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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD-SULPHUR
REFERENCE MATERIAL OREAS 56P

SUMMARY STATISTICS

Recommended Values, 95% Confidence and Tolerance Intervals

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppm)	0.746	0.724	0.768	0.739	0.753
Copper, Cu (ppm)	1064	1039	1089	1045	1083
Sulphur, S (%)	1.82	1.74	1.90	1.78	1.86

Prepared by:
Ore Research & Exploration Pty Ltd
July, 2009

SOURCE MATERIAL

Reference material OREAS 56P is a copper-gold-sulphur standard prepared from low grade material from a porphyry Cu-Au deposit, central New South Wales, Australia.

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 60g lots sealed in laminated foil pouches.

ANALYSIS OF OREAS 56P

Ten Australian commercial laboratories participated in the analytical program for gold, copper and sulphur. To evaluate the effects of batch to batch variation, samples were submitted to the laboratories in four batches, dispatched at weekly intervals. All 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 1, 3 and 5. 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. The analytical methods employed by each laboratory are given in the table captions. With the exception of Lab A each laboratory received a batch of five 60g samples for analysis. Gold (Table 1) was determined at these laboratories using a fire assay technique (40-50g charge with new pots) with AAS or ICPOES finish. Lab A determined gold in twenty replicates via instrumental neutron activation analysis (INAA) using reduced analytical subsample weights of 0.5g. Copper and sulphur were determined by aqua regia digest with ICPOES finish (Table 3). Lab F determined sulphur by Leco high temperature combustion.
Batch means for each laboratory and standard deviations (95% confidence) of these means are given in Tables 2, 4 and 6, the latter parameter providing a measure of inter-batch bias at each laboratory.
The five subsamples comprising each batch were taken at widely spaced intervals during packaging of the standard in order to maximise their representation. The twenty INAA subsamples, on which much of the homogeneity evaluation is based, were also taken at regular intervals during packaging and are similarly considered representative of the entire batch.

Table 1. Analytical results for gold in OREAS 56P (INAA - instrumental neutron activation analysis; FA-AAS - fire assay / atomic absorption spectrometry; FA-OES - fire assay / inductively coupled optical emission spectrometry; Std.Dev. and Rel.Std.Dev. are one sigma values; PDM[3] - percent deviation of lab mean from corrected mean of means; individual outliers in left-justified bold; outlying batches in bold; values in parts per million).

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-OES (40g)	Lab F FA-AAS (50g)	Lab G FA-AAS (50g)	Lab H FA-AAS (50g)	Lab I FA-AAS (50g)	Lab J FA-AAS (40g)
1	0.683	0.75	0.75	0.715	0.729	**0.66**	0.77	**0.67**	0.73	0.785
2	0.709	0.78	0.75	0.730	0.741	**0.61**	0.74	**0.66**	0.73	0.785
3	0.711	0.76	0.77	0.715	0.734	**0.64**	0.76	**0.66**	0.72	0.815
4	0.714	0.76	0.76	0.740	0.742	**0.64**	0.77	**0.66**	0.72	0.805
5	0.677	0.77	0.75	0.755	0.737	**0.62**	0.79	**0.67**	0.74	0.800
6	0.672	0.71	0.74	0.785	0.788	0.78	0.77	0.74	0.74	0.81
7	0.687	0.7	0.73	0.760	0.755	0.74	0.75	0.75	0.75	0.82
8	0.667	0.72	0.73	0.805	0.758	0.75	0.77	0.77	0.73	0.80
9	0.730	0.71	0.74	0.775	0.747	0.77	0.75	0.73	0.75	0.80
10	0.715	0.72	0.73	0.795	0.781	0.75	0.73	0.73	0.74	0.80
11	0.696	0.74	**0.87**	0.747	0.745	0.73	0.79	**0.84**	0.75	0.76
12	0.697	0.75	0.68	0.757	0.770	0.77	0.76	**0.82**	0.74	0.76
13	0.670	0.75	0.76	0.770	0.757	0.76	0.75	**0.82**	0.73	0.75
14	0.691	0.75	0.72	0.746	0.744	0.75	0.75	**0.76**	0.73	0.75
15	0.653	0.74	0.75	0.740	0.750	0.73	0.76	**0.80**	0.72	0.78
16	0.718	0.79	0.71	**0.810**	0.715	0.75	0.73	0.72	0.75	**0.845**
17	0.718	0.75	0.77	**0.900**	0.725	0.71	0.75	0.71	0.77	0.795
18	0.679	0.76	0.78	**0.800**	0.735	0.73	0.76	0.74	0.74	0.805
19	0.685	0.75	0.74	**0.845**	0.761	0.72	0.74	0.75	0.74	0.805
20	0.721	0.76	0.8	**0.695**	0.715	0.71	0.75	0.76	0.75	0.825
Mean	0.695	0.746	0.752	0.769	0.746	0.716	0.757	0.738	0.739	0.795
Median	0.694	0.750	0.750	0.759	0.745	0.730	0.755	0.740	0.740	0.800
Std.Dev.	0.021	0.024	0.038	0.048	0.019	0.053	0.017	0.056	0.013	0.025
Rel.Std.Dev.	3.09%	3.18%	5.08%	6.24%	2.61%	7.37%	2.19%	7.53%	1.72%	3.16%
PDM[3]	-6.87%	0.02%	0.75%	3.13%	0.08%	-4.01%	1.49%	-1.06%	-0.99%	6.55%

