ORE RESEARCH & EXPLORATION PTY LTD
6-8 Gatwick Road, Bayswater North, Vic 3153 AUSTRALIA
Telephone: +613-9729 0333 Facsimile: +613-9729 4777

CERTIFICATE OF ANALYSIS FOR

MULTI-ELEMENT REFERENCE MATERIAL OREAS 42P

Prepared by:
Ore Research & Exploration Pty Ltd
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REPORT 42P-2

SUMMARY STATISTICS

Recommended values and 95% confidence intervals

Constituent	Recommended value	95% Confidence interval	
		Low	High
Aluminium, Al (%)	7.06	7.01	7.11
Arsenic, As (ppm)	110	104	116
Barium, Ba (ppm)	618	594	642
Calcium, Ca (%)	0.55	0.54	0.56
Copper, Cu (ppm)	389	376	402
Gold, Au (ppb)	91	88	94
Iron, Fe (%)	8.81	8.71	8.91
Lead, Pb (ppm)	150	146	154
Magnesium, Mg (%)	0.65	0.63	0.67
Manganese, Mn (ppm)	435	410	460
Molybdenum, Mo (ppm)	9.6	9.1	10.1
Nickel, Ni (ppm)	469	457	481
Phosphorous, P (ppm)	380	350	410
Potassium, K (%)	2.35	2.28	2.42
Silicon, Si (%)	29.33	29.19	29.47
Sodium, Na (%)	0.14	0.12	0.16
Sulphur, S (ppm)	200	160	240
Titanium, Ti (ppm)	3700	3620	3780
Tungsten, W (ppm)	32	29	35
Zinc, Zn (ppm)	615	597	633

INTRODUCTION

OREAS reference materials (RM) are intended to provide a low cost method of evaluating and improving the quality of analysis of geological samples. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. To the explorationist they provide an important control in analytical data sets pertaining to exploration from the grass roots level through to prospect evaluation. To the mine geologist they enable improved performance in grade control.

As a rule only source materials exhibiting an exceptional level of homogeneity of the element(s) of interest are used in the preparation of these materials. This has enabled Ore Research & Exploration to produce a range of gold RM's exhibiting homogeneity that matches or exceeds that of currently available international reference materials. In many instances RM's produced from a single source are sufficiently homogeneous to produce a relatively coarse-grained form designed to simulate drill chip samples. These have a grain size of minus 3mm and are designated with a "C" suffix to the RM identification number. These standards are packaged in 1kg units following homogenisation and are intended for submission to analytical laboratories in subsample sizes of as little as 250g. They offer the added advantages of providing a check on both sample preparation and analytical procedures while acting as a transparent standard to the assay laboratory. The more conventional pulped standards have a grain size of minus 75 microns and a higher degree of homogeneity. These standards are distinguished by a "P" suffix to the standard identification number. In line with ISO recommendations successive batch numbers are now designated by the lower case suffixes "a", "b", "c", "d", etc.

SOURCE MATERIALS

The multi-element reference material OREAS 42P is a composite standard produced from a range of oxidised materials including Blackwood greywacke (central Victoria), Bulong laterite (Yilgarn, Western Australia), Hilton North gossan and Mount Oxide ferruginous mudstone (Mount Isa region, Queensland). The dominant constituent, a gold-bearing greywacke, was obtained from the flank of a mineralised shear zone within Ordovician flysch sediments in the Blackwood area of central Victoria. The sedimentary succession hosting the shear zone consists predominantly of medium-grained greywackes together with subordinate interbedded siltstone and slate. Hydrothermal alteration in the vicinity of the mineralisation is indicated by the development of phyllite. The shear zone, in which gold grades attain a maximum, is manifested by foliated sericitic and chloritic fault gouge and goethitic quartz veins. The very homogeneous distribution of gold on a mesoscopic scale and uniform concentration gradient away from the ore zone suggests the gold is extremely fine-grained and evenly disseminated.

