

*Operation.*—One-half gram of the sample is placed in a porcelain or platinum dish; fifty cc. water, ten cc. hydrochloric acid (1.20 sp. gr.) and twelve cc. sulphuric acid (one part sulphuric acid, 1.84 sp. gr., to three parts water) are poured on it; heat until copious fumes of sulphuric acid are given off. Allow the dish to cool, so that there will be no spattering when taking up with acid. When cool, add about ten cc. hydrochloric acid, heat to soften the sulphate of iron, add about seventy-five cc. water, and bring to a boil. Discontinue the heating and note whether there is any effervescence when boiling ceases. If there is, the liquid must be evaporated until copious fumes of sulphuric acid are given off again, then taken up as before directed. Filter at once, wash thoroughly with hydrochloric acid (1 : 1) and hot water, ignite in a platinum crucible, and weigh. Add a few drops sulphuric acid and enough hydrofluoric acid to dissolve the silica. Evaporate to dryness, heat to decompose the sulphates, cool, and weigh. The difference in the two weights is silica, which can be calculated to silicon. The whole operation can be accomplished in thirty minutes.

The following are some results, both by our method and the fusion method :

No.	Our method.	Fusion method.
1	12.08	12.01
2	12.37	12.25
3	12.09	12.08
4	13.46	13.40
5	9.05	9.03

## A METHOD FOR THE COMPLETE ANALYSIS OF IRON ORES, WITH NOTES ON SÄRNSTRÖM'S METHOD OF DETERMINING MANGANESE.

BY GEORGE AUCHY.

Received January 25, 1897.

SÄRNSTRÖM'S method of determining manganese in iron ores, as described by Messrs. Mixer and DuBois, is to precipitate the iron in dilute hot solution by sodium carbonate, care being taken to add no more of this reagent than just enough to effect the precipitation of the iron; then titrating (without filtering from the precipitated ferric oxide) with standard permanganate. The writer, in experimenting with Volhard's method,

and with Stone's modification of Volhard's method, had found that in working these methods it was necessary to add the zinc oxide in considerable excess of the amount necessary to completely precipitate the iron, for the reason that titration of the manganese in imperfectly neutralized solution gives too high results. But in Särnström's method we have, apparently, titration in incompletely neutralized solution without any tendency to high results as a consequence, as is shown by the accuracy in Messrs. Mixer and DuBois' work. It was thought by the writer that this apparent anomaly could perhaps be explained by the assumption that in Särnström's method manganese is carried down by the iron precipitate, as happens in Volhard's method, if certain precautions be not observed, and that this error compensates for the error of high results due to titration in incompletely neutralized solution. But the uniform excellence of the results given by Messrs. Mixer and DuBois operate against this view, and the following experiments made by the writer also seem to show that Särnström's method is free from these sources of error :

Manganese present. Per cent.	Manganese found. Per cent.	Number of cc. permanganate re- quired, theoretical.	Number of cc. permanganate re- quired, actual.
0.60	0.60	2	2.0
0.60	0.60	2	2.0
0.60	0.60	2	2.0
0.60	0.60	2	2.0
1.80	1.68	6	5.6
1.80	1.80	6	6.0
3.60	3.54	12	11.8
3.60	3.60	12	12.0
7.20	6.96	24	23.2
1.80	1.80	6	6.0
1.80	1.80	6	6.0
1.20	1.20	4	4.0
1.20	1.14	4	3.8
1.80	1.74	6	5.8
1.80	1.74	6	5.8
0.60	0.60	2	2.0
2.40	2.37	8	7.9
3.00	2.97	10	9.9
3.60	3.54	12	11.8
4.80	4.74	16	15.8
4.80	4.74	16	15.8

Manganese present. Per cent.	Manganese found. Per cent.	Number of cc. permanganate re- quired, theoretical.	Number of cc. permanganate re- quired, actual.
6.00	5.94	20	19.8
1.80	1.81	6	6.05
1.20	1.19	4	3.95
2.40	2.37	8	7.9
3.60	3.66	12	12.2
4.80	4.84	16	16.15
6.00	5.97	20	19.9
7.20	7.14	24	23.8

It would seem from these results that there is a tendency to slightly low results ; but that this is not due to manganese carried down with the iron precipitate seems shown by the fact that the same tendency exists when no iron is present. The following tests were made with manganese solutions free from iron, having exactly the same amounts of free acid present as in the previous experiments, and also using exactly the same amounts of sodium carbonate for neutralization.