Table 2. Batch means for gold in OREAS 56P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in ppb).

Batch Number	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	0.764	0.756	0.731	0.737	**0.634**	0.766	**0.664**	0.728	0.798
2	0.712	0.734	0.784	0.766	0.758	0.754	0.744	0.742	0.806
3	0.746	0.728	0.752	0.753	0.748	0.762	**0.820**	0.734	0.760
4	0.762	0.760	**0.810**	0.730	0.724	0.746	0.736	0.750	0.808
Overall Mean	0.746	0.744	0.769	0.746	0.716	0.757	0.741	0.739	0.793
Std.Dev.	0.048	0.032	0.070	0.032	0.113	0.018	0.128	0.019	0.045
Rel.Std.Dev.	6.4%	4.3%	9.1%	4.3%	15.8%	2.3%	17.2%	2.6%	5.6%

Table 3. Analytical results for copper in OREAS 56P (AR-OES – aqua regia digest / induct-ively coupled plasma optical emission spectrometry; abbreviations as in Table 1; 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	1096	1130	1045	1030	1050	1050	1090	1010	**1250**
2	1093	1120	1038	1120	1070	1050	1050	1015	**1450**
3	1092	1140	1042	1140	1050	1060	1070	1025	**1400**
4	1086	1110	1041	1090	1050	1040	1040	1020	**1500**
5	**1064**	1110	1035	1060	1040	1070	1060	**990**	**1450**
6	1074	1060	1033	1150	1050	1090	1130	1040	1100
7	1059	1070	1058	1150	1050	1070	1100	1030	1100
8	1068	1040	1069	**1190**	**1080**	1070	**1070**	1015	1100
9	1064	1050	1051	1140	1040	1080	1120	1055	1100
10	**1091**	1040	1025	1110	1040	1090	1110	1035	1000
11	1103	1060	1068	**1150**	1000	1060	1030	1070	**914**
12	1099	1080	1086	**1140**	1030	1070	1040	1080	**916**
13	1105	1070	1056	**1150**	1020	1090	1010	1065	**956**
14	1096	1050	1079	**1160**	1030	**1010**	1020	1060	**953**
15	1096	1070	1064	**1130**	**1070**	1060	1020	**1115**	**949**
16	1079	1060	1031	**1170**	998	1060	1160	1085	995
17	1089	1090	1035	**1190**	1010	1050	1090	1065	**1110**
18	1072	1060	**1148**	**1160**	1020	1050	1090	1090	1010
19	1051	1070	1082	**1180**	**1060**	1060	1170	1085	993
20	1064	1050	1052	**1180**	998	1060	1100	1105	963
Mean	1082	1077	1057	1140	1038	1062	1079	1053	1110
Median	1088	1070	1051	1150	1040	1060	1080	1058	1055
Std.Dev.	16	30	28	41	24	19	46	35	193
Rel.Std.Dev.	1.51%	2.8%	2.65%	3.64%	2.34%	1.77%	4.25%	3.30%	17.4%
PDM[3]	1.66%	1.14%	-0.73%	7.05%	-2.50%	-0.23%	1.32%	-1.10%	4.33%

Table 4. Batch means for copper in OREAS 56P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in ppm).

