$$\frac{0.007995\,cr}{1+r} = \text{weight of bromine in } b \text{ or in } a$$

$$p' = \frac{100}{a} \times \frac{0.012685c}{1+r} = \frac{126.85\,(b - 0.018787c)}{0.469\,a} \qquad [3]$$

$$p'' = \frac{100}{a} \times \frac{0.007995\,cr}{1+r} = \frac{79.95\,(0.023477c - b)}{0.469\,a} \qquad [4]$$

In conclusion profound thanks are expressed to Prof. A. B. Prescott, in whose laboratory and under whose supervision the work has been done.

THE DETERMINATION OF NICKEL IN NICKEL-STEEL.

BY GEO. WM. SARGENT.

THE results obtained in this laboratory by the following method have been such as to warrant my placing it before fellow-chemists, who, I believe, will find the method more reliable and more pleasant to work than most procedures now in vogue for the determination of nickel in steel.

The method in detail is as follows: Dissolve two grams of the steel in hydrochloric acid (1.1 sp. gr.), add one cc. of strong nitric acid to oxidize the iron, and evaporate to hard dryness. Take up the residue in twenty cc. hydrochloric acid (1.1 sp. gr.), evaporate to ten cc. volume, and transfer the solution to a 250 cc. separatory funnel.[1] Use warm hydrochloric acid (1.1 sp. gr.) for washing the contents of the beaker into the funnel, taking care to keep the volume as small as possible. Cool the funnel and its contents under the spigot, then introduce forty cc. of ether free from alcohol, which has just been thoroughly shaken with five cc. of strong hydrochloric acid, and shake vigorously for ten minutes, keeping the temperature from rising by frequently holding the funnel under the running water. Allow the funnel to stand a few minutes, draw off the lower aqueous layer containing the nickel, copper, manganese, and other chlorides not held by the ether, wash down the sides of the separatory funnel with hydrochloric acid (1.1 sp. gr.), shake with the

[1] See method of Chase for the "Determination of Nickel and Aluminium in Steel" in the appendix of "The Chemical Analysis of Iron," by Blair, third edition.

ether, and run the washings into the beaker with the chlorides of nickel, copper, etc.   Two washings with five to ten cc. of hydrochloric acid are sufficient to completely remove the nickel and other chlorides from the funnel.   The ethereal solution containing the ferric chloride is run into a residue bottle and later the ether recovered by distillation.   Boil the liquid containing the nickel to completely expel the ether and add to the boiling solution, diluted to at least 200 cc. an excess of ammonia and ten to twenty drops of bromine water, to precipitate any iron or manganese.   Filter, wash, redissolve in hydrochloric acid, reprecipitate with ammonia and bromine water, and filter. Combine the filtrates, boil, remove any precipitate, acidulate with hydrochloric acid, boil until the excess of bromine has been expelled, and precipitate the copper as sulphide.   The filtrate from the copper sulphide which contains nickel only, is evaporated to a volume of 100 cc., cooled and one cc. excess of ammonia added.   Now introduce into the nickel solution, which is best contained in an Erlenmeyer flask, five cc. of silver nitrate (one-half gram silver nitrate in a liter of water), and the same amount of a two per cent. solution of potassium iodide.   Run into the opalescent solution, which should have a temperature slightly lower than that of the hand, standard potassium cyanide (one cc. equal to about 0.001 gram of nickel) until the liquid becomes clear and bright.   This titration is best made with a black background, when the endreaction becomes very sharp and decided after a little practice, half a drop only being sufficient to discharge the opalescence.[1]

The potassium cyanide solution is standardized by introducing into an Erlenmeyer flask ten cc. of a solution containing a known amount of recrystallized nickel nitrate, $Ni(NO_3)_2 \cdot 6H_2O$, ten cc. of hydrochloric acid (1.1 sp. gr.), one cc. excess of ammonia, five cc. each of the silver nitrate and potassium iodide, diluting the whole to 100 cc., and running in the potassium cyanide until the solution clears.   It is necessary that a blank be made upon the silver nitrate and potassium iodide used as the indicator, under the same conditions as the standardization, and this amount deducted from each titration.

