The Eurasia Proceedings of Science, Technology,
Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2019

Volume 6, Pages 151-161

ICRES 2019: International Conference on Research in Education and Science

Synthesis, Investigation and X-Ray Powder Diffraction (XRD) of Some Oxadiazole Complexes

Amerah Jihad AHMED SHAHEEN
University of Mosul

Abstract: New ligand; 2-(1,1-dimethoxymethane) thio-5- (phenyl)-1,3,4-oxadiazole (L)(3) and its complexes with Mn(II), Fe(III), Co(II), Ni(II), Cd(II) and Zr(IV), have been synthesized and characterized on the basis of physicochemical investigations including infrared and electronic spectroscopy,magnetic moment measurements, molar conductance and elemental analysis. The structures of the compounds and some complexes have been investigated by X-ray powder diffraction and H1NMR spectra. was recorded on broker shield 300 MHz using deutrated DMSO-d6 as a solvent. for ligand. They have been found to have the formula; [M L (H2O)3Cl]Cln; M= Mn(II) n = 1, (4) and M= Fe(III), n = 2 (5), [ML (H2O) Cl)] Cl , M=. Co(II) (7), Cd(II) (8)and [NiLCl2](6) ,[ZrOL(H2O) Cl2]Cl2 (9), in molar ratio 1:1. Infrared spectral data suggest that the ligand L: behaves as a, bidentate ligand with N,S, donor sequence towards the metal ions. According to the above physicochemical data, octahedral and tetrahedral geometries were assigned for the complexes.

Keywords: Complexes, Oxadiazole, X-ray studies

Introduction

Oxadiazole ring is an electron deficient, resulting in poor hole transport but good transport properties. Although some types of metal complexes have found to be good emitters and/or excellent electron transport with excellent thermal properties (1). 1, 3,4- oxadiazoles and their derivatives are known to exhibit diversepharmacological activities such as antimicrobial, antihistaminic, anticancerous, anti- inflammatory, antihypertensive and anticonvulsant activities (2 ,3). 1,3,4 -Oxadiazole derivatives are also among the most widely employed electron conducting and hole blocking (ECHB) materials in organic light- emitting diodes (LEDs).(4).The. application of 1,3,4 -Oxadiazole derivatives lie in the field of liquid crystals.(5) .The heterocyclic thiones represent an important type of compound in the field of coordination chemistry because of their potential multifunctional donor sites, viz either exocyclic sulfur or endocyclicnitrogen (6). For this reason our aim was synthesize various 1,3,4 -oxadiazole derivatives to make notable contributions to this class of hetrocyclic compounds. There are a few reports on the metal complexes of -5-phenyl-1,3,4-oxadiazole. 2-subsituted thione.

Experimental

Chemicals

All chemicals and metal salts were commercially available pure samples and used throughout this investigation available from Merch, B.D.H Aldrich or Fluka.

Analytical and Physical Measurements

Melting point and decomposition temperature were determined using SMP30 melting point apparatus.IR spectra measurements were recorded using FTIR-Tensor 27-Burker Co.Germany 2003 as kBr pellets in the range (400-4000 cm-1) . UV- visible spectral measurements were done on Shimaduz UV-Visible

---

- This is an Open Access article distributed under the terms of the Creative Commons Attribution-Noncommercial 4.0 Unported License, permitting all non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

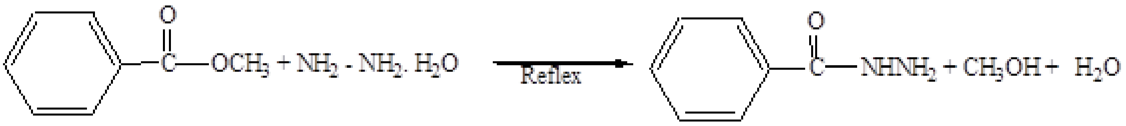
- Selection and peer-review under responsibility of the Organizing Committee of the Conference

© 2019 Published by ISRES Publishing: www.isres.org

1800 Spectrophotomer for 10-3 M complexes in DMF solvent at room temp. using 1cm quarts cell in range (200-1100) nm. The 1 HNMR analysis was recorded on Bruker Ultra Shield 300 MHz using deutrated DMSO–d6 as solvent and Me4 Si as internal reference .Elemental analysis were carried out on a CHN analyzer type 4010 model 2011 Costech Elemental Combustion. Metal contents were estimated spectrophotometrically using atomic absorption Sons AAGB Scientific Equipment. X-ray powder diffraction data were measured for the ligand (3) and complexes (5 & 7) at General Company for Geological Survey and Mining-Bagdad by using Shimadzu X –ray Diffraction 7000 model 2009 and the crystal data for complexes were analyzed by using Match Program Version 1.6 c. Molar conductance of complexes were measured at room temp. for 10-3 M in DMF using (BC 3020 professional Bench top conductivity) . Magnetic susceptibility of the complexes was measured by Bruker –BM6 using Faraday method.