COMMINUTION AND HOMOGENISATION PROCEDURES

The various constituents comprising OREAS 42P were prepared in the following manner:

a) *primary crushing in a large (36 x 50cm) jaw crusher*
b) *drying in a gas-fired rotary drier*
c) *secondary crushing in a small (10 x 20cm) jaw crusher*
d) *tertiary crushing to minus 3mm in a roller crusher*

At this stage the constituent materials were sampled, reassayed and combined in proportions designed to optimise the concentration levels of the metals of principal interest (Au, Cu, Ni, Pb and Zn). The resultant mixture was then prepared in the following manner:

e) *homogenisation in a paddle blender*
f) *milling in a gamma mill*
g) *screening to minus 75 micron in an air classifier*
h) *homogenisation in a ribbon blender*
i) *bagging into 20kg sublots*

The oversize fraction from the screening stage was re-milled and screened until a negligible amount remained. Throughout the bagging stage twenty-four 1kg test units were taken at random intervals (determined using tables of random numbers), sealed in laminated plastic bags and set aside for laboratory testing.

Prior to bottling in 1kg units each 20kg sublot was further homogenised in a tumble blender to counter the possibility of unmixing during handling. The resultant material constitutes the minus 75 micron reference material OREAS 42P.

ANALYSIS OF OREAS 42P

The certification of OREAS 42P entailed two separate programs, one primarily for major elements utilising mainly university and government research laboratories, and one for trace elements in which commercial assay laboratories played the major role.

Major Elements

Eleven laboratories participated in the major element program and are listed in the section headed Participating Laboratories. Each received one 50g test portion, randomly selected from the 1kg test units described above, with instructions to carry out duplicate major element determinations via borate fusion X-ray fluorescence spectrometry. Three of eleven laboratories used a low dilution technique while the remaining eight used conventional methods. Interlaboratory agreement is good and reflected in the very narrow 95% confidence intervals for the recommended values (refer below).

Trace Elements

Seventeen commercial and nine university and research laboratories participated in the trace element program and are listed in the section headed Participating Laboratories. The latter group was instructed to determine their standard suite of trace constituents using pressed powder pellet or low dilution borate fusion XRF analysis on the same 50g test portion or glass disc used for the major element determinations.

Each commercial laboratory received three 200g subsamples with instructions to carry out duplicate fire assay/graphite furnace AAS or ICP-MS determinations for gold on each subsample. The duplicate assays were to be performed on separate test portions using 50g charges. Selected laboratories were also instructed to conduct single gold determinations on each subsample using an aqua regia digest on a 25-30g portion. The remaining trace elements were determined by a variety of acid digestion (multi-acid/HF, perchloric acid, aqua regia, aqua regia/perchloric) and alkali fusion methods in combination with AAS, ICP-OES and ICP-MS. A limited number of laboratories also provided results by XRF pressed powder pellet. Due to limitations in capabilities some laboratories were unable to undertake certain aspects of the test program.

For each laboratory the three 200g subsamples were scoop-split from separate test units taken during the bagging stage. This two-stage nested design for the interlaboratory programme was amenable to analysis of variance (ANOVA) treatment and enabled a comparative assessment of within- and between-unit homogeneity.

For the determination of a statistical tolerance interval for gold, a 30g scoop split was taken from each of the twenty-four random test units and submitted for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 1g.

COMPARISON OF ANALYTICAL METHODS

In Tables 1-9 results for As, Ba, Cu, Au, Pb, Mo, Ni, W and Zn are summarised according to analytical method.

Table 1. Arsenic (ppm).

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	113	10	13	69
XRF-PPP & XRF-LD	106	6	10	26
Perchloric - ICPOES, AAS, ICPMS	107	6	9	27
Aqua regia - ICPOES, AAS, ICPMS	100	7	9	27
Aqua regia/perchloric - ICPOES, AAS, ICPMS	104	14	11	33
Alkaline fusion - ICPMS, ICPOES	131	-	2	6
INAA	102	-	1	24
Total			55	212

ICPOES: inductively-coupled plasma optical emission spectroscopy; AAS: atomic absorption spectroscopy; ICPMS: inductively-coupled plasma mass spectrometry; XRF-PPP and XRF-LD: pressed powder pellet and low-dilution borate fusion X-ray fluorescence; INAA: instrumental neutron activation analysis.

