MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

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4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 514

COPPER CONCENTRATE

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, AUSTRALIA, BRAZIL, CHILE, INDIA, THE NETHERLANDS, PERU AND THE UNITED STATES OF AMERICA, issued by the Bureau of Analysed Samples Ltd

ANALYSES

Mean of 4 values - mass content in %.

Line No.	SiO_2	MgO	CaO	Cu	Zn	Pb	S	Fe	Ag	Au	Cd	Mn	Ni	*Al_2O_3*	*Na_2O*	*K_2O*	*As*	*Bi*
1	2.8950	0.1700	0.2400	25.1025	0.2498	0.0900	35.5523	30.9837	0.0030	0.00140	0.0035	0.0020	0.0017	*0.4941*	*0.0540*	*0.0593*	*0.0005*	*0.0004*
2	2.8969	0.1763	0.2439	25.1625	0.2510	0.0974	35.5950	30.9900	0.0032	0.00188	0.0038	0.0020	0.0018	*0.5152*	*0.0657*	*0.0696*	*0.0006*	*0.0006*
3	2.9025	0.1780	0.2546	25.1850	0.2570	0.0987	35.7838	31.2525	0.0032	0.00190	0.0040	0.0024	0.0020	*0.5300*	*0.0709*	*0.0700*	*0.0007*	*0.00093*
4	2.9312	0.1811	0.2617	25.1860	0.2600	0.0990	35.7900	31.3250	0.0033	0.00192	0.0042	0.0026	0.0021	*0.5588*	*0.0741*	*0.0810*	*<0.001*	*0.0018*
5	2.9825	0.1817	0.2675	25.2025	0.2650	0.0998	35.9425	31.3625	0.0033	0.00193	0.0043	0.0028	0.0023	*0.6000*	*0.0750*	*0.0880*	*<0.001*	*<0.001*
6	2.9963	0.1867	0.2825	25.2200	0.2703	0.1000	35.9650	31.4125	0.0033	0.00193	0.0043	0.0029	0.0025	*0.6200*	*0.0753*	*0.0900*	*<0.002*	*<0.002*
7	3.0250	0.1900	0.2910	25.2294	0.2723	0.1025	35.9948	31.4300	0.0033	0.00194	0.0046	0.0031		*0.6275*	*0.0782*	*0.0905*	*<0.005*	*<0.002*
8	3.1338	0.1950		25.2363	0.2754	0.1031	36.0300	31.5565	0.0033	0.00195	0.0049	0.0033		*0.6585*	*0.1000*	*0.1000*	*<0.005*	*0.0045*
9		0.1954		25.2725	0.2858	0.1080	36.0900	31.6193	0.0034	0.00197	0.0049	0.0035		*0.6857*				
10				25.2734	0.2894		36.1297	31.7625	0.0035	0.00200								
11				25.3250				31.8698	0.0035	0.00240								
12				25.3325					0.0036									
13				25.3388					0.0037									
M_M	**2.9704**	**0.1838**	**0.2630**	**25.2359**	**0.2676**	**0.0998**	**35.8873**	**31.4149**	**0.0034**	**0.00193**	**0.0043**	**0.0027**	**0.0021**					
s_M	0.0826	0.0087	0.0190	0.0709	0.0137	0.0049	0.1997	0.2817	0.0002	0.00023	0.0005	0.0006	0.0004					
s_W	0.0516	0.0038	0.0053	0.0434	0.0042	0.0010	0.0796	0.0612	0.0001	0.00010	0.0003	0.0002	0.0003					

