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Determination of Manganese in Iron, Steel and Ferromanganese. I

Determination of Manganese in Iron and Steel*

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Synopsis

The suitable concentration of acids and other conditions in ferrous-permanganate method of the oxidation with persulfate were studied for the determination of manganese in iron and steel and the following results were obtained: (1) Acid concentration: using 2 g of ammonium persulfate in total volume of 200 mL, manganese was determined in the concentration range 1.2~2.4 N sulfuric acid, 0.53~1.20 N nitric acid and 0.6~1.2 N mixed acid of sulfuric acid and nitric acid (3:1) but manganese could not be determined in acidic solution with perchloric acid. (2) Boiling time: the time of boiling of the solution for the oxidation of manganese by ammonium persulfate using silver nitrate was 5 min, during which the decomposition of permanganic acid occurred. (3) Influence of phosphoric acid: when 1 g of iron and 10 mg of manganese were present, the existence of 4 ml of phosphoric acid (sp. gr. 1.7) was advisable. (4) The suitable procedure for the determination of manganese in iron and steel was established and the analysis was carried out with several samples and reliable results were obtained.

I. Introduction

With regard to the method for determining the manganese in iron and steel, there have been the sodium bismuthate method⁽¹⁾, the Volhard method and the ammonium persulfate-arsenous acid method⁽¹⁾ and so on. The bismuthate method is somewhat complicated and requires a long time and, moreover, the price of the sodium bismuthate is considerably high. The Volhard method is a rapid one, but a considerable amount of skill is required for the determination of the end point. The ammonium persulfate-ferrous sulfate-permanganate method, which has ever been used in this laboratory, does not prevail, because the experimental conditions have not been elucidated. Therefore, in order to obtain the directions for the determination of manganese in iron and steel, the investigations were carried out in detail with respect to the kind of acid, its concentration, the boiling time and the amount of the phosphoric acid, and the decisive condition was obtained. The procedure of the present method is easier than the others and has high precision.

* The 737th report of the Research Institute for Iron, Steel and Other Metals. Published in the Journal of the Japan Institute of Metals, 17-1 (1953), 35.

(1) Lundell, Hoffman, and Bright, *Chemical Analysis of Iron & Steel* (1946), 190; 195.

II. Fundamental experiment

1. Reagents

(a) Standard manganese solution

Manganous sulfate solution was used and the manganese content was determined by the sodium bismuthate method.

(b) N/10 ferrous ammonium sulfate solution

This was prepared from the ferrous ammonium sulfate crystals and its factor was determined by the standardization with N/10 potassium permanganate solution of the known concentration.

(c) N/10 potassium permanganate solution

This was prepared from the potassium permanganate crystals by the usual manner and standardized with sodium oxalate.

(d) Mixed acid

6 N sulfuric acid and 6 N nitric acid were mixed in equal proportion.

2. Experimental method

A definite amount of the standard manganese solution was added to the acidic solution of sulfuric acid, nitric acid, perchloric acid, sulfuric acid plus nitric acid, sulfuric acid plus perchloric acid and sulfuric acid plus nitric acid plus perchloric acid, respectively. 5 ml of 2 per cent silver nitrate solution and 5 ml of phosphoric acid were added to this solution and the final volume of the solution was brought up to about 200 ml. The solution was added 2 or 4 grams of ammonium persulfate and boiled, and immediately after decomposition of the excess ammonium persulfate it was cooled with cold water. After sufficient N/10 ferrous ammonium sulfate solution was added to prepare an excess of about 10 ml, the solution was at once titrated with N/10 permanganate solution and the amount of manganese was calculated.

3. Concentration of acid.

(a) Concentration of sulfuric acid

One gram of the electrolytic iron was respectively dissolved in the various amounts of 6 N sulfuric acid (acid was added in excess to the amount required to dissolve the iron). After oxidation with 3 per cent hydrogen peroxide, the excess hydrogen peroxide was decomposed. Then, 10 ml of the standard manganese solution (8.09 mg of manganese) were added the above-mentioned experimental method was followed. The results obtained are given in Table 1. The good results were obtained in the range of about 1.2 to 2.4 N sulfuric acid. When 4 grams of ammonium persulfate were used, the available range of acid concentrations was somewhat extended.

