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Synthesis of 1,3,4 oxadiazol and some derivative with Cr, Zn, V, Ag

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ABSTRACT

Background: Heterocyclic A heterocyclic compound or ring structure is a cyclic compound that has atoms of at least two different elements. Heterocyclic chemistry is the branch of organic chemistry dealing with the synthesis, properties, and applications of these heterocycles. **Organometallic** is the study of chemical compounds containing at least one chemical bond between a carbon atom of an organic compound and a metal, including alkaline, alkaline earth, transition metal, and other cases. Moreover, some related compounds such as transition metal hydrides and metal phosphine complexes are often included in discussions of organometallic compounds. The field of organometallic chemistry combines aspects of traditional inorganic and organic chemistry. **Objective** synthesis of Oxadiazol and some it's derivatives .Oxadiazol derivatives have been prepared with **Cr , Zn , V , Ag** and then theses derivatives have been diagnosed by using elemental analysis (C, H, N, S and M) and spectroscopic (IR, H^1 – NMR and UV- Visible), magnetic study and molar conductivity. **Results** many chemical compounds have been prepared for Oxadiazol and with some transition elements to give some important derivative compound with Oxadiazol and its entry into the treatment of many diseases. **Conclusions**

- Oxadiazol is a center for the synthesis of many important organo metallic compounds .
- The therapeutic effectiveness of the oxadiazol compounds has a strong effect for the treatment of diseases .
- Oxydiazol derivatives are fixed compounds and can be used in many preparation reactions .
- Physical properties measured for compounds made from oxadiazol and their derivatives show the degree of stability of these compounds chemically

The compounds derived from oxadiazol with the transitional elements have a strong effect in the treatment of many diseases as well as are the center for the preparation of many pharmaceuticals .

INTRODUCTION

Oxadiazol are penta cycle compounds include 2C & 2N & 1O and the lest derivative from Furan that write to replacement two from methylen group with 2N as pyridine and there are four isomers type oxadiazol (Rashidi, N.A. and B.N. Berad, 2013; Catalin, V., *et al*., 2013; Rashidi, N.A. and B.N. Berad, 2013)

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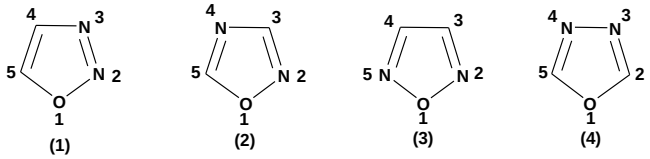
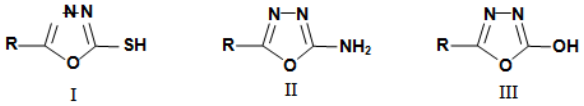


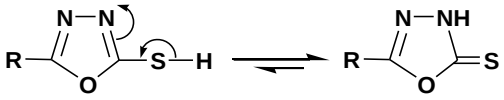
Type 4 more stable from the thermal effect and this stability give to take it inter all research (Rakesh, S., 2014; Hetzhaim, A. and K. Moee, 1966)

The greatest concern of the biological agent, especially the pharmacological importance of this compound, lies in the effective use of cystic fibrosis treatment (Jones, A., R. Helm, 2009). For the treatment of HIV, it is clear that some oxadiazol derivatives have a significant effect on diabetes, obesity (Lee, S.H., *et al*., 2008), inflammation (Mrityunjoy, K., *et al*., 2012) and anticancer (Zhang, H.Z., *et al*., 2005; Salem, D., *et al*., 2004). There are compounds containing 1, 3, 4 - oxadiazol, which are biologically effective, for example antimicrobial, antifungal, antiviral and antifungal, as well as important and useful in medical chemistry as an alternative to carboxylic acids and esters (Bostrom, J., *et al*., 2012)

The symmetric chemistry of the complex elements of the heterogeneous cyclic compounds, including the oxadiazol legand, which have been of interest to many researchers in recent years, has been attracted by the fact that these lycandes are related to the central element ions in natural systems where the complexities exhibited significant efficacy compared to lycand (Mohmoud, N., *et al*., 2013; Ibrahem, S., *et al*., 2008). Three major derivatives of 1, 3, 4 - oxadiazol (I, II, III) (Katritzky, A.R., 1986) were prepared. These three derivatives are:



A large number of compounds were prepared depending on the nature and type of the compensated aggregates at sites 2-5 in the homogeneous ring (Dhasay, D., *et al*., 2010; Hassan, A., *et al*., 1996; Naja,t Al-Obaidy, *et al*., 2006; Yadar, L., *et al*., 1996) Our interest was focused on our compounds because of the high bioavailability of the thiol group and the ability of lycand to form complexes with many transitional element ions. Loops 1, 3, and 4 - oxadiazol can be found in two totemrin forms (a, b) and can be transformed from one shape to the other depending on the reaction conditions.



