

CERTIFICATE OF ANALYSIS FOR

Copper Gold Reference Material

OREAS 59d

Prepared by:
Ore Research & Exploration Pty Ltd
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REPORT 02/446-59d

SOURCE MATERIAL

OREAS 59d is one of four Cu-Au-As-Co-Fe-Mo-Ni-S certified reference materials (CRM's) prepared by Ore Research & Exploration Pty Ltd from copper-gold ore sourced from Cloncurry, Qld, Australia. The iron oxide copper gold (IOCG) deposit is hosted in Proterozoic rocks of the Mt Isa Inlier and primary mineralisation is intimately associated with felsic to intermediate volcanic breccias. The breccias are rich in magnetite and disseminated sulphide mineralization.

COMMUNITION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- a) *drying for 24 hours at 105⁰ C;*
- b) *crushing and screening;*
- b) *preliminary homogenisation;*
- c) *milling to minus 20 microns;*
- d) *final homogenisation;*
- e) *packaging into 50g lots sealed in laminated foil pouches.*

ANALYSIS OF OREAS 59d

Ten commercial laboratories participated in the analytical program to characterise Cu-Au-As-Co-Fe-Mo-Ni-S in OREAS 59d. The analytical methods employed by each laboratory are given in Table 1. Their results together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM³) are presented in Tables 2 to 9. The parameter PDM³ is a measure of laboratory accuracy while the relative standard deviation is an effective measure of analytical precision where homogeneity of the test material has been confirmed. With the exception of Lab A, five 100g samples were submitted to each laboratory for analysis.

Gold (Table 5) was determined in five replicate assays using lead fire assay (40-50g charge with new pots) with flame AAS or ICPOES finish at nine laboratories, while Lab A determined gold (plus As, Co, Fe and Mo) in fifteen replicates via instrumental neutron activation analysis (INAA) using 0.5g analytical subsample weights. Each five samples submitted to each laboratory were taken at regular intervals during packaging of the standard in order to maximise their representation. The fifteen INAA subsamples, on which much of the homogeneity evaluation is based, were also taken at regular intervals during packaging and are considered representative of the entire batch.

Arsenic, cobalt, copper, iron, molybdenum, nickel and sulphur (Tables 2 to 4 and 6 to 9) were determined by aqua regia digest with ICPOES finish at nine laboratories and arsenic, cobalt, iron and molybdenum by INAA at one laboratory.

Table 1. Explanation of analytical methods

Code	Method
INAA	Instrumental Neutron Activation Analysis
AR*OES	Aqua Regia Digest / ICP Optical Emission Spectrometry
AR*AAS	Aqua Regia Digest / Atomic Absorption Spectrometry
FA*AAS	Fire Assay / Atomic Absorption Spectrometry
FA*OES	Fire Assay / ICP Optical Emission Spectrometry

Table 2. Analytical results for arsenic in OREAS 59d (Std.Dev. and Rel.Std.Dev. are one sigma values; PDM³ - percent deviation of lab mean from corrected mean of means; abbreviations as in Table 1; outliers in bold; values in ppm).

Replicate No.	Lab A INAA	Lab B AR*OES	Lab C AR*OES	Lab D AR*OES	Lab E AR*OES	Lab F AR*OES	Lab G AR*OES	Lab H AR*OES	Lab I AR*OES	Lab J AR*OES
1	823	722	870	775	850	830	840	715	834	780
2	822	726	860	770	860	835	840	705	837	760
3	830	765	865	765	820	824	850	725	829	751
4	829	746	860	764	820	820	850	725	847	754
5	825	697	865	763	-	816	830	715	844	780
6	824									
7	829									
8	823									
9	823									
10	820									
11	825									
12	822									
13	821									
14	829									
15	826									
Mean	825	731	864	767	838	825	842	717	838	765
Median	824	726	865	765	835	824	840	715	837	760
Std.Dev.	3	26	4	5	21	8	8	8	7	14
Rel.Std.Dev.	0.39%	3.52%	0.48%	0.66%	2.46%	0.92%	0.99%	1.17%	0.88%	1.84%
PDM ³	0.51%	-10.88%	5.3%	-6.47%	2.08%	0.55%	2.62%	-12.61%	2.16%	-6.76%

Table 3. Analytical results for cobalt in OREAS 59d (abbreviations as in Tables 1 and 2; values in ppm).

