

CERTIFICATE OF ANALYSIS FOR

Gold Ore (Andy Well Gold Mine, Western Australia) CERTIFIED REFERENCE MATERIAL OREAS 221



Document: COA-1287-OREAS221-R1

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 221.

Constituent	Certified	SD	95% Confidence Limits		95% Tolerance Limits		
Constituent	Value	30	Low	High	Low	High	
Pb Fire Assay							
Au, Gold (ppm)	1.062	0.036	1.051 1.074		1.057*	1.067*	
Aqua Regia Digestion (sample weights 10-50g)							
Au, Gold (ppm)	1.042	0.039	1.026	1.058	1.037*	1.047*	
Gas / Liquid Pycnometry							
SG, Specific Gravity (Unity)	2.98	0.053	2.95	3.00	2.96	3.00	

SI unit equivalents: ppm, parts per million \equiv mg/kg \equiv μ g/g \equiv 0.0001 wt.% \equiv 1000 ppb, parts per billion.

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 221 was prepared from a blend of Archean greenstone-hosted Wilber Lode primary ore from the Andy Well Gold Mine and barren Cambrian greenstone sourced from a quarry north of Melbourne, Australia. The Wilber Lode is a shear-hosted, narrow vein, quartz lode-style gold deposit situated within the Meekatharra-Wydgee greenstone belt in the Archean 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, chalcopyrite and gold. The host rock consists of a complex sequence of Archean metabasalt and meta-porphyritic 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.

The approximate major and trace element composition of OREAS 221 is provided in Table 2. The non-certified values contained in this table are the means of duplicate assays from one laboratory.

^{*}Gold Tolerance Limits for typical 30g fire assay and 25g aqua regia digestion methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 221 was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of the barren materials to 98% minus 75 microns;
- Crushing and milling of the ore material to 100% minus 30 microns;
- Blending in appropriate proportions to achieve the desired grade;
- Packaging in 60g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

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

- Gold via 25-50g fire assay with AAS (24 labs) or ICP-OES (4 labs) finish;
- Instrumental neutron activation analysis for Au on 20 x 1g subsamples to confirm homogeneity (1 laboratory).
- Gold via 15-50g aqua regia digestion with ICP-MS (13 labs), AAS (7 labs) or ICP-OES (1 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 (12 labs) or liquid (4 labs) pycnometry.

For the round robin program twenty 1.5kg 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 66 indicative values for major and trace element composition. 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 221 DataPack - 1.1.181025_100056.xlsx).

Results are also presented in scatter plots for gold by fire assay and aqua regia digestion (Figures 1 and 2, respectively) together with ±3SD (magenta) and ±5% (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

Table 2. Indicative Values for OREAS 221.

Table 2. Illulcative values for OREAS 221.									
Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value	
Pb Fire Assay									
Pd	ppb	9.17	Pt	ppb	9.17				
Borate Fusion XRF									
Al_2O_3	wt.%	13.30	K ₂ O	wt.%	0.285	P ₂ O ₅	wt.%	0.101	
CaO	wt.%	9.80	MgO	wt.%	7.13	S	wt.%	0.197	
CI	ppm	10.0	MnO	wt.%	0.180	SiO ₂	wt.%	50.15	
Fe ₂ O ₃	wt.%	11.70	Na₂O	wt.%	2.83	TiO ₂	wt.%	1.08	
	Thermogravimetry								
LOI ¹⁰⁰⁰	wt.%	3.36							
Laser Abl	ation ICP	-MS							
Ag	ppm	0.250	Hf	ppm	1.86	Sm	ppm	2.34	
As	ppm	9.10	Но	ppm	0.82	Sn	ppm	1.50	
Ва	ppm	150	In	ppm	0.075	Sr	ppm	111	
Ве	ppm	0.50	La	ppm	4.12	Та	ppm	0.19	
Bi	ppm	0.10	Lu	ppm	0.30	Tb	ppm	0.58	
Cd	ppm	0.075	Mn	wt.%	0.146	Te	ppm	0.30	
Ce	ppm	9.91	Мо	ppm	1.50	Th	ppm	0.43	
Со	ppm	47.9	Nb	ppm	3.43	Ti	wt.%	0.636	
Cr	ppm	254	Nd	ppm	8.12	TI	ppm	< 0.2	
Cs	ppm	0.19	Ni	ppm	111	Tm	ppm	0.31	
Cu	ppm	152	Pb	ppm	5.50	U	ppm	0.025	
Dy	ppm	3.53	Pr	ppm	1.55	V	ppm	306	
Er	ppm	2.51	Rb	ppm	5.35	W	ppm	1.90	
Eu	ppm	0.89	Re	ppm	0.008	Y	ppm	22.5	
Ga	ppm	14.8	Sb	ppm	0.50	Yb	ppm	2.47	
Gd	ppm	2.93	Sc	ppm	43.5	Zn	ppm	88	
Ge	ppm	1.63	Se	ppm	< 5	Zr	ppm	63	
Si unit aquivalente: papa porte per million = $mal/a = ua/a = 0.0001$ ut $9/ = 1000$ papa porte per billion									