Procedure:

Preparation of n - propylhydroxy benzoate derivatives (Kadhim, K., et al., 2001)

In a 100 mL circular flask, blend 0.05 ml of hydroxy derivative of benzoic acid with 75 ml of pure ethanol with 6 mL concentrated sulfuric acid. Mix the mixture for 8 hours. After finishing, add the excess ethanol. Add the remaining mixture, which is hot to 40 mL of iced water. The precipitate is then filtered and re-crystallized with cold methanol.

preparation of phenol hydrazine benzoate derivatives from ester n- propyl (Vogel, A.I., 1989):

Mix (0.03) mol of ester derivative with (0.06 mol, 20 ml) hydrazine water in a flask of capacity (150) ml. The mixture is increased for 4 hours and then 20 mL of ethanol is removed and it is repeated for 2 hours to complete the hydrazid formation. Drain the excess ethanol, cool and add to 20 mL of iced water, leave for 1 hour in the refrigerator and then filter the precipitate, wash with cold methanol and re-crystallize with ethanol.

preparation phenol for 4-(5-mercabto 1,3,4 – oxadiazol -2- yl) derivatives (Yassen, A., N. Al-Masoudi, 2003; El- Masary, A.H., et al., 2000; Abdel Kader, M., et al., 2003):

Mix (0.04) ml of the hydrazide derivative and (0.04 mol, 2.2 g) dissolved potassium hydroxide in a small amount of distilled water with 50 mL of pure ethanol and 0.04 mL 4 ml of carbon diprosulfide (CS2) Add a drop-drop with continuous stirring on a cool water bath. After completing the addition (CS2), the mixture will rise for 6 hours. Carbon-sulfide gas is detected to ensure that reaction occurs with lead acetate paper. Distill the excess ethanol and add the remaining to iced water and pour the mixture into pH (2) using concentrated hydrochloric acid (1N). The precipitate is filtered, dried and reconstituted with a mixture (water - ethanol).

preparation of complex (C10 – C1):

The complex is prepared in two ways either by mixing, stirring or refluxing the absolute ethanol solvent.

preparation of complex by refluxing (C1,C2,C5,C8) (Bassam, A.H., 2014):

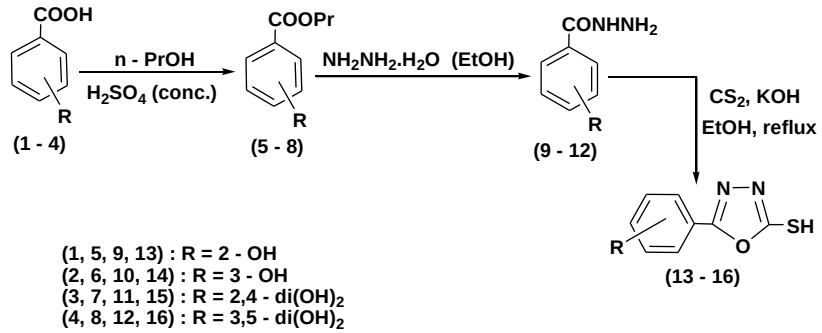
Mixed one of the nitrate elements (Cr + 2, Zn + 2) in 10 mL of ethanol with 2 mol molars in a circular flask and then mix for 5 hours. After finishing, cool the mixture and filter the precipitate and wash several Times with distilled water to dispose of excess salts and re-crystallize with absolute ethanol.

preparation of complex by stirring (C3,C4,C6,C7,C9,C10) (Moslem, H.M., 2010):

Mix 1 mic of component nitrate (V+ 2, Ag + 2) in 10 mL of ethanol with molten melanin. Leave stirring for 6 hours at room temperature. After stirring, precipitate, precipitate and recrystallize Mix (ethanol - water).

RESULT AND DISSECTIONS

Organic composites prepared by fusion measurement were identified (C,H,N,S) (FT-IR, H1-NMR) The complexities were diagnosed by precise analysis of the elements (HN S and M), molar conductivity and spectral methods (FT-IR, UV- Visible) and magnetic sensitivity.



