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SYNTHESIS AND CHARACTERIZATION OF SOME BENZOTHIAZOLOPYRAZOLINE DERIVATIVES

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Abstract:
A new route for the synthesis of benzothiazolopyrazoline derivatives has been developed using various chalcone derivatives and 2-hydrazinobenzothiazole and piperidine catalyst. The product obtained in shorter reaction times and piperidine behaves as good catalyst for the cycloaddition rof chalcone and 2-hydrazinobenzothiazole. The reaction carried out in aqueous-ethanol medium at reflux condition and product obtained in high yield.

Keywords: *Benzothiazolpyrazoline; Chalcones; Cycloaddition Reactions; Piperidine.*

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1. Introduction

Heterocycles containing nitrogen, sulphur and oxygen constitute the core structure of a number of biologically interesting compounds. Pyrazolines are an interesting group of compounds, many of which possess pharmacological properties such as analgesic, antidepressant, antipyretic and antirheumatic activities, anti-inflammatory activity and are used as potent antidiabetic agents. In recent times, pyrazolines were reported as a DP-IV inhibitors and antitumor agents[6-8]. Some nitrogen heterocyclic compounds were reported to be anticancer[9], antimicrobial[10-14] and anti-inflammatory agents[15]. Because of interesting biological activity of various pyrazoline derivatives has been focused on this group. In addition, pyrazolines have played a crucial part in heterocyclic chemistry and also used broadly in organic synthesis[16-20]. Benzothiazole and its derivatives[21-25] have been known as a class of medicinal importance. Benzothiazole derivatives represent an extensive group of heterocyclic compounds, several of which have already found application in the medical sphere as medicines[26] as well as in agriculture[27]. Many of substituted benzothiazoles are known as substances with antibacterial and antifungal activities[28-30] and are reported also to be active as antineoplastics[31] agent. Keeping in view the biological and clinical importance of benzothiazoles and pyrazolines, in this paper we report the synthesis and biological activity of new 3,5-diaryl-1-benzothiazolopyrazoline derivatives.

2. Experimental

2.1. General Procedures

Melting points were determined by the open capillary method. The purity of the compounds was controlled by thin layer chromatography (TLC). IR spectra were recorded as KBr pellets on Perkin-Elmer spectrum spectrophotometer. Carbon, hydrogen, nitrogen and sulphur were estimated by elemental analysis. ^{1}H-NMR spectra were recorded on Waters spectrometer. Mass spectra were measured on JEOL SX mass spectrometer. Substituted chalcones **(1a-d)** were prepared according to the reported methods.[32]

2.2. General Procedure for the Preparation of Compounds

A mixture of substituted chalcone (**1a-d**, 10mmol) and 2-hydrazinobenzothiazole (10mmol) in EtOH-H_2O (20 mL) and 2drops of piperidine is added as catalyst then reaction mixture was refluxed for 30 min. and then cooled to room temperature. The precipitate was separated by filtration, washed with water and recrystallized with ethanol to obtained 3, 5-diaryl-1-benzothiazolopyrazoline derivatives.

3, 5-Diphenyl-1-Benzothiazolo Pyrazoline

Obtained as white crystals yield 94%, m.p. 175-176°C; 1H NMR (δ): 3.24 (1H CH_2(Pyraz)), 3.90 (1H, CH2(Pyraz)), 5.80 (1H, CH(Pyraz)), 6.90-7.68(14H, m, Ar-H); IR(ν) max: 3428, 3016, 1596, 1495, 1443, 1390, 1280, 1120, cm-1; MS, *m/z*: 355, 354, 278, 233, 154, 126, 112, 100, 86. Anal. Calcd. For C22H17N3S: C, 74.36; H, 4.78; N, 11.83; S, 9.02. Found: C, 74.44; H, 4.68; N, 11.88; S, 9.07%.

3. Results and Discussion

Formation of 3, 5-diarylpyrazoline derivatives by the reaction of α,β-unsaturated ketones and hydrazines may take place under various reaction conditions using aqueous-ethanol as solvent. In our present case to obtained 3, 5-diaryl-1-benzothiazolopyrazoline derivatives **(3a-d)** a mixture of substituted chalcone (**1a-d**, 10mmol) and 2-hydrazinobenzothiazole (10mmol) in ethanol-water 1:1 (20 mL) was refluxed for 30 min. and then cooled to room temperature. The precipitate was separated by filtration, washed with water and recrystallized from alcohol (Scheme 1). Substituted chalcones **(1a-d)** were prepared according to the reported methods. Structures of all new compounds **(3a-d)** have been elucidated by elemental analyses, 1H NMR, Mass spectral and IR measurements. In the IR spectra of 3,5-diaryl-1-benzothiazolopyrazoline derivatives (**3a-d**) there is no carbonyl absorption but absorption bands in the region 1600-1450 cm-1 due to the presence of C=C and C=N stretching vibrations. Stretching vibrations due to the intramolecular hydrogen bonding of –OH group gave absorption in the region 3000-2500cm-1. In addition to aromatic protons, the 1H NMR spectra of these compounds exhibit double doublets for each δ CH2 proton between δ 3.23 to 3.64 and δ 3.88 to 4.00 and double doublet between δ 5.25 to 6.22 for –CH proton of the pyrazoline nucleus system.