(b) Concentration of nitric acid

One gram of the electrolytic iron was respectively dissolved in the various amounts of 7 N nitric acid (nitric acid was added in excess to the amount required to dissolve the iron). Then 10 ml of the standard manganese solution (10.50 mg of manganese) were added to this solution. The subsequent procedure was the

Table 1. (Mn added : 8.09 mg)

Acid concentration (N)	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 2(g)		$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 4(g)	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
0.3	10.26 9.82 9.96 } 10.01	+1.92	12.82 9.25 11.92 } 11.33	+3.24
0.6	8.19 9.25 9.16 } 8.87	+0.78	9.30 10.32 9.25 } 9.62	+1.53
0.9	8.19 8.16 8.16 } 8.17	+0.08	8.19 8.19 8.16 } 8.18	+0.09
1.2	8.07 8.09 8.07 } 8.08	-0.01	8.12 8.16 8.15 } 8.14	+0.05
1.5	8.12 8.09 8.07 } 8.09	± 0.00	8.11 8.12 8.12 } 8.12	-0.03
1.8	8.09 8.07 8.11 } 8.09	± 0.00	8.12 8.11 8.07 } 8.10	+0.01
2.1	8.12 8.15 8.07 } 8.11	+0.02	8.09 8.12 8.07 } 8.09	± 0.00
2.4	8.07 8.09 8.07 } 8.08	-0.01	8.07 8.11 8.07 } 8.08	-0.01
2.7	8.07 7.89 7.83 } 7.93	-0.17	8.07 8.07 8.11 } 8.08	-0.01
3.0	7.57 7.45 7.49 } 7.50	-0.59	7.59 7.77 7.89 } 7.75	-0.34

same as that described in (a) and the manganese was determined. The results obtained are given in Table 2. The satisfactory results were obtained in the range of about 0.53 to 1.22 N nitric acid.

(c) Concentration of perchloric acid

One gram of the electrolytic iron was dissolved in the various amounts of 6 N perchloric acid and small amounts of 3 per cent hydrogen peroxide (perchloric acid was added in excess to the amounts required to dissolve the iron). To this solution 10 ml of the standard manganese solution (8.81 mg of manganese) were added. The subsequent procedure was the same as that described in (a) and the manganese was determined. The results obtained are given in Table 3.

If the concentrations of acids were high, the high values of results were obtained, because the excess ferrous ammonium sulfate was oxidized. On the contrary, if the concentrations of acids were low, the results were of low values,

Table 2. (Mn added : 10.50 mg)

Acid concentration (N)	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 2(g)		$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 4(g)	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
0.18	14.37 15.13 14.83 } 14.78	+4.28	14.31 16.62 14.37 } 15.10	+4.60
0.53	10.52 10.54 10.51 } 10.52	+0.02	10.56 10.51 10.51 } 10.53	+0.03
0.84	10.49 10.54 10.52 } 10.51	+0.01	10.51 10.53 10.51 } 10.51	+0.01
1.22	10.54 10.46 10.51 } 10.50	± 0.00	10.53 10.53 10.51 } 10.53	+0.03
1.57	9.68 10.08 10.49 } 10.08	-0.42	10.13 10.36 10.32 } 10.27	-0.23
1.92	9.22 9.98 8.60 } 8.93	-1.57	9.32 9.55 9.73 } 9.53	-0.97

Table 3. (Mn added : 8.81 mg)

Acid concentration (N)	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 2(g)		$(\text{MH}_4)_2\text{S}_2\text{O}_8$ 4(g)	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
0.15	8.32 8.52 8.48 } 8.44	-0.37	8.60 8.51 8.58 } 8.56	-0.25
0.30	8.77 8.70 8.74 } 8.74	-0.07	8.72 8.68 8.72 } 8.71	-0.10
0.45	8.92 8.88 8.85 } 8.88	+0.07	8.79 8.79 8.75 } 8.78	-0.10
0.60	9.04 9.00 8.96 } 8.99	+0.18	8.80 8.87 8.86 } 8.84	+0.03
0.75	9.18 9.13 9.15 } 9.15	+0.34	9.15 9.12 9.12 } 9.13	+0.32
0.90	9.33 9.37 9.35 } 9.35	+0.54	9.21 9.18 9.20 } 9.19	+0.38

because of the incomplete oxidation of manganese. Consequently, there was no range of the acid concentration suitable for the determination of manganese. If, in the first part of the procedure, the dissolution of iron is made only with

perchloric acid, a part of the perchloric acid is reduced to liberate Cl' which, as it is, interferes with the subsequent procedure. Therefore, the solution must be heated to the fume of perchloric acid, or the dissolution must be made with the addition of hydrogen peroxide.

