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# SPECTRO-AND ELECTROCHEMICAL STUDIES OF GROUP 6 COMPLEXES WITH SALICYLIDENEIMINE-2-THIOPHENOL SCHIFF BASE

## Mostafa M.H. Khalil

Chemistry Department, Faculty of Science, Ain Shams University, Abbassia, Cairo, Egypt. e-mail : khalil62@yahoo.com

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# ABSTRACT

Interaction of the Schiff base salicylideneimine-2thiophenol (satpH<sub>2</sub>) with  $M(CO)_6$ , M= Cr and Mo, in THF under reduced pressure yielded the carbonyl derivative  $[Cr(CO)_2(satpH)_2]$  (1) and Mo(CO)<sub>4</sub>(satpH<sub>2</sub>) (2). Reaction of  $M(CO)_{k}$ , M = Mo and W with satpH<sub>2</sub> under atmospheric pressure resulted in the formation of the oxo-complexes  $M_0O_2(satp)$  (3) and  $W_2O_6(satpH_2)$  (4). Elemental analyses, spectroscopic studies of the complexes revealed that the ligand satpH<sub>2</sub> coordinated the metals, as a bidentate, from the azomethine nitrogen and either the OH or the SH groups or tridentate ligand through O.N and S. Electrochemical studies of the obtained complexes showed some reversible or irreversible redox reactions due to one electron transfer. The UV-vis spectra of the complexes in different solvents showed bands due to either metal-to-ligand or ligand-to-metal charge transfer.

## INTRODUCTION

Schiff bases and their complexes have been investigated for their interesting and important properties, such as their ability to reversibly bind oxygen [Chen & Martel (1987)], catalytic activity in the hydrogenation [Henrici & Olive (1984)], oxidation [Rao et al. (2007) and Mirckani et al. (2008)] or epoxidation of oleifens [Salvati-Niasari & Bazarganipour (2007)] and promising applications in biological activity. [You et al. (2006); Ren et al. (2002); Tarafdor et al. (2002); Raman et al. (2001, 2003,2004); Singh et al. (2004) and Thangadura

& Ihm (2004)]. Owing to the photochromic and thermochromic properties of the N-salicylidene aniline (SA) ligand, extensive studies were carried out [Zao et al. (2001); Zgierski & Grabowska (2000); Ogawa et al. (2000); Suzuki et al. (2000); Harada et al. (1999); Fukuda (2003) and Ogawa (1998)]. Schiff bases containing an ONS donor system gain more attention recently [Tarafdor et al. (2002); Raman et al. (2001); Soliman & Linert (1999a.b); Aboaly & Khalil (2001); Jayabalakrishnan & Natarajan (2002) and Soliman & Linert (2007)]. For example, complexes of salicylideneimine-2-thiophenol (satpH<sub>2</sub>) with some divalent ions such as Co(II). Ni(II). Cu(II). Zn(II) and Ru(II) were investigated [Aboaly & Khalil (2001); Jayabalakrishnan & Natarajan (2002) and Soliman & Linert (2007)]. Reactions of salicylideneimine-2-anisole (salanH); a ligand similar to satpH<sub>2</sub> with OCH<sub>3</sub> group instead of SH group, with  $M(CO)_6$ , M= Cr, MO, W gave  $Cr_2O_2(CO)_2(salan)_2$ ,  $M_2O_4(salan)_2$ , and  $W_2O_4(salan)_2$  [Ali et al. (2003)]. Spectroscopic and magnetic studies of these complexes revealed that they were paramagnetic and the metal existed in an octahedral environment. Also, the reactions between  $[M_3(CO)_{12}]$ , M = Ru and Os, and salicylideneimine-2-thiophenol Schiff base in THF under reflux gave  $[Ru(CO)_4(satpH)]$  and  $[Os(CO)_3(satpH_2)]$  complexes [Khali] et al. (2005)]. Structures of the two complexes were proposed on the basis of spectroscopic studies. Magnetic study of [Ru(CO)<sub>4</sub>(satpH)] suggested that a change in oxidation state of the ruthenium atom from zero to +1 was achieved via oxidative addition of the SH group with a proton displacement to give a low-spin  $d^7$  electronic configuration. In this paper, we report the thermal reactions of satpH<sub>2</sub> with group 6 metal carbonvls.

