Asian Journal of Chemistry Vol. 11, No. 4 (1999), 1522–1524

NOTE

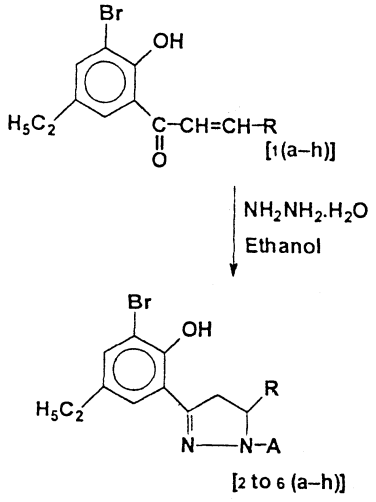
Synthesis of Some New Pyrazoline Derivatives and Related Compounds from Chalcones and their Antibacterial Activity

V.R. NAIK* and H.B. NAIK
Department of Chemistry
South Gujarat University, Surat-395 007, India

Chalcones on condensation with hydrazine hydrate in ethanol gave 2-pyrazolines which were converted to acetyl, benzoyl, *p*-acetanilide, sulphonyl and nitroso derivatives. These products have been characterised by spectral studies and screened for antibacterial activity.

Pyrazolines are important nitrogen containing heterocycles possessing diverse biological activity[1]. Some pyrazoline derivatives have shown considerable promise as chemotherapeutic agents[2]. Several workers have reported the synthesis of 2-pyrazolines by the reaction of alkyl and aryl hydrazines with α : β-unsaturated carbonyl compounds under variety of experimental conditions[3].

In the present work, 2′-hydroxy-3′-bromo-5′-ethylchalcones (1) have been reacted with hydrazine hydrate in ethanol to give 1-H-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines[4] (2). The reaction of (2) with acetic acid gave acetyl derivatives (3); similarly the reaction of (2) with benzoyl chloride gave benzoyl derivatives (4). Further, the reaction of the pyrazolines (2) with *p*-acetanilide sulphonyl chloride and sodium nitrite gave the corresponding sulphonamide derivatives (5) and nitroso derivatives (6).



For correspondence: Viral R. Naik, Brahmin Falia, At. and Po. Mandir, Via Vedchha, Tal. Jalapore, Distt:- Navsari-396 472, Gujrat, India.

2: A = —H

3: A = —$COCH_3$

4: A = —COC_6H_5

5: A = —SO_2—  —$NHCOCH_3$

6: A = —N=O

R = a: phenyl

b: 4-chlorophenyl

c: 2-hydroxyphenyl

d: 4-hydroxy phenyl

e: 4-methyl phenyl

f: 3-nitro phenyl

g: 4-methoxy phenyl

h: 4-N,N-dimethylamino phenyl

Antibacterial screenings of synthesised compounds have been carried out by cup-plate method[5]. Microorganisms employed were *Staphylococcus aureus* and *Escherichia coli.* The results were compared against tetracycline and gentamycine. All the compounds show the activity mild to moderate.

All the melting points were taken in open capillary tubes and are uncorrected. IR spectra were recorded on a Perkin-Elmer 377 spectrophotometer. All compounds gave satisfactory elemental analysis.

Preparation of 1-H-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines [2(a–h)]

A mixture of 2′-hydroxy-3′-bromo-5′-ethylchalcone (0.01 mol) and 99% hydrazine hydrate (0.015 mol) in ethanol (50 mL) was refluxed on a water-bath at 70–80°C gently for 2 h. The mixture was concentrated and allowed to cool. The resulting solid was washed with ethanol and crystallised from ethanol to obtain pyrazolines (2).

m.p. (°C;): 2a: 85; 2b: 120; 2c: 65; 2d: 88; 2e: 101; 2f: 78; 2g: 110; 2h: 94.

IR (cm^{-1}): 640–620 ν(C—Br); 3480–3300 ν(OH); 1625–1610 ν(C=N); 1520–1510 ν(N—H); 1320–1280 ν(C—N).

Preparation of 1-acetyl-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines [3(a–h)]

A mixture of (2) (0.001 mol) and acetic acid (10 mL) was refluxed on a water-bath at 70–80°C gently for 2 h. It was then concentrated. On cooling the solid separated was washed with water, dried and crystallised from ethanol to give acetyl pyrazolines (3).

m.p. (°C;): 3a: 93; 3b: 80; 3c: 82; 3d: 130; 3e: 84; 3f: 105; 3g: 96; 3h: 120.

