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**CERTIFICATE OF ANALYSIS FOR**

**Copper Gold Reference Material**

**OREAS 59a**

Prepared by:

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REPORT 02/446-59a

## SOURCE MATERIAL

OREAS 59a 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 105<sup>0</sup> 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 59a

Ten commercial laboratories participated in the analytical program to characterise Cu-Au-As-Co-Fe-Mo-Ni-S in OREAS 59a. 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 (PDM<sup>3</sup>) are presented in Tables 2 to 9. The parameter PDM<sup>3</sup> 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 59a (Std.Dev. and Rel.Std.Dev. are one sigma values; PDM<sup>3</sup> - 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	678	669	735	641	670	648	650	575	701	655
2	686	657	740	<b>627</b>	680	635	630	570	687	666
3	684	675	755	643	690	666	650	580	697	663
4	685	667	745	644	710	666	650	585	696	667
5	675	<b>633</b>	<b>710</b>	635	<b>640</b>	675	630	595	688	651
6	682									
7	693									
8	682									
9	<b>662</b>									
10	674									
11	687									
12	675									
13	676									
14	686									
15	682									
Mean	680	660	737	638	678	658	642	581	694	660
Median	682	667	740	641	680	666	650	580	696	663
Std.Dev.	7	17	17	7	26	16	11	10	6	7
Rel.Std.Dev.	1.09%	2.50%	2.28%	1.11%	3.82%	2.46%	1.71%	1.66%	0.88%	1.07%
PDM <sup>3</sup>	2.23%	-0.81%	10.7%	-4.15%	1.86%	-1.14%	-3.54%	-12.7%	4.23%	-0.78%

Table 3. Analytical results for cobalt in OREAS 59a (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	781	785	792	717	700	697	770	685	775	701
2	791	765	795	<b>692</b>	700	<b>679</b>	770	680	786	718
3	784	783	791	717	720	716	760	680	776	710
4	787	770	787	711	740	714	780	690	783	720
5	787	740	798	714	700	723	780	695	778	701
6	777									
7	794									
8	786									
9	772									
10	779									
11	787									
12	783									
13	781									
14	791									
15	796									
Mean	785	769	793	710	712	706	772	686	780	710
Median	786	770	792	714	700	714	770	685	778	710
Std.Dev.	6	18	4	10	18	18	8	7	5	9
Rel.Std.Dev.	0.83%	2.35%	0.52%	1.47%	2.51%	2.52%	1.08%	0.95%	0.59%	1.27%
PDM <sup>3</sup>	5.60%	3.40%	6.63%	-4.46%	-4.21%	-5.05%	3.86%	-7.71%	4.90%	-4.48%

Table 4. Analytical results for copper in OREAS 59a (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	3531	3700	3520	<b>3300</b>	3370	3470	3230	3456	3370
2	3401	3790	3410	3400	3320	3470	3210	3514	3440
3	3506	3740	3530	3450	3380	3480	3230	3458	3410
4	3428	3700	3520	3400	3340	3530	3230	3519	3440
5	3322	3750	3470	3400	3340	3470	<b>3300</b>	3527	3370
Mean	3438	<b>3736</b>	3490	3390	3350	3484	<b>3240</b>	3495	3406
Median	3428	3740	3520	3400	3340	3470	3230	3514	3410
Std.Dev.	84	38	50	55	24	26	35	35	35
Rel.Std.Dev.	2.44%	1.01%	1.45%	1.62%	0.73%	0.75%	1.07%	1.00%	1.03%
PDM <sup>3</sup>	-0.05%	8.63%	1.48%	-1.43%	-2.59%	1.30%	-5.79%	1.61%	-0.97%