SI unit equivalents: ppm, parts per million \equiv mg/kg \equiv μ g/g \equiv 0.0001 wt.% \equiv 1000 ppb, parts per billion. 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.

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 INAA 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 221.

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 TiO_2), laser ablation with ICP-MS (Ag to Zr), LOI at 1000°C and C + S by infrared combustion furnace 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.

Homogeneity Evaluation

The homogeneity of gold has been determined by INAA using the reduced analytical subsample method which 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 and measurement error becomes negligible.

Table 3. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples showing the
equivalent results scaled to a 30g sample mass typical of fire assay determination.

Replicate	Au	Au
No	85mg actual	30g equivalent*
1	1.062	1.093
2	1.074	1.094
3	1.081	1.094
4	1.104	1.096
5	1.121	1.096
6	1.039	1.092
7	1.074	1.094
8	1.107	1.096
9	1.095	1.095
10	1.134	1.097
11	1.088	1.095
12	1.098	1.095
13	1.113	1.096
14	1.057	1.093
15	1.116	1.096
16	1.070	1.094
17	1.150	1.098
18	1.129	1.097
19	1.072	1.094
20	1.119	1.096
Mean	1.095	1.095
Median	1.096	1.095
Std Dev.	0.029	0.002
Rel.Std.Dev.	2.64%	0.140%

^{*}Results calculated for a 30g equivalent sample mass using the formula: $x^{30g \ Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@85mg} + \bar{X}$ where $x^{30g \ Eq} =$ equivalent result calculated for a 30g sample mass $(x^{INAA}) =$ raw INAA result at 85mg $\bar{X} =$ mean of 85mg INAA results

Table 3 above shows the INAA data determined on 20 x 85mg subsamples of OREAS 221. A subsample weight of 85 milligrams was employed and the 1RSD of 0.14% calculated for a 30g fire assay or aqua regia sample (2.64% at 85mg weights) confirms the high level of gold homogeneity in OREAS 221.

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 gold homogeneity of OREAS 221 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the thirty 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 221. The test was performed using the following parameters:

- Gold fire assay 180 samples (30 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion 120 samples (20 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis, H₀: Between-unit variance is no greater than within-unit variance (reject H₀ if *p*-value < 0.05);
- Alternative Hypothesis, H₁: 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 0.47 for Au by fire assay and 0.82 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 221 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 221 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

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. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL) \pm 10%.

i.e. Certified Value ± 10% ± 2DL (adapted from Govett, 1983)



Table 4. Pooled-Lab Performance Gates for OREAS 221.

Certified		Absolute Standard Deviations					Relative Standard Deviations			5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Pb Fire Assay	Pb Fire Assay										
Au, ppm	1.062	0.036	0.989	1.135	0.953	1.171	3.43%	6.86%	10.28%	1.009	1.115
Aqua Regia D	Aqua Regia Digestion										
Au, ppm	1.042	0.039	0.963	1.121	0.924	1.160	3.78%	7.55%	11.33%	0.990	1.094
Gas / Liquid Pycnometry											
SG, Unity	2.98	0.053	2.87	3.08	2.82	3.14	1.77%	3.53%	5.30%	2.83	3.13

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ μg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.

