

rotation than in water, the calculated specific rotation suggesting the presence of the simple tartrate in the glycerol solution.

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## NOTE ON THE DETERMINATION OF SILICON IN STEEL.

BY GEORGE AUCHY.

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ALTHOUGH it has been mentioned by Dudley that silicon results in steel by Drown's method are apt to be low, on account of the iron sulphate enclosing and protecting the silicic acid from the dehydrating action of the sulphuric acid, it is probable that this fact is one not generally appreciated by chemists.

When the method is used for pig iron, as was originally intended by its author, there is no error from this cause less;<sup>1</sup> but in steel the loss amounts to from 0.01 per cent. to 0.035 per cent. silicon in medium silicon (0.15 to 0.20 per cent.) steels, which is too much to be ignored. Dr. Dudley thinks that it may be prevented by taking great pains to insure sufficient contact by stirring. Four experiments made by the writer, in which five minutes' stirring in each case was done after the appearance of sulphuric fumes, showed this to be true, only 0.006 per cent. silicon in each case being lost. But in a busy laboratory with many silicon determinations being made, or with much other work demanding the attention of the operator, this stirring adds considerably to the labor of the method, and the time it requires can hardly be allowed. Says Dr. Dudley: "If by some modification the iron salts could be kept in solution until the silica is rendered quite insoluble, it would apparently be a decided step forward with this method."

Several years ago the writer found that if aqua regia be used in the method instead of nitric acid alone, and the proportion of strong sulphuric acid be about 3.2 cc. per gram of drillings,<sup>2</sup> the iron salt in that case did not separate out until just a little before

<sup>1</sup> There is loss, it is true, of about 0.10 to 0.15 per cent. silicon in high silicon pig, but it is due to the same cause (whatever this cause may be) that in silicate analysis brings the result too low. It is a well-known fact that from 1.5 to 3 per cent. of the total silica of silicates remains in the filtrate from the first silica separation, no matter what method of dehydration be employed, or how many evaporations be made before filtration (Cameron: *Chem. News*, 69, 171). We should therefore expect the same proportion of loss in pig iron and steel analysis, or about 0.006 per cent. silicon in a 0.20 per cent. silicon steel, and about 0.10 per cent. in a 3 per cent. silicon pig iron.

<sup>2</sup> The liquid boiled down rapidly in a capacious covered casserole (evaporating dishes do not serve) on a very hot plate.

the appearance of sulphuric fumes, and it was thought therefore that this process favored the dehydration of the silicic acid. Later it was found, however, that with one evaporation the loss of silica was usually just as great or greater than in using the regular method.

Upon experimenting with the old Swedish method reintroduced by Dr. Kinsey, and which consists in the use of sulphuric acid alone,<sup>1</sup> without nitric or any other acid, it was found that the silica lost by incomplete dehydration in this method was usually less than 0.006 per cent. silicon and with stirring not resorted to at all.

Drown's method, silicon lost. Per cent.	Writer's method, silicon lost. Per cent.	Swedish method, silicon lost. Per cent.
0.018	0.016	0.002
0.012	0.028	0.000
0.018	0.018	0.002
0.020	0.028	0.006
0.016	0.026	0.000
0.022	0.020	0.006
0.012	....	0.006
0.034	....	0.012
0.012	....	0.008
0.014	....	....
0.018	0.028	0.006
0.018	0.022	0.000
0.028	0.022	0.000
0.012	0.034	0.012
0.032	....	0.006
0.024	....	0.006
0.018	....	0.006
0.010	....	0.004
0.030	....	0.002
0.020	....	....

The silicon percentages in these steels ranged from 0.15 to 0.25. Silicon lost in each case was determined by evaporating the filtrate. All evaporations were to dense fumes. The Swedish method seems to be in use in but a comparatively few (though large and important) laboratories, but for steel analysis deserves a wider popularity. In the case of high carbon steels the liquid in a covered dish may be boiled down rapidly on a thin steel plate, heated by a full Bunsen flame, to the end of the operation

<sup>1</sup> Hydrochloric acid doubtless serves as well.

without lowering the flame.<sup>1</sup> However the dehydration is just as complete when evaporated slowly.