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

Replicate No.	Lab A INAA (0.5g)	Lab B FA*AAS (50g)	Lab C FA*AAS (50g)	Lab D FA*AAS (50g)	Lab E FA*AAS (2x20g)	Lab F FA*OES (40g)	Lab G FA*AAS (50g)	Lab H FA*OES (50g)	Lab I FA*AAS (50g)	Lab J FA*AAS (50g)
1	175.1	210	201.8	174	210	188	180	184	170	210
2	195.0	190	200.7	164	205	188	200	178	180	200
3	211.6	210	199.7	181	200	185	200	<b>173</b>	170	200
4	187.7	200	<b>192.4</b>	168	210	185	190	183	170	200
5	192.1	210	198.6	172	210	191	190	182	200	200
6	203.2									
7	183.3									
8	172.5									
9	185.7									
10	188.5									
11	187.3									
12	173.4									
13	177.5									
14	187.6									
15	174.4									
Mean	186.3	204	199	172	207	187	192	180	178	202
Median	187.3	210	200	172	210	188	190	182	170	200
Std.Dev.	11.2	9	4	6	4	3	8	5	13	4
Rel.Std.Dev.	6.01%	4.38%	1.85%	3.74%	2.16%	1.41%	4.36%	2.52%	7.32%	2.21%
PDM <sup>3</sup>	-2.47%	6.79%	3.98%	-10.1%	8.36%	-1.96%	0.50%	-5.78%	-6.82%	5.74%

Table 6. Analytical results for iron in OREAS 59a (abbreviations as in Tables 1 and 2; values in weight percent).

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	18.73	18.37	17.9	>15.0	18.5	17.29	17.98	16.0	16.98	16.23
2	18.88	18.02	18.4	>15.0	18.0	17.36	17.73	16.0	17.09	16.50
3	18.64	18.46	17.9	>15.0	18.2	17.54	17.78	16.1	16.94	16.37
4	18.69	18.38	18.0	>15.0	18.5	17.44	18.05	16.1	17.20	16.49
5	18.60	17.32	17.6	>15.0	18.5	17.32	17.86	16.5	17.10	16.12
6	18.76									
7	18.93									
8	18.62									
9	18.09									
10	18.65									
11	18.99									
12	18.54									
13	18.61									
14	18.63									
15	18.92									
Mean	18.69	18.11	17.96	-	18.3	17.39	17.88	16.1	17.06	16.34
Median	18.65	18.37	17.90	-	18.5	17.36	17.86	16.1	17.09	16.37
Std.Dev.	0.22	0.47	0.29	-	0.2	0.10	0.13	0.2	0.11	0.17
Rel.Std.Dev.	1.16%	2.61%	1.60%	-	1.26%	0.58%	0.75%	1.28%	0.62%	1.01%
PDM <sup>3</sup>	4.11%	0.91%	0.07%	-	2.2%	-3.1%	-0.4%	-10.07%	-4.93%	-8.95%

Table 7. Analytical results for molybdenum in OREAS 59a (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	87	87	94	73	125	81	70	70	97	67
2	104	84	94	69	125	79	60	65	100	69
3	83	86	92	70	110	83	70	70	98	69
4	101	85	92	70	105	84	70	70	98	70
5	68	81	94	71	120	84	70	70	99	69
6	105									
7	86									
8	92									
9	96									
10	88									
11	83									
12	64									
13	82									
14	72									
15	97									
Mean	87	85	93	71	117	82	68	69	98	69
Median	87	85	94	70	120	83	70	70	98	69
Std.Dev.	12	2	1	2	9	2	4	2	1	1
Rel.Std.Dev.	14.3%	2.72%	1.18%	2.15%	7.76%	2.64%	6.58%	3.24%	1.04%	1.59%
PDM <sup>3</sup>	8.35%	5.24%	15.9%	-12.2%	45.5%	2.26%	-15.4%	-14.2%	22.2%	-14.4%

