

revolutions of the earth, this must be decided by geology; but the remains exist.

Now in which manner do the organic substances of these animals become decomposed?

The animal substance consists essentially of nitrogenated material and fat. The former is easily decomposed, the latter is very stable, a fact which has been very well known for a long time and has been shown again by exact investigations. Therefore we find the wax of cadavers in old graves, therefore the fat in the bones of mammals thousands of years old, therefore the fat on the bottom of the ocean recently found.

Whether and how far the fat was decomposed in this long period by the water splitting up glycerol and forming the free acid, for instance the fat in the bones of mammals, cannot be answered. Both, fat as well as the fatty acids form petroleum, when distilled under pressure.

We can imagine, that such remains wrapped in mud and transported by the currents in the ocean, easily accumulate and later on, under the pressure of sedimentary layers or strata, perhaps under the influence of heat too, are transformed into petroleum. This is only one of the many possibilities by which the mechanical process of the transmutation of fat into petroleum may have happened.

Under any circumstances I think I have proven that from the chemical standpoint, the formation of petroleum from animal remains has the greatest probability, as we are able now to transform every animal fat into petroleum.

THE DETERMINATION OF PHOSPHORIC ACID BY THE TITRATION OF THE YELLOW PRECIPITATE WITH STANDARD ALKALI.¹

BY HENRY PEMBERTON, JR.

I N the year 1882, I described a process for determining phosphoric acid, volumetrically, by ammonium molybdate, on the principle of Wildenstein's sulphuric acid determination, or of Gay Lussac's silver method. An *aqueous* solution of am-

¹ Read at the stated meeting of the Chemical Section of the Franklin Institute, held September 19, 1893. Communicated by the author.

monium molybdate is run into the solution of the phosphate until no further precipitate is formed.

But it is not of this process that I now have to speak. It is referred to here, in order to draw attention to the concluding paragraph of the paper, as follows:

"I have obtained very sharp and accurate results by determining the amount of yellow precipitate (formed as above, after thorough washing), by means of a standard solution of caustic alkali, using litmus as an indicator; a description of which I hope to present in a future paper. I mention it here simply to place the fact on record." (*Journ. Frank. Inst.*, **113**, 193; *Chem. News*, **46**, 7.)

At that time I did a considerable amount of work upon the last-mentioned process, with very satisfactory results. A study was made of the conditions most favorable to obtaining a phosphomolybdate precipitate of constant composition, using solutions of disodium hydrogen phosphate of known strength, and also a solution of apatite, the determinations being checked by standard methods.

Before the process was in shape for publication, however, my attention was called to work of an entirely different nature, and no description of the method was published other than that embodied in the above-quoted paragraph.

Since that time several chemists have described processes based upon the same principle.

E. Thilo, in the analysis of Thomas slag (*Chem. Ztg.*, **11**, 193), dissolves the yellow precipitate in standard ammonia, and titrates back with acid, using litmus as an indicator.

Franz Hundeshagen (*Ztschr. anal. Chem.*, **28**, 171) uses standard sodium hydroxide in excess, and titrates back with nitric acid, using phenolphthalein as an indicator.

C. E. Manby (*J. Anal. Appl. Chem.*, **6**, 82) determines the phosphorus in steel, iron, and iron ores, by dissolving the yellow precipitate in ammonia, acidifying with nitric acid, evaporating to dryness, and heating gently to expel nitric acid and ammonium nitrate. He then titrates, using the same solution and indicator as Hundeshagen employs.

James O. Handy (*J. Anal. Appl. Chem.*, **6**, 204) avoids

the evaporation and heating and titrates directly as Thilo and Hundeshagen do, using standard soda and phenolphthalein. M. Rothberg and W. A. Auchincrole (*J. Anal. Appl. Chem.* **6**, 243) also describe the same process.

Although it is now eleven years since I drew attention to this process, its advantages are so great that any information touching it, in addition to that furnished by the foregoing chemists, may be of interest. It far surpasses in quickness the process described by me (on the Gay Lussac or Wildenstein principle) and at the same time lacks nothing in accuracy. In most of the papers of the above-mentioned chemists, the process is applied to the determination of phosphorus in small quantities, as it occurs, for instance, in iron, steel, or ores. During the past year, I have had occasion to apply it to the examination of a number of phosphate rocks, as well as of strong solutions of phosphoric acid, containing over fifty per cent. P_2O_5 , and the method has been used continuously during that time. I am indebted to Mr. Edwin Harris, who had charge of most of the laboratory work of the factory, for many of the figures given below. It was seldom that two tests of the same material differed more than 0.1 per cent. in P_2O_5 , even when the total P_2O_5 present amounted to as much as forty per cent. to fifty per cent. of the substance analyzed.