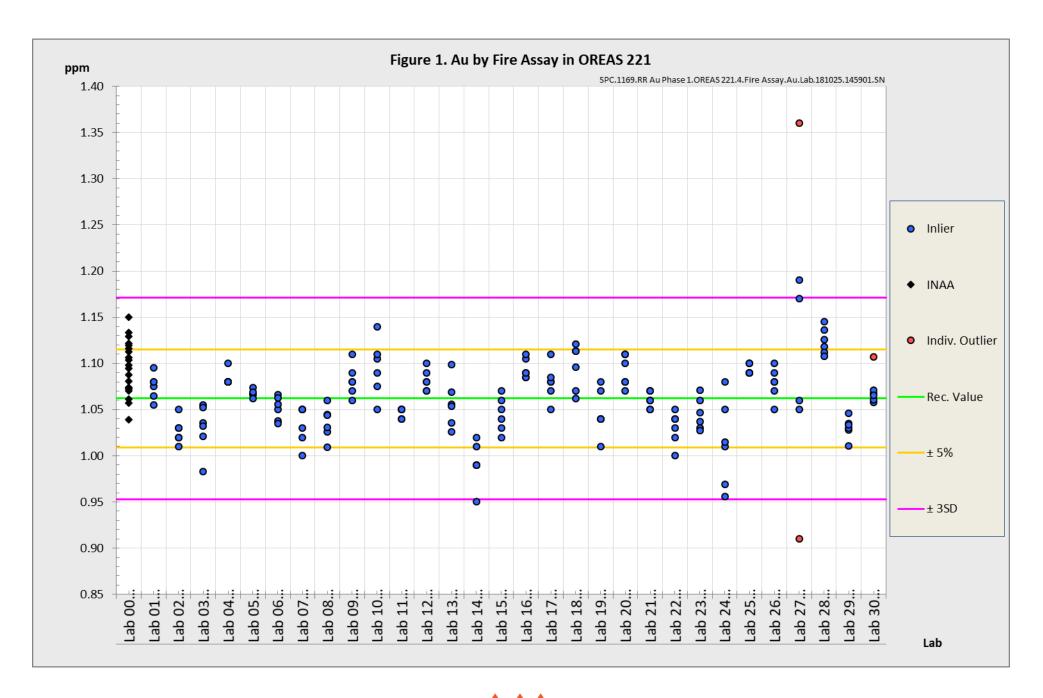
Note 1: intervals may appear asymmetric due to rounding.

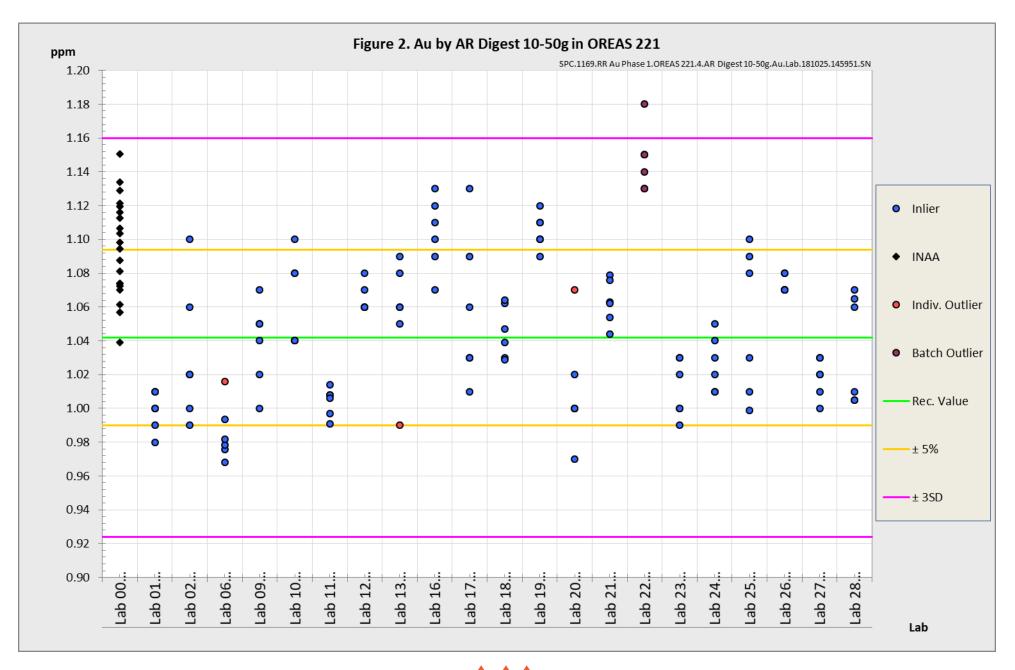
Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

