



# BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

## CERTIFICATE OF ANALYSIS

### BCS-CRM No. 393 (ECRM 752-1)

### LIMESTONE

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

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- 9 NEWELL, D.,  
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#### ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	CaO	MgO	K <sub>2</sub> O	S	L.O.I.	BaO	SrO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	F
1	0.717	0.122	0.008	0.043	0.010	55.24	0.151	0.018	0.004	43.58	0.0075	0.020	<0.02	0.004	...
2	0.674	...	0.008	0.043	0.010	55.54	0.148	0.014	...	43.54	...	...	...	...	0.001
3	0.714	0.128	0.009	0.045	0.010	55.37	0.163	0.017	0.006	43.52	0.0063	0.021	0.01	0.005	...
4	0.67	0.139	0.01	0.045	0.012	55.26	0.145	0.02	...	43.52	0.0045	0.018	0.01	...	...
5	0.705	0.124	0.010	0.039	0.010	55.64	0.165	0.03	0.007	43.38	0.006	0.018	0.03	...	<0.01
6	0.72	0.123	...	0.057	...	55.17	0.150	0.026	0.009	43.32	...	...	0.01	...	0.001
7	0.724	0.127	0.007	0.040	0.010	55.52	0.158	0.027	0.007	43.49	0.0076	0.020	0.02	0.007	...
8	0.730	0.115	0.012	0.052	0.010	55.41	0.172	0.020	...	43.50	...	...	0.02	...	...
9	0.683	0.12	...	0.043	...	55.03	0.14	0.02	...	43.18	...	...	...	...	...
<b>M<sub>M</sub></b>	<b>0.704</b>	<b>0.125</b>	<b>0.009</b>	<b>0.045</b>	<b>0.010</b>	<b>55.35</b>	<b>0.155</b>	<b>0.021</b>	<b>0.007</b>	<b>43.45</b>	<b>0.0064</b>	<b>0.019</b>	...	...	...
<i>s<sub>M</sub></i>	0.023	0.008	0.002	0.006	0.001	0.20	0.011	0.006	0.002	0.13	0.0013	0.002	...	...	...

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

Figures in bold type certified, figures in small italic type only approximate.

**M<sub>M</sub>**: Mean of the intralaboratory means. *s<sub>M</sub>*: standard deviation of the intralaboratory means.

The barium oxide content of this sample was also determined by Dr P. B. Smith, *BSc, PhD, CChem, FRSC*, (BCIRA, Birmingham) using inductively coupled plasma atomic emission spectrometry. A mean value of 0.0061% was obtained.

#### CERTIFIED VALUES (Cv)

mass content in %

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	CaO	MgO	K <sub>2</sub> O	S	L.O.I.	BaO	SrO
<b>Cv</b>	<b>0.70</b>	<b>0.12</b>	<b>0.009</b>	<b>0.045</b>	<b>0.010</b>	<b>55.4</b>	<b>0.15</b>	<b>0.02</b>	<b>0.007</b>	<b>43.4</b>	<b>0.006</b>	<b>0.019</b>
C(95%)	0.02	0.01	0.002	0.005	0.001	0.2	0.01	0.01	0.003	0.1	0.002	0.002

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. 393 (ECRM 752-1) LIMESTONE

## NOTES ON METHODS USED

### SILICA

Analysts Nos. 1, 6 and 7 determined silica by XRF<sup>1</sup>. Nos. 2 and 8 determined the acid insoluble silica gravimetrically with HF volatilization and the acid soluble silica photometrically as molybdenum blue. No. 2 followed the procedure given in B.S. 3108: 1980<sup>3</sup>. No. 3 dehydrated the silica with perchloric acid and completed gravimetrically. No. 5 dehydrated with hydrochloric acid and determined the residual silica photometrically as molybdenum blue. No. 9 decomposed the sample with hydrochloric acid, separated the silica by coagulation and completed gravimetrically (B.S. 1902: 1970)<sup>4</sup>. No. 4 used a molybdenum blue photometric method after fusion with sodium carbonate.

Analyst No. 9 also determined silica by XRF<sup>1</sup> and found 0.67%.

### ALUMINA

Analysts Nos. 1 and 7, determined alumina by XRF<sup>1</sup>. Nos. 3, 5 and 6 used AAS<sup>2</sup>. No. 4 used a photometric method with ferron. Nos. 8 and 9 used EDTA complexometric methods, No. 9 according to B.S. 1902:1970<sup>4</sup>.

Analysts Nos. 4 and 9 also determined alumina by XRF<sup>1</sup> and found 0.12% and 0.14% respectively. No. 5 also used an EDTA complexometric method and found 0.12%.

