mother liquors yielded a small quantity of dark oil which reacted with 3 ml. of 100% hydrazine hydrate in 2 ml. of ethanol. The mixture solidified on cooling. After repeated recrystallization from hot ethanol, *ca.* 1.0 g. of fluorenone was isolated as the hydrazone (m. p. 148–149°; mixed m. p. 148–149°).

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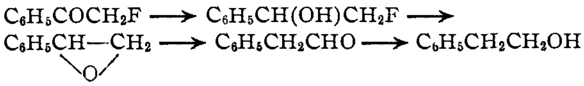
Reduction of Phenacyl Fluoride with Aluminum Isopropoxide[1]

BY WILLIAM E. TRUCE AND B. H. SACK

The original purpose of this investigation was to prepare ω-fluorostyrene by reduction of phenacyl fluoride with aluminum isopropoxide, followed by dehydration of the resulting fluorohydrin.

Phenacyl fluoride was prepared by acylating benzene with fluoroacetyl chloride[2] in the presence of aluminum chloride. After this portion of the work had been completed our attention was called to the publication of similar results.[3] However, the authors did not report the yield of phenacyl fluoride obtained nor that phenacyl chloride was an important by-product.

Phenacyl fluoride was treated with aluminum isopropoxide and isopropyl alcohol according to the procedure given for the reduction of benzophenone.[4] The product contained no fluorine. On the basis of previous results where halogen-free carbinols and ethers were obtained as reduction products of certain α-bromoketones,[4] 2-phenyl-1-ethanol was considered as a possible reduction product of phenacyl fluoride. The failure of the α-naphthylurethan of this alcohol to alter the melting point of the same derivative of an authentic sample of 2-phenyl-1-ethanol led to the conclusion that the reduction product was 2-phenyl-1-ethanol. It may have been formed by the following sequence of reactions[4]



Experimental

Preparation of Phenacyl Fluoride.—Into a mixture of 117 g. (1.50 moles) of benzene, 198 g. (1.50 moles) of technical anhydrous aluminum chloride, and 300 ml. of carbon disulfide, surrounded by ice-water, was added 114 g. (1.32 moles) of fluoroacetyl chloride (b. p. 71° (755 mm.), n^{27}D 1.3835). The addition was carried out dropwise with stirring over a period of five hours. The reaction mixture was allowed to warm up to room temperature and stirred for an additional four hours. The contents were poured into a mixture of ice and hydrochloric acid and then extracted twice with ether. This solution was treated with Drierite, the ether removed, and a vacuum rectification was carried out through a two-foot, helix-packed column. The yield of phenacyl fluoride was 46%, b. p. 94–95° (12 mm.), m. p. 27–28°.

Anal. Calcd. for C_8H_7FO: F, 13.77. Found: F, 13.78, 13.82; Cl, trace.

Phenacyl benzoate was prepared by refluxing a mixture of 1.0 g. of sodium benzoate, 5 cc. of water, 5 cc. of ethyl alcohol and 1.0 g. of phenacyl fluoride, for six hours. After chilling overnight an oil precipitated and after standing several days a small crop of long needles formed. These were recrystallized from 95% ethyl alcohol; m. p. 117–118° (lit. value 117–117.5°).[5]

The residue from the distillation of phenacyl fluoride amounted to 34 g. On recrystallization from methyl alcohol it melted at 56–57° and did not depress the melting point an authentic sample of phenacyl chloride.

Reduction of Phenacyl Fluoride.—Thirty-two grams (0.23 mole) of phenacyl fluoride was added to a mixture of 47 g. (0.23 mole) of aluminum isopropoxide in 235 cc. of isopropyl alcohol (distilled from calcium oxide) in a flask connected to a Hahn partial condenser. The solution was refluxed at such a rate that four to eight drops were distilled per minute. After thirteen hours the distillate showed a negative acetone test when treated with 2,4-dinitrophenylhydrazine reagent. The excess isopropyl alcohol was removed under reduced pressure. The residue was allowed to cool and was then hydrolyzed with cold, dilute hydrochloric acid (80 cc. of concd. acid and 400 cc. of water). The organic material was extracted with ether and, after removal of the ether, it was distilled, the material boiling at 97–102° (8 mm.) being collected; weight 16.3 g., $n^{26.8}$D 1.5238, d^{20}_4 1.024. The α-naphthyl urethan, prepared according to directions given by Shriner and Fuson,[6] melted at 116–117°. It did not depress the melting point of the α-naphthyl urethan of an authentic sample of 2-phenyl-1-ethanol.

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[1] Abstracted from the M.S. thesis of B. H. Sack.

[2] Truce, THIS JOURNAL, **70**, 2828 (1948).

[3] Gryszkiewicz-Trochimowski, Sporzynski and Wnuk, *Rec. trav. chim.*, **66**, 419 (1947).

[4] Wilds, "Organic Reactions," Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1944, Chap. 5.

[5] Zincke, *Ann.*, **216**, 308 (1882).

[6] Shriner and Fuson, "Identification of Organic Compounds," 3rd edition, John Wiley and Sons, New York, N. Y., 1940, p. 163.

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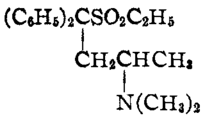
The Resolution of Ethyl 1,1-Diphenyl-3-dimethylaminobutyl Sulfone

BY B. F. TULLAR, W. WETTERAU AND S. ARCHER

In a previous paper[1] it was reported that the sulfone, I, exhibited strong analgesic action. For pharmacological reasons it became desirable to resolve this compound into its optical antipodes.



$$(C_6H_5)_2C(SO_2C_2H_5)CH_2CH(CH_3)N(CH_3)_2$$

I

The yield of ethyl benzohydryl sulfone was somewhat improved by using benzohydryl bromide rather than the chloride as the starting material. The sulfone was alkylated with dimethylaminoisopropyl chloride and sodium hydride rather than sodium amide as the condensing agent. The yield of pure I was 45% and about 29% of

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[1] Klenk, Suter and Archer, THIS JOURNAL, **70**, 3846 (1948).