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QUANTITATIVE AMMONIA DISTILLATION BY AERATION, FOR KJELDAHL, UREA AND OTHER NITROGEN ESTIMATIONS. III.[1]

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Introduction.

The object of this paper is to describe the latest developments in the aeration method of distilling ammonia, to discuss a few criticisms, to clear up some doubtful points, and finally to determin as far as possible the relative merits of air-current and steam distillations.

General Discussion.

In 1908, Kober, published two articles.[2] The first showed that under certain conditions, ammonia could be distilled quantitatively, in making Kjeldahl, urea, and other nitrogen determinations, by use of an air current. The second called attention to the fact that the long duration of the distillation of ammonia using Folin's magnesium chloride urea method was due to large amounts of magnesium chloride. Although no serious adverse criticism[3] has been made, the articles have been misquoted and misinterpreted, and the aeration method has not been generally adopted in spite of the ease and accuracy of operation and the cheapness of installation, which are essential advantages.

The method, termed by some an extension of the Folin scheme for free ammonia determination, required considerable experimentation, before the correct conditions for quantitative work were found. Folin, who at first rejected its application has very recently used it successfully on a small scale for micro-chemical determinations of total nitrogen and urea in blood and other physiological material. The method was intended for all nitrogen determinations which depend on ammonia distillation and not for Kjeldahl work only, as several text books[4] and papers[5] have implied. Since the directions and figures were given for urea and free ammonia estimations, the recommendations of Gill,[6] Allison and Grindley, and of Hen-

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[1] Kober, This Journal, 30, 1131 (1908); 32, 689 (1910).

[2] Kober, *Ibid.*, 30, 1131, and 30, 1279 (1908).

[3] J. Sebelien, *Chem. Ztg.*, 33, 785 (1909); Davis, This Journal, 31, 56 (1909); Gill and Grindley, *Ibid.*, 31, 1249; Weston and Ellis, *Chem. News*, 100, 50 (1909); *Chem. Centr.*, 11, 590 (1909); Pennington, This Journal, 32, 561 (1909); Denis, *J. Biol. Chem.*, 8, 427; V. Henriques and S. A. Gammeltoft, *Bohr-Gedachnisschrift, Skand. Archiv. Phys.*, 25, 166 (1911); Gill, Allison and Grindley, This Journal, 31, 1078 (1909); Folin, *J. Biol. Chem.*, 11, 493, 507 (1912).

[4] Abderhalden's "Handbuch der Biochemie," Vol. 5, first part, page 305; C. Neuberg, "Der Harn," first part, 545; Neubauer-Hupperts, "Analyse des Harns," 11th edition, first part, page 568.

[5] Gill, Grindley, Pennington, Denis, Henriques and Gammeltoft, *Loc. cit.*

[6] *Loc. cit.*

riques[1] and Gammeltoft are, therefore, to be taken as corroborative of Kober's first paper.

Three reasons as to why the method has not been generally accepted may well be suggested: (1) The method, where used,[2] has not been mentioned, perhaps, on the assumption that the results were the same as with the old procedure. (2) Certain conditions demanded may not have been carefully maintained. (3) The method has lacked specific directions as to the amount of aeration, the amount and concentration of alkali, precautions regarding the spattering of alkali into the standard acid, and the production of excessive heat.

Theory of Ammonia Distillation.—A short review of the theory of ammonia distillation will help make clear the basis of the method and the possible sources of error. Ammonia is a gas highly soluble in water (600–700 liters of gas to 1 liter of water at ordinary room temperature) and its absorption depends mainly on this high solubility, the acid in the standard solution serving only to combine with the ammonia, thereby removing its vapor pressure and preventing further distillation. According to many investigators, the following equation expresses the equilibrium between ammonia and water:

$$NH_3 + H_2O \rightleftarrows NH_4OH \rightleftarrows NH_4^{\cdot} + OH^-.$$

The equation means that in aqueous solutions of ammonia there are present some of each of the components NH_4OH, NH_3, $NH_4^{\cdot}$ and OH^-. According to the mass law, if we increase a component an equilibrium will shift to use up that component. In this case an increase of the OH^- ions will increase the amount of ammonia gas, making it apparent why an excess of alkali is necessary in steam distillation, and why a larger excess is needed in the aeration process. The concentration of the alkaline hydroxide is one of the factors which controls the speed of ammonia distillation but the rate is mainly dependent on the amount of surface given to the liquid, the object of boiling being to increase the surface by bubbles of steam. A rise of temperature increases the vapor pressure of the ammonia and also facilitates the distillation. To sum up, the rate and completeness of distillation depend on the concentration of the OH^- ions, on the surface given the liquid through boiling or aeration and finally on the temperature.

