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Synthesis and Characterization of Novel Pyrazoline Derivatives

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***Abstract*: Pyrazoline derivatives, are prepared from p – Amino benzoic acid via chalcones. Which has been prepared by the condensation reaction of Ethyl-4-acetamido benzoate and different ten aldehydes. These chalcones are cyclized with hydrazine hydrate and glacial acetic acid under reflux condition give pyrazoline derivatives. These compounds have been characterized by detailed spectral analysis**

***Keywords*: Claisen-schmidt condensation, chalcones, pyrazolines.**

1. Introduction

The chemistry of chalcones has generated intensive scientific interest due to their biological and industrial applications. Chalcones are natural biocides and are well known intermediates in the synthesis of heterocyclic compounds exhibiting various biological activities. Chalcones and their derivatives possess some interesting biological properties such as anti- bacterial, antifungal, insecticidal, anesthetic, anti-inflammatory, analgesic etc. [1]-[9]. Pyrazole is a class of compounds, which has many applications in different field. One of the methods for the synthesis of such compound is from unsaturated carbonyls (chalcone) by the cyclization with hydrazine and substituted hydrazine. Pyrazole and their derivatives are considered to be important for drugs and agricultural chemicals. Some substituted pyrazoles and their derivatives have been reported to possess several interesting biological activities such as hypnotic properties, antimicrobial, antitumor and antifungal. Many pyrazoles are used for the treatment of thyroid and leukemia. It has incidental antiviral activity against Herpes infections10.

2. Material methods

A. Synthesis

1) STEP 1: Synthesis of Ethyl-4-amino benzoate11

To p-Amino benzoic acid 12 gms (0.088 moles) add 80 mL of 95% v/v ethanol in a round bottom flask and 4 mL of concentrated sulphuric acid add slowly. Refluxed for 2 hour at 60oC on water bath. Then cooled the flask for several minutes. Then added 150 mL of 10 % sodium carbonate solution, which resulted in the evolution of considerable gas until the solution is neutralised. Filtered the solution and collect the precipitate. Recrystallised from rectified spirit and drying in desiccators under vacuum. (% yield 68 % w/w: m.pt: 91℃ – 92℃).

STEP 2: Synthesis of Ethyl-4-acetamido benzoate

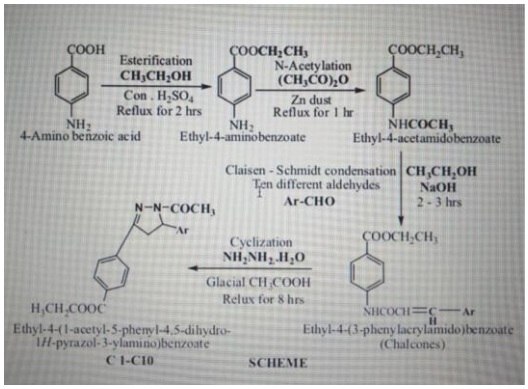
To 6.6 g of Ethyl-4-amino benzoate (0.045 moles) added 12 mL of acetic anhydride and few mgs of zinc dust. Refluxed for 1 hour. Cooled the content and filtered. Washed the precipitate with cold water and filter it. Recrystallised from rectified spirit and drying in desiccators. (% yield 64 % w/w: m.pt: 104℃ – 105℃).

2) STEP 3: Synthesis of Chalcones

To Ethyl-4-acetamido benzoate (2.07 g, 0.01 moles) added aromatic aldehydes (0.01 moles) in ethanol (20 mL) and catalytic quantity of sodium hydroxide. The mixture was stirred for 2 to 3 hours at room temperature using magnetic stirrer. The reaction was monitored by TLC and it was kept at room temperature and then cooled in an ice bath. After filtration, the product was washed with ethanol (5 mL) followed by distilled water, dried and recrystallised from ethanol to yield a pure chalcones.

3) STEP 4: Synthesis of pyrazolines12

To the ten different Chalcones (0.01 moles) added glacial acetic acid (10 mL) and hydrazine hydrate 99 % (0.01 mole). Refluxed for 8 hour on water bath at 80℃ and cool it. The resulting solid was filtered, washed with distilled water. Recrystallised from ethanol and drying in desiccators.