Table 2. Barium (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	583	52	10	55
XRF-PPP & XRF-LD	643	36	15	37
Perchloric - ICPOES, AAS, ICPMS	325	-	4	12
Aqua regia - ICPOES, AAS, ICPMS	99	27	5	15
Aqua regia/perchloric - ICPOES, AAS	140	44	5	15
Alkaline fusion - AAS, ICPOES	596	-	3	9
INAA	567	-	1	24
Total			43	168

Table 3. Copper (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	392	13	15	77
XRF-PPP & XRF-LD	379	34	9	20
Perchloric - ICPOES, AAS, ICPMS	378	12	12	36
Aqua regia - ICPOES, AAS, ICPMS	381	15	10	30
Aqua regia/perchloric - ICPOES, AAS	377	8	12	36
Alkaline fusion - AAS, ICPOES	401	-	3	9
Total			61	208

Table 4. Gold (ppb); abbreviations as in Table 1, GFAAS: graphite furnace AAS.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Lead fire assay - ICPMS, GFAAS, AAS	91	3	13	77
INAA	92	-	1	24
Aqua regia - GFAAS, ICPMS	79	7	7	21
Total			21	122

Table 5. Lead (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	151	5	14	73
XRF-PPP & XRF-LD	150	7	12	28
Perchloric - ICPOES, AAS, ICPMS	150	6	10	30
Aqua regia - ICPOES, AAS, ICPMS	137	12	9	27
Aqua regia/perchloric - ICPOES, AAS, ICPMS	145	5	11	33
Alkaline fusion - AAS, ICPMS	147	-	3	9
Total			59	200

Table 6. Molybdenum (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPMS, ICPOES, AAS	10.0	0.7	9	49
XRF-PPP & XRF-LD	9.3	1.0	9	23
Perchloric - ICPMS, ICPOES, AAS	8.5	1.1	10	30
Aqua regia - ICPMS, ICPOES, AAS	9.3	1.8	7	21
Aqua regia/perchloric - ICPOES, ICPMS, AAS	9.1	2.3	7	21
Alkaline fusion - ICPMS, AAS	9.7	-	2	6
Total			44	150

Table 7. Nickel (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	460	20	14	73
XRF-PPP & XRF-LD	472	15	10	22
Perchloric - ICPOES, AAS, ICPMS	436	24	11	33
Aqua regia - ICPOES, AAS, ICPMS	401	43	9	27
Aqua regia/perchloric - ICPOES, AAS	433	15	11	33
Alkaline fusion - AAS, ICPOES	491	-	4	12
Total			59	200

Table 8. Tungsten (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPMS, ICPOES	33	4	8	51
XRF-PPP & XRF-LD	29	3	6	17
Perchloric - ICPMS	4	-	3	9
Aqua regia - ICPMS, ICPOES	23	-	3	9
Aqua regia/perchloric - ICPMS, ICPOES	13	-	4	12
Alkaline fusion - ICPMS	34	-	2	6
INAA	31	-	1	24
Total			27	128

Table 9. Zinc (ppm); abbreviations as in Table 1.

Method	Mean	95% Confidence Interval	No. of Laboratories	No. of Results
Mixed acid/HF - ICPOES, AAS, ICPMS	638	24	15	78
XRF-PPP & XRF-LD	580	25	10	22
Perchloric - ICPOES, AAS, ICPMS	611	25	11	33
Aqua regia - ICPOES, AAS, ICPMS	617	39	9	27
Aqua regia/perchloric - ICPOES, AAS, ICPMS	620	22	12	36
Alkaline fusion - AAS, ICPOES	627	-	3	9
INAA	598	-	1	24
Total			61	226

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 42P

Recommended Value and Confidence Limits

The recommended value was determined for each element from the mean of means of accepted replicate values of accepted laboratory data sets according to the formulae

$$\overline{x}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} x_{ij}$$

$$\ddot{x} = \frac{1}{p} \sum_{i=1}^{p} \overline{x}_i$$

where

x_{ij} *is the jth result reported by laboratory i;*

p *is the number of participating laboratories;*

n_i *is the number of results reported by laboratory i;*

$\bar{x}_i$ *is the mean for laboratory i;*

$\ddot{x}$ *is the mean of means.*

Methods incorporating partial acid digestion are not reliable measures of total concentration values and were not used in determining recommended values.

