Asian Journal of Chemistry Vol. 20, No. 1 (2008), 808-810

NOTE

Synthesis and Antifungal Activity of Some Substituted Cyclohexyl Pyrazolines

PRAVEEN TRIPATHI* and SUDHA PANDEY
Department of Chemistry, St. Andrews College, Gorakhpur-273 001, India
E-mail: praveen_chem97@rediffmail.com

Some new substituted cyclohexyl pyrazolines have been synthesized. These newly synthesized substituted pyrazolines shows fungicidal activity against *Helminthosporium oryzae* and *Cephalosporium sacchari*.

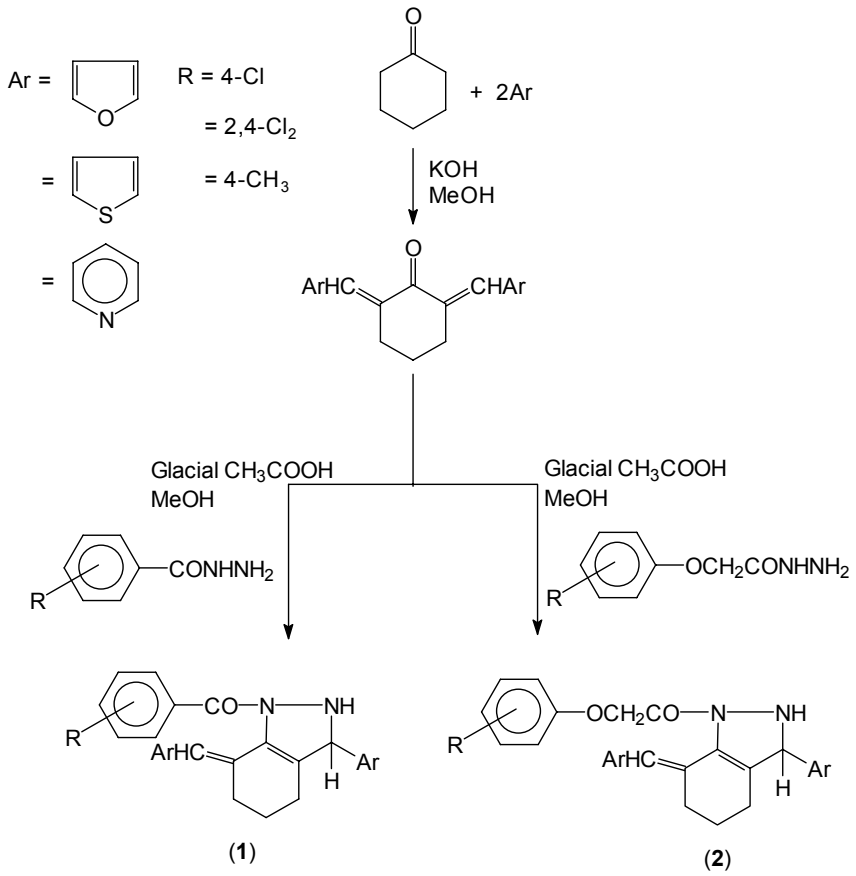
Key Words: Synthesis, Cyclohexyl pyrazolines, Antifungal activity.

A perusal of literature revealed that pyrazoline derivatives show widely differing biological properties *e.g.*, herbicidal[1], bactericidal[2], fungicidal[3], insecticidal[4] and several other biological activities[5,6]. Pyrazoline derivatives are also used in textile and cinematographic film industry[7-9].

Melting point were recorded in open capillaries and are uncorrected. The purity of compounds was checked by TLC using silica gel-G. IR spectra were recorded on Perkin-Elmer-710 spectrophotometer in nujol and ^{1}H NMR spectra on Bruker DRX-300 at 300 MHz were recorded in DMSO-d_6. Elemental analysis were recorded on Carlo Erba 1108. The reaction sequences of formation of substituted cyclohexyl pyrazolines compound is given in **Scheme-I**.