C1 R – Benzaldehyde, C2 R – Salicylaldehyde, C3 R – p – Chloro benzaldehyde, C4 R – o – Chloro benzaldehyde, C5 R – o – Nitro benzaldehyde, C6 R– m– Nitro benzaldehyde, C7 R

–p–Hydroxyl benzaldehyde, C8 R – p Dimethyl amino benzaldehyde, C9 R – 3, 4, 5 – Trimethoxy benzaldehyde, C10 R – Anisaldehyde.

Spectral Analysis

B. Compound C1

Ethyl-4-(1-acetyl-5-phenyl-4,5–dihydro-1H–pyrazol– 3 ylamino) enzoate, a white crystalline solid, was purified by crystallisation using ethanol; m.pt. 234°C; IR (KBr, cm-1): 3305.23 (-NH), 3029.14 (=C- H), 1681.70 (Acetyl carbonyl), 1607.76 (α, β - unsaturated double bond), 1514.52 (Aromatic region), 1264.57, 1178.9 (-C-O), 834.79 (Para disubstituted), 867.94 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,9H,Ar-H), δ 4.9 (d,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (s,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t,3H,CH3);MS: Mol.Wt: 351.4, m/e: 351.16 (100.0%), 352.16 (23.1%), 353.17 (2.3%). 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.1 (CH3), 39.3 (CH2), 56.2 (CH), 60.1 (CH2), 116.1 (CH), 116.0 (CH), 120.6 (C), 121.4 (CH), 126.1 (CH), 127.6 (CH), 127.0 (CH), 128.4 (CH), 130.0 (CH), 131.1 (CH), 143.2 (C), 148.1 (C), 155.8(C), 166.2 (C), 168.8 (C). Anal.Caled for C20H21N3O3; C, 68.36; H, 6.02; N, 11.96; O, 13.66. Found C, 68.31; H, 5.80; N, 11.28; O, 13.42; Confirmed the product formed.

C. Compound C2

Ethyl-4-(1-acetyl-5-(2-hydroxyphenyl) - 4, 5- dihydro-1H-pyrazol-3-ylamino) benzoate, a white crystalline solid, was purified by crystallisation using ethanol; m.pt. 221°C; IR (KBr, cm-1): 3305.54 (-NH), 3029.14 (=C-H), 1681.62 (Acetyl carbonyl), 1607.93 (α, β-unsaturated double bond), 1519.82 (Aromatic region), 1264.57, 1178.91 (-C-O), 834.72(Para di substituted), 867.72 (C-N). 1H NMR (CDCl3): δ 7.72 - 6.57 (m,8H,Ar-H),δ 5.0 (s,1H,OH), δ 4.9 (d,1H,CH), δ 4.29 (m,2H,CH2),δ 4.0 (m,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (m, 3H, CH3). MS: Mol.Wt: 367.398 m/e: 367.153 (100.0%), 368.157 (21.8%), 369.160 (2.2%), 368.150 (1.1%). Anal.Caled for C20H21N3O4; C, 65.38; H, 5.76; N, 11.44; O, 17.42. Found C, 62.31; H, 5.20; N, 10.28; O, 17.42. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.6 (CH2), 46.2 (CH), 60.9 (CH2), 115.1 (CH), 116.2 (CH), 116.2 (CH), 120.2 (C), 121.2 (CH), 128.2 (CH), 128.4 (CH), 130.7 (CH), 130.7 (C), 148.7 (C), 154.1 (C), 155.0 (C), 166.0 (C), 166.3(C), 168.8 (C); Confirmed the product for med.

