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OREAS
Certified Reference Materials

CERTIFICATE OF ANALYSIS FOR

PRIMARY QUARTZ BLANK
CERTIFIED REFERENCE MATERIAL
OREAS 22e

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 22e.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fire Assay						
Au, Gold (ppb)	< 1	IND	IND	IND	IND	IND
4-Acid Digestion						
Ag, Silver (ppm)	< 0.05	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	0.094	0.019	0.080	0.107	IND	IND
Ba, Barium (ppm)	3.92	1.08	3.23	4.60	IND	IND
Be, Beryllium (ppm)	0.061	0.018	0.050	0.072	IND	IND
Bi, Bismuth (ppm)	< 0.02	IND	IND	IND	IND	IND
Cd, Cadmium (ppm)	< 0.02	IND	IND	IND	IND	IND
Ce, Cerium (ppm)	2.26	0.187	2.20	2.31	2.02	2.50
Co, Cobalt (ppm)	0.68	0.048	0.65	0.71	IND	IND
Cr, Chromium (ppm)	6.47	1.11	5.29	7.65	IND	IND
Cs, Cesium (ppm)	0.095	0.010	0.089	0.102	IND	IND
Cu, Copper (ppm)	7.97	0.746	7.61	8.34	7.36	8.59
Fe, Iron (wt.%)	0.346	0.015	0.335	0.358	0.338	0.355
Ga, Gallium (ppm)	0.24	0.03	0.21	0.26	0.20	0.28
Hf, Hafnium (ppm)	0.23	0.05	0.21	0.26	IND	IND
In, Indium (ppm)	< 0.005	IND	IND	IND	IND	IND
K, Potassium (wt.%)	< 0.01	IND	IND	IND	IND	IND
La, Lanthanum (ppm)	1.09	0.12	1.04	1.14	IND	IND
Li, Lithium (ppm)	14.6	0.69	14.1	15.1	13.8	15.4
Mg, Magnesium (wt.%)	< 0.01	IND	IND	IND	IND	IND
Mn, Manganese (wt.%)	0.008	0.000	0.008	0.008	0.008	0.008
Mo, Molybdenum (ppm)	1.05	0.056	1.02	1.08	0.98	1.12
Na, Sodium (wt.%)	< 0.005	IND	IND	IND	IND	IND
Nb, Niobium (ppm)	1.03	0.066	0.99	1.06	IND	IND
Nd, Neodymium (ppm)	0.90	0.078	0.84	0.96	IND	IND
Ni, Nickel (ppm)	5.23	0.442	5.01	5.44	4.81	5.64
P, Phosphorus (wt.%)	< 0.005	IND	IND	IND	IND	IND
Pb, Lead (ppm)	< 1	IND	IND	IND	IND	IND
Rb, Rubidium (ppm)	0.32	0.06	0.29	0.35	IND	IND
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND	IND
S, Sulphur (wt.%)	< 0.005	IND	IND	IND	IND	IND
Sb, Antimony (ppm)	0.19	0.03	0.16	0.21	IND	IND
Se, Selenium (ppm)	< 1	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	0.20	0.009	0.19	0.21	IND	IND
Sn, Tin (ppm)	0.65	0.054	0.62	0.67	IND	IND
Sr, Strontium (ppm)	0.64	0.15	0.55	0.74	IND	IND
Ta, Tantalum (ppm)	< 0.1	IND	IND	IND	IND	IND
Tb, Terbium (ppm)	< 0.05	IND	IND	IND	IND	IND
Te, Tellurium (ppm)	< 0.05	IND	IND	IND	IND	IND
Th, Thorium (ppm)	0.68	0.054	0.64	0.71	0.57	0.79
Ti, Titanium (wt.%)	0.031	0.001	0.030	0.032	0.030	0.032

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						
Tl, Thallium (ppm)	< 0.02	IND	IND	IND	IND	IND
Tm, Thulium (ppm)	< 0.05	IND	IND	IND	IND	IND
U, Uranium (ppm)	0.13	0.04	0.11	0.16	IND	IND
V, Vanadium (ppm)	2.65	0.48	2.33	2.96	IND	IND
W, Tungsten (ppm)	0.17	0.05	0.13	0.21	IND	IND
Y, Yttrium (ppm)	0.63	0.09	0.56	0.70	IND	IND
Zn, Zinc (ppm)	4.33	1.47	3.28	5.38	IND	IND
Zr, Zirconium (ppm)	7.91	0.89	7.69	8.12	6.62	9.20

