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CERTIFICATE OF ANALYSIS FOR

Copper Gold Reference Material

OREAS 59c

Prepared by:
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REPORT 02/446-59c

SOURCE MATERIAL

OREAS 59c 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.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

a) drying for 24 hours at 105^0 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 59c

Ten commercial laboratories participated in the analytical program to characterise Cu-Au-As-Co-Fe-Mo-Ni-S in OREAS 59c. 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^3) are presented in Tables 2 to 9. The parameter PDM^3 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 59c (Std.Dev. and Rel.Std.Dev. are one sigma values; PDM[3] - 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	588	552	620	546	610	567	600	505	604	567
2	571	577	615	539	610	556	590	520	603	582
3	582	583	605	552	600	569	600	510	**590**	575
4	577	557	615	555	590	547	590	**320**	603	558
5	573	555	610	549	590	547	600	520	598	558
6	587									
7	574									
8	582									
9	571									
10	573									
11	585									
12	583									
13	580									
14	585									
15	585									
Mean	580	565	613	548	600	557	596	475	600	568
Median	582	557	615	549	600	556	600	510	603	567
Std.Dev.	6	14	6	6	10	11	5	87	6	11
Rel.Std.Dev.	1.03%	2.51%	0.93%	1.12%	1.67%	1.89%	0.92%	18.29%	1.01%	1.86%
PDM[3]	0.93%	-1.65%	6.75%	-4.54%	4.48%	-2.97%	3.79%	-17.3%	4.40%	-1.09%

Table 3. Analytical results for cobalt in OREAS 59c (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	831	760	808	697	700	713	850	695	786	724
2	809	781	804	700	700	705	870	710	788	744
3	825	**815**	802	713	720	**729**	880	700	784	731
4	811	772	799	711	720	709	870	**445**	790	711
5	804	786	**700**	702	720	698	860	710	795	718
6	825									
7	807									
8	812									
9	808									
10	817									
11	830									
12	834									
13	816									
14	832									
15	820									
Mean	818	783	783	705	712	711	**866**	652	788	726
Median	817	781	802	702	720	709	870	700	788	724
Std.Dev.	10	21	46	7	11	12	11	116	4	13
Rel.Std.Dev.	1.25%	2.62%	5.91%	1.00%	1.54%	1.63%	1.32%	17.8%	0.50%	1.74%
PDM[3]	9.34%	4.57%	4.55%	-5.87%	-4.89%	-5.05%	15.7%	-12.9%	5.33%	-3.07%

Table 4. Analytical results for copper in OREAS 59c (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	0.973	1.12	1.04	0.985	0.986	1.146	1.04	1.02	1.02
2	1.006	1.12	1.05	0.990	0.987	1.162	1.04	1.04	1.01
3	1.049	1.12	1.05	1.010	1.020	1.153	1.04	1.03	1.04
4	1.014	1.10	1.05	1.030	0.995	1.142	1.03	1.04	1.02
5	1.031	1.10	1.05	1.015	1.010	1.137	1.01	1.05	1.05
Mean	1.015	**1.11**	1.05	1.006	1.000	**1.148**	1.032	1.037	1.03
Median	1.014	1.12	1.05	1.010	0.995	1.146	1.040	1.035	1.02
Std.Dev.	0.028	0.01	0.00	0.019	0.015	0.010	0.013	0.011	0.02
Rel.Std.Dev.	2.80%	0.99%	0.43%	1.84%	1.49%	0.85%	1.26%	1.03%	1.60%
PDM[3]	-0.86%	8.64%	2.39%	-1.72%	-2.34%	12.16%	0.82%	1.28%	0.43%

