



BUREAU OF ANALYSED SAMPLES LTD

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4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 308/1

CHROME ORE

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN
 AND PERU,
 issued by the Bureau of Analysed Samples Ltd.

CO-OPERATING ANALYSTS

1	HARGREAVES, C.,	Metals & Minerals Division, Inspectorate International, Witham.
2	BLUNDELL, P. & DOUGLAS, I.,	ALS Inspection UK Ltd., Prescot.
3	CROCKER, F.H.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
4	SHAW, D.,	Ceram Research Ltd, Stoke-on-Trent.
5	JONES, S.J., BSc, CChem, MRSC,	Ridsdale & Co. Ltd., Middlesbrough.
6	HAMPSON, N.,	Alfred Knight International Ltd., St. Helens.
7	LOPEZ, N.,	A H Knight del Peru Ltda, Lima, Peru.

ANALYSES

Mean of 4 values - mass content in %.

All results relate to the dried (105°C) sample.

Analyst	FeO*	SiO ₂	Al ₂ O ₃	Cr ₂ O ₃	TiO ₂	CaO	MgO	MnO	P	Na ₂ O	K ₂ O
1	26.68	...	15.03	44.86	0.751	0.72	8.97	...	<i>0.015</i>	<i>0.019</i>	<i>0.0078</i>
2	26.40	1.193	15.27	45.07	...	0.60	8.99	0.233	<i>0.003</i>	<i>0.014</i>	<i>0.0060</i>
3	26.42	1.213	15.07	44.96	0.747	0.63	9.33	0.221	<i>0.001</i>	<i>0.023</i>	<i>0.0031</i>
4	26.72	1.180	15.07	45.19	0.745	0.65	9.13	0.221
5	26.44	1.164	15.01	44.81	0.738	0.65	9.24	0.240	<i><0.006</i>	<i>0.016</i>	<i>0.0051</i>
6	26.81	...	15.23	44.73	...	0.66	9.23	0.242	<i>0.007</i>
7	...	1.218	14.99	44.77	0.728	0.66	9.16	0.225	...	<i><0.020</i>	<i><0.005</i>
M_M	26.58	1.194	15.10	44.91	0.742	0.65	9.15	0.230			
s _M	0.18	0.023	0.11	0.17	0.010	0.04	0.14	0.010			
s _w	0.06	0.026	0.06	0.07	0.021	0.02	0.03	0.006			

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

Values given in italics are for information only

Additional Information: Analyst No.4 determined Cr(VI) using BSEN 14946 and found a value of 0.0006%.

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

*Total Iron expressed as FeO

CERTIFIED VALUES (C_v)

mass content in %

	FeO	SiO ₂	Al ₂ O ₃	Cr ₂ O ₃	TiO ₂	CaO	MgO	MnO
C_v	26.58	1.194	15.10	44.91	0.74	0.65	9.15	0.230
C(95%)	0.19	0.029	0.11	0.16	0.02	0.04	0.13	0.010

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 (C_v) by 10⁴ will yield the value in µg/g.

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NOTES ON METHODS USED

IRON (II) OXIDE

All Analysts determined total iron and reported as FeO. With the exception of Analyst No. 4, all Analysts determined FeO by titration of Fe(II) with $K_2Cr_2O_7$, Nos. 1, 3, 5 and 6 reducing Fe(III) to Fe(II) with Sn(II). Analyst No. 4 used X-Ray Fluorescence Spectrometry (XRF) with a fused bead following the standard method BS EN ISO 12677.

SILICA

Analysts Nos. 2, 3, 5 and 7 determined silica gravimetrically. Analysts Nos. 3 and 5 dehydrated with perchloric acid following a peroxide fusion, and No. 7 dehydrated with sulphuric acid, also following a fusion with Na_2O_2 . Analyst No 4 determined silica by XRF using a fused bead, following the standard method BS EN ISO 12677.

ALUMINA

Analysts Nos. 1, 2, 3 and 5 determined alumina by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). Analyst No. 1 used a peroxide fusion whilst No. 2 used an acid digestion. Analysts Nos. 4 and 6 used XRF using a fused bead, No. 4 following the standard method BS EN ISO 12677. Analyst No. 7 used Flame Atomic Absorption Spectrometry (FAAS) following a fusion with Na_2O_2 .

CHROMIUM (III) OXIDE

All Analysts determined total chromium and reported as chromium (III) oxide. Analyst No 1 oxidised chromium with ammonium persulphate; Analyst Nos. 2 and 3 fused the sample with Na_2O_2 , oxidised chromium, added an excess of Fe(II) and back titrated the excess with $K_2Cr_2O_7$, No. 2 using a potentiometric titration. Analyst No. 4 used XRF using a fused bead following the standard method BS EN ISO 12677. Analyst No. 5 fused the sample with peroxide and titrated the oxidised chromium directly with Fe(II). Analyst No. 6 titrated with $K_2Cr_2O_7$, and Analyst No. 7 determined chromium titrimetrically after a peroxide fusion and treatment with perchloric acid.

TITANIUM DIOXIDE

Analysts Nos.1 and 4 determined titanium dioxide by XRF using a fused bead, No. 4 following the standard method BS EN ISO 12678. Analysts Nos. 3 and 5 used ICP-OES. Analyst No. 7 used FAAS after fusion with sodium peroxide.

CALCIUM OXIDE

Analysts Nos.1, 4 and 6 determined calcium oxide by XRF using a fused bead, No. 4 following the standard method BS EN ISO 12679. Analysts Nos. 2, 3 and 5 used ICP-OES, No. 2 following an acid digestion. Analyst No. 7 used FAAS after digestion with hydrochloric, nitric, perchloric and hydrofluoric acids.

MAGNESIUM OXIDE

Analysts Nos.1, 4 and 6 determined magnesium oxide by XRF using a fused bead, No. 4 following the standard method BS EN ISO 12680. Analysts Nos. 2, 3 and 5 used ICP-OES, No. 2 following an acid digestion. Analyst No. 7 used FAAS after fusion with sodium peroxide.

MANGANESE OXIDE

Analysts Nos. 2, 3 and 5 determined manganese oxide by ICP-OES, No. 2 after an acid digestion. Analyst No. 4 used XRF following the standard method BS EN ISO 12677. Analysts Nos. 6 and 7 used FAAS after fusion with sodium peroxide.

PHOSPHORUS

All Analysts, apart from No.3, determined phosphorus by ICP-OES, No. 2 after acid digestion and No. 6 after fusion. Analyst No. 3 determined phosphorus photometrically after extraction of the phosphovanadomolybdate complex.

SODIUM OXIDE

All Analysts, apart from No. 2, determined sodium oxide by FAAS, Analyst No. 1 using an acid digestion. Analyst No.2 used ICP-OES following an acid digestion.

POTASSIUM OXIDE

All Analysts, apart from No. 2, determined potassium oxide by FAAS, Analyst No. 1 using an acid digestion. Analyst No.2 used ICP-OES following an acid digestion.

DESCRIPTION OF SAMPLE

BCS-CRM 308/1 is sold in the form of a powder passing a nominal 250 micron aperture sieve. It is supplied in bottles containing 100g.

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

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 308/1 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

BCS-CRM 308/1 is intended for 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.

The sample should be mixed thoroughly before each use.

STABILITY

The sample will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. When the bottle has been opened the lid should be secured immediately after use.

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 308/1 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 of the finely divided material has been assessed on the bulk material and has been found to be acceptable. Homogeneity 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.

COMMUTABILITY

BCS-CRM 308/1 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.

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