### TITANIA

Analysts Nos. 1, and 7 determined titania by XRF<sup>1</sup>. Nos. 2, 3, 4 and 8 used photometric methods; Nos. 2 and 4 with tiron, No. 3 with diantipyrylmethane and No. 8 with hydrogen peroxide. No. 5 used AAS<sup>2</sup>.

Analyst No. 5 also determined titania photometrically with diantipyrylmethane and found 0.008%.

### FERRIC OXIDE

Analysts Nos. 1 and 7 determined ferric oxide by XRF<sup>1</sup>. Nos. 2, 3, 5, 8 and 9 used 1:10 phenanthroline photometric methods, Nos. 2 and 9 according to the procedures given in B.S. 3108: 1980<sup>3</sup> and B.S. 1902: 1970<sup>4</sup> respectively. No. 4 used a photometric method with ferron and No. 6 used AAS<sup>2</sup>.

Analyst No. 5 also determined ferric oxide by AAS<sup>2</sup> and found 0.043%. No. 9 also used XRF<sup>1</sup> and found 0.046%.

### MANGANOUS OXIDE

Analysts Nos. 1 and 7 determined manganous oxide by XRF<sup>1</sup>. Nos. 2, 3 and 5 used AAS<sup>2</sup>. Nos. 4 and 8 oxidized the manganese with periodate and completed photometrically.

### LIME

Analysts Nos. 1, 6 and 7, determined lime by XRF<sup>1</sup>. The remaining analysts used complexometric methods with EDTA (Nos. 2, 4, 5 and 8) or EGTA (Nos. 3 and 9). No. 2 followed the procedure given in B.S. 3108: 1980<sup>3</sup> and No. 9 followed that given in B.S. 1902: 1970<sup>4</sup>.

Analyst No. 9 also determined lime by XRF<sup>1</sup> and found 54.96%.

### MAGNESIA

Analysts Nos. 1, 7 and 9 determined magnesia by XRF<sup>1</sup>. Nos. 2, 3, 4, 5 and 6 used AAS<sup>2</sup>. No. 8 used an EDTA complexometric method.

### POTASSIUM OXIDE

Analysts Nos. 1 and 9 determined potassium oxide by XRF<sup>1</sup>. No. 6 used AAS<sup>2</sup>. The remaining analysts used flame emission spectrometry.

### SULPHUR

Analyst No. 1, determined sulphur by high frequency combustion with iodimetric titration. Nos. 3, 6 and 7 used gravimetric methods depending upon precipitation as barium sulphate. No. 3 first carried out a chromatographic separation on an alumina column (Nydahl, Anal. Chem. 1954, **26**, 580). No. 5 used a method involving evolution of the sulphur as H<sub>2</sub>S followed by titration with 2-(hydroximercuri) benzoic acid (Murphy and Sergeant, Analyst 1974, **99**, 515).

### LOSS ON IGNITION

Determined by heating to constant weight at 1000°C.

### BARIUM OXIDE

Analysts Nos. 1 and 7 determined barium oxide by XRF<sup>1</sup>. Nos. 3 and 4 separated barium as sulphate using lead sulphate as a carrier and completed by AAS<sup>2</sup> (Bano, Analyst 1973, **98**, 655). Analyst No. 5 used AAS<sup>2</sup> without prior separation but with background correction.

### STRONTIUM OXIDE

Analysts No. 1 and 7 determined strontium oxide by XRF<sup>1</sup>. No. 3 used flame emission spectrometry. Nos. 4 and 5 used AAS<sup>2</sup>.

### SODIUM OXIDE

Analysts Nos. 3 and 6 determined sodium oxide by AAS<sup>2</sup>. The remaining analysts used flame emission spectrometry.

### PHOSPHORUS PENTOXIDE

Analysts Nos. 1 and 7 determined phosphorus pentoxide by XRF<sup>1</sup>. No. 3 separated phosphorus by precipitation with ammonia and completed photometrically as phosphovanadomolybdate.

### FLUORINE

All analysts determined fluorine directly by means of a specific ion electrode in a buffered solution

### REFERENCES:

- |   |                |  |
|---|----------------|--|
| 1 | XRF            | X-ray fluorescence spectrometry, fused cast bead technique with synthetic calibration.                     |
| 2 | AAS            | Atomic absorption spectrometry with synthetic calibration.   |
| 3 | B.S. 3108:1980 | Specification for Limestone for Making Colourless Glasses. Published by the British Standards Institution. |
| 4 | B.S. 1902:1970 | Chemical Analysis of Magnesites and Dolomites, Part 2E.  |

## DESCRIPTION OF SAMPLE

British Chemical Standard – bottles of 100g powder graded < 75µm (200 mesh) for chemical analysis.

## INTENDED USE & STABILITY

BCS-CRM 393 (ECRM 752-1) 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 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.

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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For BUREAU OF ANALYSED SAMPLES LTD

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Preliminary Edition .....	September 1981
Main Edition .....	June 1984
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