[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF WESLEYAN UNIVERSITY.]

THE DISTILLATION OF AMMONIA IN THE DETERMINATION OF NITROGEN.

BY FRANCIS GANO BENEDICT.

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THE distillation of ammonia in the moist combustion process for determining nitrogen involves the separation of the gaseous ammonia from the alkaline liquid in the distilling flask and the transportation of the gas through a tube into the receiver. While the disengagement of ammonia from a strongly alkaline liquid is readily accomplished by the aid of heat the complete removal of ammonia from the condenser tube is only affected by prolonged boiling to wash the ammonium hydroxide formed out of the tube or by conducting live steam through the tube to vaporize the ammonia which is then driven by the water vapor into the receiver.

The first method is that employed in most laboratories in this country and is designated the "official" method. The second method is that employed in many German laboratories and is here designated the German method.

In the official method the distillation is continued until about 150 cc. of the distillate have been condensed and it is assumed that all the ammonia will have been transferred to the receiver during this operation. The gaseous ammonia coming in contact with the cold water adhering to the inside of the condenser tube dissolves in it forming ammonium hydroxide. The steam generated in the flask condenses in the upper part of the tube and the water formed, slowly runs down, washing out the ammonium hydroxide. It is easily seen that a considerable amount of water would be neeessary to insure complete removal of the alkali in this way. Experience has shown that not less than 150 cc. of distillate can safely be relied on to effect this result. This requirement is both expensive and tedious. The operation requires, as a general rule, from forty-five minutes to one hour. Of this time probably not over fifteen to twenty minutes are required to drive all the ammonia out of the liquid, the remaining time being occupied in washing the ammonium hydroxide out of the condenser tube.

According to the German method, the vapors are conducted from the distilling flask through a pipe into the acid, in the receiver with no preliminary cooling, and as a result the contents of the receiver are soon heated to boiling by the latent heat of the steam. Under these conditions, it has been found that after vigorously boiling for twenty minutes all ammonia has been driven out of the alkaline liquid in the distilling flask. Inasmuch as the conditions in this flask are the same in both cases it is only correct to assume that in the official method the ammonia is entirely expelled from the liquid in the same length of time.

Under the conditions existing in the distilling flask by the German method probably no ammonium hydroxide is formed in the condenser tube as the whole system is almost immediately brought to the temperature of steam. Accordingly the gaseous ammonia is carried along mechanically by the water vapor and conducted directly into the receiver.

The great saving in time and gas of this method over the official method is, however, partially offset by the fact that the contents of the receiver at the end of the distillation must be cooled before the titration is made when (as is commonly the case) ammonium hydroxide is used as a standard alkali. Unless the receiver is well cooled, a loss of ammonia is sure to occur.

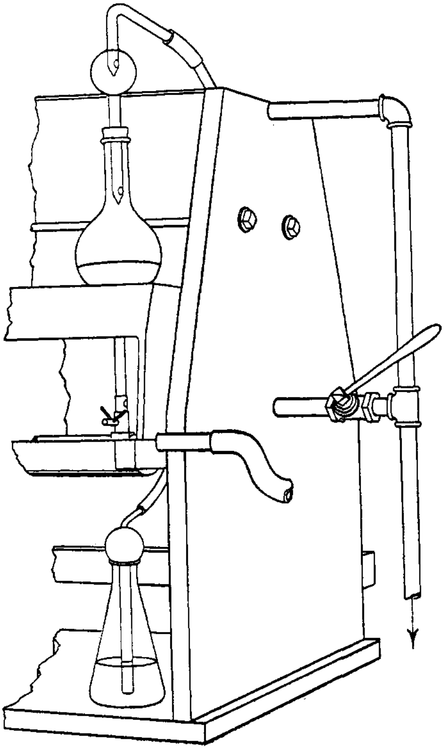
A further objection to the German method lies in the fact that a loss of acid is experienced by the passage of so much steam through the hot liquid in the receiver. This loss should not be neglected in accurate work.

The following modification is, in reality, a combination of the official and the German method, and consists briefly of distilling the liquid into a condenser containing cold water which is allowed to run out a few moments before the end of the distillation that the condenser tubes may become heated. The ammonia in the tubes is then vaporized and driven into the receiver by a current of live steam. By this means the rapid removal of ammonium hydroxide in the condenser tubes is effected without unduly heating the contents of the receiver.

The apparatus which has been in constant use in this laboratory for over a year is shown, in part, in the accompanying figure.

The principal feature is the arrangement of the condenser, a galvanized-iron tank 148 cm. long, 38 cm. deep, and 8 cm. wide.

