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OREAS
Certified Reference Materials

CERTIFICATE OF ANALYSIS FOR

QUARTZ BLANK CERTIFIED REFERENCE MATERIAL

OREAS 22d

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

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)	< 0.1	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	0.132	0.008	0.123	0.142	IND	IND
As, Arsenic (ppm)	< 1	IND	IND	IND	IND	IND
Ba, Barium (ppm)	6.17	0.63	5.66	6.67	IND	IND
Bi, Bismuth (ppm)	< 0.1	IND	IND	IND	IND	IND
Cd, Cadmium (ppm)	< 0.1	IND	IND	IND	IND	IND
Co, Cobalt (ppm)	0.85	0.071	0.77	0.93	IND	IND
Cu, Copper (ppm)	9.23	1.34	7.85	10.61	8.28	10.18
Fe, Iron (wt.%)	0.468	0.037	0.432	0.504	0.455	0.481
Hf, Hafnium (ppm)	0.22	0.03	0.19	0.24	IND	IND
Li, Lithium (ppm)	14.2	1.33	12.5	16.0	13.5	15.0
Mn, Manganese (wt.%)	0.011	0.001	0.009	0.013	0.011	0.011
Mo, Molybdenum (ppm)	2.36	0.086	2.28	2.44	2.24	2.48
Nb, Niobium (ppm)	0.88	0.081	0.79	0.97	IND	IND
Ni, Nickel (ppm)	4.38	0.63	4.05	4.70	3.67	5.09
Pb, Lead (ppm)	0.72	0.26	0.49	0.95	IND	IND
Rb, Rubidium (ppm)	0.54	0.06	0.49	0.59	IND	IND
Sb, Antimony (ppm)	0.21	0.03	0.19	0.22	IND	IND
Sn, Tin (ppm)	0.61	0.013	0.59	0.62	IND	IND
Th, Thorium (ppm)	0.67	0.10	0.55	0.78	IND	IND
Ti, Titanium (wt.%)	0.021	0.001	0.021	0.022	0.019	0.024
U, Uranium (ppm)	0.18	0.05	0.13	0.22	IND	IND
V, Vanadium (ppm)	2.63	0.50	2.12	3.15	IND	IND
W, Tungsten (ppm)	0.21	0.03	0.18	0.24	IND	IND
Y, Yttrium (ppm)	0.69	0.07	0.61	0.76	IND	IND
Zn, Zinc (ppm)	6.70	1.31	5.59	7.81	IND	IND
Zr, Zirconium (ppm)	7.02	0.600	6.31	7.73	6.31	7.73

Note: intervals may appear asymmetric due to rounding

INTRODUCTION

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

SOURCE MATERIALS

OREAS 22d has been prepared from quartz sand to which 0.5% iron oxide has been added to produce a pale grey pulp. It is characterised by extremely low background gold of less than 1 part per billion.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 22d 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 99.9% less than 75 microns;
- final homogenisation;
- packaging in 60g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

Seven commercial analytical laboratories participated in the program to characterise gold by fire assay with ICP-OES (4 labs), ICP-MS (1 lab), AAS (1 lab) or SXAAS (1 lab) finish. Elements certified via 4-acid digestion include Ag, Al, As, Ba, Bi, Cd, Co, Cu, Fe, Hf, Li, Mn, Mo, Nb, Ni, Pb, Rb, Sb, Sn, Th, Ti, U, V, W, Y, Zn and Zr with ICP-OES or ICP-MS finish.

For the round robin program ten 500g 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 two 100g scoop splits from each of three separate 500g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance. Table 1 (above) presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows indicative values. Indicative values are provided for the major and trace elements determined by borate fusion XRF (Al_2O_3 to Zn) and laser ablation with ICP-MS (Ag to Zr) and are the means of duplicate assays from Bureau Veritas, Perth. Additional indicative values by other analytical methods are present 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 or reported as less than detection limits.

