7      | 15.1  |
| Ho, ppm                           | 26.8            | 0.94                         | 24.9    | 28.6     | 24.0    | 29.6     | 3.49%                        | 6.99%  | 10.48% | 25.4      | 28.1  |
| In, ppm                           | 3.18            | 0.208                        | 2.77    | 3.60     | 2.56    | 3.81     | 6.54%                        | 13.08% | 19.62% | 3.02      | 3.34  |
| La, wt.%                          | 2.27            | 0.093                        | 2.09    | 2.46     | 2.00    | 2.55     | 4.09%                        | 8.18%  | 12.27% | 2.16      | 2.39  |
| Li, ppm                           | 3.04            | 0.33                         | 2.38    | 3.71     | 2.05    | 4.04     | 10.91%                       | 21.83% | 32.74% | 2.89      | 3.19  |
| Lu, ppm                           | 1.72            | 0.28                         | 1.16    | 2.28     | 0.88    | 2.57     | 16.37%                       | 32.74% | 49.11% | 1.63      | 1.81  |
| Mg, wt.%                          | 0.374           | 0.026                        | 0.322   | 0.426    | 0.296   | 0.452    | 6.98%                        | 13.96% | 20.95% | 0.355     | 0.393 |
| Mn, wt.%                          | 0.198           | 0.022                        | 0.153   | 0.243    | 0.130   | 0.265    | 11.36%                       | 22.71% | 34.07% | 0.188     | 0.208 |
| Mo, ppm                           | 98              | 10                           | 78      | 118      | 68      | 128      | 10.35%                       | 20.71% | 31.06% | 93        | 103   |
| Na, wt.%                          | < 0.2           | IND                          | IND     | IND      | IND     | IND      | IND                          | IND    | IND    | IND       | IND   |
| Nd, wt.%                          | 1.10            | 0.043                        | 1.01    | 1.18     | 0.97    | 1.22     | 3.93%                        | 7.86%  | 11.78% | 1.04      | 1.15  |
| Ni, ppm                           | 106             | 14                           | 79      | 133      | 65      | 146      | 12.80%                       | 25.61% | 38.41% | 101       | 111   |
| P, wt.%                           | 3.15            | 0.38                         | 2.40    | 3.90     | 2.02    | 4.28     | 11.92%                       | 23.84% | 35.75% | 2.99      | 3.31  |
| Pb, ppm                           | 573             | 16                           | 540     | 605      | 524     | 621      | 2.81%                        | 5.61%  | 8.42%  | 544       | 601   |
| Pr, ppm                           | 3670            | 193                          | 3284    | 4055     | 3092    | 4248     | 5.25%                        | 10.50% | 15.76% | 3486      | 3853  |
| Rb, ppm                           | 0.43            | 0.05                         | 0.32    | 0.53     | 0.26    | 0.59     | 12.75%                       | 25.50% | 38.25% | 0.40      | 0.45  |
| Re, ppm                           | < 0.05          | IND                          | IND     | IND      | IND     | IND      | IND                          | IND    | IND    | IND       | IND   |

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  |
| <b>4-Acid Digestion continued</b> |                 |                              |         |          |         |          |                              |        |        |           |       |
| Sc, ppm                           | 149             | 9                            | 130     | 167      | 121     | 176      | 6.11%                        | 12.23% | 18.34% | 141       | 156   |
| Sm, ppm                           | 1307            | 81                           | 1145    | 1468     | 1064    | 1549     | 6.18%                        | 12.37% | 18.55% | 1241      | 1372  |
| Sr, wt.%                          | 0.505           | 0.026                        | 0.453   | 0.558    | 0.427   | 0.584    | 5.19%                        | 10.39% | 15.58% | 0.480     | 0.531 |
| Tb, ppm                           | 57              | 3.3                          | 50      | 64       | 47      | 67       | 5.83%                        | 11.66% | 17.50% | 54        | 60    |
| Te, ppm                           | < 1             | IND                          | IND     | IND      | IND     | IND      | IND                          | IND    | IND    | IND       | IND   |
| Th, ppm                           | 805             | 150                          | 505     | 1104     | 356     | 1254     | 18.60%                       | 37.20% | 55.79% | 764       | 845   |
| Tl, ppm                           | 0.087           | 0.009                        | 0.069   | 0.105    | 0.061   | 0.114    | 10.22%                       | 20.44% | 30.66% | 0.083     | 0.092 |
| Tm, ppm                           | 3.82            | 0.274                        | 3.28    | 4.37     | 3.00    | 4.65     | 7.17%                        | 14.34% | 21.51% | 3.63      | 4.01  |
| U, ppm                            | 12.6            | 0.68                         | 11.2    | 14.0     | 10.6    | 14.6     | 5.38%                        | 10.77% | 16.15% | 12.0      | 13.2  |
| V, ppm                            | 427             | 69                           | 289     | 565      | 220     | 634      | 16.17%                       | 32.34% | 48.51% | 405       | 448   |
| Y, ppm                            | 478             | 32                           | 413     | 543      | 381     | 575      | 6.77%                        | 13.54% | 20.32% | 454       | 502   |
| Yb, ppm                           | 14.9            | 0.95                         | 13.0    | 16.8     | 12.0    | 17.8     | 6.39%                        | 12.78% | 19.17% | 14.2      | 15.6  |
| Zn, ppm                           | 921             | 133                          | 655     | 1187     | 522     | 1320     | 14.44%                       | 28.87% | 43.31% | 875       | 967   |