Synthesis of 2,6-*bis*(4-furylmethylene)cyclohexane: A mixture of cyclohexanone (0.1 mol, 9.8 mL), 2 mol of furfuraldehyde (0.1 mol, 16.4 mL) and 2 mol of KOH (11.2 g) in methanol solution is refluxed for 4 h. The solid compound was filtered, washed with water, dried and crystallized from aqueous ethanol, yield 71 %, m.p. 130 ºC.

Synthesis of 1-(4-methylbenzoyl)-3-(2-furyl)-7-(2-furylidine)-cyclohexylpyrazolines: A mixture of 2,6-*bis*(4-furylmethylene)cyclohexanone (0.01 mol, 2.5 g), 4-chlorobenzoyl hydrazone (0.01 mol, 1.7 g) and 2-3 drops of glacial acetic acid in methanol were refluxed for 4-5 h and poured into cold water. The solid obtained was filtered, washed with water dried and crystallized from aqueous ethanol to yield 67 %, m.p. 100 ºC. IR (KBr, ν_{max}, cm^{-1}) 3275 (NH), 1700 (C=O), 1460, 1500, 1560 (C=C, ArH). ^{1}H NMR (DMSO-d_6). δ 1.60 (m, 2H, >C=CH$_2$–CH$_2$), 1.96 (t, 4H, >C=C–CH$_2$), 2.20 (s, 3H, CH$_3$), 6.1-7.0 (m, 9H, ArH), 9.0 (s, 1H, NH) Anal. (%) Calcd. (found) for $C_{24}H_{22}N_2O_3$: C: 74.61 (74.57); H: 5.69 (5.63); N: 7.25 (7.20).



Scheme-I

Synthesis of 1-(4-chlorobenzoyloxyaceto-3-(2-thienyl)-7-(2-thienylidene)cyclohexylpyrazolines: A mixture of 2,6-*bis*-4-(thienyl methyl)cyclohexanone (0.01 mol, 2.86 g), 4-chlorophenoxyacetohydrazide and 2-3 drops of glacial acetic acid in methanol was refluxed for 4 h. Then poured into beaker after evaporation of methanol. The solid product thus obtained was filtered, washed, dried and crystallization from aqueous ethanol to yield 73 %, m.p. 125 ºC. IR (KBr, ν_{max}, cm^{-1}): 3320 (N–H), 2600 (S–H), 1650 (C=O), 1460, 1510, 1550 (C=C ArH). ^{1}H NMR (DMSO-d_6): δ 1.60 (m, 2H, >C=CH_2–CH_2–CH_2–), 1.94 (t, 4H, >C=C–C–CH_2–), 2.5 (s, 1H, C=C–CH–C=C–), 4.6 (s, 2H, –OCH_2 CO–), 6.0-8.2 (m, 10H, aromatic as well as conjugated olefinic proton), 9.5 (d, 1H, NH) Anal. (%) Calcd. for $C_{24}H_{21}N_2O_2S_2Cl$: C, 61.53; H, 4.48; N, 5.98; Found: C, 61.38; H, 4.37; N, 5.88.

Antifungal activity: The fungicidal activity of newly synthesized pyrazolines derivatives were evaluated against *Helminthosporium oryzae* and *Cephalosporium sacchari*. The czepaks agar method was used. The diameter of the fungus colony was measured by means of millimeter scale at time interval of 96 h. It was found that compound containing R = 4-Cl shows maximum activity. The commercial fungicide dithane M-45 was used as standard for comparison.

ACKNOWLEDGEMENTS

The authors are thankful to CDRI, Lucknow for spectral and elemental analysis. Thanks are also due to Dr. L.B. Singh Department of Chemistry, St. Andrews College for valuable suggestions.

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(*Received*: 23 November 2006; *Accepted*: 24 September 2007) AJC-5896