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Synthesis of Pyrazolopyrazol Derivative *via* Reactions of Donor Compounds with 4-(2-Hydroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one

Ashraf H. Abou-Zied[1*] and Salema A. A. El-Mansory[1]

[1]*Chemistry Department, Faculty of science, Omar Al-Mukhtar University, Al-Beida, Libya*
[*]*Email: Ashrafhz@yahoo.com*

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Abstract
4-(2-hydroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one **3** reacted with hydrazine, phenylhydrazine, semicarbazide, thiosemicarbazide, acylthiosemicarbazide and acetylthiosemicarbazide as donor compounds in ethanol in presence of acetic acid or sulphuric acid by reflux to form pyrazolopyrazole derivative s **4** to **9**, Respectively. These products which have been characterized by FT-IR, ^{1}H-NMR, ^{13}C-NMR, mass spectra and elemental analysis have been produced in good yields.

Keywords: Pyrazole, acythiosemicarbazide, thiosemicarbazide, Pyrazolopyrazol

Introduction

Heterocyclic compound and their derivatives have attracted the attention of chemists, because of broad spectrum biological and pharmacological activities associated with this class of compounds, especially those that have nitrogen, sulphur, and oxygen or the three heteroatoms (Hassan et al., 2004, 2005, and 2012).

The pyrazole ring is a prominent structural motif found in numerous pharmaceutically active compounds (Baraldi et al. 2002). Pyrazole and pyrazolone ring systems represent an important class of compounds (Khodairy, 2007), not only for their

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theoretical interest but also for anti-inflammatory, postmenopausal and osteoporosis antagonists.

Due to the ease preparation and important biological activity (Rao et al., 2012), Pyrazole framework plays an essential role in biologically active compounds (El-Assiery et al., 2004), therefore represents an interesting template for combinatorial as well as medicinal chemistry (Vaghasiya et al, 2008). pyrazoles demonstrate a variety of biological activities such as antibacterial (Trivedi et al, 2008), antifungal, antiviral, antioxidant etc. Moreover, they have played a crucial part in the development of heterocyclic chemistry and used extensively as useful synthon in organic synthesis (Ojha et al., 2008). Accordingly, this has prompted us to synthesis some of the pyrazolopyrazole derivatives starting from available and inexpensive compounds such as ethylacetoacetate, aldehyde derivative, acylthiosemicarbazide, thiosemicarbazide, benzoylhydrazine and hydrazinehydrate (Scheme 1).

