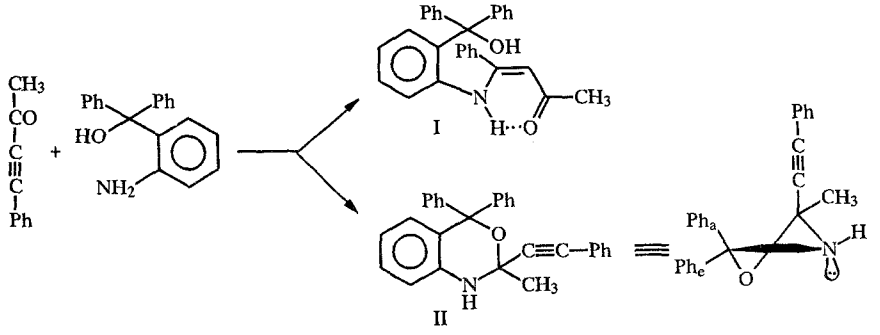
Chemistry of Heterocyclic Compounds, Vol. 29, No. 10, 1993

REACTION OF o-AMINOPHENYLDIPHENYLMETHANOL WITH α-ACETYLENIC KETONE

T. P. Kosulina, E. V. Gromachevskaya, and V. G. Kul'nevich

A reaction of acetylenic ketones having an activated triple bond with o-aminophenyldiphenylmethanol has been carried out for the first time in order to synthesize new unsaturated ketones and heterocyclic compounds containing a triple bond in the side chain, which show promise as antimycotics.

We found that the reaction of o-aminophenyldiphenylmethanol with methyl phenylethynyl ketone in an acetic acid medium proceeds by two paths: besides the known addition of N,O-dinucleophile to the triple bond without involving the hydroxyl group, with the formation of ketone (I) [1], a condensation takes place at the carbonyl group with the retention of the acetylenic fragment and obtainment of a substituted 1,2-dihydro-4H-3,1-benzoxazine (II) [2].



The reaction is carried out using an equimolar ratio of the reagents, in acetic acid at a temperature of 5°C for 2 h. After the precipitation of simultaneously formed products I and II by aqueous alcohol, their separation is carried out on a column filled with 40/100 type silica gel, eluting with benzene. The yields of compounds I and II are 55 and 25%, respectively.

[2-(2-Acetyl-1-phenylethenylamino)phenyl]diphenylmethanol (I, $C_{29}H_{25}NO_2$), mp 180-182°C; R_f = 0.17. IR spectrum: 3320 (NH); 3200 (OH); 3030, 1580, 1530 (C=CH_{arom}); 1620 (C=C); 1680 cm^{-1} (C=O). PMR spectrum (in CD_2Cl_2, δ): 1.53 (3H, s, CH_3); 5.30 (1H, s, OH); 6.60-7.82 (5H, m, C_6H_4, =CH); 7.20 (15H, s, Ph x 3); 8.62 ppm (1H, br.s, NH).

2-Methyl-2-phenylethynyl-4,4-diphenyl-1,2-dihydro-4H-3,1-benzoxazine II, $C_{29}H_{23}NO$, mp 58-59°C; R_f = 0.82. IR spectrum: 1040, 1080, 1120 (O−C−N), 3330 (NH), 2200 (C≡C), 3130, 3030, 1590, 1580 cm^{-1} (C=CH). PMR spectrum (in $CDCl_3$, δ): 2.04 (3H, s, CH_3), 4.30 (1H, br.s., NH); 6.56 (4H, m, C_6H_4); 7.02 (5H, s, Ph); 7.14 (5H, s, Ph_a); 7.20 ppm (5H, s, Ph_e), comp. [3].

REFERENCES

1. A. S. Nakhmanovich and V. N. Elokhina, Khim. Geterotsikl. Soedin., No. 3, 291 (1987).
2. E. V. Gromachevskaya, V. G. Kul'nevich, T. P. Kosulina, and V. S. Pustovarov, Khim. Geterotsikl. Soedin., No. 6, 842 (1988).

Krasnodar Polytechnical Institute, Krasnodar 350072. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 10, p. 1432, October, 1993. Original article submitted September 5, 1993.

 0009-3122/93/2910-1232$12.50 © 1994 Plenum Publishing Corporation

3. E. V. Gromachevskaya, T. P. Kosulina, V. G. Kul'nevich, Yu. Yu. Samitov, A. I. Khoyarov, and V. T. Dubonosov, Khim. Geterotsikl. Soedin., No. 1, 101 (1990).