(d) Concentration of the mixture of sulfuric acid and nitric acid (1:1)

The same procedures as that described in (a) with the various amount of the mixture of sulfuric acid and nitric acid were carried out using the electrolytic iron. The results obtained are given in Table 4. The results obtained were satisfactory in the acid concentration range 0.30~0.9 N, when 2 grams of ammonium persulfate were used, and 0.60 to 1.50 N, when 4 grams of it were used.

Table 4. (Mn added : 11.19 mg)

Acid concentration (N)	(NH ₄) ₂ S ₂ O ₈ 2(g)		(NH ₄) ₂ S ₂ O ₈ 4(g)	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
0.15	11.27 } 11.73 } 11.57 11.73 }	+0.38	12.19 } 12.21 } 12.27 12.43 }	+1.08
0.30	11.12 } 11.16 } 11.15 11.16 }	-0.04	11.73 } 11.91 } 11.81 11.79 }	+0.62
0.60	11.12 } 11.12 } 11.13 11.16 }	-0.06	11.21 } 11.27 } 11.23 11.23 }	+0.04
0.90	11.12 } 11.16 } 11.15 11.16 }	-0.04	11.15 } 11.21 } 11.18 11.19 }	-0.01
1.20	10.91 } 10.98 } 10.91 10.86 }	-0.28	11.15 } 11.23 } 11.19 11.21 }	±0.00
1.50	10.79 } 10.70 } 10.75 10.75 }	-0.44	11.10 } 11.21 } 11.15 11.15 }	-0.04
1.80	10.31 } 10.08 } 10.20 10.23 }	-0.99	10.23 } 10.17 } 10.23 10.29 }	-0.96

Altering the proportion of the mixed acid to 3:1, the experiments were carried out in the same way as above. The results are given in Table 5. These data show that the manganese can be quantitatively determined in the acid concentration range 0.60 to 1.50 N, in the case in which either 2 or 4 grams of ammonium persulfate is used.

(e) Effect of perchloric acid in the other acid medium

Various amounts of the perchloric acid were added to the solutions which were acidified in the optimum acid concentration ranges with sulfuric acid (1.80 N), nitric acid (0.84 N) or sulfuric acid plus nitric acid (3:1, 1.08 N). The experiments were carried out with the solutions thus prepared, which contained the

Table 5. (Mn added : 11.19 mg)

Acid concentration (N)	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 2(g)		$(\text{NH}_4)_2\text{S}_2\text{O}_8$ 4(g)	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
0.2	11.34 } 11.28 } 11.32 11.34 }	+0.13	11.42 } 11.38 } 11.40 11.40 }	+0.21
0.4	11.16 } 11.22 } 11.24 11.34 }	+0.05	11.22 } 11.24 } 11.22 11.19 }	+0.03
0.6	11.22 } 11.28 } 11.22 11.16 }	+0.03	11.24 } 11.22 } 11.25 11.28 }	+0.06
0.8	11.34 } 11.19 } 11.25 11.22 }	+0.06	11.22 } 11.19 } 11.21 11.22 }	+0.02
1.0	11.22 } 11.19 } 11.19 11.16 }	± 0.00	11.16 } 11.22 } 11.20 11.22 }	+0.01
1.2	11.16 } 11.22 } 11.16 11.12 }	-0.03	11.22 } 11.16 } 11.17 11.12 }	-0.02
1.6	10.91 } 10.85 } 10.80 10.66 }	-0.39	10.91 } 10.85 } 10.90 10.95 }	-0.29
2.0	10.28 } 10.47 } 10.38 10.41 }	-0.81	10.95 } 10.78 } 10.86 10.85 }	-0.33

various combinations of acids. The data listed in Table 6 are only the results which permit the quantitative determination, that is, the quantitative results can be obtained so far as the perchloric acid amount to 0.3 N. If its concentration is more than 0.3 N, the value of manganese becomes low. Therefore, it is advisable not to use the perchloric acid, if possible.