#### EXPERIMENTAL

 $M(CO)_6$ , M= Cr. Mo and W were purchased from Aldrich. The satpH<sub>2</sub> ligand was supplied from TCI-Ace, Japan. It was used without further purification. All solvents were of analytical grade and were purified by standard methods prior to their use.

Infrared measurements (KBr pellets) were performed on a Unicam-Mattson 1000 FT-IR spectrometer. Nuclear magnetic resonance measurements were carried out on a Varian 300 MHz spectrometer. Samples were dissolved in  $(CD_3)_2SO$  with TMS as internal reference. Electronic absorption spectra of *ca* 10<sup>-5</sup>M solutions were measured on a Unicam UV2-300 UV-vis spectrophotometer with 10.0 mm quartz cells. Cyclic voltammetry measurements were performed using a threeelectrode configuration cell connected to an EG and G scanning potentiostat model 372. A Pt disc was used as working electrode and a Pt wire as an auxiliary electrode. The reference was an Ag-AgCl electrode adjusted to 0.00 V versus SCE. Sample solutions (50 ml) of *ca.* 1 x  $10^{-3}$  M concentration in CH<sub>2</sub>Cl<sub>2</sub> with 0.1 M n-Bu<sub>4</sub>NPF<sub>6</sub> as supporting electrolyte were used for the measurements. A potential range of +2000 to -2000 mV, with a scan rate of 100 mVs<sup>-1</sup> was used. All the reported complexes were also characterized by elemental analyses (Perkin-Elmer 2400 CHN elemental analyzer) and mass spectrometry (Finnigan MAT SSQ 7000). Table 1 gives the elemental analysis and mass spectroscopy data of the complexes.

## $[Cr(CO)_2(satpH)_2]$

A mixture of  $Cr(CO)_6$  (0.1g, 0.45 mmol) and salicylideneimine-2thiophenol (0.1g, 0.44 mmol) in *ca.* 30 cm<sup>3</sup> THF was degassed with one freeze-thaw cycle and then heated to reflux for 7h. The color of the solution changed from pale-yellow to deep-red then to reddish brown and finally to dark green. The reaction mixture was cooled and the solvent was removed on a vacuum line. The residue was washed several times with petroleum ether and then recrystallized from hot benzene to give dark green crystals. The complex was left to dry under vaccum for several hours (yield 50 %).

#### $[Mo(CO)_4(satpH_2)]$

Similar procedure was employed as used in case of  $Cr(CO)_2(satpH)_2$  with a reaction period 1h. Dark brown crystals with a yield 75%.

#### $[MoO_2(satp)]$

A mixture of Mo(CO)<sub>6</sub> (0.10 g, 0.38 mmol) and satpH<sub>2</sub> (0.11 g, 0.38 mmol) in *ca*. 25 cm<sup>3</sup> THF was refluxed for 24 h during which the product precipitated as brown solid. The residue was isolated by filtration. The complex was washed several times with boiling petroleum ether and then recrystallized from THF (yield 56%).

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# $[W_2O_6(satpH_2)]$

 $W(CO)_6$  (0.15g, 0.43mmol) and satpH<sub>2</sub> (0.1g satp, 0.43 mmol) in about 30 ml dioxan were mixed and refluxed under atmospheric pressure for 16h. The color of the reaction mixture turns from pale yellow to reddish brown with a brown solid precipitate formed during the reaction time. The reaction mixture was cooled and the residue was isolated by filtration. The complex was washed several times with boiling petroleum ether and then recrystallized from DMF. The complex was left to dry in vacuo for several hours to give powder solid (yield 76%).

	Fou	nd (Calcd.) (	Mass Spectrometry		
Complex	С	H	N	Mol. Wt.	m/:
[Cr(CO) <sub>2</sub> (satpH) <sub>2</sub> ]	58.6(59.4)	3.9(3.9)	4.8(4.95)	566.0	528 [P-CO] <sup>+</sup>
[Mo(CO)₄(satpH₂)]	46.2 (46.7)	2.40 (2.5)	3.3 (3.2)	437.3	325 [P-4CO]⁺
[MoO <sub>2</sub> (satp)]	44.2(43.93)	2.60(2.55)	4.0(3.94)	682.5	683 [P] <sup>+</sup>
[W₂O₀(saıpH₂)]	22.6(22.52)	1.67(1.6)	1.75(2.02)	692.8	693 [P] <sup>+</sup>

Table (1): Elemental and mass spectrometry data of the complexes.

