



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS SGT LIMESTONE 1 BCS-CRM No. 513 LIMESTONE

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 ₂ O ₃	Fe ₂ O ₃	Cr ₂ O ₃	MnO	CaO	MgO	Pb	S	K ₂ O	SrO	Zn	LOI
1	0.2573	0.1018	0.1730	0.0195	...	43.9855
2	0.0085	56.0900
3	0.0010	43.7275
4	0.2248	...	0.0262	...	0.0106	55.5475	0.1975	0.0164	43.4100
5	0.2250	0.1075	0.0298	0.0013	0.0090	55.6100	0.1800	0.0010	0.0083	0.0164	0.0147	...	43.5600
6	0.2515	0.1030	0.0332	0.0016	0.0102	55.4361	0.1935	...	0.0071	0.0132	0.0176	0.0013	43.5850
7	0.0010	0.0108	...	0.0150	0.0015	...
8	0.2425	0.1000	0.0228	55.1875	43.3650
9	0.0104
10	0.0109
11	0.0262	43.4168
12	0.2088	0.1043	0.0303	...	0.0092	55.4075	0.1813	0.0008	0.0093	43.9250
13	0.1925	0.1200	0.0273	0.0009	0.0084	55.6350	0.1925	0.0009	43.7500
14	0.2375	0.1075	0.0300	0.0010	0.0100	55.8197	0.1825	0.0153	...	0.0019	43.6025
15	0.1950	0.0950	0.0225	0.0013	0.0077	55.4875	0.1600	0.0007	0.0113	0.0138	0.0190	0.0014	43.3500
16	0.1977	0.1260	0.0109	55.5759
17	0.2800	0.1100	0.0263	...	0.0100	55.7000	0.1775	0.0008	0.0200	0.0010	43.6250
M_M	0.2284	0.1075	0.0275	0.0012	0.0095	55.5906	0.1820	0.0009	0.0097	0.0150	0.0176	0.0014	43.6085
s_M	0.0284	0.0093	0.0034	0.0003	0.0011	0.2343	0.0116	0.0001	0.0016	0.0015	0.0023	0.0003	0.2093
s_w	0.0080	0.0049	0.0010	0.0003	0.0004	0.0697	0.0062	0.0001	0.0005	0.0016	0.0009	0.0001	0.0729

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 ₂ O ₃	Fe ₂ O ₃	Cr ₂ O ₃	MnO	CaO	MgO	Pb	S	K ₂ O	SrO	Zn	LOI
Cv	0.228	0.108	0.0275	0.0012	0.0095	55.59	0.182	0.0009	0.0097	0.0150	0.0176	0.0014	43.61
C(95%)	0.019	0.007	0.0025	0.0003	0.0008	0.16	0.009	0.0001	0.0015	0.0019	0.0024	0.0004	0.13

The half width confidence interval $C(95\%) = \frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:1989 section 4.

Additional Information (mass content in %)

Analyst No	TiO ₂	P ₂ O ₅	BaO	Na ₂ O	As	Cd	Ni	CO ₂	Total C	F
1	0.0063	<0.01	0.0110	<0.01
2	<0.01	0.0015	...	<0.02
3	...	<0.01	...	<0.30
4	0.0050
5	0.0059	0.0043	0.0100	<0.03
6	0.0050	0.0047	0.0097	0.0024	<0.0001	<0.0010	<0.0010	43.7967	11.9949	...
7	0.0057
12	<0.001	43.6300	11.7750	0.0024
13	0.0033	0.0032	0.0100	<0.01	<0.005
14	0.0037	0.0085	<0.001	0.0475	<0.001
15	0.0090	0.0033	<0.0001	0.0002	0.0004	0.0016
16	0.0031
17	...	<0.001	0.0150	...	<0.0001	<0.0003	<0.0002

Analyst No. 7 also provided the following information (µg/g): Y 4, Zr 6, Sc 22, Co 3, and Cu 4, each determined by XRF

Analyst No. 13 determined chlorine by potentiometric titration and reported 0.0024%

Analyst No. 17 determined Hg and Rb by XRF, and found <0.0002% and <0.0001% respectively.

