allowed to cool off with the fire-doors open, so that after a lapse of thirty-two minutes the pressure had dropped to 70 lbs. per square inch without the slightest leak developing in any part of the boiler.

It is also worthy of our attention to note the capacity shown several years ago in a test made of the boilers of the English torpedo-boat *Speedy* of 810 tons. This vessel was built by the firm of Thorneycroft & Co., and contained eight water-tube boilers designed for a working pressure of 250 lbs. per square inch. According to the contract, there was to be a test of eight hours' duration, showing an average capacity of 2,500 I.H.P., with a maximum air pressure of 3 in. of water, and a test of three hours' duration giving a mean capacity of 4,500 I.H.P., with a maximum air pressure of 5 in. of water.

In the eight hours' test an average production of 3,046 I.H.P. was obtained, and in the three hours' trial an average of 4,700 I.H.P. was obtained, and that, too, without any special forcing.

The navies of France, England, Russia, Spain, Denmark, and the United States are making applications of water-tube boilers in considerable numbers to vessels of all sizes. On large ships the Bellville or Lagrafel d'Allest boilers are used of about equal sizes, while upon the smaller craft the Oriolle, Du Temple, Normand, Thorneycroft, Yarrow, and other boilers of a similar type are applied.

The necessity of economizing space, and at the same time securing a large heating surface with the least possible amount of material, together with a greatest available capacity, as well as insensibility to variations in the intensity of working, show the principal advantages of water-tube boilers, and the reason why they are receiving a continually increasing application to the new vessels that are being built by most of the naval powers. Water-tube boilers also offer a welcome means, if it is desired, of clearing a ship when a change of boilers is required, of insuring the maintenance of its speed, if not actually increasing it, and of gaining space for a given service, thus increasing the cargo space or enlarging the radius of action.

CONTRIBUTIONS TO PRACTICAL RAILROAD INFORMATION.

Chemistry Applied to Railroads.

SECOND SERIES.—CHEMICAL METHODS.

XIII.—METHOD OF DETERMINING AMMONIA IN AMMONIUM CHLORIDE.

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OPERATION.

Have ready the apparatus shown in accompany cut or its equivalent. Put 25 c.c. of standard sulphuric acid into the smaller Erlenmeyer flask, and connect this with the apparatus as shown. Then weigh into the larger flask half a gram of the ammonium chloride, and connect this flask in its place as shown. Then add through the funnel tube 75 c.c. of caustic potash solution, and close the stop-cock of the funnel tube as soon as all the caustic potash solution has run in. Light the lamp, and bring the liquid to a boil, and continue the boiling gently until about 50 c.c. of liquid has been added to the smaller Erlenmeyer flask. Now detach this flask from the rest of the apparatus at the top end of the pipette. Wash out the pipette on the inside and the lower part of the outside with distilled water, to remove any liquid that may be adhering to it; also wash down the sides of the flask and allow to cool. Then titrate the excess of free sulphuric acid in this flask with standard caustic potash solution, using methyl-orange as indicator.

APPARATUS AND REAGENTS.

The special apparatus required by this method consists, as is seen, of a 16-oz. Erlenmeyer flask fitted with a rubber stop-per, which carries a funnel tube provided with a glass stop-cock, and an exit tube which is enlarged above the cork and has in the enlarged part an inch or two of glass balls or beads. The smaller or 8-oz. Erlenmeyer flask is fitted also with a rubber stopper which carries an exit tube and a 100 c.c. pipette, so arranged that the end of the pipette reaches almost to the bottom of the flask and always below the surface of the acid. The top of the pipette and the top of the exit tube from the 16-oz. flask are connected by means of a rubber stopper, a simple bent glass tube and a bit of rubber hose as shown. The 16-oz. flask is supported by a universal clamp on a retort stand in such a way that the glass tube connecting the two parts of the apparatus inclines a little toward the larger flask, to allow condensed water to run back. A second clamp on the same retort stand sustains the sand bath, which is adjusted to the bottom of the 16-oz. flask. A Bunsen burner protected by a sheet-iron or tin shield to keep off drafts of air furnishes the necessary heat.