PARTICIPATING LABORATORIES

- 1. Actlabs, Ancaster, Ontario, Canada
- 2. ALS, Brisbane, QLD, Australia
- 3. ALS, Lima, Peru
- 4. ALS, Loughrea, Galway, Ireland
- 5. ALS, Perth, WA, Australia
- 6. ALS, Vancouver, BC, Canada
- 7. Bureau Veritas, Abidjan, Cote D'ivoire
- 8. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 9. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 10. Bureau Veritas Geoanalytical, Perth, WA, Australia
- 11. Inspectorate (BV), Lima, Peru
- 12. Intertek Genalysis, Adelaide, SA, Australia
- 13. Intertek Genalysis, Perth, WA, Australia
- 14. Intertek Testing Services, Cupang, Muntinlupa, Philippines
- 15. MinAnalytical Services, Perth, WA, Australia
- 16. Nagrom, Perth, WA, Australia
- 17. Newcrest Services Laboratory (NSL), Orange, NSW, Australia
- 18. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 19. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 20. SGS, Randfontein, Gauteng, South Africa
- 21. SGS Australia Mineral Services, Kalgoorlie, WA, Australia
- 22. SGS Australia Mineral Services, Perth, WA, Australia
- 23. SGS del Peru, Lima, Peru
- 24. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 25. SGS Mineral Services, Townsville, QLD, Australia
- 26. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 27. Sucofindo Mineral Lab, Cibitung, West Java, Indonesia
- 28. Sucofindo Mineral Lab, Timika, Papua, Indonesia

Please note: The above numbered alphabetical list of participating laboratories <u>does</u> not reflect the Lab ID numbering on the scatter plots below.





PREPARER AND SUPPLIER

Certified reference material OREAS 221 is prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd
Tel: +613-9729 0333
37A Hosie Street
Fax: +613-9729 8338
Bayswater North VIC 3153
Web: www.ore.com.au
AUSTRALIA
Email: info@ore.com.au

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

METROLOGICAL 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 undertaken by ORE Pty Ltd) 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.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, "Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results (see ISO Guide 35:2006, Clause 10)."

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their field samples.

INTENDED USE

OREAS 221 is intended to cover all activities needed to produce a measurement result. This includes extraction, possible separation steps and the actual measurement process (the signal producing step). OREAS 221 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 221 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 221 has been prepared from primary gold ore diluted with barren greenstone. It is low in reactive sulphide (~0.20 wt.%) 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 221 refer to the concentration levels in its packaged state. There is no need for drying 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.

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.

DOCUMENT HISTORY

Revision No	Date	Changes applied
1	25 th Oct, 2018	Replaced original INAA data with new improved INAA data (a more precise method became available).
0	22 nd Dec, 2016	First publication.

QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2015 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.





CERTIFYING OFFICER

Sp

25th October, 2018

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

REFERENCES

Govett, G.J.S. (1983), ed. Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).

Hingston, R., Wellman, T. and Sternadt, G. (2014), The Geology of the Wilber Deposit, Andy Well Gold Project, Murchison District, Western Australia (pages 55-63, 9th International Mining Geology Conference 2014 - Proceedings - AusIMM).

Ingamells, C. O. and Switzer, P. (1973), Talanta 20, 547-568.

ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2015), Reference materials – Contents of certificates and labels.

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.