Asian Journal of Chemistry Vol. 18, No. 2 (2006), 1586–1588

NOTE

Synthesis and Characterization of 3-(2-Hydroxy-3,4-benzophenyl)-5-aryl-substituted-pyrazolines

V.S. JAMODE† and S.E. BHANDARKAR*

Department of Chemistry

J. Darda Institute of Engineering Technology, Lohara, Yavatmal-445 001, India

E-mail: subodhb@rediffmail.com

1-(2-Hydroxy-3,4-benzophenyl)-3-aryl-prop-2-ene-1-ones (0.01 mol) (**3a–d**) and semicarbazide/thiosemicarbazide/isonicotinic acid hydrazide (0.01 mol) were added to DMF (20 mL) and the mixture was refluxed for about 2 h. The reaction mixture was cooled and diluted with water. The semisolid so obtained was triturated with ethanol to get a solid which was recrystallized from ethanol-acetic acid mixture to obtain titled pyrazolines

Key Words: Synthesis, Substituted pyrazolines.

Pyrazolines are known to possess fungicidal[1], bactericidal[2], insecticidal[3], analgesic[4], antipyretic[5] and antiinflammatory[6] properties. Several pyrazolines are found important as pharmaceuticals. Pyrazoline derivatives acquire anti-implantation[7] and cerebroprotective[8] activity. Due to this vital biological role of pyrazoline derivatives[9–14], it was thought of interest to synthesize the titled pyrazolines.

It has been observed that substituted chalcones are the best starting compounds for the preparation of the substituted pyrazolines. The present work deals with the synthesis of some new pyrazolines and their characterization by spectral analysis (IR, ^{1}H NMR).

All melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. Thin layer chromatography on silica gel-G was used to check the purity of the compounds. ^{1}H NMR spectra were recorded on a Bruker AC300 FNMR spectrometer (300 MHz), using TMS as an internal standard. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer. Microanalysis of nitrogen was obtained on Colman 29-N analyzer.

Preparation of 2-acetyl-1-naphthol (2) from modified Nenchi's Method

In hot glacial acetic acid (80 mL), fused $ZnCl_2$ (50 g) was added and refluxed till dissolved, then powdered 1-naphthol (30 g) was added and the mixture was refluxed for about 8 h. The reaction mixture was cooled and poured in acidulated water. The solid obtained was filtered, washed with water and recrystalized from rectified spirit to obtain compounds (**2**). Physical data of the compounds is given in Table-1.

Preparation of 1-(2-hydroxy-3,4-benzophenyl)-3-aryl-prop-2-ene-1-ones (3a–d)

2-Acetyl-1-naphthol (0.01 mol) and aromatic aldehyde (0.02 mol) were added in

†Vice-Chancellor, Sant Gadge Baba Amravati University, Amravati-444 602, India.

ethanol solvent (20 mL). To this mixture KOH (10%, 10 mL) solution was added dropwise with constant stirring. The reaction mixture was kept overnight. Then this mixture was poured over crushed ice and a little HCl. The product was filtered and recrystallized from ethanol to obtain 1-(2-hydroxy-3,4-benzophenyl)-3-aryl-prop-2-ene-1-ones (**3a–d**). Their physical data is given in Table-1.

TABLE-1
PHYSICAL DATA OF SYNTHESIZED COMPOUNDS

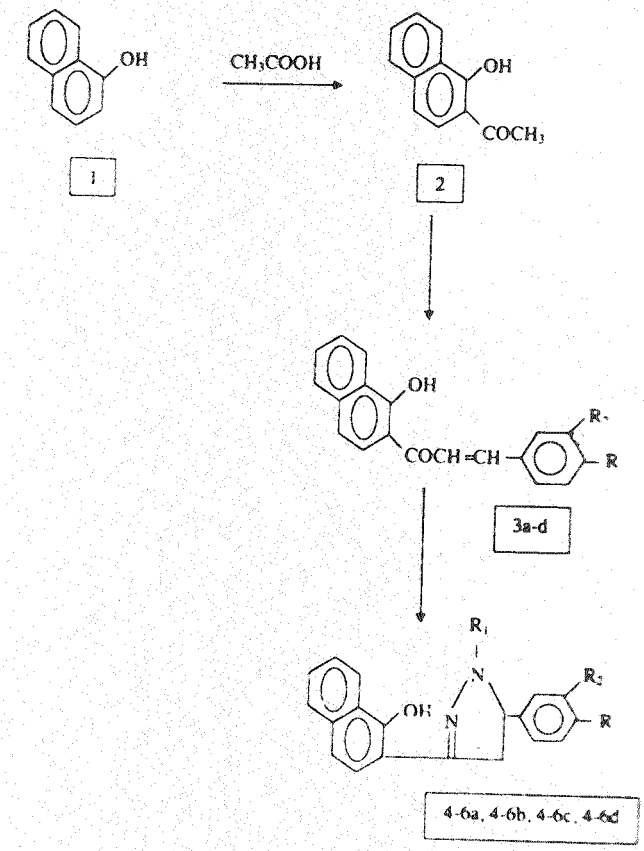
Compd	R	R_1	R_2	m.p. (°C)	Yield (%)	% N		% S		R_f value
						Found	Calcd.	Found	Calcd.	
2	—	—	—	98	72	—	—	—	—	—
3a	H	—	H	122	62	—	—	—	—	—
3b	OCH_3	—	H	144	68	—	—	—	—	—
3c	OH	—	H	146	63	—	—	—	—	—
3d	OCH_3	—	OCH_3	130	65	—	—	—	—	—
4a	H	$CONH_2$	H	211	56	14.43	14.53	—	—	0.63
5a	H	$CSNH_2$	H	190	53	12.01	12.10	9.28	9.22	0.66
6a	H	C_5H_4NCO	H	187	48	10.55	10.69	—	—	0.59
4b	OCH_3	$CONH_2$	H	195	61	11.69	11.63	—	—	0.68
5b	OCH_3	$CSNH_2$	H	165	55	11.22	11.14	8.38	8.49	0.52
6b	OCH_3	C_5H_4NCO	H	181	49	9.86	9.93	—	—	0.56
4c	OH	$CONH_2$	H	301	59	12.22	12.10	—	—	0.57
5c	OH	$CSNH_2$	H	175	47	11.47	11.57	8.71	8.82	0.58
6c	OH	C_5H_4NCO	H	188	46	10.18	10.27	—	—	0.62
4d	OCH_3	$CONH_2$	OCH_3	274	62	10.86	10.74	—	—	0.56
5d	OCH_3	$CSNH_2$	OCH_3	181	56	10.25	10.32	7.78	7.86	0.59
6d	OCH_3	C_5H_4NCO	OCH_3	179	52	9.17	9.27	—	—	0.68