The phenolphthalin solution used in standardizing the acid and alkali is made by dissolving 5 grams of the commercial material in 100 c.c. of 95 per cent. alcohol, and adding caustic potash until the solution shows a slight pinkish tint.

The methyl-orange solution is made by adding 1 gram of the commercial material to 400 c.c. of water and filtering.

The potash solution used in the 16-oz. flask is made as follows: Take a beaker holding about 40 fluid ounces and put a mark on the outside at 30 oz. capacity. Put into the beaker 200 grams of commercial caustic potash in sticks and add 36 oz. of distilled water. After solution is complete, boil down to the mark in order to remove any ammonia that may be present. Pour the liquid into a bottle for use.

The standard alkali and acid solutions are made as follows: Take about 50 grams of the best dry C. P. carbonate of soda, free from silicate, to be obtained in the market. Dissolve in distilled water and filter into a platinum dish. This is to remove any sand or dirt that may be accidentally contained in the soda. Add a little carbon dioxide, or a few drops of carbonic acid water, in order to be sure that there is a slight excess of carbonic acid present. Evaporate the solution to dryness at a temperature a little above the boiling point of water, using great care to keep out dust or dirt. When thoroughly dry transfer to a dry glass-stoppered bottle for further use. Now carefully weigh a clean ½-oz. platinum crucible, and add to it not quite a gram of the dried carbonate of soda, ignite over a Bunsen burner until the soda is just melted and weigh. This weight gives the amount of carbonate of soda used, and is the basis of the standardizing. Have previously prepared two solutions made as follows: 1. A solution of distilled water to which has been added about 26.5 grams of concentrated C. P. sulphuric acid per litre. The solution should be thoroughly mixed and allowed to cool before using. 2. A solution of caustic potash in distilled water, made by adding to it about 50 grams of commercial stick potash per litre, allowing to dissolve, and then adding to it ½ litre of milk of lime, made by slacking 70 grams of commercial caustic lime, and diluting with water to 1 litre. After the lime is added, boil for 10 or 15 minutes, then allow to settle and draw off with a pipette about 50 c.c. of the clear solution, transfer to a beaker, and add a few drops of phenolphthalin. Then run in from a burette some of the sulphuric acid solution above described, until the last drop just discharges the color and boil. If 5 or 10 minutes boiling does not bring back any of the pink color, the caustic potash solution may be regarded as free from carbonates and is ready to be proceeded with. If boiling does restore any of the pink color, the boiling with the lime must be continued, or fresh milk of lime added and boiling continued until the solution is free from carbonates by above test. After carbonates are proven absent, filter the solution into the vessel in which it is to be kept for use, taking care to avoid exposure to the air as much as possible. The two solutions thus prepared should be rendered homogeneous by stirring or shaking, and should then be allowed to stand until they are both of the temperature of about 80° F.; this being accomplished the strength of each in terms of the other must be known. For this purpose run in from a burette 40 c.c. of the acid solution into a beaker, add a few drops of phenolphthalin, and then titrate with the caustic potash solution. Two or three tests should give same figure within one or two drops. Preserve the figures thus obtained. Now put the crucible containing the fused carbonate of soda before described into a beaker, add about 50 c.c. of distilled water, and allow to dissolve. Then add about 40 c.c. of the sulphuric acid solution above described and boil 15 minutes to remove carbon dioxide, taking care that there is no loss due to effervescence. After the boiling is finished, titrate the excess of acid with the caustic potash solution, using phenolphthalin for the indicator. The relation of the acid and alkali being known as before de-