The confidence limits were obtained by calculation of the variance of the consensus value (mean of means) and reference to Student's-*t* distribution with degrees of freedom (*p*-1)

$$\hat{V}(\ddot{x}) = \frac{1}{p(p-1)} \sum_{i=1}^{p} \left(\bar{x}_i - \ddot{x}\right)^2$$

$$\text{Confidence limits } = \ddot{x} \pm t_{1-x/2}(p-1)\left(\hat{V}(\ddot{x})\right)^{1/2}$$

where $t_{1-x/2}(p-1)$ *is the 1-x/2 fractile of the t-distribution with (p-1) degrees of freedom.*

The distribution of the values are assumed to be symmetrical about the mean in the calculation of the confidence limits.

The test for rejection of individual outliers was based on the test criterion, T, and reference to tables of critical values of T at the 1% level of significance (ASTM E 178-94) as follows:

$$T_{ij} = \left|\left(x_{ij} - \bar{x}_i\right)\right| / s_i$$

where

T_{ij} *is the test criterion for the jth result of laboratory i;*

s_i *is the standard deviation of laboratory i.*

The same principles were applied in testing for outlying laboratory means. Recommended major oxide, major element and trace element concentrations are given in Tables 10-12.

Statement of Homogeneity

The variability of replicate assays from each laboratory is a result of both measurement and subsampling errors. In the determination of a statistical tolerance interval it is therefore necessary to eliminate, or at least substantially minimise, those errors attributable to measurement. One way of achieving this is by reducing the analytical subsample weight to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible. Due to limitations imposed by the analytical procedures employed, this approach was impractical for elements other than gold. Homogeneity of this metal was accordingly determined by INAA from twenty-four 1g subsamples obtained in the manner described previously and using tables of factors for two-sided tolerance limits for normal distributions (ISO Guide 3207) in which

$$\text{Lower limit is } \ddot{x} - k_2'(n, p, 1-\alpha)s$$
$$\text{Upper limit is } \ddot{x} + k_2'(n, p, 1-\alpha)s$$

Table 10. Major oxide borate fusion XRF data *(weight percent).*

Constituent	Recommended Value	95% Confidence Interval	No. of Laboratories	No. of Results
SiO_2	62.74	0.29	11	24
TiO_2	0.617	0.013	11	24
Al_2O_3	13.34	0.10	10	22
Fe_2O_3	12.60	0.15	11	24
MnO	0.056	0.003	11	24
MgO	1.07	0.03	10	23
CaO	0.77	0.01	10	21
Na_2O	0.19	0.03	10	22
K_2O	2.83	0.08	11	24
P_2O_5	0.087	0.007	11	24
BaO	0.069	0.003	8	18
SO_3	0.050	0.009	9	19
LOI	5.00	0.20	9	17

Table 11. Major element borate fusion XRF data *(expressed in elemental form; integers as ppm, rest in weight percent).*

Constituent	Recommended Value	95% Confidence Interval	No. of Laboratories	No. of Results
Silicon, Si	29.33	0.14	11	24
Titanium, Ti	3700	80	11	24
Aluminium, Al	7.06	0.05	10	22
Iron, Fe	8.81	0.10	11	24
Manganese, Mn	435	25	11	24
Magnesium, Mg	0.65	0.02	10	23
Calcium, Ca	0.55	0.01	10	21
Sodium, Na	0.14	0.02	10	22
Potassium, K	2.35	0.07	11	24
Phosphorous, P	380	30	11	24
Barium, Ba	620	30	8	18
Sulphur, S	200	40	9	19

where

n is the number of results reported by laboratory Q;

$1-\alpha$ is the confidence level;

p is the proportion of results expected within the tolerance limits;

k_2' is the factor for two-sided tolerance limits (m, σ unknown);

Table 12. Trace element recommended values and 95% confidence intervals (ppm).

Constituent	Recommended Value	95% Confidence Interval
Arsenic, As	110	6
Barium, Ba	618	24
Copper, Cu	389	13
Gold, Au	0.091	0.003
Lead, Pb	150	4
Manganese, Mn	435	25
Molybdenum, Mo	9.6	0.5
Nickel, Ni	469	12
Sulphur, S	200	40
Titanium, Ti	3700	80
Tungsten, W	32	3
Zinc, Zn	615	18

and *s* is computed according to the formula

$$s = \left[\frac{\sum_{j=1}^{n} \left(x_j - \bar{x} \right)^2}{n-1} \right]^{1/2}$$

No individual outliers were removed from the results prior to the calculation of tolerance intervals.

