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СИНТЕЗ НОВОЙ ЛИГАНДА 2- (5-МАРКАПТО-1,3,4-ОКСАДИАЗОЛА) ФЕНОЛА

Аннотация: подготовлен новый лиганд (L) L = 2- (5-маркапто-1,3,4-оксадиазол) фенол был синтезирован конденсацией этил 2-гидроксибензоат с гидразингидратом и сероуглеродом в присутствии КОН. Лиганд охарактеризован и исследованы спектральными методами ИК, ¹H-ЯМР и ¹³C-ЯМР, результаты были совместимы с предложенной структурой.

Ключевые слова: Лиганд, оксадиазол, гидроксибензоат.

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SYNTHESIS OF NEW LIGAND OF 2-(5-MERCAPTO-1,3,4-OXADIAZOL) PHENOL

***Annotation:** A new ligands $L = [2-(5\text{-mercapto-1,3,4-oxadiazol-2-yl})\text{ phenol}]$; was synthesized by condensation of ethyl 2-hydroxybenzoate with hydrazine hydrate and carbon disulfide in presence KOH.*

The ligand was characterized and studied by spectral methods of FT-IR, and $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, the results was compatible with the proposed structure.

***Key Words:** Ligand, oxadiazol, hydroxybenzoate.*

1. Introduction:

Heterocyclic compounds have occupied a prominent place among various classes of organic compounds by virtue of their diverse biological activities and chemistry [1,C.337]. 1, 3, 4-Oxadiazole derivatives are the heterocyclic that have received considerable attention during the last two decades, they found in number of pharmaceutical applications [2,C.197]. 1,3,4-oxadiazole derivatives exhibit wide range of biological activities. These activities are probably due to the presence of the $\text{N}=\text{C}-\text{O}$ group [3,C.13] Their derivatives have been known to possess antibacterial and antimicrobial [4,C.37].

2. Experimental

2.1. Apparatus and chemicals:

Materials: Salicylic acid, carbon disulfide, hydrazine hydrate, , ethanol, potassium hydroxide, dichloromethane, methanol (all from Sigma-aldrich), sodium hydroxide (BDH), anhydrous sodium sulfates (LOBAL Chemic), distilled water.

Instrumentation:

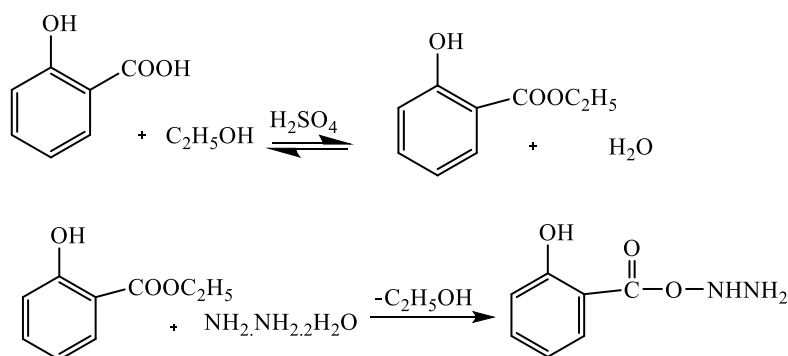
^1H NMR, ^{13}C NMR spectra were recorded on a (Bruker AVANCE) 400 MHz spectrometers and $\text{CDCl}_3\text{-D}_1$ was used as NMR solvent, with TMS as an internal standard. FT-IR spectra were recorded on a Jasco FT-IR 4100 and Shimadzu obtained by the KBr disk method.

2.2. Experimental Procedure:

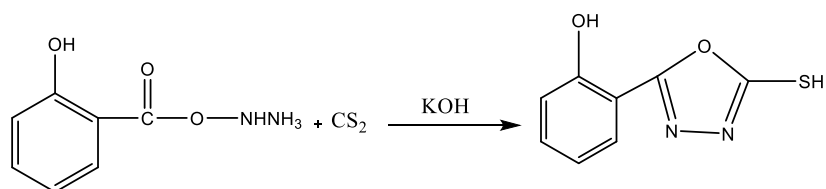
2.2.1. Synthesis of ligand 5-(2-Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole:

Distance preparation of Ester from the Salicylic acid with absolute Ethanol in the presence some drops from concentrated sulfuric acid.

