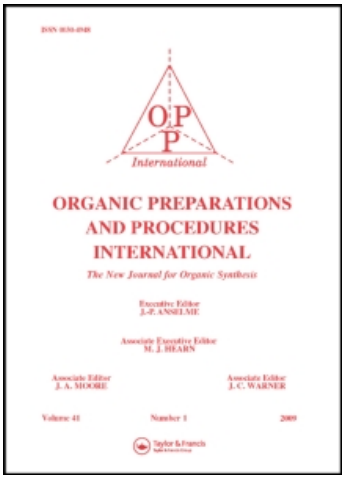

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AN IMPROVED SYNTHESIS OF 2-ETHYL-2-PHENYL-GLUTACONIMIDE

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

Submitted by (4/16/74) Hassan Y. Aboul-Enein

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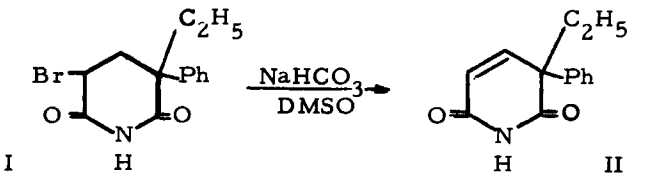
It has been found that II can be obtained by dehydrohalogenation of I using sodium bicarbonate in dimethylsulfoxide in almost quantitative yield, making this procedure superior to the one previously reported (refluxing in collidine, 74% crude yield).[1]



I II

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 μA; 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) $\bar{\nu}_{max}$ cm^{-1}: 3200 (NH), 1710 (C=O imide), 1630 (C=C); NMR ($CDCl_3$) δ 0.93 (t,3H,$\underline{CH_3}$-CH_2), 1.70-2.80 (2H,CH_3-$\underline{CH_2}$, 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, M-C_2H_4 (47.3), 172, M-NHCO (40.8), 158 (40.8), 157 (100).

ACKNOWLEDGMENT. - The author thanks Mr. Dennis Charkowski for his technical assistance with the mass spectrum. This work was supported by U. S. Public Health Service, Grant GM 12675.

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

Submitted by (9/4/74)

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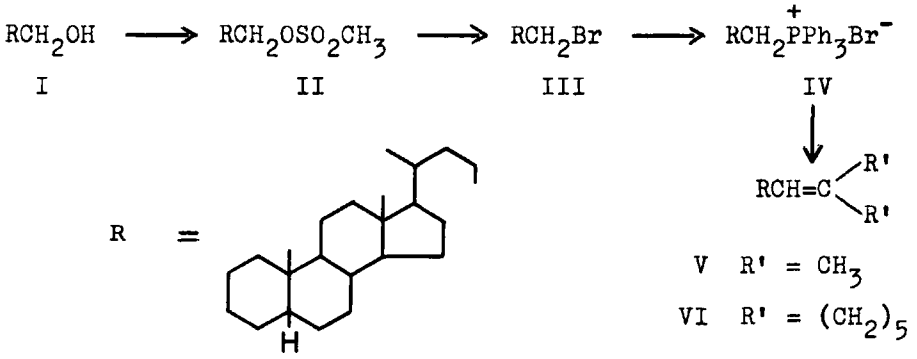
The detailed preparation of two steroidal triphenyl phosphonium salts and their reactions with several ketones[1] is described.

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 deuterochloroform on a Varian A-60 spectrometer. Rotations