### **RESULTS AND DISCUSSION**

The infrared spectrum of the satpH<sub>2</sub> ligand exhibited a strong sharp band at 3258 cm<sup>-1</sup>. This band was assigned as due to the stretching frequency of N<sup>-</sup>H group, which is expected in this region [Khalił et al. (2005) and Brezinski & Zundel (1985)]. The spectrum of satpH<sub>2</sub> also displayed a band at 1619 cm<sup>-1</sup> which could be due to vC=N. Interestigly, the IR spectrum of the ligand does not display any band due to the OH streching frequency [Khalil et al. (2005)]. However, the <sup>1</sup>H NMR spectrum of the ligand in DMSO-d<sub>6</sub> displayed two slightly broad singlets at 11.61 ppm and 9.53 ppm which probably due to the OH and N<sup>+</sup>H moities, respectively. The fact that the IR spectrum of compound satpH<sub>2</sub> did not show any OH stretching frequencies was explained as a result of the presence of intramolecular hydrogen bonding between the OH and S<sup>\*</sup> moleties [Khalil et al. (2005)].

Interaction of Cr(CO)<sub>6</sub> with satpH<sub>2</sub> in THF under reduced pressure resulted in the formation of the mononuclear complex with molecular formula  $Cr(CO)_2(satpH)_2$  1. The IR spectrum of the complex showed two medium bands at 1950 and 1870 cm<sup>-1</sup> in the terminal metal carbonyl region corresponding to symmetric and asymmetric stretching frequencies of two CO groups [Nakamoto (1997)]. The IR spectrum of the complex displayed a very strong sharp band at 1603 cm<sup>-1</sup> due to the vC=N stretching frequency. This band was shifted to lower frequency relative to that of ligand indicating that complexation occured via the nitrogen atom of the azomethine group. The number and pattern of the CO bands in the IR spectrum suggested that they were bound to the metal in cis positions of a distorted octahedral arrangement [Ali et al.(2002)]. Furthermore, the appearance of a strong band due to phenolic C-O streching frequency at 1270 cm<sup>-1</sup> indicating that coordination of the metal to the phenolic oxygen accompanied with proton displacement [Ali et al. (2002) and Collman & Hegedus (1980)]. Such type of bonding suggested that the ligand coordinated to the metal oxidatively. In addition, the far IR spectrum of the chromium complex exhibited two bands at 534 and 423 cm<sup>-1</sup>, due to Cr-O and Cr-N bonds, respectively. that confirmed the bonding of phenolic oxygen and azomethine nitrogen of the satpH<sub>2</sub> Schiff base to the metal ion [Nakamoto (1997)]. Magnetic susceptibility measurements of the complex at 298 K gave a value of an effective magnetic moment ( $\mu_{eff}$ ) of 2.70 BM. The  $\mu_{eff}$  value indicated a low-spin d' electronic configuration. This electronic configuration is in accordance with +2 formal oxidation state of the chromium in the complex. Therefore, it can be concluded that the chromium complex existed in an octahedral environment and has the proposed structure, 1. Reaction of Cr(CO)<sub>6</sub> with N-salicylidene-2-hydroxyaniline (shaH<sub>2</sub>); a ligand similar to satpH<sub>2</sub> with OH group instead of SH group, in THF under reduced pressure resulted in the formation of the dicarbonyl complex Cr(O)<sub>2</sub>(CO)<sub>2</sub>(shaH<sub>2</sub>) [El Medani (2003)]. The IR spectrum of Cr(O)<sub>2</sub>(CO)<sub>2</sub>(shaH<sub>2</sub>) complex exhibited two bands in the terminal metal carbonyl range at 1863 and 1948 cm<sup>-1</sup> due to symmetric and asymmetric stretching frequencies of two CO groups. In addition, the IR spectrum of the complex also showed two strong bands at 875 and 925 cm<sup>-1</sup> due to the presence of two Cr=O bands.