Experimental

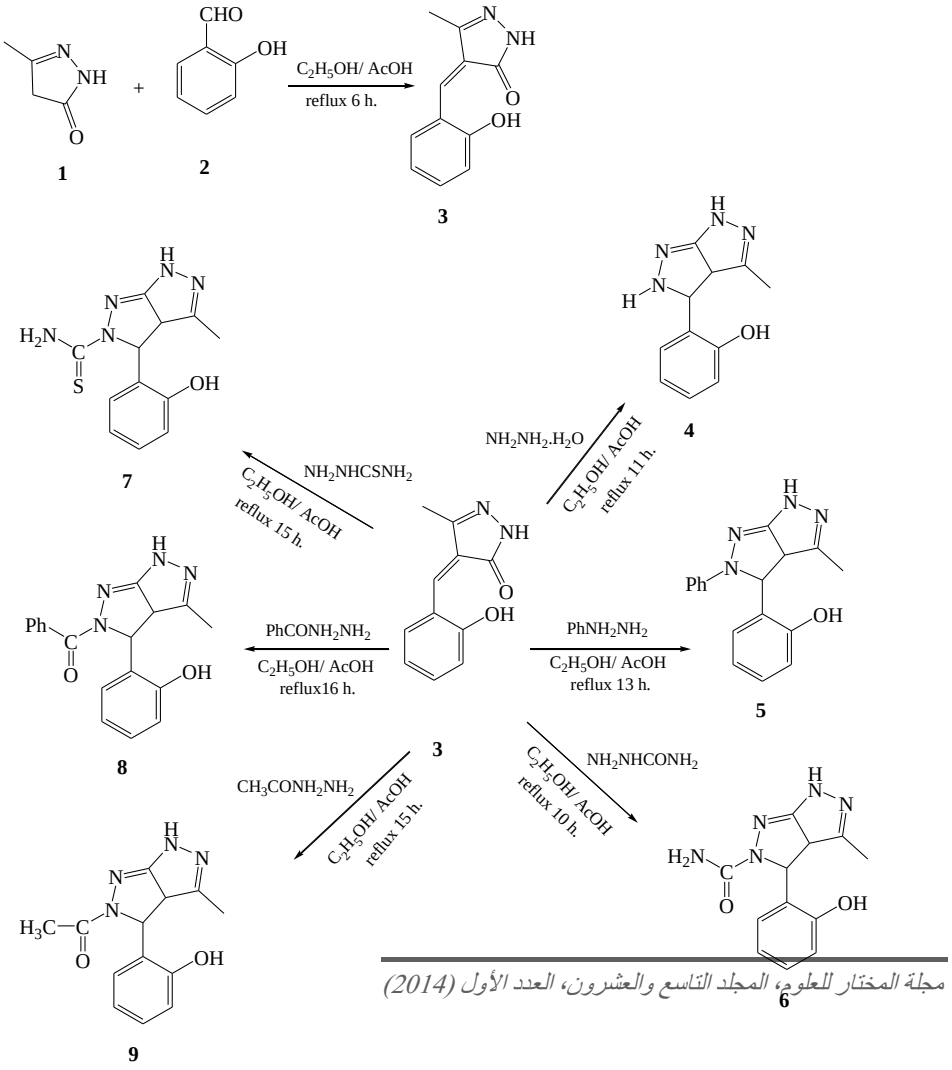
Mp's were determined with a Gallenkamp melting point apparatus and are uncorrected. The IR spectra were recorded with Thermo Nicolet 380 FT-IR spectrometers using potassium bromide pellets. ^{1}H 300 MHz and ^{13}C NMR 75 MHz spectra were recorded on a Bruker WM 300 instrument, 500 MHz ^{1}H and 125 MHz ^{13}C NMR spectra on a Bruker DRX 500 spectrometer. Chemical shifts were expressed as δ [ppm] with reference to tetramethylsilane as an internal standard, s = singlet, d = doublet, m = multiplet. The mass spectra (70 ev, electron impact mode) were recorded on an Shimadzu QP-2010 plus instrument. Elemental analyses were carried out at the micro analytical center, Cairo University, Egypt.

Starting Materials

3-methyl-1H-pyrazol-5(4H)-one **1** was prepared according to Funrniss (Funrniss, 1989), 1-Acylthiosemicarbazides were prepared according to the procedures published in literature (Hassan et al., 2007), hydrazine hydrate, thiosemicarbazide, semicarbazone, phenylhydrazine and 2-hydroxybenzaldehyde were bought from Merck and Aldrich.

Reaction of 5-methyl-2,4-dihydro-3H-pyrazol-3-one 1 with 2-hydroxybenz-aldehyde 2.

A solution of 2-hydroxybenzaldehyde **2** (0.01 mol) in 30 ml of ethanol was added drop wise with stirring at room temperature to 5-methyl-2,4-dihydro-3H-pyrazol-3-one **1** (0.01 mol) in ethanol (20 ml) and the reaction mixture was refluxed for 6 h., in presence of acetic acid (10 ml). After cooling, the mixture was left standing at room temperature, meanwhile a yellow crystalline product separated. The resulting solid was filtered, washed with ethanol, dried and recrystallized from ethanol to give 4-(2-hdroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one **3**.



Scheme 1

4-(2-hdroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one 3. This compound has mp 250-252 °C, Yellow crystals from ethanol, yield 60 %; IR (KBr, cm^{-1}): 3395 (-OH), 3370 (-NH), 3050 (Ar-CH), 3020 (C-H, methine), 2970 (-C-H, CH_3), 1690 (C=O), and 1635 (C=N) cm^{-1}; ^1H NMR (DMSO): δ 2.37 (s, 3H, CH_3); 6.50 (s, 1H, methine), 6.95-8.10 (m, 4H, phenol-H), 9.46 (s, 1H, OH), 11.28 (s, H, NH); ^{13}C NMR: δ 26.42 (CH_3), 126.22 (C-4of pyrazole), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (Ar-C), 144.32 (methine), 147.60 (C-3 of pyrazole), 173.82 (C=O); EI-MS m/z: % 202. Anal. (%) for $C_{11}H_{10}N_2O_2$: Calcd. C, 65.34; H, 4.98; N, 13.85. Found: C, 65.19; H, 5.13; N, 14.00.