The following solutions are used:

Ammonium Molybdate.—Ninety grams of the crystals are dissolved (in a large beaker) in somewhat less than one liter of water. This is allowed to settle, over night, and the clear liquor decanted into a liter flask. The small quantity of insoluble molybdic acid, always present, is dissolved in a little ammonia and added to the main solution. Should the molybdate be found to contain traces of P_2O_5 , a few decigrams of magnesium sulphate are added, ammonia being added to faint alkalinity. The whole is then made up to one liter. It is this *aqueous* solution that is used, *no nitric acid whatever being employed*. Each cc. precipitates three milligrams of P_2O_5 .

The *ammonium nitrate* solution is simply a saturated aqueous solution of the salt. Distilled water is poured into the bottle of crystals in quantity insufficient to dissolve them all. Even in

cold weather, ten cc. of this solution is amply sufficient for each test.

The *nitric acid*, used for acidifying the solution of the phosphate, has a specific gravity of 1.4 or thereabouts.

The *standard potassium hydroxide* solution is of such strength that one cc. = one mgm. P_2O_5 . One hundred cc. of it will neutralize 32.65 cc. of normal acid. It can be made from normal potassium hydroxide (that has been freed from all carbonate by barium hydroxide), by diluting 326.5 cc. to one liter. But its strength is best determined empirically by a direct test upon a phosphate solution of known strength, precipitating with ammonium molybdate and making the analysis as described below, all potassium carbonate having first been removed by barium hydroxide.

The *standard acid* has the same strength, volume for volume, as the potassium hydroxide and can be made by diluting 326.5 cc. of normal acid to one liter. In testing it against the alkali, phenolphthalein (and methyl orange) should be used.

The *indicator* can be either litmus, rosolic acid, or phenolphthalein. I have used the latter almost exclusively, as it has been shown, by J. H. Long (*Am. Chem. J.*, **11**, 84) that titrations with this indicator in the presence of ammonium salts are perfectly reliable if the amount of the ammonium salt present is not excessive, if the solution is cold, and if the phenolphthalein is used in sufficient quantity. One gram of the phenolphthalein is, accordingly, dissolved in 100 cc. of sixty per cent. alcohol, and at least 0.5 cc. of this solution is used for each titration. The washing of the ammonium phospho-molybdate is done by water. (Isbert and Stutzer, *Ztschr. anal. Chem.*, **26**, 584.) It may be of interest to quote from their results, since it is by some chemists thought necessary to wash with a neutral or acid solution of an ammonium salt. In all tests, as made by them, twenty-five cc. of the sodium phosphate solution were precipitated by ammonium molybdate and the phosphoric acid determined therefrom, as magnesium pyrophosphate in the usual manner.

When the yellow precipitate was washed with ammonium nitrate solution, there were obtained :

- (1)..... 0.1943 gram P_2O_5 in 50 cc.
 (2)..... 0.1948 gram P_2O_5 in 50 cc.

When washed with water, there was found :

- (3)..... 0.1947 gram P_2O_5 in 50 cc.
 (4)..... 0.1942 gram P_2O_5 in 50 cc.

In order to establish the fact more certainly, the precipitate was washed with unusually large quantities of water :

	cc. of water used in washing.	P_2O_5 found.
(5).....	300	0.1947 in 50 cc.
(6).....	400	0.1944 in 50 cc.
(7).....	500	0.1948 in 50 cc.
(8).....	1,000	0.1940 in 50 cc.

There is, accordingly, no danger of loss in washing the yellow precipitate with water.

The following is the method of performing the analysis :

One gram of the phosphate is dissolved in nitric acid, an excess of which can be used with impunity, and the solution filtered into a 250 cc. flask and made up to the mark. The solution can even be poured into the flask without filtering, since the presence of a little insoluble matter does not interfere in the least with the titration. Moreover, since most phosphate rocks seldom contain over ten per cent. of insoluble matter, and as this has the sp. gr. of at least 2, it occupies a volume of about 0.05 cc., an amount so small that it may be neglected. (For instance, even in the case of a phosphate rock containing forty per cent. P_2O_5 , the error is only 0.008 per cent. P_2O_5 .)

