MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

Directors:-
R. P. MEERES, *BA (Oxon), MRSC* (Managing)
G. C. FLINTOFT, *ACMA*
J. C. MEERES
M. S. TAYLOR *PhD, CChem, MRSC*

4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 517 BRAZILIAN IRON ORE

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

ANALYSES

Mean of 4 values - mass content in %. All results relate to the dried (105°C) sample.

Analyst No.	Fe	Si	Ca	Mg	Al	Ti	Mn	P	S
1	66.3408	0.5227	0.0327	0.0300	0.5163	0.0339	0.6895	0.0398	0.0088
2	66.2875	0.4952	0.0361	0.0334	0.4945	0.0316	0.6810	0.0440	0.0105
3	66.2700	...	...	...	0.5133	...	...	0.0432	0.0097
4	66.2218	0.5232	0.0322	0.0336	0.5125	0.0338	0.6822	0.0400	0.0074
5	66.2400	0.5321	0.0298	0.0279	0.5125	0.0335	0.6738	0.0364	0.0083
6	66.3925	0.5290	0.0308	0.0292	0.5141	0.0315	0.6703	0.0404	0.0080
7	66.3260	0.5106	0.0376	0.0322	0.4951	0.0341	0.6817	0.0415	0.0104
8	...	...	...	...	...	0.0340	0.6718	...	...
9	...	...	...	...	...	...	...	...	...
10	...	...	...	...	...	...	...	...	...
M_M	**66.2969**	**0.5188**	**0.0332**	**0.0311**	**0.5083**	**0.0332**	**0.6786**	**0.0408**	**0.0090**
s_M	0.0601	0.0138	0.0031	0.0024	0.0094	0.0012	0.0069	0.0026	0.0013
s_w	0.0456	0.0048	0.0007	0.0013	0.0052	0.0008	0.0047	0.0005	0.0004

Analyst No.	Na	K	V	C	Zn	Pb	Cu	LOI	Cl (soluble)
1	0.0111	0.0117	0.0047	0.0577	0.0044	...	0.0087	1.8818	...
2	0.0093	0.0094	0.0033	0.0591	0.0051	0.0025	0.0083	1.8975	...
3	0.0100	...	...	...	...	0.0032	0.0099	1.9115	...
4	0.0112	0.0120	0.0051	0.0648	0.0046	0.0022	0.0092	1.9300	0.00083
5	...	0.0110	0.0040	0.0550	0.0043	0.0031	0.0085	1.8800	...
6	0.0088	0.0090	0.0035	0.0650	0.0047	0.0031	0.0082	1.8873	...
7	0.0080	0.0100	0.0034	0.0636	0.0054	0.0030	0.0093	...	...
8	...	0.0107	0.0040	...	0.0044	0.0023	0.0083	...	0.00060
9	...	...	...	...	...	...	...	...	0.00057
10	...	...	...	...	...	...	...	...	0.00097
11	...	...	...	...	...	...	...	...	0.00076
M_M	**0.0097**	**0.0105**	**0.0040**	**0.0609**	**0.0047**	**0.0028**	**0.0088**	**1.8980**	**0.00075**
s_M	0.0013	0.0012	0.0007	0.0042	0.0005	0.0005	0.0007	0.0196	0.00017
s_w	0.0002	0.0006	0.0003	0.0010	0.0003	0.0003	0.0004	0.0138	0.00006

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

Additional Information:

Analyst No. 8 also determined Co, Ni, As and Ba by ICP-MS and reported values of 0.0003%, 0.0003%, 0.0004% and 0.0149% respectively.
The same Analyst also determined B, Se, Cd, Sb and Tl by ICP-MS and reported values of ≤0.0001%, ≤0.0002%, ≤0.0001%, ≤0.0002% and ≤0.0001% respectively

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 %

	Fe	Si	Ca	Mg	Al	Ti	Mn	P	S
Cv	**66.30**	**0.519**	**0.033**	**0.0311**	**0.508**	**0.0332**	**0.679**	**0.0408**	**0.0090**
C(95%)	0.06	0.015	0.004	0.0025	0.009	0.0011	0.007	0.0024	0.0012

	Na	K	V	C	Zn	Pb	Cu	LOI	Cl (soluble)
Cv	**0.0097**	**0.0105**	**0.0040**	**0.061**	**0.0047**	**0.0028**	**0.0088**	**1.898**	**0.00075**
C(95%)	0.0014	0.0011	0.0007	0.005	0.0004	0.0004	0.0006	0.021	0.00021

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. 517 BRAZILIAN IRON ORE

NOTES ON METHODS USED

IRON

All Analysts determined iron by titration with Cr (VI) after reduction with Sn (II).

Analyst No. 7 also determined iron by x-ray fluorescence spectrometry (XRF) and found a mean value of 66.458%.

SILICON

Analysts Nos. 1, 2 and 6 determined silicon gravimetrically as silica. Analyst No.1 dehydrated with perchloric acid whereas the other two Analysts used sulphuric acid. Analyst No. 4 determined silicon photometrically as molybdenum blue. Analysts Nos. 5 and 7 used XRF.

