XII.—*On the Determination of the "Total Carbon" in Cast-Iron.*

By ARTHUR H. ELLIOTT.

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SOME time ago, having occasion to determine the "total carbon" in some cast iron, I had much difficulty in finding a thoroughly convenient process. The following method, principally adapted from Ullgren (*Ann. Ch. Pharm.* cxxiv., 59), gives satisfactory results, and is easily worked.

Preparation of the Specimen.—Bore the iron with a drill; powder the borings in a porcelain mortar, picking out any large pieces and rejecting them.

Separation of the Carbon from the Iron.—Take a weighed quantity (from 2 to 2.5 grms.) of the powdered iron; treat it with 50 c.c. of a solution of sulphate of copper containing one part of the pure crystallised salt in 5 parts of water; and heat very gently for about 10 minutes. The iron will dissolve and metallic copper separate, the carbon remaining undissolved. The reason why sulphate of copper is used in preference to chloride of copper is, that it does not contain free acid, which, if present, would cause an evolution of hydrocarbons, and consequently a loss of carbon.*

Now add 20 c.c. of a solution of chloride of copper containing 1 part of the salt in 2 parts of water, and 50 c.c. of strong hydrochloric acid, and heat the mixture at a temperature near the boiling point for some time, until the separated copper has dissolved. Collect the carbon on a filter made of a piece of rather wide combustion-tube about 15 centimetres long, one end of which is drawn out to a point 4 millimetres wide, and stopped, first with broken glass (angular pieces about the size of small peas answer best), and then *loosely* with ignited asbestos. To see whether any carbon passes at first, mix the deeply-coloured solution with strong hydrochloric acid (to prevent the separation of basic chloride of copper), and then dilute with water; by this means any carbon that may have passed will be rendered visible; if the solution contains carbon, it should be boiled and passed

* The smell obtained when heating with sulphate of copper, is due to a small quantity of phosphoretted hydrogen.

through the filter again. Finally wash the carbon with boiling water till free from chlorides.

Conversion of the Carbon into Carbonic Acid, and Determination of the latter.—Cut the tube containing the carbon at about 2 or 3 centim. above the latter, and transfer the contents to the flask by blowing carefully into the pointed end. Wash any particles that may remain in the tube into the flask with a very fine jet of water, so as to use as little water as possible;* now add about 3 grm. of chromic acid, and attach the flask to the rest of the apparatus.

The apparatus is represented in the annexed woodcut. *a* is a tube containing soda-lime, attachable to the funnel tube *b* by a cork. *b* is provided with a glass tap. The flask *c* should hold about 200 c.c., and the bottle *d* about 60 c.c.; the latter is one-third filled with strong sulphuric acid. The U-tube *e* which is about

TOTAL CARBON IN CAST-IRON.

30 centimetres high, and about 2 centimetres in internal diameter, is filled with pumice† saturated with sulphuric acid. The sulphuric acid added to saturate the pumice should not stop the passage at the bottom of the U-tube, as this unnecessarily increases the

* If more than 15 c.c. of water is used in washing, proportionately more sulphuric acid must be added to the flask when heating.

† Previously freed from hydrochloric and hydrofluoric acids, by heating with sulphuric acid, washing and drying.

pressure to be overcome by the evolved gas. The remaining tubes are for absorbing the carbonic acid: *f* is 15 centimetres high, and 1.5 centim. in internal diameter, it is filled with good soda-lime; *g* is 9 centimetres high, and 8 millimetres in internal diameter, this is filled with pumice* saturated with sulphuric acid. Sulphuric acid is used in preference to chloride of calcium, because the latter substance does not seem competent to stop all moisture from passing it. The flexible joints must be bound with copper wire.

All being ready, add to the contents of the flask about 30 c.c. of strong sulphuric acid, through a small funnel placed in the neck of the funnel tube *b*, a small quantity at first; then shake the flask so as to mix the contents completely, and add the rest. Shake again, close the tap of the funnel-tube, and heat the flask gently till it boils. While heating the flask, the gas should not be allowed to pass the bottle *d*, faster than three bubbles in a second. When the contents of the flask boil, keep boiling for about a minute, then open the tap of the funnel-tube, remove the heat, and attach the soda-lime tube *a*. Having done this, attach an aspirator to the U-tube *g*, and draw air through the apparatus at the rate of two or three bubbles per second.

The sulphuric acid in the bottle *d* will last for three, and the soda-lime in *f* can only be trusted for two analyses; the large U-tube and the other soda-lime tube will last for about six analyses.

Test Analyses.—5 analyses of one specimen of iron gave respectively—3.40, 3.40, 3.38, 3.39, and 3.40 per cent. of carbon.

School of Chemistry,
Great Marlborough Street.

The President remarked that in preparing the specimen of iron for analysis, a selection had been taken from the borings. Now all iron is of so complex a character, that in rejecting any part of a given sample, there is a danger of rejecting a portion of the iron having a different proportion of the metal to the carbon as compared with the rest. This, however, has nothing to do with the method of analysis adopted when the specimen has been well selected.†

* Previously freed from hydrochloric and hydrofluoric acids, by heating with sulphuric acid, washing and drying.

† The large pieces proposed to be rejected are those pieces which are pushed out by the point of the drill when it has nearly passed through the iron. This can be entirely obviated by not allowing the drill to bore the iron too thin.—A. H. ELLIOTT.