Preparation of benzoylhydrazine (1)(7)

On the first step hydrazine hydrate (5g, 0.1mole) was added drop-wise to methylbenzoate (18.1g, 0.1 mole) in absolute ethanol, after adding, the mixture was refluxed for 2 hours and then cooled and the creamy solid product formed was collected by filtration, washed with cold ethanol and purified by crystallization from ethanol and dried at 50 ^{o}c as in the following equation , yield (96%), m.p 112-113 ^{o}c empirical formula. $C_7H_8N_2O$

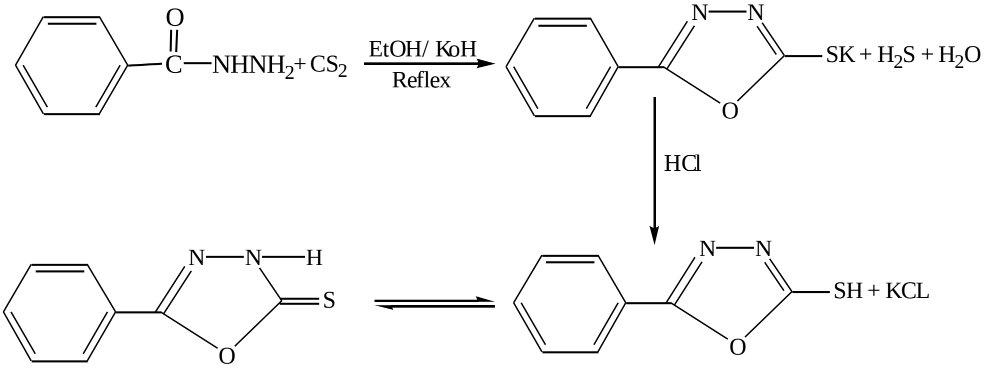


	%C	%H	%N
Calc.	61.69	5.87	20.58
Found	61.63	5.84	20.52

Preparation of 2-thio-5-phenyl -1,3,4-Oxadiazole (2) (8)

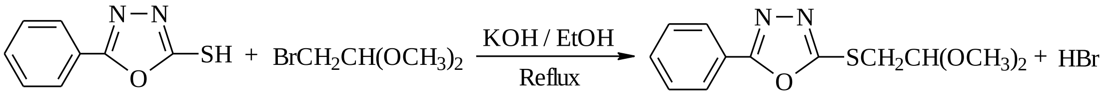
In the second step, this ligand was prepared according to the modified procedure (young wood. The hydrazide of the step 1(benzoyl hydrazine) (13.6 g, 0.1M). was allowed to react with carbon disulphide (120 ml, 0.2 M of CS_2) in the presence of ethanolic potassium hydroxide (5.6 g ,0.1M of KOH in 70 ml absolute ethanol) under reflux conditions until the libration of H_2S gas has been stopped and is detected with lead(II) acetate paper it turns grey in the presence of H_2S gas followed by acidification with dilute HCl to give yellowish precipitate, which was filtered ,wash with cold water , and dried under vacuum as in the following equation. The yield (85%) , mp 219-220 ^{o}c , empirical formula $C_8H_6N_2OS$.The yield product was characterized by CHN analysis, IR and H^1NMR.

	%C	%H	%N
Calc.	53.93	3.37	15.73
Found	53.71	3.28	15.81



Preparation of 2- thio (1,1-dimethoxyethane) -5- phenyl-1,3,4-Oxadiazole (3) (9)

A 2-thio-5-phenyl -1,3,4-Oxadiazole (A1) (17.8. g , 0.1 M) was dissolved in ethanolic potassium hydroxide (5.6g ,0.1M of KOH in 100 ml absolute ethanol) followed by slow addition of drop wise of 2-bromodimethoxyethan (16.9, 0.1M) .The mixture has been refluxed for 3 hours with stirring, followed by cooling, then evaporated to half its volume, cooled, filtered, the yellowish precipitate recrystallized from ethanol,and dried under vacuum as in the following equation. . The yield (62%), mp 195--196 °c , empirical formula $C_{12}H_{14}N_2O_3S$. The yield product was characterized by IR and H^1NMR.



	%C	%H	%N
Calc.	51.96	5.51	11.02
Found	52.03	5.80	10.89

Preparation of the Complexes(4,5,6,7,8&9)(10)

A general procedure has been adopted for the preparation of all complexes in Molar Ratio (1:1) Ligand: Metal. An ethanolic solution of the ligand (compound(3) (2.66 g, 0.01 M in 25 ml ethanol) has been added to a solution of 0.01mol of each of metal salts 0.01M in 20 ml of distilled water or ethanol solution. The mixture has been refluxed for 1/2 - 2 hours with stirring ,followed by cooling and .The product has been filtered off, recrystallized from ethanol then evaporated to half its volume, cooled, filtered, washed and dried examination of elemental analyses data, as given in Table (1&2).

Results and Discussion

The prepared ligand (3) and its complexes are solid and coloured , insoluble in water, but soluble in DMF. Table 1 revealed conductivity data obtained in DMF at 10^{-3} M .The molar conductance values of the complexes (4,7 and 8) are in the range (50-70*) ohm $^{-1}$ cm^2 mol^{-1}* indicating a (1: 1) electrolytic nature of these complexes (11) .While the molar conductance values of the complexes (5and 9) are in the range (130-140) *ohm $^{-1}$ cm^2 mol^{-1}* indicating (1:2) electrolytic nature of these complexes . On other hand, the complex (6) is non- electrolytic indicating neutral complex (12) in nature. The molar conductance values are in a good agreement with given formulations .(Tables 1&2).