After the clear solution has been poured off, it is well to treat the sand, etc., at the bottom of the beaker, with a cc. or so of hydrochloric acid, in the warmth, to insure complete solution.

It is not necessary to evaporate to dryness. Isbert and Stutzer have shown, in their paper, that when the yellow precipitate is washed with water, the soluble silica is removed, and that evaporation (to render the silica insoluble) is superfluous. Their results are corroborated by the test analysis that will be given below. In the event of its being desirable to remove silica by evaporation, for any purpose, the evaporation should be performed over a water bath, or, if on an iron plate, with great care, since, otherwise, meta or pyro phosphates are formed with results that are correspondingly low.

Twenty-five cc. of the solution (equal to 0.1 gram) are now

taken for analysis. It may be thought, by some, that an analysis made upon so small a quantity of material as one decigram, and with a standard solution representing only one milligram per cc., may be liable to errors that would not exist, when using larger quantities or stronger solutions. But it should be borne in mind that the accuracy of measurement with a twenty-five cc. pipette, is precisely the same, whether ten grams of the original substance are taken or only one gram. Any error in measuring with a pipette is, of course, entirely independent of the quantity in solution. In regard to the amount of material to be manipulated (filtered, washed, etc.) it will be remembered that the weight of the yellow precipitate is over twenty-eight times the weight of the P_2O_5 contained in it. Every milligram of P_2O_5 is accordingly represented by more than twenty-eight milligrams of precipitate. The standard alkali, although representing only 0.001 gram per cc. is in reality more than three times as strong as the decinormal solution generally employed. Of course, in the case of materials containing only ten to fifteen per cent. P_2O_5 , as in fertilizers, two or three grams can be taken for analysis, if desired, instead of one gram.

Returning, therefore, to the method of the analysis, twenty-five cc. of the solution are measured out and delivered into a beaker holding not more than 100 to 125 cc. A large beaker requires unnecessary washing to remove the free acid in washing the yellow precipitate. The solution is neutralized with ammonia—until a precipitate just begins to form—and five cc. of nitric acid of sp. gr. 1.4 added. Ten cc. of the ammonium nitrate solution are added, and the entire bulk of the solution made up to fifty to seventy-five cc. by adding water.

Heat is now applied and the solution brought to a full boil. It is then removed from the lamp, no more heat being applied and treated *at once*, with five cc. of the aqueous solution of ammonium molybdate, which is run into it from a five cc. volume pipette, the solution being stirred as the precipitate is added. The beaker is now allowed to rest quietly for about one minute, during which time the precipitate settles almost completely. The five cc. pipette is filled with the molybdate solution, and a part of its contents allowed to drop in, holding the beaker up

to the light. If a formation of a yellow cloud takes place, it is at once perceptible, in which case the remainder of the pipette full is run in, the solution stirred and allowed to settle. A third pipette full is now added as before. Should it cause no further cloud, only about one-half of its contents is added, the remainder being run into the beaker into which the filtrate and washings from the yellow precipitate are to go. In the test analyses given below, it will be shown that even when *fifteen cc. in excess* of the molybdate, were purposely used, over and above the calculated amount, the results were accurate—no molybdic acid coming down with the yellow precipitate.

It is seldom that more than fifteen cc. in all (three five cc. pipettes full) of the molybdate have to be added. Since each cc. precipitates three milligrams P_2O_5 , fifteen cc. will precipitate forty-five milligrams P_2O_5 . This is equivalent to forty-five per cent. on the 0.1 gram taken for analysis, and it is not often that any material to be examined contains over this percentage. In the analysis of materials rich in phosphoric acid, it is one of the embarrassing features of the usual process, in which the *nitric acid solution* of the molybdate is used, that, in the first place, large quantities of the precipitant have to be used (frequently several hundred cc.), and, in the second place, that the analyst is never certain that enough has been added to throw down all of the phosphoric acid. This necessitates frequent testings of small portions of the phosphate solution, or of the filtrate. There is another difficulty peculiar to the process as usually carried out in all methods in which the determination is made directly upon the phospho-molybdate itself, in that much care must be observed to keep the solution at a certain temperature, since otherwise molybdic acid contaminates the precipitate and the analysis is rendered worthless. In the process herein described, using an *aqueous* solution of the molybdate, the point at which sufficient of the precipitant has been added is easily seen. No molybdic acid separates, because, in the first place, no great excess of molybdate is added; and because, in the second place, the solution is filtered immediately, or as soon as it has settled, which requires only a minute or two. The time required from the first addition of the molybdate to the beginning of the filtra-

tion is never over ten minutes, and is generally less. The filtrate and washings from the precipitate when treated with additional molybdate solution, give, on standing on a hot plate for an hour or so, a snow-white precipitate of molybdic acid, showing that all of the phosphoric acid has been precipitated. I have observed this hundreds of times.