Line No.	*C*	*Mo*	*Cl*	*Co*	*Cr*	*F*	*Hg*	*In*	*P*	*Sb*	*Se*	*Te*	*Ti*	*Ba*	*Be*	*Ge*	*Sn*	*Tl*
1	*0.0563*	*0.0498*	*<0.001*	*0.0071*	*0.0003*	*0.0035*	*<0.0001*	*0.0001*	*0.0030*	*0.0016*	*0.01600*	*<0.001*	*0.0110*	*<0.0001*	*<0.00005*	*0.0006*	*<0.0001*	*<0.0003*
2	*0.0648*	*0.0515*	*<0.001*	*0.0075*	*0.0010*	*0.0042*	*<0.0001*	*0.0009*	*0.0046*	*0.0018*	*0.01820*	*<0.001*	*0.0144*	*<0.0003*	*<0.0001*	*<0.001*	*0.0002*	*<0.0005*
3	*0.0673*	*0.0520*	*<0.001*	*0.0090*	*0.0010*	*0.0043*	*<0.0001*	*<0.002*	*0.0046*	*<0.002*	*0.02150*	*0.0010*	*0.0154*	*0.0011*	*<0.0005*	*<0.002*	*0.0002*	*<0.001*
4	*0.0679*	*0.0531*	*0.0013*	*0.0100*	*0.0020*	*0.0048*	*0.0001*	*0.0020*	*0.0048*	*<0.0025*	*0.02180*	*0.0016*	*0.0183*	*0.0020*			*<0.001*	*0.0015*
5		*0.0550*	*0.0052*	*0.0100*	*0.0027*	*0.0050*	*0.0001*	*0.0020*	*0.0050*	*0.0037*	*0.02370*	*<0.002*	*0.0190*	*0.0022*			*<0.005*	
6		*0.0576*		*0.0103*	*0.0035*	*0.0051*	*0.0001*	*0.0022*	*0.0084*	*<0.004*	*0.02430*	*<0.005*	*0.0194*	*0.0046*			*0.0075*	
7		*0.0583*		*0.0105*	*<0.005*	*0.0066*	*<0.0005*	*0.0025*	*0.0125*	*<0.005*			*0.0200*				*0.0086*	
8		*0.0603*		*0.0113*	*<0.005*		*<0.0005*			*0.0052*								
9		*0.0605*		*0.0129*														
10		*0.0611*																
11		*0.0625*																
12		*0.0650*																
13		*0.0700*																

The above figures are those which each Analyst has decided upon after careful verification

Values given in italics are for information only

Additional Information: 2 Analysts determined U by ICP-OES and each reported <0.001%. 2 Analysts determined Re and found <0.0005% each. 2 Analysts determined Th by ICP-OES and reported values of <0.001 and < 0.0005%. One Analyst determined Ga and V by FAAS and reported values of 0.0087% and <0.05% respectively.

M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means. s_W: intralaboratory standard deviation.

CERTIFIED VALUES (Cv)

mass content in %

	SiO_2	MgO	CaO	Cu	Zn	Pb	S
Cv	**2.97**	**0.184**	**0.263**	**25.24**	**0.268**	**0.100**	**35.89**
C(95%)	0.07	0.007	0.018	0.04	0.010	0.004	0.15

	Fe	Ag	Au	Cd	Mn	Ni
Cv	**31.41**	**0.0034**	**0.00193**	**0.0043**	**0.0027**	**0.0021**
C(95%)	0.19	0.0002	0.00016	0.0004	0.0005	0.0004

The half width confidence interval **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate two sided Student's t value at the 95% confidence level for "n" acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 sections 6.1 and 10.5.2

NB: Although widely accepted within the industry "mass content in %" is neither an SI nor an IUPAC supported quantity. Multiplication of the certified value (Cv) by 10^4 will yield the value in µg/g.

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NOTES ON METHODS USED

SILICA

All results for silica, apart from No. 4, were obtained gravimetrically. No. 1 fused the sample with Na_2CO_3/K_2CO_3 and dehydrated the silica with hydrochloric acid, Nos. 2, 7 and 8 dehydrated the silica with perchloric acid (No. 8 fused the sample with sodium peroxide) and No. 6 used sulphuric acid (according to IS:3685-1966). Result No. 4 was obtained by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) after fusion.

MAGNESIUM OXIDE

Results Nos. 1, 2, 5 and 7 for magnesium oxide were obtained by Flame Atomic Absorption Spectrometry (FAAS) following acid digestion. The remaining results were obtained by ICP-OES; Result No. 3 after fusion with Na_2O_2 and No. 6 after an acid digestion.

CALCIUM OXIDE

Results 1, 3, 5 and 6 for calcium oxide were obtained using FAAS; Nos. 3 and 6 used an acid digestion. Results Nos. 2, 4 and 7 were from analysis by ICP-OES; No.7 after fusion with Na_2O_2.

COPPER

Results Nos. 1, 6, 10 and 13 for copper were obtained by electrogravimetric methods. Nos. 2, 3, 4 and 11 were obtained by titration with thiosulphate. Results 5, 7, 8, 9 and 12 were obtained by titration of copper with iodine after a sulphide separation.