4. Boiling time

In order to investigate the effect of the boiling time, the following experiments were carried out with sulfuric acid solution (1.8 N), nitric acid solution (0.84 N) and sulfuric acid plus nitric acid solution (3:1, 1.08 N), the acid concentration of each solution being kept in its optimum range, with and without addition of 1 gram of iron (the acids required to dissolve 1 gram of the electrolytic iron were added extra). 2 grams of ammonium persulfate were added to it. The solutions were boiled for various lengths of time, the measurement of which began at the evolution of large bubbles. After cooling, the preceding experiments were made and the manganese was determined. The results obtained are given in Tables 7, 8 and 9. The results were good up to about 5 minutes in the presence as well as in the absence of iron, but the boiling of above 6 minutes led to low values due to the partial decomposition of MnO_4' .

Table 6. (Mn added : 8.81 mg)

Acid concentration H ₂ SO ₄ (N)	Acid concentration HNO ₃ (N)	Acid concentration H ₂ SO ₄ + HNO ₃ (N)	Acid concentration HClO ₄ (N)	Total acid concentration (N)	Fe none		Fe 1g added	
					Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
1.65	—	—	0.15	1.80	8.78 } 8.81 } 8.80 8.80 }	-0.01	8.83 } 8.81 } 8.82 8.83 }	+0.01
2.70	—	—	0.15	2.85	8.82 } 8.84 } 8.82 8.80 }	+0.01	8.80 } 8.83 } 8.82 8.82 }	+0.01
1.80	—	—	0.30	2.10	8.78 } 8.78 } 8.77 8.75 }	-0.04	8.80 } 8.79 } 8.81 8.83 }	±0.00
2.40	—	—	0.30	2.70	8.83 } 8.80 } 8.82 8.84 }	+0.01	8.81 } 8.82 } 8.82 8.83 }	+0.01
—	0.87	—	0.21	1.08	8.80 } 8.84 } 8.81 8.78 }	±0.00	8.81 } 8.82 } 8.81 8.80 }	±0.00
—	1.06	—	0.21	1.27	8.80 } 8.80 } 7.79 8.78 }	-0.02	8.83 } 8.84 } 8.83 8.81 }	+0.02
—	1.06	—	0.30	1.36	8.83 } 8.79 } 8.81 8.80 }	±0.00	8.83 } 8.78 } 8.80 8.80 }	-0.01
0.53	0.18	0.71	0.15	0.86	8.80 } 8.79 } 8.80 8.80 }	-0.01	8.84 } 8.78 } 8.81 8.80 }	±0.00
0.90	0.18	1.08	0.15	1.23	8.81 } 8.83 } 8.81 8.80 }	±0.00	8.82 } 8.83 } 8.82 8.81 }	+0.01
0.70	0.18	0.88	0.21	1.09	8.82 } 8.80 } 8.81 8.82 }	±0.00	8.83 } 8.80 } 8.81 8.78 }	±0.00
0.90	0.18	1.08	0.21	1.29	8.82 } 8.80 } 8.81 8.80 }	±0.00	8.80 } 8.80 } 8.80 8.80 }	-0.01
0.87	0.18	1.05	0.30	1.35	8.80 } 8.72 } 8.76 8.76 }	-0.05	8.80 } 8.71 } 8.75 8.74 }	-0.06
0.90	0.18	1.08	0.30	1.38	8.76 } 8.76 } 8.77 8.78 }	-0.04	8.81 } 8.80 } 8.81 8.83 }	±0.00

5. The amounts of phosphoric acid

As a result of the experiment, it was found that, when 1 gram of the sample was used, the iron could be completely complexed with more than 4 ml of the phosphoric acid (sp. gr. 1.70) and that the resulting MnO₄' was also sufficiently stable. Therefore, 5 ml of the phosphoric acid were used, and the amount of

the phosphoric acid sufficient for 2 g sample was more than 6 ml. The details will be shown in the second report.

