

Preparation of New Pyrazol Derivatives

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ABSTRACT

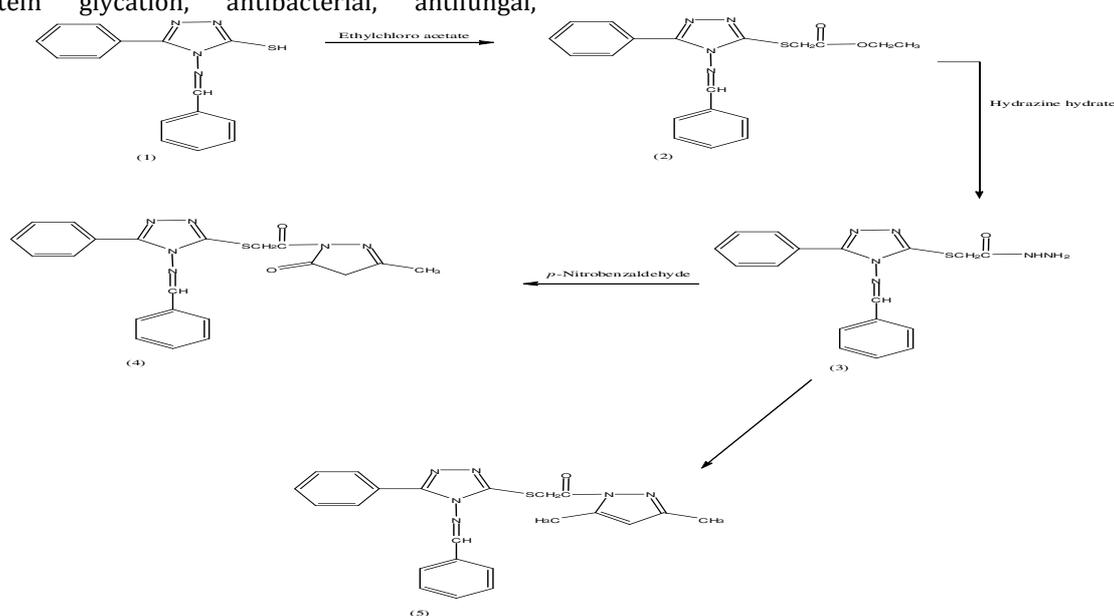
Pyrazole (4) and pyrazolone are considered a pharmacologically important active scaffold that possesses almost all types of pharmacological activities, they have been synthesized from the reaction of 2-(4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazol-3-ylthio)acetohydrazide (3) with ethylchloroacetate and acetylacetone respectively in the presence of acetic acid as catalyst, hydrazide (3) have been synthesized from nucleophilic substitution of ester (2) with hydrazine hydrate, compound (2) which result from the reaction of triazole with ethylchloro acetate, the products were characterized by Infrared spectroscopy.

Keywords: Pyrazole, pyrazolone, derivatives, synthesis, hydrazide.

1. INTRODUCTION

Pyrazoles are five-membered heterocycles that constitute a class of compounds particularly useful in organic synthesis. They are one of the most studied groups of compounds among the azole family. Indeed, a huge variety of synthesis methods and synthetic analogues have been reported over the years. The presence of the pyrazole nucleus in different structures leads to diversified applications in different areas such as technology, medicine and agriculture. In particular, they are described as inhibitors of protein glycation, antibacterial, antifungal,

anticancer, antidepressant, antiinflammatory, anti-tuberculosis, antioxidant as well as antiviral agents [1,2]. Nowadays, pyrazole systems, as biomolecules, have attracted more attention due to their interesting pharmacological properties. This heterocycle can be traced in a number of well-established drugs belonging to different categories with diverse therapeutic activities [3-10]. The structures of the pyrazole and pyrazolone synthesized from 2-(4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazol-3-ylthio)acetohydrazide are shown in scheme 1:



Scheme - 1: Synthetic pathway for compounds (4) and (5).