Note: intervals may appear asymmetric due to rounding

Table 2. Indicative Values for OREAS 22e.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Pd	ppb	*0.733*	Pt	ppb	*0.233*			
4-Acid Digestion								
As	ppm	*0.99*	Gd	ppm	*0.17*	Pr	ppm	*0.26*
Ca	wt.%	*0.007*	Ge	ppm	*0.16*	Sc	ppm	*0.18*
Dy	ppm	*0.13*	Hg	ppm	*0.023*	Yb	ppm	*< 0.1*
Er	ppm	*0.091*	Ho	ppm	*0.028*			
Eu	ppm	*< 0.05*	Lu	ppm	*0.024*			

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

INTRODUCTION

OREAS reference materials are intended to provide a low cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

SOURCE MATERIALS

OREAS 22e has been prepared from quartz sand to which 0.5% iron oxide has been added to produce a pale grey pulp. This colouring gives the material an appearance of primary origin (i.e. non-oxide). It is characterised by extremely low background gold of less than 1 part per billion.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 22e was prepared in the following manner:

- drying to constant mass at 105°C;
- preliminary blending of quartz sand with 0.5% iron oxide pigment;
- milling to approximately 99.5% less than 75 microns;
- final homogenisation;
- packaging in 10, 60 and 100g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

Ten commercial analytical laboratories participated in the program to characterise Au by fire assay with ICP-OES (4 labs), ICP-MS (5 labs) or AAS (1 lab) finish and full 48 element package by 4-acid (HF-HNO_3-HCl-$HClO_4$) digestion with ICP-OES and ICP-MS finish. Elements certified via 4-acid digestion include Ag, Al, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, Fe, Ga, Hf, In, K, La, Li, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Rb, Re, S, Sb, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Zn and Zr. Indicative values are provided for further 15 elements including Pd and Pt by Pb collection fire assay and As, Ca, Dy, Er, Eu, Gd, Ge, Hg, Ho, Lu, Pr, Sc and Yb by 4-acid digestion.

For the round robin program six 1.3kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire batch. The six samples received by each laboratory were obtained by taking one 110g split from each of the six test units. Table 1 (above) presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows indicative values. Table 3 provides performance gate intervals for the certified values based on their associated 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 22e 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 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 copper (Cu), where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 7.36 and 8.59

ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35). *Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.*

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 22e is fit-for-purpose as a certified reference material (see 'Intended Use' below).

Table 3. Performance Gates for OREAS 22e.

