MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

SGT DOLOMITE 1

BCS-CRM No. 512 DOLOMITE

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN AND GREECE, issued by the Bureau of Analysed Samples Ltd and the Society of Glass Technology

ANALYSES

Mean of 4 values - mass content in %. All results relate to the dried (105°C) sample.

Analyst No.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	SrO	LOI
1	0.3615	0.0425	...	0.0335	...	30.6920	21.5848	…	47.1025
2	…	…	…	…	0.0025	30.6400	21.6700	…	46.8600
3	...	...	…	…	…	30.8768	21.6985	…	46.6400
4	0.3568	…	0.0026	0.0257	0.0045	30.2160	21.9175	…	46.6875
5	0.3975	0.0500	…	...	0.0042	30.6300	21.3300	0.0200	46.8530
6	0.4076	0.0494	0.0024	0.0331	0.0036	30.7984	21.2432	0.0251	46.7124
7	…	…	…	…	…	…	…	0.0217	…
8	0.3300	0.0483	…	…	…	30.1275	21.3250	…	...
9	…	…	…	0.0265	…	…	…	…	46.9230
10	0.3978	0.0553	…	0.0280	0.0042	…	…	…	46.8438
11	0.3625	0.0650	0.0016	0.0268	0.0030	30.8750	21.4975	0.0200	47.0525
12	0.4025	0.0600	0.0016	0.0300	0.0030	30.7375	21.8475	…	46.8800
13	0.3875	0.0680	0.0020	0.0378	0.0035	30.4750	21.6500	...	46.5500
14	0.3900	...	…	0.0300	0.0040	30.6500	21.7000	0.0310	46.5000
M_M	**0.3794**	**0.0548**	**0.0020**	**0.0302**	**0.0036**	**30.6107**	**21.5876**	**0.0236**	**46.8004**
s_M	0.0253	0.0089	0.0005	0.0040	0.0007	0.2468	0.2177	0.0047	0.1867
s_w	0.0219	0.0032	0.0001	0.0021	0.0004	0.0779	0.0727	0.0002	0.0986

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

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	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	SrO	LOI
Cv	**0.379**	**0.055**	**0.0020**	**0.030**	**0.0036**	**30.61**	**21.59**	**0.024**	**46.80**
C(95%)	0.018	0.008	0.0006	0.003	0.0006	0.17	0.15	0.006	0.12

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

Additional Information (mass content in %)

Analyst No	Cr_2O_3	P_2O_5	*Pb*	*S*	*BaO*	Na_2O	K_2O	*Zn*	*As*	*Cd*	*Ni*	CO_2	*Total C*	*F*
1	<0.001	<0.002	…	…	<0.001	0.1460	0.0103	…	…	…	…	…	…	…
2	…	0.0020	…	…	…	<0.02	<0.02	…	…	…	…	…	…	…
3	0.0010	<0.01	…	…	…	<0.30	<0.01	…	…	…	…	…	…	…
4	…	…	…	…	…	…	0.0082	…	…	…	…	…	…	…
5	<0.01	<0.02	0.0006	<0.05	<0.01	0.0713	0.0011	<0.01	…	…	…	…	12.4000	…
6	0.0011	0.0036	<0.0010	0.0036	0.0097	0.0614	0.0042	0.0006	<0.003	<0.0010	<0.0010	47.0981	12.9355	…
7	…	…	<0.0005	<0.005	<0.0012	…	…	<0.0003	…	…	…	…	…	…
10	…	…	0.0001	0.0005	…	0.1228	…	…	…	…	…	…	11.9250	0.0123
11	0.0004	0.0032	0.0004	0.0033	0.0200	0.1075	…	…	…	…	…	…	…	0.0074
12	<0.001	0.0040	<0.0001	0.0015	<0.001	0.0700	0.0070	0.0004	…	…	0.0010	…	…	…
13	<0.001	…	0.0001	0.0085	…	0.0700	0.0083	<0.001	<0.0001	<0.0001	<0.001	46.2275	…	0.0025
14	…	…	<0.0001	…	<0.001	…	…	0.0060	<0.0001	<0.0003	<0.001	…	…	…

Analyst No. 7 also provided the following information (µg/g): Y <2, Zr 5, Sc 8, Co 2, and Cu <3, all determined by XRF

Analyst No.10 determined Chloride by potentiometric titration, and found 0.0048%

Analyst No. 14 determined Hg and Rb by XRF, and found <0.0002% and <0.0001% respectively

SGT DOLOMITE 1/BCS – CRM 512 DOLOMITE

NOTES ON METHODS USED

SILICA

Analysts Nos. 1, 5, 8, 11, 12 and 14 determined silica using X-ray Fluorescence Spectrometry (XRF). Analyst No. 4 determined the bulk of the silica gravimetrically after separation by coagulation and filtration, and the residual silica photometrically as molybdenum blue. No. 6 used Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Analyst No. 10 used Flame Atomic Absorption Spectrometry (FAAS) whilst No. 13 used Direct Coupled Plasma Optical Emission Spectrometry (DCP-OES).

ALUMINA

Analysts Nos. 1, 5, 8, 11, and 12 determined alumina using XRF. Analyst No. 6 used ICP-OES, No. 10 used FAAS and No.13 used DCP-OES.

TITANIA

Analyst No. 4 used a photometric method with 1,2-dihydroxybenzene 3,5-disulphonic acid disodium salt according to BS 2975 (1988). Analysts Nos. 6 and 12 used ICP-OES; No.11 determined titania using a diantipyrylmethane photometric method and No. 13 used DCP-OES.