Manganese present. Per cent.	Manganese found. Per cent.
0.60	0.60
0.60	0.60
0.60	0.57
1.20	1.20
2.40	2.37
3.60	3.54

There seems to be an entire absence of the tendency to high results existing in Volhard's method when titration is performed in incompletely neutralized solution. But further experiments led the writer to question whether, after all, he was right in his assumption that the titration in Särnström's method takes place in incompletely neutralized solution ; and the experiments following seem to indicate that the explanation of the mystery lies in the fact that in Särnström's method either the ferric oxide by its presence, in some way, prevents high results when solutions are incompletely neutralized, or the ferric oxide by its presence, in some way, prevents the precipitation of the manganese dioxide by titration, except the solution be thoroughly neutralized when titrated, the permanganate simply coloring the solution, and no manganese dioxide precipitating until more sodium carbonate is added. If the solution—incom-

pletely neutralized, but sufficiently so to precipitate the iron—be filtered from the precipitated ferric oxide before titration, the manganese in that case is not held up (if the solution be not too acid) but precipitates immediately upon the addition of the permanganate, and the result then becomes too high, as in Volhard's method, under similar conditions of neutralization.

Manganese present. Per cent.	Manganese found; filtered from the ferric oxide be- fore titration. Per cent.	Manganese found; filtered from the fer- ric oxide, but more sodium carbonate added before titration. Per cent.
3.60	3.66	3.60
3.60	3.63	3.60
3.60	3.66	....
3.60	3.75	....
3.60	3.75	....

It should be noted that when in the performance of the method by the regular way, the neutralization happens to be imperfect, and the permanganate therefore fails to throw down the manganese dioxide, and more sodium carbonate is consequently added, the result is apt to be too high if *much* more sodium carbonate is required, as appears from the following :

Manganese present. Per cent.	Manganese found. Per cent.
2.40	2.43
0.60	0.67
0.60	0.63

If little more sodium carbonate is required the result is not affected.

Manganese present. Per cent.	Manganese found. Per cent.
0.60	0.60
0.60	0.60
1.80	1.80
2.40	2.37
4.80	4.74
6.00	5.94
1.80	1.81
1.20	1.23

At all times an excess of sodium carbonate must of course be guarded against, but the exact point of neutralization can invariably and easily be obtained by performing the process as follows: Add seven grams of ammonium chloride; make the bulk of the solution 400 or 500 cc., add sodium carbonate *in the*

*cold* to a slight but permanent turbidity; toward the last the sodium carbonate solution is added drop by drop from a burette; when a slight turbidity appears, which, if anything, increases by standing a minute but which shows no sign of a distinct precipitate, the reaction is complete (Herschel and Schwartzberg). Bring to a boil with frequent stirring, which precipitates the iron completely; then add one cc. more of sodium carbonate solution (one pound of crystallized salt to one liter of water), and titrate with permanganate. By performing the process in this way accurate results are invariably obtained. The ammonium chloride present makes the danger of manganese precipitating through an excess of sodium carbonate very remote, even if much more carbonate be added than directed. One-tenth cc. of permanganate (when the strength is 0.0056) should be deducted from the reading of the burette, as a performance in blank shows that that much permanganate is required to give a distinct color to a solution of that bulk.

The phenomenon noted in the examination of Volhard's method, that titration in nitric acid solution requires a deduction to be made in the result of 0.02 per cent.<sup>1</sup> also occurs in Särnström's method, as was shown by two experiments.

The Särnström method may be successfully used in steel analysis provided—as Messrs. Mixer and DuBois have said—that organic matter be first destroyed. But Stone's modification of Volhard's method is preferable, since in that method the organic matter does not interfere, being carried down with the ferric oxide and filtered off, and hence the time consumed in destroying the organic matter may be saved.