A mixture of 15.2 ml ethyl salicylate and 10 ml of hydrazine hydrate were refluxed in 50 ml of 95% of absolute ethanol for 24 h. The resultant mixture was concentrated, cooled, and poured in crushed ice. solid mass thus separated out was filtered, dried, and recrystallized from methanol. Thus it is obtained of 2-hydroxy benzohydrazide.



A mixture of (1.52 g) of 2-hydroxy benzohydrazide, 0.01 mol (0.56 g) of potassium hydroxide, and 10 ml of carbon disulfide were refluxed in 30 ml of 95% absolute ethanol for 12 h. The resultant mixture was concentrated and cooled at room temperature. Then, it was acidified with diluted HCl. The solid mass thus was separated out was filtered, dried, and recrystallized from ethanol. (yield 64%, melting point = 205-207 °C).



3. Results and Discussion:

3.1. ¹H-spectroscopic measurements:

Figure 1. Explanation of ¹H-NMR (ppm) of the 5-(2-Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole in Dimethyl Sulfoxide.

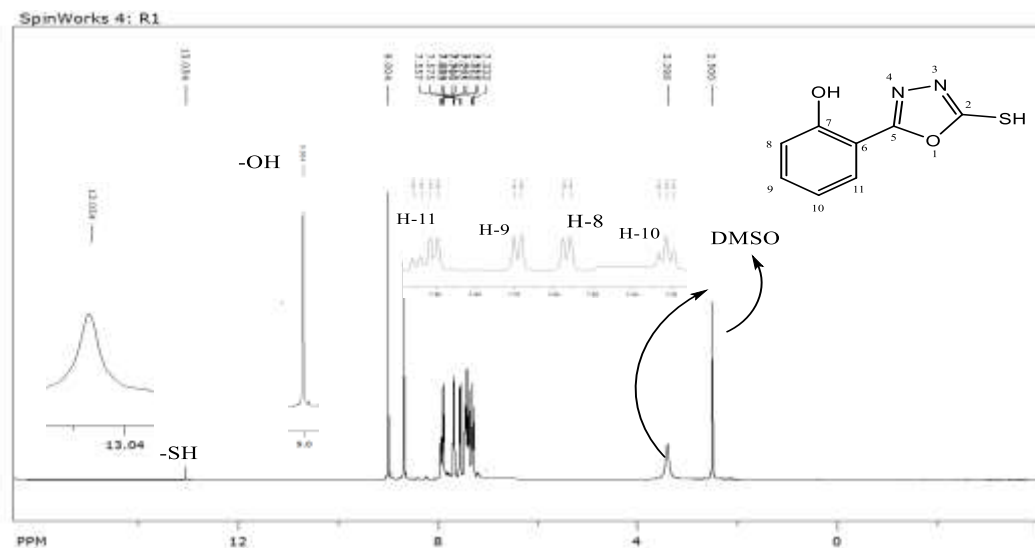
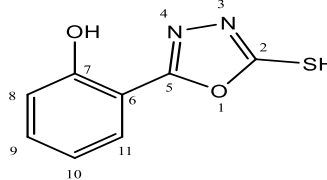


Table 1 Explanation of ¹H-NMR (ppm) of the 5-(2-Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole.

	
H-NMR(δ ,ppm)	NO
-	1
-	2
-	3,4
-	5

-	6
-	7
7.58-7.59 (d,1H,J=4Hz)	8
7.56-7.70 (dd,1H,J ₂ = J ₂ =8Hz)	9
7.30-7.33 (t,1H, J=7.2Hz)	10
7.89-7.96 (dd,1H,J ₁ =7.6, J ₂ =8Hz)	11

3.2. ¹³C-spectroscopic measurements:

Figure 2. Explanation of ¹³C -NMR (ppm) of the 5-(2- Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole.

