METHOD FOR THE DETERMINATION OF CARBON IN STEEL.

BY ANDREW A. BLAIR

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THE method given below for the determination of carbon in steel is generally used in the steel works laboratories in the Eastern part of France, and I am indebted for the details to Monsieur H. A. Brustlein of the Aciéries d'Unieux at whose words and at those of the Aciéries de la Marine at Saint Chamond the various improvements in the method have been worked out.

The method was first suggested by Wiborg[1], but was very imperfect in its original form. The greatest improvement was suggested by Monsieur de Nolly of the Laboratory of the Aciéries de la Marine at Saint Chamond and consists in the addition of phosphoric acid to the oxidizing mixture by which the iron is much more rapidly dissolved and the use of a considerable amount of chromic acid is rendered possible without the evolution of a large volume of oxygen gas.

The solutions employed are :

1. A saturated solution of chemically pure cupric sulphate.
2. An aqueous solution of chromic acid (one gram chromic acid to one cc. water).
3. A mixture of sulphuric, phosphoric and chromic acid made up as follows.

Solution of chromic acid (Sol. No. 2)	35 cc.
Water	115 "
Concentrated sulphuric acid	750 "
Phosphoric acid 1.4 sp. gr.	315 "

In preparing solution No. 2, add a few cc. of sulphuric acid and heat to boiling to destroy any organic matter that may be present.

In preparing solution No. 3, heat it to boiling also for the same purpose.

The apparatus as shown in the sketch consists of a round bottom Flask *A* of 200 cc. capacity with a long neck. The flask is closed with a rubber stopper with two holes, in one of

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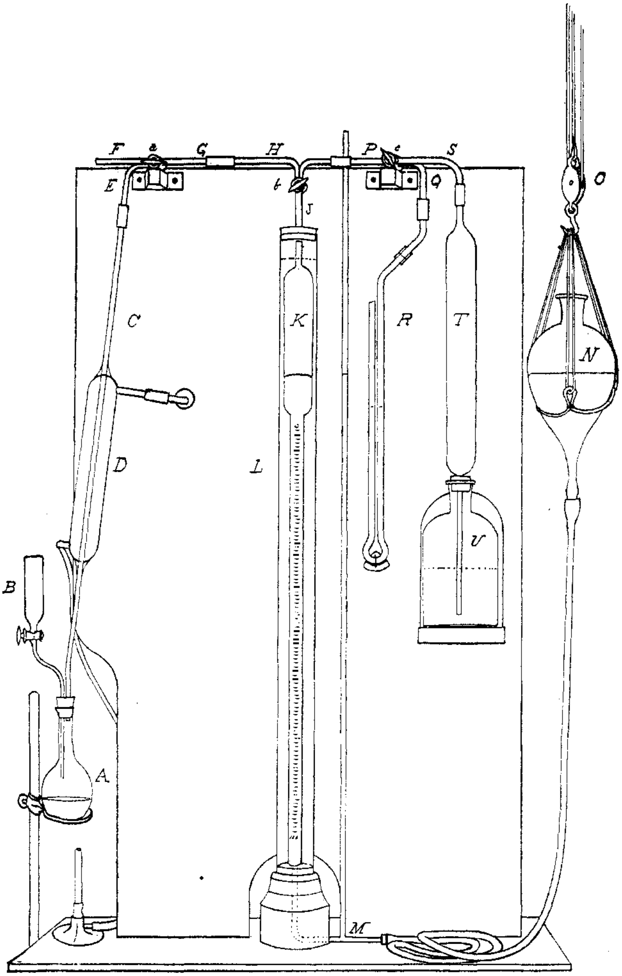
[1] Stahl und Eisen, 1882.

which is fitted the glass stopper funnel *B* and in the other the tube *C* enclosed in the condenser *D* through which a stream of water runs during the operation. The tube *C* is connected with



one tube *E* of a three way stopcock *a*, from which the second tube *F* opens into the air and the third *G* connects with the tube *H* of the three way stopcock *b*. The second tube *J* from this stopcock is fused to the burette *K* which is enclosed in the tube *L* containing water. The lower end of the burette connects with a capillary tube *M* which serves as a level tube and is in the form of a T; it is connected with the mercury reservoir *N* which is raised and lowered by the arrangement *O*. The third tube of the stopcock *b* connects with the tube *P* of the stopcock *c*, the second tube *Q* of the stopcock *c* connects with the manometer *R* and the third tube *S* with the pipette *T* which runs into the bottle *U*. The tubes of the stop cocks *b* and *c*, the manometer tube *R*, the level tube *M* and the tubes of the pipette *T* are capillaries. The manometer tube *R* contains water and serves to accurately adjust the levels when taking the reading of the burette *K*. When the manometer is shut off from the burette the approximate level is ascertained by means of the level tube *M*. The tube *F* of the stopcock *a* is used only in exceptional cases: First, when the evolution of gas is insufficient to carry the mercury far enough down the burette *K*, in which case air is drawn through it into the burette; and secondly, when the evolution of gas is so great that it is necessary to make two absorptions in the pipette *T*, in which case the residue from the first absorption is discharged through the tube F. The pipette *T* contains a solution of potassium hydroxide of 1.27 sp. gr., it is of about 400 cc. capacity. The bottle *U* is of about one liter capacity. The water in the containing tube *L* serves to keep the gas in the burette at the ordinary temperature of the laboratory. It should be protected from the heat of the burner and flask by a screen.