Spectral interpretation of (3a): IR (ν_{max}) (cm^{-1}): 3250 ν(OH), 1685 ν(C═O), 1632 ν(C═C), 1275 ν(C—O); ^{1}H NMR (δ ppm): 7.25–8.70 (m, 11Ar—H and —CH═CH—), 13.80 (s, 1H, OH).

Preparation of 3-(2-hydroxy-3,4-benzophenyl)-5-aryl-1-carboxamido/1-thiosemicarboxamido/1-isonicotinoyl-pyrazolines (4–6a , 4–6b, 4–6c, 4–6d)

1-(2-Hydroxy-3,4-benzophenyl)-3-aryl-prop-2-ene-1-ones (0.01 mole) (3a–d) and semicarbazide/thiosemicarbazide/isonicotinic acid hydrazide (0.01 mole) were added to DMF (20 mL) and refluxed for 2 h. The cooled reaction mixture was diluted with water and the semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol-acetic acid mixture to get titled pyrazolines in 42–72% yield and their physical data is given in Table-1.

Spectral interpretation of (4a): IR (ν_{max}) (cm^{-1}): 3340 ν(OH), 3237 ν(NH_2), 1652 ν(C═N), 1374 ν(C—O), ^{1}H NMR (δ ppm); 3.410–3.486 (dd, 1H, H_A), J_{AB} = 17.8 Hz, J_{AX} = 4.8 Hz, 3.959–4.048 (dd, 1H, H_B) J_{AB} = 17.8 H, J_{BX} = 1.7 Hz, 5.529–5.584 (dd, 1H, H_X), J_{AX} = 4.8 Hz, J_{BX} = 11.7 Hz, 7.21–7.78 (m, 11Ar—H), 11.14 (s, 1H, OH), 6.80 (s, 2H, NH_2).



Scheme

R = H, OCH_3, OH; $R_1 = CONH_2$, $CSNH_2$, C_6H_5NCO,; R_2 = H, OCH_3.

REFERENCES

1. N.B. Das and A.S. Mitra, *Indian J. Chem.*, **16B**, 638 (1978).
2. H.Z. Katri and S.A. Vunii, *J. Indian Chem. Soc.*, **58**, 168 (1981).
3. S.G. Roelofan, C. Arnold and Wellmgak, *J. Agric. Food Chem.*, **27**, 406 (1979).
4. M.A. Metwally, M.Y. Yusuf, A.M. Tsmaiel and F.A. Amer, *J. Indian Chem. Soc.*, **62**, 54 (1985).
5. V.G. Vomin, Z.I. Sharmova, S.Ya. Shachilova, L.D. Kulikova and A.S. Zaks, *Khim. Farm. Zh.*, **19**, 1208, (1985); *Chem. Abstr.*, **104**, 61668m (1986).
6. J.P. Dusza, J.P. Josheph and S. Burnstun, US. US4 360 680 (CI 548 362 CO7D, 231/06) (1982).
7. Y.U.M. Batulin, *Chem. Abstr.*, **70**, 2236a (1969).
8. G.E.H. Elgemeie, A.M.E. Attia, D.S. Farag and S.M. Sherif, *J. Chem. Soc. Perkin Trans.*, **1**, 1285 (1994).
9. Y.J. Fernandes and H. Parekh, *J. Indian Chem. Soc.*, **74**, 59 (1997).
10. S.S. Ganguli, M.S. Vadaria and A.R. Parikh, *J. Inst. Chem. (India)*, **68**, 1920 (1996).
11. J. Upadhyay, U. Dave and H. Parekh, *J. Indian Chem. Soc.*, **68**, 4135 (1991).
12. P. Patel, S. Koregaonkar, M. Shah and H. Parekh, *IL Farmaco*, **51**, 59 (1996).
13. V.S. Jamode and B.S. Kakde, *Indian J. Chem.*, **17B**, 622 (1979).
14. El-Taher Z. Sarhan, S.A. Mohamed, A.N. Mikhael and Alexandria, *J. Pharm. Sci.*, **7**, 41 (1993); *Chem. Abstr.*, **122**, 81233r (1995).

(*Received*: 3 December 2005; *Accepted*: 31 December 2005) AJC-4627