Table 7. (Mn added : 11.19 mg)

Boiling time (min)	Fe none		Fe 1g added	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
1	11.16 11.09 } 11.16 11.22 }	-0.03	11.20 11.24 } 11.21 11.20 }	+0.02
2	11.24 11.22 } 11.24 11.27 }	+0.05	11.18 11.20 } 11.18 11.18 }	-0.01
4	11.16 11.22 } 11.16 11.09 }	-0.03	11.24 11.20 } 11.21 11.18 }	+0.02
5	11.22 11.16 } 11.17 11.14 }	-0.02	11.18 11.16 } 11.17 11.18 }	-0.02
6	11.09 11.04 } 11.04 10.99 }	-0.15	11.08 11.05 } 11.07 11.08 }	-0.12
8	10.96 10.85 } 10.87 10.67 }	-0.32	11.05 11.02 } 11.04 11.05 }	-0.15

Table 8. (Mn added : 11.19 mg)

Boiling time (min)	Fe none		Fe 1g added	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
1	11.19 11.16 } 11.18 11.19 }	-0.01	11.17 11.17 } 11.18 11.20 }	-0.01
2	11.19 11.19 } 11.18 11.17 }	-0.01	11.24 11.20 } 11.20 11.17 }	+0.01
4	11.11 11.16 } 11.16 11.21 }	-0.03	11.17 11.17 } 11.18 11.20 }	-0.01
5	11.11 11.19 } 11.17 11.22 }	-0.02	11.17 11.24 } 11.19 11.17 }	±0.00
6	11.11 11.09 } 11.08 11.03 }	-0.11	11.15 11.17 } 11.16 11.17 }	-0.03
8	10.97 10.99 } 10.97 10.90 }	-0.22	11.11 11.07 } 11.08 11.05 }	-0.11

Table 9. (Mn added : 11.19 mg)

Boiling time (min)	Fe none		Fe 1g added	
	Mn obtained (mg)	Difference (mg)	Mn obtained (mg)	Difference (mg)
1	11.17 11.19 } 11.17 11.14	-0.02	11.21 11.26 } 11.22 11.20	+0.03
2	11.17 11.22 } 11.18 11.17	-0.01	11.24 11.19 } 11.21 11.20	+0.02
4	11.17 11.22 } 11.18 11.17	-0.01	11.26 11.24 } 11.22 11.17	+0.03
5	11.14 11.22 } 11.16 11.17	-0.03	11.14 11.17 } 11.17 11.20	-0.02
6	11.12 11.17 } 11.14 11.12	-0.09	11.09 11.05 } 11.09 11.04	-0.10
8	11.07 11.05 } 11.04 11.02	-0.15	11.01 10.98 } 11.01 11.05	-0.18

III. Determination of manganese in iron and steel

Basing upon the preceding experiments, the analytical procedure was established as follows :

1. Analytical procedure

According as the manganese content (Note. 1), a suitable amount of the sample is weighed, transferred to a 500 ml conical beaker and dissolved with the addition of 30 ml of sulfuric acid (1 : 5) and 10 ml of nitric acid (1 : 3) by heating. After the nitrous oxide is expelled by boiling, about 150 ml of water, 5 ml of 2 per cent solution of silver nitrate and 5 ml of phosphoric acid (sp. gr. 1.70) are added to the solution and a final volume is brought up to about 200 ml, and then to this solution about 2 grams of ammonium persulfate are added. After the excess of the latter is decomposed by boiling (boiling is made for about 2 minutes after the frothing of fine bubbles is replaced by the evolution of large bubbles), the solution is immediately cooled below about 30° with cold water and the standard N/10 ferrous ammonium sulfate solution is added to it until the color of permanganate disappears and then about 10 ml of the reagent is added in excess. This solution is immediately titrated back with the standard N/10 permanganate solution and the amount of manganese is calculated by the following equation :

$$\frac{\left\{ \begin{array}{l} \text{(ml of N/10 solution of)} \\ \text{(FeSO}_4\text{(NH}_4\text{)}_2\text{SO}_4\text{, added)} \end{array} \right\} - \left\{ \begin{array}{l} \text{(ml of N/10 solution of)} \\ \text{(KMnO}_4\text{, used)} \end{array} \right\} \times 0.1099}{\text{weight of sample (g)}} = \% \text{ of Mn}$$

2. Results of analyses

Table 10 shows the results obtained with various kinds of samples by the above-mentioned procedure.