The operation is conducted as follows:

Connect the pipette *T* by means of the stopcocks *b* and *c* with the burette *K* and, by lowering the mercury reservoir, fill the pipette with the potassium hydroxide solution, close the stopcock *c*, fill the burette *K* with mercury and close the stopcock *b*. Weigh one gram of drillings into the flask *A*, attach it to the apparatus, start the water through the condenser *D*, and connect the flask with the burette *K* by means of the stopcock *a*. Pour fifteen cc. of the cupric sulphate solution No. 1 into the funnel tube *B*, and let it flow into the flask. Allow it to act long enough to form a superficial deposit of copper on the drillings (one or two minutes is sufficient) then add, through the funnel tube, fifteen cc. of solution No. 2 and 135 cc. of solution No. 3. Heat the solution in the flask and raise it slowly to the boiling point. By means of the reservoir, keep the mercury in the burette and in the tube *M* nearly level. The water condensed in the tube *C* drops back into the flask and keeps the liquid of the same density, while the properly cooled gases pass into the burette.

Allow the flask *A* to cool for about five minutes and then run into it, through the funnel tube *B*, enough water to fill the flask and the tube to the stopcock *a*, thus forcing all the gas into the burette. Close the stopcock *a* and connect the burette by means of the stopcocks *b* and *c* with the manometer *R*, adjust the levels accurately and take the reading of the burette. Then by means of the stopcock *c* connect the burette with the pipette *T* and by raising and lowering the reservoir *N*, pass the gas several times back and forth to cause the potassium hydroxide to absorb all the carbon dioxide. Finally connect the burette with the manometer tube *R*, adjust the levels and take the reading of the burette.

The burette *K* should contain a few drops of water to insure the saturation of the gases with aqueous vapor. The difference between the two readings is the volume of the carbon dioxide. Observe the readings of the thermometer and barometer and reduce the volume of the carbon dioxide to that which it would occupy in the dry state at 0°C. and 760 mm. pressure.

Multiply the volume of the gas so obtained by 0.0019663 and the result is the weight of the carbon dioxide in grams.

I have constructed the apparatus as here described, and am satisfied that the results obtained by its use when the details of the method are carefully carried out are much more accurate than those arrived at by any other than the combustion method. Blank determinations at Unieux rarely give more than one-tenth cc. or two-tenths cc., when the solutions are properly purified. As results may be obtained in an hour and a half, the method should recommend itself to the chemists of steel works where determinations of carbon are called for in treated and special steels where the color method is inadmissable.

It may be noted here that the use of a condenser and the solutions given above will add to the accuracy of the method in which the carbon dioxide is weighed instead of measured.

LABORATORY OF BOOTH, GARRETT & BLAIR,
PHILADELPHIA.

A NEW SAFETY DISTILLATION TUBE FOR RAPID WORK IN NITROGEN DETERMINATIONS.

BY CYRIL G. HOPKINS.

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IN doing a large amount of work with fodders and fertilizers, involving several hundred determinations of nitrogen (by the Kjeldahl method), I have observed that the distillation tube (generally known as "Kjeldahl's connecting bulb tube," but doubtless more properly as Reitmeir's distillation tube) is frequently a source of error, due to the fact that it allows fixed alkali to be carried over mechanically. Especially is this the case when the distillation is carried on rapidly, and also when the contents of the distillation flask has a tendency to "bump."

In doing rapid work I have always found it necessary to have the lower end of these tubes reground before they would allow the liquid condensed in the bulb to run back into the distillation flask. Even after this is properly done the lower end of the tube is still open, and when the liquid boils violently or "bumps," small particles of it are often thrown up through the tube into the bulb. Sometimes these particles of strongly alkaline liquid strike the curved tube within the bulb, flow out to the end, and are forced into the tube and over into the condenser by the rapid current of vapor. This action is easily observed when the contents of the distillation flask is highly colored; and I find that the occurrence is familiar to chemists who have had much to do with nitrogen determinations.

I have obtained good results, however, by having the end of the tube within the distillation flask properly ground, and then protected by a larger glass tube, fastened to the distillation tube just below the rubber stopper, by means of a short piece of rubber tubing, and having an opening on the side and a jet at the lower end. This modification gave such good satisfaction that it finally led me to make a new distillation tube in which