



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS BCS-CRM No. 528 STANDARD GLASS SAND SGT STANDARD GLASS SAND 11

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, BELGIUM, THE CZECH REPUBLIC, FRANCE, GERMANY, ITALY, JAPAN, THE NETHERLANDS, SWEDEN, THAILAND, TURKEY AND THE UNITED STATES OF AMERICA, 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	TiO ₂	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	BaO	Cr ₂ O ₃	PbO	LOI	Mn	Sn	P ₂ O ₅	ZrO ₂
1	95.6150	2.4223	0.0480	0.1088	0.2384	0.0886	0.0898	0.8597	0.0278	...	0.0006	0.2463
2	95.6270	2.4738	0.0485	0.1205	0.2403	0.0860	0.1010	0.8630	0.0305	0.3020
3	95.5924	2.4494	0.0505	...	0.2111	0.0796	...	0.8959	0.2408
4	95.7475	2.4293	0.0459	0.1080	0.2615	...	0.0936	0.8860	0.2988
5	95.6140	2.4613	0.0485	...	0.2388	0.0815	0.1040	0.8760	0.0283	0.0010	0.0020	0.0010	0.1620	0.0165
6	95.5042	2.3630	0.0493	0.1111	0.2297	0.0899	0.1000	0.8719	0.0275	0.0011	...	0.2750
7	...	2.4625	0.0520	0.1013	0.2375	0.0848	0.1050	0.8850	0.0308	0.0009	...	0.2575	0.20	0.017
8	95.8150	2.3510	0.0466	0.1073	0.2487	0.0953	0.1043	0.8853	0.0306	0.0009	0.0006	0.2875	0.0012	0.0021
9	95.6975	0.1133	...	0.0890	0.1185	0.9000	...	0.0005	...	0.3138
10	...	2.4730	0.0465	0.1015	...	0.0876	0.0940	0.8675	0.0281	0.0009	...	0.2083
11	0.0475	0.1083	0.2205	0.0938	0.1133	...	0.0280	0.0011	0.20	0.017
12	95.6755	2.4398	0.0490	0.1178	0.2480	0.0885	...	0.8828	0.0316	0.0006	0.0005	0.2475	0.1640	...
13	95.8450	2.5455	0.0503	0.1151	0.2384	0.0874	0.1027	0.8389	0.0316	...	0.0005	0.2640
14	95.3863	2.4087	0.0442	0.1082	0.2427	0.0901	0.0908	0.8377	...	0.0009
15	95.5950	...	0.0525	0.1077	0.2200	0.8925	0.2920	0.0027	...	0.2953	0.0074
16	...	2.4800	0.0488	0.1155	...	0.0890	0.1038	0.2675	<0.001
17	95.7710	2.4438	0.0471	0.1102	0.2380	0.0983	0.1083	0.8804	0.0315	0.0010	...	0.2691
18	...	2.4593	0.0512	0.1183	0.2427	0.0905	0.0955	0.8938	0.0318	0.0005	0.0006
19	95.3950	2.4235	0.0480	0.1107	0.2331	0.0867	0.0962	0.8716	0.0296	0.0007	...	0.2750
20	95.4250	2.5198	0.0485	0.1163	0.2450	0.0895	0.1018	0.8650	0.0298	0.0006	0.0006	0.2925
M _M	95.6204	2.4474	0.0486	0.1111	0.2373	0.0887	0.1013	0.8752	0.0298	0.0008	0.0006	0.2711
s _M	0.1452	0.0484	0.0022	0.0055	0.0121	0.0045	0.0077	0.0179	0.0016	0.0003	0.0001	0.0272
s _w	0.1242	0.0312	0.0013	0.0025	0.0024	0.0025	0.0043	0.0100	0.0014	0.0002	0.0001	0.0112

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

Additional Information (mass content in %)

Analyst No. 1 determined HfO₂, SO₃, ZnO, SrO and CuO by XRF and found ≤0.0002% HfO₂, <0.05% SO₃, 0.001% ZnO, <0.01% SrO and <0.01% CuO

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	TiO ₂	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	BaO	Cr ₂ O ₃	PbO	LOI
Cv	95.62	2.447	0.0486	0.1111	0.237	0.0887	0.101	0.875	0.0298	0.0008	0.0006	0.271
C(95%)	0.09	0.025	0.0011	0.0027	0.007	0.0023	0.004	0.009	0.0010	0.0002	0.0001	0.015

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.

BCS-CRM No. 528 STANDARD GLASS SAND

SGT STANDARD GLASS SAND 11

NOTES ON METHODS USED

SILICA

Analysts Nos. 1, 6, 9, 13, 14, 19 and 20 determined silica gravimetrically: No.1 used the standard method ČSN 72 0105-2, Nos. 6, 7, 9, 13, 19 and 20 dehydrated with hydrochloric acid, No. 9 followed the Standard Method BS 2975-2:2008 and No 19 followed the Standard Method JIS M8852. No. 20 determined the residual silica using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Analyst No 14 dehydrated with perchloric acid.