A slight correction should be made to the statement made above in regard to fifteen cc. of the molybdate precipitating forty-five milligrams of P_2O_5 . This is not strictly true, for the reason that a small quantity (something over one cc.) of the molybdate is required to neutralize the solvent action of the nitric acid. Therefore, in *very* high grade phosphates a fourth five cc. pipette full may be required.

The yellow precipitate is now filtered through a filter seven centimeters in diameter, decanting the clear solution only. This is repeated three or four times, washing down the sides of the beaker, stirring up the precipitate, and washing the filter and sides of the funnel above the filter each time. The precipitate is then transferred to the filter and washed there. When the precipitate is large it cannot be churned up by the wash water and cannot be washed down to the apex of the filter. This is generally the case when there is over ten or fifteen per cent. phosphoric acid present in the substance analyzed. In such an event, I am accustomed to wash the precipitate back into the beaker, and to fill the funnel with water above the level of the filter, doing this two or three times, then washing the precipitate back into the filter. It is not necessary to transfer to the filter the precipitate adhering to the sides of the beaker.

It goes without saying that during the washing no ammonia must be present in the atmosphere of the laboratory. Inasmuch as the beaker, funnel, filter, and precipitate are small, the washing does not take long to perform. It requires, in fact, from ten to fifteen minutes, even when large precipitates (= thirty to forty per cent. P_2O_5) are handled. The precipitate and filter are now transferred together to the beaker. By pressure with the tip of the finger upon the double fold of the filter, it is easily given a sideways motion and lifted out of the funnel without any danger of breakage, the precipitate being still within it.

The alkali solution is run in until the precipitate has dissolved, at least twelve drops of the phenolphthalein (1:100) are then added, and the acid run in without delay until the pearly color disappears and the solution is colorless. The presence of the filter paper does not interfere in the least. The reaction of the indicator is not so sharp as when only acid and alkali are used, but it is easy to tell with certainty the difference caused by one drop of either acid or alkali. After deducting the volume of acid used from that of the alkali, the remainder gives the percentage of P_2O_5 directly, each cc. being equal to one per cent. P_2O_5 . Thus, if there are 28.3 cc. of alkali consumed, the material contains 28.3 per cent. P_2O_5 when one decigram is taken for analysis. From the time the twenty-five cc. are measured out until the result is obtained, from thirty to forty minutes are required.

I have applied this process to determinations of phosphoric acid in phosphates and fertilizers, and have had no experience in determining phosphorus in iron, steel, or iron ores. I am inclined to believe that in the presence of such large quantities of iron salts when using the *aqueous* solution of the molybdate it may be necessary to guard against contamination of the yellow precipitate by ferric hydrate, perhaps by using larger quantities of nitric acid than five cc., and perhaps by washing the precipitate at first, with dilute nitric acid. It may also be the case that the yellow precipitate will form more slowly.

The following analytical experiments were made in order to test the process.

Several sodium phosphate solutions were used at first, the strength of which was only approximately known, as the aim was to see how closely two readings would agree; equal quantities of the phosphate being taken for each pair of tests.

A.

	cc.
(1) KHO with rosolic acid.....	15.0
(2) KHO with rosolic acid.....	15.15

B.

	cc.
(1) KHO rosolic acid.....	30.5
(2) KHO rosolic acid.....	30.5
(3) KHO phenolphthalein.....	30.7

All titrations, after this, were made with phenolphthalein.

C.

	cc.
(1) KHO.....	31.7
(2) KHO.....	31.8

A KHO solution was now made of such strength that one cc. = one mgm. P_2O_5 .

D.

	mgm.
(1) P_2O_5	50.35
(2) P_2O_5	50.55
(3) P_2O_5	50.40

E.

	mgm.
(1) P_2O_5	51.6
(2) P_2O_5	51.55

F.

	mgm.
(1) P_2O_5	51.05
(2) P_2O_5	50.93

D, E, and F were solutions used in standardizing.

