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Certificate of Analyses

B.C.S. No. 365 (P.M.A. 71) ALCOMAX III PERMANENT MAGNET ALLOY

Specially prepared in co-operation with the Permanent Magnet Association, Sheffield, and, AFTER STANDARDIZATION BY ANALYSTS IN GREAT BRITAIN, issued by the Bureau of Analysed Samples Ltd.

The material for this standard was supplied by Darwins Ltd., Sheffield and was crushed to pass a 60 mesh sieve by Murex Ltd., Rainham, Essex. The fines were removed by sieving on a 300 mesh sieve.

CO-OPERATING ANALYSTS and FIRMS

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ANALYSES

Analyst No.	C %	Si %	S %	Mn %	Ni %	Cu %	Co %	Al %	Nb %	Ta %	Fe %
1	0·024	0·33	0·180	0·12	13·44	2·69	$24{\cdot}6_5$	8·09	0·55	0·035	*49·3*
2	0·024	0·33	0·182	0·12	13·53	2·71	$24{\cdot}5_0$	8·08	0·55	0·033	..
3	0·026	0·36	0·188	0·10	13·40	2·70	$24{\cdot}5_0$	8·20	0·58	0·037	..
4	0·026	0·32	0·18–	0·12	13·43	2·68	$24{\cdot}8_0$	8·08	..	..	..
5	0·026	0·34	0·173	0·12	13·51	2·75	$24{\cdot}6_0$	8·18	0·59	0·032	..
6	0·024	0·33	0·180	0·12	13·47	2·75	$24{\cdot}8_0$	8·10	0·57	0·031	..
7	..	..	..	..	13·48	2·76	$24{\cdot}6_0$	8·05	..	..	..
8	0·023	0·34	0·189	0·11	13·62	2·68	$24{\cdot}6_0$	8·12	0·58	0·031	..
9	0·025	0·33	0·17–	0·11	13·51	2·68	$24{\cdot}7_5$	8·13	0·58	..	..
10	0·024	0·34	0·170	..	13·59	2·83	$24{\cdot}8_0$	8·23	0·58	0·034	..
11	0·030	0·34	0·172	0·12	13·57	2·67	$24{\cdot}8_5$	8·15	0·57	0·034	..
12	0·024	0·33	0·171	0·10	13·43	2·67	$24{\cdot}7_0$	8·19	..	..	..
Average	**0·025**	**0·34**	**0·18–**	**0·11**	**13·5–**	**$2{\cdot}7_0$**	**24·7–**	**$8{\cdot}1_5$**	**0·57**	**0·033**	..

The above figures are those which each analyst has decided upon after careful verification. Figures in bold type standardized, figure in small italic type only approximate.

[P.T.O.

B.C.S. No. 365 (P.M.A. 71) ALCOMAX III PERMANENT MAGNET ALLOY

NOTES ON METHODS USED

CARBON

All analysts determined carbon by combustion in oxygen. Nos. 1, 5, 6, 9 and 10 used a non-aqueous titration method (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399); No. 1 employed a visual end-point and Nos. 5, 6, 9 and 10 potentiometric end-points. Nos. 2, 3, 4, 8, 11 and 12 determined carbon gravimetrically using large samples of at least four times normal factor weight (Bagshawe and Pinder, Analyst, 1956, **81**, 153).

Analyst No. 5 also determined carbon gravimetrically and found 0·023%.

SILICON

Analysts Nos. 1, 2, 5, 6, 9, 11 and 12 determined silicon gravimetrically. Nos. 1 and 9 carried out a double dehydration with perchloric acid according to the Standard method B.S. 1121: Part 10: 1967. Nos. 2, 6, 11 and 12 evaporated and baked with hydrochloric acid. No. 5 dehydrated with sulphuric acid. Nos. 3, 4, 8 and 10 determined silicon colorimetrically as molybdenum-blue.

Analyst No. 4 also determined silicon gravimetrically after dehydration with perchloric acid and found 0.31%.

SULPHUR

All analysts except No. 5 determined sulphur by combustion using tin as flux. No. 1 absorbed the sulphur gases in water and titrated with iodide/iodate. The other analysts absorbed in hydrogen peroxide and titrated with sodium borate. In all cases the titrant was standardized empirically using an alloy of known sulphur content. No. 5 determined sulphur gravimetrically after dissolution of the alloy in oxidizing acid.

