

14,300); in ethanol it showed λ_{\max} 261 $m\mu$ (ϵ 16,300) and 230 $m\mu$ (shoulder).

Anal. Calcd for $C_{14}H_{17}N_3O_5$: C, 50.14; H, 5.11; N, 20.89. Found: C, 50.41; H, 5.38; N, 20.76.

9-(2'-Deoxy- β -D-ribofuranosyl)adenine (II).—In 20 ml of absolute methanol, 0.33 g of I was dissolved and 3.2 ml of a solution of barium methoxide in methanol (1 *N*) was added. The mixture was kept at room temperature overnight and refluxed for 40 min. The solution was neutralized with carbon dioxide. The precipitate was filtered off and the filtrate was concentrated under reduced pressure. Recrystallization of the residue from water gave II, mp 267–268°, $[\alpha]_{25}^{20} -16.5$ (*c* 0.53, water). Ultraviolet absorption at pH 1 showed λ_{\max} 258 $m\mu$ (ϵ 15,000); at pH 11 it showed λ_{\max} 261 $m\mu$ (ϵ 14,600); in ethanol it showed λ_{\max} 260.5 $m\mu$ (ϵ 16,000) and 231 $m\mu$ (shoulder).

Anal. Calcd for $C_{10}H_{13}N_5O_3$: C, 47.8; H, 5.2; N, 27.9. Found: C, 47.74; H, 5.45; N, 27.84.

The Reaction of Diphenylcyclopropanone with Alkaline Hydrogen Peroxide

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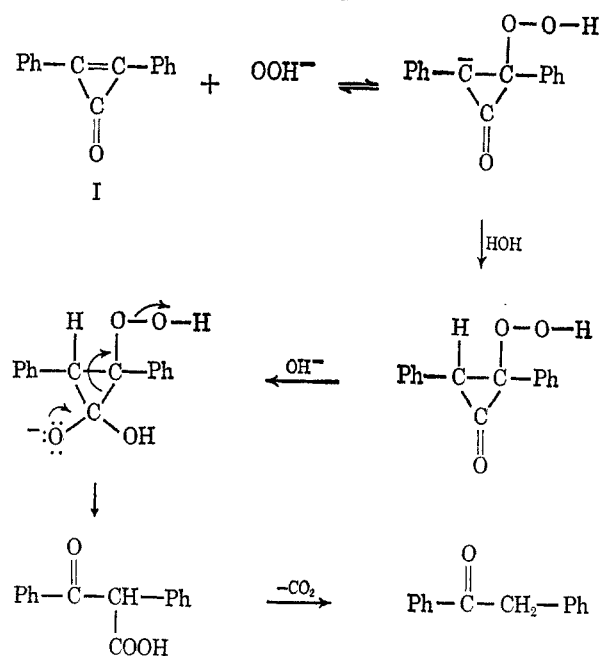
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Although the preparation of bicyclobutane or derivatives has been reported,² the heterocyclic analog, oxabicyclobutane, or its derivatives have never been made. Epoxidation of diphenylcyclopropanone (I) appeared to be a convenient method for the preparation of a derivative of oxabicyclobutane.

The reaction of diphenylcyclopropanone with hydrogen peroxide and sodium carbonate or sodium hydroxide afforded desoxybenzoin as the major product. A small amount of an intensely yellow, oily material, which was not identified, was also produced. Breslow, *et al.*,^{3,4} have found that I reacts with alcoholic sodium hydroxide to form *cis*-1,2-diphenylpropenoic acid, indicating an initial attack of hydroxide ion on the carbonyl carbon atom. It is postulated that the hydroperoxide ion, on the other hand, reacts at the carbon-carbon double bond, as in the case of an "ordinary" α,β -unsaturated carbonyl compound.⁵ However, the succeeding steps, in which ring closure to the epoxide is effected, could not take place because of unfavorable steric factors. Consequently, an alternate pathway is followed; one possible route is depicted in Scheme I.

Attempts were made to oxidize diphenylcyclopropanone with peroxyacetic acid but in each instance the starting material was recovered unchanged. Reaction of I with hydroxylamine resulted in the formation of desoxybenzoin oxime⁴ as one of the products, which points to the involvement of the carbon-carbon double bond, rather than the carbonyl group, in the first step. Attempts to oxidize I with hypochlorite ion (in aqueous dioxane) were unsuccessful, starting material

SCHEME I



being recovered each time. The hypochlorite-pyridine reagent⁶ was ineffective because of the rapidity of the reaction of I with the pyridine.⁴

Experimental Section

Reaction of Diphenylcyclopropanone with Alkaline Hydrogen Peroxide.—To a solution of 2 g (0.01 mole) of diphenylcyclopropanone⁴ in 25 ml of purified dioxane was added 4 ml of 3 *M* NaOH (or 10 ml of 5% Na_2CO_3). Two milliliters of 25% hydrogen peroxide was then added dropwise to the stirred mixture. The temperature of the reaction mixture was maintained at 25–30° during the addition and for 30 min thereafter. The addition of 60 ml of ice-water caused the separation of a yellow solid, which was filtered and allowed to air dry. The yield of the crude product was 1.5 g. Recrystallization from methanol afforded white crystals, mp 55–56°, whose infrared spectrum was identical with that of an authentic sample of desoxybenzoin.

The mother liquor from the recrystallization was evaporated and the residual solid was recrystallized again. After another such operation a yellow, oily residue (less than 0.5 g) remained.

(6) S. Marmor, *J. Org. Chem.*, **28**, 250 (1963).

Preparation of Ethyl α -Aryloxyacetoacetates and the Decomposition of Ethyl α -(4-Acetylphenoxy)acetoacetate

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Several ethyl α -aryloxyacetoacetates have been described in the literature as intermediates in the preparation of benzofurans and naphthofurans.¹ Among the analogous compounds (1a to 1f) prepared in this laboratory was ethyl α -(4-acetylphenoxy)acetoacetate (1c). It was observed that this compound, originally ob-

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(2) K. B. Wiberg and R. P. Ciula, *J. Am. Chem. Soc.*, **81**, 5261 (1959); W. R. Moore, H. R. Ward, and R. F. Merritt, *ibid.*, **83**, 2019 (1961); D. M. Lemal, F. Menger, and G. W. Clark, *ibid.*, **85**, 2529 (1963).

(3) R. Breslow and R. Peterson, *ibid.*, **82**, 4426 (1960).

(4) R. Breslow, T. Eicher, A. Krebs, R. A. Peterson, and J. Posner, *ibid.*, **87**, 1320 (1965).

(5) C. Bunton and G. Minkoff, *J. Chem. Soc.*, 665 (1949).

(1) (a) A. Hantzsch, *Ber.*, **19**, 1290, 1298, 1301 (1886); (b) R. Royer and E. Bisagni, *Bull. Soc. Chim. France*, 526 (1959); (c) W. R. Boehme, *Org. Syn.*, **33**, 43 (1953).