ZINC

Result No. 1 for zinc was obtained by analysis with Inductively Coupled Plasma Mass Spectrometry (ICP-MS); Results Nos. 2, 8 and 9 were obtained by ICP-OES (No. 2 fused with Na_2O_2, No. 8 used an acid digestion) whilst Nos. 3, 4, 5, 6 and 7 used FAAS (Nos. 3, 4, 6 and 7 used an acid digestion).

LEAD

Results Nos. 1, 2, 6 and 7 for lead were obtained by FAAS following an acid digestion, Nos. 3, 4, 8 and 9 by ICP-OES (No. 4 fused with Na_2O_2 and No. 8 used an acid digestion) and No. 5 by ICP-MS.

SULPHUR

With the exception of Nos. 1 and 9, all results for sulphur were obtained gravimetrically as $BaSO_4$. Result No. 1 was obtained using ICP-OES and No. 9 by combustion/infrared.

IRON

All results for iron, except for Nos. 7 and 9, were obtained by titration with Cr(VI) after reducing the iron. Result No. 5 was obtained by using Al to reduce Fe(III) whilst the other results were obtained using Sn(II) as the reducing agent. Result No. 7 was obtained by ICP-OES after fusion and No. 9 by FAAS after an acid digestion.

SILVER

Results Nos. 1, 3, 8, 9, 10, 11, 12 and 13 for silver were obtained using FAAS (1, 9 and 10 after acid digestion) whilst Results Nos. 2, 4, 5 and 6 were obtained by ICP-OES (No. 4 by fusion of the sample with Na_2O_2). Result No. 7 was obtained by the classical fire assay method.

GOLD

All results for gold, except for Nos. 8 and 11, were obtained using the classical fire assay method. Result No. 8 was obtained by following the method in ISO 10778, and No. 11 by ICP-OES.

CADMIUM

Results Nos. 1, 2, 3, 6, and 8 for cadmium were obtained by FAAS; No. 2 following acid digestion. The remaining results were obtained by ICP-OES.

MANGANESE

All results for manganese, apart from Nos. 2, 5 and 7 were obtained by FAAS (Nos. 4, 6, 8 and 9 used an acid digestion). Results Nos. 2, 5 and 7 were obtained by ICP-OES (No. 5 after fusion of the sample with Na_2O_2 and No. 7 using an acid digestion).

NICKEL

Results Nos. 1, 2, 3 and 6 for nickel were determined by ICP-OES (Nos. 1 and 6 used an acid digestion and No. 3 fused with Na_2O_2). The remaining results were determined by FAAS (Result 5 was obtained after an acid digestion).

ALUMINIUM OXIDE

Aluminium oxide was determined by either ICP-OES (Nos. 1, 2, 4, 6, 8 and 9) or FAAS (the remaining results).

SODIUM OXIDE

With the exception of result Nos. 8 and 10, where ICP-OES, was used, all results for sodium oxide were obtained using FAAS.

POTASSIUM OXIDE

With the exception of results Nos. 4, 5 and 8, where ICP-OES was used, all results for potassium oxide were obtained using FAAS.

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NOTES ON METHODS USED

ARSENIC

Results Nos. 1, 3, 4 and 6 for arsenic were obtained by ICP-OES. The remaining results were obtained by FAAS.

BISMUTH

Results Nos. 1, 4, 6 and 7 for bismuth were obtained by FAAS; Result No.2 by ICP-MS and Nos. 3, 5 and 8 by ICP-OES.

CARBON

Result No. 1 for carbon was obtained gravimetrically after combustion in oxygen; the remaining results were obtained by combustion/infrared techniques.

MOLYBDENUM

Results 5, 6, 8, 9, 11 and 13 for molybdenum were obtained using FAAS. The remaining results were obtained by ICP-OES.

CHLORIDE

Result No. 1 for chloride was obtained photometrically according to ASTM D512; Nos. 2 and 5 were obtained by potentiometric titration. No. 3 was obtained by a photometric method with ferrithiocyanate and No. 4 by a combustion and microcoulometric titration.

COBALT

Results Nos. 1, 2,4 and 8 for Cobalt were obtained using ICP-OES. The other results were obtained by FAAS.

CHROMIUM

Results Nos. 1, 2, 5 and 6 were obtained by ICP-OES, Nos. 3, 4, 7 and 8 by FAAS.

