

DEPARTMENT OF COMMERCE

Bureau of Standards
Certificate of Analyses

OF

STANDARD SAMPLE NO. 69

BAUXITE

All Results are Based on a Sample Dried for 2 Hours at 140° C.

ANALYSTS*	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	ZrO ₂	P ₂ O ₅	V ₂ O ₅	Cr ₂ O ₃	SiO ₂	CaO	MgO	MnO	CuO	Na ₂ O	K ₂ O	SO ₃	Loss on Ignition
1	{ 54.98 ^a 54.94 ^b 55.09 ^c }	5.63 ^d	3.04	0.07	0.12	0.03	0.03	6.32	0.05	0.02	0.55	0.03	0.18	0.05	0.06	28.81
2	55.13	5.74	3.05	.04	.12	.02		6.31	.04	.03	.56		.20	.08		28.77
3	55.08 ^e	5.61 ^d	3.09	.08	.11	.03	.05	6.39	.05	.01	.54	.03	.16	.08	.06	28.73
	55.12	5.60	3.07	.09	.10			6.21			.55					28.74
	55.06	5.74	3.10	.10	.12			6.37			.54					28.80
AVERAGES	55.06	5.66	3.07	.08	.11	.03	.04	6.32	.05	.02	.55	.03	.18	.07	.06	28.77

^a, ^b, and ^c See reverse side.
^d No ferrous iron could be detected.

^e Sample fused by exploding with Na₂O₂ and sugar carbon. See Muehlberg, Ind. and Eng. Chem., 17, 690; 1925.

INDEX TO ANALYSTS *

- | | |
|--|---|
| 1. James I. Hoffman, Bureau of Standards. | 4. M. O. Lamar, Norton Co., Worcester, Mass. |
| 2. H. V. Churchill, Aluminum Co. of America, Pittsburgh, Pa. | 5. T. R. Cunningham, The Electro Metallurgical Co., New York, N. Y. |
| 3. W. F. Muehlberg, Newburgh Steel Works, Cleveland, Ohio. | |

GEORGE K. BURGESS,
Director.

Washington, D. C.
May 3, 1926.

METHODS USED AT THE BUREAU OF STANDARDS

Drying.—As this material changes in moisture content with different atmospheric conditions, all determinations were made on a sample dried for two hours at 140° C. Experiments made at the bureau indicated that this is the lowest temperature at which a sample can be satisfactorily reproduced.

Alumina.—(a) The sample (0.5000 to 0.7500 g) was dissolved in platinum in a mixture of nitric, hydrofluoric and sulphuric acids, evaporated to fumes of sulphuric acid, treated with water and again evaporated to fumes to insure complete expulsion of hydrofluoric acid. The solution was then cooled, diluted, and filtered if a small residue remained. This small residue was fused with sodium carbonate, dissolved in water, and added to the main solution. In a volume of 200 cc containing 10 per cent (by volume) sulphuric acid, the iron, titanium, zirconium, and vanadium were precipitated with cupferron in an ice-cold solution. The cupferron precipitate was ignited, fused with pyrosulphate, dissolved in 100 cc of dilute sulphuric acid (10 per cent by volume) and again precipitated with cupferron and filtered. The combined filtrates were evaporated to fumes of sulphuric acid in platinum dishes, treated at intervals with small quantities of nitric acid until the cupferron was destroyed and then cooled and diluted to 300 to 400 cc. The aluminum was then precipitated with ammonia using methyl red as indicator, filtered, washed with a hot 5 per cent solution of ammonium nitrate, dissolved in hydrochloric acid, reprecipitated, filtered and ignited to constant weight. The precipitate was fused with pyrosulphate dissolved in dilute sulphuric acid, evaporated at a low temperature to incipient fumes of sulphuric acid to dehydrate any silica, cooled, diluted, and filtered. The small amount of silica was determined in the usual way and the P_2O_5 was determined in the filtrate. Both of these, as well as chromium, were subtracted from the original weight of Al_2O_3 .

(b) The sample was dissolved as in (a) and after expulsion of hydrofluoric acid, the alumina, iron, titanium, zirconium, phosphorus, chromium, and vanadium were precipitated with ammonium hydroxide, filtered, ignited and weighed. The SiO_2 , Fe_2O_3 , TiO_2 , ZrO_2 , P_2O_5 , Cr_2O_3 and V_2O_5 were then determined separately in this precipitate and subtracted.

(c) The filtrate from the silica determination was nearly neutralized with sodium hydroxide, warmed, and poured slowly and with constant stirring into 150

cc of a warm 10 per cent solution of sodium hydroxide, allowed to stand for one-half hour and filtered. The precipitate on the filter was dissolved in hydrochloric acid and reprecipitated. In the combined filtrates in a volume of 400 cc the aluminum was precipitated with ammonium hydroxide using methyl red as indicator. This precipitate was dissolved in hydrochloric acid and the ammonium hydroxide precipitation twice repeated (three precipitations in all). The precipitate was ignited to constant weight and corrected for SiO_2 , V_2O_5 and P_2O_5 .

