CONTRIBUTIONS TO PRACTICAL RAILROAD INFORMATION.

Chemistry Applied to Railroads.

SECOND SERIES.—CHEMICAL METHODS.

VIII.—METHOD OF DETERMINING SILICON IN STEEL.

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OPERATION.

Put 2 grams of fine borings in a 12-oz. royal Berlin porcelain casserole, and add 80 c.c. of a mixture of nitric acid, sulphuric acid, and water. Cover with a watch glass and allow action to cease. Then evaporate, if desired, over a lamp direct, until sulphate of iron begins to separate, then transfer the dish to an air-bath, heated to about 300° F., and continue the evaporation until fumes of SO_3 are given off. Allow to cool, add 250 c.c. of distilled water, and heat carefully until the sulphate of iron has all dissolved, then filter at once. Wash with water until most of the iron salts are removed, and then with dilute hydrochloric acid, as long as the washings are colored with iron salts; finally wash with water again, until the washings no longer react for chlorine, then ignite and weigh. Treat the contents of the crucible with a little dilute sulphuric acid and a few drops of hydrofluoric acid, evaporate to dryness, ignite and weigh again. The difference between the two weights is silica.

APPARATUS AND REAGENTS.

It will be observed that a porcelain casserole is designated as the characteristic piece of apparatus for this method. Of course platinum dishes and good quality porcelain evaporating dishes may be used, but, all things considered, a casserole seems best. Glass should not be used, as some of the separated silica apparently adheres to the glass, and cannot be removed by the feather or rubber tube on the end of a glass rod. Direct experiments on the same steel, everything being exactly alike, except one determination was in a porcelain casserole and the other in a beaker, show lower results in the beaker, amounting to one or two-hundredths of a per cent. The results in the porcelain were confirmed by repeated tests in platinum on the same steel. For a single determination, the air-bath recommended in "The Chemical Analysis of Iron," by A. A. Blair, 2d edition, p. 20, is very satisfactory. For a number of determinations at once, an oven gives excellent results.

The mixed acids for solution are made by adding 20 c.c. of concentrated C. P. nitric acid to 40 c.c. of distilled water, and then adding to this 20 c.c. of concentrated C. P. sulphuric acid. Where a number of determinations are to be started at the same time, it is more convenient to mix the quantity of acids required all at once.

The dilute hydrochloric acid for washing is made by adding one part concentrated C. P. acid to four parts of distilled water.

Hydrofluoric acid of good quality, practically free from residue, can now be obtained in the market, in ceresine bottles.

CALCULATIONS.

Atomic weights used, oxygen, 16; silicon, 28; molecular formula, SiO_2. Since 46.67 per cent. of the SiO_2 is silicon, if the weight found is multipled by this figure, the result will be the silicon in 2 grams of steel, and this figure multiplied by 100 and divided by two obviously gives the percentage. This may be simply stated in the following rule: Express the weight of SiO_2 found, in grams, remove the decimal point two places to the right, and multiply by 0.2333. The product will be the percentage of silicon in the steel. Thus if the silicic acid found is 0.0027 gram, the silicon is (0.27×0.2333) 0.063 per cent.

NOTES AND PRECAUTIONS.

It will be observed that this method oxidizes the silicon in the steel by means of nitric acid, and then dehydrates the silicic acid formed by means of concentrated sulphuric acid, so that it can be caught in a filter, finishes the dehydration by ignition and weighs up as SiO_2. Any residue of iron or other material not washed out is left behind after the treatment with hydrofluoric acid. There is considerable evidence that the SiO_2 is not completely dehydrated in the sulphuric acid by this method. After the water is added and the iron salts are in solution, the appearance of the silica in the liquid is more or less gelatinous, also, as is mentioned below, the SiO_2 goes into solution again under certain conditions. It is believed that if the directions are carefully followed, the results will be accurate to within perhaps half a hundredth of a per cent.

The reason for the use of a porcelain casserole has already been given.

The mixed acid gives exactly the same results as though the steel is dissolved in dilute nitric acid, and then dilute sulphuric acid added. It simplifies the manipulation a little to add the acids all at once.

A careful manipulator may succeed in evaporating over the lamp direct until the sulphuric acid fumes strongly, especially if the material is stirred continuously, but after the sulphate of iron begins to separate there is much danger of loss by spirting. The air-bath is much more sure. When a number of determinations are carried on at the same time and there is no great hurry, excellent results may be obtained by adding the mixed acids to the borings, putting the casseroles at once on a steam plate whose temperature is about 275° F., and allowing them to stand without further manipulation over night. Where the air-bath and shorter time are employed, the casserole should be set down into the air-bath, below the line of the liquid inside. With the air-bath or on the steam plate, stirring is not essential.

It has been proposed to add Nordhausen sulphuric acid to the dish after the principal part of the nitric acid has been driven off, to get the strong sulphuric acid necessary to dehydrate the SiO_2 and thus to save the time required to concentrate the sulphuric acid to the proper point. Our experience with this modification is that the Nordhausen of the market is rarely pure enough to be trusted, while if Nordhausen is made by adding SO_3 to concentrated C. P. acid, there is considerable difficulty in the manipulation of the SO_3. That which comes in tin cans is very difficult to open and put into the strong sulphuric, without at the same time being contaminated, while if that in glass bulbs is used there is much danger that some of the glass of the bulb will get into the determination. The time and labor saved by this modification is not great.

It will be observed that directions are given to filter at once, after the iron salts are dissolved. Direct experiments following the manipulation given above show that after the water is added and the iron salts are in solution, if the material is allowed to stand before filtration 24, 36, or 48 hours, quite large amounts of the silica are redissolved and lost. This statement has been disputed, but our own experiments have been confirmed by other workers. It seems probable that a few hours' standing would make very little difference, but as there seems to be no good reason for dilution until one is ready to filter, we have not thought it worth while to study the cause of the discrepancy in the statements. The manipulation which we recommend is certainly the safer.

The use of half strength hydrochloric acid has been recommended to wash out iron salts. The experiments of some chemists seem to indicate that the silica obtained as above is perceptibly soluble in hydrochloric acid of this strength. As the iron salts seem to be completely removed by the more dilute acid, we prefer to use it as a precautionary measure.

If the dilution and washing have been managed with care, the silica obtained is generally perfectly white, and the residue after the treatment of hydrofluoric acid is very small. If there is a tint of iron oxide the residue will be larger. In no case should the hydrofluoric acid treatment be omitted when determining silicon in steel.

MASTER CAR BUILDERS' CONVENTION.

The twenty-eighth annual convention of the Master Car-Builders' Association was opened on the morning of June 12, at Saratoga, N. Y., with President Grieves, of the Baltimore & Ohio Railroad, in the chair. The preliminary business, including the President's address, Secretary's and Treasurer's reports, was transacted in the new convention hall. A *résumé* of the Secretary's report shows that there are now 326 members of the Association, including active, representative and associate members. The Treasurer's report showed that the expenditures made during the past year were $8,018.84, leaving a treasury deficit of $21.30. In the report of the Executive Committee a suggestion was made that the committee be empowered to bring the subject of the Master Car-Builders'