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# Preparation and characterization of Schiff base and its complex with Cu(II) and study of their liquid crystalline properties.

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## ABSTRACT

The research includes the preparation of ligand E-H, and it was identified by spectroscopic methods Infra Red IR, Nuclear magnatic Reasonance NMR, Mass spectra· It was also prepared from the ligand a complexes with copper (11) and characterized by Infra Red spectra. Central metals ions and Schif base coordinated via O and N donor sites which was observed from IR spectra. The liquid crystal characteristics of the ligand was studied using polarized optical microscope POM and the ligand is a liquid crystal by showed nematic phase only, while no liquid crystal phase was detected in the prepared complex, and it disintegrated at 250°C.

## 1. Introduction

The emergence of liquid crystalline properties depends on the attractive forces between molecules as well as between their ends[1].The linking groups containing multiple bonds such as CH=N, that maintain the linearity and rigidity within the molecule are also satisfactory, furthermore, conjugative interaction group linking affected by the ester, - COO- and two aromatic rings also leads to some double bond character. Consequently, both the CH=N and ester groups connecting groups are considered as good to design new molecules with varying mesomorphism [2-4]. The terminal substituents would affect the polarizability of the aromatic ring to which they attached. This is attributed to increased intermolecular attractions with increasing polarity and polarizability of the molecule [5,6].The compounds of Schiff bases substituted with a hydroxyl group in the Ortho position for the Schiff are suitable compounds for the preparation of complexes with transitional elements Cu, Ni, VO and Pd [7-9]. known K. Najati and *et al*, prepared three complexes of Cu and Ni, as well as VO, these complexes are derived from the same ligand.It was found from the study the cu and Ni Complexes liquid crystals and showed the smectic phase, while the Vo complex did not show any liquid crystal phase [10].

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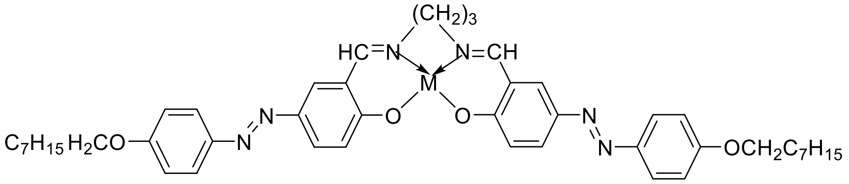


CuL $C \xrightarrow{230^{o}C} S_A \xrightarrow{255\ ^{o}C} I$ $\Delta T_{SA} = 25\ ^{o}C$

NiL $C \xrightarrow{239^{o}C} S_A \xrightarrow{261^{o}C} I$ $\Delta T_{SA} = 22\ ^{o}C$

VOL $C \xrightarrow{380\ ^{o}C} I$

## 2. Experimental

### 2.1 General

Infrared spectra were recorded with KBr pellets on a Buck – M500 spectrometer (Buck Scientific, USA). $^{1}$H- and $^{13}$C-NMR spectra were recorded on a Bruker instrument using $CDCl_3$ as solvent and TMS as internal standard. Transition temperatures were determined using a Perkin Elmer-Pyris DSC-8000 differential scanning calorimeter with a heating rate of 10°C $min^{-1}$. The phase transitions were observed with a Leitz Labor lux 12 Pol optical microscope with polarized light in conjunction with a Leitz 350 hot stage (Germany) equipped with a Vario-Orthomat. Mass spectra were recorded on an Agilent technologies 5957C spectrometer.

### 2.2 Synthesis of Schiff bases

One of molar equivalent of 4-amino acetophenone was added to a solution of the 2,4-dihydroxy Benzaldehyde (1:1) to which a few drops of acetic acid were added according to the earlier reported method [11, 12]. The mixture was heated under reflux for 2 hours, and the solid obtained upon cooling was filtered and twice recrystallized form ethanol to give pure compounds (scheme.1).