It is the writer's experience that the color method is, all things considered, as good as any for common use. But it is perhaps worth remarking that in the color method a standard *solution* is preferable to standard drillings, the time and labor of weighing out and dissolving the standard drillings for every analysis being thereby saved. In making up the standard manganese

<sup>1</sup> It is better to say that a deduction of 0.2 cc. permanganate (strength 0.0056) is required. With the weight of sample taken for analysis by Volhard's method this amounts to 0.02 per cent., as stated. But in Särnström's method only about one-third of this weight can be taken, and the deduction therefore would be 0.06 per cent. instead of 0.02 per cent. It would therefore have been better if in the article on Volhard's method the writer had directed a deduction of 0.2 cc. permanganate instead of a deduction of 0.02 per cent. in the result.

solution, the amount of nitric acid used for solution of the standard steel should, of course, be such that 100 cc. of the standard solution will contain as much acid as is used for solution of the steel to be tested. Ten cc. of the standard solution is in each case used, three cc. dilute nitric acid added, boiled with lead peroxide, etc.

#### A METHOD OF ANALYZING IRON ORE.

In making a complete analysis of iron ore it seemed an obvious application of Särnström's method to separate manganese, lime, and magnesia from iron by precipitating the iron as in that method, and filtering before titrating with permanganate. The writer had no access to Särnström's original article, but from the tone of the description of it given by Messrs. Mixer and DuBois, inferred that no such application is made by Särnström. In Crookes' Select Methods iron is separated from manganese in this way; also manganese from zinc; but there is no mention of lime and magnesia in this connection. Fresenius (old edition) gives a method by Herschel and Schwarzenberg for separating "iron sesquioxide from nickel, cobalt, zinc, manganese, and other strong bases" by neutralizing with ammonium carbonate in the cold in presence of much ammonium chloride, till the liquid loses its transparency, and does not clear up after a moment's standing, but, if anything, shows an increased cloudiness, without showing the least trace of a distinct precipitate; then bringing to a boil and boiling to expulsion of the carbon dioxide, and filtering from the completely precipitated basic iron oxide. But the writer was not sure that lime and magnesia were included in the term "strong bases;" and moreover, had much doubt as to the efficacy of the separation in the case of alumina. Experiments were therefore made on his own account, which showed that in carrying out the process, as already described in the first section of this article, namely, neutralization with sodium carbonate in the cold to slight but permanent turbidity, but with no trace of a distinct precipitate showing, then bringing to a boil, adding afterwards one cc. more of sodium carbonate solution, and filtering from the iron oxide, it occurs:

1. That in the absence of alumina all the zinc, manganese, and lime are found in the filtrate from the ferric oxide, but not

all the magnesia, which latter requires therefore a repetition of the operation for a complete separation from the iron.

2. That if seven grams of ammonium chloride be added to the solution before neutralization with sodium carbonate and precipitation of the iron, the separation of the magnesia, as well as that of the manganese, zinc, and lime, is, in that case, thorough and complete in one precipitation.

3. That if alumina be present, however (seven grams of ammonium chloride being also present), the alumina not only is not itself completely separated from the iron in one precipitation, but the small amount of alumina precipitating with the iron also carries with it a small amount of the magnesia or lime, or both (the test was made by adding phosphate and ammonium chloride to the concentrated filtrate from a basic acetate precipitation), and therefore two, and very often three precipitations are necessary to get the ferric oxide completely free from alumina, lime, and magnesia.<sup>1</sup>

4. That the presence of alumina in solution with the manganese, after filtration from the ferric oxide, interferes with the titration with permanganate, as the addition of the small quantity of sodium carbonate necessary to a thorough neutralization before titration, partially precipitates the alumina, and this keeps the manganese dioxide precipitate from balling together, thereby preventing any observation of the color of the liquid; while if enough sodium carbonate be added to completely precipitate the alumina—although the titration can then be successfully performed—the result will be too low, the alumina apparently carrying down some of the manganese.