Replicate No.	Lab A INAA	Lab B AR*OES	Lab C AR*OES	Lab D AR*OES	Lab E AR*OES	Lab F AR*OES	Lab G AR*OES	Lab H AR*OES	Lab I AR*OES	Lab J AR*OES
1	1005	902	971	842	860	913	1040	830	933	868
2	998	915	966	838	860	910	1020	830	933	848
3	992	927	957	835	840	897	1060	850	927	839
4	994	915	960	834	840	900	1060	850	935	843
5	999	858	969	843	-	867	1040	840	927	858
6	990									
7	997									
8	999									
9	1000									
10	991									
11	999									
12	993									
13	994									
14	994									
15	998									
Mean	996	903	965	838	850	897	1044	840	931	851
Median	997	915	966	838	850	900	1040	840	933	848
Std.Dev.	4	27	6	4	12	18	17	10	4	12
Rel.Std.Dev.	0.42%	2.97%	0.62%	0.48%	1.36%	2.03%	1.60%	1.19%	0.41%	1.38%
PDM ³	10.8%	0.49%	7.29%	-6.74%	-5.45%	-0.18%	16.1%	-6.57%	3.57%	-5.32%

Analytical results for copper in OREAS 59d (abbreviations as in Tables 1 and 2; values in wt.%).

Table 4.

Replicate No.	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES
1	1.399	1.58	1.49	1.435	1.45	1.617	1.47	1.479	1.49
2	1.419	1.56	1.48	1.465	1.44	1.609	1.45	1.486	1.47
3	1.420	1.60	1.49	1.440	1.44	1.624	1.48	1.480	1.47
4	1.418	1.58	1.50	1.360	1.42	1.617	1.46	1.476	1.49
5	1.314	1.59	1.48	-	1.44	1.628	1.49	1.480	1.45
Mean	1.394	1.582	1.488	1.425	1.438	1.619	1.470	1.480	1.474
Median	1.418	1.580	1.490	1.438	1.440	1.617	1.470	1.480	1.470
Std.Dev.	0.046	0.015	0.008	0.045	0.011	0.007	0.016	0.003	0.017
Rel.Std.Dev.	3.27%	0.94%	0.56%	3.18%	0.76%	0.45%	1.08%	0.23%	1.14%
PDM ³	-5.11%	7.70%	1.30%	-2.99%	-2.10%	10.22%	0.08%	0.77%	0.35%

Table 5. Analytical results for gold in OREAS 59d (abbreviations as in Table 1 and 2; values in ppm).

Replicate No.	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
	INAA (0.5g)	FA*AAS (50g)	FA*AAS (50g)	FA*AAS (50g)	FA*AAS (2x20g)	FA*OES (40g)	FA*AAS (50g)	FA*OES (50g)	FA*AAS (50g)	FA*AAS (50g)
1	0.749	0.79	0.810	0.751	0.85	0.836	0.81	0.812	0.81	0.85
2	0.750	0.79	0.794	0.720	0.84	0.818	0.80	0.806	0.78	0.84
3	0.764	0.78	0.840	0.726	0.83	0.769	0.79	0.835	0.77	0.85
4	0.763	0.79	0.825	0.736	0.82	0.815	0.79	0.785	0.77	0.88
5	0.795	0.79	0.837	0.749	-	0.815	0.80	0.791	0.78	0.88
6	0.792									
7	0.753									
8	0.755									
9	0.766									
10	0.778									
11	0.770									
12	0.783									
13	0.777									
14	0.769									
15	0.781									
Mean	0.770	0.788	0.821	0.736	0.830	0.811	0.798	0.806	0.782	0.860
Median	0.769	0.790	0.825	0.736	0.830	0.815	0.800	0.806	0.780	0.850
Std.Dev.	0.015	0.004	0.019	0.014	0.013	0.025	0.008	0.020	0.016	0.019
Rel.Std.Dev.	1.89%	0.57%	2.34%	1.86%	1.56%	3.07%	1.05%	2.44%	2.10%	2.18%
PDM ³	-3.96%	-1.66%	2.48%	-8.10%	3.58%	1.16%	-0.41%	0.56%	-2.41%	7.32%

Table 6. Analytical results for iron in OREAS 59d (abbreviations as in Tables 1 and 2; values in wt.%).