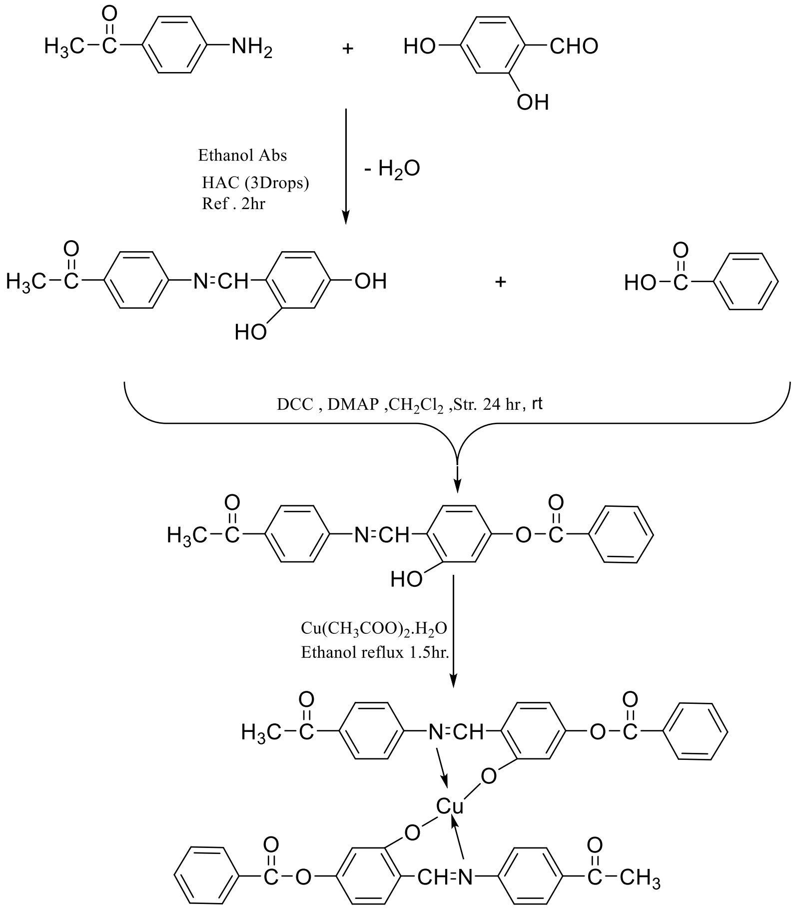
### 2.3 Synthesis of esters 4-(((4-acetylphenyl)imino)methyl)-3-hydroxyphenyl benzoate (E-H)

Molar equivalents of acids and phenols (0.01 mol) were dissolved in 25 ml of DCM to the resulting mixture, DCC (0.002 mol) and DMAP (0.002 mol) were added and the mixture was kept stirring at room temperature for 6 hours following the reported procedure [13,14].The precipitated solid (by product) was filtered off, and the filtrate was evaporated, and the residue was twice crystallized from ethanol (Scheme .1). Chemical formula : $C_{22}H_{17}NO_4$; yellow solid; yield 72%; $^{1}$H-NMR spectrum ($CDCl_3$, 400 MHz): δ (ppm) 2.64 ppm ( S , 3H ), 6.87 - 8.22 ppm (m ,9H , Ar-H ), 8.67 ppm (S , 1H, CH=N), 13.26 ppm (S , 1H , OH ), ( Fig. 1). $^{13}$C-NMR ($CDCl_3$, 100 MHz): δ (ppm) 26.8 ppm ($CH_3$), 55.6 ppm ($COCH_3$),111.4-155.4 ppm (Ar-C),153.0 ppm (CH=N),164.2 ppm (C-OH), 197.2 ppm (COO), (Fig 2). (IR ($cm^{-1}$): 3344 (OH) 3064 (C-H aromatic), 2927-2854 (C-H aliphatic), 1734 (C=O ester), 1680 (C=O ketone), 1647 (CH=N) , ( Fig. 3), MS (m/z) 359.2 [$M^{+}$] , ( Fig. 4).

