NEW SYNTHESIS OF INDOLIZINEDIONE FROM β-ALANINE

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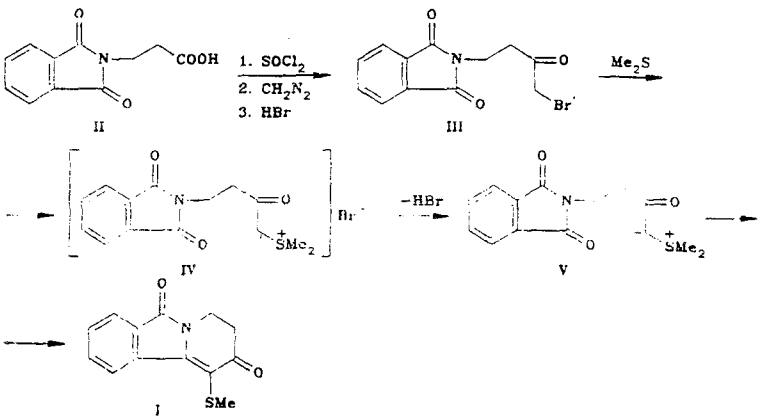
Recently the indclizine structure has been prepared by an intramolecular Witting reaction [1]. We have now synthesized the indolizinedione (I) via intramolecular cyclization of the keto stabilized sulfur ylid (V) by heating in toluene. The ylid was prepared by the following reactions from N-phthalyl-β-alanine (II).



1-Bromo-4-phthalimido-2-butanone (III). Yield 70%, mp 114°C (ether–hexane). IR spectrum (Nujol): 1770, 1730, 1705 (C=O), 1605 cm^{-1}.

^1H NMR spectrum ($CDCl_3$): 3.14 (2H, t, J = 7 Hz, CH_2CO), 3.95 (2H, t, J = 7 Hz, CH_2N), 4.27 (2H, s, CH_2Br), 7.84 ppm (4H, s, C_6H_4).

Dimethyl(2-oxo-4-phthalimidobutyl)sulfonium Bromide (IV). Yield 87%, mp 119-120°C (acetone). IR spectrum (Nujol): 1770, 1710, 1700 (C=O), 1610 cm^{-1}. ^1H NMR spectrum (CF_3COOH): 2.67 [6H, s, $(CH_3)_2S$], 2.90 (2H, t, J = 7 Hz, CH_2CO), 3.81 (2H, t, J = 7 Hz, CH_2N), 4.52 (2H, s, CH_2S), 7.52 ppm (4H, s, C_6H_4).

1-Dimethylsulfuranylidene-4-phthalimido-2-butanone (V). Yield 95%, mp 145°C (decomp.). IR spectrum (Nujol): 1770, 1705 (C=O), 1610, 1540 cm^{-1} (C=O). ^1H NMR spectrum ($CDCl_3$): 2.47 (2H, t, J = 7 Hz, CH_2CO), 2.86 [6H, s, $(CH_3)_2S$], 3.59 (1H, s, CH), 3.97 (2H, t, J = 7 Hz, CH_2N), 7.83 ppm (4H, m, C_6H_4).

1-Methylthio-3,4-dihydropyridino[2,1-a]isoindol-2,6-dione (I). Yield 65%, mp 164-165°C (acetone). IR spectrum (Nujol): 1770, 1712 (C=O), 1664, 1572 cm^{-1}. ^1H NMR spectrum ($CDCl_3$): 2.39 (3H, s, CH_3S), 2.86 (2H, t, J = 7 Hz, CH_2CO), 4.14 (2H, t, J = 7 Hz, CH_2N), 7.68 and 8.90 ppm (4H, M, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$): 17.76 (q, C_{11}), 35.97 (t, C_3), 36.82 (t, C_4), 113.38 (s, C_1), 123.83 (d, C_{10}), 127.55 (d, C_7), 129.83 (s, C_{10b}), 131.92 (d, C_8), 133.04 (d, C_9), 134.47 (s, C_{10a}), 151.11 (s, C_{6a}), 165.35 (s, C_6), 190.87 ppm (s, C_2). M^+ 245.

Elemental analytical data for I and III-V agreed with that calculated.

LITERATURE CITED

1. W. Flitsch and K. Pandl, Annalen, No. 8, 649 (1987).

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