Two different samples of Florida phosphate rock were examined.

I.

	Per cent.
(1) P_2O_5	= 29.68
(2) P_2O_5	= 29.84

II.

(1) P_2O_5	= 31.28
(2) P_2O_5	= 31.34

The following were solutions of phosphoric acid:

I.

	Per cent.
(1) P_2O_5	= 46.78
(2) P_2O_5	= 46.69

II.

(1) P_2O_5	= 44.41
(2) P_2O_5	= 44.63

III.

(1) P_2O_5	= 48.95
(2) P_2O_5	= 48.80

The effect of an excess of ammonium molybdate was tried. The soluble part of an acid phosphate was made up to a definite volume and fifty cc. taken for analysis. It was found to contain 8.28 per cent. P_2O_5 . The test was repeated, using fifteen

cc. of the molybdate more than was used in the first trial. Result = 8.36 per cent. P_2O_5 equal to a difference of 0.08 per cent.

The following tests show that it is not necessary to remove silicic acid by evaporating to dryness before precipitating with the ammonium molybdate.

A sample of Florida phoshate rock contained :

	Per cent.
(1) P_2O_5 silica removed	31.21
(2) P_2O_5 silica not removed	31.21

Another sample of the same rock was also tried :

Silica removed.		Per cent.
(1) P_2O_5	=	30.5
(2) P_2O_5	=	30.7
Average P_2O_5	=	30.6
Silica not removed.		Per cent.
(1) P_2O_5		30.6
(2) P_2O_5		30.7
Average P_2O_5		30.65

When silica is not removed the filtrate from the yellow precipitate has a yellow tinge.

The relation between the P_2O_5 in the precipitate and the potassium hydrate was established by determining the strength of a solution of disodium hydrogen phosphate by precipitation as the ammonium magnesium salt, and also by testing it by this titration process. The phosphate of soda solution was weighed (not measured), and the magnesia precipitate, after filtering, was dissolved and reprecipitated with ammonia. (Gooch, *Am. Chem. J.*, 1, 405.) The results are given in Table I, the last column giving the amounts of P_2O_5 obtained on a basis of ten grams of the solution :

TABLE I.

	Weight of Na_2HPO_4 solution. Grams.	Weight of $Mg_2P_2O_7$ obtained. Grams.	Equal to P_2O_5 . Grams.	Grams P_2O_5 in 10 grams of solution.
I.....	75.824	1.2471	0.7956	0.10494
II.....	101.167	1.6637	1.0614	0.10492
III.....	101.622	1.6733	1.0676	0.10505
Average.....				0.10497

Therefore, ten grams of the solution contain 0.10497 gram P_2O_5 .

Weighed portions of the same solution were now precipitated with molybdate and the precipitate titrated with alkali. The results are given in Table II, the last column of which gives the number of cc. of alkali equivalent to ten grams of the solution. The indicator was phenolphthalein :

TABLE II.

	Weight of Na_2HPO_4 solution. Grams.	Number of cc. of KHO sol. required.	= Number of cc. KHO sol. required for 10 grams solution.
I.....	7.4975	79.05	= 105.4
II.....	7.8255	82.40	= 105.3
Average.....			= 105.35

It follows, therefore, that 105.35 cc. alkali = 0.10497 gram P_2O_5 ; therefore,

$$(1) 100 \text{ cc. alkali} = 99.64 \text{ milligrams } P_2O_5.$$

The HCl solution was now titrated against the alkali, using phenolphthalein as the indicator. 99.00 cc. alkali were found to equal 99.05 cc. acid.

The HCl was then standardized against pure sodium carbonate.

(With methyl orange, cold :)

$$(a) 1.1291 \text{ grams } Na_2CO_3 = 65.45 \text{ cc. HCl}$$

(With phenolphthalein, boiling :)

$$(b) 1.1934 \text{ grams } Na_2CO_3 = 69.2 \text{ cc. HCl}$$

Therefore, 100 cc. =

$$(a) 1725. \text{ milligrams } Na_2CO_3$$

$$(b) 1725. \text{ milligrams } Na_2CO_3$$

Since 99.00 alkali = 99.05 acid, it follows that :

$$(2) 100 \text{ cc. alkali} = 1726 \text{ milligrams } Na_2CO_3$$

It has already been shown by (1) that 100 cc. alkali = 99.64 milligrams P_2O_5 . Therefore, combining (1) and (2) we obtain 99.64 milligrams P_2O_5 = 1726. milligrams Na_2CO_3 .

