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Synthesis and Characterization of Various Pyrazolines From Chalcones

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Abstract: ***Some novel series of pyrazoline derivatives were synthesized from Chalcones. Various Pyrazoline derivatives were prepared by reflux reaction of Chalcone with Phenyl Hydrazine Hydrate in ethanolic solution. The structures of the newly synthesized Pyrazoline derivatives have been characterized by spectral data.***
Keywords: Pyrazolines, chalcones, spectral analysis.

I. INTRODUCTION

Because bacterial resistance to antibacterial drugs is rapidly developing, it is critical to develop new scaffolds for the design and synthesis of new antibacterial agents to help in the fight against harmful germs. Chalcones are a class of natural and synthetic compounds that have a wide variety of pharmacological actions, including antibacterial[1], antitumour[2], anticancer[3], antitubercular[4], antiinflammatory[5], antioxidant[6], antimalarial[7], antileishmanial[8], and other properties. The biological activity of chalcones is determined to be due to the presence of a reactive, -unsaturated keto group. In this study, chalcones were made by condensing different ketones with aromatic aldehyde via claisen Schmidt condensation. According to available evidence, N-containing heterocyclic compounds derived from chalcones have a wide range of activities[9-17], including possible cytotoxic, antibacterial, antiviral, anti-inflammatory, anaesthetic, and mydriatic properties.

II. MATERIALS AND METHOD

All of the reagents and solvents used were of the highest quality. Which were used without being purified further. Melting points were measured using standardized melting point equipment. UV-Visible absorption spectra in DMSO were recorded using a SHIMADZU UV-1800 spectrometer with more than a concentration range of 104 M. In KBr pellets, IR spectra were obtained on an FT-IR-Alpha Bruker IR spectrometer from 4000 cm1 to 400 cm1. The 1H and 13C NMR spectra in DMSO-d6 were acquired at 500 MHz using an AV500 Resolution Multinuclear FT-NMR Spectrometer with tetramethylsilane as the internal standard. In sterile distilled water, a working stock of 100 mg/ml concentration of the given drug samples identified as "pyrazoline derivatives" was prepared.

III. SYNTHESIS

(Z)-2-benzylidene-N1-(((2-hydroxy-3-nitrophenyl)(4-methoxyphenyl)methyl)carbamothioyl)-N3-(((2-hydroxyphenyl)(4-methoxyphenyl)methyl)carbamothioyl) malonamide (0.01 mole) In 100% ethanolic medium, malonamide and hydrazine hydrate (0.01 mole) were refluxed for 1 hour. When the mixture was filtered under suction and recrystallized from ethanol, it was cooled and poured on crushed ice, and recrystallized from ethanol.

IV. EXPERIMENTAL DATA

Comp. Code	Chemical formula	Molecular weight	Elemental analysis	Appearance	Melting point	Yield
V(1)	$C_{44}H_{41}N_7O_6S$	795.28	C (66.40%) H (5.19%) N (12.32%) O (12.06%) S (4.03%)	Brown	185^0C	

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V(2)	$C_{42}H_{41}N_6O_6SCl$	792.25	C (63.59%) H (5.21%) N (10.59%) O (12.10%) S (4.04%) Cl (4.47)	Brown	182^0C	50%
V(3)	$C_{41}H_{39}N_7O_8S$	789.26	C (62.35%) H (4.98%) N (12.41%) O (16.20%) S (4.06%)	Yellow	180^0C	53%
V(4)	$C_{41}H_{40}N_6O_7S$	760.27	C (64.72%) H (5.30%) N (11.05%) O (14.72%) S (4.21%)	Brown	165^0C	55%
V(5)	$C_{42}H_{42}N_6O_6S$	758.29	C (66.47%) H (5.58%) N (11.07%) O (12.65%) S (4.22%)	White	162^0C	47%

