

## Preparation and Characterization of some Transition Metal Complexes with OleylXanthate and 1,10- Phenanthroline

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(Received 2/10/2018 ; Accepted 25/10/2018)

### ABSTRACT

Xanthate complexes of the general formula  $[M\{Ag(oleylXant)_2\}_2]$  (A) and  $[MM'(oleylXant)_4]$  (B) were  $M =$  Copper (II), Nickel (II), Cobalt (II)  $M' =$  Cadmium(II), Zinc(II). These complexes were reacted with bidentate Lewis base (1,10-phenanthroline). The result of this reaction gave the complexes  $[M(phen)][\{Ag(oleylxant)_2\}_2]$  (I.A) and  $[M(phen)][M'(oleyl-xant)_4]$  (I.B), which have been prepared and measured by an infrared, magnetic susceptibility measurements, conductance measurements, electronic spectra and elemental analysis electronic absorption spectra. These data indicate gave the result that the complexes of the type (A & B) are a square planer geometry while the complexes of the type (I.A), (I.B) have an octahedral geometry.

**Keywords:** Xanthate, Cobalt(II), Nickel(II), Copper(II), 1,10-phenanthroline.

### تحضير وتشخيص بعض من معقدات العناصر الانتقالية مع اولاييل زانثيت و 1,10-فينانثرولين

#### الملخص

حضرت معقدات الزانثيت ذات الصيغة العامة  $[M\{Ag(oleylXant)_2\}_2]$  (A) و  $[MM'(oleylXant)_4]$  (B) عندما  $M =$  Cu(II), Ni(II), Co(II), Zn(II)  $M' =$  Zn(II), Cd(II) ثم مفاعلة هذه المعقدات مع قاعدة لويس ثنائية السن (1,10-فينانثرولين). الناتج من هذا التفاعل اعطى معقدات  $[M(phen)][\{Ag(oleylxant)_2\}_2]$  (I.A) و  $[M(phen)][M'(oleyl-xant)_4]$  (I.B).

شخصت هذه المعقدات بواسطة تحليل العناصر وطيف الاشعة تحت الحمراء والقياسات التوصيلية والاطياف الالكترونية وقياسات الحساسية المغناطيسية وقد دلت هذه القياسات على اتخاذ المعقدات من النوع (B,A) بنية المربع المستوي بينما اتخذت من النوع (I.A) و (I.B) بنية ثماني السطوح.

**الكلمات الدالة:** زانثيت، الكوبلت الثنائي، النيكل الثنائي، النحاس الثنائي، 1,10-فينانثرولين.

### INTRODUCTION

Xanthate complexes and their reaction products with variety Lewis bases have been extensively studied (Xiong *et al.*, 1997; Gable *et al.*, 1985; Travnick *et al.*, 1999). Xanthate,  $S_2COR$ , Comprise an important class of 1,1-dithiolate ligands with many applications ranging from flotation agents to radical polymerization (Coucovanis, 1970; Coucovanis, 1986; Coote and Radom, 2004; Haiduc, 2007; Tiekink and Haiduc, 2005).

Xanthate mostly refers to a salt with the formula  $ROCS_2 \cdot M^+$  ( $R =$ alkyl;  $M^+ = Na^+, K^+$ ) (IUPAC, 1997). Zeise is the one who discovered the xanthates for the first time in 1822 (Zeis, 1980), and was used by industry as flotation agents for the thiophilic minerals of the transition metals such as copper, zinc, cobalt, nickel and gold (Han, 1992). They use it as reagents for the separation and

quantitative determination of a large number of cations. Xanthate of a few of the transition metals have been prepared in last years to determine their compound and structure (Exarchos *et al.*, 2001).

