



Certified Reference Materials

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CERTIFICATE OF ANALYSIS FOR

GOLD OXIDE ORE

CERTIFIED REFERENCE MATERIAL

OREAS 257

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 257

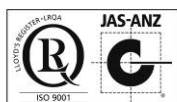
Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Pb Fire Assay						
Au, Gold (ppm)	14.18	0.264	14.10	14.26	14.15*	14.21*
Aqua Regia Digestion						
Au, Gold (ppm)	13.96	0.284	13.82	14.09	13.92 [†]	13.99 [†]
Gas / Liquid Pycnometry						
SG, Specific Gravity (Unity)	2.69	0.071	2.66	2.73	2.67	2.71

*Gold Tolerance Limits for typical 30g fire assay charge weight determined from 20 x 85mg NAA results and the Sampling Constant (Ingamells & Switzer, 1973);

[†]Gold Tolerance Limits for typical 25g aqua regia sample weight determined as above;

Please note: intervals may appear asymmetric due to rounding.

The homogeneity of OREAS 257 is of a level such that **sampling error is negligible** for a conventional fire assay or aqua regia determination.



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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

Certified Reference Material (CRM) OREAS 257 was prepared from gold-bearing Wilber Lode oxide ore from the Andy Well Gold Project. The Wilber Lode is a shear-hosted, narrow vein, quartz lode-style gold deposit situated within the Meekatharra-Wydney greenstone belt in the Archaean Yilgarn Craton of Western Australia. The common primary mineral assemblage as stated by Mason and Harris (2011, 2012, cited in Hingston et al, 2014) is quartz, calcite, chlorite, fuchsite, pyrite, galena, sphalerite, chalcocopyrite and gold. The host rock consists of a complex sequence of weathered Archaean meta-basalt and meta-porphyrific rocks derived from a primary mineralogy of albite, actinolite, chlorite, sericite, biotite, calcite, zoisite, muscovite, quartz and titanate. The Andy Well deposit is located approximately 45km north of Meekatharra in the Murchison region of Western Australia. OREAS 257 is one of a suite of five oxide gold CRMs ranging in gold content from 1.2 to 14ppm.

The approximate major and trace element composition of OREAS 257 is provided in Table 2. The non-certified values contained in this table are the means of six assays from one laboratory.

Table 2. Approximate major and trace element data for OREAS 257.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Pd	ppb	4	Pt	ppb	4			
Aqua Regia Digestion								
Ag	ppm	2.17	Ga	ppm	5.52	Sc	ppm	7.94
As	ppm	50	Gd	ppm	1.01	Sm	ppm	0.94
Be	ppm	0.14	Ho	ppm	0.20	Sr	ppm	16.5
Bi	ppm	0.74	La	ppm	6.02	Tb	ppm	0.16
Ce	ppm	12.5	Li	ppm	15.5	Th	ppm	1.33
Co	ppm	23.7	Nd	ppm	5.79	U	ppm	0.28
Cu	ppm	134	Ni	ppm	100	W	ppm	0.87
Dy	ppm	0.99	Pr	ppm	1.34	Y	ppm	5.65
Er	ppm	0.50	Rb	ppm	6.01	Yb	ppm	0.42
Eu	ppm	0.28	S	wt.%	0.109			
4-Acid Digestion								
Ag	ppm	2.15	Ge	ppm	0.060	Sc	ppm	19.7
Al	wt.%	5.05	Hf	ppm	1.29	Sm	ppm	1.46
Ba	ppm	291	Ho	ppm	0.32	Sr	ppm	39.7