If the solution contains salts which form insoluble hydroxides or oxides, these will, in proportion to their amount, reduce the concentration of the

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[1] *Loc. cit.*

[2] Levene and Kober, *Amer. J. Physiol.*, **23**, 328 (1909); Saccharine Report, U. S. Dept. of Agriculture, Referee Board of Consulting Scientific Experts, from the Herter Lab. The statements in these publications that the total nitrogen estimations were made according to the Kjeldahl-Gunning process are not quite correct: they were practically all distilled according to the aeration method.

alkali and thereby delay the distillation. If the salts are not completely precipitated and remain in considerable excess, they may further hinder the distillation by a shifting of the equilibrium, which was referred to in a former paper.[1]

$$MgCl_2 + NH_4OH \rightleftarrows Mg(OH)_2 + NH_4Cl.$$

Such conditions are found in Folin's magnesium chloride urea estimation in which ammonia is distilled in the presence of 15 grams of magnesium chloride (anhydrous) and 20 cc. of 7.5% solution of sodium hydroxide. This means that about 3 grams of magnesium chloride are precipitated as hydroxide and about 12 grams of the salt remain in solution.

In discussing this equation, $MgCl_2 + NH_4OH \rightleftarrows Mg(OH)_2 + NH_4Cl$, Folin[2] presents as an analogous equation, $Na_2SO_4 + 2NH_4OH \rightleftarrows 2NaOH$ $(NH_4)_2SO_4$. He argues that, since ammonia can be easily obtained quantitatively from this reaction, it should be expected from the first reaction. In reality these reactions are not analogous. The first contains both a liquid and a solid phase. The second is a one-phase (liquid) system. In other words, one is a heterogeneous, the other a homogeneous equilibrium.

Kober stated that magnesium chloride is the cause of slowness in distliling ammonia—a fact which Folin admits. Kober also said that the last traces of ammonia would be difficult to remove. To this statement Folin objects on the ground that no experimental evidence was produced to show how much ammonia was not removed in the time allowed by the Folin method. Folin having carefully worked out the time needed for quantitative results, experiments on this point seemed unnecessary, for the error, according to the theory, can be reduced by sufficient boiling to any desired amount, *other substances remaining unchanged.*

Folin has made a series of experiments using known amounts of ammonium sulfate with 500 cc. of water and 15 grams of magnesium chloride. The average of the first series of 24 distillations with 25.5 cc. of 0.1 *N* ammonium sulfate solution showed a deficiency of 0.075 cc. of 0.1 *N* ammonia, while the second series with 51 cc. of the same solution lacked 0.93 cc. when distilling for the same length of time, *i. e.*, 1 hour. His third series, similar to the second, distilling 20 minutes longer, showed a deficiency of 0.32 cc. These results fully corroborate the statement made by Kober that "the last traces of ammonia would be hard to remove."

Unfortunately S. R. Benedict, Neuberg and Folin inferred from Kober's article that he condemned the Folin method as unsuitable for urea determinations. On the contrary, Kober hoped by pointing out the possible sources of error, to increase the trustworthiness of the process. He had

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[1] This Journal, 30, 1281 (1909).

[2] *J. Biol. Chem.*, 11, 509 (1912).

been and was using the magnesium chloride method, distilling by aeration as a matter of convenience.

In this connection, it would be well to consider two recent statements: (1) Hawk[1] has said that it is sufficient in distilling ammonia in the Kjeldahl determination, to make the solution alkaline. When distilling in the presence of magnesium and calcium salts, this is certainly incorrect. Folin's results, given above, show that simply making the solution alkaline may lead to low results, unless the time required for boiling is greatly increased. (2) Folin[2] has suggested distilling until the distillate is no longer alkaline to litmus paper. Small but appreciable amounts of ammonia may in this way escape detection; and if the distillation is slow, because of the slight excess of alkali, considerable error may result. As a rule, an appreciable excess of acid or of alkali is required to affect test papers (see experimental part of the paper).