### MATERIAL AND METHOD

The purest chemical materials which are supplying from Fluka and BDH. The instrument Brucker Tensor 27 Co. (400-4000  $\text{cm}^{-1}$ ) FTIR spectrophotometer with KBr disc was used for recording infra red spectra data. The measurements of conductivity using  $10^{-3}$  M of the complexes solution in dimethylformamide and conductivity's meter PCM<sub>3</sub> jenway at ambient temperature. The spectrophotometer Shimadzu UV-visible (UV-160) was used for recording electronic spectra data using  $10^{-3}$  M of complexes solution in dimethylformamide at room temperature and cells of 1 cm quartz. The contents of metal were estimated by AA670 atomic absorption spectrophotomet, also the recoding data of melting points were carried out with the apparatus of Electrothermal 9300. The measurement of magnetic was recorded at room temperature on the solids using Brucker BM6 instrument by Faraday's process.

#### preparing of Potasium Oleyl Xanthate

We added oleyl alcohol (63.2  $\text{cm}^3$ , 0.2 mol) to (11.2 g, 0.2 mol) of aqueous KOH solution with continous stirring. An ice bath was used to cool down the mixture. Then, adding with stirring (12.03  $\text{cm}^3$ , 0.20 mol) of  $\text{CS}_2$  drop wise for (60 min.) in salted ice bath. The resulted precipitate which was formed have been extracted by (150 ml) diethyl ether, finally, we must filtrated the resulted yellow product, then washed it using diethyl ether as a washing solution, at the end dried it with a vacuum.

#### Preparation of $[\text{M}\{\text{Ag}(\text{oleylXant})_2\}_2]$ Complex (I.A.)

**M = Co(II), Ni(II), Cu(II)**

Silver nitrate that was dissolved in distilled water has been added (0.169 gm, 0.001 mole) to (0.7642 gm, 0.002 mole) olylxant which was dissolved in a mixture of dimethyl formamide (DMF) and distilled water in ratio of (20:80%) then added to a solution that contains the complex  $\text{K}_2[\text{Ag}(\text{oleylxant})_2]_2$  added to the solution (0.005 mole) from salt metal M [(0.145 gm) hydrated cobalt nitrate salt and (0.145 gm) hydrated nickel nitrate and (0.124 gm) copper sulphate penta hydrate] which was dissolved in distilled water. The precipitate should be filtered then washed with ether and dried under vacuum.

#### Preparation of $[\text{MCd}(\text{oleylXant})_4]$ Complex (I.B.)

**M=Co(II), Ni(II), Cu(II).**

Cadmium nitrate (0.344 gm, 0.001 mole) solution was dissolved in a mixture of distilled water acetone (in 20:80% ratio) was added to ligand solution (0.004 mole, 0.344 gm) dissolved in ethanol (20 ml). Then (0.001 mole) from salt metal M [(0.290 gm) cobal nitrate hexa hydrate, (0.290gm) nickel nitrate hexa hydrate (0.249gm) penta hydrate of copper sulphate] was added to the former mixture with continuous stirring. A precipitated formed washed with ether filtrate precipitate formed and was which was dried under vacuum.

#### Preparation $[\text{MZn}(\text{oleylXant})_4]$ Complex (I.C.)

The zinc chloride has been added (0.001 mole, 0.136 gm) dissolved in a mixture of distilled water and ethanol in a 20:80% ratio of ligand solution (0.004 mole, 1.5289 gm) that dissolved in ethanol (20 ml) that makes the complex  $[\text{K}_2\text{Zn}(\text{oleylxant})_4]$  then metal salts (0.001 mole) of added (0.001 mole) from salt metal M [(0.290 gm) hydrated cobalt nitrate and (0.290 gm) hydrated nickel nitrate and (0.249 gm) copper sulphate] was added to the former mixture filtrate precipitate formal, washed with ether and dry it using vacuum.

