

external diameter a little less than half the difference between the internal diameter of the large jar and the external diameter of the small one, and should extend ten cm. above the top of the outer vessel, serving instead of the flanges and grooves. A glass stop-cock with as large capacity as possible is clamped rigidly in convenient position above the apparatus, and to it a lever arm of light wood is bound. To the end of the arm another light sliver or a wire is attached, which rests on the float and operates the valve.

Operation of regulator is as follows: One of the exit tubes may be connected to a manometer, or both may go to burners as may be desired. Gas enters through the stop-cock and long tube into the container, and in so doing lifts the container, at the same time closing the valve. If outlets are closed the container will rise until the valve is entirely closed, in which position it will stand. When the exit tubes are opened the container falls, re-opening the valve, admitting gas at the same rate at which it is consumed, and delivering it at a pressure which is measured by *the weight of the inner vessel, plus or minus the resistance due to friction*. If the apparatus is well made the resistance amounts to only one or two mm. on the manometer, and it is only exerted during the changes of position of the container. If the pressure in the pipes falls to that in the apparatus, the float falls to the bottom, opening the valve to its fullest capacity, and allowing the gas to flow through unhindered.

Thanks are expressed to Mr. J. T. Faig for the sketches herewith given, and to Professors P. C. Freer and F. G. Novy for assistance rendered in various ways.

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ARBOR, APRIL 14, 1898.

## USE OF HYDROFLUORIC ACID IN THE DETERMINATION OF MANGANESE IN IRON AND ORES.

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Received May 13, 1898.

WILLIAMS' method for the determination of manganese is not faultless but is nevertheless the best method for the iron chemist who has to determine manganese in iron, steel, ferro-manganese, and ores.

It may be said, in passing, that a solution of ferrous sulphate is preferable to a solution of oxalic acid. An objection to the method, though not believed by most chemists to be valid, is that strong nitric acid and potassium chlorate do not completely precipitate manganese. As regards iron and steel, it can be said positively that strong nitric acid, free from nitrous fumes, and potassium chlorate, properly used, will completely precipitate the last traces of manganese. In regard to ores, more will be said later.

Another objection urged against this method, especially in the case of pig irons, is the delay and annoyance caused by the silicon clogging the filter and being generally troublesome.

The use of hydrofluoric acid in the determination of manganese in high silicon pig irons, is not new and is mentioned in Blair's "Analysis of Iron," second edition, page 111; but as there directed it necessitates an evaporation, which takes time. The direct application of the acid was therefore tried, and after some failures, successfully. In practice, the iron is dissolved and the manganese precipitated with potassium chlorate as in the regular method. As soon as the manganese is precipitated, which will be completed in a few minutes, a few drops of hydrofluoric acid (more or less according to the amount of silicon present) are added, boiled several minutes or until the hydrofluoric acid is evaporated. Then a little more potassium chlorate (about one gram) is added and the solution boiled as low as desired. The last addition of potassium chlorate has not been proved necessary but is added for safety. By using the acid in this manner the operation takes practically no longer than would a sample of steel containing a few hundredths of a per cent. of silicon. Not only will the solution filter rapidly and the fine silty manganese dioxide dissolve much more easily in the ferrous sulphate solution, but the graphitic carbon will be found to be nearly or entirely oxidized owing to the absence of the enveloping silica which otherwise would protect the particles of carbon from the oxidizing influence of the nitric acid and potassium chlorate.

In the case of pig iron, the benefit of the hydrofluoric acid is simply that it eliminates the silicon and consequently gets rid of a source of annoyance and delay.

A short time since, in doing some work on manganese ore, it was

discovered that hydrofluoric acid plays a much more important part than had been anticipated. In working ores it has always been found that the first addition of potassium chlorate did not completely precipitate the manganese, and after filtering, a second precipitation, and sometimes a third, was made in the filtrate. In the case of the ores above mentioned it was decided to use hydrofluoric acid on one of the samples to see if it would prove as beneficial as in the case of pig iron. On adding potassium chlorate to the filtrate, as in the other cases, no precipitate of manganese dioxide appeared, and this led to further experiments which proved, at least as far as this particular lot of ores was concerned, that the addition of hydrofluoric acid renders the first precipitation by potassium chlorate complete. In this case, as in the case of pig iron, only a few drops of acid were added a few minutes after the potassium chlorate. After a little practice one can judge by the amount of floating gelatinous silica about how much hydrofluoric acid to add. If the first addition does not clear the solution in a minute or two a few drops more can be added.

In beginning the use of the acid with pig irons, it was added to the solution and the silicon eliminated before the manganese was precipitated, as directed by Blair; but some bad results were obtained about this time, and knowing that some reagents will prevent a precipitation while they will not dissolve the precipitate after it is formed, the acid was used as above stated. The bad results may or may not have been due to this, but as it is just as easy to add the acid after the precipitation as before, it has always been so done. We have had long experience with the use of this acid in working pig iron and know it is safe and advantageous. Our work on ores is very limited, but as it appears to be even more useful in this class of work, we take pleasure in bringing it to the attention of those in this particular line of work.

In regard to the effect of the acid on the beakers, although it etches and blurs them, they have been used in this laboratory every day for a period of five or six months. The lower part of the beakers become partially opaque and not suitable for most other work, but by keeping a number of beakers for manganese determinations only, their life will be found to be very little diminished, provided the hydrofluoric acid is used carefully.