BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

UKAS REFERENCE MATERIALS 4004

CERTIFICATE OF ANALYSIS

BCS-CRM No. 520

ZINC CONCENTRATE

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, CHINA, HUNGARY, INDIA and The NETHERLANDS, issued by the Bureau of Analysed Samples Ltd.

ANALYSES

Mean of 4 values - mass content in %.

Analyst	SiO_2	Al_2O_3	MgO	CaO	Cu	Zn	Pb	S	Fe	Ag	As	Cd
1	1.6950	0.3307	0.2875	0.1278	0.5702	52.5575	0.8223	31.5975	9.7625	0.0036	0.0228	0.0720
2	1.7990	0.3503	0.2496	0.1194	0.5543	52.5075	0.7991	31.9595	9.9869	0.0039	0.0214	0.0747
3	1.7583	0.3130	0.2620	0.1226	0.5710	52.5533	0.8128	31.8148	9.8618	0.0035	0.0231	0.0722
4	…	0.3490	0.2630	0.1370	0.5830	52.6000	0.8218	31.8400	9.9600	0.0036	…	0.0719
5	1.8518	0.3383	0.2583	0.1180	0.5457	52.2025	0.8213	31.6050	9.9100	0.0036	…	0.0699
6	1.7285	0.3395	0.2708	0.1160	0.5815	52.5100	0.8238	…	9.8300	0.0034	0.0230	0.0745
7	1.7083	...	0.2870	0.1348	0.5455	52.5623	0.8133	…	9.7755	…	0.0247	0.0695
8	1.8450	0.3175	0.2675	…	0.5625	52.4125	0.8500	32.0775	9.9400	0.0037	0.0248	0.0773
9	1.7996	0.3411	0.2552	0.1316	0.5353	52.1215	0.8063	31.5196	9.7917	0.0040	0.0216	0.0706
10	1.7958	0.3370	0.2426	0.1128	0.5851	52.8326	0.8321	31.8160	9.9493	0.0035	0.0201	0.0770
11	1.8000	0.3283	0.2875	0.1278	0.5928	52.6600	0.8225	31.8300	9.9580	0.0035	0.0223	0.0740
12	…	…	…	…	0.5940	…	0.8150	…	…	0.0038	0.0240	0.0780
13	…	…	…	…	…	…	…	…	…	…	…	0.0736
M_M	**1.7781**	**0.3345**	**0.2665**	**0.1248**	**0.5684**	**52.5018**	**0.8200**	**31.7844**	**9.8842**	**0.0036**	**0.0228**	**0.0735**
s_M	0.0540	0.0123	0.0156	0.0083	0.0198	0.1989	0.0129	0.1806	0.0827	0.0002	0.0015	0.0028
s_w	0.0253	0.0055	0.0047	0.0029	0.0126	0.0880	0.0057	0.0706	0.0496	0.0001	0.0002	0.0014

M_M: Mean of the laboratory mean values. **s_M**: standard deviation of the laboratory mean values.. **s_W**: average within laboratory standard deviation.

CERTIFIED VALUES (Cv)

mass content in %

	SiO_2	Al_2O_3	MgO	CaO	Cu	Zn
Cv	**1.78**	**0.334**	**0.266**	**0.125**	**0.568**	**52.50**
C(95%)	0.04	0.009	0.011	0.006	0.013	0.14
Minimum weight (g)	0.25	0.2	0.2	0.2	0.2	0.2

	Pb	S	Fe	Ag	As	Cd
Cv	**0.820**	**31.78**	**9.88**	**0.0036**	**0.0228**	**0.0735**
C(95%)	0.009	0.14	0.06	0.0002	0.0011	0.0017
Minimum weight (g)	0.2	0.2	0.25	0.2	0.2	0.2

The half width confidence interval C(95%) is an expression of the uncertainty of the certified value, where C(95%) = $\frac{t \times s_M}{\sqrt{n}}$ and "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

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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BCS-CRM No. 520
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NOTES ON METHODS USED

SILICON DIOXIDE

Analysts Nos. 1, 6 and 7 determined silicon dioxide by X-Ray Fluorescence Spectroscopy (XRF). Nos. 2, 3, 8 and 11 used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES), No. 2 after dissolution with $HCl/HNO_3/HF$ and No 3 after alkali fusion. Analysts Nos. 5, 9 and 10 used a gravimetric method, dehydrating with perchloric acid.

ALUMINIUM OXIDE

With the exception of Analyst No. 10, who used Flame Atomic Absorption Spectrometry (FAAS), all of the Analysts determined aluminium oxide using ICP-OES, Nos. 1, 3 and 6 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$.

MAGNESIUM OXIDE

Apart from Analyst No. 7, who used XRF, all of the Analysts determined magnesium oxide by ICP-OES, Nos. 1, 3 and 6 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$.

