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SYNTHESIS OF NICKLE (II) COMPLEX WITH NEW LIGAND DI (FURANYL METHYLENE HYDRAZONE) PIPERAZINE (DFMHP)

Annotation: *The synthesise of a new ligand (L) di(Furanyle Methylene Hydrazone) Piperazine (DFMHP) was carried by condensation of 1,4 di formyle piperazine with hydrazine mono hydrate ,Then add furfural to getting ligand (L). Then the reaction of this ligand with Nickle(ll) ion were carried out using metal Chloride salt by the (1:2) molar ration respectively conduced . [Ni_2LCl_4]*

The ligand and complexes were characterized and studied on the basis of (FT-IR) and (^{1}H-NMR, ^{13}C-NMR). the results were comparative with the proposed structures.

Key words: *reactions of condensation , Hydrazine Hydrate, Piperazine.*

СИНТЕЗ КОМПЛЕКСА НИКЕЛЯ (II) С НОВЫМ ЛИГАНДОМ ДИ (ФУРАНИЛМЕТИЛГИДРАЗОНО) ПИПЕРАЗИНОМ (DFMHP)

Аннотация: *В исследовании был проведен синтез нового лиганда (L) ди (фуранилметилгидразона) пиперазина (DFMHP) путем конденсации 1,4-диформилпиперазина с моногидратом гидразина, затем добавляли фурфурол для получения лиганда (L).*

Затем реакцию этого лиганда с ионом Nickle (ll) проводили с использованием соли хлорида металла с помощью соответственно проведенного молярного соотношения (1: 2). [Ni2LCl4]

В заключении лиганд и комплексы были охарактеризованы и изучены на основе (FT-IR) и (1 H-ЯМР, 13 C-ЯМР). результаты были сопоставимы с предлагаемыми структурами.

Ключевые слова: *реакции конденсации, гидразин гидрат, пиперазин.*

1.Introduction:

Schiff base ligands have been widely studied in the field of coordination chemistry mainly due to their facile synthesis, easy availability and good solubility in common solvents. They are generally known as azomethine compounds due to the presence of azomethine bond [1, C. 1555].

Piperazine is an organic compound that consists of a six-membered ring containing two nitrogen atoms at opposite positions in the ring. Piperazine ring is extensive structural elements in drug finding, with a large variety of successes experienced in biological applications such as drug designing [2,C. 1744].In the early 1900's piperazine was used about the turn of the century for the treatment of gout. Its first successful use in helminthiasis led to extensive use as a human and animal antihelminthic, for more than 50 years, the drug is used in the treatment of infections caused by Ascaris lumricoides and Enterobius vernicularis8 and certain of its compounds have been investigated for the treatment of cancer radiation sickness and angina pectoris [3, C. 282].

2.Experimental

2.1.Apparatus and chemicals:

All the chemicals used were purchased from both Merck and Sigma Aldrich companies and used without further purification. FT-IR spectra was recorded using Jasco Japanese taype (A) Infrared Spectrophotometer Fourier Transform FT-IR-4100 (KBr).

2.2.Experimental Procedure:

2.2.1. Synthesis of the ligand (L):

The Schiff base, (L) was prepared by adding 1,4 di formyle piperazine(1.44 g , 10 mmol) to hydrazine mono hydrate (2.5 ml , 30 mmol) then add furfural (2.5 ml , 30 mmol) in ethanol 99% (50 ml). The mixture was refluxed with stirring for 16 h. then the volume of the resultant was reduced to ~20 ml .on cooling the content for 7 days at 0℃. Yellow crystals were separated out and washed with ethanol (2 × 5 ml) followed by diethyl ether (2 × 5 ml). The solid was recrystallized by hot ethanol.