Table 1.Analytical data and some physical properties of the complexes

Comp. No.	Metal Salt	Wt(g) Metal Salt	Wt(g) L	Ω Ohm^{-1}.cm^2.mol^{-1}	Complexes	m.p or d ^{0}C	Color
4	$MnCl_2.6H_2O$	1.97	2.66	70	$[MnL(H_2O)_3Cl]Cl$	214	pale brown
5	$FeCl_3$	1.62	2.66	130	$[FeL(H_2O)_3Cl]Cl_2$	140	deep brown
6	$NiCl_2.6H_2O$	2.34	2.66	10	$[NiLCl_2]$	152	olive
7	$CoCl_2.6H_2O$	1.8	2.66	50	$[CoL(H_2O)Cl]Cl$	193	blue
8	$CdCl_2$	1.83	2.66	70	$[CdL(H_2O)Cl]Cl$	218	pale brown
9	$ZrOCl_2.8H_2O$	3.22	2.66	170	$[ZrOL(H_2O)Cl_2]Cl_2$	130	pale brown

d = decomposition temp.

Table 2.Elemental analysis of the complexes

Comp.No.	Complexes	% Found (Calc.) CHN , M			
		C	H	N	M
4	[MnL $(H_2O)_3Cl]Cl$	(30.41)	(4.60)	(6.45)	(12.67)
		30.50	4.91	6.71	12.40
5	[FeL $(H_2O)_3Cl]Cl_2$	(28.05)	(4.25)	(5.95)	(11.90)
		28.22	4.35	6.00	12.02
6	[NiLCl_2]	(34.46)	(5.22)	(7.31)	(15.32)
		34.77	5.45	7.61)	15.53
7	[CoL (H_2O) Cl]Cl	(32.83)	(5.47)	(3.81)	(14.67)
		33.00	5.77	4.00	14.87
8	[CdL (H_2O)Cl]Cl	(29.01)	(4.83)	(6.15)	(24.61)
		28.89	5.11	6.45	24.88
9	[ZrOL (H_2O) $Cl_2]Cl_2$	(26.13)	(2.57)	(5.54)	(18.01)
		26.00	2.84	(5.83	17.87

Infrared Spectra

The ligand (3) is basically composed of different groups of potent ability to coordinate with the metal ions. The coordination sites of the ligand involved in the bonding with metal ions had been determined by careful comparison of the infrared spectra of those compounds with that of the parent ligand. The IR spectra of the compound (2) shows two bands a weak band at 2350cm^{-1} which is characteristic of stretching vibration of the SH group and sharp band at 3434 cm^{-1} due to NH stretching vibration (13).These bands which subsequently disappeared in the ligand L(compound 3,Fig.1) ,indicating a vibration substitution occurred on position 2, and a new absorption bands appeared at1397cm^{-1}, which has been attributed to bending frequency of -SCH_2 group. This is further supported by the appearance of the band at 652 cm^{-1}due to stretching vibration of C-S bond and, these bands shifted to a higher value in complexes as shown in Fig. 2 for (complex 4), ν- C-S (672)& ν- S-CH2 (1415) cm-1 (14). In the spectra of the ligand (3) showed, a weak band at 2925 cm–1due to sym.& asym. C-H aliphatic of CH2 group, and his band remains unaltered on complexation(15) .The IR spectra of the ligand (3) show a strong band in the region 1608cm-1, which is characteristic of the azomethane(16)indicating coordination of the L through azomethine nitrogen atoms to the metal center (through the nitrogen atom, of the oxadiazole ring) and, this band shifts to a higher value on coordination as shown in (complex 4) □-C=N at (1618) cm-1 .This view is further supportednby the appearance of a band corresponding to the metal–nitrogen(17) stretching vibration at 460–501 cm–1 in the complexes and for complex 4 has been appeared at 501 cm-1 .The spectra also shows a broad band at 946cm-1 due to N-N group shifts towards higher frequency (1000-1020) cm-1 on complexation (18). ,while in complex(4) this band appears at 1014 cm-1 .In the ligand 3 spectra shows a characteristic band at 1077cm-1 which was assigned to the stretching was assigned to C-O-C group of the oxadiazole ring and this band remains unaltered on complexation indicating the absence of coordination through oxygen of this group (19) .The aqua complexes contain weak to medium a broad band at (3099-3477) cm-1 due to stretching vibration OH of water (20) and a sharp shoulder at (1117) cm-1 may be assigned to bending vibration of water molecules are coordinated, confirmed by occurrence of additional strong and sharp band at 672-720 cm-1 due to OH rocking vibrations. Also is further supported by the appearance of a band corresponding to the metal– oxygen stretching vibration at 546–571 cm-1and for complex 4 3477, ν-OH2766, ν-OH2 571, ν-M-OH2.(21). In addition the coordination of chloride could not be inferred from infrared spectra of the complexes because the band occurred beyond the range of our infrared spectrophotometer, whereas for Cl- ionic has been checked by $AgNO_3$..All chloride complexes show a band at 584- 618 cm-1 and for complex (4) 584, cm-1 has been attributed to ionic chloride (15) as shown in (Table 3).

Table 3. Selected I.R. bands of the ligand and its complexes (cm^{-1})

Comp. No.	**ν-C=N**	**ν- N-N**	**ν- C-S**	**ν-M-N**	**ν- M-S**	**Others**
3	*1608*	*946*	*652*	-	-	*1397, ν- S-CH_2* *1024,ν- C-O-C* *2925,ν- C-H*
4	1618	1014	672	501	410	584, ν- IonicCl- 3477, ν- OH_2 766, ν- OH_2 571, ν- M-OH_2
5	1600	1018	676	460	423	600, ν- ionic C^{l-} 3420, ν- OH_2 752, ν- OH_2 546 ν- M-OH_2
6	1580	1019	665	462	415	-
7	1585	1020	666	476	410	618 ν- Ionic Cl^- 3300 ν- OH_2 680 ν- OH_2 558 ν- M-OH_2
8	1592	1000	675	470	416	612ν- Ionic Cl^- 3475 ν- OH_2 690 ν- OH_2 556 ν- M-OH_2
9	1575	1007	675	480	420	600 ν- Ionic Cl^- 3472ν- OH_2 741ν- OH_2 552 ν- M-OH_2

