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**CERTIFICATE OF ANALYSIS FOR**

**Copper Gold Reference Material**

**OREAS 59b**

Prepared by:

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REPORT 02/446-59b

## SOURCE MATERIAL

OREAS 59b 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 59b

Ten commercial laboratories participated in the analytical program to characterise Cu-Au-As-Co-Fe-Mo-Ni-S in OREAS 59b. 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 59b (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	712	716	770	<b>677</b>	710	688	670	610	739	696
2	714	712	780	664	710	695	690	605	735	703
3	720	693	760	664	<b>760</b>	711	690	605	738	674
4	726	668	770	662	720	703	690	615	741	684
5	709	698	775	667	720	691	670	610	740	709
6	714									
7	710									
8	711									
9	721									
10	712									
11	722									
12	718									
13	715									
14	724									
15	713									
Mean	716	697	<b>771</b>	667	724	698	682	<b>609</b>	739	693
Median	714	698	770	664	720	695	690	610	739	696
Std.Dev.	5	19	7	6	21	9	11	4	2	14
Rel.Std.Dev.	0.75%	2.72%	0.96%	0.90%	2.86%	1.34%	1.61%	0.69%	0.32%	2.05%
PDM <sup>3</sup>	2.22%	-0.45%	10.1%	-4.81%	3.35%	-0.42%	-2.64%	-13.1%	5.44%	-1.05%

Table 3. Analytical results for cobalt in OREAS 59b (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	922	964	977	<b>844</b>	820	849	900	815	934	866
2	942	943	985	829	840	865	920	815	932	872
3	941	933	975	830	880	893	910	815	<b>928</b>	836
4	952	908	<b>992</b>	827	860	865	920	830	933	847
5	937	933	976	834	840	857	920	810	933	876
6	934									
7	923									
8	929									
9	949									
10	940									
11	952									
12	939									
13	938									
14	947									
15	937									
Mean	939	936	981	833	848	866	914	817	932	859
Median	939	933	977	830	840	865	920	815	933	866
Std.Dev.	9	20	7	7	23	17	9	8	2	17
Rel.Std.Dev.	0.98%	2.16%	0.75%	0.81%	2.69%	1.92%	0.98%	0.93%	0.26%	2.00%
PDM <sup>3</sup>	5.24%	4.95%	9.97%	-6.64%	-4.94%	-2.94%	2.46%	-8.41%	4.48%	-3.66%

Table 4. Analytical results for copper in OREAS 59b (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	5926	6430	5810	6150	5500	5750	5310	5950	5750
2	5806	6440	5800	5800	<b>5670</b>	5770	5340	5949	5820
3	5817	6440	5710	6000	5540	5790	5380	5930	5600
4	5617	6450	5810	5650	5510	5800	5480	6009	5650
5	5645	<b>6350</b>	5770	5650	5440	5760	5370	5940	5850
Mean	5762	<b>6422</b>	5780	5850	5532	5774	<b>5376</b>	5956	5734
Median	5806	6440	5800	5800	5510	5770	5370	5949	5750
Std.Dev.	129	41	42	221	85	21	64	31	107
Rel.Std.Dev.	2.24%	0.64%	0.73%	3.77%	1.54%	0.36%	1.20%	0.52%	1.87%
PDM <sup>3</sup>	-0.04%	11.40%	0.26%	1.48%	-4.04%	0.16%	-6.74%	3.31%	-0.53%

Table 5. Analytical results for gold in OREAS 59b (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	280.4	300	318	<b>268</b>	370	307	290	294	300	310
2	302.4	310	312	287	350	295	300	293	290	320
3	300.9	300	322	285	350	<b>321</b>	320	315	290	330
4	306.1	300	321	286	340	303	330	301	290	330
5	290.0	290	324	289	340	306	310	302	300	330
6	279.9									
7	293.7									
8	297.3									
9	284.7									
10	283.4									
11	309.6									
12	293.1									
13	293.2									
14	281.9									
15	302.5									
Mean	293.3	300	320	283	<b>350</b>	306	310	301	294	324
Median	293.2	300	321	286	350	306	310	301	290	330
Std.Dev.	9.7	7	5	9	12	9	16	9	5	9
Rel.Std.Dev.	3.32%	2.36%	1.52%	3.01%	3.50%	3.08%	5.10%	2.92%	1.86%	2.76%
PDM <sup>3</sup>	-3.37%	-1.15%	5.34%	-6.75%	15.3%	1.0%	2.1%	-0.82%	-3.13%	6.78%

Table 6. Analytical results for iron in OREAS 59b (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	21.24	21.26	20.9	>15.0	21.2	20.01	19.99	18.2	19.55	18.67
2	21.29	21.36	21.1	>15.0	21.1	20.24	20.16	18.3	19.64	18.76
3	21.22	20.44	21.2	>15.0	20.8	19.91	20.20	18.4	19.31	18.06
4	21.38	19.82	21.1	>15.0	20.7	19.90	20.38	<b>18.8</b>	19.48	18.12
5	21.11	20.66	20.8	>15.0	20.8	20.06	20.14	18.4	19.35	18.92
6	21.15									
7	20.91									
8	20.81									
9	21.09									
10	21.06									
11	21.25									
12	21.11									
13	21.01									
14	21.28									
15	21.12									
Mean	21.14	20.71	21.02	-	20.9	20.02	20.17	18.42	19.47	18.51
Median	21.12	20.66	21.10	-	20.8	20.01	20.16	18.40	19.48	18.67
Std.Dev.	0.15	0.63	0.16	-	0.2	0.14	0.14	0.23	0.14	0.39
Rel.Std.Dev.	0.71%	3.05%	0.78%	-	1.04%	0.69%	0.69%	1.24%	0.71%	2.11%
PDM <sup>3</sup>	5.51%	3.38%	4.94%	-	4.44%	-0.03%	0.71%	-8.04%	-2.82%	-7.61%