Reaction of 3 with donor compounds

General procedures

Each of the donor compounds (hydrazinehydrate, phenylhydrazine, semicarbazide, thiosemicarbazide, benzoylhydrazine, acetylhydrazine) (0.01 mol) was dissolved in 20 ml absolute ethanol with 10 ml of acetic acid or two drop of sulphoric acid and added to a solution of compound **3** (0.01 mol) in 25 ml ethanol. Each mixture was heated under reflux for certain time as follows: hydrazinehydrate 11h, phenylhydrazine 13h, semicarbazide 10 h, thiosemicarbazide 15 h, benzoylhydrazine 16h and acetyl-hydrazine 15h. Each mixture was cooled and left standing for 48h at room temperature, meanwhile a pale yellow or yellow crystalline product separated. The resulting solid material was filtered and the precipitate was washed with ethanol, dried and recrystallized from ethanol to give compounds **4-9** respectively.

Reaction of 3 with hydrazine hydrate to give compound 4.

2-(1,3a,4,5-tetrahydro-3-methylpyrazolo[3,4-c]pyrazol-4-yl)phenol 4. This compound has mp 273-275 °C, Pale yellow crystals from ethanol, yield 78 %; IR (KBr, cm^{-1}): 3410 (-OH), 3395, 3328 (2NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 1635 (C=N) and 1590 (C=C, Ar) cm^{-1}; ^1H NMR (DMSO): δ 2.37 (s, 3H, CH_3), 3.11 (d, 1H, H-3a), 3.34 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 9.12 (s, 1H, OH), 11.18 (s, H, NH) and 11.21 (s, 1H, NH); ^{13}C NMR: δ 26.42 (CH_3), 32 .22 (C-4), 39.20 (C-3a), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (aryl-C), 147.60 (C-3) and 155.50 (C-6a),; EI-MS m/z: % 216. Anal. (%) for $C_{11}H_{12}N_4O$ Calcd. C, 61.10; H, 5.59; N, 25.91. Found: C, 60.91; H, 5.43; N, 26.00.

Reaction of 3 with phenylhydrazine to give compound 5.

2-(1,3a,4,5-tetrahydro-3-methyl-5-phenylpyrazolo[3,4-c]pyrazol-4-yl)-phenol 5. This compound has mp 298-300 °C, Pale yellow crystals from ethanol, yield 78 %; IR (KBr, cm^{-1}): 3410 (-OH), 3395 (-NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 1590 (Ar, C=C) and 1635 (C=N) cm^{-1}; ^{1}H NMR (DMSO): δ 2.37 (s, 3H, CH_3), 3.25 (d, 1H, H-3a), 3.78 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 6.95-8.10 (m, 5, aryl-H), 9.36 (br, 1H, OH), 11.21 (br, H, NH); ^{13}C NMR: δ 26.63 (CH_3), 37.22 (C-4), 39.20 (C-3a), 117.20, 129.60, 135.50, 142,80 (aryl-C), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (phenol-C), 152.50 (C-3) and 155.50 (C-6a); EI-MS m/z: % 292. Anal. (%) for $C_{17}H_{16}N_4O$, Calcd. C, 69.85; H, 5.52; N, 19.17. Found: C, 69.18; H, 5.43; N, 19.30

Reaction of 3 with semicarbazide to give compound 6.

3,3a-dihydro-3-(2-hydroxyphenyl)-4-methylpyrazolo[3,4-c]pyrazole-2(6H)-carbo-xamide 6. This compound has mp 333-335 °C, yellow crystals from ethanol, yield 78 %; IR (KBr, cm^{-1}): 3410 (-OH), 3395, 3225 (-NH_2, NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 1590 (Ar, C=C), 1635 (C=N) and 1660 (C=O) cm^{-1}; ^{1}H NMR (DMSO): δ 2.37 (s, 3H, CH_3), 3.28 (d, 1H, H-3a), 3.78 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 8.16 (s, 2H, NH_2), 9.27 (s, 1H, OH) and 11.33 (s, H, NH); ^{13}C NMR: δ 26.73 (CH_3), 37.22 (C-4), 39.20 (C-3a), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (phenol-C), 152.5 (C-3), 155.50 (C-6a) and 165.33 (C=O), ; EI-MS m/z: % 259. Anal. (%) for $C_{12}H_{13}N_5O_2$, Calcd. C, 55.59; H, 5.05; N, 27.01. Found: C, 56.05; H, 5.23; N, 27.28.

