MAIN EDITION

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 309
SILLIMANITE

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

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ANALYSES

Mean of 4 values - mass content in %.

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

Analyst No.	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	*MnO*	*Li_2O*	*LOI*
1	34.2	61.0	1.52	1.96	0.23	0.17	0.32	0.50	*0.02*	*0.01*	*0.08*
2	34.2	61.4	1.53	1.82	0.21	0.18	0.30	0.43	...	...	*0.06*
3	34.1	61.1	1.47	2.00	...	0.18	0.32	0.48	...	...	*0.10*
4	34.1	61.2	1.56	...	...	0.12	0.28	0.42	*0.02*	...	*0.08*
5	34.1	61.3	1.48	1.82	0.23	0.21	0.43	0.52	...	...	*0.11*
6	34.0	61.2	1.50	1.90	...	0.14	0.35	0.46	...	...	...
7	34.2	60.9	1.51	2.02	0.21	0.18	0.38	0.43	...	...	...
8	34.2	60.8	1.54	1.89	0.21	0.15	0.30	0.44	*0.04*	...	*0.18*
M_M	**34.1**	**61.1**	**1.51**	**1.92**	**0.22**	**0.17**	**0.34**	**0.46**	...	...	...
s_M	0.1	0.3	0.04	0.09	0.02	0.03	0.05	0.04	...	...	...

The following additional results have been supplied by Analyst No. 1 using direct reading spectroscopy:
SrO 0.003%, BaO 0.006%

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.

CERTIFIED VALUES (Cv)

mass content in %

	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
Cv	**34.1**	**61.1**	**1.51**	**1.92**	**0.22**	**0.17**	**0.34**	**0.46**
C(95%)	0.1	0.2	0.03	0.08	0.02	0.03	0.05	0.04

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. 309 SILLIMANITE

NOTES ON METHODS USED

SILICA

All Analysts determined silica gravimetrically after fusing the sample with sodium carbonate/borax mixture and dissolving the melt in hydrochloric acid[1,2]. No. 2 carried out three evaporations. In all cases the small amount of silica which remained in the filtrate after the double or triple evaporation was determined separately. All Analysts except No. 8 determined this gravimetrically in the R_20_3 precipitate obtained with ammonia. No. 8 determined the residual silica photometrically.

ALUMINA

Analysts Nos. 1 and 2 determined alumina gravimetrically as oxinate according to the Standard method B.S. 1902: 1952, Addendum No. 1 (1957) Part 2. The other Analysts determined alumina by difference after weighing the ignited R_20_3 precipitate and deducting Fe_2O_3 and $Ti0_2$[1].

Analyst No. 1 also determined alumina gravimetrically as Al_2O_3 after removing iron and titanium by solvent extraction with cupferron/chloroform; the result by this method was 61.0%. No. 3 also used an oxine method and found 60.8%.

FERRIC OXIDE

Analysts Nos. 1, 2, 3, 4, 6 and 8 determined iron by photometric methods. Nos. 1 and 2 fused the R_20_3 precipitate with bisulphate and determined iron in the extract with 1:10 phenanthroline. No. 3 treated a separate sample with hydrofluoric/sulphuric acid, fused the residue with sodium carbonate/borax mixture, extracted with dilute hydrochloric acid and determined iron photometrically with 1:10 phenanthroline. Nos. 4 and 6 treated separate samples with hydrofluoric/sulphuric acid, fused the residue with bisulphate, extracted with dilute sulphuric acid and determined iron photometrically with thiocyanate (No. 4) or thioglycollic acid (No. 6). No. 8 fused the R_20_3 precipitate with bisulphate and determined iron with thioglycollic acid. Nos. 5 and 7 treated the sample with hydrofluoric/ sulphuric acid and titrated with titanous chloride[1].

TITANIUM DIOXIDE

All Analysts determined titanium photometrically with hydrogen peroxide. Nos. 1, 6 and 8 fused the R_20_3 precipitate with bisulphate, extracted the melt with dilute sulphuric acid and added hydrogen peroxide[1]. Nos. 2, 3, 5 and 7 fused separate samples with sodium carbonate/borax mixture, extracted the melt with dilute sulphuric acid and applied the peroxide method[1].

LIME

All analysts except No. 1 determined lime by atomic absorption spectroscopy. No. 1 used direct reading spectroscopy.

Analyst No. 1 also found 0.22% by X-ray fluorescence.

MAGNESIA

All Analysts determined magnesia in the filtrate from the lime determination by precipitation as magnesium ammonium phosphate and ignition to magnesium pyrophosphate. Nos. 2 and 6 evaporated with nitric acid to remove ammonium salts before precipitation.

Analyst No. 1 also found 0.16% by X-ray fluorescence.

SODIUM AND POTASSIUM OXIDES

Analysts Nos. 1, 2, 4, 6, 7, 8 and 9 treated the sample with hydrofluoric/sulphuric acid and determined the alkalis with a flame photometer. Nos. 3 and 5 treated the sample according to the J. Lawrence Smith method and determined sodium by the triple acetate method. No. 5 used an aliquot from the same solution to determine potassium as cobaltinitrite[1]; No. 3 determined potassium with a flame photometer after decomposition with hydrofluoric/perchloric acid.

Analyst No. 5 also determined the alkalis spectrographically and found 0.30% Na_2O and 0.46% K_20.

MANGANOUS OXIDE

Photometric method using periodate.

LITHIUM OXIDE

Flame photometric method.

LOSS ON IGNITION

All analysts except No. 8 ignited at 1000°C. No. 8 ignited at 1200°C.

REFERENCES

1. Analysis Committee of the Society of Glass Technology, The Chemical Analysis of Sillimanite in the form of a Fired Block, Trans. Soc. Glass, Technology, 1953, **37**, 97-112.
2. Bennett and Hawley, Methods of Silicate Analysis, The British Ceramic Research Association, 1958.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded <90µm (170 mesh) for chemical analysis.

INTENDED USE & STABILITY

The 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. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

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For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

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