Analysts Nos. 2, 3, 4, 5, 12, 15 and 17 used X-Ray Fluorescence Spectrometry (XRF), No 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analyst No. 8 determined silica by ICP-OES after a fusion with sodium carbonate.

Analysts Nos. 7 and 13 also determined silica by XRF and found 95.6% and 95.72% respectively.

ALUMINA

Analysts Nos. 1 and 13 determined alumina by Flame Atomic Absorption Spectrometry (FAAS), No. 13 using the method published in Glass Technology 34 239 1993.

Analysts Nos. 2, 3, 4, 5, 12 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 7, 8, 10, 16, 18, 19 and 20 used ICP. Analysts Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 and No. 20 prepared the sample solution according to the standard method DIN 52342-2.

Analyst No. 14 determined alumina used a photometric method with chromazurol s.

Analyst No. 1 also determined alumina by complexometric titration with ethylenediaminetetra-acetic acid (EDTA) according to the standard method ČSN 70 0628-1 and found 2.406%. Nos. 7 and 13 also used XRF and found 2.45% and 2.47% respectively.

TITANIA

Analysts Nos. 1, 13 and 14 determined titania photometrically, Nos. 1 and 13 used tiron, No. 1 following the standard method ČSN 72 0112-2) and No 13 that published in Glass Technology 34 239 1993. Analyst No. 14 used chromotropic acid to develop the colour.

Analysts Nos. 2, 3, 4, 5, 12 and 15 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 7, 8, 10, 11, 16, 17, 18, 19 and 20 determined titania by ICP. Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 whilst No. 20 digested the sample with hydrofluoric and sulphuric acids before fusing the insoluble residue with a mixture of sodium carbonate and sodium tetraborate.

Analyst Nos. 7, 11 and 13 also determined by XRF and found 0.05%, 0.05%, and 0.051% respectively. In addition No. 13 also used the ICP method described in Glass Technology 34 239 1993 and found 0.0484%

FERRIC OXIDE

Analyst Nos. 1, 9, 13 and 14 determined ferric oxide photometrically. No 1 used 2-2' bipyridine and followed the standard method ČSN 72 0110-2. ; Analyst No. 9 used mercaptoacetic acid and followed the standard method BS 2975:1958; Nos. 13 and 14 used 1:10 Phenanthroline.

Analysts Nos. 2, 4, 12, 15 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 7, 8, 10, 11, 16, 18, 19 and 20 used ICP. Analysts Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 and No. 20 prepared the sample solution according to the standard method DIN 52342-2.

Analyst No. 1 also determined ferric oxide by FAAS and found 0.1107%. Analysts Nos. 7, 11 and 13 also used XRF and obtained 0.11%, 0.1% and 0.107% respectively. In addition No. 13 also used the ICP method described in Glass Technology 34 239 1993 and found 0.1143%

CALCIUM OXIDE

Analysts Nos. 1, 7, 13 and 14 determined calcium oxide by FAAS. No 13 used the method published in Glass Technology 34 239 1993).

Analysts Nos. 2, 3, 4, 5, 12, 15 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 8, 11, 18, 19, and 20 used ICP. Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 whilst No. 20 digested the sample with hydrofluoric and sulphuric acids before fusing the insoluble residue with a mixture of sodium carbonate and sodium tetraborate.

Analyst Nos. 7, 11 and 13 also determined calcium oxide by XRF and reported 0.25%, 0.27%, and 0.23% respectively.

MAGNESIUM OXIDE

Analysts Nos. 1, 7, 9 and 14 determined magnesium oxide by FAAS; No. 9 followed the standard method BS 2975-2: 2008.

Analysts Nos. 2, 3, 5, 12 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 8, 10, 11, 13, 16, 18, 19 and 20 used ICP. Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 and No. 20 digested the sample with hydrofluoric and sulphuric acids before fusing the insoluble residue with a mixture of sodium carbonate and sodium tetraborate.

Analyst Nos. 7, 11 and 13 also determined magnesium oxide by XRF and found 0.11%, 0.1% and 0.12% respectively.

SODIUM OXIDE

Analysts Nos. 1, 6, 7, 13, 14 and 20 determined sodium oxide with FAAS. No 13 followed the method published in Glass Technology 34 239 1993 and No 20 digested the sample with hydrofluoric, perchloric and hydrochloric acids.

Analysts Nos. 2, 4, 5 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 8, 10, 11, 16 and 18 used ICP, Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids.

Analysts No. 9 and 19 used atomic emission spectrophotometry (AES), No 9 following the standard method BS 2975-2: 2008 and No 19 according to the Standard Method JIS M8852.

Analysts Nos. 7, 11 and 13 also determined sodium oxide by XRF and obtained 0.14%, 0.1%, and 0.10% respectively.

POTASSIUM OXIDE

Analysts Nos. 1, 6, 7, 13, 14, 15 and 20 determined potassium oxide using FAAS. No 13 followed the method published in Glass Technology 34 239 1993 and No 20 digested the sample with hydrofluoric, perchloric and hydrochloric acids.

