DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE No. 81

GLASS SAND

ANALYST*	Fe_2O_3†	Al_2O_3	TiO_2	ZrO_2	CaO	MgO	Loss on ignition
1	0.0724[a], .0720[b]	0.261	0.090	0.036	0.018	0.013	0.23
2	.076[a], .073[b]	.247	.102	.032	.028	.008	
3	.0726[b]	.256	.089	.038	.020	.014	.22
4	.068[c]		.10		.051	.01	
5	.077[d]	.294	.090	.009	.034	.008	
6	.070[b]	.272	.090	.033	.024	.015	.234
	.079[d]	.289	.112	.038	.04	.01	.23
8	.080[d]	.238	.091	.039	.020	.018	
9	.072[b]	.276	.092	.032	.024	.014	
10	.067[c]	.25	.095	.02	.03	.045	
Averages	.0732‡	.265	.095	.031	.029	.016	.23

[a] Gravimetric method, FeS-Fe_2O_3.
[b] Electrometric method, $SnCl_2$-$K_2Cr_2O_7$.
[c] Colorimetric method KCNS.
[d] Volumetric method H_2S-$KMnO_4$.

† A ferric oxide content of 0.061 per cent is indicated when a 10 g sample of the sand is simmered for one hour in 50 ml of HCl (sp. gr. 1.19) in an Erlenmeyer flask with a funnel resting in the neck.

‡ Recommended value of 0.072.

*LIST OF ANALYSTS

1. H. B. Knowles, Bureau of Standards.
2. M. O. Lamar, Norton Co., Worcester, Mass.
3. K. O. Sanders, Hazel-Atlas Glass Co., Washington, Pa.
4. W. C. Taylor, Corning Glass Works, Corning, N. Y.
5. R. H. Lardin, Pittsburgh Plate Glass Co., Creighton, Pa.
6. A. R. Payne, Fostoria Glass Co., Moundsville, W. Va.
7. D. D. Schurtz and C. E. Reed, The Sharp-Schurtz Co., Lancaster, Ohio.
8. R. W. Goodwin, Glass Technology Laboratory, Incandescent Lamp Department, General Electric Co., Cleveland, Ohio.
9. C. A. Walworth, The Libbey-Owens Sheet Glass Co., Charleston, W. Va.
10. C. Sundstrom, The Solvay Process Co., Syracuse, N. Y.

Washington, D. C.
May 1, 1928

George K. Burgess
Director.

(See other side)

METHODS USED
at the
BUREAU OF STANDARDS
in the
ANALYSIS OF STANDARD GLASS SAND No. 81

The sample as bottled at the Bureau of Standards showed a loss of 0.02 per cent on drying at 110° C. Drying of the sample is not required unless the analyst has reason to believe that it has been unduly exposed and may have absorbed moisture.

TOTAL IRON AS FERRIC OXIDE

The Bureau of Standards values for total iron as ferric oxide were obtained by both gravimetric and electrometric procedures. The procedure for the gravimetric determination is as follows:

Transfer 25 g of the dried sample to a liter platinum dish, moisten with water and add 200 ml of HF and 10 ml of dilute H_2SO_4 (1:1). Evaporate to fumes of H_2SO_4, cool, rinse the walls of the dish with water, and again evaporate to fumes of H_2SO_4. Treat the contents of the dish with 100 ml of water, heat to boiling, filter through a No. 42 Whatman paper, wash with hot water, and reserve the filtrate (A). Ignite the residue in the paper, fuse with a small amount of Na_2CO_3, and dissolve the melt in dilute H_2SO_4 (1:5). Filter, wash the residue, and combine the filtrate with the original filtrate (A). Again ignite the paper and insoluble matter. Fuse with a minimum quantity of $K_2S_2O_7$, dissolve in dilute H_2SO_4 (1:5) and combine the solution with the reserved filtrate (A). Neutralize the solution with NaOH, dilute to 250 ml and then add 2 to 3 ml of H_2SO_4. Thoroughly saturate the solution with H_2S, and digest, preferably over night. Filter and wash with diluted H_2SO_4 (1:99), saturated with H_2S. To the filtrate add 10 g of tartaric acid and again saturate with H_2S. Slowly add dilute NH_4OH (1:2) until the solution is ammoniacal and pass in H_2S for 10 to 15 minutes. Allow the iron sulphide to settle, filter through a paper of close texture and wash thoroughly with a 1 per cent solution of ammonium tartrate-ammonium sulphide, always keeping the funnel filled. Place the filter and its contents in the beaker in which the precipitation was made and treat with 25 ml of HNO_3 (specific gravity 1.42) and 5 ml of H_2SO_4 (specific gravity 1.84). Cover, and cautiously heat until fumes of H_2SO_4 are evolved. If the solution still contains organic matter, cautiously add HNO_3 (specific gravity 1.42) dropwise and continue the treatment until the solution clears and all organic matter is destroyed. To make sure of the separation of the titanium, etc., cool, rinse the cover glass, dilute to 200 ml with H_2O and to the clear solution add 5 g of tartaric acid. Again thoroughly saturate with H_2S, make ammoniacal, digest, filter, wash the precipitate, and dissolve it in H_2SO_4 and HNO_3 as before. Cool the sulphuric acid solution, dilute to 150 ml with H_2O, heat to boiling and precipitate the iron with NH_4OH. Filter, wash the precipitate a few times with a hot 2 per cent solution of NH_4NO_3 and ignite the precipitate and paper. Cool, treat with a few drops of dilute H_2SO_4 and a few ml of HF, and evaporate to dryness. Cautiously expel the H_2SO_4 and ignite to constant weight at approximately 1,000° C. under good oxidizing conditions. The weight of oxide found must be corrected for the oxide found in two or three runs on the reagents carried through all operations.

The electrometric titration method was based on the complete solution of the sample and removal of platinum as in the gravimetric method, reduction with stannous chloride and titration with a standard solution of potassium dichromate as described by Hostetter and Roberts, J. Am. Chem. Soc., **41**, p. 1337; 1919; Lundell and Knowles, J. Am. Ceram. Soc., **11**, p. 119, 1928; and Bull. Am. Ceram. Soc., **6**, p. 100, 1927.

Determinations, not recorded in the table of results, were also made by the hydrogen sulphide, sulphur dioxide, and colorimetric methods with the following results:

Colorimetric method	0.068 per cent Fe_2O_3
	.069 per cent Fe_2O_3
	.070 per cent Fe_2O_3
Reduction by hydrogen sulphide	.0769 per cent Fe_2O_3
	.0785 per cent Fe_2O_3
Reduction by sulphur dioxide	.0745 per cent Fe_2O_3
	.0749 per cent Fe_2O_3

Of the above methods the colorimetric method normally gives low and the others high results. Correct results can be obtained by proper modifications of the colorimetric method, but at an immoderate expenditure of effort. High results are unavoidable in any of the modifications of the hydrogen sulphide reduction method and are difficult to avoid in the corresponding sulphur dioxide method. Both the gravimetric and electrometric methods can give satisfactory results. The former is the most accurate of all the methods, but requires numerous precautions and very careful manipulation. The latter is rapid and is entirely satisfactory, provided platinum is excluded and the blank correction is properly determined.

Careful determinations of iron in sample No. 81 by the various methods are subject to errors at least as large as the following:

Gravimetric	±0.0005
Electrometric	± .001
Hydrogen sulphide reduction	+ .006
Sulphur dioxide reduction	+ .003
Colorimetric	− .004

OTHER CONSTITUENTS

The remaining constituents in sample No. 81 were determined, in general, by the procedures described by W. F. Hillebrand in "The Analysis of Silicate and Carbonate Rocks," United States Geological Survey Bulletin No. 700.

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