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

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	0.581	0.60	0.622	0.549	0.64	0.603	0.56	0.572	0.57	0.63
2	**0.545**	**0.57**	0.638	0.558	0.63	0.614	0.57	0.577	0.57	0.63
3	0.572	0.60	0.614	**0.519**	0.62	0.611	0.58	**0.559**	0.58	0.64
4	0.562	0.59	0.627	0.552	0.62	0.629	0.58	0.574	0.57	0.65
5	0.587	0.60	0.613	0.567	**0.60**	0.592	0.59	0.580	0.58	0.63
6	0.600									
7	0.580									
8	0.581									
9	0.557									
10	0.558									
11	0.581									
12	0.566									
13	0.584									
14	0.590									
15	0.578									
Mean	0.575	0.592	0.623	0.549	0.619	0.610	0.576	0.572	0.574	0.636
Median	0.580	0.600	0.622	0.552	0.620	0.611	0.580	0.574	0.570	0.630
Std.Dev.	0.015	0.013	0.010	0.018	0.013	0.014	0.011	0.008	0.005	0.009
Rel.Std.Dev.	2.56%	2.20%	1.65%	3.30%	2.09%	2.25%	1.98%	1.41%	0.95%	1.41%
PDM[3]	-3.36%	-0.49%	4.69%	-7.72%	4.05%	2.49%	-3.18%	-3.78%	-3.51%	6.91%

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

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	20.44	19.13	19.7	>15.0	19.9	19.32	21.557	18.0	18.27	17.96
2	20.03	19.81	19.7	>15.0	19.6	18.65	21.754	18.6	18.58	18.43
3	20.18	20.14	19.6	>15.0	19.8	19.24	21.685	18.2	18.52	18.15
4	20.01	19.46	19.4	>15.0	20.2	19.10	21.531	**11.3**	18.50	17.64
5	19.95	19.67	19.6	>15.0	19.9	19.01	21.524	18.4	18.83	17.70
6	20.48									
7	19.98									
8	20.15									
9	19.92									
10	20.04									
11	20.52									
12	20.14									
13	20.21									
14	20.51									
15	20.38									
Mean	20.20	19.64	19.60	-	19.88	19.06	21.61	**16.90**	18.54	17.98
Median	20.15	19.67	19.60	-	19.90	19.10	21.56	18.20	18.52	17.96
Std.Dev.	0.22	0.38	0.12	-	0.22	0.26	0.10	3.14	0.20	0.33
Rel.Std.Dev.	1.07%	1.93%	0.62%	-	1.09%	1.37%	0.48%	18.6%	1.08%	1.81%
PDM[3]	3.23%	0.40%	0.19%	-	1.62%	-2.55%	10.5%	-13.6%	-5.23%	-8.11%

Table 7. Analytical results for molybdenum in OREAS 59c (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	181	181	192	153	205	180	200	160	210	129
2	185	190	194	150	225	173	200	165	212	130
3	177	189	192	147	235	181	190	160	212	120
4	190	183	190	157	220	175	200	**105**	212	133
5	163	186	192	156	220	173	200	160	214	133
6	173									
7	175									
8	183									
9	178									
10	189									
11	194									
12	186									
13	182									
14	177									
15	190									
Mean	181	186	192	153	221	176	198	150	212	129
Median	182	186	192	153	220	175	200	160	212	130
Std.Dev.	8	4	1	4	11	4	4	25	1	5
Rel.Std.Dev.	4.42%	2.06%	0.74%	2.73%	4.90%	2.18%	2.26%	16.8%	0.60%	4.14%
PDM[3]	0.30%	2.68%	6.11%	-15.7%	22.1%	-2.51%	9.4%	-17.1%	17.1%	-28.7%

Table 8. Analytical results for nickel in OREAS 59c (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	50	63	52	50	52	60	50	53	40
2	52	61	53	50	51	60	54	54	40
3	53	59	52	50	55	70	52	53	40
4	51	60	54	50	56	60	**34**	54	39
5	52	62	52	50	54	60	56	54	39
Mean	52	**61**	53	**50**	54	**62**	49	54	**40**
Median	52	61	52	50	54	60	52	54	40
Std.Dev.	1	2	1	0	2	4	9	1	1
Rel.Std.Dev.	2.21%	2.59%	1.70%	0.00%	3.87%	7.21%	17.9%	1.00%	1.38%
PDM[3]	-2.42%	15.4%	-0.53%	-5.45%	1.36%	17.2%	-6.96%	1.36%	-25.1%

