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AN IMPROVED SYNTHESIS OF 2-ETHYL-2-PHENYL-GLUTACONIMIDE

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JAMES A. MOORE

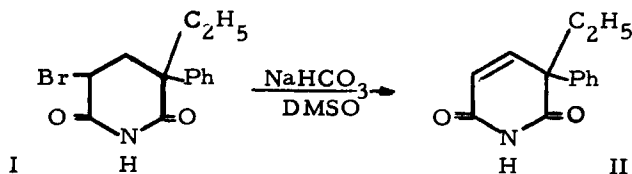
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AN IMPROVED SYNTHESIS OF 2-ETHYL-2-PHENYL-GLUTACONIMIDE

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It has been found that II can be obtained by dehydrohalogenation of I using sodium bicarbonate in dimethylsulfoxide in almost quantitative yield, making this procedure superior to the one previously reported (refluxing in collidine, 74% crude yield).¹



EXPERIMENTAL

A Finnigan Model 1015 SL quadrupole instrument was used. The conditions at which the mass spectrum was taken were: electron energy, 70 eV; emission current 200 μ A; high voltage 3KV; sensitivity, 10^{-7} ; scan time, 10 seconds.

A solution of 296 mg (1 mmole) of I (prepared according to the procedure of Urech *et al.*¹ and 300 mg of sodium bicarbonate in 2 ml of dimethylsulfoxide was heated at 80° for 1 hr. The solution was cooled and diluted with 100 ml of water, then extracted with methylene chloride (2 x 50 ml). The combined extracts were washed once with 100 ml of water, dried (MgSO_4), filtered, and the solvent was removed under reduced pressure to give 120 mg (96%) of II after recrystallization from ethanol; mp. 165-7°; lit.¹ mp. 163-5° (from acetone-petroleum ether); ir (KBr) $\bar{\nu}_{\text{max}}$ cm^{-1} : 3200 (NH), 1710 (C=O imide), 1630 (C=C); NMR (CDCl_3) δ 0.93 (t, 3H, $\text{CH}_3\text{-CH}_2$), 1.70-2.80 (2H, $\text{CH}_3\text{-CH}_2$, two quartets because of the asymmetric center at C_2 and vinylic coupling), 6.37 (doublet of doublets, 1H, vinylic proton at C_4), 6.8 (d, 1H, vinylic proton at C_5) and 7.03 (s, 5H, aromatic protons); mass spectrum m/e (relative intensity) 215, M^+ (5.2), 187, $\text{M-C}_2\text{H}_4$ (47.3), 172, M-NHCO (40.8), 158 (40.8), 157 (100).

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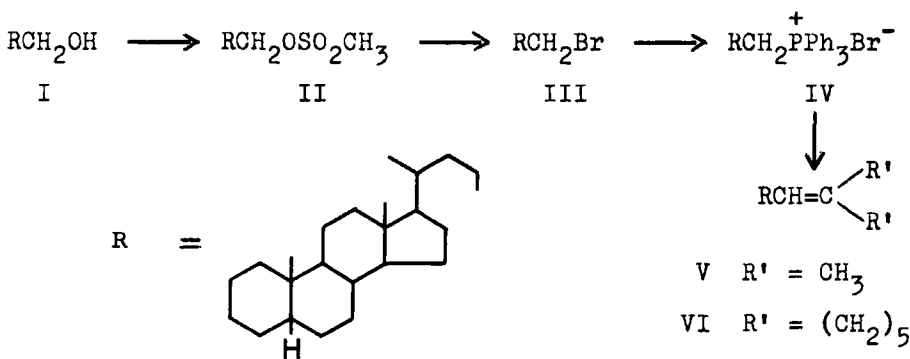
THE SYNTHESIS OF STEROLS WITH MODIFIED
SIDE CHAINS BY THE WITTIG REACTION

Submitted by J. E. Herz and S. Cruz M.
(9/4/74)

Departamento de Quimica
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Mexico, D. F., MEXICO

The detailed preparation of two steroidal triphenyl phosphonium salts and their reactions with several ketones¹ is described.

SCHEME I



EXPERIMENTAL

Melting points were determined on a Kofler Hotstage and are uncorrected. Infrared spectra were measured on a Perkin-Elmer Model 421 in chloroform. NMR spectra were determined in deuteriochloroform on a Varian A-60 spectrometer. Rotations