

alent of the stearic acid contained in the lecithin, and calculating to the respective glycerol esters the remaining saturated acids (as stearic acid), the oleic acid, and the linolic acid, the following summary is obtained as the composition of the oil of corn :

	Per cent.
Cholesterol .....	1.37
Lecithin.....	1.49
Stearin (?) .....	3.66
Olein .....	44.85
Linolin .....	48.19
Total .....	99.56

### SOME ERRORS IN THE DETERMINATION OF NITROGEN.<sup>1</sup>

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THE determination of nitrogen was made by the ordinary Kjeldahl method. The metallic mercury used in the digestion was measured in a capillary tube, one end of which is doubly bent so as to form a loop, the short arm of which is turned back upon itself near the end while the long arm serves as a handle. The loop is made sufficiently narrow to pass into the mercury bottle, and of sufficient length to retain, when raised above the liquid, the exact quantity of mercury required for a single determination. By blowing in the longer arm the mercury is emptied into the digestion flask.

Heavy copper flasks were used in the distillation with much satisfaction, the sodium hydroxide solution (containing the necessary amount of potassium sulphide) being added in sufficient excess to "bump" before the contents may become dry, thus serving as a signal that the distillation has gone far enough.

Two common sources of error in the nitrogen determination were found and investigated. In titrating an acid solution in an open vessel with standard ammonia solution a very appreciable error is introduced by the volatility<sup>2</sup> of the ammonia, although the only possible loss is from the tip of the burette and from the falling drops.

<sup>1</sup> From advance sheets of the author's thesis, "The Chemistry of the Corn Kernel," for the degree of Doctor of Philosophy, Cornell University, 1898, which will be published as Bulletin No. 53 of the University of Illinois Agricultural Experiment Station.

<sup>2</sup> Rempel has already shown that dilute ammonia solution drawn into beakers or evaporating dishes and then titrated suffers marked loss.—*Ztschr. angew. Chem.*, 1889, 331.

In the following work ammonia of about one-sixth normal strength was used, the hydrochloric acid being of such strength that three cc. were equivalent to approximately four cc. of ammonia. The hydrochloric acid was measured from an automatic overflow pipette of fifteen cc. capacity, and the ammonia from an automatic overflow burette graduated to 0.05 cc. and drawn to a fine tip at the outlet. The pipette and burette were each provided with three-way stop-cocks through which the standard solutions were drawn from the stock bottles by means of siphons. Perfectly neutral water free from ammonia and carbon dioxide was used for diluting. Lacmoid served as the indicator and gave an exceedingly sharp end-reaction.

By titrating in beaker flasks with the tip of the ammonia burette well below the top of the flask the following results were obtained, the length of time taken in making the titration being also given :

No.	Hydrochloric acid taken. cc.	Ammonia required. cc.	Time, minutes.
1.....	15	20.10	1
2.....	15	20.08	1
3.....	15	20.12	1
4.....	15	20.30	2
5.....	15	20.25	2
6.....	15	20.40	3

By titrating in an Erlenmeyer flask attached to the burette by means of a rubber stopper,<sup>1</sup> provided with a capillary tube for relieving the pressure, the following results were obtained :

No.	Hydrochloric acid taken. cc.	Ammonia required. cc.	Time, minutes.
1.....	15	19.82	1
2.....	15	19.83	1
3.....	15	19.81	3
4.....	15	19.81	5

As from three to five minutes are taken to make a titration when the amount of ammonia required is not known, as in ordinary nitrogen determinations, the error<sup>2</sup> from titrating in open vessels becomes an important factor, the total variation in the two series of experiments above noted amounting to six-tenths

<sup>1</sup> By using a stopper which has been bored nearly through from the small end by a large borer, the flask may easily be given a free rotary motion.

<sup>2</sup> Confirmed by recent (unpublished) work of Dr. F. L. Kortright, of Cornell University.

cc. or three per cent. of the ammonia required. The fact that the density of ammonia gas is but little more than half that of air explains its rapid upward diffusion from an open vessel.

Another error in nitrogen determinations may occur in the distillation by loss of ammonia from the receiving flask in case there is not sufficient acid *above the end of the delivery-tube* to neutralize all of the ammonia distilled over.