Table 9. Analytical results for sulphur in OREAS 59c (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	2.80	4.51	3.75	4.62	4.04	4.18	3.80	3.58	3.74
2	2.80	4.39	3.76	4.68	3.99	3.94	3.95	3.59	3.82
3	**3.20**	4.38	3.84	4.71	4.12	3.66	3.85	3.57	3.73
4	2.79	4.41	3.78	4.77	3.99	4.16	**2.60**	3.60	3.66
5	**2.54**	4.35	3.71	4.76	3.96	3.82	3.90	3.65	3.68
Mean	2.83	4.41	3.77	4.71	4.02	3.95	3.62	3.60	3.73
Median	2.80	4.39	3.76	4.71	3.99	3.94	3.85	3.59	3.73
Std.Dev.	0.24	0.06	0.05	0.06	0.06	0.22	0.57	0.03	0.06
Rel.Std.Dev.	8.33%	1.38%	1.26%	1.30%	1.56%	5.63%	15.8%	0.90%	1.67%
PDM[3]	-28%	13%	-3.56%	21%	2.89%	1.15%	-7.35%	-7.85%	-4.63%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 59c

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

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

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

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.*

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).

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

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

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

$$S = 1.483\ \underset{j=1.....n}{median} / x_j - \underset{i=1.....n}{median}\ (x_i) /$$

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

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 59c.

Constituent	Certified value	95% Confidence interval	
		Low	High
Arsenic, As (ppm)	574	553	596
Cobalt, Co (ppm)	749	712	785
Copper, Cu (wt.%)	1.02	1.01	1.04
Gold, Au (ppm)	0.595	0.576	0.614
Iron, Fe (wt.%)	19.6	18.6	20.5
Molybdenum, Mo (ppm)	181	161	201
Nickel, Ni (ppm)	53	52	54
Sulphur, S (wt.%)	3.91	3.66	4.15

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

$$\text{Lower limit is } \ddot{x} - k'_2(n, p, 1-\alpha)s''_g$$
$$\text{Upper limit is } \ddot{x} + k'_2(n, p, 1-\alpha)s''_g$$

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 1.00 and 1.04 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 (IS0 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

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

$$s_g' \text{ is the grand standard deviation computed from the transformed (i.e. means - adjusted) results}$$

according to the formula

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

$$\text{where } \bar{x}_i' \text{ is the transformed mean for laboratorty } i$$

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 59c.
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 59c.

Constituent	Certified value	Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High
Arsenic, As (ppm)	574	563	586
Cobalt, Co (ppm)	749	737	760
Copper, Cu (wt.%)	1.02	1.00	1.04
Gold, Au (ppm)	0.595	0.590	0.600
Iron, Fe (wt.%)	19.6	19.3	19.9
Molybdenum, Mo (ppm)	181	175	187
Nickel, Ni (ppm)	53	51	55
Sulphur, S (wt.%)	3.91	3.81	4.01

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 59c

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)	574	15	544	604	529	619	2.60%	5.21%	7.81%	546	603
Co (ppm)	749	17	714	783	697	800	2.31%	4.61%	6.92%	711	786
Cu (wt.%)	1.02	0.03	0.97	1.08	0.94	1.11	2.74%	5.48%	8.21%	0.97	1.07
Au (ppm)	0.595	0.019	0.557	0.632	0.539	0.651	3.15%	6.30%	9.45%	0.565	0.625
Fe (wt.%)	19.6	0.4	18.7	20.4	18.2	20.9	2.25%	4.51%	6.76%	18.6	20.5
Mo (ppm)	181	9	163	199	154	208	5.01%	10.0%	15.0%	172	190
Ni (ppm)	53	4	45	61	41	64	7.21%	14.4%	21.6%	50	56
S (wt.%)	3.90	0.16	3.57	4.22	3.41	4.39	4.19%	8.38%	12.6%	3.70	4.09

Note - intervals may appear asymmetric due to rounding

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