5. That if the alumina be precipitated by ammonia in slight excess, boiling off the excess, no manganese is carried down, and the manganese in the filtrate from the alumina may be conveniently and accurately determined by titration with permanganate.

With these facts ascertained, the scheme for analysis of iron ores would obviously be as follows:

<sup>1</sup> It should be noted, however, that the experiments on this point were made with a solution containing also titanous acid, equivalent to one per cent.; so that it is probably the titanous acid which carries down the alumina. But as titanous acid in small amount is so frequently a constituent of iron ores, it was not thought necessary to investigate this point.

Take one-half gram of the ore for analysis. Dissolve and separate silica as usual. To the filtrate from the silica add two hornspsoonsful (about seven grams) of ammonium chloride, and dilute to 275 cc. (or 300 cc. in rich ores). Add sodium carbonate solution (or the main part of the neutralization may conveniently be made with the solid carbonate<sup>1</sup>) from a burette, toward the last, after the solution turns from yellow to red in color, drop by drop, till the appearance of a faint cloudiness which persists on standing a minute, or even increases, without, however, showing any trace of a distinct precipitate.<sup>2</sup> Then bring to a boil with frequent stirring, the ferric oxide beginning to precipitate as soon as the heat is applied. Boil a few minutes. Add five drops more of sodium carbonate from the burette (about three-tenths cc., sodium carbonate solution of strength one pound crystallized salt to a liter of water) and stir. Allow to stand until the ferric oxide precipitate has completely settled. Filter and wash (not by decantation) with hot water.<sup>3</sup> The ferric oxide precipitate contains besides the titanitic acid and phosphoric acid, also a small portion of the alumina, lime, and magnesia. Dissolve on the filter in hot hydrochloric acid, add the solution of the alumina later obtained, and precipitate with ammonia. Filter and weigh, and estimate alumina by difference. The filtrate from this precipitate—containing the small amount of magnesia or lime, or both, coming down with the iron in the first precipitation—is added to the main lime and magnesia solution after the latter has been freed from the manganese, as described later.

The filtrate from the first ferric oxide precipitate containing all the manganese, and all but a small part of the lime, alumina, and magnesia (and 0.01 or 0.02 per cent. of phosphoric acid if the phosphorus in the ore be extremely high, afterwards precipitated with the alumina) is brought to a boil and boiled a minute to expel traces of carbon dioxide (a very necessary pre-

<sup>1</sup> Ammonium carbonate, if free from organic matter, doubtless answers just as well, or probably better, as ammonium salts are desired in the solution, while sodium salts are of no use. But the writer prefers sodium carbonate on account of cheapness, and on account of being free from organic compounds, which interfere with the subsequent titration with permanganate.

<sup>2</sup> In observing the liquid, it is well to do so both by holding it between the light and the operator, and by reflected light. The cloudiness is best recognized, however, by working by reflected light.

<sup>3</sup> A nine cm. filter paper is hardly large enough, and either a size or two larger should be used, or else two nine cm. filters. After the first two washings the precipitate should be stirred up by the jet.

caution), ammonia added in slight excess, and the excess boiled off. The precipitated alumina, free from manganese, but carrying a little magnesia when much is present, is allowed to settle, filtered (by siphon preferably), and washed with hot water. Dissolve in hydrochloric acid and add to the ferric oxide solution. The filtrate from the alumina is brought to a boil and titrated with standard permanganate. Deduct from the reading of the burette the number of cc. of the permanganate taken up by the organic matter of the ammonium chloride used—previously ascertained by a trial in blank—and calculate the percentage of manganese. Add a small crystal or two of oxalic acid, and some hydrochloric acid to get the precipitated manganese again into solution (the manganese dioxide cannot be filtered off because it carries magnesia). Destroy the excess of oxalic acid by titrating with permanganate solution. Evaporate down and precipitate and filter the manganese (as sulphide) as directed in Blair's Chemical Analysis of Iron, and finish as usual—but determining the lime by titration with permanganate.