V(1): N-(((5-amino-2-hydroxyphenyl)(4-methoxyphenyl)methyl)carbamoyl)-3-(3-((1-hydroxynaphthalen-2-yl)(4-methoxyphenyl)methyl)thioureido)-5-phenyl-4,5-dihydro-1H-pyrazole-4-carboxamide:
IR (KBr) cm-^{1}: 3705(Ar-OH stretching), 1413(C=N stretching) 1650 (C=O) 3315 (N-H stretching for 2^0 amines) 1491 (C=S), 1078 (C-N stretching) 1363 (OH bending). 755 (N-H wagging)
^{1}HNMR (CDCl3) δ: 13.01(s,1H NH), 8.12 (d,1H,NH), 9.92 (d,1H,NH Pyrazoline ring) 4.12 (dd, 1H, CH pyrazoline ring) 2.9 (d,1H CH pyrazoline ring) 9.65 (s,1H,Ar-OH), 5.26(s,1H,CH), 7.26, 7.35, 6.99, 6.86 (m, 4H, Ar-H) 8.05, 7.43, 7.22,(m, 3H, Ar-H), 7.32, 7.36. 7.26, 7.33, 7.35(m 5H Ar-H) 3.81(s,3H CH_3)
^{13}CNMR (CDCl3) δ: 52.2 (1C, CH, of pyrazoline ring) 46.23 (1C, CH of Pyrazoline ring) 175.4 (1C, C=O) 184 (1C C=S) 61 (1C, CH) 57.1 (1C OCH_3) 155, 135,136, 120,127 (6C-C_6H_5) 128 (6C-C_6H_5)
V(2): N-(((3-chloro-4-hydroxyphenyl)(4-methoxyphenyl)methyl)carbamoyl)-3-(3-((4-hydroxy-3-methylphenyl)(4-methoxyphenyl)methyl)thioureido)-5-phenyl-4,5-dihydro-1H-pyrazole-4-carboxamide:
IR (KBr) cm-^{1}: 3698(Ar-OH stretching), 1415(C=N stretching) 1654 (C=O) 3215 (N-H stretching for 2^0 amines) 1491 (C=S), 1078 (C-N stretching) 1363 (OH bending). 755 (N-H wagging)
^{1}HNMR (CDCl3) δ: 13.07(s,1H NH), 8.1 (d,1H,NH), 9.92 (d,1H,NH Pyrazoline ring) 4.1 (dd, 1H, CH pyrazoline ring) 2.81 (d,1H CH pyrazoline ring) 9.63 (s,1H,Ar-OH), 5.16(s,1H,CH), 7.21, 7.32, 6.89, 6.85 (m, 4H, Ar-H) 8.05, 7.43, 7.22,(m, 3H, Ar-H), 7.32, 7.35. 7.25, 7.32, 7.35(m 5H Ar-H) 3.81(s, 3H CH_3)
^{13}CNMR (CDCl3) δ: 52.11 (1C, CH, of pyrazoline ring) 47.23 (1C, CH of Pyrazoline ring) 175.4 (1C, C=O) 184 (1C C=S) 61 (1C, CH) 57.1 (1C OCH_3) 155, 135,136, 120,127 (6C-C_6H_5) 128 (6C-C_6H_5)
V(3): 3-(3-((4-hydroxy-3-methylphenyl)(4-methoxyphenyl)methyl)thioureido)-N-(((2-hydroxy-5-nitrophenyl)(4-methoxyphenyl)methyl)carbamoyl)-5-phenyl-4,5-dihydro-1H-pyrazole-4-carboxamide:
IR (KBr) cm-^{1}: 3705(Ar-OH stretching), 1413(C=N stretching) 1650 (C=O) 3315 (N-H stretching for 2^0 amines) 1491 (C=S), 1078 (C-N stretching) 1363 (OH bending). 755 (N-H wagging)
^{1}HNMR (CDCl3) δ: 13.07(s,1H NH), 8.2 (d,1H,NH), 9.92 (d,1H,NH Pyrazoline ring) 4.15 (dd, 1H, CH pyrazoline ring) 2.7 (d,1H CH pyrazoline ring) 9.65 (s,1H,Ar-OH), 5.14(s,1H,CH), 7.21, 7.32, 6.89, 6.85 (m, 4H, Ar-H) 8.05, 7.43, 7.22,(m, 3H, Ar-H), 7.32, 7.35. 7.25, 7.32, 7.35(m 5H Ar-H) 3.81(s, 3H CH_3)
^{13}CNMR (CDCl3) δ: 52.11 (1C, CH, of pyrazoline ring) 47.23 (1C, CH of Pyrazoline ring) 175.4 (1C, C=O) 184 (1C C=S) 61 (1C, CH) 57.1 (1C OCH_3) 155, 135,136, 121,126 (6C-C_6H_5) 128.2 (6C-C_6H_5)