Improvements and Details of the Aeration Method.

(*1*) *Amount of Aeration.*—Usually the term "amount of aeration" conveys no exact idea because the average laboratoy lacks a meter or gauge for measuring the volume of air. We have, therefore, constructed the following crude but suitable apparatus by means of which we have found that of the six or more suction pumps in this laboratory giving about the same vacuum, the volume varies from 20 to 500%.

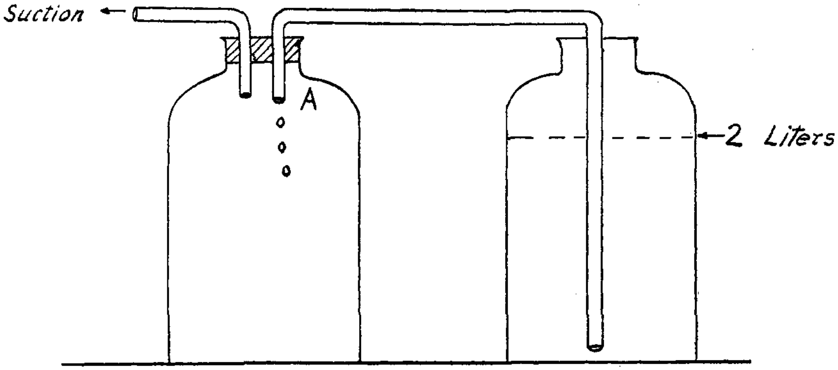

Fig. 1.—Air Gauge.

The volume of air is guaged by the time it takes to draw a certain volume of water from one bottle to another connected by means of a glass tube (11.0 mm. wide). Thus if it takes 15 seconds for 2 liters of water to pass the point A the pumps draw over 480 liters of air per hour.

Before attempting to fix the amount of aeration necessary for ammonia distillation the speed of three pumps, or the amount of air drawn over in an hour was determined by means of the gauge described above.

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[1] "Practical Physiological Chemistry," fourth edition, p. 401.

[2] *J. Biol. Chem.*, 11, 510 (1912).

Pump.	Time required for two liters of water to pass point (A).	Liters of air per hour.	Remarks.
A........	11 seconds	650	Glass pump, of German make.
B........	45 seconds	160	Ordinary aspirator.
C........	52 seconds	139	Geissler's.

In order to test the rate of distillation with each of these pumps, the following experiments were made: 0.0595 gram of ammonia as ammonium sulfate solution was distilled according to directions given in the latter part of this paper. At intervals of 10 minutes or more, the standard acid was titrated and the amount of ammonia which distilled over was thus determined.

	Pump A.		Pump B.		Pump C.	
Time.	Amount of NH_3. Grams.	Per cent. of total.	Amount of NH_3. Grams.	Per cent. of total.	Amount of NH_3. Grams.	Per cent. of total.
10 minutes............	0.0565	94.8	0.05460	91.6	0.0418	70.1
20 minutes............	0.0587	98.5	0.0563	94.4	0.0507	85.0
35 minutes............	0.0593	99.5	0.0581	97.4	0.0548	92.0
50 minutes............	0.0596	100.1	0.0507	98.5	0.0551	92.6
65 minutes............	..	...	0.0591	99.1	0.0576	96.4
90 minutes............	..	...	0.0598	100.3	0.0583	97.8
Correction for reagents.	..	99.7	..	99.9	..	97.4

The figures given in the table are not to be taken as showing the percentage accuracy of the aeration method, as the experimental error with a half dozen titrations is apt to be large. 400 to 600 liters of air seems to be sufficient for the average amount of ammonia obtained in Kjeldahl work. If a weaker pump is used, or if larger quantities of ammonia are to be distilled, more aeration must be used.

(2) *Amount of Heat.*—As these results were obtained with the aid of the heat of neutralization it was of interest to determin the rate and completeness of distillation without utilizing this heat.[1]

In the following experiments a saturated solution of sodium and potassium sulfate was used in place of concentrated sulfuric acid and its equivalent of alkali. The solution in each case was cooled to 20–25° before aerating by means of pump A. The amount of ammonia distilled every 5 minutes from 20 cc. of 0.2 N NH_3 was determined by titration.

