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A RAPID METHOD FOR THE DETERMINATION OF SILICON IN SILICO-SPIEGEL AND FERRO-SILICON.

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IN 1894 Furnace "A" at the Edgar Thomson Steel Works made the first silico-spiegel ever manufactured in this country. A determination of silicon and manganese was required in each cast, the silicon especially, as soon as possible.

The "aqua regia" method was the one tried. Various strengths of acid were used, but it was found almost impossible to get the metal into solution, even after passing the crushed "shot" through bolting-cloth. As high as 400 cc. aqua regia was used on one-half gram, the aqua regia being four parts nitric acid and three parts hydrochloric acid.

The fusion method, as recommended by Williams,¹ was also tried, but we were never able to make a determination in less than six to eight hours. The furnace made only a short run on silico-spiegel and for the time the matter was dropped. Last year we again made some silico-spiegel and were successful in finding a method for determining the silicon in a rapid and accurate manner and give the method in detail below.

Preparation of the Sample.—For the success of the method it is essential that the sample should be in a very fine state of subdivision. Grind in an agate or crush in a diamond mortar so that the sample will pass through a sieve made of bolting-cloth.

¹ *Trans. Am. Inst. Min. Eng.*, 17, 542.

Operation.—One-half gram of the sample is placed in a porcelain or platinum dish; fifty cc. water, ten cc. hydrochloric acid (1.20 sp. gr.) and twelve cc. sulphuric acid (one part sulphuric acid, 1.84 sp. gr., to three parts water) are poured on it; heat until copious fumes of sulphuric acid are given off. Allow the dish to cool, so that there will be no spattering when taking up with acid. When cool, add about ten cc. hydrochloric acid, heat to soften the sulphate of iron, add about seventy-five cc. water, and bring to a boil. Discontinue the heating and note whether there is any effervescence when boiling ceases. If there is, the liquid must be evaporated until copious fumes of sulphuric acid are given off again, then taken up as before directed. Filter at once, wash thoroughly with hydrochloric acid (1 : 1) and hot water, ignite in a platinum crucible, and weigh. Add a few drops sulphuric acid and enough hydrofluoric acid to dissolve the silica. Evaporate to dryness, heat to decompose the sulphates, cool, and weigh. The difference in the two weights is silica, which can be calculated to silicon. The whole operation can be accomplished in thirty minutes.

The following are some results, both by our method and the fusion method :

No.	Our method.	Fusion method.
1	12.08	12.01
2	12.37	12.25
3	12.09	12.08
4	13.46	13.40
5	9.05	9.03

A METHOD FOR THE COMPLETE ANALYSIS OF IRON ORES, WITH NOTES ON SÄRNSTRÖM'S METHOD OF DETERMINING MANGANESE.

BY GEORGE AUCHY.

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SÄRNSTRÖM'S method of determining manganese in iron ores, as described by Messrs. Mixer and DuBois, is to precipitate the iron in dilute hot solution by sodium carbonate, care being taken to add no more of this reagent than just enough to effect the precipitation of the iron; then titrating (without filtering from the precipitated ferric oxide) with standard permanganate. The writer, in experimenting with Volhard's method,