In the following work a quantity of a very dilute solution of ammonium chloride was prepared by exactly neutralizing standard hydrochloric acid with standard ammonia and diluting with ammonia-free water. A quantity of this solution equivalent to twelve cc. of standard ammonia was placed in a distillation flask with an excess of sodium hydroxide and distilled into fifteen cc. of standard hydrochloric acid diluted to about forty cc., the end of the delivery-tube from the condenser dipping *well into the acid solution*. The relation of the standard acid and ammonia solutions was such that fifteen cc. hydrochloric acid were equivalent to 19.82 cc. ammonia. Six distillations were made, in each case ammonium chloride equivalent to twelve cc. of standard ammonia solution being taken. Following are the amounts of standard ammonia solution required to neutralize the excess of acid :

No.	Required. cc.	Calculated. cc.	Error. cc.
1.....	8.20	7.82	0.38
2.....	7.85	7.82	0.03
3.....	7.93	7.82	0.11
4.....	8.60	7.82	0.78
5.....	7.84	7.82	0.02
6.....	7.95	7.82	0.13

Two of these are practically exact, the other four showing errors varying from 0.11 cc. to 0.78 cc. of standard ammonia.

This work was repeated with the distillation from quantities of ammonium chloride equivalent to fifteen cc. of standard ammonia solution, the other conditions being as before. Following are the amounts of standard ammonia solution required to neutralize the excess of acid :

No.	Required. cc.	Calculated. cc.	Error. cc.
1 .....	6.10	4.82	1.28
2 .....	5.40	4.82	0.58
3 .....	5.95	4.82	1.13
4 .....	6.20	4.82	1.38
5 .....	5.65	4.82	0.83
6 .....	5.18	4.82	0.36

Diluting the residues in the distillation flasks with ammonia-free water, and distilling, gave no further addition of ammonia in any case.

It was observed that in both trials the greatest errors occurred with Nos. 1 and 4. A careful inspection of the apparatus showed all connections to be perfect. It was observed, however, that the delivery-tubes from Nos. 1 and 4 did not reach as far into the acid solution as most of the others.

With the thought that possibly ammonia escaped from the receiving-flasks, the following six distillations were made, in each the quantity of ammonium chloride employed being equivalent to 19.32 cc. of standard ammonia solution; thus, exactly five-tenths cc. of standard ammonia should have been required to neutralize the excess of acid. Some lacmoid indicator was added to the acid solutions in receiving-flasks Nos. 1, 3, and 5; strips of moistened red litmus paper were also hung in the necks of these flasks. During the process of distillation, receiving-flasks 2, 4, and 6 were agitated to keep their contents thoroughly mixed.

It was observed that, during the process of distillation, in receiving-flasks 1, 3, and 5 the liquid above the end of the delivery-tube turned blue, while a layer of liquid below this remained red; also that the moistened red litmus paper hung in the necks of these flasks turned blue.

In titrating the excess of acid the amounts of standard ammonia required were as follows:

No.	Required. cc.	Calculated. cc.	Error. cc.
1 .....	2.60	0.50	2.10
2 .....	0.50	0.50	0.00
3 .....	2.27	0.50	1.77
4 .....	0.53	0.50	0.03
5 .....	1.99	0.50	1.49
6 .....	0.50	0.50	0.00

The explanation for the separation of the liquid in the receiving-flasks into two layers as described is to be found in the different densities of aqueous solutions of ammonia and hydrochloric acid.

In subsequent work I have used delivery-tubes reaching to the very bottom of the receiving flasks, and contracted at the end to an aperture of but four or five mm. diameter. This insures considerable agitation of the contents of the receiving flask produced by irregularities in the boiling of the liquid in the distillation flask.

This loss of ammonia shown to have taken place from the very dilute solution in the receiving flask after cooling by an efficient condenser emphasizes the results of the preceding work on titration and the importance of avoiding a common error in that process.

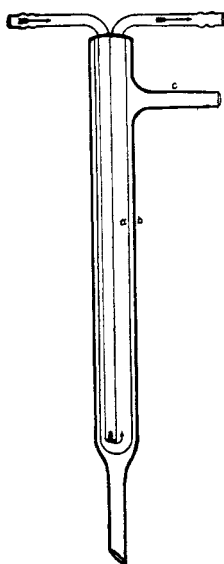
### A CONDENSER FOR EXTRACTION WORK.<sup>1</sup>

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MAINLY to avoid the constant trouble of having atmospheric moisture condense upon the outer surface of a Liebig or Allihn condenser and run down over the extraction apparatus, the following form of condenser was designed:

This condenser is made entirely of glass, and consists of a *thin* glass tube *a* twenty-five mm. *outside* diameter and twenty-five cm. long, provided with two glass tubes about six mm. in diameter, one reaching to near the bottom of *a*, sealed in for water inlet and outlet. The tube *a* is surrounded by a stronger glass tube *b* of thirty mm. *inside* diameter sealed on at the top and narrowed at the lower end to a ten mm. tube which extends eight mm. below and is ground off obliquely at the end. About



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