Table 8. Analytical results for nickel in OREAS 59a (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	46	48	46	<50	47	50	44	47	31
2	45	49	44	<50	<b>44</b>	50	44	47	32
3	46	47	46	<50	48	50	44	46	31
4	45	48	45	<50	48	50	44	46	32
5	43	47	45	<50	49	50	44	46	31
Mean	45	48	45	-	47	<b>50</b>	44	46	<b>31</b>
Median	45	48	45	-	48	50	44	46	31
Std.Dev.	1	1	1	-	2	0	0	0	1
Rel.Std.Dev.	2.72%	1.75%	1.85%	-	4.08%	0.00%	0.00%	0.87%	1.74%
PDM <sup>3</sup>	-2.28%	3.80%	-1.85%	-	2.50%	8.58%	-4.45%	0.55%	-31.8%

Table 9. Analytical results for sulphur in OREAS 59a (abbreviations as in Tables 1 and 2; values in weight percent).

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	3.11	3.43	3.09	3.52	3.12	3.06	2.95	2.73	2.86
2	2.98	3.52	2.94	3.67	3.03	3.05	2.95	2.73	2.94
3	3.05	3.40	3.06	3.58	3.19	3.11	2.95	2.74	2.89
4	3.04	3.53	3.04	3.74	3.19	3.16	3.00	2.75	2.94
5	2.82	3.39	3.03	3.44	3.24	2.96	3.00	2.70	2.85
Mean	3.00	3.45	3.03	<b>3.59</b>	3.15	3.07	2.97	2.73	2.90
Median	3.04	3.43	3.04	3.58	3.19	3.06	2.95	2.73	2.89
Std.Dev.	0.11	0.07	0.06	0.12	0.08	0.07	0.03	0.02	0.04
Rel.Std.Dev.	3.76%	1.93%	1.86%	3.31%	2.58%	2.43%	0.92%	0.66%	1.48%
PDM <sup>3</sup>	-1.25%	13.7%	-0.20%	18.2%	3.81%	0.98%	-2.25%	-10.1%	-4.68%

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 59a

### 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

$$S = 1.483 \text{ median } |x_j - \text{median}(x_i)|$$

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

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 59a.

Constituent	Certified value	95% Confidence interval	
		Low	High
Arsenic, As (ppm)	666	635	696
Cobalt, Co (ppm)	743	715	772
Copper, Cu (ppm)	3439	3389	3489
Gold, Au (ppb)	191	182	200
Iron, Fe (wt.%)	17.9	17.4	18.5
Molybdenum, Mo (ppm)	80	71	89
Nickel, Ni (ppm)	46	44	48
Sulphur, S (wt.%)	3.04	2.86	3.21

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 ( $m, \alpha$  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 3383 and 3495 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

The 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

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

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 59a.

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 59a.

Constituent	Certified value	Tolerance limits 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Arsenic, As (ppm)	666	651	680
Cobalt, Co (ppm)	743	730	757
Copper, Cu (ppm)	3439	3383	3495
Gold, Au (ppb)	191	187	195
Iron, Fe (wt.%)	17.9	17.6	18.3
Molybdenum, Mo (ppm)	80	77	84
Nickel, Ni (ppm)	46	45	47
Sulphur, S (wt.%)	3.04	2.95	3.12

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 3 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 59a

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)	666	17	631	700	614	717	2.57%	5.13%	7.70%	632	699
Co (ppm)	743	18	708	779	690	797	2.39%	4.78%	7.17%	706	780
Cu (ppm)	3439	72	3295	3583	3223	3655	2.10%	4.19%	6.29%	3267	3611
Au (ppb)	191	15	160	222	145	238	8.10%	16.2%	24.3%	182	201
Fe (wt.%)	18.0	0.2	17.5	18.4	17.2	18.7	1.36%	2.73%	4.09%	17.1	18.9
Mo (ppm)	80	7	67	94	60	100	8.27%	16.5%	24.8%	76	84
Ni (ppm)	46	1	44	49	42	50	2.77%	5.54%	8.31%	44	48
S (wt.%)	3.04	0.13	2.77	3.31	2.64	3.44	4.42%	8.83%	13.2%	2.89	3.19

Note - intervals may appear asymmetric due to rounding

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