3a: R1= R2 = R3= R4= R5= H
3b: R2= OH, R1= R3= R4= R5= H
3c: R1= Cl, R2 = R3= R4= R5=H
3d: R1= CH3, R2 = R3= R4= R5= H

In conclusion, we have synthesized a series of new 3, 5-diaryl-1-benzothiazolopyrazoline derivatives. These substituted benzothiazolo- pyrazolines are very stable compounds, which are expected to biological active against various bacterial and fungal stains.

References

[1] Jung J C, Watkins E B and Avery M A, Heterocycles, 2005, 65, 77.
[2] Kumar B, Pathak V, Rani S, Kant R, Tewari I C, Int. J. Microbio Res, 1(2), 2009, 20
[3] Bansal E, Shrivastava V K and Kumar A, Eur J Med Chem., 2001, 36, 81.
[4] Ahn J H, Kim H M, Jung S H, Kang S K, Kim K R, Rhee S D, Yong S D, Cheon H G and Kim S S, Bioorg Med Chem Lett., 2004, 14, 4461.
[5] Villhauer E B, Brinkman J A, Naderi C B, Dunning B E, Mangold B L, Mone M D, Russell M E, Weldon S C and Hughes T E, J Med Chem., 2002, 45, 2362.
[6] Amr A E, Indian J Heterocycl Chem., 2000, 10, 49.
[7] Hamman A G, Fahmy A F M, Amr A E and Mohamad A M Indian J Chem., 2003, 42(B), 1985.
[8] Hamman A G, Abdel Hafez N A, Midura W H. and Mikolajczyk M, Z Naturforsch., 2000, 55(B), 417.
[9] Amr A E and Abou-Ghalia M H, Amino Acids, 2004, 26, 283.
[10] Hammam A G, Sharaf M A. and Abdel-Hafez N A, Indian J Chem., 2001, 40B, 213.
[11] Amr A E, Mohamed A M and Ibrahim A A, Z Naturforsch, 2003, 58b, 861.
[12] Ankhiwala M. D. and Naik H. B. (1990) J. Indian Chem. Soc., 67, 258.
[13] Attia A, Abdel-Salam O I, Amr A E, Stibor I and Budesinsky M, Egypt J Chem., 2000, 43, 187.
[14] Sharma V and Sharma KV, E-Journal of Chem., 2009, 6(2), 348.
[15] Fahmy H H and Soliman G A, Arch Pharm Res., 2001, 24,180.
[16] Tomilovi Yu V, Okonnishnikova G P, Shulishov E V and Nefedov O M, Russ Chem Bt., 1995, 44, 2114.
[17] Desai M. D. and Desai K. K. Asian J. Chem., 2002, 14(2), 995.
[18] Bhaskarreddy D, Padmaja A, Ramanareddy P V and Seenaiah B, Sulfur Lett., 1993, 16, 227.
[19] Padmavathi V, Sumathi R P, Chandrasekhar B N and Bhaskarreddy D, J Chem Research, 1999, 610.
[20] Bhaskarreddy D, Chandrasekhar B N, Padmavathi V and Sumathi R P, Synthesis, 1998, 491
[21] Russo F, Romeo G, Santagati N A, Caruso A, Cutuli V and Amore D, Eur J Med Chem., 1994, 29, 569.
[22] Baltork I M, Khosropour A R and Hojati S F, Monatshefte fur Chemie – Chemical Monthly, 2007, 138, 663.
[23] Katsura Y, Inoue Y, Nishino S, Tomoi M and Takasugi H, Chem Pharm Bull (Tokyo), 1992, 40, 1818.
[24] Kuhler T C, Swanson M, Shcherbuchin V, Larsson H, Mellgard B and Sjostrom J E, J Med Chem., 1998, 41, 1777.
[25] Krasovskii A N, Grin A N, Soroka I. I., Kochergin P M and Bogamyreva E I, Pharma Chem J., 1977, 11(7), 900.
[26] Kashiyama E, Hutchinson I and Chua M S, J Med Chem., 1999, 42, 4172.
[27] Pulkrábek J, Šroller J and Zahradnícek J, Rostlinná Výroba, 1999, 45, 379.
[28] Foltínová P, Sutoris V and Blockinger G, Folia Microbiologica, 1978, 23, 225.

[29] Sharma T C, Pawar S R and Reddy N J, Acta Chim Hung., 1983, 112, 159.
[30] Afsah S A and Nayer S A, Asian J Chem., 1996, 8, 419.
[31] Krieg M and Billitz J, Molecular and Biochemical Pharmacology, 1996,51,1461.
[32] Narender T, Reddy K P, Tetrahedron Letters, 2007, 48(18), 3177

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