Comparing these results with those immediately preceding, it is evident that *the heat of neutralization, although slightly accelerating the distillation at the beginning, has no effect on the final results.* This is also true when a slight excess of alkali is used (Solution III).

To make absolutely sure that the quantitative distillation is not dependent upon heat, another series of experiments was made and the completeness of distillation tested by Nesslerizing the residues in the Kjeldahl flasks.

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[1] We are indebted to the editor for suggesting experiments to determin the effects of the heat of neutralization.

	Solution I. 125 cc. saturated sulfate. 25 cc. saturated alkali. 20 cc. 0.2 N NH_3.		Solution II. Same as I except 1/2 the amount of sulfate.		Solution III. Same as I except 1/2 the amount of alkali.	
Time.	Cc. 0.1 N NH_3.	% NH_3.	Cc. 1 N NH_3.	% NH_3.	Cc. 1 N NH_3.	% NH_3.
5 minutes....	20.15	49.8	22.9	55.1	9.37	23.1
10 minutes....	9.37	72.8	9.80	79.2	7.0	40.4
15 minutes....	4.81	84.7	4.55	90.5	5.38	53.7
20 minutes....	2.59	91.2	2.10	95.7	4.12	63.9
25 minutes....	1.45	94.7	1.20	98.7	3.64	72.8
30 minutes....	0.89	97.0	0.55	100.0	2.90	80.1
35 minutes....	0.55	98.3	0.21	100.4	1.96	84.7
40 minutes....	0.26	99.0	0.10	100.7	1.75	89.2
45 minutes....	0.27	99.5	0.04	100.8	1.33	92.5
50 minutes....	0.09	99.7	0.08	101.0	0.83	94.5
55 minutes....	0.04	99.9	0.00	...	0.70	96.2
60 minutes....	0.00	...	...	...	0.50	97.6
65 minutes....	0.14	100.2	...	...	0.41	98.5
70 minutes....	...	...	...	...	0.27	99.2
75 minutes....	...	...	...	...	0.17	99.6
80 minutes....	...	...	...	...	0.09	99.7
85 minutes....	...	...	...	...	0.04	99.9
90 minutes....	...	...	...	...	0.01	99.9

Twenty cc. of H_2SO_4 (sp. gr. = 1.84) with 4 volumes of water and 0.2 gram of ammonium sulfate were cooled to room temperature. The saturated alkali was added in three parts, cooling after each addition so that the acid when neutralized was not above room temperature. After aerating for 105 minutes the amount of ammonia undistilled was estimated with the Nessler reagent. In the three experiments which we made less than 0.05 cc. 0.1 N NH_3 was found in each flask. This again shows that the heat does not influence the final result.

(3) *Amount and Concentration of Alkali.*—To show the most efficient amount and concentration of alkali for the aeration process, a series of distillations with varying amounts of saturated solution[1] was made. 52 cc. of a saturated solution of sodium hydroxide is necessary to neutralize the 20 cc. of concentrated sulfuric acid used for digestion.

Substance.	Amount taken. Grams.	Saturated NaOH used. Cc.	0.1 N NH_3 found in residue with Nessler's reagent. Cc.
$(NH_4)_2SO_4$..............	0.235	55	0.3
$(NH_4)_2SO_4$..............	0.235	65	0.05
$(NH_4)_2SO_4$..............	0.235	75	0.1
$(NH_4)_2SO_4$..............	0.235	75	0.05
$(NH_4)_2SO_4$..............	0.235	75	0.05
$(NH_4)_2SO_4$..............	0.235	85	0.5 –1.0
$(NH_4)_2SO_4$..............	0.235	95	0.5 –1.0

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[1] Caustic soda of 48° Bé., obtainable in steel drums from the Niagara Alkali Company, is used for Kjeldahl work in this laboratory. This saves the trouble of making the solution from small quantities and insures a uniform concentration.

