

the slight advantage gained thereby is hardly worth the extra trouble for works practice.)

If the filter paper used to wrap the drillings, or the ferro-cyanide itself, contains sulphates, the latter are reduced during the annealing, and their sulphur given off as sulphuretted hydrogen in the evolution flask, so that a blank determination becomes necessary. The blank can be conveniently determined upon a pure iron, by dissolving 5 grammes of the iron in concentrated hydrochloric acid, and absorbing the sulphuretted hydrogen as usual. By annealing another 5 grammes of the iron in the usual way, and determining the sulphur contents as in the direct estimation, any difference due to the above causes can be determined.

The iodine and thiosulphate solutions may be standardized by means of a steel or iron, the sulphur contents of which have been accurately determined by the "aqua regia" or Bamber's method. The value of the solutions in sulphur so obtained, should agree closely with that got by calculation after standardizing the thiosulphate against a standard solution of potassium permanganate. The latter is very easily performed, and is a very useful check on the working of the process. The solutions of iodine and thiosulphate used are of such strength, that 1 cubic centimetre equals 0.005 per cent. sulphur on 5 grammes of the example.

The cadmium chloride solution is prepared by dissolving 20 grammes of cadmium chloride in water with the aid of a few drops of hydrochloric acid. Ammonia is added until the precipitate of cadmium hydrate formed, completely dissolves. Acetic acid is then added until the solution becomes slightly acid, when a further 20 cubic centimetres are added in excess, and the solution made up to 2 litres.

I find the cadmium solution acidified with acetic acid, and containing ammonium acetate, the best absorbent to use. It does not absorb the hydrocarbons, phosphides, etc., simultaneously evolved with the sulphur compounds, to the extent that solutions of salts of zinc, lead, and copper do, or as do the alkaline solutions frequently used for this purpose. I have frequently tested the point by filtering off the cadmium sulphide before titration, and comparing the results obtained by titrating the washed sulphide, with those got by direct titration.

The three steels each contain over 1 per cent. of both nickel and chromium. No. 8620 is in the cast condition, R. 4290 in the tempered, and No. 8672 in the annealed state. The latter contained under 0.05 per cent. of hardening carbon, and practically the whole of the sulphur was obtained as cadmium sulphide by direct evolution. This is most important, and I believe that therein lies the solution of the problem of how to obtain

the whole of the sulphur in a sample of iron or steel evolved as sulphuretted hydrogen by hydrochloric acid. It is, of course, absolutely necessary that the sample should be completely decomposed by the acid. In the case of irons containing titanium, as Blair⁹ has shown, this is not the case, and some of the sulphur may remain in the evolution flask in the insoluble residue. I have experimented with an iron containing 0.2 per cent. of titanium. The sulphur content, obtained by a gravimetric method which included the fusion of the insoluble graphitic residue after treatment with aqua regia, was 0.080 per cent. The direct evolution gave 0.017 and 0.018 per cent. After annealing at 800 deg. C. the amount obtained was 0.066 per cent. The special treatment (referred to in the last column of results in the table) consisted in placing the crucible containing the sample in the muffle at 950 deg. C., and leaving at that temperature for five minutes. The gas was then turned off, and the muffle allowed to cool down to just below 800 deg. C. The gas was then relighted, and the temperature of the muffle kept at about 790 deg. C. for fifteen minutes. The crucible was then brought out to the front of the muffle and allowed to cool slowly. The sample containing titanium after this treatment gave 0.076 per cent., which suggests that by a proper preliminary treatment the whole of the sulphur may be obtained even from pig iron containing titanium by the evolution process.

Iron No. 471 was found to give off the whole of its sulphur as sulphuretted hydrogen by direct evolution (i.e., without any preliminary treatment).

As it is my custom to have all iron samples annealed before treatment in the evolution flask, I experimented with this sample in order to test the effect of the annealing on the amount of sulphur obtained. The results are given in the table.

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1 Chemical News, vol. lxxv., p. 47.

2 Journal of the Society of Chemical Industry, 1896, p. 218.

3 Journal of the Iron and Steel Institute, 1902, No. 1, p. 851.

4 Iron Age, May 8, 1902, p. 14.

5 Analyst, April 1903.

6 Chemical News, vol. xc., p. 326.

7 Ibid., vol. xciii., p. 201.

8 Stahl und Eisen, 1908, No. 8, p. 249.

9 Transactions of the American Institute of Mining Engineers, vol. xxxi., p. 748.

TABLE OF RESULTS

Description of Sample	Gravimetric Method Silicon per Cent.	Un-annealed	Evolution Method Annealed (Temperatures)							Special Treatments	Remarks
			700° C.	750° C.	800° C.	850° C.	900° C.	950° C.			
Steel, No. 8620	0.061	{ 0.025 0.026	0.046 0.045	0.058 0.058	0.061 0.060	0.059 0.059	0.053 0.054	0.046a 0.051b			a) Paper burnt off; drilling slightly oxidised. b) Drillings quite bright after annealing instead of grey, as in the case when annealed at a lower temperature.
Steel, R. 4290	0.051	{ 0.030 0.031	0.045 0.048	0.049 0.050	0.051 0.051	0.050 0.047	0.048 0.046	} ..	0.050		The Duplicate results were obtained by different workers using different apparatus.
Steel, No. 8627	0.054	0.053			{ 0.053 0.054	{	0.048		{ 0.054 0.055		
Iron, No. 471	0.085	{ 0.085 0.086	0.083 0.087	 0.085	0.084 0.085	0.080 0.081	0.066 0.071	{ }	0.084		Do.
Iron, No. 726	0.053	0.028		0.052	0.051	0.048			0.051		Do.