SGT LIMESTONE 1/BCS – CRM 513 LIMESTONE

NOTES ON METHODS USED

SILICA

All Analysts, with the exception of Nos. 4, 6, 12 and 15 determined silica using X-ray Fluorescence Spectrometry (XRF). Analyst No. 4 determined silica according to BS 3108:1980, No. 6 used Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and No. 12 used Flame Atomic Absorption Spectrometry (FAAS). Analyst No. 15 used Direct Coupled Plasma Optical Emission Spectrometry (DCP-OES).

ALUMINA

All Analysts except for Nos. 6, 12 and 15 determined alumina using XRF. Analyst No. 6 used ICP-OES, No. 12 used FAAS and No. 15 used DCP-OES.

FERRIC OXIDE

Analyst Nos. 4 and 11 determined ferric oxide using the 1-10 phenanthroline photometric method according to BS 3108:1980. Analysts Nos. 5, 8, 13, 14 and 17 used XRF, No. 6 used ICP-OES; No. 12 used FAAS and No. 15 used DCP-OES. Analyst No. 13 also determined ferric oxide by FAAS and found 0.025%.

CHROMIUM OXIDE

Analysts Nos. 3, 5 and 14 determined chromium oxide by XRF. Analyst No. 6 used ICP-OES, No. 13 used FAAS, and No. 15 used DCP-OES.

MANGANESE OXIDE

Analysts Nos. 2, 5, 14, 16 and 17 determined manganese oxide by XRF. Analysts Nos. 4, 12 and 13 used FAAS, No. 6 used ICP-OES and Analyst No. 15 used DCP-OES.

CALCIUM OXIDE

All Analysts except Nos. 4, 6, 8, 12 and 15 determined calcium oxide by XRF. Analyst No. 4 titrated with ethylenediaminetetraacetic acid (EDTA), according to BS 3108:1980 and No. 6 titrated with 1,2-di(2-aminoethoxy)ethane- NNN'N' -tetra-acetic acid, whilst Analysts Nos. 12 and 15 determined calcium oxide according to BS 6463.

MAGNESIUM OXIDE

All Analysts except Nos. 4, 6, 12 and 15, determined magnesia by XRF. Analyst No. 4 titrated with EDTA, according to BS 3108:1980; No. 6 used ICP-OES; No. 12 used FAAS and No. 15 used DCP.

LEAD

Analyst No. 5 determined lead using ICP. Analysts Nos. 7 and 17 used XRF; Nos. 12 and 13 used FAAS and No. 15 used polarography.

SULPHUR

All Analysts except Nos. 6 and 7 determined sulphur using high frequency combustion and infra-red absorption. Analyst No. 6 determined sulphur gravimetrically as barium sulphate and Analyst No. 7 used XRF.

POTASSIUM OXIDE

Analysts Nos. 4 and 6 determined potassium oxide using FAAS. Analysts Nos. 5 and 14 used XRF whilst No. 15 used DCP-OES.

STRONTIUM OXIDE

All Analysts except Nos. 6 and 15 determined strontium oxide by XRF. Analyst No. 6 used FAAS and No. 15 used DCP-OES.

ZINC

Analyst No. 6 determined zinc by FAAS, whilst Analysts Nos. 7, 14 and 17 used XRF. Analyst No. 15 used DCP-OES.

LOSS ON IGNITION

All Analysts determined the loss on ignition gravimetrically by heating at $1025^{\circ} \pm 25^{\circ} \text{C}$ to constant weight.

TITANIA

All Analysts except Nos. 4, 6, 13 and 14 determined titania by XRF. Analyst No. 4 used a photometric method with 1,2-dihydroxybenzene 3,5-disulphonic acid disodium salt according to BS 2975 (1988), Nos. 6 and 14 used ICP-OES whilst No. 13 determined titania photometrically with diantipyrylmethane.