FLUORIDE

All results for fluoride were obtained using ion selective electrodes, No. 2 after distillation and Nos. 4, 6 and 7 after an alkaline fusion and subsequent separation of the hydroxides.

MERCURY

Result No. 1 for mercury was obtained by cold-vapour Atomic Absorption Spectrometry. All other results, apart from No.5, were obtained using ICP-OES. No. 5 was obtained using a flow injection mercury system.

INDIUM

Result No. 1 for indium was obtained by ICP-MS. Results 2 and 5 were from FAAS methods and Results 3,4, 6 and 7 from ICP-OES analyses.

PHOSPHORUS

Results Nos. 1, 2, 3, 5 and 6 for phosphorus were obtained by ICP-OES. Result No. 4 was from a photometric method in which the phosphovanadomolybdate complex was extracted into an organic solvent. Result No. 7was also from the phosphovanadomolybdate photometric method, this time without extraction.

ANTIMONY

Results Nos. 1, 2, 3, 4, and 5 for antimony were obtained by ICP-OES; Nos. 6, 7 and 8 by FAAS.

SELENIUM

Results Nos. 1, 3 and 6 for selenium were obtained by FAAS; Results Nos. 2 and 4 by ICP-OES and No. 5 by a photometric method using 3,3'-diamino-benzydene.

TELLURIUM

Result No. 1 for tellurium was obtained by hydride generation atomic absorption spectrometry; No. 2 by ICP-OES and the remaining results by FAAS.

TITANIUM

All results for titanium were obtained by ICP-OES apart from No. 7 which was obtained by FAAS.

BARIUM

All of the results for barium were obtained by ICP-OES.

BERYLLIUM

All of the results for beryllium were obtained by ICP-OES.

GERMANIUM

All three results for germanium were obtained by ICP-OES.

TIN

Results Nos. 1, 2, 4 and 7 for tin were obtained by ICP-OES, Result No. 3 by ICP-MS and Nos. 5 and 6 by FAAS.

THALLIUM

Results Nos. 1, 2 and 3 for thallium were obtained by ICP-OES. No. 4 was obtained using a photometric method.

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CO-OPERATING ANALYSTS

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Pattinson & Stead (2005) Ltd, Middlesbrough, UK.
Raghavendra Spectro Metallurgical Laboratory, Bangalore, India.
Standard & Reference Laboratories, Malaga, Western Australia, Australia.

DESCRIPTION OF SAMPLE

BCS-CRM 514 is a copper concentrate available in the form of a powder passing a nominal 100 µm aperture. It is supplied in bottles containing 100 g which have been purged with nitrogen.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd (BAS).

BAS is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS-CRM 514 as defined in ISO Guide 34:2009 section 3.1, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006.

INTENDED USE

This sample is intended for uses such as the verification of the accuracy and repeatability of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments, for establishing values for secondary reference materials and for training purposes.

In order to ensure that a fully representative sample is taken users should take a minimum sub-sample size of 1.0g. Users of this material should be aware that the use of a smaller sub-sample size will invalidate the certified values and the associated 95% confidence limits.

BCS-CRM 514 should be used without drying. The contents of the bottle should be thoroughly mixed before a sample is taken and should be exposed to the air for the shortest possible time. Unused material should be stored under an inert gas atmosphere, for example in a desiccator.

STABILITY

BCS-CRM 514 will remain stable only provided that the bottle remains sealed and is stored under an inert gas atmosphere. When the bottle has been opened the lid should be secured immediately after use. Even so the values given above are those obtained at the time of certification and BAS is not responsible for changes occurring once the bottle has been opened.

BAS will undertake to monitor the stability of the remaining stock material regularly.

TRACEABILITY

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above.

Most of the analytical methods used in the characterisation of this CRM were either international or national standard methods or methods which are technically equivalent. All laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 514 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's t value at the 95% confidence level for the number of results. Homogeneity has been assessed on the bulk material and has been found to be acceptable. It has not, therefore, been included in the calculated measurement uncertainty. The stability of this CRM and its transportation also make negligible contributions to the overall uncertainty of the certified values.

COMMUTABILTY

BCS-CRM 514 is intended to be used in the same physical form as that used by the participating analysts and therefore commutability is not of relevance in respect of this CRM.

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd may be obtained from the address below.

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