standard conditions are adhered to, very accurate results may be quickly obtained.

The scope of its usefulness, however, is somewhat limited and a great number of details, apparently trivial, affect the results to a considerable extent.

---

## THE DETERMINATION OF SILICA IN IRON ORES CONTAINING ALUMINA.

BY GRAHAM DEAN.

Received April 18, 1906.

I HAVE found that if the insoluble residue left on the treatment of an iron ore with acids is ignited for a short time the aluminum which it contains is converted into a form which dissolves in concentrated hydrochloric acid. On the basis of this fact the following rapid method for the determination of silica in such ores has been developed.

One gram of the finely powdered ore is dissolved on the hot plate in concentrated hydrochloric acid, using an initial amount of 25 cc. Boil until the iron is completely dissolved, adding two or three drops of nitric acid near the end. If pyrites is present, more nitric acid may be required. Nearly all iron ores will yield to this treatment; if an ore does not, the results are worthless.

Evaporate the solution to dryness, take up the residue with a small amount of concentrated hydrochloric acid, boiling till the ferric chloride is all dissolved. Dilute slightly, filter and wash thoroughly with hot hydrochloric acid (1 : 1). If a determination of alumina is required, this filtrate must be examined as well as the solution obtained below.

Transfer the moist filter and residue to a platinum crucible and burn the filter carefully, as usual. Then set the crucible upright, put on the cover and ignite for two or three minutes over a Dangler burner. I have found that the proper temperature is best secured in a dull or carbon covered platinum crucible. The ignition must not be continued too long and the temperature must be properly regulated.[1] I have repeatedly attempted to

[1] Dr. Hillebrand explains the results obtained by Mr. Dean by calling attention to the fact that the aluminum of kaolin is rendered soluble in hydrochloric acid by gentle ignition. See McNeil: This Journal, 28, 592. If the ignition is too intense, the aluminum becomes insoluble again. If this explanation is correct, the method would not be reliable for ores containing other silicates than kaolin.—EDITOR.

obtain good figures by use of bright and shining platinum crucibles but have never been able to do so.

When cold the contents of the crucible are transferred to a beaker, the cake pulverized with a glass rod and digested for seven or eight minutes with boiling concentrated hydrochloric acid.[1] The residue should feel soft and mushy under pressure unless the silica of the ore existed as free sand. Dilute slightly, filter and wash thoroughly. The filtrate may be combined with the first filtrate for the determination of aluminum. The residue, after ignition, may be considered as practically pure silica.

The precipitation of the alumina by phenylhydrazine,[2] I have found most satisfactory.

The figures given below are an average set selected from a large number of experimental determinations made with Mesabi ores containing in some instances a high percentage of alumina, a large portion of which was proved to be retained in the residue previous to separation and extraction. The results under the heading "Ignition" were obtained by the method given above.

| | Ignition. | Sodium carbonate. fusion. | Hydrofluoric acid. |
|---|---|---|---|
| 1 | 12.86 | 12.83 | 12.94 |
| 2 | 22.96 | 22.82 | 22.99 |
| 3 | 17.50 | 17.40 | 17.56 |
| 4 | 5.16 | 5.00 | 5.10 |
| 5 | 6.05 | 6.00 | 5.99 |
| 6 | 13.89 | 13.80 | 13.70 |
| 7 | 9.84 | 9.92 | 10.06 |

The following amounts of alumina were found in residues obtained by the new method: 0.06, 0.04, 0.10, 0.11, 0.02, 0.07, 0.05, 0.09 and 0.10 per cent. Before the separation by ignition and treatment with hydrochloric acid the residues retained from 4.25 to 10 per cent. of alumina. It is evident that the separation is sufficiently complete for technical purposes.

OLIVER IRON MINING CO.,
HIBBING, MINN.

[1] One-fourth gram of potassium chlorate added at this stage will give slightly higher alumina results, which are also more concordant with fusion alumina. No time is lost, since the seven or eight minutes allotted will suffice to remove chlorine from the solution as well as alumina from residue.

[2] Campbell and Hess: This Journal, 21, 776. E. T. Allen: Ibid., 25, 423.