### 2.4 Synthesis of Complex Bis(2-(((4-acetylphenyl)imino)methyl) (benzoyloxy)phenoxy) copper (Cu-E-H)

Copper(II) acetate monohydrate was dissolved in ethanol and added to a solution of free ligand in ethanol. The reaction mixture was stirred and refluxed for 1.5 hours. The brownish crystals were filtered and recrystallized from a solvent mixture of DMF (Scheme .1), [15, 16].

Chemical formula : $C_{44}H_{32}CuN_2O_8$; Green solid; yield 72%; (IR ($cm^{-1}$): 2875 (C-H aliphatic), 1737 (C=O ester), 1676 (C=O ketone), 1612 (CH=N) , ( Fig. 5).



**Scheme 1.** Steps of preparation the ligand and their complex with Cu(II)

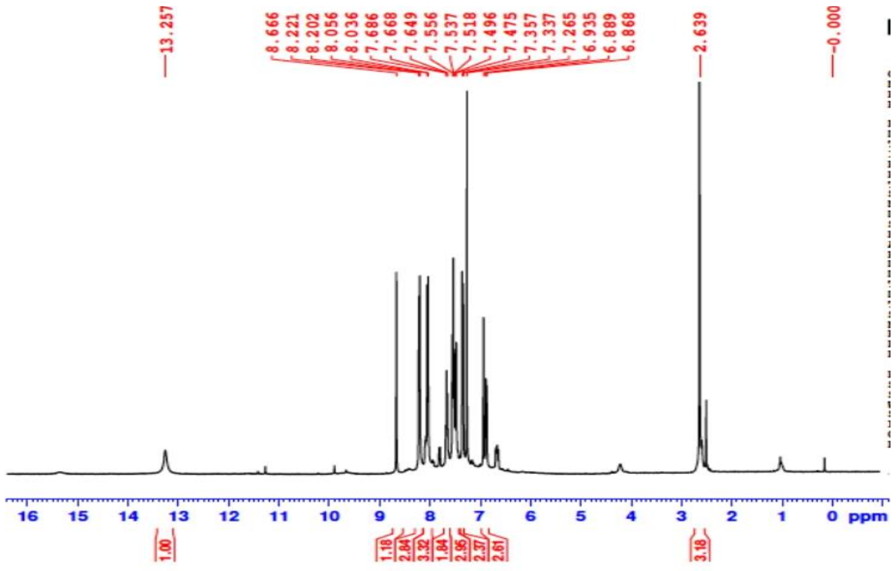
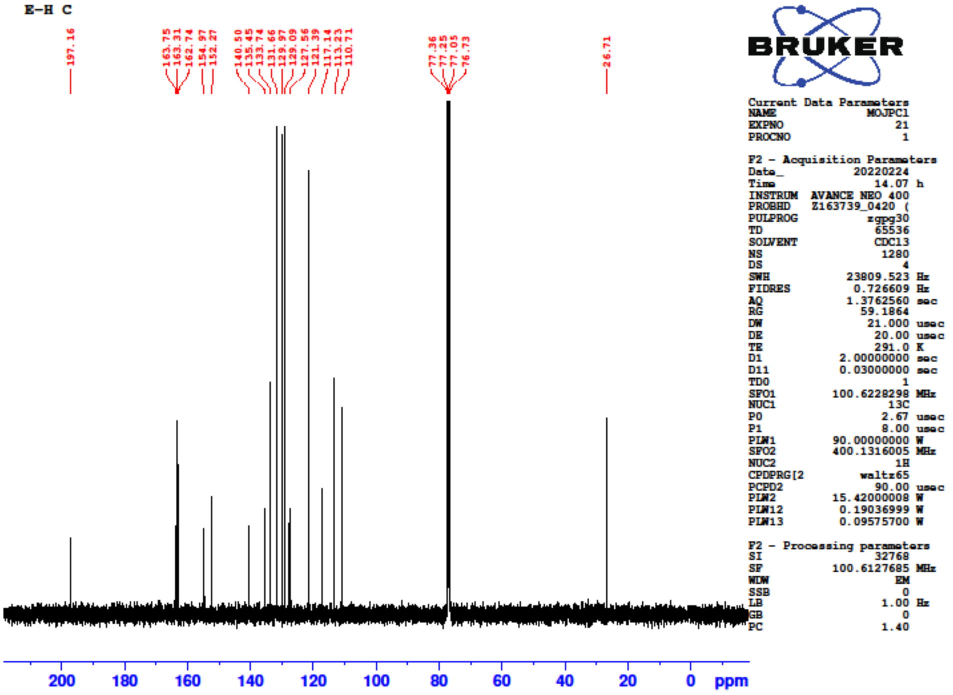