PHOSPHORUS PENTOXIDE

All Analysts except Nos. 6 and 13 determined phosphorus pentoxide by XRF. Analyst No. 6 used ICP-OES and No. 13 extracted the molybdenum blue complex and determined photometrically.

BARIUM OXIDE

All Analysts except Nos. 6 and 15 determined barium oxide using XRF. Analyst No. 6 used ICP-OES and No. 15 used DCP-OES.

SODIUM OXIDE

All Analysts except Nos. 6 and 15 determined sodium oxide using XRF. Analyst No. 6 used FAAS and No. 15 used DCP-OES.

ARSENIC

Analyst No. 6 determined arsenic photometrically with silver diethyldithiocarbamate after separation as arsine. Analysts Nos. 12 and 15 used the Gutzeit method. Analyst No. 17 used XRF.

CADMIUM

Analyst No. 6 determined cadmium by ICP. Analyst No. 15 used polarography and No. 17 used XRF.

NICKEL

Analyst No. 6 determined nickel by ICP. Analysts Nos. 14 and 17 used XRF whilst No. 15 used DCP-OES.

CARBON DIOXIDE

Analyst No. 6 determined carbon dioxide gravimetrically according to Bennett and Reed, Chemical Methods of Silicate Analysis (1971), and No. 12 used a thermogravimetric technique.

TOTAL CARBON

Analyst No. 6 determined total carbon gravimetrically as carbon dioxide, and No. 12 used high frequency combustion infra-red absorption.

FLUORINE

All Analysts determined fluorine using ion selective electrodes.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

- | | | |
|---|------------------------------------------------|-------------------------------------------------------------------|
| 1 | BURTON, R., <i>MSc</i> , | Sheffield Hallam University, Sheffield. |
| 2 | CROUDACE, I., | Southampton Oceanography Centre, Southampton. |
| 3 | NEVE, L. Ms, <i>MSc, MRSC</i> , | University of Leeds, Leeds. |
| 4 | KAKLOPOULOS, B., | Agricultural Research and Analytical Laboratories, Athens, GREECE |
| 5 | OLIVER, G. J., <i>BSc, PhD, CChem, MRSC</i> , | CERAM Research Ltd., Stoke-on-Trent. |
| 6 | PAGE-GIBSON, J.E., <i>BSc, CChem, MRSC</i> , | Ridsdale & Co. Ltd., Middlesbrough, |
| 7 | POTTS, P., <i>BSc, PhD, DSc, CChem, FRSC</i> , | Open University, Milton Keynes. |

ANALYSTS representing MANUFACTURERS and USERS

- | | | |
|----|-----------------------------------------|-----------------------------------------------------|
| 8 | CARRARD, M. Ms, | St. Gobain Glass UK Ltd., Eggborough. |
| 9 | CROOK, D., <i>MRSC</i> , | Corus Llanwern |
| 10 | ALLEN, M., | Corus, Port Talbot |
| 11 | FLOWER, M., Mrs., | Glass Technology Services Ltd., Sheffield. |
| 12 | JACKSON, P., | Buxton Lime Industries Ltd., Buxton. |
| 13 | JAMIESON, S., <i>MSc, CChem, MRSC</i> , | Pilkington European Technology Centre Ltd., Lathom. |
| 14 | JONES, N., Mrs., | WBB Minerals, Group Central Laboratory, Whiston. |
| 15 | MANSELL, J., | Omya UK, North Ferriby. |
| 16 | MARRIOTT N. J, <i>MEng, PhD</i> , | Calumite Ltd, Scunthorpe. |
| 17 | WEEDON, N., | Longcliffe Quarries Ltd, Brassington. |

DESCRIPTION OF SAMPLE

Bottles of 100g of finely divided material for chemical analysis

INTENDED USE & STABILITY

This sample is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

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

The sample should be dried at a temperature of 105°C for two hours before use.

TRACEABILITY

The traceability of this CRM is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

This Certified Reference Material has been prepared in accordance with the recommendations specified in ISO Guides 30 to 35, available from the International Standards Organisation in Geneva.

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