

**CERTIFICATE OF ANALYSIS FOR**

**SOIL LITHOGEOCHEM / BLANK**

**REFERENCE MATERIAL**

**OREAS 25a**

**Table 1. Fusion XRF - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 25a**

Constituent (wt.%)	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion XRF</b>						
Aluminium Oxide, Al <sub>2</sub> O <sub>3</sub>	18.24	0.304	18.01	18.47	18.12	18.36
Calcium Oxide, CaO	0.438	0.007	0.433	0.442	0.434	0.442
Iron(III) Oxide, Fe <sub>2</sub> O <sub>3</sub>	9.77	0.144	9.66	9.88	9.71	9.84
Manganese Oxide, MnO	0.063	0.004	0.061	0.065	IND	IND
Phosphorus Oxide, P <sub>2</sub> O <sub>5</sub>	0.117	0.006	0.113	0.122	0.115	0.119
Potassium Oxide, K <sub>2</sub> O	0.599	0.007	0.593	0.604	0.590	0.607
Silicon Dioxide, SiO <sub>2</sub>	56.7	0.84	56.0	57.3	56.4	56.9
Sodium Oxide, Na <sub>2</sub> O	0.191	0.008	0.185	0.196	IND	IND
Titanium Oxide, TiO <sub>2</sub>	1.93	0.051	1.89	1.97	1.91	1.94

Note: intervals may appear asymmetric due to rounding.

**Table 2. Fusion ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 25a**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	9.25	0.131	9.19	9.31	9.04	9.46
Barium, Ba (ppm)	151	8.0	146	157	148	155
Calcium, Ca (wt.%)	0.302	0.024	0.286	0.318	0.288	0.316
Cerium, Ce (ppm)	51	1.6	50	52	49	52
Cesium, Cs (ppm)	6.36	0.368	6.09	6.62	6.15	6.56
Chromium, Cr (ppm)	125	13	116	134	118	131
Cobalt, Co (ppm)	8.05	0.529	7.62	8.48	7.73	8.37
Dysprosium, Dy (ppm)	4.31	0.189	4.19	4.42	4.12	4.49
Erbium, Er (ppm)	2.76	0.159	2.69	2.84	2.63	2.90
Europium, Eu (ppm)	0.80	0.031	0.78	0.82	0.76	0.84
Gadolinium, Gd (ppm)	3.79	0.222	3.64	3.94	3.61	3.97
Gallium, Ga (ppm)	25.4	1.35	24.4	26.3	24.7	26.0
Hafnium, Hf (ppm)	11.1	0.62	10.7	11.5	10.6	11.6
Holmium, Ho (ppm)	0.92	0.049	0.88	0.96	0.88	0.96
Iron, Fe (wt.%)	6.72	0.161	6.63	6.80	6.60	6.83
Lanthanum, La (ppm)	23.3	1.11	22.6	24.0	22.3	24.2
Lutetium, Lu (ppm)	0.45	0.038	0.42	0.48	0.42	0.48
Magnesium, Mg (wt.%)	0.324	0.011	0.319	0.329	0.311	0.337
Manganese, Mn (wt.%)	0.049	0.003	0.047	0.051	0.048	0.050
Neodymium, Nd (ppm)	20.0	0.64	19.6	20.4	19.3	20.7
Niobium, Nb (ppm)	26.5	1.18	25.6	27.3	25.7	27.2
Phosphorus, P (wt.%)	0.049	0.006	0.043	0.055	IND	IND
Potassium, K (wt.%)	0.493	0.023	0.477	0.510	0.480	0.507
Praseodymium, Pr (ppm)	5.33	0.227	5.19	5.48	5.15	5.51
Rubidium, Rb (ppm)	60	2.3	58	61	58	61
Samarium, Sm (ppm)	3.90	0.229	3.77	4.02	3.68	4.12
Scandium, Sc (ppm)	13.5	0.57	12.8	14.2	IND	IND
Silicon, Si (wt.%)	25.85	0.804	25.30	26.41	25.49	26.22
Strontium, Sr (ppm)	49.4	2.47	47.7	51.2	47.5	51.4
Tantalum, Ta (ppm)	1.99	0.123	1.91	2.07	1.87	2.11
Terbium, Tb (ppm)	0.66	0.051	0.63	0.68	0.61	0.70
Thorium, Th (ppm)	16.4	0.81	15.9	17.0	16.0	16.9
Thulium, Tm (ppm)	0.43	0.030	0.42	0.45	0.41	0.46
Tin, Sn (ppm)	4.83	0.92	4.18	5.48	IND	IND
Titanium, Ti (wt.%)	1.14	0.033	1.12	1.16	1.12	1.16
Tungsten, W (ppm)	2.89	0.284	2.73	3.05	IND	IND
Uranium, U (ppm)	3.51	0.119	3.43	3.60	3.42	3.60
Vanadium, V (ppm)	164	9.3	157	170	159	169
Ytterbium, Yb (ppm)	2.89	0.170	2.80	2.97	2.77	3.01
Yttrium, Y (ppm)	25.1	1.50	24.2	26.1	24.2	26.1
Zinc, Zn (ppm)	46.8	5.8	44.0	49.6	IND	IND
Zirconium, Zr (ppm)	398	15.3	387	409	384	411

Note: intervals may appear asymmetric due to rounding.