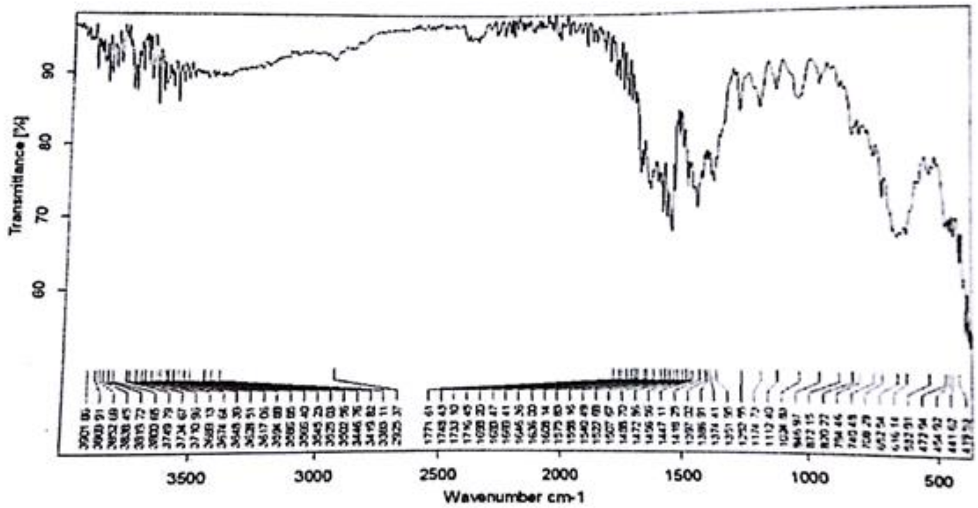


Figure 1. Ft-IR spctrum of the ligand (3)

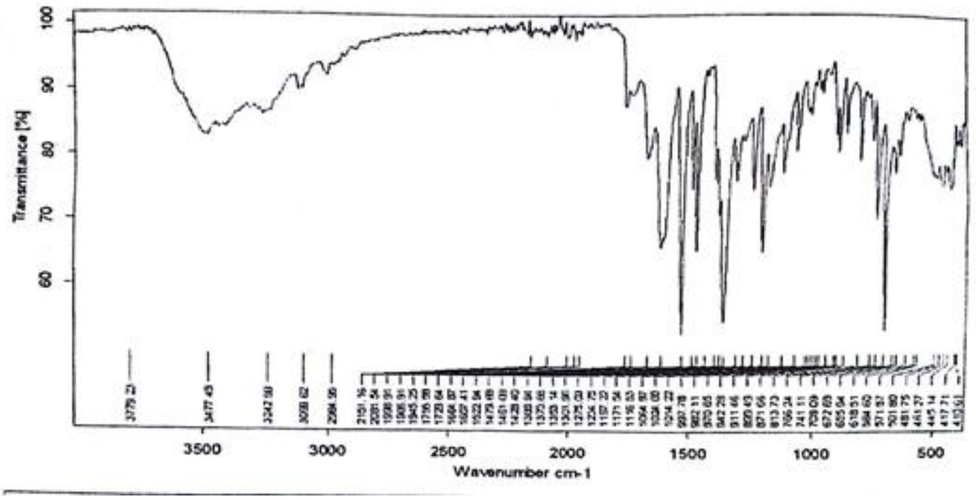


Figure 2. Ft-IR spctrum of the complex 4

Electronic Spectra and Magnetic Moment

The electronic spectra of the ligand (3) and its complexes) in DMF solution have been recorded giving ultraviolet spectra, d-d spectra and charge transfer spectra (Table 4). The ultraviolet spectra for ligand exhibited two bands in the UV intervals at 31847 cm^{-1} and 377351 cm^{-1}, assigned to $n \rightarrow \pi^*$ (C=S), and $\pi \rightarrow \pi^*$ (C=N) transitions, respectively. All these transitions were also found in the spectra of the complexes but they were shifted to lower frequencies confirming the coordination of the ligand to the metal ions(23). . The electronic spectrum of Fe(III) complex (5) exhibited , only sextet term $^6A_{1g}$ of the d^5 configuration octahedral geometry and does not split by the ligand field. Consequently, all the excited states have different spin multiplicity from the ground term and transition to them is forbidden ,and many weak bands were observed at 9794,10928,13333 $cm^{-1}$and assigned as due to transition from $^6A_{1g}$ to $^4T_{2g}$ (G) and $^4T_{2g}$ (G), and are characteristic of an octahedral geometry as shown in (Fig. 4). In addition, high intensity band was observed in the ultraviolet region of the spectra at 27472cm^{-1} and assigned to C-T transition. Complex 5 shows magnetic moments at room temperature calculated from the corrected magnetic susceptibilities is 5.38 B.M revealing the presence of five unpaired electrons are presen in the complex molecule and indicating high spin octahedral complex(24). The experimental electronic spectrum of Co(II) complex (7)showed less intense band in the region 14833 cm^{-1} which correspond to the $^4A_2$ (F) $\rightarrow$ $^4T_1$ (P) ν_3 transition. This band is specific to the Co(II) ion in tetrahedral stereochemistry, a geometry also confirmed by the magnetic moment of the complex which is 4.61 B.M .and the blue colour. The other two bands due to $^4A_2$ (F) $\rightarrow$ $^4T_2$ (F) ν_1 and $^4A_2$ (F) $\rightarrow$ $^4T_1$ (F) ν_2 transitions (24) were absence because they fall below the limits of our instrument. While in the spectrum of the Ni(II) complex(6) exhibits electronic spectrum . One strong band at 13881, 14566 and 37593cm^{-1}, respectively and assigned to transitions $^3A_{1g}$ (F) $\rightarrow$$^3T_2$ g(F) ν_1 , $^3A_{1g}$ (F) $\rightarrow$ $^3T_{1g}$ (F) ν_2 and A_{1g} (F) $\rightarrow$ $^3T_1$g (p) ν_3 and this tetrahedral geometry is also supported by the value of the magnetic moment of 3.85 B.M(25). The electronic spectrum of manganese (II) complex 4 exhibited bands at 14880 cm^{-1} and 32362 cm^{-1} since the d-d transitions as shown in Fig.3 , doubly forbidden from the term $^6A_{1g}$ towards , the quartet terms $^4T_1$g (G) , $^4T_2$g (G) , $^4A_{1g}$, ^{4}Eg, exhibit a very small intensity and are concealed by the intraligand transitions. The absorption band at 32362 cm^{-1} can be attributed to the charge transfer of ligand to metal. The complex 4 shows magnetic moment 5.09 B.M basing on this discussion a high spin octahedral arrangement may be proposed around Mn(II) ion in complex(4) (26). The two transitions of the ligand are found also in the spectra of the Cd(II) and Zr(IV) complexes but they shifted towards a lower frequencies, confirming the coordination of the ligand to these ions. They were found to be diamagnetic as expected for d^{10}system,(24) Table 4.