Table 7. Analytical results for molybdenum in OREAS 59b (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	122	131	138	<b>112</b>	135	122	110	110	149	93
2	146	130	138	109	150	123	110	105	146	96
3	143	128	136	108	170	<b>128</b>	110	110	148	91
4	132	124	136	107	110	123	110	105	148	94
5	130	127	136	106	135	123	110	110	149	101
6	146									
7	137									
8	117									
9	123									
10	132									
11	134									
12	134									
13	121									
14	107									
15	121									
Mean	130	128	137	108	140	124	110	108	148	95
Median	132	128	136	108	135	123	110	110	148	94
Std.Dev.	11	3	1	2	22	2	0	3	1	4
Rel.Std.Dev.	8.56%	2.14%	0.80%	2.12%	15.8%	1.93%	0.00%	2.54%	0.70%	4.01%
PDM <sup>3</sup>	5.75%	4.44%	11.6%	-11.6%	14.2%	1.01%	-10.2%	-11.9%	20.7%	-22.5%

Table 8. Analytical results for nickel in OREAS 59b (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	50	45	<50	47	50	44	45	28
2	45	53	44	<50	46	50	42	45	29
3	44	51	44	<50	50	50	44	45	27
4	42	51	44	<50	46	50	44	45	28
5	43	<b>47</b>	44	<50	48	50	44	46	29
Mean	44	<b>50</b>	44	-	47	<b>50</b>	44	45	<b>28</b>
Median	44	51	44	-	47	50	44	45	28
Std.Dev.	2	2	0	-	2	0	1	0	1
Rel.Std.Dev.	3.59%	4.35%	1.01%	-	3.53%	0.00%	2.05%	0.90%	2.97%
PDM <sup>3</sup>	-1.93%	12.3%	-1.48%	-	5.65%	11.4%	-2.82%	0.59%	-37.1%

Table 9. Analytical results for sulphur in OREAS 59b (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	3.93	4.65	3.88	4.63	4.12	4.34	3.90	3.63	3.91
2	3.96	4.63	3.91	4.63	4.20	4.23	3.90	3.66	3.93
3	3.44	4.61	3.88	5.05	<b>4.34</b>	4.30	3.90	3.65	3.80
4	3.26	4.81	3.88	4.86	4.21	4.24	4.00	3.68	3.79
5	3.90	4.70	3.90	4.83	4.18	4.20	3.90	3.67	3.94
Mean	3.70	4.68	3.89	4.80	4.21	4.26	3.92	3.66	3.87
Median	3.90	4.65	3.88	4.83	4.20	4.24	3.90	3.66	3.91
Std.Dev.	0.32	0.08	0.01	0.18	0.08	0.06	0.04	0.02	0.07
Rel.Std.Dev.	8.76%	1.71%	0.36%	3.68%	1.92%	1.33%	1.14%	0.53%	1.88%
PDM <sup>3</sup>	-9.93%	14.0%	-5.28%	16.9%	2.51%	3.78%	-4.55%	-10.9%	-5.67%

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 59b

### 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 59b.

Constituent	Certified value	95% Confidence interval	
		Low	High
Arsenic, As (ppm)	701	682	719
Cobalt, Co (ppm)	892	853	931
Copper, Cu (ppm)	5765	5636	5894
Gold, Au (ppb)	303	294	313
Iron, Fe (wt.%)	20.0	19.2	20.8
Molybdenum, Mo (ppm)	123	110	135
Nickel, Ni (ppm)	45	43	47
Sulphur, S (wt.%)	4.11	3.79	4.42

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 5689 and 5840 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 59b.

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 59b.

Constituent	Certified value	Tolerance limits 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Arsenic, As (ppm)	701	691	710
Cobalt, Co (ppm)	892	882	903
Copper, Cu (ppm)	5765	5689	5840
Gold, Au (ppb)	303	300	307
Iron, Fe (wt.%)	20.0	19.7	20.4
Molybdenum, Mo (ppm)	123	118	127
Nickel, Ni (ppm)	45	44	46
Sulphur, S (wt.%)	4.11	4.00	4.21

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 59b

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)	701	17	667	734	651	750	2.37%	4.73%	7.10%	665	736
Co (ppm)	892	24	845	939	821	963	2.65%	5.30%	7.95%	847	937
Cu (ppm)	5765	182	5401	6130	5218	6313	3.16%	6.33%	9.49%	5477	6054
Au (ppb)	303	17	270	337	253	354	5.57%	11.1%	16.7%	288	319
Fe (wt.%)	20.0	0.5	19.1	21.0	18.6	21.5	2.40%	4.81%	7.21%	19.0	21.0
Mo (ppm)	123	14	94	151	80	165	11.5%	23.0%	34.5%	116	129
Ni (ppm)	45	2	41	49	39	50	4.18%	8.36%	12.5%	43	47
S (wt.%)	4.13	0.13	3.86	4.40	3.73	4.53	3.25%	6.51%	9.76%	3.92	4.34

Note - intervals may appear asymmetric due to rounding

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