The writer uses 50 cc. of dilute sulphuric acid (1 : 4) for 2.351 grams drillings steel, and observes that solution is complete before the sulphuric acid becomes concentrated. But in the determinations of silicon in steel given by Drown's method in the first table the proportion of sulphuric acid used was for part of them 10 cc. concentrated acid to 1 gram drillings and for the remainder 3.5 cc. per gram drillings. The following results from a 3.55 per cent. silicon pig iron show that in pig iron analysis as regards the point under discussion Drown's method is as effective as the old Swedish.

Drown: silicon lost, per cent.	0.168,	0.14,	0.14,	0.164,	0.133,	0.152
Swedish " " " "	0.096,	0.096,	0.096			

That these losses are due not to the iron sulphate protecting the silicic acid, but to the cause already referred to in a foot-note, is shown by the results of a number of tests made by the Swedish method and with five minutes' stirring after appearance of fumes in which the respective losses were 0.152 per cent. and 0.112 per cent. silicon; and also by another test by the Swedish method, in which two evaporations were made before filtration and in which the loss was 0.108 per cent. silicon. Incidentally it is shown by these and the above tests that the hydrofluoric acid treatment usually insisted upon should not be included in a silicon determination, unless the silica remaining in the filtrate be recovered or allowed for. The same is, of course, true also of ferrosilicons.

The following results from different ferrosilicons give some hint of the proportion these mutually opposing errors are apt to bear to each other.

The silica lost is about 3 per cent. of the total as usual. The hydrofluoric acid residues seem to be apt to run somewhat less.

Silicon lost, per cent.	0.43,	0.23
H. F. residues, per cent.	0.16,	0.24, 0.14

In the following table total silicon (silicon as usual plus silicon in filtrate) results by the Swedish method are compared with total silicon results by Drown's method.

<sup>1</sup> An impossible operation in Drown's method; impossible also, usually, in this method, in the case of low carbon steels and wrought irons. For such, it is perhaps better to use hydrochloric acid instead of sulphuric.

Steel.	Drown's method. Per cent.	Swedish method. Per cent.
A	0.190	0.190
"	0.190	0.196
"	0.196	....
"	0.192	....
B	0.178	0.184
"	0.178	0.182
"	0.182	....
"	0.190	....
C	0.204	0.202
"	0.202	0.204
"	0.202	0.200

LABORATORY OF HENRY DISSTON & SONS'  
STEEL WORKS, PHILADELPHIA.

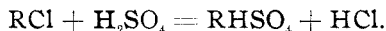
## ON THE DECOMPOSITION OF THE CHLORIDES OF ALKALI METALS.

By C. W. VOLNEY.

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### PART I.

THE action of sulphuric acid on the chlorides of the alkali metals is usually expressed by



It has been observed that concentrated sulphuric acid acts with considerable violence on the alkaline chlorides at the common temperature; that an acid, which has been diluted with one-quarter or one-third of its weight of water, acts with less violence, developing hydrochloric acid gas on the application of heat; and it is known that, if less acid than as above is used, the hydrochloric acid gas is developed only on applying considerable heat. Thus we observe that in the manufacture of sulphate by the Leblanc process, 2 molecules of salt are used with 1 molecule of sulphuric acid (60° Bé) and that, whereas, only about 70 per cent. of the hydrochloric acid gas is driven off in the pans, the mass has further to be calcined at a high temperature in the calcining furnaces, to complete the decomposition. We have, therefore, *a priori* to conclude that, in order to effect a thorough decomposition of the chloride by sulphuric acid, the application of heat is necessary; that the decomposition takes place periodically, as the temperature is raised, and that it is generally presumed that