scribed, it is easy to find the amount of the sulphuric acid solution, corresponding to the carbonate of soda taken. But one point still remains uncertain—viz., whether the boiling has removed all the carbon dioxide. To decide this point add to the solution which has just been titrated with the potash solution, and which the last drop of potash rendered pink, one drop of the acid solution, or enough to just completely discharge the color and boil again. If the color does not reappear on boiling, the figures already obtained may be regarded as satisfactory. If the color does reappear, run in 1 or 2 c.c. of the acid and boil again. The amount of acid thus run in must be added to the 40 c.c. used at first. After boiling, say 5 minutes more, titrate with the potash solution, noting how much of it is required to bring back the pink color, and adding this amount to the amount of potash solution previously used. Now test as before for the absence of carbon dioxide, and if it is proven not present, find the total number of c.c. of the sulphuric acid solution which are equivalent to the carbonate of soda used. From this, as described below, the amount of sulphuric acid [H_2SO_4] in 1 c.c. of the acid solution may be obtained. But convenience in the subsequent use of the acid solution makes it desirable that each c.c. of it should contain a definite proportion of the molecular weight of sulphuric acid, say one-fourth or 0.0245 grams, H_2SO_4. If sufficiently concentrated C. P. sulphuric acid has been used in making the solution to start with, the figure obtained as above will be larger than this, and as shown in the calculation below, a certain amount of water must be added, which should be done, the solution being agitated by stirring or shaking, and then allowed to stand until the following day, when a new determination of its strength should be made by means of carbonate of soda as above described. The figure thus obtained will show whether further addition of water is necessary. When all the water needed has been added, not less than two determinations of the strength of the acid should be made by means of carbonate of soda, as described above, which duplicates should show the value of 1 c.c. to be not less than 0.0244 gram nor more than 0.0246 gram of sulphuric acid [H_2SO_4].

The standard acid having been obtained, it remains to make the caustic potash solution so that 1 c.c. equals 1 c.c. of the acid solution. For this purpose run, say, 40 c.c. of the standard acid into a beaker and titrate with the caustic potash, using phenolphthalin as indicator. If fairly good caustic potash has been used in making the solution, this operation will show that water must be added. If the operation shows that the solution is too weak, it is better to throw it away and start again, using more of the potash per litre. The figure obtained enables, as is shown below, the amount of water that must be added, to be calculated. This amount of water should be added, the solution agitated by stirring or shaking, and allowed to stand until the following day, when a new test should be made. The figure thus obtained will show whether further addition of water is necessary. After all the water has been added, not less than two tests should be made, and each of these should show that the two solutions are alike to within one-tenth of a c.c. in 40.

CALCULATIONS.

An example of all the calculations is given herewith. Atomic weights used : nitrogen, 14 ; hydrogen, 1 ; potash, 39.1 ; sulphur, 32. Molecular formula : ammonium chloride, NH_4Cl ; ammonia, NH_3 ; caustic potash, KOH ; sulphuric acid, H_2SO_4 ; water, H_2O.

I. Standardizing the sulphuric acid. Suppose that 40 c.c. of the sulphuric acid as mixed requires 36.4 c.c. of the caustic potash as mixed to exactly neutralize it, this figure having been obtained by two or three closely agreeing tests. This means that 1 c.c. of the sulphuric acid solution is equal to [36.4 ÷ 40] 0.91 c.c. of the potash solution, and that 1 c.c. of the potash solution is equal to [40 ÷ 36.4] 1.0989 c.c. of the acid solution. Next, suppose the fused carbonate of soda in the crucible weighs 0.9864 gram, and that 45 c.c. of the