From the amount of ammonia in the residue (distilling 90 minutes) it is evident that a small excess of alkali gives low results, while larger amounts of alkali leave an inappreciable amount of ammonia in the residue. On the other hand, a very large excess is no advantage as it increases the amount of liquid to be aerated. We, therefore, recommend the use of 75 cc. of sodium hydroxide solution with the specific gravity of about 1.46–1.48 with 20 cc. concentrated sulfuric acid.

(4) *Precautions Against Spattering of Alkali and Excessive Heat.*—The chief difficulty caused through lack of precautionary measures are, as previously stated, excess of heat and the contents of the Kjeldahl flask boiling over. Another objection is the danger of high vacuum produced by adding the saturated alkali, too slowly.

The following scheme overcomes these obstacles and the operation of starting the aeration becomes simple and without danger.

(5) *New Directions and Details of Manipulations.*—The melt in the Kjeldahl flask after digestion, is diluted with four parts by volume of ammonia free water and cooled to room temperature. After connecting the Kjeldahl flask (K) to the absorption bottle (S) and placing it in a water bath (W) the suction is slightly reduced by means of screwed pinch-cock (X) and the alkali cylinder (C) is attached. As the alkali enters, the Kjeldahl flask is shaken in a rotatory fashion until all of the alkali has been drawn over. The bath is then removed and the Kjeldahl flask set at an angle on a suberite ring. This prevents the possibility of alkali spattering into the absorption bottle (round, wide-mouth, 16-ounce bottle) which should be half full of standard acid and ammonia free water. The water aids in absorbing the ammonia but if the bottle is too full there is danger of losing some of the standard acid through spattering.

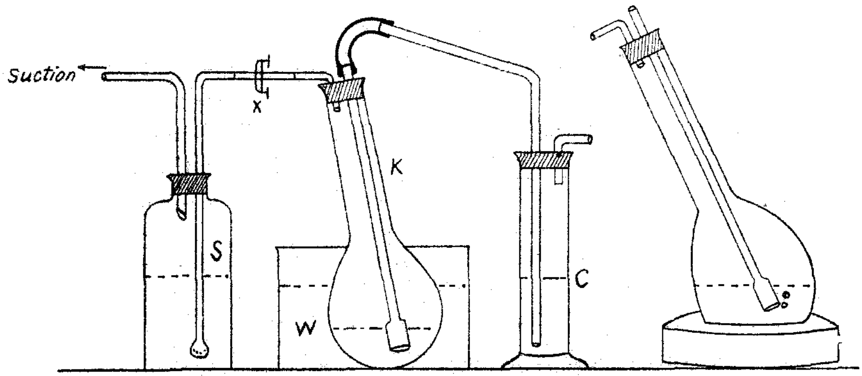

Fig. 2.—The Aeration Apparatus with Alkali Cylinder.

By means of the pinch-cock (X) aeration may be closed off completely and the absorption bottle removed with safety, for the purpose of adding more standard acid, titrating, etc. It is best, however, before doing this, to decrease the suction in order to avoid exposing the system to too high a vacuum and to prevent spattering when the flask is removed from the circuit. A number of distillations, usually from 3 to 6, may be run in series. In disconnecting a series it is, of course, necessary, to begin with the flask farthest from the pump. As in all aeration procedures, a wash bottle containing sulfuric acid should be attached to the system in order to purify the air from ammonia.

The use of a water bath during the addition of the alkali has made the method easier, safer and possibly more accurate. The water of the bath acts as a condenser on the upper part of the Kjeldahl flask which, if the flask is shaken, absolutely prevents superheating and boiling over.

The rubber stopper in the Kjeldahl flask should be short and should fit very tightly. Otherwise water condensing between the stopper and the glass will serve as a absorption medium and an appreciable amount of ammonia will remain undistilled.

If the flat bottom Kjeldahl flask is used (the round bottom ones are preferable) a safety valve should be inserted between the source of the vacuum and the first absorption bottle so that the Kjeldahl flask is not exposed to too great a vacuum.

When the melt is an unknown amount, as is the case in digesting large quantities of organic matter, such as food, filter paper, etc., a rough estimation of the dilution necessary may be made by holding an empty Kjeldahl flask next to the melt and adding water until the heights of liquids are the same. The amount of water used may be taken as a volume of the melt. Another scheme is to roughly weigh the Kjeldahl flask, empty and after digestion. The difference in weight, after allowing for the potassium sulfate, corresponds to the amount of sulfuric acid.