Table 2. Indicative Values for OREAS 22d

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Fire Assay								
Pd	ppb	*< 5*	Pt	ppb	*< 5*			
Borate Fusion XRF								
Al_2O_3	wt.%	*0.265*	Fe_2O_3	wt.%	*0.635*	Pb	ppm	*< 10*
As	ppm	*< 10*	K_2O	wt.%	*0.014*	SiO_2	wt.%	*98.84*
Ba	ppm	*< 10*	MgO	wt.%	*0.035*	Sn	ppm	*< 10*
CaO	wt.%	*0.015*	MnO	wt.%	*0.020*	SO_3	wt.%	*0.011*
Co	ppm	*7.50*	Na_2O	wt.%	*0.050*	TiO_2	wt.%	*0.023*
Cr	ppm	*25.0*	Ni	ppm	*< 10*	U	ppm	*< 10*
Cu	ppm	*< 10*	P_2O_5	wt.%	*0.005*	Zn	ppm	*< 10*
Thermogravimetry								
LOI^{1000}	wt.%	*0.130*						
Laser Ablation ICP-MS								
Ag	ppm	*< 0.1*	Ho	ppm	*0.045*	Sn	ppm	*0.60*
As	ppm	*0.70*	In	ppm	*0.038*	Sr	ppm	*1.20*
Ba	ppm	*5.75*	La	ppm	*1.26*	Ta	ppm	*0.055*
Be	ppm	*0.60*	Lu	ppm	*0.020*	Tb	ppm	*0.040*
Bi	ppm	*0.015*	Mn	wt.%	*0.010*	Te	ppm	*< 0.2*
Cd	ppm	*< 0.1*	Mo	ppm	*2.10*	Th	ppm	*0.77*
Ce	ppm	*2.41*	Nb	ppm	*0.88*	Ti	wt.%	*0.022*
Co	ppm	*0.90*	Nd	ppm	*1.06*	Tl	ppm	*< 0.2*
Cr	ppm	*18.5*	Ni	ppm	*6.00*	Tm	ppm	*0.030*
Cs	ppm	*0.13*	Pb	ppm	*0.75*	U	ppm	*0.16*
Cu	ppm	*9.00*	Pr	ppm	*0.28*	V	ppm	*3.20*
Dy	ppm	*0.21*	Rb	ppm	*0.48*	W	ppm	*0.25*
Er	ppm	*0.10*	Re	ppm	*< 0.01*	Y	ppm	*1.14*
Eu	ppm	*0.025*	Sb	ppm	*0.25*	Yb	ppm	*0.15*
Ga	ppm	*0.35*	Sc	ppm	*0.25*	Zn	ppm	*12.5*
Gd	ppm	*0.22*	Se	ppm	*< 5*	Zr	ppm	*13.8*
Hf	ppm	*0.50*	Sm	ppm	*0.25*			
4-Acid Digestion								
Be	ppm	*0.066*	In	ppb	*< 5*	Se	ppm	*< 1*
Ca	wt.%	*0.010*	K	wt.%	*0.008*	Sm	ppm	*0.22*
Ce	ppm	*2.43*	La	ppm	*1.20*	Sr	ppm	*1.14*
Cr	ppm	*11.9*	Lu	ppm	*0.013*	Ta	ppm	*0.036*
Cs	ppm	*0.10*	Mg	wt.%	*0.009*	Tb	ppm	*< 0.05*
Dy	ppm	*0.15*	Na	wt.%	*0.009*	Te	ppm	*< 0.05*
Er	ppm	*< 0.1*	Nd	ppm	*1.03*	Tl	ppm	*< 0.02*
Eu	ppm	*< 0.05*	P	wt.%	*0.001*	Tm	ppm	*< 0.05*
Ga	ppm	*0.26*	Pr	ppm	*0.32*	Yb	ppm	*< 0.1*
Gd	ppm	*0.20*	Re	ppm	*< 0.002*			
Ge	ppm	*0.065*	S	wt.%	*< 0.01*			
Ho	ppm	*< 0.05*	Sc	ppm	*0.20*			

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

Table 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 22d Datapack.xlsx**).

STATISTICAL ANALYSIS

Certified Values, Standard Deviations, Confidence and Tolerance Limits have been determined for each analytical method following removal of individual and laboratory outliers (Table 1). Certified Values are the mean of means after outlier filtering. The 95% Confidence Limit is a measure of the reliability of the certified value, i.e. the narrower the Confidence Interval the greater the certainty in the Certified Value. It should not be used as a control limit 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. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.

Performance Gates (Table 3) are 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.

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 ($1-\alpha=0.99$) at least 95% of subsamples ($\rho=0.95$) will have concentrations lying between 8.28 and 10.18ppm. 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 (IS0 Guide 35).

The homogeneity of OREAS 22d 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 28 certified values, no failures were observed indicating no evidence to reject the null hypothesis.