Dividing each by its molecular weight, we have :

$$\text{for } P_2O_5 \dots \dots \dots \frac{99.64}{142.06} = 0.7014$$

$$\text{for } Na_2CO_3 \dots \dots \dots \frac{1726}{106.1} = 16.27$$

$$\text{Therefore } P_2O_5 : Na_2CO_3 = 0.7014 : 16.27 = 1 : 23.2.$$

In other words, 23.2 molecules of Na_2CO_3 are required to neutralize the yellow precipitate containing one molecule P_2O_5 .

The above figures are based upon the following atomic weights:

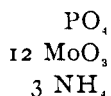
Mg	= 24.3
O	= 16
P	= 31.03
Na	= 23.05
C	= 12

$$\text{Mg}_2\text{P}_2\text{O}_7 = 63.80 \text{ per cent. } \text{P}_2\text{O}_5.$$

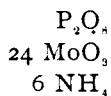
There is some uncertainty as to the correct atomic weight of magnesium. If $\text{Mg} = 24$ (instead of 24.3 as taken above) with $\text{Mg}_2\text{P}_2\text{O}_7 = 63.98$ per cent. P_2O_5 , the ratio of Na_2CO_3 to $\text{P}_2\text{O}_5 = 23.1$ to 1 (instead of 23.2 to 1). It is difficult to obtain absolutely pure Na_2CO_3 ; any impurity in it will make the ratio of Na_2CO_3 to P_2O_5 too high.

Practically, therefore, twenty-three molecules of Na_2CO_3 are required for one molecule P_2O_5 . This agrees with Hundeshagen's results.

I have never seen any explanation as to why twenty-three molecules of alkali are required to neutralize one molecule of the ammonium phospho-molybdate. A discussion of the subject, therefore, may be of interest. Hundeshagen has shown (*loc. cit.*) that (neglecting any water of crystallization) the yellow precipitate, after thorough washing with water, has the following composition:

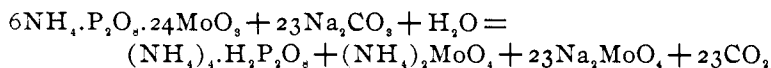


Or, doubling the formula, for the sake of clearness:



R. T. Thomson has shown in his researches on indicators (*Chem. News*, 47, 127) that of the three hydrogen atoms in H_3PO_4 , two must be saturated with alkali before the reaction with phenolphthalein will be neutral, the next drop of alkali after this causing the red color to appear. Writing the formula

differently, $H_4P_2O_8$ must become $R_4H_2P_2O_8$ (R being the radical NH_4 , or any alkali metal). Therefore, when the yellow precipitate is broken up by alkali, only four of the six molecules of NH_4 are required to form (with the P_2O_8 of the precipitate) a phosphate of ammonium that is neutral to the indicator. The remaining two molecules of NH_4 unite with one molecule of MoO_3 , yielding also a salt that is of neutral reaction. This leaves twenty-three molecules of MoO_3 , representing the "net available acidity" (if I may use the expression) of the ammonium phosphomolybdate. These twenty-three molecules of MoO_3 , of course, require twenty-three molecules of Na_2CO_3 (or its equivalent of KHO) to form Na_2MoO_4 . Q. E. D. The following is the formula representing the reaction:



It may be well to give a short résumé of this method. One gram of phosphate rock, or two or three grams of fertilizer are dissolved in nitric acid, and without evaporating to dryness diluted to 250 cc. The solution need not be filtered. Twenty-five cc. of the solution are delivered into a four-ounce beaker and neutralized with ammonia—until a precipitate just begins to form—and then treated with five cc. of HNO_3 of 1.4 specific gravity. Ten cc. of a saturated solution of ammonium nitrate are added and the solution diluted to a volume of fifty to seventy-five cc. It is then brought to a full boil, removed from the lamp and five cc. of the *aqueous* solution of ammonium molybdate added. This is followed by a second and a third five cc., if necessary, the precipitate allowed to settle, and filtered at once through a seven centimeter diameter filter. It is washed thoroughly with water by decantation and on the filter. The filter and precipitate are transferred bodily to the beaker. Standard alkali is then run in and at least 0.5 cc. of phenolphthalein (one per cent. solution) added, and then standard acid, until the color vanishes. Each cc. of alkali equals one milligram of phosphorus pentoxide.