Table 2 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion continued								
Be	ppm	0.64	K	wt.%	1.12	Tb	ppm	0.26
Bi	ppm	0.96	La	ppm	5.93	Te	ppm	0.43
Ca	wt.%	0.577	Li	ppm	23.1	Th	ppm	2.00
Cd	ppm	0.15	Lu	ppm	0.17	Ti	wt.%	0.186
Ce	ppm	16.1	Mg	wt.%	2.13	Tl	ppm	0.38
Co	ppm	25.9	Mn	wt.%	0.034	Tm	ppm	0.15
Cs	ppm	0.67	Mo	ppm	10.6	U	ppm	0.54
Cu	ppm	136	Na	wt.%	0.237	V	ppm	125
Dy	ppm	1.55	Nb	ppm	1.52	W	ppm	16.5
Er	ppm	0.96	Nd	ppm	6.87	Y	ppm	8.00
Eu	ppm	0.50	Ni	ppm	109	Yb	ppm	0.94
Fe	wt.%	3.21	P	wt.%	0.016	Zr	ppm	42.9
Ga	ppm	11.8	Pr	ppm	1.80			
Gd	ppm	1.47	Rb	ppm	51			
Laser Ablation								
Ag	ppm	2.45	Ho	ppm	0.29	Sn	ppm	0.60
As	ppm	58	In	ppm	0.038	Sr	ppm	34.0
Ba	ppm	285	La	ppm	7.27	Ta	ppm	0.15
Be	ppm	0.70	Lu	ppm	0.11	Tb	ppm	0.24
Bi	ppm	0.88	Mn	wt.%	0.037	Te	ppm	0.30
Cd	ppm	< 0.1	Mo	ppm	9.90	Th	ppm	1.73
Ce	ppm	13.6	Nb	ppm	1.69	Tl	ppm	< 0.2
Co	ppm	28.2	Nd	ppm	5.36	Tm	ppm	0.14
Cs	ppm	0.62	Ni	ppm	133	U	ppm	0.50
Cu	ppm	146	Pb	ppm	26.5	V	ppm	146
Dy	ppm	1.43	Pr	ppm	1.55	W	ppm	15.7
Er	ppm	0.83	Rb	ppm	44.9	Y	ppm	8.06
Eu	ppm	0.46	Re	ppm	< 0.01	Yb	ppm	0.92
Ga	ppm	11.6	Sb	ppm	1.10	Zn	ppm	55
Gd	ppm	1.24	Sc	ppm	18.0	Zr	ppm	44.8
Ge	ppm	1.23	Se	ppm	< 5			
Hf	ppm	1.33	Sm	ppm	1.18			
Borate Fusion XRF								
Al ₂ O ₃	wt.%	9.98	Fe ₂ O ₃	wt.%	4.60	P ₂ O ₅	wt.%	0.036
BaO	ppm	305	K ₂ O	wt.%	1.36	S	wt.%	0.112
CaO	wt.%	0.850	MgO	wt.%	3.57	SiO ₂	wt.%	74.30
Cl	ppm	45.0	MnO	wt.%	0.050	TiO ₂	wt.%	0.320
Cr ₂ O ₃	ppm	560	Na ₂ O	wt.%	0.335	V ₂ O ₅	ppm	245
Cu	ppm	124	Ni	ppm	114	Zr	ppm	44.4
Thermogravimetry								
LOI ¹⁰⁰⁰	wt.%	4.20						

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.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 257 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and milling of the gold ore material to 100% minus 30 microns;
- homogenisation;
- packaging in 60 and 100g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

Twenty-eight commercial analytical laboratories participated in the program to certify gold (as reported in Table 1) by the following methods:

- Gold via 25-40g fire assay with AAS (20 labs), ICP-OES (4 labs) or gravimetric (4 labs) finish;
- Instrumental neutron activation analysis for Au on 20 x 85mg subsamples to confirm homogeneity (1 laboratory).
- Gold via 15-50g aqua regia digestion with ICP-MS (13 labs), AAS (7 labs) or ICP-OES (2 lab) finish. It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. Aqua regia is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions which can include the ratio of nitric to hydrochloric acids, acid strength, temperatures, leach times and secondary digestions.
- Specific gravity by gas (13 labs) or liquid (4 labs) pycnometry.

For the round robin program twenty 1kg 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 1kg test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance. Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 149 indicative values for major and trace element composition. Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty ~85 milligram sample portions (see Table 3 below) and by a nested ANOVA program for both fire assay and aqua regia digestion (see '**nested ANOVA**' section). Table 4 provides performance gate intervals for the certified values based on their pooled 1SD's. Tabulated results of all elements (including Au INAA analyses) together with uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM³) are presented in the detailed certification data for this CRM (**OREAS 257 DataPack.xlsx**).

Table 3. Neutron Activation Analysis of Au (ppm) on 20 x 85mg subsamples.

Replicate No	NAA 0.085g
1	13.50
2	13.40
3	13.10
4	13.30
5	13.60
6	13.10
7	13.40
8	13.20
9	13.20
10	13.30
11	12.90
12	13.10
13	13.40
14	13.50
15	13.40
16	13.50
17	13.50
18	13.50
19	13.30
20	13.50
Mean	13.34
Median	13.40
Std Dev.	0.184
Rel.Std.Dev.	1.38%
PDM ³	-5.97%

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.