D. Compound C3

Ethyl-4-(1-acetyl-5-(4-chlorophenyl)-4,5-dihydro-1H-pyrazol-3-amino)benzoate, a white crystalline solid, was purified by crystallisation using ethanol;m.pt.230°C; IR (KBr, cm-1): 3305.35 (-NH), 3029.39 (=C-H), 1681.96 (Acetyl carbonyl), 1607.97 (α, β-unsaturated double bond), 1519.87 (Aromatic region), 1264.44, 1178.91 (-C-O), 834.49 (Para di substituted), 867.94 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29(d,2H,CH2), δ 4.0 (m,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t, 3H,CH3). MS; Mol. Wt: 385.844; m/e: 385.119 (100.0%), 387.116 (32.0%), 386.123 (21.6%), 388.120 (6.9%), 387.126 (2.2%), 386.116 (1.1%). Anal.Caled for C20H20ClN3O3; C, 62.26; H, 5.22; Cl, 9.19; N, 10.89; O, 12.44. Found C, 62.31; H, 5.01; Cl, 9.91; N, 10.24; O, 12.42. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 56.2 (CH), 60.9 (CH2), 116.1 (CH), 116.2 (CH), 120.2 (C), 120.2 (C), 128.4 (CH), 128.4 (CH), 128.7 (CH), 128.7 (CH) 130.7 (CH), 132.7 (C), 141.6 (C), 148.8 (C), 155.0 (C), 166.0 (C), 168.2 (C); Confirmed the product formed.

E. Compound C4

Ethyl-4-(1-acetyl-5-(2-chlorophenyl)-4,5-dihydro-1H-pyrazol-3-ylamino) benzoate, a white crystalline solid, was purified by crystallisation using ethanol; m.pt. 228°C; IR (KBr, cm-1): 3305.48 (-NH), 3029.36 (=C-H), 1681.55 (Acetyl carbonyl), 1607.98 (α, β-unsaturated double bond), 1519.92 (Aromatic region), 1264.35, 1178.91 (-C-O), 834.61 (Para di substituted), 867.94 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (m,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t, 3H, CH3). MS; Mol.Wt: 385.844; m/e: 385.119 (100.0%), 387.116 (32.0%), 386.123 (21.6%), 388.120 (6.9%), 387.126 (2.2%), 386.116 (1.1%). Anal.Caled for C20H20ClN3O3; C, 62.26; H, 5.22; Cl, 9.19; N, 10.89; O, 12.44; Found C, 62.31; H, 5.01; Cl, 9.91; N, 10.24; O, 12.42; 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 38.3 (CH2), 47.6 (CH), 60.9 (CH2), 116.1 (CH), 116.2 (CH), 120.2 (C), 120.2 (C), 126.4 (CH), 128.2 (CH), 128.4 (CH), 128.7 (CH), 130.7 (CH), 130.7 (CH), 143.5 (C), 148.8 (C), 155.0 (C), 166.0 (C), 168.2 (C); Confirmed the product formed.

F. Compound C5

Ethyl-4-(1-acetyl-5-(2-nitro phenyl)-4, 5- dihydro-1H-pyrazol-3-ylamino) benzoate, a yellowish white crystalline solid, was purified by crystallisation using ethanol; m.pt. 239°C; IR (KBr, cm- 1): 3305.48 (-NH), 3029.76 (=C-H), 1681.08 (Acetyl carbonyl), 1607.02 (α, β-unsaturated double bond), 1519.61 (Aromatic region), 1264.21, 1178.91 (-C-O), 834.41 (Para di substituted), 867.93 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (m,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t, 3H, CH3). MS; Mol. Wt: 868.39; m/e: 47.98 (100.0%). Anal.Caled for C20H20N5O5; C, 27.66; H, 2.32; N, 69.29; O, 18.53; Found C, 27.31; H, 2.01; N, 68.29; O, 18.42. 13C NMR spectrum of compound showed 20. carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39 (CH2), 52.6 (CH), 60.9 (CH2), 116.2 (CH), 116.2 (CH), 120.2 (C),126.8 (CH), 127 (C), 127 (CH), 128 (CH), 128.6 (CH), 130.7 (CH), 130.7 (CH), 142 (C), 148.8 (C), 155 (C), 166.0 (C), 168.3 (C); Confirmed the product for med.