**Fig. 1.** $^1$H-NMR spectrum for E-H



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PROCNO                  1

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PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1280
DS                      4
SWH             23809.523 Hz
FIDRES           0.726609 Hz
AQ              1.3762560 sec
RG                59.1864
DW                 21.000 usec
DE                  20.00 usec
TE                  291.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P0                   2.67 usec
P1                   8.00 usec
PLW1          90.00000000 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          15.42000008 W
PLW12          0.19036999 W
PLW13          0.09575700 W

F2 - Processing parameters
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WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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**Fig. 2.** $^{13}$C-NMR spectrum for E-H

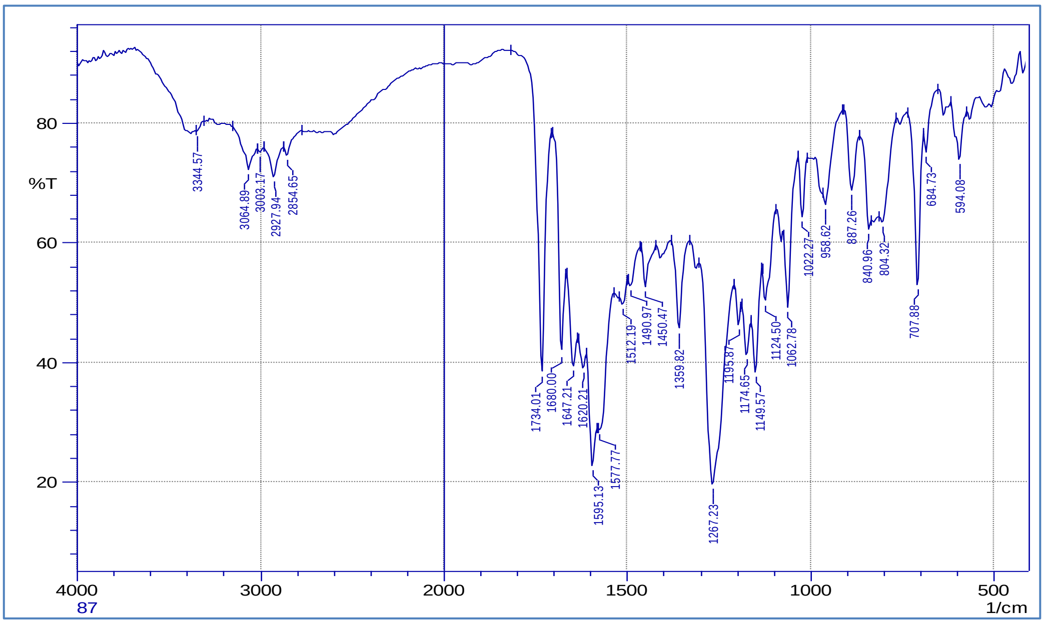


**Fig.3.** IR spectrum for E-H

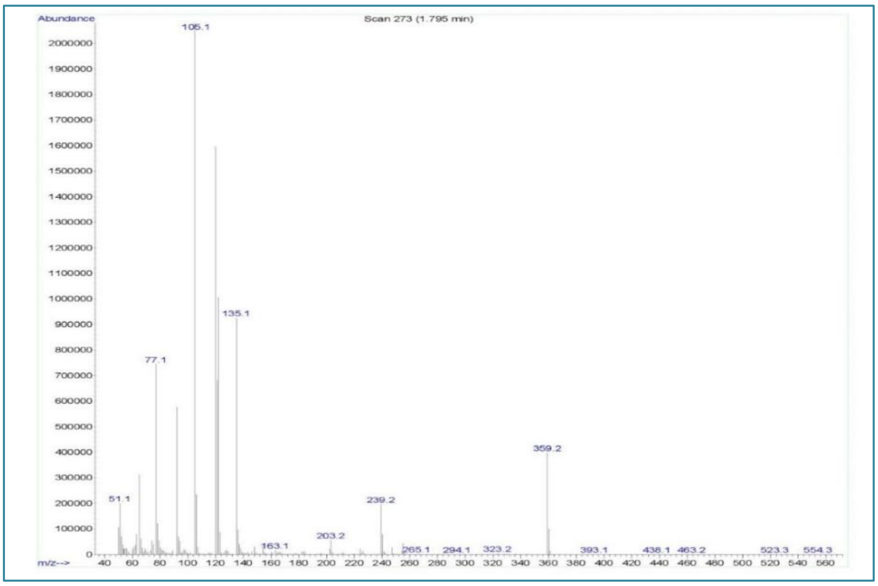


**Fig.4.** Mass spectrum for E-H

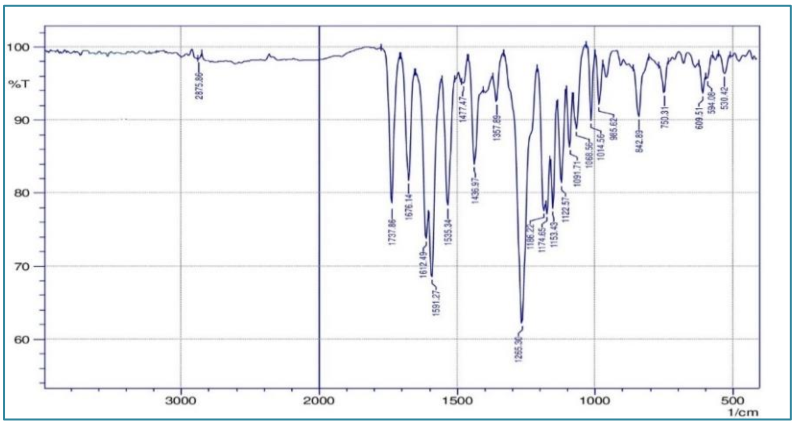


**Fig.5.** IR spectrum for Cu-E-H

## 3. Discussion

The ratio of lateral to terminal. attractive forces determines the type of liquid crystal intermediate phase in organic compounds that have these characteristics[17].It was found from studying the ligand E-H with a polarizing microscope that the ligand is a liquid crystal and it showed the nematic phase at 182˚C and turned into a liquid state at 196˚C with a thermal range 14 $^{O}$C (Fig 6). As a result of the presence of electronic succession along the longitudinal axis and the presence aft variation in thepolarity the molecule, but as a result of the presence of the carbonyl group at the of molecule,which provides a strong dipole moment across the axis of the molecule which enhances the emergence of the