Precise analysis of elements was used to determine the composition of organic compounds and complexities as follows:

Precise analysis of elements for organic compound C . H . N:

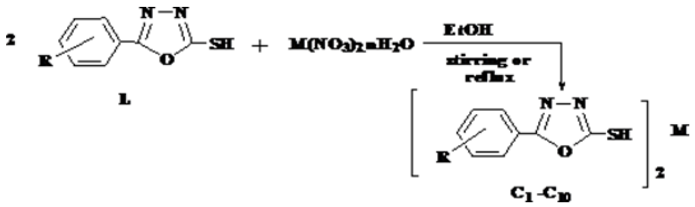
The elements were analyzed carefully and as in Table (3-1) where the results gave practical values similar to the calculated theoretical results. This indicates the validity of the chemical structures of the prepared organic compounds

Table 3-1: Physical properties and values of accurate analysis of elements of organic compounds

Comp. No.	Yield %	M.P. °C	(cal./found) %			
			C	H	N	S
9	88	146 - 148	55.24 55.67	5.30 5.10	18.41 18.12	-
10	83	217 - 219	55.24 55.58	5.30 5.25	18.41 18.22	-
11	80	260 - 263	55.24 56.50	5.30 5.06	18.41 18.23	-
12	59	253 - 256	55.24 54.81	5.30 5.11	18.41 18.46	-
13	79	198 - 202	49.46 50.11	3.12 3.19	14.42 14.22	16.50 15.75
14	69	220 - 223	49.46 49.65	3.12 3.28	14.42 14.36	16.50 16.33
15	88	223 - 225	49.46 49.10	3.12 3.23	14.42 15.11	16.50 16.05
16	6	226 – 227	49.46 49.50	3.12 3.10	14.42 14.52	16.50 16.35

Precise analysis of elements for complex:

The precise analysis of the elements and measurement of the physical properties of the prepared and conductive conductivity was performed. The results were good and are shown in Table (3.- 2.)



C_1: R= 2- OH , M = Cr ; C_2: R=2- OH , M = Zn ; C_3: R= 3-OH , M = V

C_4: R = 3 – OH , M = Ag ; C_5 = R 2,4 – di(OH)$_2$, M=Cr ; C_6 : R=2,4 – di(OH)$_2$,M=V

C_7: R=2,4 – di(OH)$_2$, M=Ag ; C_8: R=3,5 – di(OH)$_2$, M=Cr

C_9: R = 3,5 – di(OH)$_2$, M=V ; C_{10}: R = 3,5 – di(OH)$_2$, M = Ag

Table 3-2: Physical properties and values of accurate analysis of elements of complex

Com. No.	Yield %	M.P. ^{o}C	Color	Condu. Ω^{-1}.M^2	(cal./found) %				
					C	H	N	S	M
C_1	40	298-300	Beige	4.6	35.79 35.71	2.98 2.88	10.43 10.40	11.93 11.91	20.95 20.81
C_2	55	Over 311	Brown	8.7	39.77 39.68	3.31 3.25	11.60 11.51	13.25 13.15	12.15 12.00
C_3	80	262-264	Dark gray	9.3	39.38 39.28	3.28 3.11	11.48 11.35	13.12 13.10	13.03 13.00
C_4	36	Over 312	Dark violet	5.2	40.01 40.00	3.33 3.17	11.67 11.44	13.33 13.22	11.63 11.55
C_5	48	298-300	Light yellow	6.8	35.79 35.68	2.98 2.78	10.43 10.59	11.93 11.87	20.95 20.87
C_6	71	Over 310	Dark blue	7.1	39.38 39.19	3.28 3.35	11.48 11.37	13.12 12.89	13.03 13.11
C_7	50	232-233	Beige	6.9	40.01 40.19	3.33 3.25	11.67 11.80	13.33 13.25	11.63 11.56
C_8	50	231-232	Light brown	8.1	33.77 33.65	2.81 2.78	9.85 9.79	11.25 11.17	19.77 19.67
C_9	80	298-300	Dark green	7.5	36.95 36.66	3.07 3.23	10.77 10.88	12.31 12.21	12.23 12.35
C_{10}	40	245-246	Dark blue	6.6	37.51 37.12	3.12 3.10	10.94 10.65	12.50 12.59	10.90 10.88

Spectrum (IR):

An IR spectrum is used to diagnose organic compounds and complexes. All vehicles were identified using a KBr disk.