IR (cm^{-1}): 640–620 ν(C—Br); 3480–3300 ν(OH); 1625–1610 ν(C=N); 1320–1280 ν(C—N); 1680–1640 ν(C=O).

Preparation of 1-benzoyl-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines [4(a–h)]

A mixture of (2) (0.001 mol) and benzoyl chloride (0.0011 mol) was dissolved in dry pyridine (10 mL) and stirred at room temperature for 1 h. It was then treated with dil. hydrochloric acid (2 N). The solid obtained was filtered, washed with water and cold NaOH (2%), dried and crystallised from glacial acetic acid to give benzoyl pyrazolines (4).

m.p. (°C;): 4a: 94; 4b: 75; 4c: 97; 4d: 102; 4e: 109; 4f: 108; 4g: 98; 4h: 111.

IR (cm^{-1}): 640–620 ν(C—Br); 3480–3300 ν(OH); 1625–1610 ν(C=N); 1320–1280 ν(C—N); 1680–1640 ν(C=O).

Preparation of 1-*p*-acetanilide sulphonyl-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines [5(a–h)]

Compound (2) (0.001 mol) in pyridine (10 mL) was cooled in ice-bath and to it *p*-acetanilide sulphonyl chloride (0.0011 mol) was added. The mixture was stirred for 1 h at room temperature and it was then treated with cold dil. hydrochloric acid (2 N). The resulting solid was filtered, washed with water and crystallised from ethanol to give *p*-acetanilide sulphonyl pyrazolines (5).

m.p. (°C;): 5a: 101; 5b: 106; 5c: 120; 5d: 110; 5e: 117; 5f: 85; 5g: 114 ; 5h: 125.

IR (cm^{-1}): 640–620 ν(C—Br); 3480–3300 ν(OH); 1625–1610 ν(C=N); 1320–1280 ν(C—N); 1180–1140 ν(S=O); 1520–1500 ν(N—H); 1690–1660 ν(C=O).

Preparation of 1-nitroso-3-(2′-hydroxy-3′-bromo-5′-ethyl phen-1′-yl)-5-aryl-2-pyrazolines [6(a–h)]

Compound (2) (0.002 mol) was dissolved in 1 : 1 hydrochloric acid (2 mL) and then cooled in ice-bath. Cold sodium nitrite solution (6 mL, 10%) was added dropwise to the mixture with continuous stirring. The mixture was stirred for further 30 min at room temperature. The solid residue was filtered, washed with distilled water, dried and crystallised from ethanol to give nitroso pyrazolines (6).

m.p. (°C;): 6a: 98; 6b: 85; 6c: 110; 6d: 100; 6e: 112–114; 6f: 93; 6g: 108; 6h: 105.

IR (cm^{-1}): 640–620 ν(C—Br); 3480–3300 ν(O—H); 1625–1610 ν(C=N); 1320–1280 ν(C—N); 1580–1560 ν(N=O).

ACKNOWLEDGEMENT

The authors are thankful to Department of Chemistry, South Gujarat University, Surat, for providing laboratory facilities.

REFERENCES

1. T.C. Sharma, M.M. Bokadia and N.J. Reddy, *Indian J. Chem.*, **19B,** 228 (1980); N. Stamper and B.F. Avcock, *J. Am. Chem. Soc.*, **76,** 2786 (1954); C.P. Singh and A.C. Ojha, *J. Indian Chem. Soc.*, **57,** 1172 (1980).
2. A. Burger, Medicinal Chemistry, John Wiley and Sons (1970).
3. M.G. Joshi and K.N. Wadodkar, *Indian J. Chem.*, **20B,** 1090 (1981).
4. S.R. Modi and H.B. Naik, *Indian J. Heterocyclic Chem.*, **3,** 133 (1993); P.A. Mehta and H.B. Naik, *Orient J. Chem.*, **14,** 159 (1998).
5. F. Kavanagh, Analytical Microbiology, Academic Press, New York, p. 125 (1963).

(*Received*: 2 April 1999; *Accepted*: 31 July 1999) AJC-1828