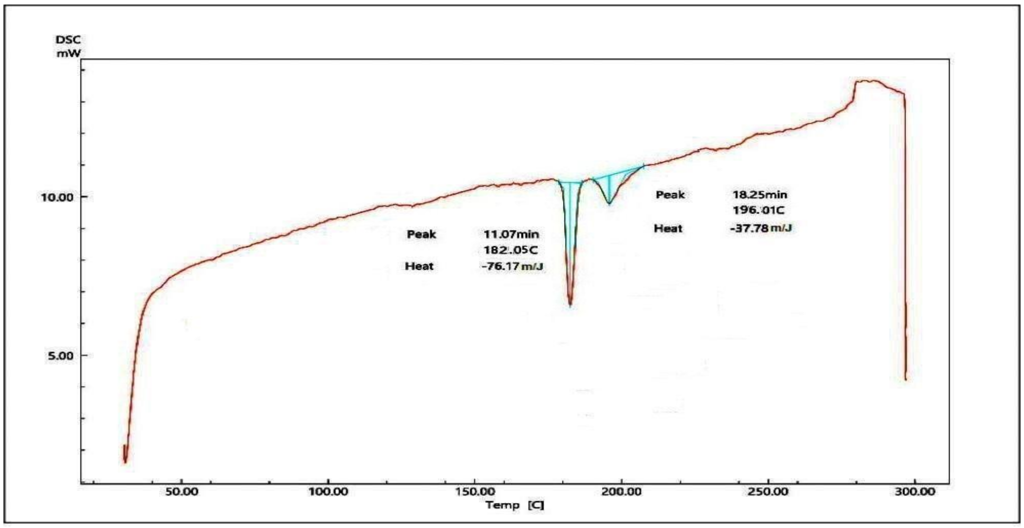
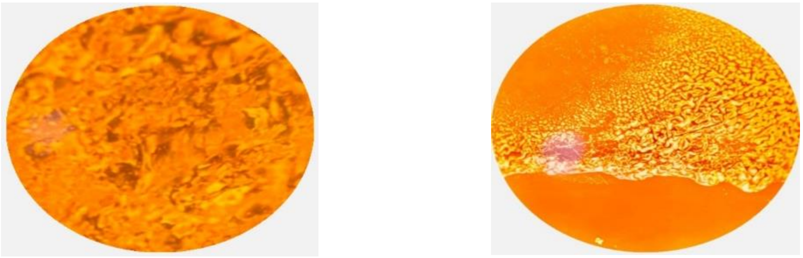


**Fig.6:** DSC thermogram for E-H



**a** **b**

**Figure7:** a) Marbled texture in Nematic phase for compound E-H on

smectic phase but this phase was not diagnosed in the ligand because these forces are not sufficient for the emergence of smectic phase [18].The nematic phase took the marbled texture in heating and schlieren texture in cooling [18], ( Fig. 7). The derived complex from ligand E-H has not been diagnosed with any liquid crystalline phase and dissociation at 250 °C . This is due to the high parrier at the complex solidity, which led to its disintegration directly

## 4. Conclusion

The study showed that the ligand has only the nematic phase, which means that the terminal attractive forces are higher than the lateral ones, but with a non-wide thermal range, because one of the ligand sides not compensated by a terminal group that enhances the terminal attractive forces, as well as the presence of a carbonyl group on the other side.

Liquid crystalline phase and dissociation, because one of the factors that promote the appearance of liquid crystalline properties in compounds is the presence of a percentage of hardness and flexibility, and if either of them is on it, it causes the loss of these characteristics.

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# تحضير وتشخيص قاعدة شف ومعقداتها مع النحاس (II) ودراسة خصائصها البلورية السائلة

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## معلومات البحث

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## الكلمات المفتاحية

Schiff base, liquid crystals, Cu(II) complexes.

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## الملخص

يتضمن البحث تحضير الليكاند E-H، والذي تم تشخيصه بالطرق الطيفية طيف الاشعة تحت الحمراء ، الرنين النووي المغناطيسي وطيف الكتلة · كما تم تحضير معقد الليكاند مع النحاس (II) والذي تم تشخيصه من خلال طيف الاشعة تحت الحمراء. اظهرت النتائج أن التناسق بين أيون الفلز وقاعدة شف يكون عبر المواقع المانحة للأكسجين والنيتروجين. تمت دراسة خصائص البلورات السائلة لليكاند ومعقد النحاس باستخدام المجهر الضوئي المستقطب. أوضحت النتائج ان الليكاند يظهر الصفاتت البلورية سائلة من خلال الطور النيماتي الموضح فقط، في حين لا توجد صفات بلورية سائلة في المعقد المحضر، والذي تفكك عند درجة حرارة 250 درجة مئوية.

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