MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 301/1 (ECRM 651-1)
LINCOLNSHIRE IRON ORE

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.	Fe	SiO_2	CaO	MgO	Al_2O_3	TiO_2	MnO	P	S	Na_2O	K_2O	*CO_2*	*H_2O*	*LOI*
1	23.83	7.40	22.7	1.69	4.21	0.16	1.21	0.36	0.39	0.06	0.31	*22.2*	*5.5*	*25.5*
2	23.89	7.40	22.4	1.74	4.22	0.18	1.24	0.35	0.41	0.05	0.29	*22.2*	*5.5*	*25.9*
3	23.89	7.43	22.8	1.67	4.31	0.16	1.29	...	...	0.06	0.31	*22.9*	...	*25.6*
4	23.85	7.42	22.6	1.74	4.22	0.17	1.27	0.37	0.40	...	...	*21.8*	*5.4*	*26.0*
5	23.85	7.41	22.5	1.75	4.31	0.16	1.28	0.35	0.41	0.07	0.34	*22.0*	*4.8*	*25.5*
6	23.86	7.38	22.5	1.75	4.26	0.16	1.22	0.34	0.40	0.08	0.34	*21.9*	*5.1*	*25.9*
7	23.79	7.37	22.6	1.76	4.27	0.16	1.24	0.35	0.40	0.08	0.31	*22.0*	*5.4*	*26.0*
M_M	**23.85**	**7.40**	**22.6**	**1.73**	**4.26**	**0.16**	**1.25**	**0.35**	**0.40**	**0.07**	**0.32**	...	...	...
s_M	0.04	0.03	0.2	0.04	0.05	0.01	0.04	0.02	0.01	0.02	0.02	...	...	...

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 %

	Fe	SiO_2	CaO	MgO	Al_2O_3	TiO_2	MnO	P	S	Na_2O	K_2O
Cv	**23.85**	**7.40**	**22.6**	**1.73**	**4.26**	**0.16**	**1.25**	**0.35**	**0.40**	**0.07**	**0.32**
C(95%)	0.04	0.02	0.2	0.04	0.04	0.01	0.03	0.02	0.01	0.02	0.03

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.

Note. The dried sample is hygroscopic and rapidly reabsorbs moisture if exposed to the atmosphere. Analysis should preferably be carried out on the "as received" sample and corrected for moisture content.

EQUIVALENT % ELEMENT CONTENT OF OXIDE CONSTITUENTS

	Si	Ca	Mg	Al	Ti	Mn	Na	K
M_M	**3.46**	**16.1**	**1.04**	**2.25**	**0.10**	**0.97**	**0.05**	**0.26**

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NOTES ON METHODS USED

IRON

All Analysts determined iron by titration of the reduced solution with standard dichromate solution. Nos. 1, 5 and 7 used the Standard Method B.S. 4158: Part 1: 1967 which depends on reduction of the ferric iron with hydrogen sulphide. No. 3 used a similar method. Nos. 2, 4 and 6 reduced with stannous chloride.

Analyst No. 5 also used a stannous chloride reduction method and found 24.00%. No. 6 also used the British Standard Method and found 23.64%.

Analyst No. 4 also determined ferrous iron and found 7.4%; this will result in an ignition gain of 1.1%.

SILICA

All Analysts determined silica gravimetrically after double dehydration with perchloric acid. Nos. 1, 4, 5 and 6 followed the procedure of the Standard Method B.S. 4158: Part 3: 1972.

LIME

Analysts Nos. 1 and 4 separated Group III elements by double precipitation with ammonia and separated manganese with bromine/ammonia; the lime was then precipitated as oxalate and titrated. No. 2 separated the bulk of the iron by extraction into amyl acetate, then separated any remaining Group III elements together with manganese by means of bromine/ammonia and finally precipitated lime as oxalate and completed gravimetrically by ignition to calcium oxide. No. 3 separated Group III elements by double precipitation with ammonia and determined lime gravimetrically by ignition of the oxalate; a correction was made for co-precipitated manganese which was determined photometrically. Nos. 5 and 6 carried out a basic acetate separation followed by a bromine/ammonia separation, then precipitated lime as oxalate and completed by titration. No. 7 separated lime by a basic acetate separation followed by bromine/ammonia separation but completed by titration with EDTA.