Replicate No.	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
	INAA	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES
1	28.89	25.96	27.1	>15.0	28.7	27.62	30.672	25.5	25.83	25.29
2	28.75	26.05	26.8	>15.0	28.7	27.74	30.376	25.7	25.66	24.62
3	29.43	26.95	27.5	>15.0	28.1	28.11	30.937	26.3	25.64	24.43
4	29.11	26.72	26.9	>15.0	28.0	27.31	30.673	26.6	25.68	24.50
5	28.94	25.94	27.1	>15.0	-	27.54	30.568	26.0	25.65	24.96
6	28.85									
7	28.99									
8	28.91									
9	28.71									
10	28.94									
11	28.98									
12	28.87									
13	28.71									
14	28.69									
15	29.13									
Mean	28.93	26.32	27.08	-	28.38	27.66	30.65	26.02	25.69	24.76
Median	28.91	26.05	27.10	-	28.40	27.62	30.67	26.00	25.66	24.62
Std.Dev.	0.19	0.48	0.27	-	0.38	0.29	0.20	0.44	0.08	0.36
Rel.Std.Dev.	0.67%	1.81%	0.99%	-	1.33%	1.07%	0.66%	1.71%	0.30%	1.45%
PDM ³	7.81%	-1.89%	0.92%	-	5.75%	3.10%	14.2%	-3.03%	-4.26%	-7.72%

Table 7. Analytical results for molybdenum in OREAS 59d (abbreviations as in Tables 1 and 2; values in ppm).

Replicate No.	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
	INAA	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES	AR*OES
1	297	283	320	223	365	304	340	250	344	214
2	288	290	318	219	355	305	330	250	339	190
3	294	293	320	219	345	304	340	265	337	189
4	291	287	324	222	315	301	340	250	341	181
5	304	274	322	219	-	290	340	250	337	204
6	286									
7	284									
8	305									
9	310									
10	295									
11	301									
12	301									
13	309									
14	295									
15	290									
Mean	297	285	321	220	345	301	338	253	339	196
Median	295	287	320	219	350	304	340	250	339	190
Std.Dev.	8	7	2	2	22	6	4	7	3	13
Rel.Std.Dev.	2.73%	2.58%	0.71%	0.88%	6.26%	2.07%	1.32%	2.65%	0.86%	6.75%
PDM ³	-4.39%	-8.00%	3.4%	-29.0%	11.2%	-3.04%	9.0%	-18.4%	9.42%	-36.9%

Table 8. Analytical results for nickel in OREAS 59d (abbreviations as in Tables 1 and 2; values in ppm).

Replicate No.	Lab B AR*OES	Lab C AR*OES	Lab D AR*OES	Lab E AR*OES	Lab F AR*OES	Lab G AR*OES	Lab H AR*OES	Lab I AR*OES	Lab J AR*OES
1	61	72	65	50	72	80	64	65	49
2	62	72	65	100	71	70	62	65	47
3	63	72	64	50	69	80	66	65	47
4	61		64	50	71	70	64	65	46
5	66	72	64	-	68	80		65	48
Mean	63	72	64	63	70	76	64	65	47
Median	62	72	64	50	71	80	64	65	47
Std.Dev.	2	0	1	25	2	5	2	0	1
Rel.Std.Dev.	3.31%	0.00%	0.85%	40.0%	2.34%	7.21%	2.55%	0.56%	2.41%
PDM ³	-7.59%	6.3%	-4.93%	-7.73%	3.63%	12.2%	-5.52%	-4.08%	-30.0%

Table 9. Analytical results for sulphur in OREAS 59d (abbreviations as in Tables 1 and 2; values in wt.%).