## EXPERIMENTAL RESULTS AND DISCUSSION

The preparation of xanthate ligand was carried out with the reaction of oleyl alcohol and KOH and CS<sub>2</sub>; preparation of the complexes was done by the reaction of the AgNO<sub>3</sub> or Cd(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O or ZnCl<sub>2</sub>, CoCl<sub>2</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O and CuCl<sub>2</sub>.6H<sub>2</sub>O directly with xanthate ligand. Conductivity's values in DMF solvent for the complexes (A) and (B) have been ranged (13.8 - 24.6 Ω<sup>-1</sup> cm<sup>2</sup>mol<sup>-1</sup>), (Table 1) which considered to be standard values for the type of non electrolyte in the solvent used (Varma and Prabhakaran, 1989), while the complexes (I.A), (I.B), range from (51.0-67.5) which were found to be 1:1 electrolyte in dimethyl formamide (Aggarwal, 1985).

### Infra Red Studies

The band of infra red's spectra at (1196 cm<sup>-1</sup>) in the ligand spectra which was assigned to ν(C-O). in complexes. This band was shifted to a higher frequency and observed in the range (1215-1268 cm<sup>-1</sup>) and ν(C-S) shifted to lower frequency (1018-1052 cm<sup>-1</sup>) (Serrano *et al.*, 2003). Also, new band of infer red spectra recorded (418-468 cm<sup>-1</sup>), this range is the evidence for the metal's coordination to sulphur ν(M-S) (Rathore *et al.*, 2007), (Table 2).

### The Studies of Electronic Spectra

The ligands and their complexes ultra violet-visible spectra in (10<sup>-3</sup>M) in dimethylformamide have been recorded and tabulated as showing in (Table 2) the UV-Vis. The complex spectra of the Co(II) (1, 3, 5) show two absorption bandsat (11407 – 11520 cm<sup>-1</sup>) (16197 -1629 cm<sup>-1</sup>) which were assigned to (A<sub>1g</sub> → B<sub>1g</sub>) and (A<sub>1g</sub> → E<sub>g</sub>) respectively. The transitions was in a square planar geometry (Bailar *et al.*, 1975).

Cobalt complexes (2, 4) show three bands of absorption at the rang (9982-1062 cm<sup>-1</sup>), (16297-16340 cm<sup>-1</sup>) which were assigned to (<sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>1g</sub>(F)), <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>A<sub>2g</sub>(F)) and (<sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>1g</sub>(P)) transitions due to the configuration of octahedral (Martell, 1971).

The Nickel(II) complex (7, 9, 11) shows two bands of absorption (15187-15276cm<sup>-1</sup>) and (22791-23084 cm<sup>-1</sup>) due to (<sup>1</sup>A<sub>1g</sub> → <sup>1</sup>A<sub>2g</sub>) and (<sup>1</sup>A<sub>1g</sub> → <sup>1</sup>B<sub>1g</sub>) transitions respectively in square planar geometry. As well as complexes (8,12) which was showed three bands of absorption at (11962-12483 cm<sup>-1</sup>), (17961-18536 cm<sup>-1</sup>) and (23582-24196 cm<sup>-1</sup>), due to (<sup>3</sup>A<sub>2g</sub>(F) → <sup>3</sup>T<sub>2g</sub>(F)), (<sup>3</sup>A<sub>2g</sub>(F) → <sup>3</sup>T<sub>1g</sub>(F), (<sup>3</sup>A<sub>2g</sub>(F) → <sup>3</sup>T<sub>1g</sub>(P)).

The Cu(II) complexes (13, 15, 17) give abroad absorption band at (14625-1480 cm<sup>-1</sup>), which was assigned to the combination of (<sup>2</sup>B<sub>1g</sub> → <sup>2</sup>A<sub>1g</sub>) and (<sup>2</sup>B<sub>1g</sub> → <sup>2</sup>E<sub>g</sub>) transition in an square planar geometry (Figgis, 1967). The complexes (14, 16, 18) show abroad absorption band in the region (12964-13766 cm<sup>-1</sup>), which was assigned to (<sup>2</sup>E<sub>g</sub> → <sup>2</sup>T<sub>2g</sub>) transition in octahedral geometry that resulted from the combination of three transitions (<sup>2</sup>B<sub>1g</sub> → <sup>2</sup>A<sub>1g</sub>), (<sup>2</sup>B<sub>1g</sub> → <sup>2</sup>B<sub>2g</sub>) and (<sup>2</sup>B<sub>1g</sub> → <sup>2</sup>E<sub>g</sub>) (Lever, 1984).