Table 10.

Kind of sample	Mn obtained by bismuthate method (%)	Mn obtained by this method (%)	Difference (%)
C-steel	0.33 } 0.33 0.33 } 0.33 }	0.33 } 0.33 0.33 } 0.33 }	± 0.00
C-steel	0.51 } 0.51 0.51 } 0.51 }	0.51 } 0.51 0.51 } 0.50 }	± 0.00
Si-Mn steel	0.89 } 0.89 0.89 } 0.89 }	0.88 } 0.88 0.88 } 0.88 }	-0.01
N-steel	1.22 } 1.22 1.22 } 1.22 }	1.21 } 1.21 1.20 } 1.22 }	-0.01
B-steel	1.75 } 1.75 1.75 } 1.76 }	1.75 } 1.75 1.75 } 1.75 }	± 0.00
Mn-steel	2.31 } 2.31 2.30 } 2.32 }	2.33 } 2.32 2.31 } 2.32 }	+0.01

Note :

(1) The amounts of samples used for the analyses are weighed according as the manganese content as shown in Table 11.

Table 11.

Mn content (%)	Sample taken (g)
Less than 0.5	2.0
0.5 1.5	1.0
1.5 2.0	0.5
2.0 5.0	0.25

(2) If a sample contains chromium and vanadium, it is treated and titrated in the same way as the preceding analytical procedure, but afterwards the resulting solution is further added with 2 grams of ammonium persulfate and boiled again to complete oxidation. Boiling is continued until the excess of ammonium persulfate is decomposed, and then 5 ml of the hydrochloric acid (1 : 4) are added to decompose manganate or manganese dioxide completely. Expelling the chlorine by boiling, the solution is cooled with cold water. After cooling, an excess of about 5 ml of N/10 ferrous ammonium sulfate solution are added and the excess of the latter is immediately titrated with N/10 potassium permanganate solution and the amount of manganese is calculated by the following equation :

$$\frac{(F_1 - V_1) - (F_2 - V_2) \times 0.1099}{\text{weight of sample (gram)}} = \% \text{ of Mn}$$

F_1 = ml of N/10 ferrous ammonium sulfate solution added initially,

V_1 = ml of N/10 potassium permanganate solution required for the first titration,

F_2 = ml of N/10 ferrous ammonium sulfate solution added afterwards,

V_2 = ml of N/10 potassium permanganate solution required for the second titration.

(3) As to the amounts of silver nitrate, 5 ml of its 2 per cent solution were used in the above-mentioned analytical procedure, but it has been indicated⁽²⁾ that the minimum amount of silver nitrate needed is 0.1 ml of its 0.2 per cent solution. Therefore, if the sample contains chromium and vanadium, it is advisable to use the minimum amount of silver nitrate, if possible, as the amount of the white precipitate of silver chloride decreases and the end point is easily recognized in the last titration.

Summary

(1) The various investigations were carried out on the ferrous-permanganate method for determining manganese in iron and steel in which the oxidation was made with ammonium persulfate.

(2) The optimum concentration range of acid was from about 1.2 to 2.4N in the sulfuric acid solution and this range was somewhat extended when 4 grams of ammonium persulfate were used. It was from 0.3 to 0.9 N in the sulfuric acid plus nitric acid (1:1) solution and from 0.6 to 1.5 N when 4 grams of ammonium persulfate were used. The quantitative results were obtained in the acid concentration range 0.4 to 1.2 N in the mixed acid solution whose component acids were mixed in the proportion 3:1 and from 0.52 to 1.22 N in the nitric acid solution, both ranges being independent of the amounts of ammonium persulfate. The determination could not be carried out in the perchloric acid solution. In all the acidic solutions described above, the quantitative results were obtained within about 5 minutes' boil with large bubbles. In the extent of about 10 mg of manganese, the reliable results were obtained with the addition of more than 4 ml of the phosphoric acid.

(3) According to the above results, an analytical procedure for the determination of manganese in iron and steel could be established and the results obtained by this procedure were shown.

(2) O. V. Datsenko, *Zavodskaya Lab.*, 16 (1950), 784-6. C. A., 45 (1951), 975.