Batch Number	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	1092	1122	1040	1088	1052	1054	1062	1018	**1450**
2	1066	1052	1047	1138	1045	1080	1115	1035	1080
3	1100	1066	1070	**1146**	1020	1070	1024	1069	**938**
4	1071	1066	1050	**1176**	1007	1056	1122	1086	990
Overall Mean	1082	1077	1052	1137	1031	1065	1081	1052	1114
Std.Dev.	32	62	26	73	43	25	93	62	463
Rel.Std.Dev.	3.0%	5.8%	2.5%	6.4%	4.1%	2.3%	8.6%	5.9%	41.5%

Table 5. Analytical results for sulphur in OREAS 56P (AR-OES – aqua regia digest / induct-ively coupled plasma optical emission spectrometry; Leco – high temperature combustion; abbreviations as in Table 1; values in percent; NR – not reported).

Replicate No.	Lab B AR-OES	Lab C AR-OES	Lab D AR-OES	Lab E AR-OES	Lab F Leco	Lab G AR-OES	Lab H AR-OES	Lab I AR-OES	Lab J AR-OES
1	1.814	1.98	1.586	1.73	1.77	1.71	1.93	1.73	1.73
2	1.809	1.91	1.552	1.77	1.77	1.73	1.88	1.71	1.85
3	1.795	1.89	1.572	1.81	**1.72**	1.73	1.89	1.74	1.75
4	1.812	1.98	1.590	1.68	1.76	1.71	1.86	1.72	1.79
5	**1.755**	1.93	1.566	1.66	1.79	1.75	1.86	**1.68**	1.81
6	1.771	1.87	1.534	2.07	1.83	1.77	2.00	1.78	1.98
7	1.763	**1.92**	1.577	2.08	1.82	1.74	1.93	1.75	1.97
8	1.732	1.82	1.590	2.02	1.83	1.72	**1.90**	1.74	1.96
9	1.749	1.86	1.587	2.04	1.82	1.74	1.98	1.80	1.93
10	**1.815**	1.86	1.560	**1.90**	1.80	1.76	1.99	1.77	**1.27**
11	1.859	**2.13**	1.744	1.96	1.87	1.70	1.85	NR	1.93
12	1.886	**2.24**	1.736	1.98	1.87	1.74	1.85	NR	1.91
13	1.887	**2.16**	1.715	1.94	1.87	1.76	1.83	NR	1.96
14	1.848	**2.12**	1.715	1.93	1.88	1.63	1.83	NR	1.94
15	1.839	**2.19**	1.715	1.95	1.84	1.69	1.81	NR	1.93
16	1.830	1.83	1.652	2.00	1.84	1.73	1.97	1.85	1.91
17	1.798	1.85	1.685	2.01	1.87	1.70	1.86	1.83	**2.11**
18	1.765	1.85	1.877	1.96	1.91	1.73	1.86	1.92	1.93
19	1.791	1.83	1.748	1.97	1.98	1.71	1.96	2.02	1.89
20	1.791	1.86	1.747	1.96	1.87	1.73	1.88	1.98	1.82
Mean	1.805	1.954	1.652	1.921	1.836	1.724	1.896	1.801	1.869
Median	1.803	1.900	1.621	1.960	1.835	1.730	1.880	1.770	1.920
Std.Dev.	0.044	0.136	0.093	0.125	0.059	0.031	0.058	0.101	0.166
Rel.Std.Dev.	2.41%	6.96%	5.65%	6.49%	3.21%	1.78%	3.08%	5.62%	8.90%
PDM[3]	-1.06%	7.09%	-9.44%	5.28%	0.59%	-5.51%	3.91%	-1.28%	2.40%

Table 6. Batch means for sulphur in OREAS 56P (outlying batches in bold; Overall Means are means of batch means; Std.Dev. and Rel.Std.Dev. are two sigma values for batch means; values in percent).