### Measurements of Magnetic Susceptibility

The values of magnetic moments for the complexes which were tabulated in (Table 1), have been measured at (25°C). magnetic moment's values for Co(II) complexes (1,3,5) are (2.38-2.53 B.M) suggesting a square planar geometry, while Ni(II) complexes (7,9,11) are diamagnetic. The magnetic moment values of the other Co(II) and Ni(II) complexes (2, 4, 6, and 8, 10, 12) were in the range (4.52-4.80B.μ) and (3.35-3.50B.μ) respectively, in an octahedral geometry (Gable, 1980).

The values of magnetic moment for four Cu(II) complexes (13, 15, 17) are (1.42-1.62B.μ) that correspond to the spin only value irrespective of the stereo chemistry, which suggests a square planer geometry around Cu(II) ion. The values of magnetic moment for hexa coordinated Cu(II) complexes (14, 16, 18) (Table 1) are in the range (1.78 – 1.94B.μ). The observed range is similar to other reported values of octahedral Cu(II) complexes (Saha and Gayen, 1983). Fig. (1) show the preposed structure of the prepared complexes.

**Table 1: Analytical and some physical properties of the prepared complexes**

No.	Compound	Color	m.p	Molar conductivity $\Omega^{-1} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$	%M	%yield	$\mu_{\text{eff}}$ B.M
L	$\text{C}_{19}\text{H}_{35}\text{OS}_2\text{K}$	Yellow	232	-----	-----	-----	-----
1	$[\text{Co}\{\text{Ag}(\text{oleylxant})_2\}_2]$	Green	143	14.5	3.83 (3.57)	62	2.46
2	$[\text{Co}(\text{phen})_3][\text{Co}\{\text{Ag}(\text{oleylxant})_2\}_2]$	Yellow	194	58.3	2.72 (2.69)	83	4.52
3	$[\text{CoCd}(\text{oleylxan})_4]$	Green	152	16.7	4.17 (3.81)	76	2.38
4	$[\text{Co}(\text{phen})_3][\text{CoCd}(\text{oleylxan})_4]$	Green	176	62.8	3.25 (2.82)	90	4.80
5	$[\text{CoZn}(\text{oleylxan})_4]$	Yellow	115	24.6	4.12 (3.93)	68	2.53
6	$[\text{Co}(\text{Phen})_3][\text{CoZn}(\text{oleylxant})_4]$	Green	184	53.8	3.14 (2.89)	92	4.62
7	$[\text{Ni}\{\text{Ag}(\text{oleylxant})_2\}_2]$	Yellow	119	18.0	3.78 (3.56)	77	Dia
8	$[\text{Ni}(\text{phen})_3][\{\text{Ag}(\text{oleylxant})_2\}_2]$	Yellow	165	52.4	2.93 (2.68)	65	3.35
9	$[\text{NiCd}(\text{oleylxan})_4]$	Green	132	13.8	4.15 (3.80)	83	Dia
10	$[\text{Ni}(\text{phen})_3][\text{Cd}(\text{oleylxan})_4]$	Green	172	56.9	3.35 (2.81)	61	3.50
11	$[\text{NiZn}(\text{oleylxan})_4]$	Yellow	187	17.3	4.34 (3.92)	96	Dia
12	$[\text{Ni}(\text{phen})_3][\text{Zn}(\text{oleylxan})_4]$	Yellow	228	67.4	3.29 (2.88)	75	3.38
13	$[\text{Cu}\{\text{Ag}(\text{oleylxant})_2\}_2]$	Yellow	146	18.4	4.27 (3.84)	87	1.42
14	$[\text{Cu}(\text{phen})_3][\{\text{Ag}(\text{oleylxant})_2\}_2]$	Yellow	192	51.0	3.12 (2.89)	90	1.78
15	$[\text{CuCd}(\text{oleylxan})_4]$	Yellow	127	22.8	4.43 (4.10)	74	1.56
16	$[\text{Cu}(\text{phen})_3][\text{Cd}(\text{oleylxan})_4]$	Brown	218	63.2	3.45 (3.04)	85	1.94
17	$[\text{CuZn}(\text{oleylxan})_4]$	Green	215	23.9	4.57 (4.23)	76	1.62
18	$[\text{Cu}(\text{phen})_3][\text{Zn}(\text{oleylxan})_4]$	Brown	221	67.5	3.59 (3.11)	89	1.86