Reaction of 3 with thiosemicarbazide to give compound 7.

3,3a-dihydro-3-(2-hydroxyphenyl)-4-methylpyrazolo[3,4-c]pyrazole-2(6H)-carbo-thioamide 7. This compound has mp 321-323 °C, yellow crystals from ethanol, yield 80 %; IR (KBr, cm^{-1}): 3450 (-OH), 3435, 3220, (-NH_2, NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 1635 (C=N), 1590 (Ar, C=C) and 1360 (-C=S) cm^{-1}; ^{1}H NMR (DMSO): δ 2.37 (s, 3H, CH_3), 3.28 (d, 1H, H-3a), 3.78 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 9.27 (s, 1H, OH), 11.33 (s, H, NH); ^{13}C NMR: δ 26.73 (CH_3), 37.22 (C-4), 39.20 (C-3a), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (phenol-C), 152.5 (C-3), 155.50 (C-6a) and 180.65 (C=S),; EI-MS m/z: % 275. Anal. (%) for $C_{12}H_{13}N_5OS$, Calcd. C, 52.35; H, 4.77; N, 25.43, S, 11.65. Found: C, 51.90; H, 5.35; N, 27.38, S, 11,55.

Reaction of 3 with benzoylhydrazine to give compound 8.

(3a,4-dihydro-4-(2-hydroxyphenyl)-3-methylpyrazolo[3,4-c]pyrazol-5(1H)-yl) (phenyl)methanone 8. This compound has mp 362-365 °C, yellow crystals from ethanol, yield 66 %; IR (KBr, cm^{-1}): 3435 (-OH), 3240 (NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 1590 (Ar-C=C), 1610 (-C=O) and 1635 (C=N) cm^{-1}; ^1H NMR (DMSO): δ 2.33 (s, 3H, CH_3), 3.25 (d, 1H, H-3a), 3.88 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 6.95-8.10 (m, 5H, Ar-H), 9.27 (s, 1H, OH) and 11.33 (s, H, NH); ^{13}C NMR: δ 26.73 (CH_3), 37.22 (C-4), 39.20 (C-3a), 117.20, 129.60, 135.50, 142,80 (aryl-C), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (phenol-C), 152.50 (C-3), , 155.50 (C-6a) and 165.34 (C=O); EI-MS m/z: % 320. Anal. (%) for $C_{18}H_{16}N_4O_2$, Calcd. C, 67.47; H, 5.05; N, 17.49. Found: C, 67.57; H, 4.95; N, 17.38.

Reaction of 3 with acetylhydrazine to give compound 9.

1-(3a,4-dihydro-4-(2-hydroxyphenyl)-3-methylpyrazolo[3,4-c]pyrazol-5(1H)-yl) ethanone 9. This compound has mp 192-194 °C, Pale yellow crystals from ethanol, yield 66 %; IR (KBr, cm^{-1}): 3410 (-OH), 3240, (NH), 3060 (Ar-H), 2970 (-C-H, CH_3), 2880 (C-H, CH_3-CO), 1635 (C=N), 1630 (-C=O) and 1590 (Ar-C=C) cm^{-1}; ^1H NMR (DMSO): δ 2.33 (s, 3H, CH_3), 3.10 (s, 3H, CH_3-CO), 3.25 (d, 1H, H-3a), 3.88 (d, 1H, H-4), 6.50-7.80 (m, 4H, phenol), 9.27 (s, 1H, OH), 11.33 (s, H, NH); ^{13}C NMR: δ 26.73 (CH_3), 35.45 (CH_3-CO), 37.22 (C-4), 39.20 (C-3a), 126.92, 126.91, 127.44, 128.28, 132.11, 132.34 (phenol-C), 152.50 (C-3), 155.50 (C-6a), 165.34 (C=O); EI-MS m/z: % 258. Anal. (%) for $C_{13}H_{14}N_4O_2$, Calcd. C, 60.46; H, 5.45; N, 21.79. Found: C, 60.37; H, 5.05; N, 21.67.

