to maintain fluidity, for 10 minutes longer. Cool and extract the fused melt with 25 or 30 cc. of hot water.

By fusing with sodium carbonate alone, the vanadium will be converted into the soluble sodium vanadate, but part of the chromium, which is usually present in the special steels, will be oxidized to sodium chromate. The addition of charcoal and the continuation of the fusion for 10 minutes reduces the sodium chromate to chromic oxide but does not affect the extraction of the vanadium, so that a very sharp separation of chromium from vanadium is obtained. When the disintegration of the melt is complete, filter and wash well with hot water. The filtrate will contain all of the vanadium, but no chromium. The filtrate should be either colorless or slightly tinged with yellow, due to the presence of sodium sulphide, if the sulphates were not completely washed out of the precipitate obtained with sodium carbonate.

Add to the filtrate and washings 20 cc. of sulphuric acid (sp. gr. 1.51). Boil the solution and run in 3 or 4 cc. of a 3 per cent. solution of potassium permanganate. Boil for 5 minutes, run in sulphur dioxide until all the potassium permanganate is reduced to manganous sulphate and the solution has turned blue from the reduction of the vanadic acid to divanadyl sulphate.

Evaporate the solution until fumes of sulphuric anhydride begin to come off freely. Cool the solution and dilute with water to about 60 cc. and titrate the warm solution with $N/20$ potassium permanganate. If the excess of potassium permanganate is added and the solution boiled down before reduction with sulphur dioxide and then the excess of the sulphur dioxide destroyed by evaporating to sulphur anhydride fumes, and the warm diluted solution titrated, at about 50°, the oxidation of the divanadyl sulphate to vanadic acid is rapid and the end reaction sharp.

Since the oxidation of the vanadium is from V_2O_4 to V_2O_5, the hydrogen equivalent of elemental vanadium must be the atomic weight of vanadium, and one cc. of $N/20$ potassium permanganate is equivalent to 0.00256 gram of elemental vanadium.

CHEMICAL LABORATORY, UNIVERSITY OF MICHIGAN,
ANN ARBOR.

DIRECT COMBUSTION OF STEEL FOR CARBON AND SULPHUR.

BY HELEN ISHAM AND JOSEPH AUMER.

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In the preparation of Standardized Steel samples at the Bureau of Standards the criterion of homogeneity of the bar has been its carbon content, determined at varying points along the length and radius. This has necessitated many carbon determinations for each bar, and has led to an inquiry into the relative merits of the solution and combustion method and the direct combustion method. Results at the Bureau of Standards seemed to indicate that the direct combustion method, while giving higher results than the solution and combustion method, did not give a complete evolution of the carbon as carbon monoxide or carbon dioxide, since the unburned residue, after the magnetic oxide of iron had been separated from it by grinding and sifting, gave an average carbon content, by the solution and combustion method, corresponding to 0.01 per cent. carbon in the original sample.

The investigation has been continued at the University of Illinois with results which would lead to the conclusion that while the direct combustion method gives results which are somewhat higher than those of the solution method, still the discrepancy is not so great as the first experiments would seem to indicate, and might almost come within the limits of experimental error. The determination of carbon in the residue shows that when the steel is burned in oxygen for twenty minutes at such a temperature that it is found to be completely fused together when withdrawn from the crucible, the carbon content in the unburned residue corresponds to an average of 0.003 per cent. of carbon in the original sample.

Along with this work a series of experiments have been conducted in the hope of devising a method by which sulphur might be evolved as sulphur dioxide or trioxide and determined simultaneously with the carbon. Previous work on this subject[1] has shown that sulphur is much less readily evolved from iron than is carbon. The results of this work show that with a basic protection, as powdered alumina, between the platinum of the crucible and the steel, some sulphur is retained by the burned steel, some by the alumina, and some is evolved in gaseous compounds. With an acid protection for the platinum, as powered silica, one source of trouble is removed, and the sulphur is distributed between the iron residue and the evolved gases. It has been found impossible, so far, to heat the steel surrounded by silica to a sufficiently high temperature, and for a long enough time, to get complete evolution of the sulphur before a slag forms and runs into the platinum. Further work is being done, using highly acid clay boats for the steel, which have so far proved a complete protection for the platinum, but one hour's heating in open air has not been sufficient to remove all the sulphur from the steel.

Experimental.

