Bromine is not a suitable precipitant for proteids in vegetable materials.

The Stutzer method seems to be the method open to the fewest objections.

Acknowledgment is due Mr. H. W. Primrose, formerly assistant chemist, for assistance in the analytical work.

The above investigation was carried out in the laboratory of the North Carolina Agricultural Experiment Station with the permission of Professor W. A. Withers, chemist.

DETERMINATION OF CARBON IN FERROCHROME.

BY A. A. BLAIR.

Received August 14, 1900.

THE method in general use for the determination of carbon in ferrochrome may be briefly described as follows: Place 1 gram of the finely ground ferrochrome in a porcelain or platinum boat with 25 grams of fused potassium bisulphate and insert the boat in a porcelain tube in a gas furnace. Fit each end of the tube with a rubber stopper carrying a glass tube, and fill the forward part of the tube with lumps of cupric oxide. Connect the tube in the forward stopper with a U-tube containing strong sulphuric acid and chromic acid, a second U-tube containing dry pumice, a third containing dried, not fused, calcium chloride, the weighed absorption apparatus, and a guard tube. Connect the tube in the rear stopper with sources of purified oxygen and air. Start the oxygen through the apparatus and heat the tube carefully, beginning at the forward end which contains the oxide of copper, until the entire length of the tube inside the furnace is at a dull red heat in order to fuse the contents of the boat. Replace the oxygen with air, detach and weigh the absorption apparatus.

This does not seem very troublesome, but in practice several difficulties arise that make the method not only unsatisfactory, but very unreliable.

The sulphuric acid, both that evolved from the potassium bisulphate as sulphuric acid, and that evolved as sulphurous acid and oxidized to sulphuric acid by the oxide of copper and oxygen, acts on the rubber stoppers and sometimes carbonizes them sufficiently to give results several times greater than the actual carbon content of the ferrochrome. The spattering of the bisulphate, no matter how carefully the heat is applied, generally covers the inside of the tube around the boat and cements boat and tube together. The absorption of sulphuric acid by the oxide of copper causes the latter to swell, usually breaking the tube in the second, and sometimes while it is cooling, in the first determination.

To avoid these difficulties and sources of error, I have devised the apparatus shown in the cuts.

Fig. 1 shows the platinum boats and cover. The smaller boat, 150 mm. long and 25 mm. wide, fits inside the larger and the cover is so arranged that any particles from the melting mass thrown on it run into the larger boat and thus keeps the tube itself perfectly clean.

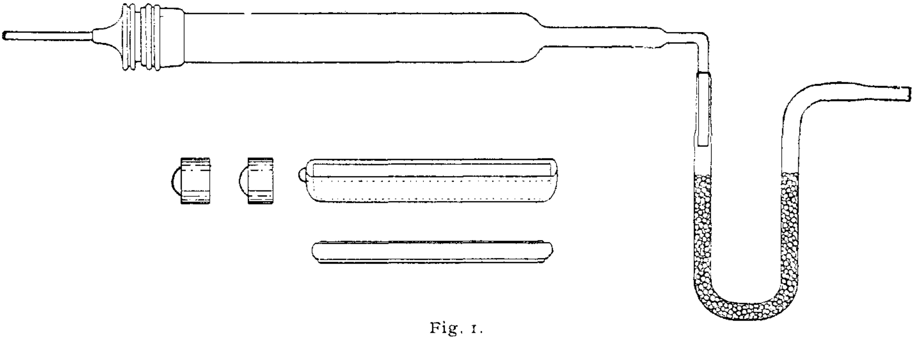

Fig. 1.

The platinum tube, shown also in Fig. 1, is 400 mm. long and 30 mm. in diameter and is closed with a ground joint at the rear. The forward end for a distance of 75 mm. is contracted to 12 mm. and filled with platinized asbestos. It is then further contracted to 6 mm. in diameter, and a piece of glass tubing filled with glass beads is fused to it after it is bent downwards at an angle of 90°. The platinized asbestos facilitates the oxidation of the sulphurous acid evolved from the fused mass. The plugs are made of pumice wrapped with platinum foil, and are pushed in after the boat. They serve to fill the back end of the tube and prevent the diffusion of the evolved gases and consequent condensation of sulphuric acid around the ground joint.

Fig. 2 shows the general arrangement of the apparatus. The purifying apparatus for oxygen and air is shown in detail in Fig. 3. The tubes A and B contain respectively potassium hydroxide and concentrated sulphuric acid. It is almost impossible with this form of tube to throw out the contained liquid either forward or backward. In Fig. 2, A is the platinum tube, and B the glass tube containing beads fused to the contracted end of the platinum tube at C. D, D are glass flasks containing a solution of 150 grams of chromic acid and 300 cc. of strong sulphuric acid to the liter. These flasks stand on a copper plate and are heated by a Bunsen burner. The solution serves to oxidize any sulphurous acid that may have escaped oxidation in the contracted part of the platinum tube. E is a U-tube filled with glass beads, and acts as a condenser; F contains pumice saturated with chromic acid, and G contains dried calcium chloride. The absorption apparatus and guard tube follow. The latter is connected by a rubber tube with the gasometer, shown in the cut, which acts as an aspirator and serves to relieve the pressure in the apparatus, which, on account of the condensation of strong sulphuric acid in the tube B and the high spe-