Table 3. Performance Gates for OREAS 22d

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
Fire Assay											
Au, ppb	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
4-Acid Digestion											
Ag, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	0.132	0.008	0.117	0.148	0.109	0.156	5.90%	11.80%	17.70%	0.126	0.139
As, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	6.17	0.63	4.91	7.43	4.28	8.06	10.23%	20.46%	30.69%	5.86	6.48
Bi, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cd, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Co, ppm	0.85	0.071	0.71	0.99	0.64	1.06	8.36%	16.71%	25.07%	0.81	0.89
Cu, ppm	9.23	1.34	6.55	11.91	5.21	13.25	14.51%	29.02%	43.53%	8.77	9.70
Fe, wt.%	0.468	0.037	0.394	0.542	0.357	0.579	7.91%	15.82%	23.73%	0.444	0.491
Hf, ppm	0.22	0.03	0.16	0.27	0.13	0.30	13.54%	27.07%	40.61%	0.20	0.23
Li, ppm	14.2	1.33	11.6	16.9	10.2	18.2	9.35%	18.70%	28.06%	13.5	14.9
Mn, wt.%	0.011	0.001	0.008	0.013	0.007	0.015	11.20%	22.40%	33.59%	0.010	0.011
Mo, ppm	2.36	0.086	2.19	2.53	2.10	2.62	3.64%	7.28%	10.91%	2.24	2.48
Nb, ppm	0.88	0.081	0.72	1.04	0.64	1.12	9.15%	18.30%	27.45%	0.84	0.92
Ni, ppm	4.38	0.63	3.12	5.64	2.49	6.27	14.41%	28.82%	43.23%	4.16	4.60
Pb, ppm	0.72	0.26	0.19	1.24	0.00	1.51	36.58%	73.16%	109.7%	0.68	0.75
Rb, ppm	0.54	0.06	0.42	0.66	0.35	0.72	11.43%	22.85%	34.28%	0.51	0.57
Sb, ppm	0.21	0.03	0.15	0.27	0.12	0.30	14.65%	29.31%	43.96%	0.20	0.22
Sn, ppm	0.61	0.013	0.58	0.63	0.57	0.64	2.18%	4.37%	6.55%	0.57	0.64
Th, ppm	0.67	0.10	0.48	0.86	0.38	0.95	14.27%	28.53%	42.80%	0.63	0.70
Ti, wt.%	0.021	0.001	0.020	0.023	0.019	0.024	3.61%	7.22%	10.83%	0.020	0.022
U, ppm	0.18	0.05	0.08	0.28	0.03	0.33	27.71%	55.42%	83.12%	0.17	0.19
V, ppm	2.63	0.50	1.64	3.63	1.14	4.13	18.89%	37.77%	56.66%	2.50	2.77
W, ppm	0.21	0.03	0.15	0.27	0.12	0.30	15.00%	30.00%	45.00%	0.20	0.22
Y, ppm	0.69	0.07	0.54	0.83	0.46	0.91	10.85%	21.69%	32.54%	0.65	0.72
Zn, ppm	6.70	1.31	4.07	9.33	2.75	10.64	19.63%	39.25%	58.88%	6.36	7.03
Zr, ppm	7.02	0.600	5.82	8.22	5.22	8.82	8.54%	17.08%	25.62%	6.67	7.37

Note: intervals may appear asymmetric due to rounding

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

PARTICIPATING LABORATORIES

Acme, Vancouver, BC, Canada
ALS, Brisbane, QLD, Australia
ALS, Vancouver, BC, Canada
Amdel (BV), Adelaide, SA, Australia
Intertek Genalysis, Perth, WA, Australia
SGS, Perth, WA, Australia
Ultra Trace (BV), Perth, WA, Australia

PREPARER AND SUPPLIER

Certified reference material OREAS 22d is prepared, certified and supplied by:

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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 22d is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of Ag, Al, As, Au, Ba, Bi, Cd, Co, Cu, Fe, Hf, Li, Mn, Mo, Nb, Ni, Pb, Rb, Sb, Sn, Th, Ti, U, V, W, Y, Zn and Zr in geological samples;
- for the verification of analytical methods for Ag, Al, As, Au, Ba, Bi, Cd, Co, Cu, Fe, Hf, Li, Mn, Mo, Nb, Ni, Pb, Rb, Sb, Sn, Th, Ti, U, V, W, Y, Zn and Zr;
- for the calibration of instruments used in the determination of the concentration of Ag, Al, As, Au, Ba, Bi, Cd, Co, Cu, Fe, Hf, Li, Mn, Mo, Nb, Ni, Pb, Rb, Sb, Sn, Th, Ti, U, V, W, Y, Zn and Zr.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 22d has been prepared from a barren quartz sample. In its unopened state under normal conditions of storage it has a shelf life beyond ten years.

INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 22d refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

HANDLING INSTRUCTIONS

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

TRACEABILITY

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis. The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs) for a particular analytical method, analyte or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

LEGAL NOTICE

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

QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2008 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

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