Replicate No.	Lab B AR*OES	Lab C AR*OES	Lab D AR*OES	Lab E AR*OES	Lab F AR*OES	Lab G AR*OES	Lab H AR*OES	Lab I AR*OES	Lab J AR*OES
1	2.28	4.30	3.60	4.67	4.11	4.08	3.65	3.35	3.61
2	2.21	4.29	3.52	4.70	4.11	3.43	3.65	3.34	3.51
3	2.44	4.35	3.53	4.33	4.03	4.08	3.75	3.30	3.50
4	2.95	4.35	3.55	4.54	4.05	3.55	3.65	3.37	3.48
5	2.38	4.33	3.51	-	3.93	3.97	3.65	3.37	3.52
Mean	2.45	4.32	3.54	4.56	4.05	3.82	3.67	3.34	3.52
Median	2.38	4.33	3.53	4.61	4.05	3.97	3.65	3.35	3.51
Std.Dev.	0.29	0.03	0.04	0.17	0.07	0.31	0.04	0.03	0.05
Rel.Std.Dev.	12.0%	0.65%	1.01%	3.69%	1.83%	8.09%	1.22%	0.85%	1.43%
PDM ³	-33.7%	16.9%	-4.23%	23.3%	9.4%	3.34%	-0.77%	-9.56%	-4.71%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 59d

Certified Value and Confidence Limits

The certified value is the mean of means of accepted replicate values of accepted participating laboratories computed according to the formulae

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

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

where

x_{ij} is the j th 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 ;

$\bar{\bar{x}}$ is the mean of means.

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).

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

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

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 from each laboratory data set was based on *z* scores (rejected if $|z_i| > 2.5$) computed from the robust estimators of location and scale, *T* and *S*, respectively, according to the formulae

$$S = 1.483 \frac{\text{median} / x_j - \text{median} (x_i)}{j=1 \dots n \quad i=1 \dots n}$$

$$z_i = \frac{x_i - T}{S}$$

where

T is the median value in a data set;

S is the median of all absolute deviations from the sample median multiplied by 1.483, a correction factor to make the estimator consistent with the usual parameter of a normal distribution.

In certain instances statistician's prerogative has been employed in discriminating outliers. Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in bold italics (red in bar charts) and have been omitted in the determination of certified values. The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It 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.

Table 10. Certified values and 95% confidence intervals for OREAS 59d.

Constituent	Certified value	95% Confidence interval	
		Low	High
Arsenic, As (ppm)	820	791	850
Cobalt, Co (ppm)	899	854	944
Copper, Cu (wt.%)	1.47	1.42	1.52
Gold, Au (ppm)	0.801	0.784	0.819
Iron, Fe (wt.%)	26.8	25.7	28.0
Molybdenum, Mo (ppm)	310	284	337
Nickel, Ni (ppm)	68	63	72
Sulphur, S (wt.%)	3.70	3.23	4.17

Note: Intervals may be asymmetric due to rounding

Statement of Homogeneity

The standard deviation of each laboratory data set includes error due to both the imprecision of the analytical method employed and to possible inhomogeneity of the material analysed. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material analysed and, in particular, to deficiencies in accuracy of each analytical method. In determining tolerance intervals for elements other than gold that component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \bar{x}_i + \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^p n_i}$$

where

x_{ij} is the j th raw result reported by laboratory i ;

x'_{ij} is the j th transformed result reported by laboratory i ;

n_i is the number of results reported by laboratory i ;

p is the number of participating laboratories;

\bar{x}_i is the raw mean for laboratory i .

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$\text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha) s_g''$$

$$\text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha) s_g''$$

where

n is the number of results;