G. Compound C6

Ethyl-4-(1-acetyl-5-(3-nitrophenyl) - 4, 5- dihydro-1H-pyrazol-3-ylamino) benzoate, a yellowish white crystalline solid, was purified by crystallisation using ethanol; m.pt. 218°C; IR (KBr, cm-1): 3305.79 (-NH), 3029.30 (=C-H), 1681.54 (Acetyl carbonyl), 1607.60 (α, β-unsaturated double bond), 1519.68 (Aromatic region), 1264.57, 1178.91 (-C-O), 834.77 (Para di substituted), 867.90 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (m,1H,NH), δ 2.02 (t,3H,CH3), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t, 3H, CH3). MS; Mol. Wt: 868.39; m/e: 47.98 (100.0%). Anal.Caled for C20H20N5O5; C, 27.66; H, 2.32; N, 69.29; O, 18.53; Found C, 27.31; H, 2.01; N, 68.29; O, 18.42; 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 56.7 (CH), 60.9 (CH2), 116.2 (CH), 116.2 (CH), 120.2 (C),127 (CH), 127 (C), 127 (CH), 128 (CH), 128.6 (CH), 130.7 (CH), 130.7 (CH), 143.5 (C), 148.7 (C), 155 (C), 166.0 (C), 168.3 (C); Confirmed the product for med.

H. Compound C7

Ethyl -4-(1-acetyl-5-(4-hydroxy phenyl)-4, 5- dihydro-1H-pyrazol-3-ylamino) benzoate, a white crystalline solid, was purified by crystallisation using ethanol; m.pt. 226°C; IR (KBr, cm-1): 3305.88 (-NH), 3029.78 (=C-H), 1681.31 (Acetyl carbonyl), 1607.92 (α, β-unsaturated double bond), 1519.69 (Aromatic region), 1264.35, 1178.91 (-C-O), 834.24 (Para di substituted), 867.91 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 5.0 (s,1H,OH), δ 4.9 (s,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (s,1H,NH), δ 2.02 (t,3H,CH2), δ 2.0 - 1.8 (d,2H,CH2), δ 1.30 (t, 3H, CH3). MS: Mol. Wt.: 367.4; m/e: 367.15 (100.0%), 368.16 (22.0%), 369.16 (3.1%), 368.15 (1.1%). Anal.Caled for C20H21N3O4; C, 65.38; H, 5.76; N, 11.44; O, 17.42. Found C, 62.21; H, 5.70; N, 11.28; O, 17.42. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 56.2 (CH), 60.9 (CH2), 115.7 (CH), 115.7 (CH), 116.2 (CH), 120.2 (C), 121.2 (CH), 128.4 (CH), 128.4 (CH), 130.7 (CH), 130.7 (C),136.1 (C), 148.7 (C), 155.1 (C), 156.5 (C), 166.0 (C), 168.3 (C); Confirmed the product formed.

I. Compound C8

Ethyl-4-(1-acetyl-5- (4-(dimethylamino) phenyl) - 4, 5-dihydro-1H-pyrazol-3-ylamino) benzoate, a white brown crystalline solid, was purified by crystallisation using ethanol; m.pt. 237°C; IR (KBr, cm-1): 3305.55 (-NH), 3029.14 (=C-H), 1681.01 (Acetyl carbonyl), 1607.01 (α, β-unsaturated double bond), 1519.79 (Aromatic region), 1264.57, 1178.91 (- C-O), 834.26 (Para di substituted), 867.53 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.54 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (m,2H,CH2), δ 4.0 (d,1H,NH), δ 2.85 (m,6H,2CH3), δ 2.02 (t,2H,CH2), δ 2.0 - 1.8 (d,3H,CH3), δ 1.30 (t, 3H, CH3). MS: Mol. Wt: 394.47; m/e: 394.20 (100.0%), 395.20 (25.4%), 396.21 (2.8%). Anal.Caled for C22H26N4O3; C, 66.99; H, 6.64; N, 14.20; O, 12.17. Found C, 66.21; H, 6.40; N, 14.20; O, 12.17. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 40.3 (CH3), 40.3 (CH3), 56.7 (CH), 60.9 (CH2), 114.1 (CH), 114.1 (CH), 116.2 (CH), 116.2 (CH), 120.2 (C), 127.9 (CH), 127.9 (CH), 130.7 (CH), 130.7 (CH),133.1 (C), 147.6 (C), 148.7 (C), 155.1 (C), 166.0 (C), 168.3 (C); Confirmed the product formed.

