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Study of 3-(1-hydroxy naphthalene-2-yl)-5-(furan-2-yl)-1-substituted pyrazolines

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Abstract

2-acetyl-1-naphthol 2 is prepared by Modified Nenchi's method which on treatment with furfuraldehyde and KOH gives 1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-ones 3 in excellent yield. The chalcone 3 when subjected to hydrazine / phenyl hydrazine/ semicarbazide / 2,4 dinitro phenyl hydrazine / isonicotinic acid hydrazide in DMF solvent gives 3-(1-hydroxy naphthalene-2-yl)-5-(furan-2-yl)-1-substituted pyrazolines 4, 5, 6, 7 and 8 in 35-45% yield. The structural assignments to the compounds 4, 5, 6, 7 and 8 are based on their elemental analysis and spectral data.

Keywords: semicarbazide, pyrazolines, cerebroprotective

Introduction

Pyrazolines with sulphonamidoaryl substituent at 3-position show cerebroprotective, antidepressant activity, anti-implantation activity, hypoglycemic activity [1-5]. Due to this vital biological roll of pyrazoline derivatives, it was thought of interest to synthesize titled pyrazoline derivatives. Thus we present herein the synthesis of the titled compounds. It has been observed that substituted flavanones are the best starting compounds for the preparation of 4-aryl derivatives of pyrazoline. Present work also deals with the synthesis of some new pyrazolines and their characterization by spectral analysis (IR, ¹H NMR).

Material and Method

All the melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. ¹H NMR spectra were recorded on a Bruker AC300 FNMR spectrometer (300MHz), using TMS as an internal standard. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer. Thin Layer Chromatography on silica gel-G, was used to check the purity of the compounds. Microanalysis of nitrogen was obtained on Colman 29-N analyzer.

Procedure for the synthesis of 2-acetyl-1-naphthol 2

In hot glacial acetic acid (80ml), fused ZnCl₂(50 gm) was added and refluxed till dissolved, then powdered 1-naphthol (30gm) was added and the mixture was refluxed for about 8 hours then cooled & poured in acidulated water. The solid obtained was filtered, washed, dried and recrystallized from rectified spirit to obtain compound 2. Physical data of the compound is given in table 1.

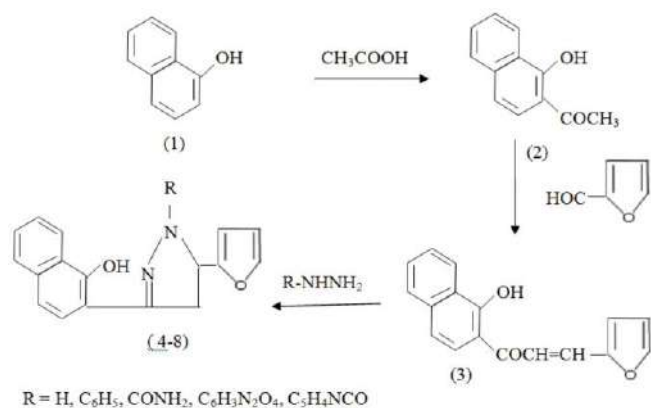
Table 1: Physical and analytical characterization data of newly synthesized compounds

| S. No. | Compound No | Molecular formula | R | Melting Point °C | % Yield | % Nitrogen | | R.F. Value |
|--------|-------------|---|---|------------------|---------|------------|------------|------------|
| | | | | | | Found | Calculated | |
| 1 | 2 | C ₁₂ H ₁₀ O ₂ | - | 98°C | 72% | - | - | |
| 2 | 3 | C ₁₇ H ₁₂ O ₃ | - | 126°C | 71% | - | - | |
| 3 | 4 | C ₁₇ H ₁₄ N ₂ O ₂ | H | 110°C | 38% | 10.00 | 10.07 | 0.79 |
| 4 | 5 | C ₂₃ H ₁₈ N ₂ O ₂ | C ₆ H ₅ | 100°C | 40% | 7.81 | 7.91 | 0.88 |
| 5 | 6 | C ₁₈ H ₁₅ N ₃ O ₃ | CONH ₂ | 140°C | 45% | 12.99 | 13.08 | 0.87 |
| 6 | 7 | C ₂₃ H ₁₆ N ₄ O ₆ | C ₆ H ₃ N ₂ O ₄ | 120°C | 40% | 12.56 | 12.61 | 0.78 |
| 7 | 8 | C ₂₃ H ₁₇ N ₃ O ₃ | C ₅ H ₄ NCO | 117°C | 35% | 10.91 | 10.97 | 0.84 |

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2: IR (KBr): 1650 (C=O), 3412 (-OH)

NMR (CDCl₃ + DMSO-d₆): δ 2.35 (s, 3H, CH₃), δ 7.11-6.88 (m, 6H, Ar-H), δ 9.83 (s, 1H, -OH).

**Synthesis of 1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-one (3)**

2-acetyl-1-naphthol (0.01 mole) and furfuraldehyde (0.02 mole) were added in ethanol solvent (20ml). To this mixture KOH (10%, 10ml) solution was added dropwise with constant stirring. The reaction mixture was kept overnight. Then the mixture was poured over crushed ice & little HCl. The product was filtered and recrystallized from ethanol to obtain the compounds (3). The physical data is given in Table 1.

3: IR (KBr): 1650 (C=O), 3412 (-OH), 1155 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.98-7.46 (m, 9H, Ar-H), δ 8.057 (d, 1H =CH), δ 8.099 (d, 1H =CH), δ 9.63 (s, 1H, -OH)

Synthesis of 3 - (1-hydroxy naphthalen - 2 - yl)-5-(furan-2-yl) - 1-substituted pyrazolines (4-8):

1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-ones (0.01 mole) & hydrazine / phenyl hydrazine / semicarbazide / 2,4 dinitro phenyl hydrazine / isonicotinic acid hydrazide (0.01 mole) were added to DMF (20 ml) and refluxed for 2 hours. The cooled reaction mixture was diluted with water & the semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol-acetic acid mixture to get titled pyrazolines in 45-51% yield. The physical data is given in Table 1

4: IR (KBr): 3000 (N-H), 3402 (-OH), 1151 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.90-7.80 (m, 6H, Ar-H), δ 8.051 (d, 1H =CH), δ 8.091 (d, 1H =CH), δ 9.62 (s, 1H, -OH)

5: IR (KBr): 1600 (C=C), 3410 (-OH), 1148 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.89-7.90 (m, 11H, Ar-H), δ 8.049 (d, 1H =CH), δ 8.081 (d, 1H =CH), δ 9.53 (s, 1H, -OH)

6: IR (KBr): 1723 (C=O), 3405 (-OH), 1160 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.81-7.40 (m, 6H, Ar-H), δ 8.041 (d, 1H =CH), δ 8.079 (d, 1H =CH), δ 9.49 (s, 1H, -OH), δ 6.6 (s, 2H, NH₂)

7: IR (KBr): 1530, 1550 (NO₂), 3412 (OH), 1155 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.81-7.60 (m, 9H, Ar-H), δ 8.045 (d, 1H =CH), δ 8.081 (d, 1H =CH), δ 9.43 (s, 1H, -OH)

8: IR (KBr): 1611 (C=O), 3477 (-OH), 1170 (C-O-C) NMR (CDCl₃ + DMSO-d₆): δ 6.95-7.81 (m, 10H, Ar-H), δ 8.043 (d, 1H =CH), δ 8.083 (d, 1H =CH), δ 9.50 (s, 1H, -OH)

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