$1 - \alpha$ 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, α unknown);

s_g'' is the corrected grand standard deviation

The meaning of these tolerance limits may be illustrated for copper, where 99% of the time at least 95% of subsamples will have concentrations lying between 1.45 and 1.49 wt.%. 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 corrected grand standard deviation, s_g'' , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula

$$s_g'' = \frac{\sum_{i=1}^p (s_i (1 - \frac{s_i}{s_g'}))}{\sum_{i=1}^p (1 - \frac{s_i}{s_g'})}$$

where

$1 - (\frac{s_i}{2s_g'})$ is the weighting factor for laboratory i ;

s_g' is the grand standard deviation computed from the transformed (i.e. means-adjusted) results

according to the formula

$$s_g' = \left[\frac{\sum_{i=1}^p \sum_{j=i}^{n_i} (x'_{ij} - \bar{x}'_i)^2}{\sum_{i=1}^p n_i - 1} \right]^{1/2}$$

where \bar{x}'_i is the transformed mean for laboratory i

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error. For gold a more simplified procedure was used in the determination of homogeneity. This entailed using the high precision INAA data alone, obtained on an analytical subsample weight of 0.5g (compared to 40-50g for the fire assay method). By employing a sufficiently reduced subsample weight in a series of determinations by the same method, analytical error becomes negligible in comparison to subsampling error. The corresponding standard deviation at a 50g subsample weight can then be determined from the observed standard deviation of the 0.5g data using the known relationship between the two parameters (Kleeman, 1967). The homogeneity of gold was then determined from tables of factors for two-sided tolerance limits for normal distributions. The high level of repeatability indicated by the low coefficients of variation in Table 1 (particularly the 0.5 g Becquerel data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 59d.

No outliers were removed from the INAA results prior to the calculation of tolerance intervals for gold, however for the other elements outliers were removed prior to the calculation of s_g' and a weighting factor of zero was applied to those data sets where $s_i / 2s_g' > 1$ (i.e. where the weighting factor $1 - s_i / 2s_g' < 0$).

Table 11. Certified values and tolerance limits for OREAS 59d.

Constituent	Certified value	Tolerance limits 1- α =0.99, ρ =0.95	
		Low	High
Arsenic, As (ppm)	820	807	834
Cobalt, Co (ppm)	899	889	910
Copper, Cu (wt.%)	1.47	1.45	1.49
Gold, Au (ppm)	0.801	0.796	0.807
Iron, Fe (wt.%)	26.8	26.6	27.1
Molybdenum, Mo (ppm)	310	303	317
Nickel, Ni (ppm)	68	66	69
Sulphur, S (wt.%)	3.70	3.59	3.81

Note: Intervals may be asymmetric due to rounding

Performance Gates

Performance gates 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 and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. Sources of measurement error include inter-lab bias, analytical precision (repeatability) and inter-batch bias (reproducibility).

Two methods have been employed to calculate performance gates. The first method uses the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers. 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 individual analyses (excluding the INAA data for gold) generated from the certification program.

Table 12 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 12. Performance Gates for OREAS 59d

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
As (ppm)	820	20	780	861	759	882	2.49%	4.97%	7.46%	779	861
Co (ppm)	899	16	868	931	852	946	1.75%	3.50%	5.25%	854	944
Cu (wt.%)	1.47	0.03	1.41	1.53	1.38	1.57	2.15%	4.29%	6.44%	1.40	1.54
Au (ppm)	0.801	0.027	0.748	0.855	0.721	0.882	3.35%	6.70%	10.1%	0.761	0.841
Fe (wt.%)	26.8	0.5	25.8	27.9	25.3	28.4	1.96%	3.91%	5.87%	25.5	28.2
Mo (ppm)	310	16	279	342	263	358	5.10%	10.2%	15.3%	295	326
Ni (ppm)	68	4	59	76	55	80	6.01%	12.0%	18.0%	64	71
S (wt.%)	3.85	0.23	3.39	4.31	3.16	4.54	5.97%	11.9%	17.9%	3.66	4.04

Note - intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

Acme Analytical Laboratories, Vancouver, BC, Canada
Amdel Laboratories, Wangara, WA, Australia
Analabs, Townsville, QLD, Australia
ALS Chemex, North Vancouver, Ontario, Canada
ALS Chemex, Orange, NSW, Australia
ALS Chemex, Townsville, QLD, Australia
Becquerel Laboratories, Lucas Heights, NSW, Australia
Genalysis Laboratory Services, Maddington, WA, Australia
OMAC Laboratories, Loughrea. Co. Galway, Ireland
Ultra Trace, Canning Vale, WA, Australia

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