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V(4): N-(((2,4-dihydroxyphenyl)(4-methoxyphenyl)methyl)carbamoyl)-3-(3-((4-hydroxy-3-methylphenyl)(4-methoxyphenyl)methyl)thioureido)-5-phenyl-4,5-dihydro-1H-pyrazole-4-carboxamide:

IR (KBr) cm-^{1}: 3695(Ar-OH stretching), 1413(C=N stretching) 1650 (C=O) 3315 (N-H stretching for 2^{0} amines) 1491 (C=S), 1078 (C-N stretching) 1363 (OH bending). 755 (N-H wagging)

^{1}HNMR (CDCl3) δ: 13.27(s,1H NH), 8.1 (d,1H,NH), 9.91 (d,1H,NH Pyrazoline ring) 4.10 (dd, 1H, CH pyrazoline ring) 2.8 (d,1H CH pyrazoline ring) 9.63 (s,1H,Ar-OH), 5.16 (s,1H,CH), 7.31, 7.33, 6.89, 6.84 (m, 4H, Ar-H) 8.05, 7.43, 7.24,(m, 3H, Ar-H), 7.32, 7.34. 7.26, 7.32,7.35(m 5H Ar-H) 3.81(s,3H CH_3)

^{13}CNMR (CDCl3) δ: 52.11 (1C, CH, of pyrazoline ring) 47.23 (1C, CH of Pyrazoline ring) 175.4 (1C, C=O) 182.1 (1C C=S) 61 (1C, CH) 57.01 (1C OCH_3) 155, 134,136, 120,127.5 (6C-C_6H_5) 128 (6C-C_6H_5)

V(5): 3-(3-((4-hydroxy-3-methylphenyl)(4-methoxyphenyl)methyl)thioureido)-N-(((2-hydroxy-5-mehtylphenyl)(4-methoxyphenyl)methyl)carbamoyl)-5-phenyl-4,5-dihydro-1H-pyrazole-4-carboxamide:

IR (KBr) cm-^{1}: 3707(Ar-OH stretching), 1415(C=N stretching) 1651 (C=O) 3310 (N-H stretching for 2^{0} amines) 1491 (C=S), 1077 (C-N stretching) 1362 (OH bending). 756 (N-H wagging)

^{1}HNMR (CDCl3) δ: 13.17(s,1H NH), 8.1 (d,1H,NH), 9.82 (d,1H,NH Pyrazoline ring) 4.2 (dd, 1H, CH pyrazoline ring) 2.81 (d,1H CH pyrazoline ring) 9.63 (s,1H,Ar-OH), 5.16(s,1H,CH), 7.21, 7.02, 6.89, 6.65 (m, 4H, Ar-H) 8.15, 7.43, 7.22,(m, 3H, Ar-H), 7.43, 7.31. 7.25, 7.32, 7.31(m 5H Ar-H) 3.81(s,3H CH_3)

^{13}CNMR (CDCl3) δ: 50.43 (1C, CH, of pyrazoline ring) 46.23 (1C, CH of Pyrazoline ring) 174.4 (1C, C=O) 186.5 (1C C=S) 61.1 (1C, CH) 55.1 (1C OCH_3) 153, 135,137, 121,128 (6C-C_6H_5) 128 (6C-C_6H_5)

