chemistry has been a matter of thought from time to time for some years, but the matter took definite shape in its present form in the autumn of 1907.

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NOTE.

The Determination of Ammonia without a Condenser.—Often it is desired to determine the nitrogen content of a substance without distillation and it was with the idea of devising a suitable general method that this work was undertaken. Kober[1] describes a method for the determination of nitrogen in urine without using a condenser. The same method is used under various circumstances where heat is not desired. Kober's method is to make a solution of urine by digestion similar to the regular Kjeldahl and after the solution is complete to slightly dilute it. Sodium hydroxide solution is added by drawing it over from another vessel by means of suction. After the solution is alkaline, air is drawn through at as rapid rate as possible without drawing over some of the solution. The ammonia carried over with the air is absorbed by standard acid. A specially strong suction pump is needed.

Cottonseed meal was selected for the experiments here described because of its variable nitrogen content and its difficulty of digestion. At first the method as carried out by Kober gave very discordant results. This was due to two difficulties: (1) Some of the ammonia was not absorbed by the acid, and (2) all the ammonia was not carried over.

Various forms of absorption tubes were tried until finally the one giving best results was a Folin tube supplemented by a tube filled with glass beads wetted with acid, as shown in Fig. 1.

The second difficulty seemed to arise from the facts that the solution, which was just brought to boiling by the heat of neutralization, cooled too rapidly; and that the stream of air drawn by the ordinary glass pump was not strong enough to carry over the ammonia by the time the solution cooled.

If the flask (Kjeldahl) containing the solution were placed in an asbestos box, the solution kept warm for an hour and a half. To the solution was added scrap aluminium which reacts rapidly with the alkali, setting free hydrogen. With these modifications the results checked with results obtained by the ordinary Kjeldahl.

It was also found that the digestion could be made with sulphuric acid and aluminium, and the results check with digestion using salicylic acid and mercury. The air must be drawn through at least an hour, but it generally requires somewhat longer to remove all ammonia.

[1] *Jour. Amer. Chem. Soc.*, 30, 1131.

Using 30 cc. of sulphuric acid for digestion of ½ gram of substance, about 100 cc. of water were added before neutralization. The heat of neutralization was then just sufficient to make the solution boil. If a larger quantity of substance is used the solution must be kept near the boiling point with a rose burner for about fifteen minutes in order to get all the ammonia removed.