Table 4. Electronic spectra data of the complexes

Comp.No.	Electronic transitions of d-d cm^{-1}			
	υ_1	υ_2	υ_3	CT
4	14880	-	-	32362
5	9794	10928	13333	27472
6	13881	14566	37593	37812
7	-	-	14833	35668
8	-	-	-	31446,37250,41615
9	-	-	-	31440,37133,41611

CT = charge transfer band

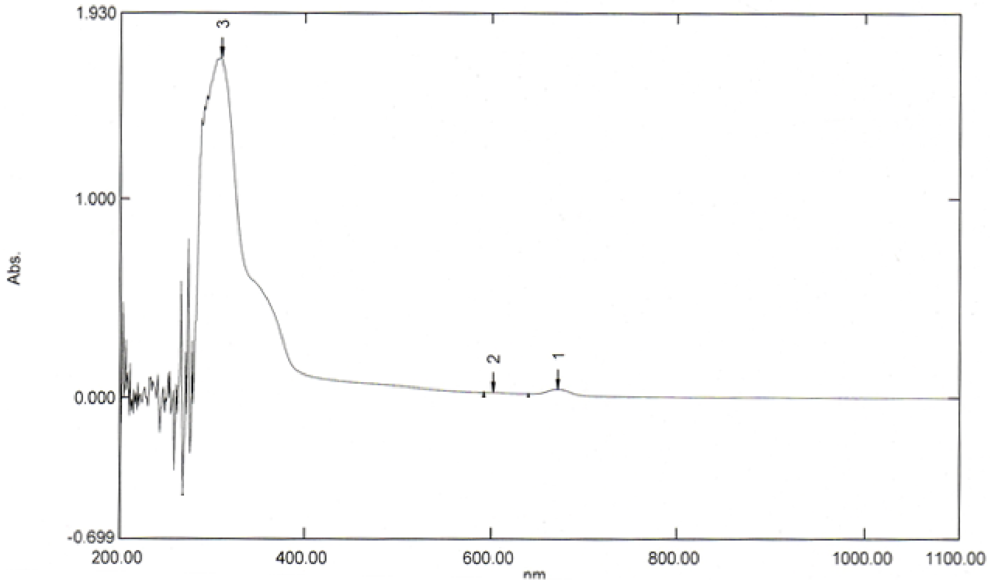


Figure 3. UV-Visible spectrum of the complex 4

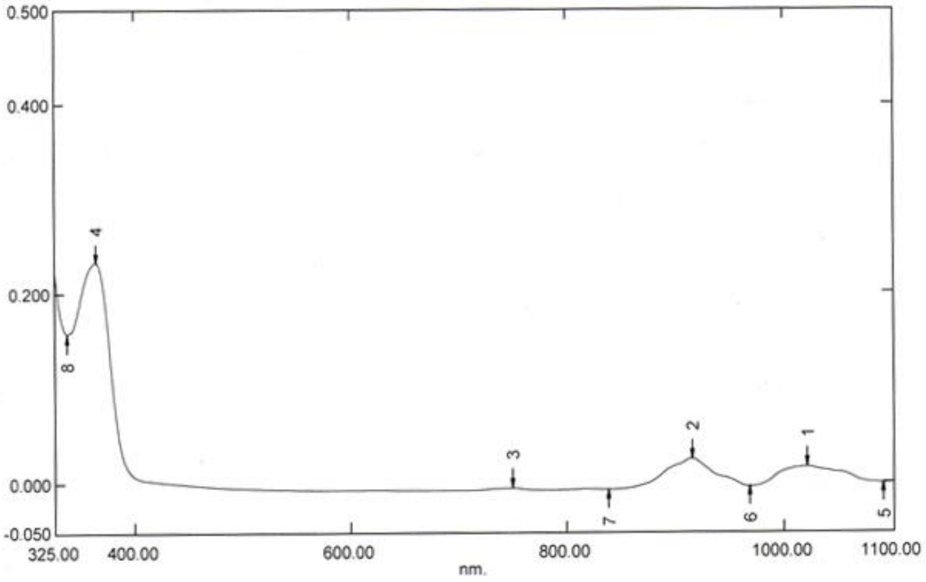


Figure 4. UV-Visible spectrum of the complex 5

^{1} H NMR Spectra

Compound 2 exhibits a sharp singlet at 10.7 ppm due to SH group and this peak is expected for thiol- 1,3,4-oxadiazole signal, While compound 3 exhibits a singlet signal at δ = 4.52 ppm due to CH_2-S group protons attached to S atom and oxadiazole ring ,and this is an electron withdrawing group. On the other hand, aromatic protons were observed in region 7.15–7.46 ppm , the signal at ($\delta = 3.24$ ppm) is assigned to O- CH_3 group and absorption signal at (δ=4.47 ppm) due to CH group (22) .

X-ray Powder Diffraction Analysis

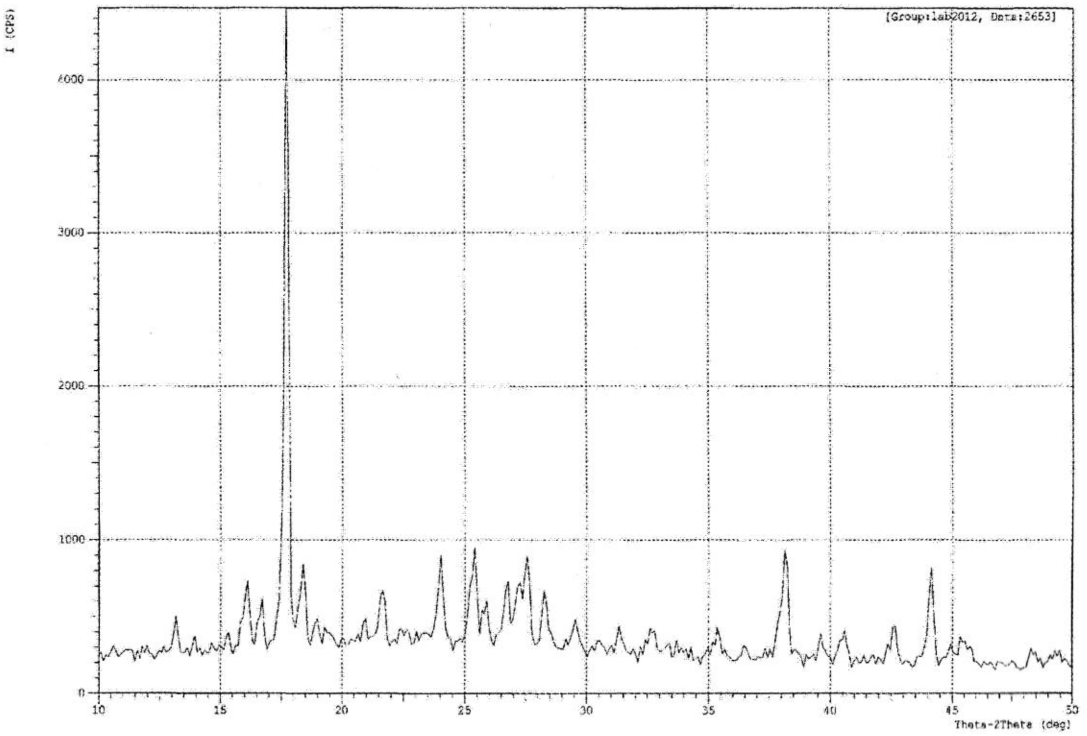
X -ray powder diffraction analysis of the parent metal salts from which they were prepared metal complexes was carried out to determine the type of crystal system, lattice parameters, and the cell volume .The XRD patterns of complex4 indicate a crystalline nature for metal complexes. Indexing of the diffraction patterns was performed using high Score Plus Software (Match program)(27). For Fe(III) and Co(II) complexes, for example ,their Miller indices (hkl) along with observed and calculated 2Ø angle , d values, and relative intensities , From the indexed data the unit cell parameters were also calculated and the powder XRD patterns of the compounds are completely different from those of the starting materials (28), demonstrating the formation of coordination compounds. It is found that Fe(III) and Co(II) complexes have orthorhombic and tetragonal structure. Moreover, using diffraction data, the mean crystallite sizes of the complexes, D ,were determined according Scherrer equation ($D = 0.9\, \lambda / (\beta \cos \theta)$), where λ X-ray wavelength (1.5406 $\text{A}^{-\circ}$), θ is Bragg diffraction angle, and β is the full width at half maximum of the diffraction peak (29,30) as shown in Table 5.