This method of analysis has the advantage over the basic acetate in that the precipitation of the iron takes place in small, instead of large, bulk of solution, with a consequent saving of time in evaporation; the separation from the manganese by the former process, moreover, being thorough and complete, while by the basic acetate, two basic acetate precipitations are necessary for a thoroughly accurate separation. If two basic acetate precipitations be made, the proposed method is very much quicker, but is also quicker if only one basic acetate be made. In the basic acetate method, no separate precipitation and filtration of the alumina is involved, but this precipitation and filtration requires considerably less time than is required to evaporate down the filtrate from the basic acetate precipitation to a workable bulk. Another advantage in the proposed method is in the determination of manganese by titration instead of by the rather tedious and troublesome gravimetric process. Still another is in the fact that the iron precipitate by the proposed method is one that shows no tendency to run through the filter, settles more rapidly, and is washed with much more ease than the basic acetate precipitate; moreover, this precipitation is simpler and more likely to be successfully performed by a beginner.

Following are some manganese results obtained in this way :

No.	Manganese present.	Manganese found.
	Per cent.	Per cent.
1	3.60	3.60
2	3.60	3.60
3	3.60	3.60
4	3.60	3.60
5	3.60	3.60
6	3.60	3.54
7	3.60	3.60
8	3.60	3.36

The last result of the series is a poor one, some of the manganese evidently having precipitated with the alumina. This seemed a very singular circumstance in view of the fact that it is laid down by authorities that manganese is completely separated from alumina by precipitation of the latter with ammonia, and boiling off the excess ; all the writer's other experiments also went to prove the same thing. It was thought that perhaps the manganese had come down with the iron precipitate instead of with the alumina, but a careful examination of the iron precipitate showed no trace of manganese. Finally it occurred to the writer that a little carbon dioxide might have been in solution at the time of the alumina precipitation—it was remembered that the ammonia had been added before the liquid had been brought to a boil. To see whether in such an event manganese would precipitate a test was made by taking standard manganese solution, adding to it the amount of hydrochloric acid used in ore analysis, nearly neutralizing with sodium carbonate as usual, heating, but not to boiling, then adding the ammonia in slight excess, and bringing to a boil. Manganic oxide was seen to precipitate ; hence the necessity for the precaution already spoken of, of boiling the liquid before precipitating the alumina with ammonia.

That titration of the manganese solution, obtained in this way, by permanganate succeeds when no carbon dioxide is present, is shown by the following tests :

No.	Manganese present.	Manganese found.
	Per cent.	Per cent.
1	3.00	3.03
2	1.20	1.19
3	1.80	1.80
4	2.40	2.40

If in the ore to be analyzed, zinc, nickel, and cobalt are to be tested for, the scheme of analysis just described must be modified as follows :

In the filtrate from the ferric oxide precipitate the alumina, manganese, nickel, cobalt, and zinc with ammonium sulphide, filter, dissolve in hydrochloric acid, filter from sulphides of nickel and cobalt remaining undissolved, and make a basic acetate separation in small bulk, and separate the zinc, nickel, cobalt, and manganese as usual, except that manganese is determined by titration, in solution made neutral by adding ammonia and boiling off the excess, instead of being determined as phosphate.

Another scheme of analysis by which alumina is directly estimated and iron is determined volumetrically in the same sample is to make *two* ferric oxide precipitations by neutralization with sodium carbonate as described, uniting the filtrates, precipitating, filtering, and weighing the alumina (if zinc, nickel, and cobalt are not to be tested for), which may afterwards be tested for iron ; titrating the filtrate from the alumina for manganese, etc. ; the ferric oxide precipitate being dissolved, reduced, and titrated for iron, no separate iron determination being made. But if this procedure be followed it is absolutely necessary to make a third ferric oxide precipitation as described, by way of precaution ; and if much titanitic acid be present the writer is doubtful if even three precipitations will always free the ferric oxide entirely from alumina (and from magnesia or lime, or both, which comes down as long as the alumina does). although he has not, as yet, made any experiments on this point. In the table of manganese results, numbers 2 to 5, inclusive, were obtained in this way.