When first trying the method, it is well after distillation to test the Kjeldahl residue with 5 cc. of Nessler's solution. If it shows an appreciable amount of ammonia, there is trouble in manipulating the apparatus —either the tube is not far enough into the liquid, or the stopper is loose. Another suggestion is to add a second absorption bottle containing some standard acid. If the liquid, titrated with alkali or tested with Nessler's reagent, shows ammonia, the probabilities are that the first absorption bottle is too full or the aeration is too rapid.

Comparison of the Steam and the Aeration Method.

The method of distilling ammonia by boiling with strong alkali has been accepted too long and too generally to need proof of its efficiency. Yet there are disadvantages, three of which the aeration method, at least in part, overcomes. These are expense, time and attention.

The steam process requires a metal rack and block tin condensers, and the expense of maintenance, gas and breakage of flasks is no small item. About 15 minutes heating is necessary to bring the liquid to boiling, and from 40 to 90 minutes to distil off the ammonia, during which period constant attention is required to prevent bumping or evaporation to dryness.

The chief piece of apparatus for the aeration method is a suction pump. The cost of operation is practically nothing. With a fairly good pump, 25 cc. of 0.1 N NH_3 can be distilled quantitatively in about an hour, 95% of the gas going over in the first 10 minutes. Once set up the determination may be left with perfect safety. As the system is closed throughout, the estimation cannot be affected by dust or fumes of the laboratory.

The question then is "are accurate results obtained with the aeration method?" To test the accuracy of the process we attempted to determin first, to what extent ammonia is not distilled off, by testing the residue after distillation with Nessler's reagent; second, to what extent it is not absorbed by the standard acid; and finally the sources of error not included in the first two possibilities, *e. g.*, spattering of alkali and ammonia in the reagents.

(*1*) *The amount of ammonia not distilled.*

Substance.	Amount taken. Grams.	0.1 N NH_3 in residue. Cc.
$(NH_4)SO_4$	0.235	0.05–0.1
$(NH_4)SO_4$	0.235	0.1
$(NH_4)SO_4$	0.235	0.05–0.1
Alanine	0.1004	0.05
Alanine	0.1001	0.05
Blank (H_2SO_4 + NaOH)	..	0.00

The ammonia was estimated by adding to the residue in the Kjeldahl flask 5 cc. of Nessler's reagent and comparing with standards similarly prepared and containing known amounts of ammonium hydroxide. From the above results it is evident that in carefully conducted determinations practically all the ammonia is distilled off.

(*2*) *The Amount of Ammonia not Absorbed.*—To determin the amount of ammonia not absorbed, a second absorption bottle containing standard acid was put into the circuit so as to catch any ammonia escaping from the first absorption apparatus. The liquid in the second flask was tested for ammonia by titrating with standard alkali or by adding 5 cc. of Nessler's reagent.

Substance.	Amount taken. Grams.	NH_3 with Nessler.	NH_3 by titration.
Alanine	0.10055	.....	Negative
Alanine	0.1004	Negative	.....
$(NH_4)_2SO_4$	0.235	.....	Negative
$(NH_4)_2SO_4$	0.235	Trace	.....

The error due to incomplete absorption of ammonia is evidently negligible.

(*3*) *Spattering of Alkali and Impurity of Reagents.*—To determin the error due to spattering of alkali or to ammonia in the reagent, a series of distillation was conducted, using in each 20 cc. of sulfuric acid and 75 cc. of a saturated sodium hydroxide solution.

Substance.	Alkali found in distillate determined as NH_3. Grams.
H_2SO_4 and NaOH	0.00024
H_2SO_4 and NaOH	0.00024
H_2SO_4 and NaOH	0.00024
H_2SO_4 + 10 grams K_2SO_4	0.00024

The error being constant is probably due to impure reagents and as such can be easily corrected in the results.

(4) *The Amount of Ammonia Absorbed.*—The final test of the method rests upon the amount of ammonia absorbed or upon the % of nitrogen obtained with pure substances.

The following results were taken from the note book of K. Sugiura who made the experiments to determin the purity of glycine for work with the amino acids. He used the aeration method for small quantities according to Folin, and for larger quantities according to the method described in this paper.

