

with water and kneading with a glass rod it became viscous and finally solidified, and after repeated crystallization from hot alcohol it melted at 125°.

Nitro-4-nitro-2'-methyl phenylether is a yellow crystalline substance which is soluble in sulphuric ether, petroleum ether, benzene, nitrobenzene and aniline. An attempt to obtain an amino derivative by reducing with tin and hydrochloric acid was unsuccessful, owing to its decomposing when an attempt was made to evaporate the solution to crystallization. The results of the analysis for nitrogen together with its mode of formation, would lead us to the conclusion that it is a dinitro methyl phenylether. Two analyses for nitrogen gave 10.55 and 10.35 per cent. Calculated, 10.22 per cent.

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THE DETERMINATION OF MANGANESE IN IRON AND STEEL.

BY JOHN V. R. STEHMAN.

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WALTERS,¹ has shown that ammonium persulphate in the presence of a small amount of silver nitrate can be employed to advantage in place of lead peroxide, for the oxidation of manganese in the colorimetric method.

The writer has been using lead peroxide as an oxidizing agent and titrating the permanganic acid, with a standard solution of sodium arsenite after first filtering free from lead oxides, through an asbestos filter.²

In attempting to replace the lead peroxide by the ammonium salt and titrating as before, the silver salt, of course, caused trouble. If, however, the silver salt is thrown out of solution before titration is begun, as insoluble silver chloride, the determination of the permanganic acid by a standard solution of sodium arsenite may be readily accomplished.

The silver salt can be precipitated by a solution of sodium chlo-

¹ *Age of Steel*, November, 1901; This Journal, 24, R. 12.

² Blair's "Analysis of Iron," third edition.

ride, giving to the solution a fine white background, in which the end-reaction is well marked. It was found necessary with pig irons, to filter free from graphite after solution, as the graphite gives to the solution a peculiar color, in which it was difficult to distinguish the end-reaction.

The following is the method adopted: Weigh off into a 6-inch test-tube 0.2 gram of the sample; add 10 cc. nitric acid (sp. gr. 1.20), heat to boiling in hot water-bath till dissolved, and free from nitrous fumes. Filter into a 10 by 1-inch test-tube (using a small filter), wash filter twice with hot water containing a little nitric acid, and add through the filter to the contents of tube, 15 cc. of a solution of silver nitrate (1.33 grams to the liter). One gram of ammonium persulphate is now added and the solution brought to the boiling-point, using a free flame; heating is continued for about one minute after oxidation begins. The tube and contents are now cooled rapidly under the spigot and poured into a No. 2 beaker, washing out the tube with 30 or 40 cc. of water; 5 cc. of a saturated solution of sodium chloride are now added to the solution in the beaker, silver chloride being precipitated immediately, and titration is begun at once with standard sodium arsenite. The sodium arsenite solution is standardized against standard permanganate or it may be standardized by an iron in which the manganese has been accurately determined. The former is the more rapid and will be found to check closely with the latter.

The ammonium persulphate, as pointed out by Walters,¹ causes complete oxidation only when damp; he therefore adds 10 cc. of water to each pound of the salt the day before used. A determination by the above method may be made in twenty-five minutes in the case of pig irons; with steels, where filtration is not necessary and the sample can be dissolved directly in a large test-tube, the method is more rapid than the color method in which lead peroxide is used as an oxidant.

Below are a few results obtained in pig irons.

¹ *Loc. cit.*

Mn present. Per cent.	Mn found. Per cent.	Mn present. Per cent.	Mn found. Per cent.
0.415	0.423
0.415	0.414
0.415	0.414	1.00	0.990
0.415	0.420	1.00	0.989
0.442	0.440	1.00	0.982
0.442	0.460	1.00	0.980
0.442	0.460	1.00	1.010

LABORATORY OF THE E. AND G. BROOKE IRON CO.,
BIRDSBORO, PA.

THE RAPID DETERMINATION OF CARBON BY COMBUSTION.

BY GEORGE AUCHY.

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SOME experiences with the dry method of combustion having led the writer to the suspicion that the employment of a rapid rate of gas flow is occasionally attended with loss on account of incomplete oxidation by the copper oxide of the products of ignition and combustion, it was thought that it might be advantageous to use the Shimer apparatus with the modification of having the copper oxide in the crucible instead of in a tube.

In this plan the products of combustion are not forced through the copper oxide by the current of oxygen, but are carried in through the apparatus by the current of oxygen *after* oxidation by the copper oxide has been effected. Therefore, the time of contact with the copper oxide is entirely independent of the rate of gas flow employed, and the carbon compounds will be just as thoroughly oxidized by the copper oxide with a fast as with a slow rate. The carbon dioxide is formed and driven to the top of the layer of copper oxide by the heat of the blast-lamp. Is this period of contact with the copper oxide sufficient for complete oxidation? If so, the rapidity of the passage of oxygen is limited only by the capacity of the potash solution to absorb carbon dioxide rapidly and completely. With solution of 1.400 sp. gr. this capacity is very great.

This plan of having the copper oxide in the crucible had been tried by Dr. Shimer himself who in his original article speaks of