

DEPARTMENT OF COMMERCE

**Bureau of Standards**  
**Certificate of Analyses**

OF

STANDARD SAMPLE NO. 1A

**ARGILLACEOUS LIMESTONE**

[All results are based on a sample dried for one hour at 105° to 110° C.]

ANALYST*	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	CaO	SrO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	S	SO <sub>3</sub>	C	CO <sub>2</sub>	Loss on Ignition
1.....	14.12	1.57	4.17	0.16	0.14	0.036	41.23	<del>0.13</del> *0.13	2.17	0.43	0.69	0.24	0.047	0.53	33.65	34.53
2.....	14.02	1.65	4.16	.17	.16	.044	41.47	.11	2.23	.31	.62	.29	.04	.69	33.36	-----
3.....	14.00	1.57	4.10	.11	.11	-----	41.50	.10	2.17	.41	.77	.24	.056	-----	33.62	34.54
4.....	14.10	1.69	4.35	-----	-----	-----	41.32	-----	2.26	-----	-----	.18	1.02	-----	-----	34.56
5.....	14.14	1.57	4.27	-----	.18	-----	41.19	.17	2.14	1.21	.62	.29	.049	-----	-----	34.54
-----	14.18	1.57	4.20	1.25	.18	1.06	41.64	-----	2.22	.39	.76	.27	1.02	-----	-----	34.49
7.....	14.16	1.66	4.33	-----	.16	-----	41.28	-----	2.22	.38	.68	.23	.03	-----	-----	34.60
8.....	14.11	1.59	4.14	.16	.15	.04	41.23	-----	2.24	-----	-----	.20	-----	-----	-----	34.54
9.....	14.10	1.63	4.07	.20	.15	-----	41.24	.14	2.15	.45	.74	.26	.03	-----	-----	34.59
10.....	13.98	1.63	4.20	.18	.15	.03	41.35	.10	2.19	.32	.74	.28	-----	-----	33.51	34.57
11.....	14.20	1.68	4.27	-----	.108	-----	41.29	.106	2.11	.40	.78	.29	.03	-----	33.51	34.54
Averages....	14.11	1.63	4.16	.16	.15	.038	41.32	<del>.12</del>	2.19	.39	.71	.25	.04	.61	33.53	34.55

<sup>1</sup>Not included in averages.

11-10546

**\*LIST OF ANALYSTS**

1. H. B Knowles, Bureau of Standards.
2. L. G. Sprague, Universal Atlas Cement Co., Northampton, Pa.
3. T. Clum, North American Cement Corp., Catskill, N. Y.  
M. Samuelson, North American Cement Corp., Security, Md.
4. J. R. Schwab, Lehigh Portland Cement Co., Allentown, Pa.
5. E. A. Ledyard, Riverside Cement Co., Riverside, Calif.
6. W. P. Eckdahl, Universal Atlas Cement Co., Buffington, Ind.

7. F. C. Crumbliss, Pennsylvania-Dixie Cement Corp., Richard City, Tenn.
8. R. Stotts, Pennsylvania-Dixie Cement Corp., Des Moines, Iowa.
9. J. Clifford Evans, Nazareth Cement Co., Nazareth, Pa.
10. F. A. Martin, C. L. Ford, Raymond Wilson, research laboratory, Portland Cement Association, Chicago, Ill.
11. J. G. Hackman, Alpha Portland Cement Co., Easton, Pa.

Washington, D. C.  
January 8, 1931.

*George K. Burgess*

Director.

\*A recent redetermination of this value at the NBS, by flame photometry, indicated 0.23 percent SrO (Oct. 1, 1954).

METHODS USED AT THE BUREAU OF STANDARDS IN THE ANALYSIS OF  
STANDARD ARGILLACEOUS LIMESTONE NO. 1a

The results for silica, carbon dioxide, and the oxides of calcium, strontium, magnesium, sodium, and potassium were obtained, in general, by the methods described by W. F. Hillebrand in "The Analysis of Silicate and Carbonate Rocks," United States Geological Survey Bulletin No. 700.

Ferric oxide was determined on 1 g samples by precipitating the iron twice as the sulphide from an ammoniacal ammonium tartrate solution and finally precipitating with ammonia. The resulting precipitate was cautiously ignited to oxide and corrected for any silica it might contain.

Alumina was determined by difference. The sum of the percentage of oxides of iron, titanium, and phosphorus, as separately determined, was subtracted from the percentage of oxides obtained by precipitation with ammonia.

Titania was determined by the usual colorimetric procedure after fusing the ignited precipitate obtained by cupperron in the filtrates from the iron determination, dissolving the melt in 10 per cent sulphuric acid and treating with hydrogen peroxide.

Manganese was determined on complete solutions of 25 g samples by oxidation with sodium bismuthate and titration with ferrous sulphate.

Total sulphur was determined by decomposing 25 g samples with nitric acid (specific gravity 1.42) and bromine, evaporating with hydrochloric acid to remove the nitric acid and finally precipitating with barium chloride. The resulting barium sulphate was digested in concentrated hydrochloric acid and again precipitated from a 1 per cent hydrochloric acid solution, filtered, washed, ignited, and weighed.

Sulphur trioxide was determined by treating 25 g samples with dilute hydrochloric acid (1:1), evaporating to near dryness, diluting with hot water, and filtering. The insoluble residue was digested with concentrated hydrochloric acid, diluted with hot water, filtered, and washed. The combined filtrates were neutralized, made 1 per cent by volume with hydrochloric acid and precipitated with barium chloride in the usual manner. The resulting barium sulphate was digested with concentrated hydrochloric acid, evaporated to near dryness and again precipitated from a 1 per cent hydrochloric acid solution. The final precipitate of barium sulphate after filtering, washing, igniting, and weighing was corrected for any silica it might contain by treating with hydrofluoric and sulphuric acids.

Phosphorus pentoxide was determined by dissolving 10 g samples in nitric acid and evaporating to dryness. After removal of silica and its volatilization with hydrofluoric acid, any remaining nonvolatile residue was fused with sodium carbonate, dissolved in nitric acid and combined with the main solution. The phosphorus was then precipitated as phosphomolybdate, filtered, washed, dissolved in ammonium hydroxide, twice precipitated as magnesium ammonium phosphate, and finally determined as magnesium pyrophosphate.

Carbon was determined by combustion, after separation by the method described by A. C. Fieldner, W. A. Selvig, and G. B. Taylor in Technical Paper No. 212, of the Bureau of Mines, on the "Determination of Combustible Matter in Silicate and Carbonate Rocks."

Loss on ignition was determined by cautiously heating 1 g samples in covered platinum crucibles for one hour in an electric muffle at approximately 1,000° C.