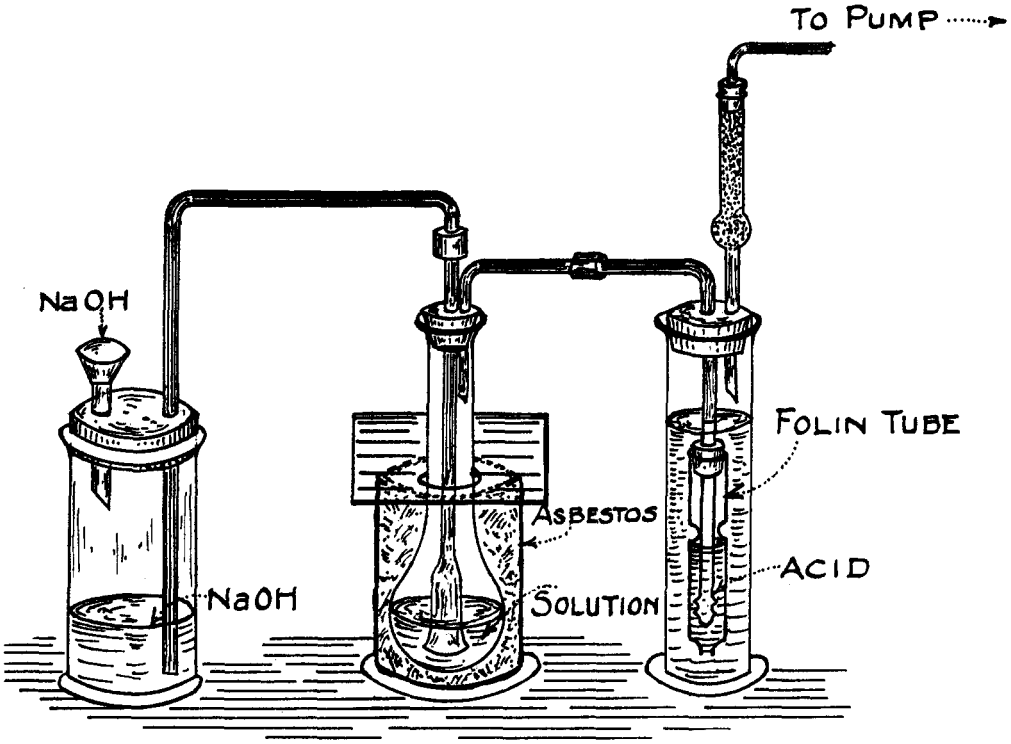

FIGURE 1

Some of the results obtained by the Kjeldahl and by the absorption methods are as follows:

Per cent. ammonia.		
Kjeldahl.	Absorption.	
7·73	7·78	Both digested with Hg and salicylic acid: air drawn through 1 hour.
7·73	7·75	Absorption digested with Al: air drawn 1 hour.
7·07	7·14	Both Hg and salicylic acid: air 1 1/4 hours.
6.97	7·04	Both digested with Al 90 min.: air drawn through 1 1/4 hours.
7·65	7·61	Hg and salicylic acid; air 1 hour.
7·48	7·41	Hg and salicylic acid: air 1 hour.
7·57	7·65	Hg and salicylic acid for Kjeldahl: Al for absorption: air drawn through 1 hour.

From these experiments the conclusion is reached that the method described by Kober for urine, if modified as described, will give good results with other nitrogen-containing bodies. R. O. E. DAVIS.

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3-METHYL-HEPTANE.

BY LATHAM CLARKE.

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This paper contains an account of the synthesis and properties of the octane, 3-methyl-heptane, $CH_3CH_2\underset{\displaystyle CH_3}{\underset{|}{CH}}CH_2CH_2CH_2CH_3$, and forms a continuation of the study of the octanes which was begun some time ago in this laboratory.[1]

3-Methyl-heptane has already been prepared and described by Mlle. Welt,[2] but as her product boiled from 110° to 120°, it was evidently too impure for the careful study of the physical constants, which is one of the objects of this research. The method of preparation according to Wurtz was employed by Mlle. Welt, who treated amyl iodide and ethyl iodide with sodium.

In this study of 3-methyl-heptane one method was used, which guaranteed a pure product, and alone of those considered was simple enough to be used with ease. It consisted of the following reactions: Ethyl normal propyl acetoacetate was made and saponified, forming methyl normal butyl ketone, which on treatment with ethyl magnesium bromide produced 3-methyl-heptanol. This last compound was converted into the corresponding iodide and reduced, yielding the desired octane.

Summary of Reactions.

$CH_3CH_2CH_2I + CH_3COCH(Na)CO_2C_2H_5 \longrightarrow$

$CH_3CO\underset{\displaystyle CH_2CH_2CH_3}{\underset{|}{CH}}-CO_2C_2H_5$, ethyl normal propyl acetoacetate $\longrightarrow$

$CH_3CO\,CH_2CH_2CH_2CH_3$ methyl normal butyl ketone or 2-hexanone $\longrightarrow$

$CH_3CH_2\underset{\displaystyle CH_3}{\underset{|}{C}}(OH)CH_2CH_2CH_2CH_3$, 3-methyl-3-heptanol $\longrightarrow$

$CH_3CH_2-\underset{\displaystyle CH_3}{\underset{|}{C}}-(I)-CH_2CH_2CH_2CH_3$, 3-methyl-3-iodoheptane $\longrightarrow$

[1] A full list of the octanes hitherto described will be found in the January, 1909, number of THIS JOURNAL, 31, 107.

[2] *Ann. Chim. Phys.* [7], 6, 121.