

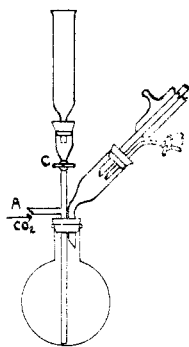
## NOTE

**Laboratory Preparation of Acetaldehyde.**—Recent work by Adams and Williams<sup>1</sup> has shown that in the preparation of acetaldehyde by the oxidation of alcohol, it is important to remove the aldehyde from the flask as fast as it is formed in order to avoid its further oxidation. By employing a mercury-sealed stirring device they were able to increase greatly the yields formerly obtainable. The present work was undertaken in order to simplify the apparatus, shorten the time required and increase the yield further.

In this work the stirring was performed by a stream of carbon dioxide and the apparatus was so adjusted that samples of the liquid refluxing in the condenser could be withdrawn at any time. Using the directions of Adams and Williams with this apparatus the yields of aldehyde-ammonia for 50 g. and 25 g. of alcohol were, respectively, 55.5% and 62%. At the conclusion of each run the contents of the flask were boiled for 30 minutes while the stream of carbon dioxide was running through the apparatus. Tests at this time showed the presence of unoxidized alcohol, and the addition of excess acid and dichromate resulted in the production of more aldehyde. This makes it evident that the amount of acid called for was not sufficient, though Adams and Williams used double the amount recommended by Gattermann.<sup>2</sup>

In the present work the full amount of acid called for by calculation has been used. Various methods of mixing the components have been compared, and oxidizing agents other than sodium dichromate have been employed, also other acids than sulfuric. The effect of the concentration of the oxidizing mixture (important) has been studied.

A mixture of nitric and sulfuric acids, and sodium (or potassium) dichromate gives the best results of the oxidizing agents which were tried.



It was found preferable first to place in the flask the alcohol to be oxidized and then run in the mixed acids and dichromate. The yield is better when small amounts of alcohol are oxidized at a time, and the minimum quantity seems to be about 25 g. The yield is likewise raised when the reaction is run rapidly; for this reason ice water is admitted to the condenser instead of warm water as has formerly been the rule. The apparatus has also been so arranged that it need not be taken down for recharging.

A 2-liter round-bottom flask is fitted with a 2-hole stopper bearing a thistle tube with stopcock and side-tube, and a bent adapter which leads to a reflux condenser. The thistle tube leads nearly to the bottom of the flask where it is drawn to a diameter of about 2 mm. The reflux

<sup>1</sup> Adams and Williams, *THIS JOURNAL*, 43, 2420 (1921).

<sup>2</sup> "Practical Methods of Organic Chemistry," Macmillan Co., 1920, p. 167.

condenser has an internal diameter of about 12 mm. and the water jacket should be at least 80 cm. in length. A sampling tube (B) with stopcock is fitted as shown at the bottom of the condenser. Near the top of the condenser a thermometer is hung by a thread and pin placed in the stopper at the outlet. A vertical glass tube leads from the condenser to the absorption bottles, which are two 500cc. high-form Drechsel wash bottles. About 7.5 meters of rubber tubing is loosely coiled about the absorption bottles and all is placed in a large pail of ice and salt. The water for the condenser is passed through the rubber tube and enters the condenser at about 5°.

Twenty-five g. of alcohol is placed in the flask; the first absorption bottle contains 150 cc. of ether dried over sodium, and the second, 125 cc. The oxidizing mixture consists of 115 g. of nitric acid (sp. gr. 1.42) poured onto 60 g. of sodium (or potassium) dichromate, with the further addition of 220 cc. of water and 20 g. of sulfuric acid (sp. gr. 1.84). When potassium dichromate is used, 300 cc. of water will be required. The alcohol is heated to boiling with a small flame, the flame is then lowered and the oxidizing mixture added at such a rate that the thermometer in the condenser reads between 20° and 25°. The introduction of the oxidizing mixture usually takes about 20 minutes. Carbon dioxide is supplied from a large Kipp generator and is run in at (A) with sufficient speed to keep the contents of the flask in active motion. When all the oxidizing mixture has been introduced the contents of the flask are boiled for an additional 10 minutes or until tests at (B) show that no more aldehyde is passing over. Dry ammonia gas is passed into the absorption bottles in the usual manner, and after standing in an atmosphere of ammonia for 3 to 4 hours the crystals of aldehyde-ammonia are collected, washed, dried, and weighed; yield, 70 to 72%. At the conclusion of a run the carbon dioxide tube is slipped off, and the tube leading to absorption bottles clamped. Air pressure at (B) forces the contents of the flask from (A); fresh absorption bottles are now attached and the apparatus is ready for another run.

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## NEW BOOKS

**Kapillarchemie.** Eine Darstellung der Chemie der Kolloide und verwandter Gebiete.

By Prof. Dr. HERBERT FREUNDLICH, Fellow of the Kaiser-Wilhelm-Institute für physikalische Chemie und Elektrochemie. Second fully revised edition. Akademische Verlagsgesellschaft M. B. H., Leipzig, 1922. xvi + 1181 pp. 157 fig. 24 × 15.5 cm.

I consented to review the second edition of this book within a short time thinking that it would be like most second editions—a few new added chapters and a bolstering up of the old arguments with the experimental material that has appeared since the issuance of the first edition (1909).

This particular second edition is, however, a new book with the old