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Heating an equimolar mixture of Mo(CO)<sub>6</sub>, and satpH<sub>2</sub> in THF under reduced pressure resulted in the formation of the tetracarbonyl derivatives [Mo(CO)<sub>4</sub>(satpH<sub>2</sub>)], 2. The IR spectrum of the complex showed bands due to the ligand moiety with the corresponding shifts, Table 2. Also, it displayed four carbonyl bands in the terminal metal carbonyl region with a symmetry  $2a_1+b_1+b_2$  due to symmetric and asymetric streching frequencies. The number and pattern of the CO bands suggested that the molybdenum existed in an octahedral environment and the ligand occupied two equatorial positions [Nakamoto (1997)]. The <sup>1</sup>H NMR spectrum of 2 displayed signals similar to that of the ligand with an appropriate shift of the signal of the CH-N proton. The fact that IR spectrum of the complex displayed an OH band at 3423 cm<sup>-1</sup>, the range of free OH group, indicated that the OH group did not participate in the coordination. The observation of vOH band in the IR spectrum of the complex may be due to break down of the hydrogen bond in the free ligand during complex formation. Therefore, according to the spectroscopic studies, the satpH<sub>2</sub> coordinated the metal through SH and azomethine nitrogen.

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OH (2)

Refluxing of Mo(CO)<sub>6</sub> and satpH<sub>2</sub> in THF under atmospheric produced the mononuclear pressure for 16h dioxo-complex [MoO<sub>2</sub>(satp)], **3**. The infrared spectrum of **3** displayed two stretching bands at 925 and 906 cm<sup>-1</sup> due to symmetric and asymmetric frequencies of two terminal cis-Mo=O bonds [Nakamoto (1997)]. The infrared spectrum of the molybdenum complex displayed a band at 1610 cm<sup>-1</sup> due to v(C=N) stretching frequency of the azomethine group, in addition to new bands at 553 and 475 cm<sup>-1</sup> due to Mo-O and Mo-N bonds, respectively [Nakamoto (1997)]. The <sup>1</sup>H NMR spectrum of 3 showed no signals for OH or SH groups, suggesting participation of the two groups in coordination with proton displacement and molybdenum exist in +6 formal oxidation state. However, interaction of N-salicylidene-2hydroxyaniline ( $shaH_2$ ) with Mo(CO)<sub>6</sub> in air gave two complexes MoO(sha) and  $Mo_2O_4(sha)_2$  depending on the reaction period [E]-Medani (2003)]. It can be concluded that the type of ligand and the reaction conditions play a vital role in determining the type of products isolated.

From the spectroscopic data one can conclude that  $satpH_2$  ligand coordinated to molybdenum through O. N and S as tridentate ligand. This indicate that the complex exist in square pyramidal structure forming an oligomer chain through oxygen in the solid state. However, in donor solvents such as DMSO. DMF, EtOH or py, the oligomeric chains are broken and monomeric cis-dioxomolybenum complexes are obtained in solution [Tomić et al. (2007)].

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(3)

Interaction of  $[W(CO)_6]$  with satpH<sub>2</sub> under atmospheric pressure resulted in the formation of a brown complex with a molecular formula of  $[W_2O_6(satpH_2)]$ , 4. The IR spectrum of the tungsten complex showed the ligand bands with appropriate shifts as a result of complex formation (Table 2). The C=N stretching vibration of the tungsten complex displayed a shift of 20 cm<sup>-1</sup> to lower energy relative to that of ligand (Table 2) indicating the coordination of the azomethine nitrogen atom to the metal. The infrared spectrum of 4 also displayed two strong vibrational bands at 978, 900 and very strong band at 813 cm<sup>-1</sup> due to symmetric and asymmetric frequencies of terminal W=O bonds. The band at 813 cm<sup>-1</sup> in the IR spectrum of the present tungsten complex might be due to two trans W=O groups [Nakamoto (1997)]. In addition, stretching infrared bands were observed at 642, 554 and 444 cm<sup>-1</sup> corresponding to W-O-W, W-O and W-N bonds, respectively (Table 2). Magnetic measurements showed diamagnetic properties for 4. According to the proposed structure, tungsten may have +6 formal oxidation state with d<sup>0</sup> electronic configuration. The <sup>1</sup>H NMR spectrum of 4 displayed a signal at 11.60 due to OH group and absence of the SH signal. This probably due to coordination of SH group to the metal without proton displacement, i.e., the ligand reacted oxidatively.



 Table (2): Important IR data for salicylideneimine-2-thiophenol and its complexes.