sulphuric acid as mixed are run into the solution of this carbonate of soda ; also that after boiling it requires 9.2 c.c. of the potash solution to neutralize the excess of acid ; also that it is found that the carbon dioxide is not quite all removed by the first boiling, and that 1 c.c. more of the acid is put in for the second boiling, and that after this second boiling it requires 0.4 c.c. of the potash solution to neutralize the excess of acid, and that test shows that the second boiling removed all the carbon dioxide. It is evident that 46 [45 + 1] c.c. of the acid have been used altogether, and that 9.6 [9.2 + .4] c.c. of the potash solution have been used to neutralize the excess of acid. But 1 c.c. of the potash solution is equal to 1.0989 c.c.
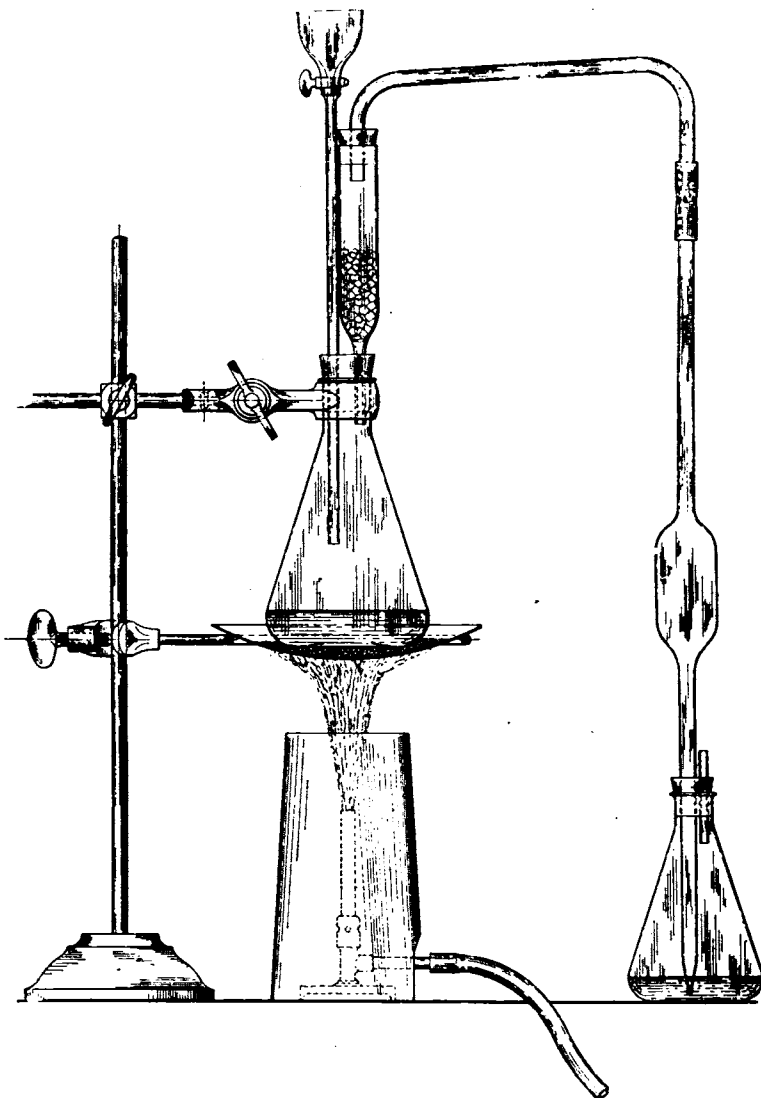
APPARATUS FOR DETERMINING AMMONIA IN AMMONIUM CHLORIDE.

of the acid, or 9.6 c.c. of the potash solution are equal to [1.0989 × 9.6] 10.55 c.c. of the acid solution. Hence the amount of the acid solution used up by the 0.9864 gram of carbonate of soda is 35.45 [46 − 10.55] c.c. or 1 c.c. of the acid solution is equivalent to [0.9864 ÷ 35.45] 0.027825 gram carbonate soda. But the ratio of the molecular weights of carbonate of soda [Na_2CO_3] to sulphuric acid [H_2SO_4] is as 106 to 98. Hence each c.c. of the sulphuric acid solution contains [106 : 98 :: 0.027825 : x] 0.025725 gram sulphuric acid. But, as previously stated, it is more convenient to have the acid and alkali solutions some even ratio of the molecular weight, and therefore a solution is wanted which contains [98 ÷ 4] 0.0245 gram of sulphuric acid per cubic centimetre. To obtain this water must be added to the solution in question. The amount of this is found by the following ratio, $a : b :: x : c$, in which a represents the strength of the acid as determined, in this case 0.025725 gram, b the strength of the acid desired, in this case 0.0245 gram, c the total volume of the solution we are working with, say 15000 c.c., and x the volume of the solu-

tion after the water is added, which in the case supposed is [0.025725 × 15000 ÷ 0.0245] 15750 ; or [15750 — 15000] 750 c.c. of water must be added.