RESULTS WITH FOLIN'S MICRO-METHOD.

Substance.	Amount taken. Grams.	Per cent. of N found.	Per cent. of N, theoretical.
Glycine	0.00798	18.03	...
Glycine	0.00798	18.28	...
Glycine	0.00798	18.17	...
Glycine	0.00798	18.21	...
Glycine	0.00798	18.21	...
Average		18.18	
Correction due to pipet		0.18	
		18.36	18.67

RESULTS WITH THE AERATION METHOD.

Substance.	Amount taken. Grams.	Per cent. of N found.	Per cent. of N, theoretical.
Glycine	0.1011	18.63	...
Glycine	0.1011	18.66	...
Glycine	0.1000	18.70	...
Average		18.66	18.67

According to these results, the Folin micro-method gives very useful results but the percentage accuracy, as might be expected, is lower than with our method. This is in harmony with the percentage accuracy which Folin[1] obtained with his method.[2] These experiments also show that the aeration method gives accurate results.

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[1] *J. Biol. Chem.*, **11**, 501 (1912).

[2] Folin states in his micro-method that digestion for 6–8 minutes is sufficient to convert the nitrogenous substances into ammonia. Since the ratio of nitrogen to sulfuric acid and K_2SO_4 is about the same as in the usual process, there is no reason to suppose that less time is required to complete digestion. Bang (*Biochem. Z.*, **51**, 194 (1913)) using similar amounts of substances digests at least 3 hours.

Boric Acid as an Absorption Medium for Ammonia.—L. W. Winkler[1] has suggested the use of boric acid in place of the standard acid in the distillation of ammonia. This may be a satisfactory absorption medium when distilling by steam, but a number of experiments made in this laboratory have proved it unsuitable in connection with aeration. To determin the amount of ammonia not absorbed by the boric acid a second absorption bottle containing a little standard acid was put in the circuit. In every case the second absorption liquid showed an appreciable quantity of ammonia when tested with Nessler's reagent.

Litmus Paper as Indicator in Dilute Solutions.—A series of experiments was made in this laboratory to determin the accuracy and reliability of litmus paper as an indicator in dilute solutions. The amounts of alkali or acid necessary for immediate reaction and time required for reaction of small amounts are given in the following tables.

In the first instance the test paper[2] was dipped quickly in and out of the solution in the second series *it was allowed to soak with stirring until change of color became apparent.* In each series the tenth normal solution used, was diluted with 100 cc. of distilled water.

A.

Cc. NH_4OH 0.1 *N*.	0.1.	0.2.	0.3.	0.4.	0.5.	1.0.	1.5.	2.0.	3.0.	4.0.	5.0.
Immediate reaction	—	—	—	—	—	—	—	—	—	—	+
Time of reaction, in min	3.75	2.5	1.45	1.08	0.5	0.3	0.2	0.15	0.1	0.05	0.0

B. Cc. 0.1 *N* NaOH	0.1.	0.2.	0.3.	0.4.	0.5.	1.0.	2.0.	3.0.	4.0.	5.0.
Immediate reaction	—	—	—	—	—	—	—	—	—	+

C.

0.1 *N* H_2SO_4	0.1	0.2	0.3	0.4	0.5
Immediate reaction	—	—	—	+	+

In dilute solutions the time necessary for reaction varies inversely as the concentration. These results show that litmus paper simply dipped into the liquid as is ordinarily done is not a satisfactory indicator of very small amounts of acid or alkali.

Summary.

We have shown:

(1) That the amount of ammonia left in the distillation residue after aeration for an hour is, with moderate amounts of ammonia, inappreciable.

(2) That the completeness of distillation is not dependent upon heat.

(3) That the absorption in the aeration method is complete.

(4) That the results with the method are fairly accurate.

(5) That litmus paper used in the ordinary way as recommended by Folin and others, is not suitable for the detection of small amounts of ammonia in distillates.

(6) That boric acid, recommended by Winkler for an absorption medium, cannot be used for the aeration method.

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[1] *Chem. Zentr.*, 23, 1936; *Z. angew. Chem.*, 26, 231–232.

[2] Squibbs "best."