Table 5 .Crystal data and structure refinement for complexes

No	**Complex**	**5**
1	Molecular Formula	$FeC_{12}H_{20}N_2O_6SCl_3$
2	Molecular weight	482
3	Crystal system	Orthorhombic
4	Space group	Pca (61)
5	Unit cell parameters (A^{o})	a= 11.101 , b= 12.229 , c= 9.079
6	Cell Volume (A^{o3})	1271.93
7	Z	8
8	θ range , deg	2.08 – 30.52
9	Index ranges	$0 \le h \le 2$, $0 \le k \le 3$, $0 \le L \le 4$

No	**Complex**	7
1	Molecular Formula	$CoC_{12}H_{16}N_2O_4SCl_2$
2	Molecular weight	414
3	Crystal system	Tetragonal
4	Space group	P-4(81)
5	Unit cell parameters (A^{o})	a= 7.22 A^{o} , c= 14.33 A^{o}
6	Cell Volume (A^{o3})	1253.37 A^{o}
7	Z	4
8	θ range , deg	10.55 – 50.06
9	Index ranges	$0 \le h \le 6$ $0 \le k \le 4$ $0 \le L \le 2$

No.	Metal Salt	Cobalt chloride hexa hydrate
1	Molecular Formula	$CoCl_2.6H_2O$
2	Molecular weight	237.93
3	Crystal system	monoclinic
4	Space group	P21/c(14)
5	Unit cell parameters (Ao)	a=8.86,b=7.07, c=13.12, β=97.28
6	Cell Volume (Ao3)	815.21
7	Z	4
8	θ range , deg	14.26-69.36
9	Index ranges	$-5 \le h \le 6$



Scheme 1. Powder x-ray diffraction (PXRD) of complex 4

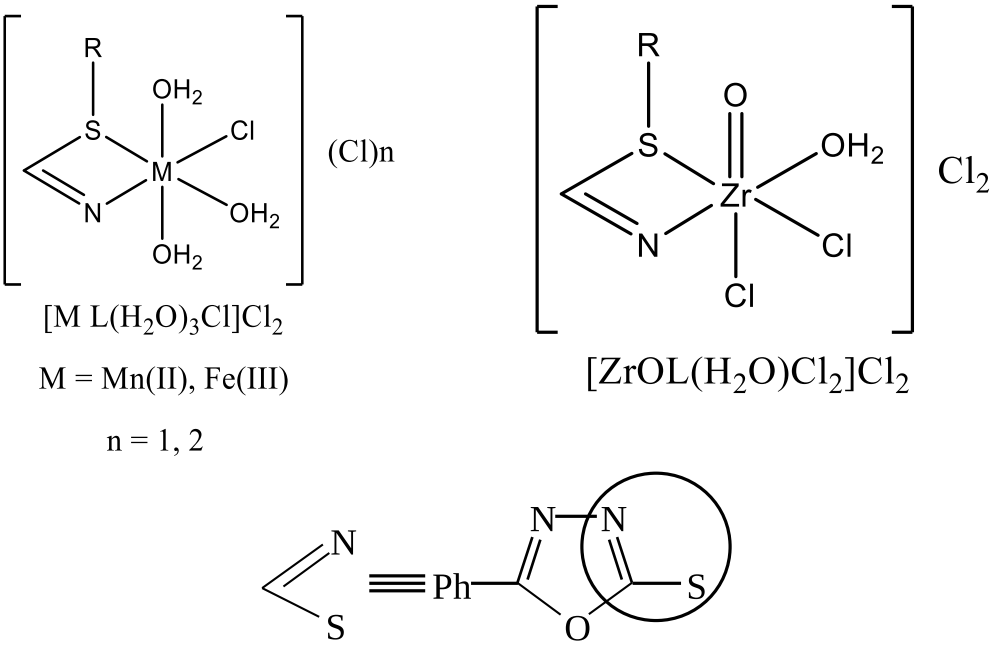


Figure 5. Proposed Structure of some complexes

Conclusion

Simple efficient methods have been followed to synthesize new ligand of oxadiazole derivative and its complexes and they were characterized by physicochemical measurements and spectral studies. Moreover, the newly synthesized ligand(3)and complexes (5&7) were evaluated for XRD analysis to estimate the crystal geometries and we concluded that an octahedral structure has been suggested for manganese, iron and zirconium complexes as shown in (Fig. 5).Whereas cadmium, nickel and cobalt complexes have tetracoordinate geometries. The IR spectroscopic and analytical data showed that ligand (L) (3) acts as bidentate coordinated to the metal ions through sulfur thioether and azomethine nitrogen atoms.

Acknowledgement

The author would like to thank Head of Chemistry Department in Education college for Pure Science of Mosul University for providing laboratory facilities, the author also is thankful to Central Geological Survey and Mining-Baghdad for the use of Shimadzu X -ray Diffraction 7000 model 2009.

References

Kalagudasi G, Manjula P, Ramesh, V., Rashmisheoy and Sidappa P."(2007), Transition metal complexes with a new tridentate ligand, 5-_6-(5- mercapto-1,3,4 -oxadiazol -2-yl) pyridin -2-yl-1,3,4-oxadiazole-2-thiol", *J. Serb. Chem. Soc. 72,* (4) 357–366.

Almasirad, A; Tabatabai, S.A and Faizi, M; (2004) "Synthesis and biological activity of 1,3,4- oxadiazole derivatives"; Biorg. Med. Chem. Lett; 14, pp. 6057,.

Abdel Hamid,M; , (2008)" Synthesis and cyclization reactions with oxadiazole dervatives and their enzymaic activity; Acta. Chim. Slov; 55,. 492-501

Bhat, M. A; Siddiqui, N and Khan, S.A; (2008)." Synthesis of novel 3-(4-Acetyl-5H-methyl-5-substituted phenyl-4,5-dihydro-1,3,4-oxadiazol-2-yl)-2H-chromen -2-ones as potential anticonvulsant agent, Polish. Pharm. Soc; 65 , pp.235-239.

Shabbir Akazi (2011)", Synthesis and characterization of a new bidentate ligand 5 -substituted-(2- methyl-5 -nitro-1- imidazomethyl) -1,3,4oxadiazole 2-thione and its metal complexes of Ag(I), Cu(II) and Zn(II)", E -J. of Chemistry 8(S1), S127-S136.

