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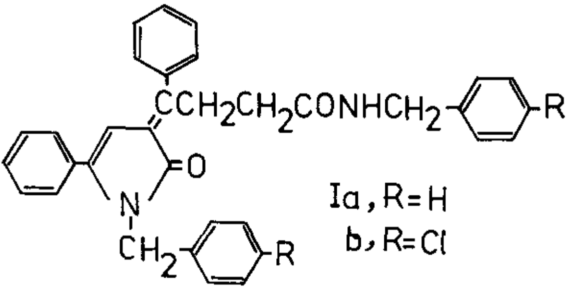
PREPARATION OF NEW 3-SUBSTITUTED N-BENZYL-5-PHENYLPYRROLIN-2-ONES

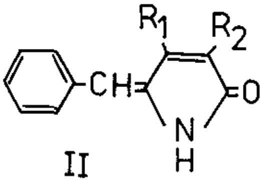
Ali Abdul Hussain Saeed

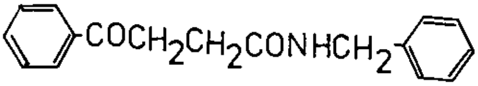
Department of Chemistry, College of Science, University of Basrah,
Basrah, Iraq

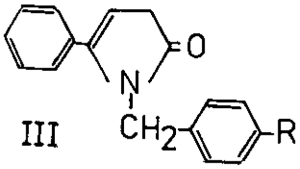
Abstract —— Amination of γ-benzoylpropionic acid by benzyl and p-chlorobenzylamine gave new 3-substituted N-benzyl-5-phenylpyrroline-2-ones in high yield. The products were identified by nmr, ir, mass, and uv-visible spectroscopy.

It was reported that from the condensation between β-dicarbonyls and α-aminoketone or ethyl oximinoacetoacetate[1] tetrasubstituted pyrroles were obtained. 2,5,N-Trisubstituted pyrroles were obtained from the condensation of ammonia, hydrazine, hydroxylamine or primary amines with α-diketones[2-5] or from cyclization of acylsuccinic esters with ammonia or primary amines[6]. Whereas, the condensation between γ-benzoylpropionic acid and aniline, o-toluidene or cyclododecaylamine gave N-substituted γ-benzoylpropionamide[7]. In this work we have obtained an orange product, 3-substituted N-benzyl-5-phenylpyrrolin-2-one I, by refluxing for 2 h a mixture of equimolar amounts of γ-benzoylpropionic acid and benzyl or p-chlorobenzylamines. Our condensation is basically akin to the preparation of the orange product II from aldehydes or ketones with 3,4-disubstituted pyrrolin-2-one[8]. Our reaction may occur through formation of 3,4-unsubstituted 5-phenyl-N-benzylpyrrolin-2-one III and N-benzyl-γ-benzoylpropionamide IV as intermediates, then the two moieties are reacted to give I. In a separate experiment, IV has been isolated and identified, but unfortunatelly we were unable to isolate III [Ia; 95% yield, mp 160-161°C, ir (nujol mull) cm^{-1} 3550-3310 (N-H stretching),