Ten pieces of block tin pipe 4 mm. internal diameter and 70 cm. long are soldered as usual in the bottom of the tank with ends about 6 cm. long extending through for connection with the receiver and bulb. The upper ends are fastened to one side of the tank and each is provided with a rubber tube to which a Hopkins'[1] safety distilling bulb is attached.



At one end of the tank and near the bottom a piece of standard half inch pipe is inserted, fitted with a valve or gate which is in turn connected with a T. An overflow is provided near the top of the tank and is connected with the T, the lower arm of which is connected with a sink or drain. The tank which is filled from a water tap at the other end is fastened on a stout frame 35 cm. above the table. The space below is occupied by the receivers and bulb tubes used to prevent back suction of liquid.

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[1] This Journal, 18, 227.

With the form of apparatus above described the distillation is carried out as follows: The gas is turned on full and the burners are all lighted.[1] As the liquid in the distilling flask is quite warm from the heat of the dilution of the sulphuric acid and the heat generated by the reaction between the acid and the alkali, the flasks are all vigorously boiling, as a rule, inside of four or five minutes. When all are boiling the time is noted and at the expiration of fifteen minutes the valve is opened, allowing the water to flow out of the condenser. As the water runs out the cold water at the bottom is first withdrawn, the layer of hot water at the top gradually settling and warming the block tin pipes. Just before the layer of hot water begins to flow out the whole condenser tube becomes hot enough to prevent any condensation of water and the live steam blows through the tube into the receiver. This is allowed to continue till the water in the receiver is warmed slightly, the degree of warming being readily determined by occasionally placing the hand on the receiver. As soon as a rise in temperature is perceptible the receiver and bulb are disconnected. This operation requires from four to six minutes after opening the water valve in the apparatus here used. The contents of the flask are therefore vigorously boiling for from nineteen to twenty-two minutes. Repeated tests have shown that all the ammonia is driven off under these conditions. With excessive amounts of ammonia, the boiling operation can be prolonged five minutes before opening the valve. This is seldom necessary, however. Ordinarily, twenty-five to thirty minutes from the lighting of the gas under the flasks to the removal of the receivers suffice to complete the distillation. The titrations can be made immediately.

The length of time necessary to draw off the water in the condenser tank to such a level as to cause live steam to blow through the block tin pipes, is determined by the sizes of the waste pipe and the condenser. With very little experience, it is not difficult to determine how long after boiling the valve should be opened to produce the desired effect at the end of twenty minutes.

The condenser tank here described was not constructed pri-

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[1] The use of Jena glass digestion as well as distillation flasks is strongly recommended. With such flasks the full free flame may be employed with impunity. The distillation flasks contain approximately 500 cc. and are about half filled with the alkaline liquid.

marily for this method of distillation. It could undoubtedly be diminished very materially in size with equally satisfactory results.

Obviously the principle is capable of application to numerous forms of condensers. It is only necessary to observe that the flasks are vigorously boiling for at least nineteen or twenty minutes. In a local laboratory the principle has been easily applied to a two-tube condenser constructed out of an old ether can. This apparatus has also given excellent satisfaction for over a year.

MIDDLETOWN, CONNECTICUT.

THE GLUTEN CONSTITUENTS OF WHEAT AND FLOUR AND THEIR RELATION TO BREAD-MAKING QUALITIES.

BY H. A. GUESS.

Received March 22, 1900.

DURING the last eighteen months the writer has been pursuing some investigations in the chemical factors which determine bread-making qualities of wheats and flours, and the notes here submitted, representing some results of the work, are offered as contributions to the chemical literature of an as yet rather neglected subject.

As aids in the work advantage was taken of published notes by other investigators, acknowledgments being more particularly due to the papers of M. E. Fleurent,[1] and of Mr. G. L. Teller.[2]

The result of considerable preliminary work in the estimation of the proteids of both wheats and flours, and subsequent comparisons with results of milling and baking tests, led the writer to confine his attention more particularly to the determination of the gluten constituents gliadin and glutenin, as being apparently the chief determining factors in bread-making qualities.

In the estimation of the gliadin and glutenin, five nitrogenous bodies were considered as being present in the samples,—gliadin,

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[1] "Sur la composition immédiate du gluten des cereals."—*Compt. rend.*, 123, 327. "Determination of Gluten in Wheat Flour."—Third Congress of Applied Chemistry. Vienna, August, 1898.

[2] "Concerning Wheat and its Mill Products: A report of progress of investigations in the chemistry of wheat."—Arkansas Expt. Station, 1898.