THE DETERMINATION OF GRAPHITIC CARBON IN CAST AND PIG IRON.

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GRAPHITIC carbon in cast iron is usually determined by dissolving the iron in dilute nitric acid and estimating the residual graphite by combustion, either by the wet or the dry-method; or by drying and weighing direct on weighed paper filters, burning off the graphite and paper and subtracting the weight of the ash and silicon.

The combustion method, while generally considered to be the most accurate, requires considerable time, great care, and close attention, and even then failures are common. For these reasons it is not well adapted to the requirements of a laboratory in connection with foundries and manufacturing plants, where time is an important consideration. In these cases the weighed paper method is generally resorted to, though, in a less measure, it is open to the same objections as the combustion method. Aside from the time required for two dryings in the air-bath of an hour to an hour and a half each, and three weights with time for cooling each time, one must take care that the paper is thoroughly dried without being charred and that no loss occurs in the transfers of the filter-paper from funnel to weighing-bottle and back again.

Under the conditions there appeared to be no reason why the graphite could not be filtered, burned off and determined by direct loss on a Gooch. At first, in order to make the experiment inexpensive, a porcelain Gooch was used, but it was found to be very difficult to get sufficient heat through the thick and comparatively clumsy porcelain crucible to burn off all the graphite. Furthermore, when the attempt was made to ignite the crucible and freshly prepared asbestos felt previous to filtering the graphite, the bottom of the crucible usually broke off. Being convinced that the method was practicable, however, a platinum Gooch was obtained and this difficulty overcome.

Then much annoyance was experienced in filtering. When the iron contained much silicon, enough would separate with the graphite to clog the asbestos felt, and hinder the filtration, sometimes even making it impossible. As hydrofluoric acid had

been used to eliminate the silicon in the determination of manganese, it was tried for the same purpose in the graphite. The result was very satisfactory.

The silicon was disposed of, and the filtration made easy and rapid. Also many determinations can be made on the same asbestos felt while in the first case only one could be made without changing the felt. It remained to be ascertained whether the hydrofluoric acid had any effect on the accuracy of the result in any way.

To determine this point a number of analyses were made in pairs, in one case with hydrofluoric acid and in the other without. The results checked closely and proved that the hydrofluoric acid did not affect the accuracy of the method.

Determinations by this method with and without hydrofluoric acid were also checked with duplicate samples by the paper method. The results agreed closely. In fact, in some cases where a number of determinations were made on one sample, those by the Gooch showed more uniformity than those by weighed papers. This was attributed to the fact that the weight of the paper may vary before and after filtering. The details of the method are as follows :

One gram of pig or cast iron is dissolved in nitric acid (sp. gr. 1.12), better without boiling. When solution is complete, or nearly so, a few drops of hydrofluoric acid are added, more or less according to the amount of silicon present, and boiled a short time.

This boiling will drive off the hydrofluoric acid or nearly so, and insure complete solution of the iron. The acid solution is then diluted with four or five times its volume of water and filtered by slight suction on a Gooch.

Care should be taken in the selection of asbestos for this purpose. It should be digested in hydrochloric acid and, in case of a freshly prepared felt, it should be ignited until there is no further loss of weight, before the graphite is thrown on it.

After the first determination it is only necessary to wash it each time. This is advisable as each ignition has a tendency to open up the felt, and the washing under suction settles the asbestos down in place and makes as good a filtering medium as in the first determination.

It also washes out any fine particles of asbestos which might possibly be lost in the subsequent ignition. After washing with hot dilute hydrochloric acid and hot water, the crucible with contents is dried an hour or an hour and a half at 120°, the length of time it is dried depending somewhat on the amount of graphite.

The crucible is then cooled, weighed, the graphite burned off, and the crucible cooled and weighed again, the difference between the two weights being the graphite.

The advantages of the method are, a very considerable saving of time, less liability to error by doing away with the paper filter, and the avoidance of the necessity of such close attention since a variation in the temperature of the air-bath that would be fatal to a paper filter will not affect the graphite. Also, in the absence of the paper, the air-bath can be run at a much higher temperature, thus making the thorough drying of the graphite certain.

A few experiments were made with samples containing about three per cent. of graphite to determine the loss by boiling in nitric acid.

It was found that when using nitric acid (sp. gr. 1.20) and boiling from one to three hours, there was a loss of from 0.16 per cent. to 0.32 per cent. In using nitric acid (sp. gr. 1.12), however, the graphite was apparently not affected when boiled one hour after the iron was dissolved and the solution ready to filter.

In some cases there was a loss of a few hundredths of a per cent. and in others a slight gain. In all cases, however, the difference was within the limit of the legitimate error of manipulation and simply showed that there was no oxidation of the carbon by the nitric acid.

From these experiments it was pretty clearly demonstrated that there is no loss of graphite when using nitric acid (sp. gr. 1.12), even if the boiling is continued much longer than necessary.