A tabulated summary of the results on carbon, taken from the standard analyses published by the Bureau of Standards, and from personal work, will show to what extent the two methods differ. The averages

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[1] *The Analyst*, 25, 141.

from the Bureau of Standards results have been taken from only those reports in which both the direct combustion and the solution and combustion methods have been reported by the same analyst, and are therefore called "selected:"

Sample No.	Nature of steel.		1 Sol. and comb. method.	2 Direct comb. method.	Diff. 2.− 1.	Analytical source.
1	Bessemer..........	0.1	0.073	0.080	+0.007	B. of S. average, selected.
2	"	0.2	0.205	0.207	+0.002	" " " " "
3	"	0.2	0.189[1]	0.193[1]	+0.004	H. Isham.
4	"	0.4	0.436	0.447	+0.011	B. of S. average, selected.
5	"	0.4	0.412[2]	0.423[1]	+0.011	H. Isham.
6	Basic open hearth...	0.1	0.145	0.144	—0.001	B. of S. average, selected.
7	" " " ...	0.2	0.236	0.241	+0.005	" " " " "
8	" " " ...	0.2	0.240	0.255	+0.015	H. Isham.
9	" " " ...	0.2	0.255	0.257	+0.002	J. Aumer.
10	" " " ...	0.4	0.422	0.423	+0.001	B. of S. average, selected.
11	" " " ...	0.4	0.424	0.425	+0.001	H. Isham.
12	" " " ...	0.6	0.602	0.603	+0.001	B. of S. average, selected.
13	" " " ...	0.6	0.591	0.589	—0.002	H. Isham.
14	" " " ...	0.8	0.831	0.837	+0.006	B. of S. average, selected.
15	" " " ...	1.0	1.048	1.049	+0.001	" " " " "
	Average............................				+0.004	

An attempt to explain the low results in the solution and combustion method by loss of hydrocarbons during solution in acid copper potassium chloride led to the determination of hydrocarbons by passing air through a gas wash-bottle containing steel dissolving in copper potassium chloride with 5 cc. concentrated hydrochloric acid in 300 cc. solution, then through a combustion furnace, a purifying chain of anhydrous copper sulphate, silver nitrate solution, and calcium chloride to remove hydrochloric acid and water, then a weighed soda lime tube. The soda lime tube did not show any increase in weight during the solution of the steel.

The residues from samples Nos. 8, 11, and 13 were analyzed for carbon by grinding in a steel mortar, sifting out the magnetic oxide of iron and dissolving the unburned steel in copper potassium chloride, filtering, and burning the residue. No. 8, basic open hearth 0.2, gave from 7 grams of steel a residue of carbon equivalent to 0.004 per cent. carbon in the original steel. No. 11, basic open hearth 0.4, gave from 9 grams steel a carbon residue equivalent to 0.003 per cent. carbon in the original steel. No. 13, basic open hearth 0.6, gave from 13 grams steel a residue containing carbon equivalent to 0.003 per cent. carbon in the original steel. Frequent blanks were run to insure an efficient purifying chain.

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[1] Average of four determinations.

[2] Average of three determinations.

For the sulphur determinations the gases from the burning steel were passed over heated platinized asbestos, and then through a saturated solution of barium chromate in 2 per cent. hydrochloric acid. After the combustion was completed the solution was made alkaline with ammonium hydroxide, the barium salts filtered out, and the soluble chromate, equivalent to the sulphur trioxide introduced, determined by titration against an excess of standard ferrous sulphate and potassium bichromate solutions. This method was finally adopted in preference to the precipitation by barium chloride, after absorbing the sulphur dioxide and trioxide in chromic acid solution, since the precipitation of barium sulphate in chromic acid solution is inaccurate, and the reduction of the chromic acid too tedious. The one precaution necessary is the complete conversion of all sulphur dioxide into trioxide before absorption by the barium chromate. The work on sulphur will be continued.

The apparatus used for the direct combustions in oxygen has consisted of a Gooch tubulated platinum crucible, such as is used for moisture determinations in mineral analysis, the tubulated cover being sealed in with fused sodium meta tungstate. The outlet tube was joined by lead glass to a tube carrying platinized asbestos, heated with the low flame from a bunsen burner, followed by a U-tube containing the barium chromate solution, and then by the usual purifying and absorbing chain. The combustion was carried on in oxygen, which was passed over heated platinized asbestos and the usual purifying chain before entering the crucible. The steel was introduced in a little platinum bucket, which had been previously filled with alumina or silica and a hole punched down the center with a glass rod. If this protecting media is very finely divided and is light the danger of the steel running through it and coming in contact with the platinum is slight, especially in the case of alumina. The bucket rests on a layer of alumina or silica on the bottom of the crucible.

Conclusions.

1. The direct combustion of steel gives results for carbon which average 0.004 per cent. higher than results by the solution and combustion method.
2. After combustion in oxygen the burned steel contains carbon amounting to about 0.003 per cent. carbon in the original sample.
3. Combustion is complete after 20 minutes heating at the full heat of a gas blast lamp.
4. The evolution of sulphur is incomplete under the conditions giving practically complete evolution of the carbon.

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