Certified Values are the means of accepted laboratory means after outlier filtering. The NAA data (see Table 3) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 257.

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.*

Indicative (uncertified) values (Table 2) 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 the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where inter-laboratory consensus is poor.

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 4 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.

Table 4. Performance Gates for OREAS 257.

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, ppm	14.18	0.264	13.65	14.71	13.39	14.97	1.86%	3.72%	5.59%	13.47	14.89
Aqua Regia Digestion											
Au, ppm	13.96	0.284	13.39	14.52	13.11	14.81	2.03%	4.06%	6.10%	13.26	14.66
Gas / Liquid Pycnometry											
SG, Unity	2.69	0.071	2.55	2.84	2.48	2.91	2.64%	5.27%	7.91%	2.56	2.83

Tolerance Limits (ISO Guide 3207) were determined by NAA using the reduced analytical subsample method that utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material (a sampling error is thereby induced) and measurement error becomes negligible.

In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.07% calculated for a 30g fire assay or aqua regia sample (1.38% at 85mg weight) confirms the very high level of gold homogeneity in OREAS 257. The homogeneity is of a level such that **sampling error is negligible** for a conventional fire assay or aqua regia determination.

Please note that these RSD's and tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.

The meaning of tolerance limits may be illustrated for gold by fire assay, where 99% of the time ($1-\alpha=0.99$) at least 95% of subsamples ($\rho=0.95$) will have concentrations lying between 14.15 and 14.21ppm. 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 gold homogeneity of OREAS 257 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty-eight round robin laboratories received six samples per CRM and these samples were made up of paired samples from three different, non-adjacent sampling intervals. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between-units to that of the variance within-units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 257. The test was performed using the following parameters:

- Gold fire assay – 168 samples (28 laboratories each providing analyses on 3 pairs of samples);

- Gold aqua regia digestion – 132 samples (22 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis, H_0 : Between-unit variance is no greater than within-unit variance (reject H_0 if p -value < 0.05);
- Alternative Hypothesis, H_1 : Between-unit variance is greater than within-unit variance.

P -values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The dataset was filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the p -value. This process derived p -values of 0.72 for Au by fire assay and 0.88 for Au by aqua regia digestion. Both p -values are insignificant and the Null Hypothesis is retained.

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 257 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 257 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. Actlabs, Coquimbo, Curarta, Chile
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Perth, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. ANSTO, Lucas Heights, NSW, Australia
10. Bureau Veritas, Abidjan, Cote D'ivoire
11. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
12. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. Bureau Veritas Kalassay, Kalgoorlie, WA, Australia
15. Bureau Veritas Minerals, Hermosillo, Sonora, Mexico
16. Intertek Genalysis, Adelaide, SA, Australia
17. Intertek Genalysis, Perth, WA, Australia
18. Intertek Testing Services, Cupang, Muntinlupa, Philippines
19. McClelland Laboratories Inc., Sparks, Nevada, USA
20. MinAnalytical Services, Perth, WA, Australia

21. Nagrom, Perth, WA, Australia
22. Newcrest Services Laboratory (NSL), Orange, NSW, Australia
23. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
24. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
25. SGS, Randfontein, Gauteng, South Africa
26. SGS del Peru, Lima, Peru
27. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
28. SGS Mineral Services, Townsville, QLD, Australia
29. Shiva Analyticals Ltd, Bangalore North, Karnataka, India

PREPARER AND SUPPLIER

Certified reference material OREAS 257 is prepared, certified and supplied by:



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

INTENDED USE

OREAS 257 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of gold by fire assay, gold by aqua regia digestion and specific gravity by pycnometry in geological samples;
- for the verification of analytical methods (gold fire assay, gold aqua regia digestion and specific gravity by pycnometry);
- for the calibration of instruments used in the determination of gold or specific gravity.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 257 has been prepared from gold-bearing Wilber Lode oxide ore from the Andy Well Gold Project. It is low in reactive sulphide and in its unopened state and under normal conditions of storage 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 257 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

A handwritten signature in black ink, appearing to read 'S. Hamlyn'.

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

REFERENCES

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