MAGNESIA

Analysts Nos. 1 and 6 determined magnesia by atomic absorption spectroscopy. Nos. 2, 4 and 5 determined magnesia in the filtrate from the lime determination by precipitation as magnesium ammonium phosphate and gravimetric determination as $Mg_2P_2O_7$. Nos. 3 and 7 used the filtrate from the lime determination but titrated with EDTA.

Analysts Nos. 2 and 5 also used atomic absorption spectroscopy and found 1.74% and 1.69% respectively. No. 6 also determined magnesia gravimetrically as the pyrophosphate and found 1.84%.

ALUMINA

Analysts Nos. 1, 2, 3, 4 and 7 separated aluminium together with other Group III elements by precipitation with ammonia solution ; iron was separated by treatment with sodium hydroxide solution and the aluminium was precipitated as oxinate and determined by titration with bromate[1] (Nos. 1, 4 and 7) or gravimetrically (Nos. 2 and 3). Nos. 5 and 6 used atomic absorption spectroscopy.

Analyst No. 5 also determined aluminium as oxinate by titration and found 4.26% expressed as alumina.

TITANIA

Analysts Nos. 1, 2, 3, 4, 6 and 7 determined titania photometrically with hydrogen peroxide. Nos. 1, 2, 3 and 6 carried out a preliminary separation with cupferron[1], No. 4 first separated titanium, together with aluminium, as the phosphate. No. 5 determined titania photometrically with diantipyrylmethane.

Analyst No. 5 also used a cupferron/hydrogen peroxide method and found 0.17%.

MANGANESE OXIDE

Analysts Nos. 1, 2 and 4 determined manganese oxide by titration. No. 1 oxidized with persulphate/silver nitrate and titrated with arsenite/nitrite solution (Analoid Method No. 53), No. 2 oxidized with bismuthate and titrated with ammonium ferrous sulphate/permanganate. No. 4 oxidized with persulphate/silver nitrate and titrated with arsenite. Nos. 3, 5, 6 and 7 determined manganese oxide photometrically after oxidation with periodate.[1]

PHOSPHORUS

All Analysts except No. 5 determined phosphorus by titration after precipitation as ammonium phosphomolybdate. Nos. 1, 6 and 7 oxidized the phosphorus by evaporation with perchloric acid and eliminated arsenic by treatment with hydrobromic acid before precipitating the phosphorus.[1] No. 5 determined phosphorus photometrically as phosphovanadomolybdic acid according to the Standard method BS 4158 : Part 2 : 1970.

SULPHUR

Analysts Nos. 1, 2, 4, 5 and 6 determined sulphur gravimetrically as barium sulphate. Nos. 1, 2, 5 and 6 used a method involving reduction of the iron with hydroxylammonium chloride.[1] No. 7 determined sulphur by combustion in the presence of tungstic oxide using nitrogen as carrier gas.[2]

Analyst No. 4 also used a combustion method and found 0.40%.

ALKALIS

Analysts Nos. 1, 2, 3 and 7 determined alkalis with a flame photometer. Nos. 5 and 6 used atomic absorption spectroscopy.

CARBON DIOXIDE

All Analysts determined carbon dioxide gravimetrically by evolution with dilute acid and absorption in soda-asbestos.[1]

COMBINED WATER

All Analysts determined combined water by heating the dried sample at 1000°C in a stream of dry air and collecting the evolved water in magnesium perchlorate.[1]

LOSS ON IGNITION

All Analysts except Nos, 3 and 7 determined loss on ignition by heating at 1000°C for 1 hour. No. 3 heated at 1050°C to constant weight. No, 7 heated at 1000°C to constant weight.

[1] British Iron and Steel Research Association: Report on Recommended Methods of Analysis for Iron Ores MG/D/56/59.

[2] British Iron and Steel Research Association: Report on the Determination of Sulphur in Non-Metallic Materials, MG/D/412/70.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded < 180µm (85 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

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