[1] See "Determination of Nickel in Nickel-Steel," by E. D. Campbell and W. H. Andrews : This Journal. 17, 127.

The presence of varying quantities of ammonium chloride in the nickel solution to be titrated has no effect on the titration. This is evident from the following :

Taken.			Found.				
NH$_4$Cl present. Grams.	Ni(NO$_3$)$_2$. cc.	Weight of Ni. Gram.	Readings. First.	Second.	Difference.	Blank.	Weight of Ni. Gram.
I	20	0.02	0.0	20.2	20.2	0.45	0.01996
3	20	0.02	20.2	40.5	20.3	0.45	0.02007
7	20	0.02	0.0	20.2	20.2	0.45	0.01996

I cc. potassium cyanide solution = 0.001011 gram nickel.

Some of the results obtained by the foregoing method I append :

Mark.	Solution of KCN equivalent to Ni. cc.	Weight of nickel found. Gram.	Nickel. Per cent.
389	27.65	0.02795	1.397
389	27.65	0.02795	1.397
389	27.55	0.02785	1.392
390	28.60	0.02891	1.445
390	28.55	0.02886	1.443
382	30.40	0.03073	1.536
382	30.20	0.03053	1.527
387	31.00	0.03134	1.567
387	30.70	0.03104	1.552
387	31.00	0.03134	1.567
109	51.00	0.05156	2.578
109	51.30	0.05190	2.595

I cc. potassium cyanide = 0.001011 gram nickel.

The above determinations were made on steels in which the manganese did not exceed one-tenth per cent. and in which there was no copper.

Different amounts of nickel were mixed with solutions containing two grams of iron and the nickel determined therein :

Taken.		Found.	
Ni(NO$_3$)$_2$ solution. cc.	Weight of nickel. Gram.	KCN solution equivalent to Ni. cc.	Weight of nickel. Gram.
30	0.03	29.60	0.02992
30	0.03	29.05	0.02937
40	0.04	39.60	0.04003
40	0.04	39.70	0.0401
45	0.045	44.05	0.0445

For the estimation of nickel in ferronickel, dissolve twenty grams of the sample in aqua regia, evaporate to hard dryness take up with hydrochloric acid (1.1 sp. gr.), transfer to a liter flask, and dilute to the mark with water. Remove fifty cc., to which add five cc. strong hydrochloric acid, and concentrate to one-sixth its volume. Transfer to the separatory funnel, and proceed as with the steel.

A sample of ferronickel which, by an unknown method contained 33.93 per cent. of nickel, yielded 33.80, 34.00, 33.88, and 33.96 per cent. Another sample which, according to one chemist, contained 34.95 per cent. of nickel, gave 35.15 and 35.26 per cent.

The above-described method is, as can readily be seen, a combination of a modified portion of the method of Chase, with a slightly changed part of the procedure given by Campbell and Andrews. In the course pursued by Chase, two ether separations are necessary to remove the ferric chloride, while the separation, as worked by us, need not be repeated ; but little ferric chloride is left, and the two ammonia precipitations are sufficient to completely remove the iron from the nickel.

With regard to the titration with potassium cyanide, that may be accomplished in the presence of most all the salts of ammonium and the mixed alkalies except the nitrates. The excess of ammonia should not in any case exceed three cc.[1]

While in steels, about which one knows nothing, it will be necessary to hunt for copper, yet in the general run of furnace work, copper is absent, and knowing such to be the case, the working with the unpleasant gas, hydrogen sulphide, is done away with entirely. As far as time is concerned, the method is short, requiring no longer time than the method of Chase.

In conclusion, I wish to here acknowledge the assistance of Mr. Clarence Ebaugh and Mr. John K. Faust in working out this method.

[1] See " Estimation of Cyanogen," by Wm. J. Sharwood: This Journal. **19,** 415.