	IR data (cm <sup>-1</sup> )				NMR
Compound					
	v (OH)	v (C=N)	v (C≖C)	v(CO)	
satpH <sub>2</sub>		1619(s)	1462 (m)	***	11.61(s,OH), 9.75(s, CH-
- I +					N), 9.53 (s, NH <sup>*</sup> ), 6.51-
					7.57(m, aromatics)
(d)		1603(m)	1483(\$)	1950, 1870	
			1461(s)		
(2)	3423	1608(s)	1460(s)	2017.1980,	11.62(s,OH) 9.58(s, CH-N),
·-•				1944, 1890	6.47-7.63 (m, aromatics),
			1457(s)		4.32(s, SH)
(3)	******	1604(s)	1460(vs)	******	9.57(s, CH-N), 6.23-7.59(m,
					aromatics)
(4)	*	1600(m)	1483(s)	*****	11.60, 9.57(s, CH-N), 6.38-
			1456(s)		7.62(m, aromatics)

### **UV-vis studies**

The electronic absorption spectra of the satpH<sub>2</sub> and its the complexes have been measured in three different solvents; EtOH, DMSO and CH<sub>2</sub>Cl<sub>2</sub>. Table 3. The absorption spectra of satpH<sub>2</sub> and complexes in DMSO are shown in Fig. 1. Two electronic transition bands were observed for satpH<sub>2</sub> in the range of 279-282 and 323-329 nm due to  $\pi$ - $\pi$ <sup>\*</sup> of the aromatic system and n- $\pi$ <sup>\*</sup> transitions, respectively [Khalil et al. (2005)]. The position of the first band at 279-282 nm was slightly influenced by the solvent polarity confirming the local  $\pi$ - $\pi$ <sup>\*</sup> nature of the

transition leading to these bands. The other band at 323-329 ascribed to transition between the  $\pi$ -orbital largely localized on the central C=N bond, influenced by the charge transfer within the entire molecule. This band is commonly observed in o-hydroxy Schiff bases [Maurya et al. (1999)].

For all complexes, the band occurred in the range of 272-283 nm showed an insignificant effect by chelation. On the other hand, the band at 323-329 nm showed a slight shift to longer wavelength, Table 3, with a considerable change in the molar absorbitivity on going from ligand to complex. indicating coordination of ligand to metals through the azomethine moiety. Similar phenomena was observed for the complexes of salicylideneimine-2-anisole [Ali et al. (2003)]. In addition, the complexes exhibited absorption bands at 410-500 nm. which could be due to metal-to-ligand charge transfer transitions as for complexes 1 and 2 or due to ligand-to-metal charge transfer as for complexes 3 and 4 [Khalil et al. (2001)].



Fig. (1): Absoprption spectra of satpH<sub>2</sub> and its complexes in DMSO: (---) satpH<sub>2</sub>. (---) [Cr(CO)<sub>2</sub>(satpH)<sub>2</sub>]. (-.-.) [Mo(CO)<sub>4</sub>(satpH<sub>2</sub>)]. (....)[MoO<sub>2</sub>(satp)] and (-....)[W<sub>2</sub>O<sub>6</sub>(satpH<sub>2</sub>)].

Compound	Solvent	λ <sub>max</sub> (nm)
satpH <sub>2</sub>	CH <sub>2</sub> Cl <sub>2</sub>	283, 329
	EtOH	282,329
	DMSO	281, 323
[Cr(CO)2(satpH)2]	CH <sub>2</sub> Cl <sub>2</sub>	272, 327 <sup>a</sup> , 411
	EtOH	271, 310, 409
	DMSO	275, 323, 420,500 <sup>b</sup>
[Mo(CO) <sub>4</sub> (satpH <sub>2</sub> )]	CH <sub>2</sub> Cl <sub>2</sub>	276 <sup>a</sup> , 339 <sup>a</sup> , 448 <sup>a</sup>
	EtOH	273, 285 <sup>a</sup> , 320 <sup>a</sup> , 427 <sup>a</sup>
	DMSO	281, 305,460°
MoO <sub>2</sub> (satp)	CH <sub>2</sub> Cl <sub>2</sub>	281, 326 <sup>°</sup> , 437 <sup>ª</sup>
	EtOH	274, 333, 431
	DMSO	272 <sup>a</sup> , 326, 383 <sup>a</sup> , 460 <sup>a</sup>
W <sub>2</sub> O <sub>6</sub> (satpH <sub>2</sub> )	CH <sub>2</sub> Cl <sub>2</sub>	286, 335, <u>3</u> 83, 455 <sup>a</sup>
	EtOH	279, <u>3</u> 31, <u>370</u> , 457 <sup>a</sup>
	DMSO	281, 328,373, 462 <sup>a</sup>

Table (3): UV-vis data for salicylideneimine-2-thiophenol and itscomplexes in different solvents.