II. Standardizing the caustic potash solution. Suppose that it is found that 40 c.c. of the standard acid requires 31.2 c.c. of caustic potash solution as made to exactly neutralize it. This means that water must be added and the amount may be found by the proportion $a : b :: x : c$, in which a represents the number of c.c. of standard acid used, in this case 40, b the number of c.c. of potash solution used, in this case 31.2 c.c., c the total volume of the solution we are working with, say 15000 c.c., and x the volume of the solution after the water is added, which in the case supposed is [40 × 15000 ÷ 31.2] 19230, or [19230 — 15000] 4230 c.c. of water must be added. The reaction between sulphuric acid and caustic potash being represented by the equation $H_2SO_4 + 2 [KOH] = K_2SO_4 + 2[H_2O]$ or by weight $98 + 112.2 = 174.2 + 36$, it must be remembered that, since 1 c.c. of each solution is the equivalent of the other, the actual amount of caustic potash [KOH] in each c.c. of the solution is [112.2 ÷ 4] 0.02805 gram ; that is, if a solution containing any substance which reacts with sulphuric acid is so made that 1 c.c. equals 1 c.c. of the acid, the value of 1 c.c. of the solution in question may be found by writing the equation which expresses the reaction, together with the molecular weights, and dividing the molecular weight as given in the equation of the substance sought by the same figure that is required to give the known strength of the standard sulphuric acid. Further, the quotients thus obtained may be used interchangeably according to the work in hand. Thus 1 c.c. of the standard sulphuric acid, or 1 c.c. of the standard caustic potash, is equivalent to 0.02355 gram of potash [K_2O], or to 0.020 gram of caustic soda [NaOH], or to 0.0155 gram of soda [Na_2O], or to 0.0265 gram of carbonate of soda, or to 0.0085 gram of ammonia [NH_3].

III. Ammonia in ammonium chloride. Suppose, as is noted below, that methyl-orange has been used in standardizing the acid and alkali, and that 1 c.c. of acid equals 1 c.c. of alkali. Also that 6.6 c.c. of the caustic potash solution are required to neutralize the excess of sulphuric acid in the 8 oz. flask. It is evident that [25 — 6.6] 18.4 c.c. of the standard sulphuric acid have been used up by the ammonia from the ammonium chloride. But each c.c. of the standard acid is equivalent to 0.0085 gram of ammonia, consequently the ammonia in the half gram is [0.0085 × 18.4] 0.1564 gram, or [0.1564 × 100 ÷ 0.500] 31.28 per cent. If, on the other hand, phenolphthalin has been used as indicator in standardizing the acid and alkali, and it has been found, as described below, that 50 c.c. of the standard acid require 49.5 c.c. of standard potash when methyl-orange is used as indicator, it is evident that 1 c.c. of the potash equals [50 ÷ 49.5] 1.0101 c.c. of the acid. But under these conditions with methyl-orange as indicator, it would require 6.53 c.c. of potash to neutralize the acid left in the flask in the case supposed above, or [6.53 × 1.0101] 6.6 c.c. of the acid are neutralized by the potash, showing as before [25 — 6.6] 18.4 c.c. of the acid neutralized by the ammonia ; the remainder of the calculation is as above.

NOTES AND PRECAUTIONS.

It will be observed that this method separates the ammonia from the ammonium chloride by decomposing it with caustic potash, removing it from the solution by boiling and catching the ammonia gas along with some of the condensed water in standard sulphuric acid solution, no special condenser or aspirator being required.

If the boiling is conducted too rapidly, some of the water on the glass beads or balls is apt to be carried along mechanically, and may find its way into the acid flask. This water is principally condensed steam, but may contain some of the potash solution mechanically carried along from the boiling solution below. The error introduced by any of the potash solution getting into the acid solution is obvious.

On the other hand, if the boiling is too slow, especially toward the last of the operation, when the whole apparatus is filled with steam and the acid has become somewhat warm, or if a cold draft of air strikes the apparatus, there is a tendency for the acid to be sucked back toward the alkali flask. The 100 c.c. pipette allows all the acid to be sucked up into the bulb without any of it getting back into the alkali flask, but there is danger of error if air is allowed to bubble back through the acid solution in the bulb, since some of this solution may be carried mechanically by the air bubbles back into the alkali flask. By having the apparatus in a quiet place, and managing the heat properly, the absorption of the ammonia takes place quietly and there is no regurgitation.