2. Experimental

2.1. Materials

All the reagents used were of analar or chemically pure grade. Solvents were purified and dried according to standard procedures.

The compound (1) was prepared according to the method of work in reference [11].

2.2. Synthesis of ethyl 2-((4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazole-3-yl)thio)acetate(2):

Compound (1) (1g, 0.003 mol) was dissolved in ethanol (20ml) The stirrer is started and (0.414g,0.003mol) of K_2CO_3 was added,after cooling the (0.48ml) of ethylchloro acetate was added drop by drop.After cooling, the product was filtered,dried and recrystallized from ethanol. [12]

2.3. Synthesis of 2-((4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazole-3-yl)thio)acetohydrazide(3)

Ester (1) (3g, 0.007 mol) was dissolved in ethanol (25 ml) after that (0.36 ml) of hydrazine hydrate was added drop by drop, the mixture was refluxed. After cooling, the product was filtered, dried and recrystallized from ethanol. [13]

2.4. 1-(2-(4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazol-3-ylthio)acetyl)-3-methyl-1H-pyrazol-5(4H)-one (4)and 2-(4-(benzylideneamino)-5-phenyl-4H-1,2,4-triazol-3-ylthio)-1-(3,5-dimethyl-1H-pyrazol-1-yl)ethanone (5):

Compound (3)(0.25 g, 0.0079 mole) was treated with (ethylacetoacetate or acetylacetone) respectively, acetic acid (0.5 ml) in absolute ethanol (10 ml) was heated under reflux for (7 hours). The reaction mixture was cooled and the formed precipitate was filtered off to give the final product. [14]

3. RESULTS AND DISCUSSION

Scheme (1) that shown the synthesized of compounds (2), (3), (4) and (5).Name, Chemical formula, molecular weight, color, melting points and yields of. FT-IR spectraldata are also shown in Table 2.

Table - 1: Physical Properties for Compounds (1), (2), (3), (4) and (5)

No.	Chemical Formula	Color	Melting Point (°C)	Yield (%)
(2)	$C_{19}H_{19}O_2N_4S_2$	Yellow	190-192	67
(3)	$C_{17}H_{16}N_6OS$	Pale yellow	238-240	60
(4)	$C_{21}H_{18}N_6O_2S$	Brown	205-208	56

(5)	$C_{22}H_{20}N_6OS$	Brown	188-190	52
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3.1. FT-Infrared spectra

From the recorded IR spectrum of compound (2), the wave numbers of corresponding groups are; 3096 cm^{-1} (CH aromatic), 2963 cm^{-1} (CH aliphatic), 1736 (CO ester), 1602 (brod band, overlape C=N imine and CO amide).

IR spectrum of compound (3), the wave numbers of corresponding groups are; 3263- 3200 cm^{-1} (NH_2), 3056 cm^{-1} (CH aromatic), 2918 cm^{-1} (CH aliphatic), 1679 cm^{-1} (CO amide), 1621 and C=N imine).

IR spectrum of compound (4), the wave numbers of corresponding groups are; disappearance band at 3333 cm^{-1} of NH_2 amine, and appearance band at 3166 (NH amide), 3078 cm^{-1} (CH aromatic), 2904 and 2849 cm^{-1} (CH aliphatic), 1665 cm^{-1} (overlape CO amide and C=N imine).

IR spectrum of compound (5), the wave numbers of corresponding groups are; disappearance band at 3333 cm^{-1} of NH_2 amine, and appearance band at 3101 cm^{-1} (NH amide), 3024 cm^{-1} (CH aromatic), 2929 and 2830 cm^{-1} (CH aliphatic), 1686 cm^{-1} (overlape CO amide and C=N imine).

4. CONCLUSION

Pyrazole and pyrazolonewas synthesized andcharacterized spectral techniques. These compound exhibited significant activity against allthe tested microorganisms.

5. REFERENCES

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