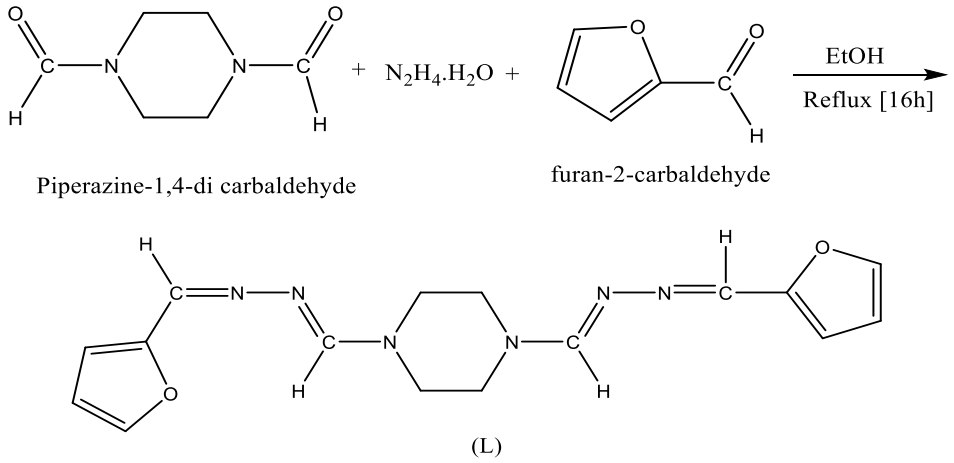

Figure 1. chiff reaction

2.2.2. Synthesis of metal complexes:

The hot methanolic solution (30mL) of corresponding metal salts (L:Metal) = (1:2) for Ni(II), was mixed with hot methanolic solution of the respective ligands and refluxed by using a water bath. . After 20 h of stirring the volume of the solution was reduced to ~20 ml and filtered. The solid resultant was obtained and washed with methanol (3 × 3 ml), followed by diethyl ether (2 × 5 ml).

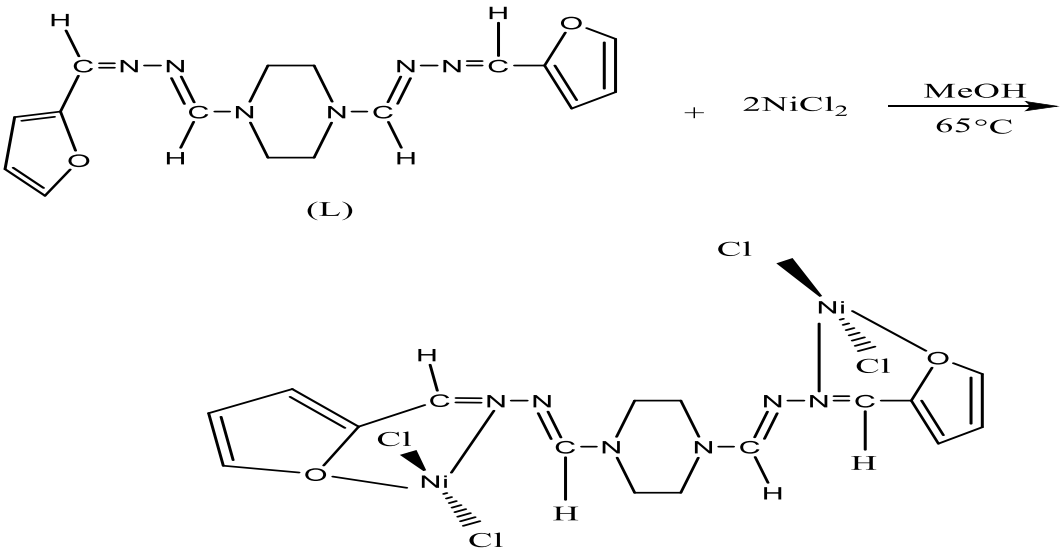

Figure 2. complex

3.Results and Discussion:

3.1. 1H-spectroscopic measurements:

Figure 3 . Explanation of ^{1}H-NMR (ppm) of the Di (Furanyle Methylene Hydrazono) Piperazine (DFMHP)

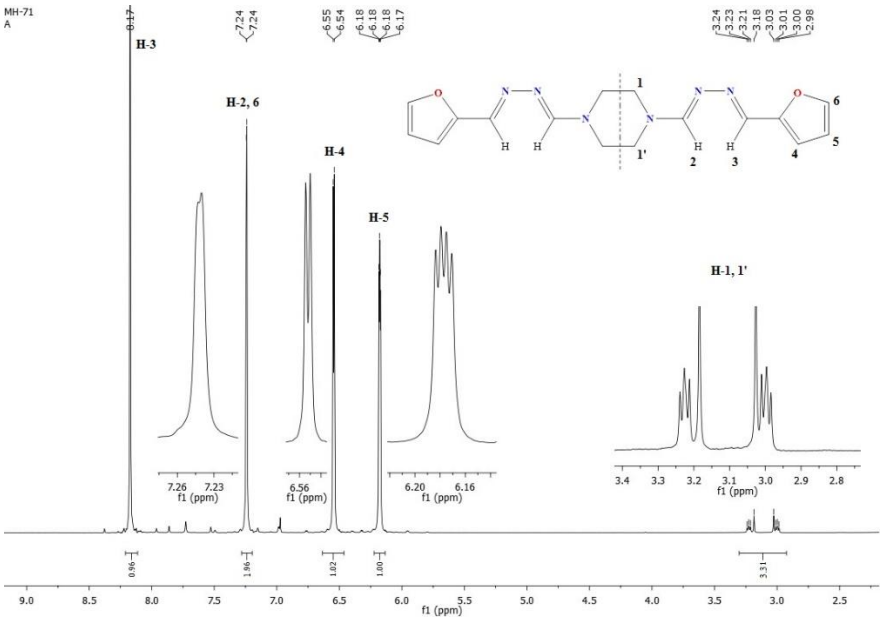

Table 1.