J. Compound C9

Ethyl-4-(1-acetyl-5-(3, 4, 5-trimethoxy phenyl)-4, 5-dihydro-1H-pyrazol-3-ylamino) benzoate, a white crystalline solid, was purified by crystallisation using ethanol; m.pt. 237°C; IR (KBr, cm-1): 3305.45 (-NH), 3029.50 (=C-H), 1681.19 (Acetyl carbonyl), 1607.04 (α, β-unsaturated double bond), 1519.85 (Aromatic region), 1264.33, 1178.91 (- C-O), 834.55 (Para di substituted), 867.95 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,6H, Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (m,2H,CH2), δ 4.0 (d,1H,NH), δ 3.73 (m,9H,3CH3), δ 2.02 (t,2H,CH2), δ 2.0 - 1.8 (d,3H,CH3), δ 1.30 (t, 3H, CH3). MS: Mol. Wt: 441.48; m/e: 441.19 (100.0%), 442.19 (26.2%), 443.20 (3.1%), 443.19 (1.5%). Anal.Caled for C23H27N3O6 C, 62.57; H, 6.16; N, 9.52; O, 21.74. Found C, 62.21; H, 6.40; N, 9.20; O, 21.74. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 56.3 (CH3), 56.3 (CH3), 56.7 (CH3), 57.3 (CH), 60.9 (CH2), 104.3 (CH), 104.3 (CH), 116.2 (CH), 116.2 (CH), 120.2 (C), 130.7 (CH), 130.7 (CH), 137.2 (C), 137.8 (C),148.7 (C), 150.6 (C), 150.6 (C), 155.1 (C), 166.0 (C), 168.3 (C); Confirmed the product formed.

K. Compound C10

Ethyl-4-(1-acetyl-5-(4-methoxy phenyl)-4, 5- dihydro-1H-pyrazol-3-ylamino) benzoate, a white crystalline solid, was purified by crystallization using ethanol; m.pt. 234°C; IR (KBr, cm-1): 3305.76 (-NH), 3029.01 (=C-H), 1681.76 (Acetyl carbonyl), 1607.97 (α, β-unsaturated double bond), 1519.62 (Aromatic region), 1264.19, 1178.91 (-C-O), 834.25 (Para di substituted), 867.89 (C-N). 1H NMR (CDCl3): δ 7.72 – 6.57 (m,8H,Ar-H), δ 4.9 (s,1H,CH), δ 4.29 (d,2H,CH2), δ 4.0 (m,1H,NH), δ 3.73 (m,3H,CH3), δ 2.02 (t,2H,CH2), δ 2.0 - 1.8 (d,3H,CH3), δ 1.30 (t, 3H, CH3); MS m/z (M+) 369.08, m/z (B+) 269.16. MS: Mol. Wt: 381.43; m/e: 381.17 (100.0%), 382.17 (24.0%), 383.18 (2.6%), 383.17 (1.1%). Anal.Caled for C21H23N3O4; C, 66.13; H, 6.08; N, 11.02; O, 16.78. Found C, 66.21; H, 6.40; N, 11.20; O, 16.74. 13C NMR spectrum of compound showed 20 carbon atoms. δ 14.1 (CH3), 23.4 (CH3), 39.3 (CH2), 55.3 (CH3), 56.7 (CH2), 60.9 (CH2), 114.1 (CH), 114.1 (CH), 116.2 (CH), 116.2 (CH), 120.2 (C), 128.9 (CH), 128.9 (CH), 130.7 (CH), 130.7 (CH), 135.8 (C), 148.7 (C), 155 (C), 158.1 (C), 166.0 (C), 168.3 (C); Confirmed the product formed.

3. Conclusion

All the synthesized compounds were characterized by TLC, Melting point, elemental analysis, IR, Mass, and 1H NMR. Analysis indicated by the symbols of the elements is very close to the theoretical values.

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