our purposes because the boiling points of phosphorus trichloride and acrylyl chloride are within a degree of one another. We therefore adopted the method using benzoyl chloride for the preparation of aliphatic acid chlorides first reported by Brown.[5] This synthesis gave us good yields of a pure product.

Acknowledgment.—This work was performed as a part of a research project sponsored by The General Tire and Rubber Company of Akron, Ohio.

Experimental

A mixture of 216 g. (3 moles) of acrylic acid, 844 g. (6 moles) of benzoyl chloride, and 0.5 g. of hydroquinone was distilled at a fairly rapid rate through an efficient 25-cm. distilling column. The distillate was collected in a receiver containing half a gram of hydroquinone, immersed in ice. When the temperature at the top of the column, which remained between 60 and 70° for most of the distillation, had reached 85° the distillation was discontinued. The crude product, weighing between 215–225 g., was then redistilled through the same column and the fraction boiling at 72–74° at 740 mm. was collected. The weight of the final product was 185–195 g., or 68–72%.

(5) Brown, THIS JOURNAL, **60**, 1325 (1938).

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Preparation of 1-Phenyl-6-methylhendecane

BY WILLIAM E. TRUCE AND JOHN T. WISE

A pure sample of 1-phenyl-6-methylhendecane was desired for infrared and ultraviolet absorption studies. It was prepared by treating 1-phenyl-5-pentylmagnesium bromide with 2-heptanone, followed by dehydration and subsequent hydrogenation.

Experimental

1-Phenyl-5-pentanol[1] was prepared by treating 1-phenyl-3-propylmagnesium bromide with a two-fold excess[2] of ethylene oxide; yield 68%, b. p. 136° (5 mm.), $n^{20}D$ 1.5158.

1-Phenyl-5-bromopentane was prepared by treating the corresponding alcohol with anhydrous hydrogen bromide[3]; yield 80.3%, b. p. 144° (12 mm.), $n^{20}D$ 1.5332. *Anal.* Calcd. for $C_{11}H_{15}Br$: Br, 35.1. Found: Br, 35.1.

To the Grignard reagent prepared from 319.5 g. (1.40 m.) of 1-phenyl-5-bromopentane, 34.1 g. (1.40 m.) of magnesium and 600 ml. of ether, 159.6 g. (1.40 m.) of 2-heptanone was added over a period of five hours. After standing for thirty-six hours, the reaction mixture was hydrolyzed with cold, dilute hydrochloric acid. The alcohol was extracted with ether. After removing the ether, the crude product was refluxed for twenty hours with twice its volume of 90% formic acid.[4] The mixture was made alkaline with aqueous sodium hydroxide and the crude olefin(s) was extracted with ether. After removing the ether, the residue was distilled over sodium in an atmosphere of nitrogen; b. p. 158° (5 mm.), $n^{20}D$ 1.4979; 40.1% conversion based on 1-phenyl-5-bromopentane. The product gave positive tests for unsaturation with bromine and potassium permanganate. *Anal.* Calcd. for $C_{18}H_{28}$: C, 88.45; H, 11.55. Found: C, 88.9; H, 11.1.

A portion of the material (24.4 g.) was reduced practically quantitatively in the presence of Raney nickel catalyst in a Parr type hydrogenerator at 90–100° and 51 p. s. i. hydrogen pressure for four hours. The resulting hydrocarbon, 1-phenyl-6-methylhendecane, was filtered and distilled, b. p. 136–137° (1 mm.), $n^{20}D$ 1.4874. It gave negative tests for unsaturation with bromine and potassium permanganate. *Anal.* Calcd. for $C_{18}H_{30}$: C, 87.8; H, 12.2. Found: C, 88.0; H, 12.3.

Retention of the benzene ring was demonstrated by infrared analysis, and the ultraviolet absorption spectrum of the compound agreed well with that expected of a monoalkylbenzene.[5]

(1) v. Braun, *Ber.*, **44**, 2872 (1911).

(2) Huston and Langham, *J. Org. Chem.*, **12**, 90 (1947).

(3) v. Braun, Deutsch and Schmatloch, *Ber.*, **45**, 1258 (1912); "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 246.

(4) Soffer, Strauss, Trail and Sherk, THIS JOURNAL, **69**, 1684 (1947).

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Basic Ketals of Benzophenone

BY PRICE TRUITT AND W. D. COMPTON[1]

The recent success of Benadryl[2] as a potent histamine antagonist suggested the possibility that the structurally related diphenyl-di-(2-dialkylaminoethoxy)-methane might exhibit a similar potency.

Although Fourneau and Chantalou[3] have reported certain similar cyclic acetals, namely, 2-phenyl-4-dialkylaminomethyldioxalane-1,3, their method of synthesis was unsuccessful when applied to preparation of the compounds reported in this paper.

Even though other procedures gave some of the desired ketals, the preferred method of synthesis was by the addition of anhydrous potassium carbonate to a refluxing solution of diphenyldichloromethane and the appropriate 2-dialkylaminoethanol. The diphenyl-di-(2-dialkylaminoethoxy)-methanes prepared in this manner were very viscous liquids which hydrolyzed rapidly when in contact with diluted hydrochloric acid. Benzophenone was obtained from this hydrolysis. It was necessary to prepare the disuccinates or dimethiodides of these basic ketals in order to obtain pure crystalline products.

Neither of the disuccinate salts prepared in this work showed appreciable antihistamine activity.

Experimental[4]

The 2-dimethylaminoethanol, 2-diethylaminoethanol and 2-piperidinoethanol were obtained from Eastman Kodak Company and distilled before use.

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(2)(a) Parke, Davis & Co. Trade Mark. (b) Rieveschl and Huber, Paper 41, Division of Medicinal Chemistry, American Chemical Society Meeting, Atlantic City, 1946.

(3) Fourneau and Chantalou, *Bull. soc. chim.*, **12**, 845 (1945).

(4) Melting points were taken with a Fisher-Johns melting point apparatus.