ACTA CHEMICA SCANDINAVICA 19 (1965) 2420–2442

Short Communications

Preparation of Dimethylallylamine

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Dimethylallylamine has recently been introduced as a component of a buffer system in Edman's [1] phenylisothiocyanate method of peptide degradation. The methods of its preparation described in the literature [2,3] run with fairly poor yields. The present author found the following procedure to give a more satisfactory yield.

500 ml of benzene, 200 ml of 40 % aqueous solution of dimethylamine and 30 ml of a 50 % solution of sodium hydroxide are introduced into a 2 liter flask supplied with a condenser, 50 cm in length, a stirrer, a drop funnel, and a thermometer. The cooling liquid pumped through the condenser from a storage tank with cooled 60 % EtOH should have a temperature slightly below −2°C. During the course of 10–15 min 30 ml of allyl chloride is dropwise introduced under stirring. The temperature of the reaction mixture is not allowed to rise above 36° and leakage is carefully to be avoided. After stirring for 1 h another lot, 30 ml, of allyl chloride is slowly introduced, again not allowing the temperature to rise above 36°. After stirring for 1 h 120 ml of 50 % sodium hydroxide are added without any rise in temperature and then dropwise 60 ml of allyl chloride, again keeping the temperature below 36°. The stirring is continued overnight. The following day 250 ml of 50 % w/v sodium hydroxide are added and the reaction mixture is transferred to a 2 liter separatory funnel. After removing the alkaline aqueous layer the benzene layer is poured into 800 ml of 2 N hydrochloric acid cooled with ice under stirring. The acid aqueous layer is collected, cooled with ice and a 50 % sodium hydroxide solution added as long as the floating layer of dimethylallylamine is increasing. The amine is collected, dried with solid potassium hydroxide and fractionated in a Widmer column. A fraction distilling at 61° is discarded. The main fraction, about 100 g, distills over at 62–63°. The yield depends largely upon the extent to which leakages can be avoided.

1. Blombäck, B., Blombäck, M., Edman, P. and Hessel, B. *Biochim. Biophys. Acta*, No. 25478. *In press.*
2. Weston, A. W., Ruddy, A.W. and Suter, C.M. *J. Am. Chem. Soc.* **65** (1943) 674.
3. Cope, A.C. and Towlo, P.H. *J. Am. Chem. Soc.* **71** (1949) 3423.

Received November 4, 1965.