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
Pb Fire Assay											
Au, ppb	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
4-Acid Digestion											
Ag, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	0.094	0.019	0.056	0.131	0.038	0.150	19.90%	39.80%	59.69%	0.089	0.098
Ba, ppm	3.92	1.08	1.75	6.08	0.67	7.16	27.63%	55.25%	82.88%	3.72	4.11
Be, ppm	0.061	0.018	0.025	0.097	0.007	0.115	29.47%	58.94%	88.41%	0.058	0.064
Bi, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cd, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	2.26	0.187	1.88	2.63	1.70	2.82	8.30%	16.60%	24.89%	2.15	2.37
Co, ppm	0.68	0.048	0.58	0.78	0.54	0.83	7.09%	14.17%	21.26%	0.65	0.72
Cr, ppm	6.47	1.11	4.26	8.69	3.15	9.80	17.12%	34.24%	51.36%	6.15	6.80
Cs, ppm	0.095	0.010	0.076	0.115	0.066	0.125	10.32%	20.64%	30.97%	0.091	0.100
Cu, ppm	7.97	0.746	6.48	9.47	5.74	10.21	9.35%	18.70%	28.05%	7.58	8.37
Fe, wt.%	0.346	0.015	0.317	0.376	0.302	0.391	4.31%	8.61%	12.92%	0.329	0.364
Ga, ppm	0.24	0.03	0.17	0.31	0.13	0.34	14.52%	29.04%	43.56%	0.23	0.25
Hf, ppm	0.23	0.05	0.14	0.33	0.09	0.38	20.80%	41.60%	62.40%	0.22	0.25
In, ppm	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
La, ppm	1.09	0.12	0.86	1.33	0.74	1.45	10.80%	21.60%	32.40%	1.04	1.15
Li, ppm	14.6	0.69	13.2	16.0	12.6	16.7	4.72%	9.44%	14.16%	13.9	15.4
Mg, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Mn, wt.%	0.008	0.000	0.007	0.009	0.007	0.009	3.41%	6.83%	10.24%	0.008	0.008
Mo, ppm	1.05	0.056	0.94	1.16	0.88	1.22	5.36%	10.72%	16.08%	1.00	1.10
Na, wt.%	< 0.005	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
4-Acid Digestion											
Nb, ppm	1.03	0.066	0.90	1.16	0.83	1.23	6.42%	12.84%	19.26%	0.98	1.08
Nd, ppm	0.90	0.078	0.75	1.06	0.67	1.14	8.69%	17.37%	26.06%	0.86	0.95
Ni, ppm	5.23	0.442	4.34	6.11	3.90	6.55	8.46%	16.91%	25.37%	4.96	5.49
P, wt.%	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pb, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Rb, ppm	0.32	0.06	0.21	0.43	0.15	0.49	17.91%	35.82%	53.73%	0.30	0.34
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sb, ppm	0.19	0.03	0.12	0.25	0.09	0.28	17.20%	34.40%	51.60%	0.18	0.20
Se, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	0.20	0.009	0.18	0.21	0.17	0.22	4.42%	8.85%	13.27%	0.19	0.21
Sn, ppm	0.65	0.054	0.54	0.75	0.48	0.81	8.42%	16.85%	25.27%	0.61	0.68
Sr, ppm	0.64	0.15	0.34	0.95	0.19	1.10	23.70%	47.40%	71.10%	0.61	0.68
Ta, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Te, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	0.68	0.054	0.57	0.78	0.52	0.84	7.97%	15.93%	23.90%	0.64	0.71
Ti, wt.%	0.031	0.001	0.029	0.034	0.027	0.035	4.26%	8.52%	12.78%	0.030	0.033
Tl, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tm, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
U, ppm	0.13	0.04	0.05	0.21	0.01	0.26	30.89%	61.77%	92.66%	0.13	0.14
V, ppm	2.65	0.48	1.68	3.61	1.20	4.09	18.20%	36.41%	54.61%	2.52	2.78
W, ppm	0.17	0.05	0.06	0.27	0.01	0.33	32.26%	64.51%	96.77%	0.16	0.18
Y, ppm	0.63	0.09	0.45	0.81	0.36	0.90	14.22%	28.44%	42.67%	0.60	0.66
Zn, ppm	4.33	1.47	1.38	7.28	0.00	8.76	34.04%	68.08%	102.11 %	4.12	4.55
Zr, ppm	7.91	0.89	6.14	9.68	5.25	10.56	11.20%	22.39%	33.59%	7.51	8.30

Note: intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Perth, WA, Australia
4. ALS, Vancouver, BC, Canada

5. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
6. Bureau Veritas Geoanalytical, Perth, WA, Australia
7. Intertek Genalysis, Adelaide, SA, Australia
8. Intertek Genalysis, Perth, WA, Australia
9. SGS Australia Mineral Services, Perth, WA, Australia
10. SGS del Peru, Lima, Peru

PREPARER AND SUPPLIER

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It is available in unit sizes of 10, 60 and 100g (single-use laminated foil pouches) and 1kg (plastic jars).

INTENDED USE

OREAS 22e is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of Ag, Al, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, Fe, Ga, Hf, In, K, La, Li, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Rb, Re, S, Sb, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Zn and Zr in geological samples;
- for the verification of analytical methods for Ag, Al, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, Fe, Ga, Hf, In, K, La, Li, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Rb, Re, S, Sb, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Zn and Zr;
- for the calibration of instruments used in the determination of the concentration of Ag, Al, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, Fe, Ga, Hf, In, K, La, Li, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Rb, Re, S, Sb, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Zn and Zr.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 22e has been prepared from barren quartz blended with a small amount of iron oxide (0.5%). In its unopened state and under normal conditions of storage it 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 22e refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

HANDLING INSTRUCTIONS

The material is almost entirely made from crystalline silica (quartz) of which the fine dust is a known respiratory hazard. Respirable (<10 microns) crystalline silica has the potential to cause silicosis. Mandatory PPE includes safety glasses and dust masks when handling this material (see 'AIOH Position Paper (2009)' for further details').

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

CERTIFYING OFFICER

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

REFERENCES

AIOH Position Paper (2009) – Respirable Crystalline Silica and Occupational Health Issues (last accessed 20th October, 2016).

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.