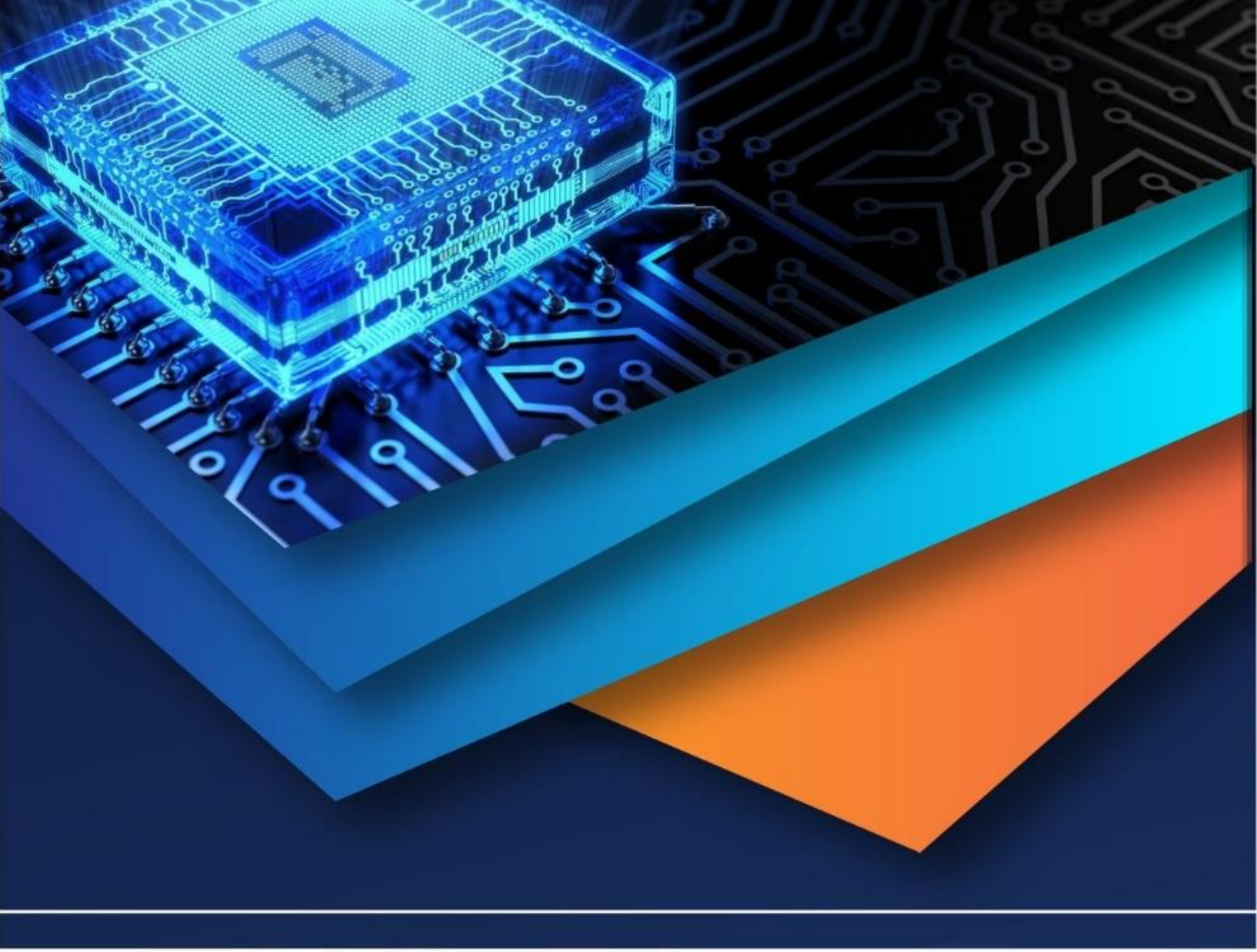
V. CONCLUSION

In this study, we have successfully synthesized new pyrazolines derivatives compounds. The proper analysis of the synthesis of new pyrazolines derivatives has been systematically evaluated by FT-IR, NMR, Mass spectrometry.

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