Ia, R=H
b, R=Cl

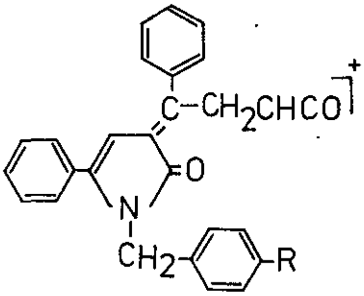

II


IVa, R=H; R=Cl, IVb

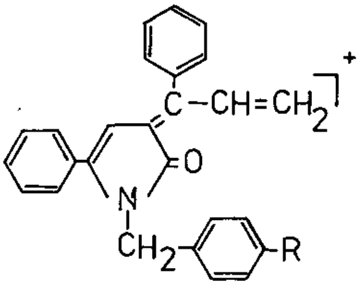

III

R=H or Cl

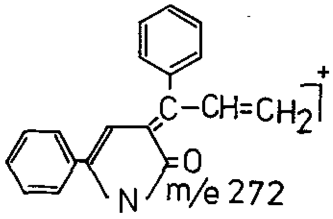
1960-1670 (pyrrolin C=O), 1650-1615 (secondary amide C=O), 1610-1585 (phenyl and N-H bending), nmr (d_6-DMSO) δ 7.04-7.44 (phenyls protons), 3.30 (benzyl $-CH_2-$ protons), 11.39 (N-H proton), 2.24-2.49 triplet ($-CH_2-$ attached to carbonyl), 4.17-4.82 triplet ($-CH_2-$ attached to unsaturated carbon), 5.62 singlet (pyrroline ring proton), [λ_{max}^{EtOH} nm 400, 280, 261, 210]. Anal. Calcd for $C_{34}H_{30}N_2O_2$: C,81.93; H,6.02; N,5.62. Found: C,81.14; H,6.04; N,5.59 [Ib; 90% yield, mp 200-210°C, ir (nujol mull) cm^{-1} 3530-3350 (N-H stretching), 1680 (pyrrolin C=O), 1650-1620 (secondary amide C=O), 1610-1590 (phenyl and N-H bending), nmr ($CDCl_3$) δ 7.06-8.17 (phenyls protons), 3.61 (benzyl $-CH_2-$ protons), 10.79 (N-H protons), 2.39-2.56 triplet ($-CH_2-$ attached to carbonyl), 4.00-4.27 triplet ($-CH_2-$ attached to unsaturated carbon), 5.62 singlet (pyrrolin ring proton), [$\lambda_{max}^{CH_2Cl_2}$ nm 525, 402, 270, 232]. Anal. Calcd for $C_{34}H_{28}N_2Cl_2O_2$: C,71.96; H,4.94; N,4.94; Cl, 12.52. Found: C,71.98; H,4.97; N,4.96; Cl, 12.44 [IVb; 95% yield, mp 148-149°C, ir (nujol mull) cm^{-1} 3230 (N-H stretching), 1670 (benzoyl C=O), 1615 (amide C=O), 1585 (phenyl and/or N-H bending), [λ_{max}^{EtOH} nm 498, 481, 279, 246]. Anal. Calcd for $C_{17}H_{16}NClO_2$: C,67.66; H,5.31; N,4.64; Cl,11.78. Found: C,67.65; H, 5.32; N,4.62; Cl,11.85. The mass spectra of Ia and Ib show a base peak at m/e 91 and 125 which represent the benzyl and p-chlorobenzyl fragment ions, and peaks at high m/e values for the fragment ions V, VI, and VII. The presence of these fragment ions confirms our suggested structure I. Other peaks are at m/e 409($M^{\cdot+}$-89,1%) and at 443($M^{\cdot+}$-123,66%), and only the spectrum of Ia exhibited a molecular ion peak at m/e 498(39%). The mass spectrum of IVb exhibited a base peak at m/e 140, strong peaks at m/e 125, 105, and 77 for the fragment ions p-chlorobenzyl (34%), benzoyl (36%), and phenyl (28%), respectively, less intense peaks at m/e 301 (molecular ion), and at 283(11%) for the fragment ion VIII ($M^{\cdot+}-H_2O$ molecule). The presence of the latter fragment ion in the mass spectrum of IVb confirms our suggested mechanism in the formation of the moiety III intermediate which leads finally to I. The uv-visible spectra of Ia and Ib in



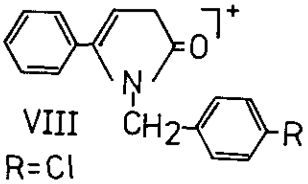
Va; R= H, m/e 391(22%)
b; R=Cl, m/e 425(21%)



VIa; R=H, m/e 363(31%)
b; R=Cl, m/e 397(60%)



m/e 272
Ia(17%); Ib(73%)



VIII
R=Cl
m/e 283(11%)

ethanol and methylene chloride, respectively, showed long wavelength bands at 400, 525 nm which indicate that I is highly conjugated compound. Measurements of the elemental analysis, ir, nmr, uv-visible, and mass spectra have previously described[5,9,10].

REFERENCES

1. A. H. Jackson, 'Comprehensive Organic Chemistry of Heterocyclic Compounds', eds. by P. G. Sammes, Pergamon Press, 1979, p. 296.
2. H. S. Broadbent, W. S. Burham, R. K. Olsen, and R. M. Sheeley, J. Heterocycl. Chem., 1968, 5, 757.
3. R. Ramasseul and A. Rassat, Bull. Soc. Chim. France , 1970, 4330.
4. W. Keller-Schierlein, M. L. Mihailovic, and V. Prelog, Helv. Chim. Acta, 1958, 41, 220.
5. A. A. H. Saeed, J. Chem. Eng. Data, 1985, in press.
6. M. Pesson, D. Humbert, M. Dursin, and H. Tecker, Compt. Rend. (C), 1971, 272, 478.
7. A. A. H. Saeed, Iraqi J. Sci., 1982, 23, 42.
8. H. Plieninger and M. Decker, Annalen., 1956, 598, 198.
9. A. A. H. Saeed, J. Heterocycl. Chem., 1982, 19, 113.
10. A. A. H. Saeed and E. K. Ebraheem, J. Heterocycl. Chem., 1983, 20, 1739.

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