Preparation of 1,2-Dialkylcyclohexanes.—Preliminary tests showed that the 1,2-dialkylcyclohexenes were resistant to hydrogenation at room temperature and 1–2 atmospheres of hydrogen using platinum as catalyst. Hydrogenation occurred smoothly in a bomb at 150° and a starting hydrogen pressure of 2400 p. s. i. using 0.5–1.0 g. of Raney nickel catalyst for 2–5 g. of cyclohexene. Absolute ethanol (30–50 ml.) was used as solvent in all cases. After filtration the solution was evaporated to approximately 10 ml. and poured into water. The mixture was extracted with benzene and the combined extracts dried over sodium sulfate. After evaporation of the solvent the residue was treated with concentrated sulfuric acid to remove any cyclohexenes. The mixture was diluted with water and was extracted with benzene which was washed with dilute sodium hydroxide and water and dried over sodium sulfate. On distillation colorless liquids were obtained. Data on the cyclohexenes prepared are given in Table III.

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RECEIVED JUNE 21, 1949

Preparation of 2-Pyridylmethanol[1]

BY WALTER M. EDWARDS[2] AND PEYTON C. TEAGUE[3]

Harries and Lenart[4] and Graf, *et al.*,[5] have reported the preparation of 2-pyridylmethanol by methods which require several steps beginning with readily obtainable derivatives of pyridine. This report describes a simple method of preparation of 2-pyridylmethanol from 2-picoline, which does not involve the isolation of any intermediate product. 2-Picolyllithium was prepared by the hydrogen–metal interchange reaction between 2-picoline and phenyllithium. The picolyllithium was oxidized with a slow current of air to form the desired 2-pyridylmethanol.

A compound, $C_{12}H_{12}N_2$, was obtained as a by-product of the reaction. This compound was not identified but is probably 1,2-dipyridylethane, which would be expected[6] as the coupling product of two pyridyl radicals.

Experimental[7]

2-Picolyllithium was prepared by the procedure of Finkelstein and Elderfield[8] from 46.5 g. of 2-picoline. When the reaction was complete, the source of nitrogen was removed. Dry, carbon dioxide-free air was drawn at the rate of 2 cc. per minute into the flask and over the surface of the solution. The oxidation was carried out with stirring and without heating or cooling for eight hours, at which time the bright red color had disappeared and the mixture was light yellow. From time to time, more anhydrous ether was added to replace that lost by evaporation.

For the separation of the product, 6 *N* hydrochloric acid was added until the solution was acid to congo red. The aqueous layer was separated, made alkaline with sodium carbonate, saturated with sodium chloride, and extracted several times with chloroform. After the chloroform layer had been dried over anhydrous calcium sulfate for twenty-four hours, the chloroform and 2-picoline was distilled from the mixture at atmospheric pressure. The picoline boiled at 128° and was identified by the melting point of its picrate (168°). It weighed 11.3 g. or 24% of the original amount added.

The mixture of free bases was then fractionally distilled under reduced pressure. The following fractions were obtained: Fract. 1 (70–103° at 3–4 mm.), 11.2 g.; fract. 2 (103–136° at 3–4 mm.), 8.0 g., fract. 3 (138–170° at 1–2 mm.), 0.9 g.; residue, 16 g.

Identification of 2-Pyridylmethanol.—Fraction 1 on redistillation boiled completely at 111–115° at 16 mm. *Anal.* Calcd. for C_6H_7NO: N, 12.84. Found: N, 12.64. The picrate melted at 150° and the chloroplatinate at 179°. These data are in agreement with the values of Harries and Lenart.[4]

The yield of 11.2 g. was 20.5% based on the 2-picoline added or 27.0% on the basis of 2-picoline consumed in the reaction.

Compound $C_{12}H_{12}N_2$.—Fraction 2 redistilled at 112–114° at 1–2 mm. The distillate solidified on standing. After recrystallization from ligroin, its melting point was 49°. *Anal.* Calcd. for $C_{12}H_{12}N_2$: C, 78.23; H, 6.57; N, 15.21. Found: C, 77.98; H, 6.67; N, 15.05. Molecular weight determination by the Rast camphor method. Calcd.: 184. Found: 197, 196, 193; av. 195. This product was not further characterized.

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RECEIVED APRIL 27, 1949

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(1) Abstracted from the thesis of Walter M. Edwards, submitted in partial fulfilment of the requirements for the M.S. degree at the University of Georgia, Athens, Georgia.

(2) Present address: Ohio State College, Columbus, Ohio.

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(4) Harries and Lenart, *Ann.*, **410**, 107 (1915).

(5) Graf, *et al.*, *J. prakt. Chem.*, **146**, 88 (1936).

(6) Gilman and Pacevitz, THIS JOURNAL, **61**, 1603 (1939).

(7) All melting points and boiling points are corrected.

(8) Finkelstein and Elderfield, *J. Org. Chem.*, **4**, 365 (1939).

Polyvinyl Bromide[1]

BY DAVID EDELSON AND RAYMOND M. FUOSS

In connection with our work on polyelectrolytes,[2] we attempted to prepare a polymeric quaternary compound by the addition of tertiary amines to polyvinyl bromide. The desired product was not obtained, but the results of some of our experiments seem worth reporting. Vinyl bromide is one of the earliest known vinyl compounds; Regnault[3] prepared it by treating ethylene dibromide with alkali. Staudinger[4] studied its polymerization and noted that the polymer readily loses hydrogen bromide.

We heated 0.5 cc. of 30% hydrogen peroxide and 10 g. of vinyl bromide[4a] in a bomb at 47° for twenty-four hours and obtained no polymer, although Güyer and Schütze[5] report complete conversion in twenty hours at 60°. About 30% conversion was obtained in two days at 60° from a (deoxygenated) solution of vinyl bromide (10 g.) in toluene (8 g.) saturated with benzoyl peroxide. The product was white, but darkened on drying under vacuum at 30°; Parr bomb bromine averaged to 70.2% (theoretical 74.77%). Fair results were obtained by photochemical

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(1) Project NR 054-002 of the Office of Naval Research.

(2) R. M. Fuoss and G. I. Cathers, *J. Polymer Sci.*, **2**, 12 (1947); **4**, 97 (1949); R. M. Fuoss and U. P. Strauss, *ibid.*, **3**, 246 (1948); G. I. Cathers and R. M. Fuoss, *ibid.*, **4**, 121 (1949).

(3) V. Regnault, *Ann. chim.*, [II] **59**, 358 (1935).

(4) H. Staudinger, M. Brunner and W. Feisst, *Helv. Chim. Acta* **13**, 805 (1930).

(4a) We are indebted to the Dow Chemical Company for the sample of vinyl bromide on which these experiments were made.

(5) A. Güyer and H. Schütze, *Helv. Chim. Acta*, **17**, 1544 (1934).