FERRIC OXIDE

Analysts Nos. 1, 11, 12 and 14 analysed ferric oxide by XRF. Analysts Nos. 4 and 9 used a 1-10 phenanthroline photometric method, No 6 used ICP-OES; No. 10 used FAAS and No. 13 used DCP-OES. No. 10 also used XRF and found 0.028% and No. 11 also used FAAS and found 0.028%.

MANGANESE OXIDE

Analysts Nos. 2, 5, 12 and 14 determined manganese oxide by XRF. Analysts Nos. 4, 10 and 11 used FAAS, No.6 used ICP-OES and No. 13 used DCP-OES.

CALCIUM OXIDE

All Analysts except Nos. 4, 6, 8 and 13 determined calcium oxide by XRF. Analysts Nos. 4 and 6 titrated with 1,2-di(2-aminoethoxy)ethane-NNN′N′-tetra-acetic acid (EGTA), whilst No. 13 titrated with ethylenediaminetetra-acetic acid (EDTA).

MAGNESIUM OXIDE

All Analysts except Nos. 4, 6, 8 and 13, determined magnesia by XRF. Analyst No. 4 titrated with trans -1,2-diaminocyclohexane – NNN′N′ -tetra-acetic acid (DCTA), No. 6 used ICP-OES and No. 13 titrated with EDTA.

STRONTIUM OXIDE

All Analysts except No. 6 determined strontium oxide by XRF. Analyst No. 6 used FAAS.

LOSS ON IGNITION

All Analysts determined the loss on ignition gravimetrically by heating at 1025° ± 25° C to constant weight.

CHROMIUM OXIDE

Analysts Nos. 1, 3, 5 and 12 determined chromium oxide by XRF. Analyst No. 6 used ICP-OES, No. 11 used FAAS and No.13 used DCP-OES.

PHOSPHORUS PENTOXIDE

All Analysts except Nos. 6 and 11 determined phosphorus pentoxide by XRF. Analyst No. 6 used ICP-OES and No. 11 extracted the molybdenum blue complex and determined photometrically.

LEAD

Analysts Nos. 5, 7 and 14 determined lead using XRF. Analysts Nos. 6 and 12 used ICP-OES, Nos. 10 and 11 used FAAS and No. 13 used polarography.

SULPHUR

Analysts Nos. 5, 7, 11 and 12 determined sulphur using XRF. Analyst No 6 used a gravimetric method with barium sulphate whilst Analyst No. 10 used high frequency combustion and infrared absorption. Analyst No. 13 used the method in BS 6463.

BARIUM OXIDE

All Analysts except for No. 6 determined barium oxide by XRF. Analyst No. 6 used ICP-OES.

SODIUM OXIDE

All Analysts except Nos. 6, 10 and 13 determined sodium oxide using XRF. Analysts Nos. 6 and 10 used FAAS, whilst No. 13 used DCP-OES.

POTASSIUM OXIDE

All Analysts except Nos. 4, 6 and 13 determined potassium oxide using XRF. Analysts Nos. 4 and 6 used FAAS, and No. 13 used DCP-OES.

ZINC

Analysts Nos. 5, 7, 12 and 14 determined zinc by XRF. Analyst No. 6 used FAAS and No. 13 used DCP-OES.

ARSENIC

Analyst No. 6 determined arsenic photometrically with silver diethyldithiocarbamate after separation as arsine. Analyst No. 13 used the Gutzeit method and No. 14 used XRF.

CADMIUM

Analyst No. 6 determined cadmium by ICP. Analyst No. 13 used polarography and No. 14 used XRF.

NICKEL

Analyst No. 6 determined nickel by ICP. Analyst Nos. 12 and 14 used XRF and No. 13 used DCP-OES.

CARBON DIOXIDE

Analyst No. 6 determined carbon dioxide using the gravimetric method according to Bennett and Reed, Chemical Methods of Silicate Analysis (1971); and No. 13 used the evolution technique described in BS 6463.

TOTAL CARBON

Analysts Nos. 5 and 10 determined total carbon using high frequency combustion and infrared absorption. Analyst No. 6 determined gravimetrically as carbon dioxide.

FLUORINE

All Analysts determined fluorine using ion selective electrodes.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

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4	KAKLOPOULOS, B.,	Agricultural Research and Analytical Laboratories, Athens, Greece.
5	OLIVER, G. J., *BSc, PhD, CChem, MRSC*	CERAM Research Ltd., Stoke-on-Trent.
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7	POTTS, P., *BSc, PhD, DSc, CChem, FRSC*	Open University, Milton Keynes.

ANALYSTS representing MANUFACTURERS and USERS

8	CARRARD, M. Ms,	St. Gobain Glass UK Ltd., Eggborough.
9	FLOWER, M. Mrs.,	Glass Technology Services Ltd., Sheffield.
10	JACKSON, P.,	Buxton Lime Industries Ltd., Buxton.
11	JAMIESON, S., *MSc, CChem, MRSC*	Pilkington European Technology Centre Ltd., Lathom.
12	JONES, N. Mrs.,	WBB Minerals, Group Central Laboratory, Whiston.
13	MANSELL, J.,	Omya UK, North Ferriby.
14	WEEDON, N.,	Longcliffe Quarries Ltd, Brassington.

DESCRIPTION OF SAMPLE

BCS-CRM 512 is sold in the form of finely divided material passing a nominal 250 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.

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 512 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.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 512 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 512 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 512 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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