Analysts Nos. 2, 3, 4, 5, 10, 12 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 8 and 18 used ICP after digesting the sample with a mixture of hydrofluoric and nitric acids.

Analysts No. 9 and 19 used atomic emission spectrophotometry (AES); No 9 following the standard method BS 2975-2: 2008 and No 19 according to the Standard Method JIS M8852.

Analysts Nos. 7 and 13 also determined potassium oxide by XRF and found 0.86% and 0.89% respectively.

BARIUM OXIDE

Analyst No. 1 determined barium oxide by FAAS.

Analysts Nos. 2, 5 and 17 used XRF; No. 2 following the method described in X-Ray Spectrometry 24 205-218 1995.

Analysts Nos. 6, 7, 8, 10, 11, 12, 13, 18, 19 and 20 used ICP, Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 and No 20 digested the sample with hydrofluoric, perchloric and hydrochloric acids.

Analysts Nos. 7 and 11 also determined barium oxide by XRF and found 0.05%, and 0.033% respectively.

CHROMIUM OXIDE

Analysts Nos. 5, 14 and 17 determined chromium oxide by XRF.

Analysts Nos. 6, 8, 10, 11, 18, 19 and 20 used ICP. Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids, No 19 followed the standard method JIS M8852 and No. 20 digested the sample with hydrofluoric and sulphuric acids before fusing the insoluble residue with a mixture of sodium carbonate and sodium tetraborate.

Analyst Nos. 7 and 12 used FAAS.

Analyst No. 9 used the standard method BS 2975:1958 in which the colour is developed with diphenylcarbazide.

Analyst No 11 also determined chromium oxide by XRF and reported <0.05%

LEAD OXIDE

Analysts Nos. 1 and 12 determined lead oxide using FAAS.

Analysts Nos. 8, 13, 18 and 20 used ICP, Nos. 8 and 18 digested the sample with a mixture of hydrofluoric and nitric acids and No 20 digested the sample with hydrofluoric, perchloric and hydrochloric acids.

LOSS ON IGNITION

All Analysts determined the loss on ignition gravimetrically by heating at $1000^{\circ} \pm 25^{\circ}$ C to constant weight.

MANGANESE

Analysts Nos. 5 and 15 determined manganese by XRF whilst No. 8 used ICP after a digestion with hydrofluoric and nitric acids.

TIN

Analyst No. 5 determined tin by XRF whilst No. 8 used ICP after a digestion with hydrofluoric and nitric acids.

PHOSPHORUS PENTOXIDE

Analysts Nos. 5, 7 and 15 determined phosphorus pentoxide by XRF.

ZIRCONIUM OXIDE

Analysts Nos. 5, 7 and 15 determined phosphorus pentoxide by XRF.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

- 1 BAUER, L., Sklářský ústav Hradec Králové s. r. o. (Glass Institute Ltd.) Hradec Králové, Czech Republic.
- 2 BURTON, R., *MSc*, Sheffield Hallam University, Sheffield.
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- 4 JITWATCHARAKOMOL, T., Department of Science Service, Ministry of Science & Technology, Thailand.
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ANALYSTS representing MANUFACTURERS and USERS

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- 14 MALHEIRO, M., St Gobain Recherche, Aubervilliers, France.
- 15 MAYHER, J., Guardian Glass, Carleton, USA.
- 16 MICHIELS, D., AGC Flat Glass Europe, Jumet, Belgium.
- 17 MORAN, T., Sibelco UK, Whiston.
- 18 SMOLDERS, S., Phillips Research Europe, Eindhoven, The Netherlands.
- 19 TSUJINO, T., Nippon Sheet Glass, Techno Research Co Ltd., Konoike Itami, Japan.
- 20 STRUBEL, C., Schott AG, Corporate Research & Technology Development, Mainz, Germany.

DESCRIPTION OF SAMPLE

Bottles of 100g of finely divided material for chemical analysis passing a nominal 250 micron aperture.

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, for establishing values for secondary reference materials and for training purposes.

It 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.

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. Provided that the material is stored in a suitable environment there will be no contribution to the uncertainty from the long term stability of this CRM.

TRACEABILITY

The traceability of this CRM has been established in accordance with principles of ISO Guides 30 – 35 and the International Vocabulary of Basic and General Terms in Metrology.

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 methods used 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.

Nine of the participating laboratories were accredited to ISO/IEC 17025 at the time of the analysis, although not necessarily for all of the constituents determined and not necessarily for the analysis of sand. It has been established statistically that there is no difference between the results of the accredited and the non-accredited laboratories.

Bureau of Analysed Samples Ltd is the Reference Material Producer as defined in ISO Guide 34:2009 section 3.1 and is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006. The Society of Glass Technology has acted as a collaborator, as defined in ISO Guide 34:2009 section 3.1 and provided substantial advice during the certification of this material.

Bureau of Analysed Samples Ltd is a UKAS Accredited Reference Material Producer No 4004.

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

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