Spectrum (IR) for hydrazine acids:

The IR spectrum of the acid hydrzidates showed a shock absorption of the carbonyl (amide) group at the low frequency at (1680 - 1600) cm -1 for the carbon uptake value for the esters prepared at high frequency (1750 - 1680) cm -1, Another for the formation of acid hydrazidate is the appearance of a strong absorption pack at the site (3600 - 3000) cm -1 for extending the (-NH) group that interferes with phenolic (-OH).

Spectrum (IR) for oxadiazol derivatives:

The disappearance of the carbonylamide uptake package at 1680 - 1600 cm -1 and the emergence of a new uptake at 1620 - 1600 cm -1 for the group (C = N) and the emergence of an absorption package at (1190 - 1170) cm -1 with Absorption at (1390 - 1350) cm -1 for the symmetric and asymmetric S (C = S) of the group as well as the emergence of an absorption package at (790 - 740) cm -1 for the extension of the CS group and the asymmetric (1070 - 1031) cm -1 and (1300 - 1200) cm - 1 respectively and these couplings are clear evidence of the formation of oxydiazol ring.

Spectrum (IR) for complex (C10 – C1):

The IR spectrum of the complexes showed the same absorption packs in the lycandates (oxadiaziazole derivatives) or the approach to the oxydiazole derivatives, which is as follows: A C-C uptake of the gas ring in the region (1600-1400 cm -1) (C = N) in the non- homogeneous five-ring area in the region (1630-1600) cm -1, the aromatic extension band (CN) of the five-ring ring due to the presence of the rzonzone process in the region

(1500- 1400) cm -1, (OH) in the region (1400-1300) cm -1, COC Asymmetric Absorption Package for Area (1300-1200) cm -1 and corresponding J region (1100-1000) cm -1 in the ring five asymmetrical, absorption stretchable bending package (N-H) Altotomreh with the group (C

= N) for the five-ring asymmetrical in the region .

(1300-1200) cm -1, and a weak tachromatic tectonic bundle (C = S) with C-SH in the area (1200-1100) cm -1 in addition to the absorption packages of the gasoline ring protons for Ortho sites in the region (770-700) cm -1 and the location Bara in the region (900-800) cm - 1 and sites dead in the region (900-800) cm 1 - All these specialties are present in the spectra of the complex as we observed in the derivatives of Oxydiazol.

The complex spectra showed new packages in the region (230-220) cm -1, dating to the metal and nitrogen nitrate, as well as a package in the region (400-350) cm -1 for the metal bed and sulfur atom in the five-ring and the other wide package in the region (3500) -3100) cm-1 for the OH in the water molecule involved in the complex structure and the reason for the beam width due to an interstitial hydrogenase between the water molecules. These new beams give us an indication of the complexities, metal bonds, sulfur atoms and nitrogen.

^{1}H – NMR:

Spectrum (H1-NMR) is used to diagnose organic compounds containing hydrogen atoms.

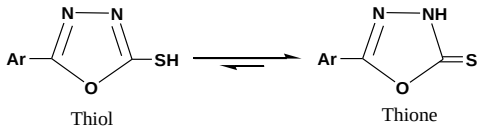
^{1}H – NMR for hydrazine acids:

The nucleotide spectrum (H1-NMR) for acid hydrazidate showed a weak and wide unilateral bundle at the region (2-2.5) ppm for protonin (-NH2), and bronton amyide (CO- NH-NH) 7.5-8 (ppm) This displacement is the result of the effect of the induction of the electrons of the neighboring group (-CO-) of the group (-NH-).

Proteins of the benzene ring for the phenol-derived derivatives of the phenol were found at Ortho site as in the compound (9) multi-pack in the region (6.8-7.9) ppm, whereas the proton at site (3) only gave a multiple pack at (6.81-6.94) While the proton in situ (4) also gave a multi-pack at the region (7.31-7.36) ppm while the proton gave the site (6) a package at the site (7.8) ppm. the benzene ring protons in the compound (11), the phenol- compensated derivative of the phenol at the site Barra, gave two types of couplings due to the similarity of the protons close to the group (-OH) and the second close to the carbonyl group of amides. The beams in the region were between 6.9-7.7 ppm . The protons at the site (6) gave packets at the site (7.69-7.79) ppm while the protons at the site (3) gave packets at (6.78-6.83) ppm and for the proton the phenol group gave a weak(3,5 -6) part million.

^{1}H -NMR for oxadiazol derivative:

The NMR spectrum of the oxydiazole derivatives showed a wide monocrystalline package in the region (2.5-3.5) ppm of the N-H and S-H groups, showing a balance between the titomer forms of thiol and thion when the proton moves as shown below:



Thiol Thione

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