After a test has been finished it is essential to wash out the

whole apparatus and to remove the glass stopcock in the funnel tube, otherwise this stopcock becomes fast.

Much of the phenolphthalin of the market apparently contains something which combines with alkali, without showing change of color. If this is not satisfied with alkali as directed, the reaction will not be quite so delicate.

Standard sulphuric acid solution is made by adding to a clean clear glass 5-gall. bottle 15 litres of distilled water, and then weighing out and adding to it 397.5 grams of concentrated C. P. sulphuric acid. It is better to set the water in the bottle in motion by stirring with a clean glass rod before adding the sulphuric acid. After the acid is in it is essential to agitate thoroughly by stirring and shaking, but not advisable to draw air through for this purpose, as this causes the liquid to take up carbon dioxide, which interferes with its subsequent usefulness with phenolphthalin. It is not desirable to standardize on the same day, both on account of temperature, and also because it is very difficult by any practicable method of agitation to get so large a bulk of liquid entirely homogeneous without standing. If the first standardizing shows that it is essential to add, say, 750 c.c. of water, it is better to add only 700, since the liquid should be standardized once more any way, and too much water must of course be avoided. The second addition of water is usually less than 100 c.c. Both agitation and standing over night are essential after each addition of water.

Caustic potash solution is made in the same kind of bottle and in the same amount as the acid solution. The same precautions should be taken in regard to stirring, and allowing to stand over night, as in the case of the acid. It is well known that caustic potash solution, if properly made as above described, contains a small amount of caustic lime in solution. Of course this lime will appear in the comparison with the standard acid. If now the water used in the first addition contains a little carbon dioxide, a little of the lime will be precipitated on standing over night and weaken the solution a little. It is therefore not advisable to add quite as much of the water shown by calculation the first time, as in case of the acid.

The two solutions, as will be observed, are made in quite large amounts, and considerable pains are taken to have them right, since other work depends upon them. Both of the solutions are kept on a shelf somewhat higher than the burettes, and both are drawn into the burettes by means of glass tube syphons with glass cocks at the lower ends. In accurate work it is of course essential to draw out and throw away the liquid which has been standing exposed between the cock and the lower end of the syphon tube before filling the burette. The air which goes in to replace the liquid in the large glass bottles should bubble through caustic potash solution in order to keep out carbon dioxide. Potash bulbs are used for this purpose.

The fact that phenolphthalin is sensitive to carbon dioxide in water solution and to carbonates and bicarbonates may lead to serious error unless sufficient care is taken to add enough acid to decompose all carbonates and bicarbonates and then expel the gas by boiling before subsequent titration with caustic potash. An illustration will make the matter clear. Let us suppose that in obtaining the relation between carbonate of soda and sulphuric acid in standardizing the acid, the carbon dioxide is not quite all removed by boiling, when we attempt to measure the excess of the acid by means of the caustic potash solution. We add this solution drop by drop and ultimately reach a point when all the free sulphuric acid is satisfied with the caustic potash, but since phenolphthalin in presence of carbonic acid or carbon dioxide in water solution does not change color until part at least of this carbonic acid is also satisfied with caustic potash, we do not get our end reaction when the sulphuric acid is all satisfied, as should be the case, but rather after some further addition of caustic potash. The error is obvious, and there is always uncertainty if carbon dioxide or carbonates are present when using phenolphthalin as indicator. Even carbon dioxide in the standard sulphuric acid solution, or carbonates in the caustic potash solution, will cause difficulty. Possibly other indicators do not give so much trouble from this cause, but all that we have ever tried are so much less sensitive and sharp at the end reaction than phenolphthalin, provided the conditions are right, that we prefer to take the extra trouble. Positive experiments show that if the solution is rendered clearly acid with standard acid, and boiled for 10 minutes or even less, the carbon dioxide will all be expelled, so that if the directions are closely followed, the results will be fairly accurate. It is obvious that if the distilled water used in making the standard acid contains carbon dioxide, there will always be some present, with a consequent liability to uncertainty in the final results. Presence or absence of carbon dioxide in the standard acid can be proved by titrating some of the acid cold with