Najat A.obaidy,K.K.Abid, Yusra, (2006)." Transition metal complexes of 1,3,4-oxadiazole ligands",National J .of Chem.,24,629-641

H.L. Yale, K. Losee, J. Martins, M. Holsing, F.M. Pery and J. Bernstein, (1953), "Chemotherapy of experimental tuberculosis. VIII. The synthesis of acid hydrazides, Their derivatives and related compounds", J. Am. Chem. Soc., 75, 1933

Yong Wood(1955) ,"The cyclization of 3- acyl dithiocarbazates ester", J.Am.Chem.Soc.,77.

Amira Jihad Al- Shaheen, Tamem A . Al- Muhger, (2016) " Synthesis, Spectroscopic and X- ray Powder Diffraction(XRD) of Some Oxadiazole Complexes. " International Journal of Enhanced Research in Science ,Technology & Engineering , ISSN :2319-7463 , Vol.5 issue 6 , June-, p.1339.

Amira Jihad Al- Shaheen, Mustafa R.A.Al-Bayati and Amal M.A.Abid Allah.,(2018) "Synthesis and Characterization of some Metals(II) complexes with 2-(6- methoxy naphthalene-2-yl)Propionic acid(2- hydroxyl benzylidene) hydrazine ligand" . International Journal of

Enhanced Research in Science ,Technology & Engineering , ISSN :2319-7463 , ISSN: 2319-7463, Vol 7 Issue 4 april-, Impact Factor: 4.059.

Geary W.J(1971).,"The use of conductivity measurement in organic solvent for Characterization of coordination compounds" , Coord. Chem. Rev., 7, 8

Al- Sabaawi S.A. A .J. Al- Shaheen, (2019)., "Preparation and Physicochemica l Investigation of Manganese (II) complexes with Hydrazone Ligands.,Raf .J .Sc.,Vol .28 ,No.2, Specia l Issue for third Scientific Conference of Chemistry pp.204-210.

Mamata Singh R .J. Butcher, N.K. Singh., (2008).," Syntheses and X- Structural Studies of the novel complexes", [Ni(en)2(3-pyt)2] and [Cu(en)2](3- pyt)2 based on 5-(3-pyridyl)-1,3,4-oxadiazole-2-thione, Polyhedron 27, 3151–3159.

Hoggarth, (1949), "Compounds Related to Thiosemicarbazi-3-Phenyl-1,2,4- tri azole derivatives" ,J Chem . Soc 2, 113

Cotton F .A. and G. Wilkinson, (1980) ," Advance Inorganic Chemistry", 4th Ed. , John Wiley and Sons,Interscienic,NewYork,P.783

Bellamy L.J. (1966)., "The infrared Spectra of complex molecules 3rd ed. Methuen,London.

Dogan ,Alev Sakyan Iffet KA+A+C ,Esma.,(2014)" Synthesis and characterization of amino acid Schiff base complexes", J Solution Chemistry, 33,1539-1547,

Satapathy S.and Sahoo B,(1970).,"Salicyl-alazine metal chelates and their IR spectra".,J.Inorg. Nucl . Chem., 12, 2223-2227

William K., (1991) "Organic spectroscopy, 3rd ed.,Macmillan Education Ltd,London,49 54,60- 75

Gamo (1960)., "Infrared spectra of water of crystallization in some inorganic chlorides and Sulfate","Bull .Chem.Soc., 34,760,1430

Chira Bhattacharjee, Pankak Gosurimi, Sankar Neogi and Saptarshi Dhibar . (2010),, "Transition metal complexes of furfuraldohyde and hydrazine hydrate", J.of Sci & Technol., 5(2), 81,87.

Tok T. T., O. Ozasik, D. Sarlgol, and A. U. Baran (2015). , "Synthesis and Molecular Modeling Studies of Naproxen-Based Acyl Hydrazone Derivatives.", *Turk. J.Chem.* 39(1), 54-83

D. Suttan(1998)., " Electronic Spectra ofTransition Metal Complexes", Mc. Grow- Hill Publishing London, pp. 11-21.

D. Nicholls(1975) , " The Chemistry of iron, cobalt and nickel", Pergamon Press Oxford, pp.1087.

Spina C., M. Pleniceau and C. Tigae(2008) , "Biologically Active New Fe(II), Co(II), Ni(II)," Chem. Soc.,73 (4), 415-421.

Lever ,A.B.P (1968), "Inorganic Electronic Spectroscopy," Amsterdam- London, New -Y ork Soc.

Match!(2003-2007)"Crystal impact,phase Identification from Powder Diffraction",version1.6c Licensed to Ankara Uni.Dep.of Geological Engineering site license(c),(http://www. Crystali mpact. com.

Stout, C. H L.H. Jensen (1968), "X-ray Structure Determination", Macmillan, New.York.

Amira Jihad Al- Shaheen,(2017)., "Physicochemical Studies and X-ray Powder Diffraction of New Homodinuclear Schiff Base Ccomplexes", International Journal of Enhanced Research in Science ,Technology & Engineering , ISSN :2319-7463 , ISSN: 2319-7463, Vol. 6 Issue 10, October-2017, Impact Factor: 4.059.

Aroyo, Mois I. Ulrich Müler and Hans Wondratschek (2007), "Hisiorical Introduction:, International Tables for Crystallography", (Springer) A1 (1:1): 2-5. Doi: 1107/97809.

Author Information

Amerah Jihad Ahmed Shaheen
University of Mosul
Mosul/Iraq
Contact E-mail: *a.inorganic@yahoo.com*