**Table 3. 4-Acid ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 25a**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Four Acid Digestion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	8.87	0.425	8.42	9.32	8.58	9.16
Antimony, Sb (ppm)	0.67	0.048	0.65	0.70	IND	IND
Barium, Ba (ppm)	147	4.7	145	149	141	152
Beryllium, Be (ppm)	1.02	0.17	0.87	1.17	IND	IND
Bismuth, Bi (ppm)	0.35	0.034	0.33	0.37	0.30	0.40
Calcium, Ca (wt.%)	0.309	0.008	0.301	0.317	0.298	0.319
Cerium, Ce (ppm)	48.9	2.71	47.0	50.8	45.9	51.9
Cesium, Cs (ppm)	6.46	0.334	6.15	6.77	6.23	6.69
Chromium, Cr (ppm)	115	8.1	109	120	105	124
Cobalt, Co (ppm)	8.20	0.663	7.58	8.82	7.87	8.53
Copper, Cu (ppm)	33.9	2.29	32.3	35.5	32.2	35.6
Gallium, Ga (ppm)	25.9	1.77	24.4	27.4	24.7	27.1
Hafnium, Hf (ppm)	4.53	0.53	4.03	5.04	4.26	4.80
Iron, Fe (wt.%)	6.60	0.157	6.48	6.71	6.38	6.82
Lanthanum, La (ppm)	21.8	2.02	20.2	23.5	20.3	23.4
Lead, Pb (ppm)	25.2	1.87	23.8	26.6	23.7	26.8
Lithium, Li (ppm)	36.7	1.34	35.8	37.7	35.2	38.3
Magnesium, Mg (wt.%)	0.327	0.017	0.316	0.339	0.316	0.339
Manganese, Mn (wt.%)	0.047	0.002	0.046	0.048	0.046	0.049
Molybdenum, Mo (ppm)	2.55	0.158	2.41	2.69	2.40	2.70
Nickel, Ni (ppm)	45.8	4.05	43.0	48.7	43.8	47.9
Niobium, Nb (ppm)	22.4	1.71	20.9	23.9	20.9	23.9
Phosphorus, P (wt.%)	0.048	0.002	0.046	0.050	0.046	0.050
Potassium, K (wt.%)	0.482	0.014	0.473	0.492	0.465	0.500
Rubidium, Rb (ppm)	61	3.9	58	64	57	64
Scandium, Sc (ppm)	13.7	1.05	12.8	14.7	IND	IND
Sodium, Na (wt.%)	0.134	0.004	0.131	0.136	0.127	0.140
Strontium, Sr (ppm)	48.5	1.99	47.0	49.9	45.9	51.0
Sulphur, S (wt.%)	0.051	0.002	0.049	0.053	IND	IND
Tantalum, Ta (ppm)	1.60	0.134	1.47	1.72	1.44	1.75
Thallium, Tl (ppm)	0.35	0.030	0.31	0.39	0.32	0.38
Thorium, Th (ppm)	15.8	0.97	15.1	16.5	14.9	16.8
Tin, Sn (ppm)	4.06	0.196	3.94	4.19	3.82	4.30
Titanium, Ti (wt.%)	0.977	0.059	0.915	1.038	0.945	1.008
Tungsten, W (ppm)	2.10	0.201	1.89	2.30	1.79	2.40
Uranium, U (ppm)	2.94	0.088	2.92	2.96	2.79	3.09
Vanadium, V (ppm)	157	8.3	150	165	152	163
Yttrium, Y (ppm)	12.3	2.1	10.5	14.0	11.5	13.0
Zinc, Zn (ppm)	44.4	2.60	42.9	45.9	42.2	46.6

Note: intervals may appear asymmetric due to rounding.