CALCIUM

Analysts Nos. 1 and 2 determined calcium using Flame Atomic Absorption Spectrometry (FAAS). Analysts Nos. 4 and 6 used Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) whilst Analysts Nos. 5 and 7 used XRF.

MAGNESIUM

Analysts Nos. 1, 4 and 6 determined magnesium using ICP-OES. Analyst No. 2 used FAAS, whilst Nos. 5 and 7 used XRF.

ALUMINIUM

All Analysts, except for Nos. 5 and 7 determined aluminium using ICP-OES. Analysts Nos. 5 and 7 used XRF.

TITANIUM

All Analysts, except for Nos. 5, 7 and 8 determined titanium using ICP-OES. Analysts Nos. 5 and 7 used XRF, whilst No. 8 used Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

MANGANESE

Analysts Nos. 1, 4, 5 and 6 determined manganese by ICP-OES. Analyst No. 2 used FAAS, Analyst No. 7 used XRF and Analyst No. 8 used ICP-MS.

PHOSPHORUS

Analysts Nos. 1 and 2 determined phosphorus photometrically as the phosphovanadomolybdate complex after extraction with 4-methyl pentan-2-one. Analysts Nos. 3, 4 and 6 used ICP-OES whilst Nos. 5 and 7 used XRF.

SULPHUR

Analyst No. 1 determined sulphur gravimetrically as barium sulphate. Analyst No. 2 used a redox titration with potassium iodate after combustion with a flux and the remaining Analysts used methods based on infra-red absorption following combustion.

SODIUM

Analysts Nos. 1, 2, 4 and 7 determined sodium using FAAS. The remaining Analysts used ICP-OES.

Analyst No. 7 also determined sodium using ICP-OES and reported a mean value of 0.0075%.

POTASSIUM

Analysts Nos. 1, 2, 4 and 7 determined potassium using FAAS. Analysts Nos. 5 and 6 used ICP-OES whilst No. 8 used ICP-MS.

VANADIUM

Analysts Nos. 1, 4, 5 and 6 determined vanadium using ICP-OES. Analyst No. 2 used FAAS, Analyst No. 7 used XRF and Analyst No. 8 used ICP-MS.

CARBON

Analyst No. 1 determined carbon by non-aqueous titration after combustion in a stream of oxygen. Analyst No. 2 used a coulometric titration after combustion and the remaining Analysts used methods based on infra-red absorption after combustion.

ZINC

Analysts Nos. 1 and 2 determined zinc using FAAS. Analysts Nos. 4, 5, 6 and 7 used ICP-OES and Analyst No. 8 used ICP-MS.

LEAD

Analysts No. 2 determined lead by FAAS. Analysts Nos. 3, 4, 5, 6 and 7 used ICP-OES whilst No. 8 used ICP-MS.

COPPER

Analysts Nos. 1, 2 and 4 determined copper by FAAS. Analysts Nos. 3, 5, 6 and 7 used ICP-OES whilst No. 8 used ICP-MS.

LOSS ON IGNITION

All Analysts determined the loss on ignition gravimetrically by heating at 1000° ± 25° C to constant weight.

CHLORIDE (soluble)

Analysts Nos. 4, 9 and 10 determined soluble chloride by ion chromatography. Analyst No. 9 extracted 5g of sample into 25ml of deionised water by shaking for 24 hours before diluting to 50ml whilst No. 10 gently boiled 1g of sample in about 150ml of deionised water for 10min before diluting to 200ml. Analyst No. 8 used a specific ion electrode after extraction in an ultrasonic bath. Analyst No. 11 determined soluble chloride by titration with silver nitrate using potassium chromate as the indicator in a method loosely based on BS EN 480-10:1997.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

1 JONES, S.J., *BSc, CChem, MRSC,* Ridsdale & Co. Ltd., Middlesbrough.
2 CROCKER, F.H., Pattinson & Stead (2005) Ltd, Middlesbrough.

ANALYSTS representing MANUFACTURERS and USERS

3 SNOWDEN, Y., Corus Testing Solutions, Scunthorpe.
4 WEERDT, J. de, Corus Analytical Department, Projects & Special Analysis, IJmuiden, Holland.
5 DAVIES, M., Corus Strip Products, Port Talbot.
6 CROOK, D., Corus Strip Products, Llanwern.
7 RICHMOND, H., Corus Testing Solutions, Redcar.
8 COLLINS, P., Corus Research, Development & Testing, Swinden Technology Centre, Rotherham.
9 JOHNSON, K. M., *BSc, PhD,* Ceram Research Ltd, Stoke on Trent.
10 JOHNSON, K. M., *BSc, PhD,* Ceram Research Ltd, Stoke on Trent.
11 ATHERTON, S., *BSc, MSc,* UK Analytical Ltd., Leeds.

DESCRIPTION OF SAMPLE

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

Seven 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 iron ore. 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 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 may be obtained from the address below.

For BUREAU OF ANALYSED SAMPLES LTD.
NEWHAM HALL
NEWBY
MIDDLESBROUGH
ENGLAND
TS8 9EA

Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk

R P MEERES,
Managing Director

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