For the experiments on these methods a solution was used containing iron equivalent to forty per cent. ; alumina equivalent to ten per cent. ; manganese 3.60 per cent. ; lime fifteen per cent. ; magnesia fifteen per cent. ; phosphoric acid two per cent. ; titanitic acid one per cent. These experiments need not be given in detail.

Further experimenting—which the writer at present has not the opportunity to perform—is intended. For instance, in the first method of separation described—precipitation of the iron by

neutralization with sodium carbonate, followed by a second precipitation of the iron (and alumina) by ammonia—when zinc, nickel, and cobalt are present, experiment might perhaps prove it feasible to completely precipitate these metals as sulphides by hydrogen sulphide in the filtrate from the first ferric oxide precipitate; afterwards precipitating the alumina by ammonia and determining the manganese by titration as directed in the scheme for analysis when no zinc, nickel, and cobalt are present. Nickel and cobalt could undoubtedly be completely precipitated in this way, and the writer has hopes that the zinc, also, would be completely thrown down from the dilute, nearly neutral solution. At least by the aid of sodium dithionate, the zinc would be completely precipitated.<sup>1</sup> In that case, this method of separation would be simpler and more convenient than the one suggested. In the method suggested it would perhaps be better to separate the zinc, nickel, and cobalt from the alumina by ammonia and ammonium chloride instead of by a basic acetate precipitation.

No experiments have been made by the writer regarding the accuracy of the separation of the magnesia from the iron and alumina by these two precipitations described in the first method (precipitation by neutralization with sodium carbonate, followed by precipitation by ammonia). It was not thought necessary to experiment on this point, because authorities unite in considering the separation of iron and alumina from lime and magnesia by a precipitation of the former by ammonia to be sufficiently accurate—one precipitation being considered by Blair sufficient to separate all the lime and magnesia from the iron and alumina; in this method two are used—only one by ammonia, but the first precipitation, by neutralization, in all probability separates nearly as large a proportion of the magnesia as a precipitation by ammonia would do, and quite as large a proportion of it as a basic acetate precipitation would effect. But it is considered that the basic acetate separation of magnesia from iron and alumina is rather better than the separation by ammonia; and if desired, the second ferric oxide precipitation of the method suggested may of course be made by basic acetate. But in this event it is the writer's opinion that the precipitation may be made in much less

<sup>1</sup> J. Ribon: *Bull. Soc. Chim.*, 50, 518.

bulk (say 400 cc.) to separate the iron and alumina from the trifling amount of magnesia remaining from the first separation by neutralization, than is ordinarily required.

A quicker way of performing the first method than the one described (zinc, nickel, and cobalt being absent) would be to precipitate the aluminum and manganese by ammonium sulphide in the filtrate from the first ferric oxide precipitate; then filtering, dissolving in hydrochloric acid, separating alumina by precipitating with ammonia, boiling off excess; titrating the filtrate for manganese; filtering off the precipitated manganese dioxide; and adding filtrate to the main lime and magnesia solution (which has meanwhile been evaporated down) to recover the lime and magnesia (if any) carried down by the ammonium sulphide precipitation of the alumina and manganese. But, as before stated, the manganese dioxide precipitate carries down magnesia; but doubtless not when the magnesia present is small in amount as would be the case here. This point also, however, must be tested by experiment.

The Särnström reaction may evidently also be made use of in zinc ores for separating iron, alumina, and manganese from zinc, previous to titration of the latter with ferrocyanide. The method at present most in use, at least in the West, seems to be that of von Schulz and Low. But Mr. Bertrand Hinman finds their method inapplicable to eastern ores (on account of the insolubility of these ores, and also on account of the higher percentage of iron which makes the separation from zinc by ammonia and ammonium chloride an incomplete one) except certain modifications be adopted. Mr. George C. Stone, however, finds that even with these modifications the method does not work with New Jersey ores, and originates another method in which the iron and aluminum are separated from the manganese and zinc by precipitation with barium carbonate; and the latter titrated with ferrocyanide, manganese being determined in a separate portion, and the zinc then estimated by difference. Very accurate results are obtained by this method, but it would be just as accurate and convenient to apply Särnström's method as follows: Obtain the ore in hydrochloric acid solution. If not enough iron be present add ferric chloride solution in sufficient