**Table 4. Aqua Regia ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 25a**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Aqua Regia Digestion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	5.85	0.78	5.16	6.54	5.69	6.00
Barium, Ba (ppm)	56	7	49	62	54	58
Bismuth, Bi (ppm)	0.30	0.025	0.28	0.33	IND	IND
Calcium, Ca (wt.%)	0.150	0.014	0.138	0.162	IND	IND
Cerium, Ce (ppm)	33.1	5.3	27.1	39.0	31.6	34.5
Cesium, Cs (ppm)	4.45	0.86	3.48	5.41	4.34	4.55
Chromium, Cr (ppm)	73	5.2	68	78	69	76
Cobalt, Co (ppm)	5.72	0.69	5.17	6.26	5.48	5.96
Copper, Cu (ppm)	24.9	2.8	22.6	27.2	23.1	26.6
Gallium, Ga (ppm)	20.6	2.6	18.5	22.7	19.8	21.4
Iron, Fe (wt.%)	5.99	0.459	5.60	6.38	5.81	6.17
Lead, Pb (ppm)	21.0	2.01	19.5	22.6	19.8	22.3
Manganese, Mn (wt.%)	0.042	0.003	0.040	0.045	0.041	0.044
Nickel, Ni (ppm)	26.9	3.4	23.8	29.9	25.7	28.0
Phosphorus, P (wt.%)	0.037	0.004	0.034	0.041	0.036	0.039
Potassium, K (wt.%)	0.131	0.009	0.123	0.139	IND	IND
Scandium, Sc (ppm)	8.64	0.799	7.97	9.32	8.28	9.01
Strontium, Sr (ppm)	17.3	1.18	16.4	18.3	16.5	18.1
Thallium, Tl (ppm)	0.20	0.02	0.17	0.23	IND	IND
Thorium, Th (ppm)	10.7	0.97	9.8	11.6	9.9	11.5
Tin, Sn (ppm)	2.70	0.27	2.42	2.99	2.52	2.89
Uranium, U (ppm)	1.49	0.18	1.29	1.69	1.38	1.59
Vanadium, V (ppm)	117	10.9	107	127	114	120
Yttrium, Y (ppm)	4.56	0.74	3.74	5.38	4.31	4.81
Zinc, Zn (ppm)	30.1	3.4	27.1	33.0	28.4	31.7

Note: intervals may appear asymmetric due to rounding.

**Table 5. Miscellaneous - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 25a**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fire Assay</b>						
Gold, Au (ppb)	< 2	NA	NA	NA	NA	NA
Palladium, Pd (ppb)	< 1	NA	NA	NA	NA	NA
Platinum, Pt (ppb)	< 1	NA	NA	NA	NA	NA
<b>IR Combustion Furnace</b>						
Carbon, C (wt.%)	1.56	0.078	1.50	1.62	1.54	1.58
Sulphur, S (wt.%)	0.044	0.007	0.040	0.048	IND	IND
<b>Thermogravimetry</b>						
Loss On Ignition, LOI (wt.%)	11.70	1.49	10.59	12.81	11.62	11.78

Note: intervals may appear asymmetric due to rounding; NA = Not Applicable

**Table 6. Indicative Values for OREAS 25a**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Fusion XRF</b>								
BaO	ppm	151	MgO	wt.%	0.579	V2O5	ppm	269
Cl	ppm	< 10	Ni	ppm	31.2	Zn	ppm	46.7
Co	ppm	10.0	S	wt.%	0.052	Zr	ppm	135
Cr2O3	ppm	167	Sr	ppm	56			
<b>Fusion ICP-OES/MS</b>								
Ag	ppm	0.570	Ge	ppm	2.02	Re	ppm	< 0.1
As	ppm	9.83	In	ppm	< 0.2	S	wt.%	0.046
B	ppm	39.2	Li	ppm	35.1	Sb	ppm	1.02
Be	ppm	0.94	Mo	ppm	2.99	Te	ppm	< 5
Bi	ppm	0.40	Na	wt.%	0.126	Tl	ppm	0.30
Cd	ppm	< 2	Ni	ppm	55			
Cu	ppm	39.1	Pb	ppm	24.4			
<b>Four Acid Digestion ICP-OES/MS</b>								
Ag	ppm	0.168	Ge	ppm	0.22	Sm	ppm	3.41
As	ppm	9.94	Ho	ppm	0.46	Tb	ppm	0.41
Cd	ppm	0.041	In	ppm	0.091	Te	ppm	0.10
Dy	ppm	2.67	Lu	ppm	0.23	Tm	ppm	0.30
Er	ppm	1.50	Nd	ppm	17.0	Yb	ppm	1.48
Eu	ppm	0.64	Pr	ppm	4.71	Zr	ppm	159
Gd	ppm	2.91	Se	ppm	2.86			
<b>Aqua Regia Digestion ICP-OES/MS</b>								
Ag	ppm	0.035	Ho	ppm	0.20	Rb	ppm	31.4
As	ppm	2.84	In	ppm	0.081	Re	ppm	< 0.05
Au	ppb	1	La	ppm	13.0	S	wt.%	0.050
B	ppm	5.92	Li	ppm	23.7	Sb	ppm	0.18
Be	ppm	0.65	Lu	ppm	0.057	Se	ppm	0.87
Cd	ppm	0.041	Mg	wt.%	0.193	Sm	ppm	2.26
Dy	ppm	1.15	Mo	ppm	1.36	Ta	ppm	0.099
Er	ppm	0.50	Na	wt.%	0.040	Tb	ppm	0.24
Eu	ppm	0.43	Nb	ppm	0.52	Te	ppm	< 0.02
Gd	ppm	1.74	Nd	ppm	11.7	Ti	wt.%	0.036
Ge	ppm	0.13	Pd	ppm	< 0.01	Tm	ppm	0.062
Hf	ppm	0.47	Pr	ppm	3.35	Yb	ppm	0.41
Hg	ppm	0.053	Pt	ppm	0.004	Zr	ppm	19.0