Note: intervals may appear asymmetric due to rounding.

**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  $\text{La}_2\text{O}_3$  by fusion ICP, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 2.77 and 2.89 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).

The homogeneity of OREAS 465 has also been evaluated in an ANOVA study for all certified analytes. This study tests the null hypothesis that no statistically significant difference exists between the *between-unit variance* and the *within-unit variance* (i.e. p-values <0.05 indicate rejection of the null hypothesis). Of the 99 certified values, no failures were observed indicating no evidence to reject the null hypothesis.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 465 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PARTICIPATING LABORATORIES

1. ALS, Brisbane, QLD, Australia
2. ALS, Lima, Peru
3. ALS, Loughrea, Galway, Ireland
4. ALS, Perth, WA, Australia

5. ALS, Vancouver, BC, Canada
6. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
7. Bureau Veritas Geoanalytical, Perth, WA, Australia
8. Intertek Genalysis, Adelaide, SA, Australia
9. Intertek Genalysis, Perth, WA, Australia
10. Intertek Testing Services, Cupang, Muntinlupa, Philippines
11. Intertek Testing Services, Shunyi, Beijing, China
12. Nagrom, Perth, WA, Australia
13. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
14. SGS Australia Mineral Services, Perth (Newburn), WA, Australia
15. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
16. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
17. SGS Mineral Services, Townsville, QLD, Australia
18. SGS South Africa Pty Ltd, Booyens, Gauteng, South Africa
19. SGS Vostok Limited, Chita, Russian Federation
20. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
21. UIS Analytical Services, Centurion, South Africa

## PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 465 has been prepared, certified and is supplied by:

|                                    |   |
|------------------------------------|---|
| ORE Research & Exploration Pty Ltd | Tel: +613-9729 0333   |
| 37A Hosie Street                   | Fax: +613-9729 8338   |
| Bayswater North VIC 3153           | Web: <a href="http://www.ore.com.au">www.ore.com.au</a>     |
| AUSTRALIA                          | Email: <a href="mailto:info@ore.com.au">info@ore.com.au</a> |

It is available in unit sizes of 10g in laminated foil pouches or 1kg in plastic jars.

## INTENDED USE

OREAS 465 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 465 has been prepared from ore grade/waste REE bearing ore (TREO = 9.88%). The source materials (waste, low and medium grade REE ores) were found to be highly hygroscopic and this property was destroyed by roasting the materials at 900°C for 2 hours. Following re-equilibration of the materials to laboratory atmosphere the hygroscopic moisture content was deemed acceptable (~0.5% H<sub>2</sub>O-).

OREAS 465 has been packaged in single-use, 10g units in laminated foil pouches and 1kg units in plastic jars. 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 derived by 4-acid digestion and by fusion with ICP-OES/MS refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis.

In contrast the certified values derived by lithium borate fusion XRF and for LOI at 1000°C are on a dry sample basis. This is standard laboratory protocol for fusion XRF determinations and requires the removal of hygroscopic moisture by drying in air to constant mass at 105°C. If the reference material is not dried prior to analysis, the certified values should be corrected to the moisture-bearing basis.

## 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 and non-certified (indicative) values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

## 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.

## QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2008 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.



## LEGAL NOTICE

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

## CERTIFYING OFFICER



---

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.

Jaireth, S., Hoatson D.M., Mieзитis, Y. Ore Geology Reviews 62 (2014) 72-128 - Geological setting and resources of the major rare-earth-element deposits in Australia.