Batch Number	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	1.81	1.94	1.57	1.73	1.77	1.73	1.88	1.73	1.79
2	1.75	1.85	1.57	2.05	1.82	1.75	1.98	1.77	1.96
3	1.86	**2.17**	1.73	1.95	1.87	1.70	1.83	NR	1.93
4	1.80	1.84	1.74	1.98	1.89	1.72	1.91	1.92	1.89
Overall Mean	1.805	1.951	1.652	1.929	1.838	1.724	1.900	1.804	1.892
Std.Dev.	0.091	0.302	0.188	0.278	0.107	0.034	0.117	0.205	0.153
Rel.Std.Dev.	5.0%	15.5%	11.4%	14.4%	5.8%	2.0%	6.2%	11.4%	8.1%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR 56P

Recommended 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).

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

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

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

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

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

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 left-justified bold and bold, respectively, and have been omitted in the determination of recommended 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 recommended value, i.e. the narrower the confidence interval the greater the certainty in the recommended value.

Table 7. Recommended values and 95% confidence intervals for 56P.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	0.746	0.724	0.768
Copper, Cu (ppm)	1064	1039	1089
Sulphur, S (%)	1.82	1.74	1.90

Note - intervals may appear 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 dataset. 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 jth raw result reported by laboratory i*;

x'_{ij} *is the jth 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*;

ρ *is the proportion of results expected within the tolerance limits*;

k'_2 *is the factor for two-sided tolerance limits* (m, σ *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 1045 and 1083 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 (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

$1-(\frac{s_i}{2s'_g})$ *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

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

where $\bar{x}'_i$ is the transformed mean for 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.

Table 8. Recommended values and tolerance limits for 56P.

Constituent	Recommended value	Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	0.746	0.739	0.753
Copper, Cu (ppm)	1064	1045	1083
Sulphur, S (%)	1.82	1.78	1.86

Note - intervals may appear asymmetric due to rounding

For gold a more simplified procedure was used in the determination of homogeneity. This entailed using the high precision INAA data alone, obtained on a reduced analytical subsample weight of 0.5g. By employing a sufficiently reduced subsample weight in a series of determinations by the same method, analytical error should become negligible in comparison to subsampling error. The standard deviation at a 50g subsample weight has been 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, in particular the 0.5g INAA data, is consistent with the narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in 56P.
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 $s_l / 2s_g' > 1$ (i.e. where the weighting factor $1 - s_l / 2s_g' < 0$).

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 deviations are then calculated for each lab's results and then each SD is tested for outlying status using z-score discrimination (rejected if $|z_i| > 2.5$). The 1SD used to calculate performance gates is the mean of the remaining (accepted) lab standard deviations. Table 9 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.
Both methods should be used with caution 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 9. Performance Gate for OREAS 56P

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
Au (ppm)	0.746	0.021	0.705	0.787	0.684	0.808	2.75%	5.50%	8.24%	0.709	0.783
Cu (ppm)	1064	30	1004	1124	975	1153	2.80%	5.60%	8.40%	1011	1117
S (wt.%)	1.82	0.07	1.68	1.97	1.61	2.04	3.92%	7.85%	11.8%	1.73	1.91

Note - intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

ALS Chemex, Garbutt, QLD, Australia
ALS Chemex, Orange, NSW, Australia
Amdel Laboratories, Orange, NSW, Australia
Amdel Laboratories, Thebarton, SA, Australia
Amdel Laboratories, Wangara, WA, Australia
Lab A Laboratories, Lucas Heights, NSW, Australia
Genalysis, Maddington, WA, Australia
SGS Analabs, Garbutt, QLD, Australia
SGS Analabs, Welshpool, WA, Australia
Ultra Trace, Canning Vale, WA, Australia

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