<sup>a</sup>:shoulder, <sup>b</sup>. broad, <sup>c</sup>: weak

### **Electrochemical studies**

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The electrochemical behaviour of satpH<sub>2</sub> and its chromium, molybdenum and tungsten complexes were investigated in CH<sub>2</sub>Cl<sub>2</sub> with a scan rate of 100 mVs<sup>-1</sup> (Fig. 2). The cyclic voltammogram of the free satpH<sub>2</sub> displayed three irreversible peaks; one cathodic and two anodic. The cathodic peak which occurred at Ep<sub>c</sub> = -0.47 V was assigned to the reduction of the azomethine group. On the other hand, the two anodic peaks occurred at Ep<sub>a</sub> = 0.28 and 0.98 V might be corresponded to oxidation of SH and OH groups, respectively [Kadish (1982)]. The cyclic voltammograms of the [Cr(CO)<sub>2</sub>(satpH)<sub>2</sub>], [MoO(satpH<sub>2</sub>)]<sub>2</sub> and  $[W_2O_6(\text{satp})]$  complexes were consistent with one irreversible metal based reduction (Ep<sub>c</sub> = -1.21 and -1.29 V, and -1.32 V, respectively) due to the reduction of the metal. The ligand did not show exhibit any redox acivity in the potential range where the metal reductions are occurred. These reduction peaks were shifted to more negative potentials from Cr(11) to W(VI) which was consistent with complex formation.



Fig. (2): The cyclic voltammetry plots of: (a)  $satpH_2$ , (b)  $[Cr(CO)_2(satpH)_2]$  (c)  $[MoO_2(satp)]$  and (d)  $W_2O_6(satpH_2)]$  in  $CH_2Cl_2$ .

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الملخص العربى

**مصطفى محمد حسن خليل** قسم الكيمياء – كلية العلوم – جامعة عين شمس

(Cr - M حب M(CO)<sub>6</sub> بقاعل فاعدذ شيف سالسيلدين إميتو -2-ثيوفينول (satpH<sub>2</sub>) مع مداسي كربونيل الفلز M(CO)<sub>6</sub> محب M(CO)<sub>2</sub> و أو M ف مسديب رباعسي هسيدرو فسيوران تحت ضغط متخفض نتج عنه متراكين (1)<sub>2</sub>(satpH)<sub>2</sub>(2) و (2) [(2) [(Mo(CO)<sub>4</sub>(satpH<sub>2</sub>)] - حيث تم استبدال جزئي لمحموعات الكربونيل. من ناحية أحري فإن تفاعل M(CO)<sub>6</sub> و محبث M - M أو W تحسب الطغط الجوى أنتج متراكبات أوكسو لها التركيب الجريتي (3) (MoO<sub>2</sub>(satp فريب MoO<sub>2</sub>(satpH<sub>2</sub>)]. أظهرت نتائج تحاليل العناصر والقياسات الطيفية أد عدا الميحاند يتناسق كتنائي العطاء من حسلال ذرة نيتسروجين بحمسوعة الميستاين و ذرة الاكسجين أو الكيريت أو كتلامي العطاء من خلال ذرات النيتروجين. الاكسسجين و الكسيريت. أوضحت دراسات الكيمياء الكهرية وجود تعاعلات أكسدة واختزال منعكسة أو غير منعكسه نتسيجة لانقال الكترون واحد. تم مناقشة أطباف UV-Vis للمتراكبات النائعة في مدينات عنافة وقد حرم امتصاص نتيجة انقال السحنة من العلز الي الليجاند أو من الميجاند الناقين وحود حرم امتصاص نتيجة انقال السحنة من العلز الي الميجاند أو من الميجاند النائية وقد أطهرت وحود حرم