Result and discussions

4-(2-Hdroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one **3** can be formed by reaction of 5-methyl-2,4-dihydro-3H-pyrazol-3-one **1** (0.01 mol.) with 2-hydroxybenzaldehyde (0.01 mol.) in ethanol as a solvent in presence of acetic acid. Structural assignment of products **3** is based on spectral data and on combustion analysis. The elemental analysis of **3** supports the gross formula $C_{11}H_{10}N_2O_2$, and the mass spectrum gives a correct molecular ion at m/z 202 (11 %). The IR spectrum of **3** shows NH absorption bands at ν = 3370, 3050 (=C-H, Ar), 2970 (-C-H, CH_3), 1635 (-C=N), 1690 (-C=O), -OH group at 3395 cm^{-1}. The ^1H-NMR spectrum reveals one broad signals at δ = 11.28 related to pyrazole-NH, broad single at 2.37 for C-H of

CH_3, a broad singlet for C-H of methine, and at 9.46 abroad signal appears due to OH group. The ^{13}C-NMR spectrum of **3** confirms the previous ^{1}H-NMR spectral data by the appearance of signals at 26.42 (CH_3), 147.60 (C-3), 126.22 (C-4), 144.32 (=C-H, methine), 173.82 (C=O) and 129.91, 127.44, 128.28, 132.11, 132.34 (aryl-c).

The mixture of compound **3** with one molar equivalents of hydrazine hydrate in ethanol as solvent at room temperature in presence of acetic acid was stirred for 3h at room temperature. This mixture was heated under reflux for 15h, cooled to room temperature. The mixture was left standing for 48h at room temperature, meanwhile a pale yellow crystalline product separated. The resulting solid was filtered, washed with ethanol, dried and recrystallized from ethanol to give 2-(1,3a,4,5-tetrahydro-3-methylpyrazolo[3,4-c]pyrazol-4-yl)phenol **4** .

The IR spectrum of **4** shows absorption bands characteristic of NH groups at 3395, 3210, strong OH group at 3410, aryl hydrogen absorption at 3060, methyl hydrogen absorption at 2970 in addition to C=N absorption at 1635 cm^{-1}. ^{1}H-NMR spectrum of **4** clearly supports the presence of two different broad signals centered at δ 11.21 and 11.18 ppm due to the two pyrazol-NH. There is abroad signal at 9.12 ppm due to presence of OH. In the ^{13}C-NMR spectrum the methyl groups of pyrazole and C-3 resonate at δ = 2.37 and 147.60 ppm, respectively. Further peaks at 32.22 ppm (C-4), at 39.20 (C-3a) and 155.50 ppm (C-6a), besides the aromatic carbons support the assigned structure. Elemental analysis of **4** suggests a gross formula $C_{11}H_{12}N_4O$. This is also confirmed by the mass spectrum which exhibited the molecular ion at m/z 216 (19 %).

The IR spectrum of **4** shows absorption bands characteristic of NH groups at 3395, 3210, strong OH group at 3410, aryl hydrogen absorption at 3060, methyl hydrogen absorption at 2970 in addition to C=N absorption at 1635 cm^{-1}. ^{1}H-NMR spectrum of **4** clearly supports the presence of two different broad signals centered at δ 11.21 and 11.18 ppm due to the two pyrazol-NH. There is abroad signal at 9.12 ppm due to presence of OH. In the ^{13}C-NMR spectrum the methyl groups of pyrazole and C-3 resonate at δ = 2.37 and 147.60 ppm, respectively. Further peaks at 32.22 ppm (C-4), at 39.20 (C-3a) and 155.50 ppm (C-6a), besides the aromatic carbons support the assigned structure. Elemental analysis of **4** suggests a gross formula $C_{11}H_{12}N_4O$. This is also confirmed by the mass spectrum which exhibited the molecular ion at m/z 216 (19 %).

