

temperatures be not expressed. Fortunately this good practice of always giving both temperatures, as $\frac{4}{4}^{\circ}\text{C.}$, $\frac{1}{4}^{\circ}\text{C.}$, $\frac{1}{15}^{\circ}\text{C.}$, is now becoming common.

THE DETERMINATION OF SULPHUR IN PIG IRON.

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THERE are two general methods in use for the determination of sulphur in pig iron; the evolution method and the oxidation method. Attention has been repeatedly called to the fact that the residue from many pig irons after treatment with dilute hydrochloric acid contained sulphur, while in some the aggregate evolved as hydrogen sulphide and remaining unacted on in the residue was decidedly less than the amount obtained by the oxidation method. The cause of this discrepancy has been clearly pointed out by Prof. Phillips¹ in his admirable paper on the "Evolution Method for the Determination of Sulphur in White Cast Iron."

Following the line of Prof. Phillips' work I found that it was possible to convert all the volatile sulphur compounds into hydrogen sulphide by passing the evolved gases mixed with hydrogen through a tube filled with pumice and heated to redness. The long boiling that proved necessary and the passage of so much distilled hydrochloric acid and water through the red hot tube made the method too troublesome for ordinary use.

During this investigation I received a sample of pig iron for the determination of its sulphur contents, and used for this purpose not only the oxidation method but the new method as well. The results were as follows:

	Per cent.
1. Sulphur by oxidation.....	0.032
2. Sulphur evolved as hydrogen sulphide.....	0.000
3. { Sulphur obtained as hydrogen sulphide after passing	
{ through red hot pumice	0.005
{ Sulphur obtained by fusion of residue.....	0.057
Total sulphur	0.062

This seemed to point to the fact that the ferric chloride in the oxidation method held barium sulphate in solution. Mr. P. W.

¹ This Journal, 17, 891.

Shimer made a careful determination of sulphur in this sample by the oxidation method and obtained 0.042 per cent. I repeated all my determinations but the results remained the same. I then made a determination by Bamber's method¹ and found 0.064 per cent. Finally, I fused the residue of carbon, silica, etc., filtered off before precipitating the sulphuric acid in the oxidation method, and obtained 0.030 per cent. sulphur unacted on by hydrochloric and nitric acids. Mr Shimer corroborated these results. In other words, we now have in pig irons :

1. Sulphur evolved, by solution of the iron in hydrochloric acid as hydrogen sulphide.

2. Sulphur evolved as other compounds not absorbed by alkaline lead salts or oxidized by bromine or potassium permanganate.

3. Sulphur in some form, unacted on by boiling hydrochloric acid, but oxidized by nitric acid or aqua regia.

4. Sulphur in some form, unacted on by nitric acid, hydrochloric acid, or aqua regia.

The simplest and most satisfactory method for the treatment of pig irons containing sulphur in the latter form is that of Bamber. It is essentially as follows: Dissolve five grams or a five factor weight (6.878 grams) in strong nitric acid, add two to five grams of potassium nitrate, evaporate to dryness in a platinum capsule and ignite. Treat with water with the addition of a little sodium carbonate, filter, and wash with water containing sodium carbonate. Acidulate with hydrochloric acid, evaporate to dryness, redissolve in water with a few drops of hydrochloric acid, and precipitate boiling with barium chloride.

The pig iron referred to above contains appreciable amounts of titanium and vanadium and Mr. Shimer and I are now engaged in studying the residue insoluble in hydrochloric acid. The results of our investigation we hope will be of sufficient interest to warrant their presentation to the Society.

¹J. *Iron and Steel Inst.*, 1894, 1, 319.