<sup>1</sup> This Journal. 17, 473.

amount. Add about seven grams ammonium chloride; dilute to 300 cc.; neutralize in the cold with sodium carbonate till the liquid loses its transparence as previously described; bring to a boil and boil a minute; add one cc. sodium carbonate solution and mix; titrate for manganese with permanganate; filter from the mixed iron and manganese precipitate (according to Crookes, page 200, no zinc comes down with the manganese dioxide precipitated in this way) and wash with hot water; and titrate the filtrate with ferrocyanide for zinc. Instead of filtering and washing the mixed ferric oxide and manganese dioxide precipitate, however, it would be better to dilute to a definite volume, and, after mixing, decant through a filter an aliquot portion.

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The writer takes this opportunity to make a few additional remarks on the determination of phosphorus in steel and cast iron. In a recent paper on that subject he advocated the use of double filters of Schleicher and Shüll's No. 579 or 589 black ribbon for the filtration of the yellow phosphomolybdate precipitate by aid of the pump, using the same filters for a number of different filtrations in succession. To this suggestion he wishes to add that these filters can very well be also used again the following day, or after having been dried out, for the same purpose; but that, for such an extended use, the number 589 black ribbon answers better than No. 579. The filters should not be unfolded when placed away for another use.

In filtering from the undissolved zinc through cotton wool, as in the method described in the article referred to, the same cotton wool may be made to answer for two consecutive filtrations, provided the percentage of phosphorus in the second of the two be not too low (under 0.05 per cent.). Below 0.05 per cent., fresh cotton should be used so as to get as rapid a filtration as possible. Several uses of the same filter clog it considerably. Instead of performing this filtration by the pump, it is more convenient, owing to the extreme quickness and facility with which the liquid is drawn through the cotton wool, to use the mouth, by means of a clean rubber tubing of convenient length connected with the filtering flask.

Twenty-five cc. sulphuric acid (two of water to one of acid) was suggested for acidification of the phosphorus solution pre-

vious to the addition of a mustard-spoonful of zinc. If with the zinc in use it is found that this amount of acid dissolves it too quickly, or not quickly enough, the proper amount of acid to be used is found by varying the quantity added by one or two-tenths cc. If, for instance, twenty-five cc. are found to dissolve the zinc too quickly, so that the color of the reduced solution is only wine colored instead of green, as it should be, then 24.9 cc. will usually be found the right quantity for use on every occasion. It should perhaps be pointed out to those who have never used the Emmerton process that in titrating the reduced solution, the end point of the titration does not for some reason manifest itself with such suddenness as in the reductor process, and care must therefore be taken to note that the pink coloration is a distinct and permanent one.

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[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF CINCINNATI.]

## LI. THE USE OF ALUMINUM FOR CONDENSERS.

By T. H. NORTON.

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IN connection with the extended use of aluminum in this laboratory for various forms of apparatus, water-baths, air-baths, Bunsen burners, hot water filtering funnels, etc., it seemed desirable to study the availability of the metal for condensation processes.

For this purpose a condenser was constructed as follows: The outer jacket was of glass; the inner tube was of aluminum and possessed the following dimensions: length, 122 cm., external diameter, one cm., inner diameter, eight and one-half mm., weight per meter, twenty-nine grams. At a distance of fifteen cm. from the end, the tube was bent at right angles. This permitted of connection with a distilling flask, without allowing the condensing vapors to come in contact with any substance but aluminum. It might be mentioned here that in order to bend an aluminum tube of these dimensions satisfactorily, it is necessary to fill it with molten lead, and further, that several distillations with water are requisite in order to remove completely slight traces of lead adherent to the surface of the aluminum, after this operation.

The method of testing the condenser was to distil a measured

<sup>1</sup> Read before the American Chemical Society at the Springfield Meeting.