From the INAA data set an estimated tolerance interval of ±4ppb at an analytical subsample weight of 50g was obtained (using the sampling constant relationship of Ingamells and Switzer, 1973) and is considered to reflect the actual inhomogeneity of the material under test. The meaning of this tolerance interval may be illustrated for gold (refer Table 13) where 99% of the time at least 95% of 50g-sized subsamples will have concentrations lying between 87 and 95ppb. 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 two-stage nested design adopted for the interlaboratory programme entailed each laboratory completing two replicate fire assay determinations on each of the three units received. This enabled gold homogeneity to be independently evaluated using an Analysis

Table 13. Recommended value and tolerance interval for gold

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99$, $\rho=0.95$	
		Low	**High**
Gold, Au (ppb)	91	87	95

Table 14. ANOVA table.

Source	Sum of squares	Degrees of freedom	Mean square
Between units	295	22	13.4 (MS1)
Within units	360	33	10.9 (MS2)

of Variance (ANOVA) approach. The results of this treatment, modified for unbalanced data, are summarised in Table 14. The between-unit mean square is of similar magnitude to the within-unit mean square for which:

$$\text{Test statistic} = MS1/MS2 = 1.23$$

and the critical values for the F-test (one-sided) are:

1.89 at the 5% significance level
2.47 at the 1% significance level.

We may conclude, therefore, that there is no evidence to indicate that the between-units variance for gold is greater than that within units and that the homogeneity of the entire batch of OREAS 42P is of an acceptable level.

PARTICIPATING LABORATORIES

Amdel Laboratories Ltd, Thebarton, SA, Australia
Analabs Pty Ltd, Cooee, TAS, Australia
Analabs Pty Ltd, East Brisbane, QLD, Australia
Analabs Pty Ltd, Townsville, QLD, Australia
Analabs Pty Ltd, Welshpool, WA, Australia
Anglo American Research Laboratories Pty Ltd, Johannesburg, South Africa
Assaycorp Pty Ltd, Pine Creek, NT, Australia
Australian Assay Laboratories Pty Ltd, Balcatta, WA, Australia
Australian Laboratory Services Pty Ltd, Bendigo, VIC, Australia
Australian Laboratory Services Pty Ltd, Stafford, QLD, Australia

Australian Laboratory Services Pty Ltd, Malaga, WA, Australia
Australian National University, Canberra, ACT, Australia
Becquerel Laboratories, Lucas Heights, NSW, Australia
CSIRO Division of Exploration and Mining, Floreat Park, WA, Australia
Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia
La Trobe University, Bundoora, VIC, Australia
Minlab, Malaga, WA, Australia
SGS Australia Pty Ltd, Queens Park, WA, Australia
Ultra Trace, Bentley, WA, Australia
University of Adelaide,SA, Australia
University of Melbourne, Parkville, VIC, Australia
University of New South Wales, NSW, Australia
University of Newcastle, Callaghan, NSW, Australia
University of Queensland, Brisbane QLD, Australia
University of Tasmania, Hobart, TAS, Australia
Western Mining Corporation Ltd, Kalgoorlie, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The multi-element geochem reference material, OREAS 42P has been prepared and certified and is supplied by:

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

Telephone	(03) 9729 0333	International	+613-9729 0333
Facsimile	(03) 9729 4777	International	+613-9729 4777

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

INTENDED USE

OREAS 42P is a reference material intended for the following:

i) for the calibration of instruments used in the determination of the concentration of major and trace elements;
ii) for the verification of analytical methods;
iii) for the preparation of secondary reference materials of similar composition;
iv) as an arbitration sample for commercial transactions.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 42P has been prepared from rock samples obtained within the oxidised zone at various mineralised localities. It is therefore considered to have long-term stability under normal storage conditions.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The recommended values for OREAS 42P refer to the concentration levels after removal of hygroscopic moisture by drying in air to constant mass at 105^0 C. In its undried state a hygroscopic moisture content of approximately 1.13% has been established. If the reference material is not dried by the user prior to analysis, the recommended values should be corrected to the moisture-bearing basis.

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: Dr Paul Hamlyn

ACKNOWLEDGMENTS

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