



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 388

ZIRCON

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

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

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ANALYSES

Mean of 4 values - mass content in %. All results relate to the dried (110°C) sample.

Analyst No.	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	P ₂ O ₅	ZrO ₂ *	HfO ₂	ThO ₂	U ₃ O ₈	Y ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	LOI
1	32.6	0.282	0.227	0.053	0.120	66.1	1.31	0.016	0.037	0.139	0.04	0.04	<0.01	<0.01	0.28
2	32.6	0.302	0.231	0.049	0.127	66.2	0.02	0.03	0.15
3	1.17	0.019	0.031	0.144
4	1.16	0.019	0.035
5	32.6	0.052	0.108	66.1	0.03	0.02	0.01	0.01	0.19
6	32.7	0.289	0.237	0.044	0.115	66.2	1.39	0.020	0.028	0.121	0.03	<0.01	0.02	<0.01	0.16
7	32.8	66.2	0.01	0.01	0.18
8	32.7	0.286	0.228	0.050	0.130	66.3	1.32	0.141	0.04	<0.01	0.02	<0.01	0.22
9	0.117
10	0.134
11	...	0.298	0.238	0.046	1.34	0.019	0.037	0.135
M_M	32.7	0.291	0.232	0.049	0.122	66.2	1.28	0.019	0.034	0.136	0.04	<0.05	<0.02	<0.03	0.20
<i>S_M</i>	0.1	0.009	0.006	0.004	0.010	0.1	0.10	0.002	0.004	0.009

* Includes HfO₂ (ZrO₂ = 64.9% by difference)

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_M: Mean of the intralaboratory means. S_M: standard deviation of the intralaboratory means.

The following additional information was supplied by Analysts Nos. 3 and 4:-

La 0.003%, Ce 0.008%, Nd 0.004%, Sm 0.001%, Eu 0.0004%, Yb 0.03%, Lu 0.005%.

CERTIFIED VALUES (C_v)

mass content in %

	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	P ₂ O ₅	ZrO ₂ *	HfO ₂	ThO ₂	U ₃ O ₈	Y ₂ O ₃
C _v	32.7	0.291	0.232	0.049	0.122	66.2	1.28	0.019	0.034	0.136
C(95%)	0.1	0.011	0.007	0.004	0.009	0.1	0.10	0.002	0.005	0.012

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. 388 ZIRCON

NOTES ON METHODS USED

SILICA

All Analysts fused the sample with sodium or potassium carbonate/boric acid and dissolved the melt in hydrochloric and sulphuric acids; after evaporation the silica was coagulated with polyethylene oxide, filtered off and determined gravimetrically. The residual silica in the filtrate was determined photometrically as yellow silicomolybdic acid. Unless stated otherwise the filtrate from the silica determination was also used for the determination of titania, alumina, lime and magnesia.

Analysts No. 8 also found 32.6% by X-ray fluorescence spectrometry.

ALUMINA

All Analysts, except No. 2 determined alumina using X-ray fluorescence spectrometry. No. 2 used atomic absorption spectrometry.

Analyst No. 6 also determined alumina by atomic absorption spectrometry and obtained a mean value of 0.298%.

TITANIA

All Analysts, except No. 2, determined titania using X-ray fluorescence spectrometry. No. 2 used a photometric method with diantipyrylmethane.

Analyst No. 6 also determined titania by atomic absorption spectrometry and obtained a mean value of 0.238%.

FERRIC OXIDE

Analysts Nos. 1, 6 and 11 determined ferric oxide using X-ray fluorescence spectrometry. No. 2 used atomic absorption spectrometry. Nos. 5 and 8 determined ferric oxide photometrically with 1,10-phenanthroline.

PHOSPHORUS PENTOXIDE

Analysts Nos. 1, 6 and 10 determined phosphorus using X-ray fluorescence spectrometry calibrated with synthetic standards. (Nos. 1 and 10 used fused bead techniques, No. 6 used a compressed disc). Nos. 2, 8 and 9 used phosphovanadomolybdate photometric methods. Nos. 2 and 8 fused the sample with sodium hydroxide or carbonate and extracted with water whereas No. 9 fused with a mixture of boric acid and lithium fluoride, extracted with sulphuric acid and removed zirconium by extraction with cupferron/chloroform. Analyst No. 5 fused the sample with sodium hydroxide, extracted with water, precipitated phosphorus as phosphomolybdate and completed gravimetrically as lead molybdate.

ZIRCONIA (+ Hafnia)

All Analysts determined zirconia (+ hafnia) gravimetrically after precipitation with cupferron; corrections were applied for iron and titanium.

HAFNIA

Analysts Nos. 1, 6, 8 and 11 determined hafnia using X-ray fluorescence spectrometry. No. 3 used ICP-MS and No. 4 used Instrumental Neutron Activation Analysis^{1,2}.

THORIA

Analysts Nos. 1, 6 and 11 determined thoria by X-ray fluorescence spectrometry. No. 3 used ICP-MS and No. 4 used Instrumental Neutron Activation Analysis^{1,2}.

Analyst No. 3 also determined thoria using laser ablation - ICP-MS on a fused glass bead and obtained a mean value of 0.019% ThO₂.

URANIA

Analysts Nos. 1, 6 and 11 determined urania using X-ray fluorescence spectrometry. No. 3 used ICP-MS and No. 4 used Instrumental Neutron Activation Analysis^{1,2}.

Analyst No. 3 also determined urania using laser ablation - ICP-MS on a fused glass bead and obtained a mean value of 0.031% U₃O₈.

YTTRIA

Analysts Nos. 1, 6, 8 and 11 determined yttria using X-ray fluorescence spectrometry. No. 3 used ICP-MS.

Analyst No. 3 also determined yttria using laser ablation - ICP-MS on a fused glass bead and obtained a mean value of 0.14% Y₂O₃.

LIME

Analyst No. 1 determined lime by titration with EGTA in strongly alkaline solution. Nos. 5, 6 and 8 used Flame Atomic Absorption Spectrometry.

MAGNESIA

Analyst No. 1 determined magnesia by titration with EDTA in ammoniacal solution: a correction was made for lime. Nos. 5, 6 and 8 used Flame Atomic Absorption Spectrometry.

ALKALIS

All Analysts determined alkalis by Flame Atomic Emission Spectrometry.

LOSS ON IGNITION

Loss on ignition was determined by heating at 1000°C.

References:-

1. Potts, P.J. et al. (1981), Chemical Geology, 34,331-352.
2. Potts, P.J. et al. (1985), Chemical Geology, 48, 145-155.

Abbreviations:-

ICP-MS: Inductively Coupled Plasma-Mass Spectrometry

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded 75 micron (200 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.

TRACEABILITY

The traceability of BCS-CRM 388 to the SI is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

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For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
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<i>Preliminary Edition</i>	July 1973
<i>Main Edition</i>	September 1975
<i>Main Edition, incorporating values for P₂O₅</i>	August 1981
<i>Main Edition with certified values for HfO₂, Y₂O₃, U₃O₈, ThO₂ and revised values for Fe₂O₃</i>	October 1992
<i>Main Edition with revised values for TiO₂ and Al₂O₃</i>	July 1993
<i>Main Edition (revised with C(95%) values for each certified constituent and recalculated M_M and s_M values)</i>	August 2015