Compound **5** shows a characteristic pale yellow color. The gross formula of **5** was confirmed by the mass spectrum, which exhibited the molecular ion at m/z 292 (9 %).

The IR spectrum shows absorption at 3395 (NH), 3410 (OH), and 1635(C=N) cm^{-1}. The ^{1}H-NMR spectrum of **5** displays one broad signals at 11.21 ppm for pyrazole-NH, in addition to the aromatic protons. In ^{13}C-NMR spectrum C-3, C-3a, C-4 and C-6a resonate at δ = 151.50, 39.20, 37.22 and 155.50 ppm, respectively; further peaks are at δ = 26.63 (CH_3) and aromatic carbon.

Structural assignment of product **6** is based on spectral data and on combustion analysis. The elemental analysis of **6** supports the gross formula $C_{12}H_{13}N_5O_2$, and the mass spectrum gives a correct molecular ion at m/z 259 (11 %). The IR spectrum of **6** shows NH_2 and NH absorption bands at υ = 3395 and 3320, carbonyl group at 1660 cm^{-1}. The ^{1}H-NMR spectrum reveals two broad signals with the ratio 2:1 at δ = 8.16, 11.33 related to NH_2, pyrazole-NH, respectively. The ^{13}C-NMR spectrum of **6** confirms ^{1}H-NMR spectral data by the appearance of signals at 26.73 (CH_3), 152.50 (C-3), 37.22 (C-4), 39.20 (C-3a), 151.50 (C-6a), and 165.33 (CO).

The IR spectra of **7** in KBr disc shows absorption characterized NH_2, NH groups at 3435, 3220, and OH group at 3410 cm^{-1}. The ^{1}H-NMR spectrum of **7**clearly shows the presence of aryl protons and pyrazol-NH and NH_2. The ^{13}C-NMR of **7** shows signals at 26.73, 152.50, 37.22, 39.20 and 155.50 due to CH3, -C-3, C-4, C-3a and C-6a, respectively and at 180.65 for (C=S), in addition to the aryl carbons. The molecular formulae of compound **7** is supported by elemental analysis and mass spectra which give the expected molecular ion peaks. Structural assignment of products **8, 9** is based on spectral data and on combustion analysis (Experimental section).

Conclusion

The reactions and the heterocyclic products here provide insight into the reactions between the electron donating (hydrazine, phenyl hydrazine, semicarbazide, thiosemicarbazide, acylthiosemicarbazide and acetylthiosemicarbazide) and 4-(2-hydroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one **3** to form pyrazolopyrazole derivatives expected biological activity. Thus, electron donor may act as a mediator and as a building block in heterocyclization of 4-(2-hydroxybenzylidene)-3-methyl-1H-pyrazol-5(4H)-one **3**. The results reported here supplement the rich chemistry of donor compound and **3**.

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تخليق مشتقات البيرزولو بيرزول من خلال التفاعل بين المركبات المانحه للالكترونات و 4-(2-هيدروكسى بنزاليدين)-3-ميثيل-H1-بيرازول-5(H4)-اون

اشرف حسن ابوزيد و سليمه المبروك عبدالمولى المنصورى

الملخص

تفاعل 4-(2-هيدروكسى بنزاليدين)-3-ميثيل-1H-بيرازول-5(H4)-اون مع الهيدرازين هيدريد، الفنيل هيدرازين، سيمى كاربازيد، الثيوسيمى كاربازيد، البنزويل هيدرازين، بالإضافة إلى الأسيتيل هيدرازين كمركبات مانحه للالكترونات في الإيثانول في وجود حمض الخليك أو الكبريتيك بالتسخين ليتكون مشتقات البيرازولوبيرازول 4-9 بالتتابع. هذه النواتج تم تحديد التركيب الكيميائي لهذه المركبات الجديدة باستخدام الأشعة تحت الحمراء، الرنين النووي المغناطيسي لنواتي ذرة الهيدروجين والكربون 13، مطياف الكتلة والتحليل الدقيق لعناصر الكربون والهيدروجين والنيتروجين وهذه النواتج تم الحصول عليها بكميات جيدة.

مفتاح الكلمات: البيرزول، اسيل ثيو سيمى كاربازيد، ثيو سيمى كاربازيد، بيرازولو بيرزول