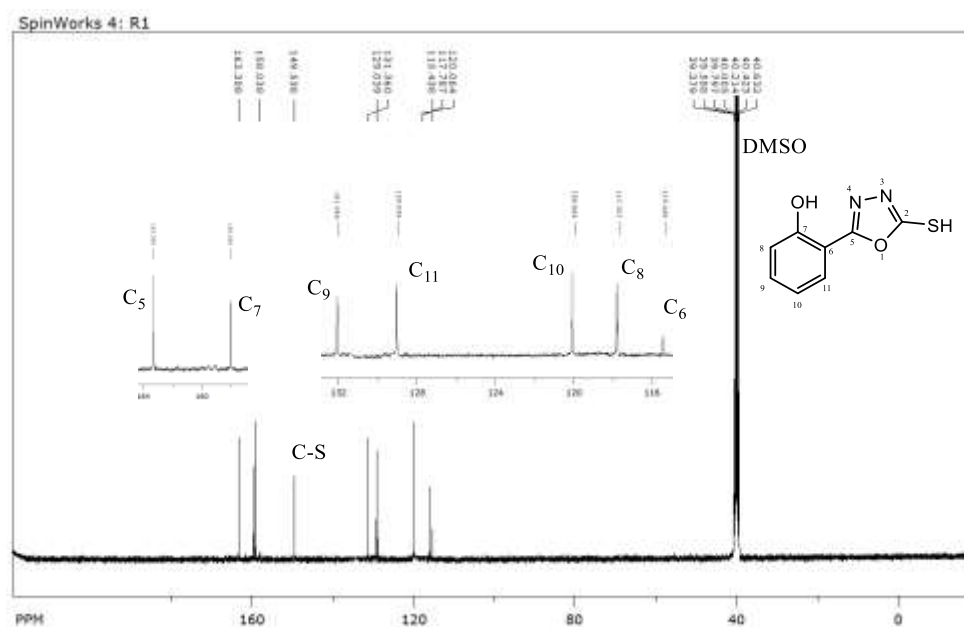


Table 2 Explanation of ^{13}C -NMR (ppm) of the 5-(2- Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole.

Chemical Shift Ppm	NO	Chemical Shift Ppm	NO
158.03	7	-	1
131.36	9	149.54	2(C-S)
120.06	10	163	5
129.04	11	115.44	6

3.3 Infrared Spectra:

The infrared spectra for the present compounds taken in the range 400-4000 cm^{-1} help to indicate regions of absorption vibrations. The main stretching modes are for $\nu(\text{C}=\text{N})$ and $\nu(\text{O}-\text{H})$.

Figure 3. FT-IR absorption spectra of ligand (L).

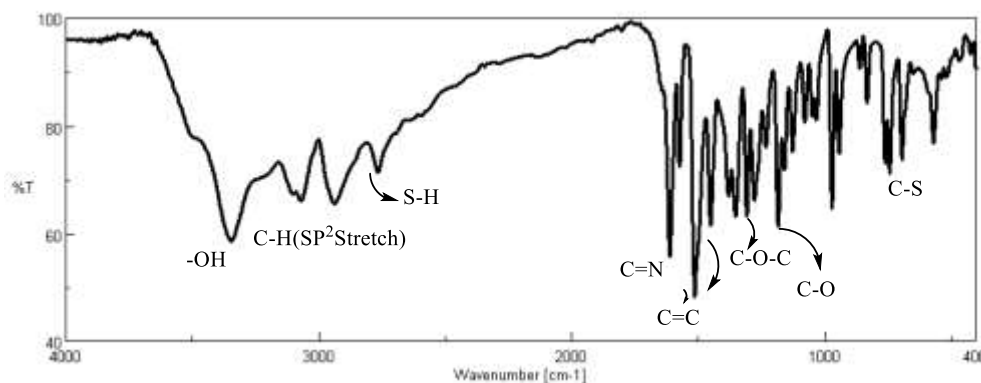


Table 3 Explanation of IR of the 5-(2- Hydroxyphenyl)-2-mercapto-1,3,4-oxadiazole.

compou nd	$\nu(\text{O}-\text{H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{C})$	$\nu(\text{C}-\text{H})\text{SP}^2$	$\nu(\text{N}-\text{N})$	$\nu(\text{C}-\text{O}-\text{C})$	$\nu(\text{C}-\text{O})$	$\nu(\text{S}-\text{H})$	$\nu(\text{C}-\text{S})$
L	334 4 _{st}	1611 _s t	1450- 1573 _{st}	3069 _w	1078	1277	118 3	2765	741

4. Conclusion:

The synthesis of a new ligand (L) [2-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol]; was reaction of by condensation of ethyl 2-hydroxybenzoate with hydrazine hydrate and carbon disulfide in presence KOH to lead ligand (L).

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