Blank determinations were carried through all of these and succeeding determinations and corrections were made as necessary.

Silica.—The sample (1.000 g) was fused in a platinum crucible with 8 to 10 g of sodium carbonate. The melt was cooled, dissolved in 150 cc of a 10 per cent solution of sulphuric acid, and evaporated in a platinum dish to moderate fumes of sulphuric acid. The mass was then taken up in 150 to 200 cc of warm water, and heated until salts were in solution. The silica was then filtered off and washed first with 5 per cent sulphuric acid and then with warm water. The evaporation and filtration was repeated. The combined papers were then ignited and the SiO_2 determined in the usual way by treatment with hydrofluoric and sulphuric acids. The silica which escaped the two dehydrations was recovered in the alumina determination and added. In this case the recovery amounted to 0.05 per cent.

Iron Oxide (Fe_2O_3).—The iron oxide was determined in the cupferron precipitate obtained in the alumina determination under (b) and in the sodium hydroxide precipitate obtained in the alumina determination under (c). The Zimmermann-Reinhardt method was used. Platinum was removed by treatment with hydrogen sulphide and the hydrogen sulphide was in turn boiled out and the last trace destroyed by boiling with a few drops of potassium permanganate before the stannous chloride reduction was started.

Titania and Zirconia.—The sample (2.00 to 4.00 g) was dissolved as in (a) under alumina and the iron, titanium and zirconium were then precipitated with sodium hydroxide as in (c) under alumina. Only one sodium hydroxide precipitation is necessary. The precipitate was dissolved in hydrochloric acid, diluted,

300 cc, 4 g tartaric acid added, gassed with hydrogen sulphide until the iron was reduced, rendered ammoniacal and gassed a little longer. The iron sulphide was then filtered off and washed with dilute ammonium sulphide water containing 10 g of ammonium chloride per liter. The filtrate and washings were acidified with sulphuric acid, digested for 5 to 10 minutes and any sulphides that separated were filtered off. The filtrate was then boiled to expel hydrogen sulphide and the titanium and zirconium precipitated by cupferron in a cold solution in a volume of 200 cc and an acidity of 10 per cent by volume (H_2SO_4). The precipitate was first dried, then ignited, treated with hydrofluoric and sulphuric acids to eliminate silica, and weighed as $TiO_2 + ZrO_2$. See Lundell and Knowles, *J. Ind. Eng. Chem.*, **12**, 562; 1920.

The precipitate of titania and zirconia was fused with a small amount of pyrosulphate, the melt dissolved in 50 cc of 10 per cent sulphuric acid, and sufficient hydrogen peroxide added to oxidize all of the titanium (an excess does no harm). Diammonium phosphate (0.5 g) was then added, the solution was allowed to stand over night, the precipitate was filtered off, washed with 5 per cent ammonium nitrate, ignited and weighed as ZrP_2O_7 . The ZrP_2O_7 was calculated to ZrO_2 and subtracted from the total weight $TiO_2 + ZrO_2$. See Lundell and Knowles, *J. Am. Chem. Soc.*, **42**, 1439; 1920.

Manganese (MnO).—Manganese was determined by the bismuthate method after the vanadium was removed by a sodium hydroxide precipitation.

Vanadium, Chromium, and Phosphorus.—After dissolving 5.00 g of the sample as in (a) under alumina, the solution was nearly neutralized with sodium hydroxide and poured into an excess of sodium hydroxide containing sodium peroxide. The solution was digested on the steam bath for one hour, cooled,

and filtered. The chromium was determined, colorimetrically in the alkaline filtrate. After the chromium was determined, the solution was acidified with nitric acid and the vanadium determined colorimetrically by development of the color with hydrogen peroxide.

The hydrogen peroxide was then practically all destroyed by boiling for one-half hour, and the phosphorus precipitated by molybdate reagent. The molybdate precipitate was filtered off, dissolved in ammonium hydroxide, the phosphorus precipitated with magnesia mixture and finally weighed as $Mg_2P_2O_7$.

Sodium and Potassium.—Sodium and potassium were determined by the J. Lawrence Smith method as described in "The Analysis of Silicate and Carbonate Rocks," by W. F. Hillebrand, *Bull.* 700 U. S. Geological Survey.

Calcium and Magnesium.—The sample (5.00 g) was dissolved as in (a) under alumina. The solution was diluted to a volume of 200 cc, nearly neutralized with sodium hydroxide and poured into 150 cc of a 10 per cent solution of sodium hydroxide containing 1 g of sodium carbonate. The precipitate, which contained all of the calcium and magnesium, was dissolved in hydrochloric acid, the iron, titanium, etc., was precipitated with ammonium hydroxide and removed by filtration. The precipitate was dissolved and the ammonium hydroxide precipitation repeated. The calcium was determined by precipitation as the oxalate in the combined filtrates. The magnesium was determined by precipitation as magnesium ammonium phosphate in the filtrate from the calcium determination.

Loss on Ignition.—Loss on ignition was determined by igniting 0.5000 g samples to constant weight in a partially covered platinum crucible. The sample was heated slowly at first and finally over a moderate blast.