**Table 2: IR bands (cm<sup>-1</sup>) and electronic spectral data of ligand and prepared complexes**

No.	Compound	UV bands (cm <sup>-1</sup> )	IR spectra		
			$\nu(\text{C-O})$	$\nu(\text{C-S})$	$\nu(\text{M-S})$
L	C <sub>19</sub> H <sub>35</sub> OS <sub>2</sub> K		1196	1055	-----
1	[Co{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	11407-16235	1230	1027	438
2	[Co(phen) <sub>3</sub> ][Co{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	9982, 16340, 21320	1223	1039	427
3	[CoCd(oleylxan) <sub>4</sub> ]	11485, 16197	1246	1027	442
4	[Co(phen) <sub>3</sub> ][CoCd(oleylxan) <sub>4</sub> ]	10624, 16327, 21354	1232	1027	428
5	[CoZn(oleylxan) <sub>4</sub> ]	11520-16296	1233	1045	465
6	[Ni{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	10463, 16297, 20918	1228	1052	424
7	[Ni{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	15187, 22791	1245	1020	454
8	[Ni(phen) <sub>3</sub> ][Ni{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	12483, 17961, 23582	1237	1025	432
9	[NiCd(oleylxan) <sub>4</sub> ]	15276, 23084	1215	1048	462
10	[Ni(phen) <sub>3</sub> ][Cd(oleylxan) <sub>4</sub> ]	12395, 23084	1209	1050	418
11	[NiZn(oleylxan) <sub>4</sub> ]	15228, 22935	1268	1036	468
12	[Ni(phen) <sub>3</sub> ][Zn(oleylxan) <sub>4</sub> ]	11962, 18219, 24196	1252	1042	430
13	[Cu{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	14739, 23495	1246	1034	446
14	[Cu(phen) <sub>3</sub> ][Cu{Ag(oleylxant) <sub>2</sub> } <sub>2</sub> ]	13567, 22597	1235	1042	426
15	[CuCd(oleylxan) <sub>4</sub> ]	14801, 22973	1238	1027	456
16	[Cu(phen) <sub>3</sub> ][Cd(oleylxan) <sub>4</sub> ]	12964, 23720	1224	1035	435
17	[CuZn(oleylxan) <sub>4</sub> ]	14625, 24720	1265	1018	450
18	[Cu(phen) <sub>3</sub> ][Zn(oleylxan) <sub>4</sub> ]	13766, 24256	1254	1028	424



## CONCLUSION

A new hetrobimecallic complexes were prepared from some transition metal ions, two series were used in this paper, the first one including Cobalt(II), Nickel(II), Copper(II), while the second one included the non-transition metal ions which were Zinc(II), Cadmium(II). As well as the ligand which was used named olyelxanthate, all the data were recorded experimentally for the resulted complexes as it assigned in this work, type complexes were hetrobimetallic tetra xanthate which react with 1,10-phenathroline to produce octahedral complexes.

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