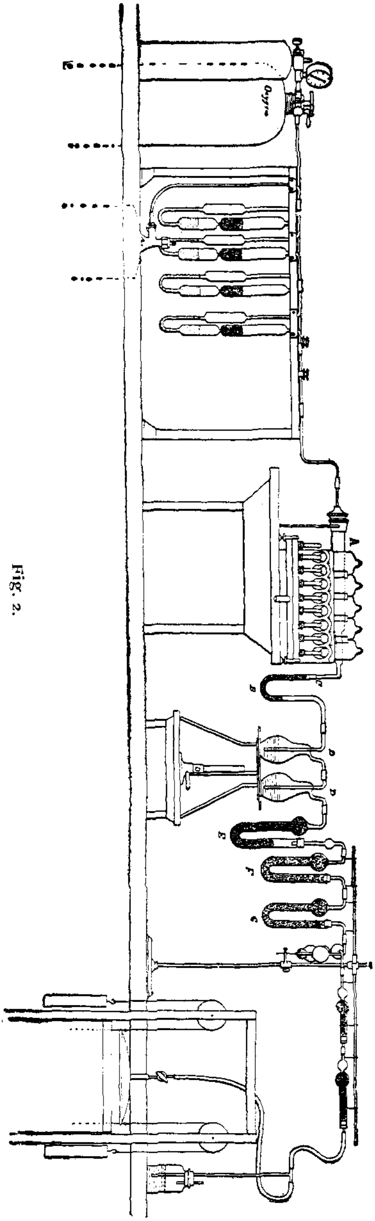

Fig. 2.

cific gravity of the liquid in the flasks D, D, would otherwise be excessive. The details of this connection are shown in Fig. 4.

The method is as follows: Place 25 grams of pure potassium bisulphate in the small boat (Fig. 1), and fuse it over a Bunsen burner or blast-lamp to destroy any carbonaceous matter and allow it to cool. When cold spread 1 gram of the finely ground sample evenly over the surface of the fused mass, place the boat inside the larger boat, arrange the cover, place the boats in the tube, insert the plugs and close the tube with the ground joint. Connect the apparatus as shown in Fig. 3, and start a slow current of oxygen through the apparatus. Light the burners under the forward end of the tube which contains the platinized asbestos, and when this is red hot light the burner under the forward end of the boat and light the others successively until the tube is red hot for its entire length where the boat rests. Keep the tube hot for twenty minutes, replace the oxygen with air, turn out the lights, allow the air to run about thirty minutes and detach and weigh the absorption apparatus with the usual precautions.

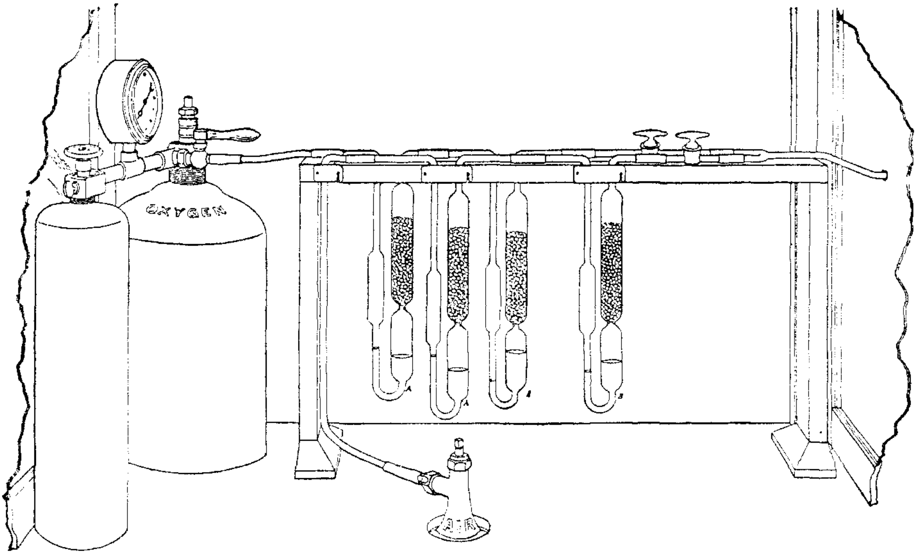

Fig. 3.

As a large amount of sulphurous acid is produced it is necessary to heat the tube very gradually in order to keep an excess of oxygen in the tube to oxidize all the sulphurous acid. As the sulphuric anhydride does not condense readily, a slow current of gas is requisite, and a combustion requires about two hours and a half.

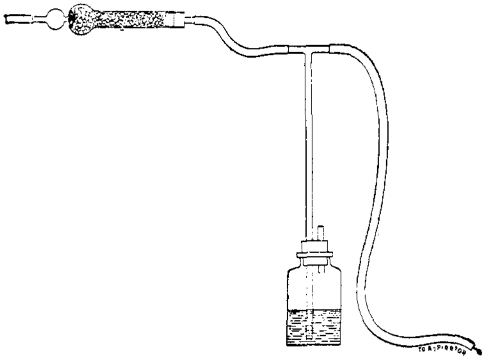

Fig. 4.

At the end of the operation the tube is opened and the plugs and boats removed. The fused mass is readily removed from the boats which, with the cover, are washed and ignited, ready for another combustion. About three determinations can be made in a day. Duplicate determinations agree within 0.01 per cent.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 51.]

THE ELECTROLYTIC OXIDATION OF TOLUENE.

BY AARON MERZBACHER AND EDGAR F. SMITH.

Received September 7, 1900.

THE oxidation of toluene by electrolytic oxygen has been tried by Jos. H. James.[1] The products were a minute quantity of liquid, with an ester-like odor, and a resin, together with unaltered toluene.

Our attention has been attracted to this interesting subject and we have repeated the experiments, varying the conditions, of course, with the hope of finally changing the methyl side-chain to carboxyl.

The apparatus used in the electrolysis was similar to that

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[1] This Journal, 21, 890.