## 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

Reference material OREAS 25a was sourced from an in situ layer of mature soil developed over early tertiary tholeiitic basalt in outer eastern Melbourne, Victoria, Australia.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 25a was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and multi stage milling;
- homogenisation;
- packaging in 10g and 60g units into laminated foil pouches and in 1kg units into plastic jars.

## ANALYTICAL PROGRAM

Ten commercial analytical laboratories participated in the program to characterise the elements reported in Tables 1 to 6. The following methods were employed:

- Lithium borate fusion for full suite X-ray fluorescence (9 laboratories)
- Sodium peroxide fusion or lithium borate fusion for full suite ICP-OES and ICP-MS (10 laboratories)
- Four acid digestion for full suite ICP-OES and ICP-MS (9 laboratories)
- Aqua regia digestion for full suite ICP-OES and ICP-MS (9 laboratories)
- Fire assay with ICP-OES and ICP-MS for Au, Pd and Pt (9 laboratories)
- Infra-red combustion furnace for C and S (9 laboratories)
- Thermogravimetry for LOI (10 laboratories)

For the round robin program eleven 700g test units were taken at predetermined intervals during the bagging stage, immediately following final blending, and are considered representative of the entire batch. The six samples received by each laboratory were obtained

by taking two 110g scoop splits from each of three separate 700g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity. Tabulated results, together with uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are available upon request for this CRM (Datapack for OREAS 25a.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 (see Tables 1-5). 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.

Indicative values (Table 6) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) interlaboratory consensus is poor; or iii) a significant proportion of results are outlying or reported as less than detection limits.

**Standard Deviation** values (1SDs) are reported in Tables 1-5 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.

As a guide two or more analytical results lying outside the 2SD window may be regarded as warning or rejection, and rejection for single results lying outside the 3SD window in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

**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 by 4-acid digestion, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 32.2 and 35.6 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).

The homogeneity of OREAS 25a has also been evaluated in an ANOVA study for all certified analytes. This study indicates no evidence that between-unit variance is greater than within-unit variance.

Based on the statistical analysis of the results of the interlaboratory certification program it can be concluded that OREAS 25a is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## **PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL**

Reference material OREAS 25a has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd  
6-8 Gatwick Road  
Bayswater North VIC 3153  
AUSTRALIA

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

### **INTENDED USE**

OREAS 25a is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of analytes reported in Tables 1-5 in geological samples
- for the verification of analytical methods for analytes reported in Tables 1-5
- for the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1-5

### **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 25a was sourced from an in situ layer of mature soil developed over early tertiary tholeiitic basalt in outer eastern Melbourne, Victoria, Australia. 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 THE CORRECT USE OF THE REFERENCE MATERIAL**

The certified values for lithium borate fusion XRF and for LOI are on a dry basis whilst all other certified values are reported on an "as received" basis.

### **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.

### **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**)

## **PARTICIPATING LABORATORIES**

Acme Analytical Laboratories, Vancouver, BC, Canada  
Activation Laboratories, Ancaster, Ontario, Canada  
ALS, Brisbane, QLD, Australia  
ALS, Callao, Lima, Peru  
ALS, Vancouver, BC, Canada  
BV Amdel, Adelaide, SA, Australia  
BV Ultra Trace, Perth, WA, Australia  
Intertek Genalysis, Perth, WA, Australia  
SGS Mineral Services, Booyens, Gauteng, South Africa  
SGS Mineral Services, Toronto, Ontario, Canada

## **REFERENCES**

ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.  
ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.  
Hamlyn, C. L. (2012), Datapack for OREAS 25a.