A NEW LABORATORY METHOD FOR THE PREPARATION OF ACETAMIDE.

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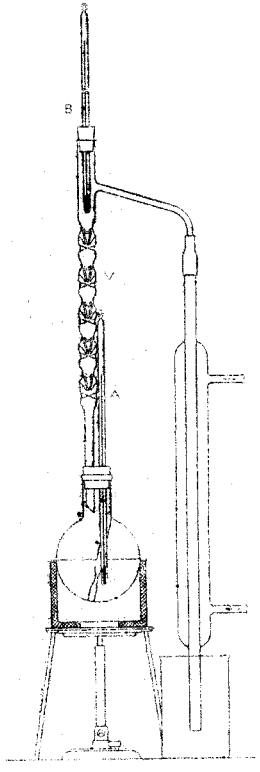
Of the methods now in general use for the preparation of acetamide, none is entirely satisfactory. In most cases the yield is extremely small and the length of time for the preparation is considerable. Some of the methods require apparatus that is cumbersome and expensive, and considerable skill in manipulation is necessary. The following method can be used by the average student working with ordinary laboratory apparatus. The method, furthermore, gives a good yield, and can be carried out in one laboratory period of four hours. It is based on the experiments of Rosanoff, who showed that an excess of acetic acid is favorable to the formation of acetamide.[1]



Arrange an apparatus as shown in accompanying figure. A 250 cc. round-bottom flask is fitted with a cork carrying a thermometer, *A*, and an ordinary 15 cm. (large size) Vigreux column, *V*.[2] The top of the Vigreux column is closed by a cork carrying a thermometer, *B*. The side arm of the column is bent downward and connected with an upright condenser. The flask is placed on an asbestos air bath, and a boiling tube is placed in the flask to insure even boiling.

Place 42 grams of powdered commercial ammonium carbonate in the flask and add gradually 125 grams of glacial acetic acid. Connect the flask with the Vigreux column and heat gently until the ammonium carbonate is all dissolved. Increase the temperature until the liquid boils. (The distillate should not come over faster than 30 drops per minute—better, only 20 drops per minute for the first half hour.) Gradually increase the heat until the thermometer in the flask indicates 223°, the boiling point of acetamide. Discontinue the heating and cool the residue left in the flask by pouring into a beaker, or evaporating dish, and stirring to avoid caking. Grind the material in a mortar and wash several times with ether, in which it is only slightly soluble, and finally press out on drying paper and allow to stand until the ether evaporates.

[1] This Journal, 33, 974 (1911).

[2] The Vigreux column may be replaced by the simple Hempel column containing glass beads, but the yield obtained is somewhat lower.

This material is almost pure acetamide. If it is desired to obtain a sample free from all impurities, it may be recrystallized from a large volume of ether. If the sample is colored yellow, due to the charring of the corks, it can be obtained colorless and practically pure by distilling from a small retort. However, such distillation gives rise to a small amount of acetonitrile, which lowers the melting point of the crystals.

The yield is about 40 grams, or 85–90% of the theoretical amount calculated from the nitrogen content of the ammonium carbonate. The actual time required for the experiment is four hours.

This method has been used in the laboratories of organic chemistry at Cornell University for one year and has proven very satisfactory.

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A MODIFIED METHOD FOR THE PREPARATION OF TRIETHYLAMINE.

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Considerable difficulty has been experienced in the investigation of triethylammonium nitrite,[1] in the preparation of triethylamine. In order to obtain the base in a pure condition, it was thought feasible to prepare tetraethylammonium bromide by the method described by Scott;[2] next, to convert it into the quaternary hydroxide by the interaction with moist silver oxide, which on heating would decompose into triethylamine and ethylene. But, as has been already noticed,[3] the method was not found suitable for the purpose; the lack of success has been, reasonably enough, assigned to the higher atmospheric temperature of this tropical country. Next Hofmann's[4] method was tried; and in spite of the want of the indespensable details of this classical memoir, the base has been prepared by it. But the yield being comparatively poor, it was thought worth while investigating the exact condition under which the triethylamine can be conveniently prepared.

Of the two alkyl halides, ethyl iodide and bromide, the latter has been found easier to prepare, the former invariably yielding a mixture of all four bases under different circumstances. Seventy-five cc. of ethyl bromide and 50 cc. of strong ammonia (sp. gr. 0.88) were taken in a 750 cc. thick glass flask, sufficient absolute alcohol was then added to effect solution, and the flask immediately closed with a rubber cork and tied with wire. Next the flask was placed in a steam oven for three hours. After cooling, it was opened and the alcohol (which may be again used for the

[1] Ray and Rakshit, *J. Chem. Soc.*, **101**, 216 (1912).

[2] *J. Chem. Soc.*, **95**, 1200 (1909).

[3] *Loc. cit.*

[4] *Phil. Trans.*, **1850**, Pt. L, 121.