CALCIUM OXIDE

Analysts Nos. 6 and 7 determined calcium oxide using XRF whilst Nos. 9 and 10 used FAAS. The remaining Analysts used ICP-OES, No. 1 and 3 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$.

COPPER

Most of the Analysts determined copper by ICP-OES, No. 1 and 3 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$. Nos. 6 and 7 used XRF, Nos. 9 and 10 used FAAS and No. 12 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

ZINC

Analysts Nos. 1, 2, 6, 7 and 8 used XRF to determine zinc. No. 5 used ICP-OES whilst the remaining Analysts used a complexometric titration with a visual end point, No. 4 following the Indian Bureau of Mines method and Nos. 9 and 10 BS ISO 12739:2006.

LEAD

Analysts Nos. 6 and 7 determined lead using XRF. Analysts Nos. 9 and 10 used FAAS whilst No. 12 used ICP-MS. The remaining Analysts used ICP-OES, Nos. 1, 3 and 8 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$.

SULPHUR

Analyst Nos. 1, 2 and 3 used a combustion-infrared technique, calibrated with pure chemicals, to determine sulphur. Analyst No. 5 used a combustion technique, followed by acid/base titration whilst the other Analysts used a gravimetric technique, No.4 following the Indian Bureau of Mines method.

IRON

Analysts Nos. 1, 6 and 7 determined iron using XRF. Nos. 5, 9 and 10 titrated with Cr(VII), No. 5 after reduction with Ti(III) and Nos. 9 and 10 after reduction with Sn(II). The other Analysts all used ICP-OES, No. 2 after dissolution with $HCl/HNO_3/HF$.

SILVER

Most Analysts used ICP-OES to determine silver, No. 1 after acid digestion, but Nos. 3, 6, 8 and 9 used FAAS, Nos. 6 and 8 after acid digestion. No. 12 used ICP-MS.

ARSENIC

Except for Analysts Nos. 9 and 10, who used a photometric method with silver diethyldithiocarbamate, and No. 12, who used ICP-MS, all of the Analysts determined arsenic by ICP-OES, Nos. 1, 3, 6 and 8 after acid digestion and No. 2 after dissolution with $HCl/HNO_3/HF$.

CADMIUM

Analyst No. 7 determined cadmium by XRF. Apart from Nos, 9 and 10, who used FAAS, and No. 12, who used ICP-MS, all of the other Analysts used ICP-OES, Nos. 1, 3, 6 and 8 after acid digestion.

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CO-OPERATING ANALYSTS

1	BLUNDELL, P.,	ALS Inspection UK, Prescot.
2	HURDITCH, P.,	AMG Superalloys UK. Ltd., Rotherham.
3	JIANG, J.,	Inspectorate (Shanghai) Ltd., Shanghai, China.
4	CHATTOPADHYAY, S.,	Inspectorate Griffith India Pvt Ltd., Bhubaneswar, India.
5	KONDOROSI, G.,	Dunaferr Labor NonProfit Ltd., Dunaujvaros, Hungary.
6	DERUITER, G.,	A H Knight International Ltd., St Helens.
7 & 13	CULLEY, N.,	LMA cv, Sleeuwijk, The Netherlands.
8	CHRISTEN, M.,	Laboratory Services International Rotterdam BV, Rotterdam, The Netherlands.
9	CROCKER, F.H. & ATKINSON, M.J.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
10	JONES, S.J., *BSc, CChem, MRSC,*	Ridsdale & Co. Ltd., Middlesbrough.
11	ROCHE, T. *CSi, CChem, FRSC, Eurchem,*	Alex Stewart International, Liverpool.
12	COLLINS, P.,	TATA Steel – Technical, Rotherham.

DESCRIPTION OF SAMPLE

BCS-CRM 520 is sold in the form of finely divided material passing a nominal 150 micron aperture and packed into bottles of 100g.

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

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Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS-CRM 520 as defined in BS EN ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with BS EN ISO 17034 and ISO Guide 35.

INTENDED USE

This sample is intended for the verification 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 not less than the minimum weight stated on the certificate for that element, this being the lowest sample mass used by any of the Co-operating Analysts contributing to the certified value. Users of this material should be aware that the use of a smaller sub-sample size may invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use and should be used as received, i.e. without drying.

STABILITY

BCS-CRM 520 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. Once the bottle has been opened the lid should be secured immediately after use.

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 520 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 representative samples taken from the batch and 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 520 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:

For BUREAU OF ANALYSED SAMPLES LTD.
NEWHAM HALL
NEWBY
MIDDLESBROUGH
ENGLAND
TS8 9EA
Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk
R P MEERES,
Managing Director March 2019