MANGANESE

All analysts determined manganese colorimetrically after oxidation with periodate according to the Standard method B.S. 1121: Part 23: 1951.

NICKEL

Analysts Nos. 1 and 5 carried out a preliminary separation of copper as the sulphide and determined nickel gravimetrically with dimethylglyoxime. Nos. 2, 3, 6, 7, 8, 10, 11 and 12 determined nickel volumetrically. All except No. 6 precipitated with dimethylglyoxime (No. 11 carried out a double precipitation) and determined nickel cyanometrically according to the Standard method B.S. 1121: Part 37; 1961. No. 6 separated nickel with dimethylglyoxime and titrated with EDTA. Nos. 4 and 9 determined nickel colorimetrically with dimethylglyoxime.

COPPER

Analysts Nos. 1, 2, 3, 8 and 12 precipitated copper with hydrogen sulphide and determined it iodometrically according to the Standard method B.S. 1121: Part 5: 1953. Nos. 4, 6, 7, 9, 10 and 11 determined copper colorimetrically with *bis*-cyclohexanone oxalyldihydrazone. No. 5 separated copper as sulphide and determined it colorimetrically with diethyldithiocarbamate after extraction into carbon tetrachloride.

Analyst No. 9 also determined copper by atomic-absorption spectroscopy and found 2·64%.

COBALT

Analysts Nos. 1, 2, 3, 4, 5, 7 and 12 determined cobalt volumetrically by addition of an excess of potassium ferricyanide solution in the presence of ammonium citrate buffer; the excess was titrated potentiometrically with cobalt sulphate solution. No. 1 carried out a preliminary separation of the cobalt with phenylthiohydantoic acid. No.s 6, 8, 9, 10 and 11 determined cobalt colorimetrically with nitroso-R-salt according to the Standard method B.S. 1121: Part 30: 1954.

ALUMINIUM

Analysts Nos. 1, 2, 3, 5 and 10 determined aluminium either volumetrically or gravimetrically via the oxinate. Nos. 1, 2 and 3 used the Standard method B.S. 1121: Part 4: 1953 in which interfering elements are removed by mercury-cathode electrolysis followed by precipitation with cupferron. Nos. 5 and 10 precipitated the oxinate directly from an ammoniacal tartrate solution containing cyanide. Nos. 4, 7, 8, 9, 11 and 12 determined aluminium volumetrically with EDTA; sufficient EDTA was added to complex all the alloying elements, then ammonium fluoride was added to form a complex with aluminium, liberating an equivalent amount of EDTA which was titrated with standard zinc solution. No. 6 separated iron with sodium hydroxide, then determined aluminium by addition of potassium fluoride and titration of the liberated hydroxyl ions with hydrochloric acid.

NIOBIUM

Analysts Nos. 1, 2, 6, 8, 9, 10 and 11 separated niobium, together with tantalum, by means of phenylarsonic acid. Nos. 1 and 11 determined it gravimetrically, making a correction for tantalum which was determined separately. Nos. 2, 6 and 10 determined niobium colorimetrically with pyridylazoresorcinol (PAR). No. 3 separated niobium, together with tantalum, by hydrolysis from a reduced solution, purified by re-precipitation as mixed magnesium niobate and tantalate and determined gravimetrically as the oxide, a correction being made for tantalum. No. 5 separated niobium from other elements, including tantalum, by ion-exchange and determined it gravimetrically after precipitation with cupferron.

TANTALUM

Analysts Nos. 1, 2, 3 ,6 10 and 11 separated tantalum, together with niobium, by means of phenylarsonic acid. No. 1 fused ignited oxides with bisulphate, extracted with ammonium oxalate solution and determined tantalum colorimetrically with pyrogallol (Kidman, Darwent and White, Metallurgia, 1960, **62**, 125). Nos. 2, 3, 6, 8, and 10 separated tantalum from niobium by solvent extraction with *iso*-butyl methyl ketone and determined it colorimetrically with pyrogallol (Nos. 2and 3) or phenylfluorone (Nos. 6 and 10). No. 11 separated tantalum in the same way but determined it gravimetrically. No. 5 separated tantalum from other elements, including niobium, by ion-exchange, precipitated it with cupferron using zirconium as a carrier, and finally determined it by emission spectroscopy.

IRON

The iron was separated together with other Group III elements by precipitation with ammonia and the precipitate was dissolved reduced with stannous chloride and titrated with dichromate.

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For BUREAU OF ANALYSED SAMPLES LTD.,
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