device consists simply of an ordinary Argand gas burner with chimney, as made for illuminating purposes, with the addition of a simple hood or tent of asbestos and sheet-iron to go over the top of the chimney and confine the heat. It is surprising what a wide range of temperatures this simple apparatus gives command of. It is very perfectly adapted for the ignition of antimonous sulphide in carbon dioxide, an operation which can be carried out with great nicety at 400°, but which is difficult and uncertain when a Bunsen burner is used as the source of heat. Many other operations, distillations, digestions, etc., are carried on advantageously in this way, the great merit of the arrangement consisting in the superior control of the temperature. It is, for example, well adapted to the conversion of calcium oxalate into carbonate.

THE DETERMINATION OF GRAPHITE IN PIG-IRON.[1]

BY P. W. SHIMER.

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THE purpose of this note is to call attention to a source of error in the determination of graphitic carbon, made by the usual method of solution in hydrochloric acid. Although the method is tedious, because of the necessary treatment of the separated carbon with caustic potash, alcohol and ether, the text-books seem to give it preference; and it is, perhaps, used more generally than the method of solution in dilute nitric acid. Solution in hydrochloric acid usually gives higher graphitic-carbon results than solution in nitric acid, and many, therefore, consider it more trustworthy, the inference being that the lower results obtained by nitric acid are due to the loss of some of the finely-divided graphite by reason of the oxidizing action of the solvent. But, experiments made in Dr. Drown's laboratory, about seventeen years ago, showed no appreciable oxidation of graphite in the fifteen or twenty minutes' boiling required for the solution of a sample of pig-iron.

The point I desire to bring out here is, that the high results in graphitic carbon obtained by solution in hydrochloric acid are due to the presence, in the graphitic residue, of titanium carbide,[2]

[1] To be read at the Atlanta meeting of the American Institute of Mining Engineers.

[2] See *Trans. Am. Inst. Min. Eng.*, 15, 455.

and possibly of other insoluble carbides, the carbon of which is, of course, included with the graphite in the final determination. In the nitric acid method, the titanium carbide is easily dissolved, and its carbon appears with the combined carbon, when the latter is determined by difference between graphitic and total carbon.

The method by solution in dilute sulphuric acid is open to the same objection as that by solution in hydrochloric acid; for titanium carbide is insoluble in sulphuric acid, and, I may add, it is also unattacked by hydrofluoric acid, and by a boiling solution of caustic potash.

The following is an analysis of a pig-iron unusually high in titanium:

	Per cent.
Silicon	3.650
Phosphorus	1.145
Sulphur	0.010
Manganese	0.226
Graphitic carbon	3.206
Combined carbon	0.128
Titanium	0.399
Iron (by difference)	91.236
	100.000

The total carbon in this iron was determined by dissolving in an acidified solution of double chloride of copper and potassium, and subsequent combustion. The graphite was determined by solution in dilute nitric acid and combustion. A determination of graphite, made by solution in hydrochloric acid and combustion, gave 3.327 per cent. of graphite, a result 0.121 per cent. higher than that obtained by the nitric acid method. The amount of carbon combined as titanium carbide (TiC) with 0.399 per cent. of titanium is 0.1 per cent., which counts as graphite in the determination by solution in hydrochloric acid. The results may be set down as follows:

	Per cent.
Total carbon	3.334
Graphite by nitric acid solution	3.206
Graphite by hydrochloric acid solution	3.327

The error in the hydrochloric acid method falls heavily upon

the resultant estimate of combined carbon, which is determined by difference, as appears below :

	Per cent
Combined carbon, when graphite is determined by nitric acid	0.128
" " " " hydrochloric "	0.007

An experiment was made to determine the action of boiling nitric acid (1.20 sp. gr.) on the graphite from this iron. A sample of two grams was dissolved in hydrochloric acid (1.10 sp. gr). After washing the graphitic residue with water, it was boiled gently for one hour with nitric acid (1.20 sp. gr.), with the addition of a little water from time to time, to keep up the bulk of the solution. The graphite as thus determined, was 3.203 per cent. against 3.206 per cent., by direct solution in nitric acid, showing that the treatment, for one hour, with boiling nitric acid, had dissolved out the titanium carbide without having attacked the graphite. The graphite in this high silicon iron, however, was coarse and perhaps unusually resistent to the oxidizing action of nitric acid. It is proposed to make similar experiments on the graphite from a variety of pig-irons.

The 0.128 per cent. of combined carbon is made up as follows :

	Per cent.
Carbon combined with 0.399 per cent. titanium as TiC	0.100
Combined carbon soluble in hydrochloric acid (probably combined with iron and manganese)	0.007
Carbon possibly existing as insoluble carbide other than titanium carbide	0.021

A careful mechanical separation of a few grams of titanium carbide was made from several pounds of this iron by use of the long, slightly inclined glass plane described in the paper before the Institute referred to above.

Besides titanium and carbon in this separation, there is some vanadium, apparently also existing as an insoluble carbide, which would account for a part of the above 0.021 per cent. of combined carbon. This investigation is, however, still under way.

The writer has never encountered a pig-iron free from tita-

nium, the amount found varying usually from 0.05 to 0.40 per cent. In irons with a coarsely crystalline fracture, the cubical crystals of titanium carbide may always be found when carefully looked for. The conclusion seems to be fair that the hydrochloric acid method includes, with the graphite determined by it, the carbon existing as insoluble titanium carbide. With pig-irons containing from 0.05 to 0.40 per cent. of titanium, the graphite so determined will be from 0.013 to 0.100 per cent. too high, while the combined carbon will be correspondingly low.

It follows that more light would be thrown upon the condition of the carbon in pig-iron by making three determinations: *viz.*, one of total carbon, one of the carbon insoluble in hydrochloric acid, and one of graphite by the nitric acid method. We would thus have determinations of graphitic carbon; carbon combined with iron and manganese, soluble in hydrochloric acid; and carbon combined as carbides insoluble in hydrochloric acid. In high-silicon, low-sulphur titanic irons, the insoluble form of combined carbon exceeds the carbon existing as soluble carbides of iron and manganese. It is important to know how the carbon is combined. One-tenth per cent. of carbon combined with titanium in the condition of disseminated microscopic crystals, probably has no effect on the hardness of pig-iron; while the same amount of carbon, combined with iron and manganese, would have an appreciable hardening effect. Practically, therefore, it may be desirable to have the carbon existing as carbides insoluble in hydrochloric acid appear with the graphite as determined by the hydrochloric acid method, although the actual graphite can be determined only by solution in nitric acid. At all events it is essential to know by what method graphite has been determined, in order to draw conclusions from determinations of graphitic and combined carbon in pig-iron.

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