Explanation of ^{1}H-NMR (ppm) of the Di(Furanyle Methylene Hydrazono) Piperazine (DFMHP)

Chemical Shift δ ppm	Proton Number	No,H
2.98-3.24	2H ,dd	**1,1ó**
7.24	1H,d	**2,6**
8.17	1H,s	**3**
6.54- 6.55	1H,d	**4**
6.17-6.18	1H,dd	**5**

Figure 4. Explanation of ^{13}C -NMR (ppm) of the Di (Furanyle Methylene Hydrazono) Piperazine (DFMHP)

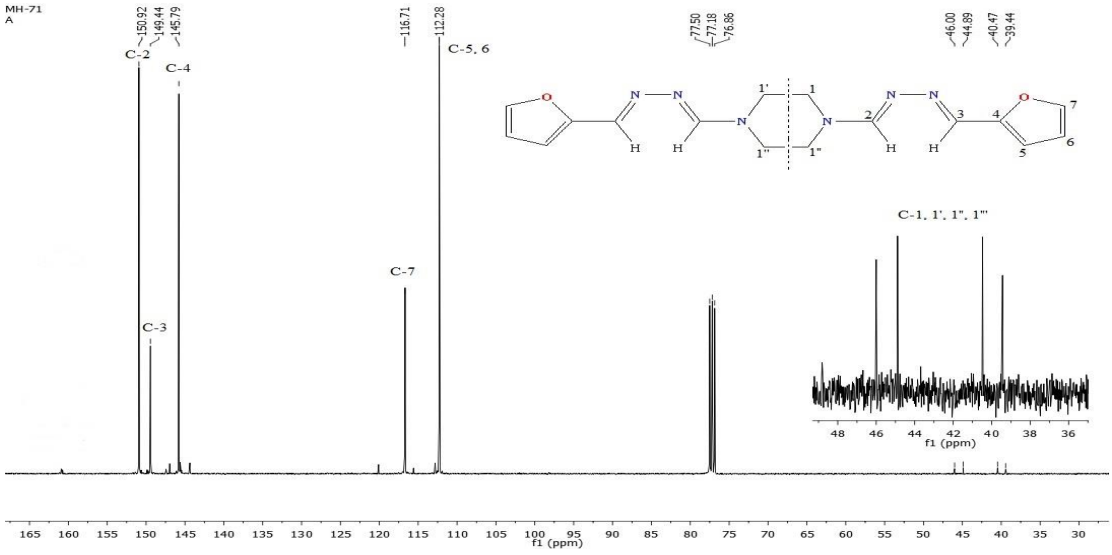

Table 2.

Explanation of ^{13}C-NMR (ppm) of the Di (Furanyle Methylene Hydrazono) Piperazine (DFMHP)

Chemical Shift δ ppm	No
39.434-40.47-44.89-46.00	**1,1ó ,1ő ,1ő**
150.92	**2**
149.44	**3**
145.97	**4**
112.28	**5,6**
116.71	**7**

3.3. Infrared Spectra:

The infrared spectra for the present compounds taken in the range 400-4000 cm^{-1} help to indicate regions of absorption vibrations. The main stretching modes are for ν(C=N) and ν(C-O).

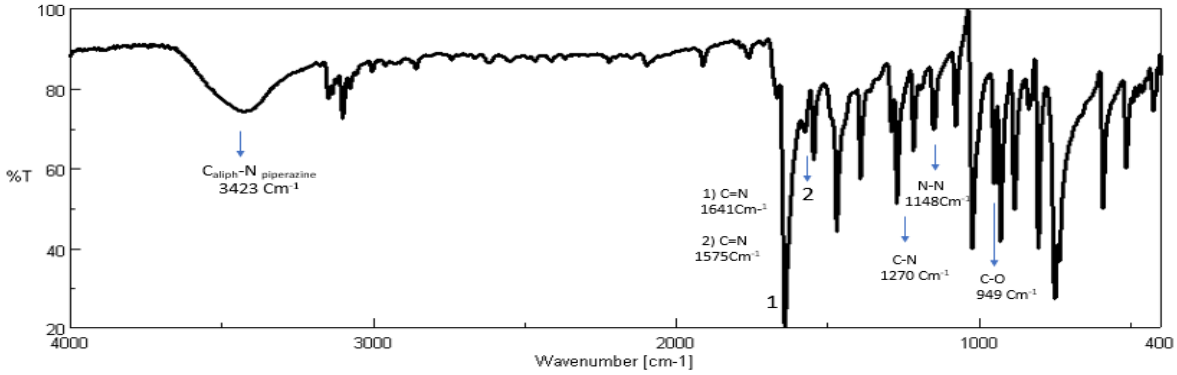

Figure 5. FT-IR absorption spectra of ligand (L)

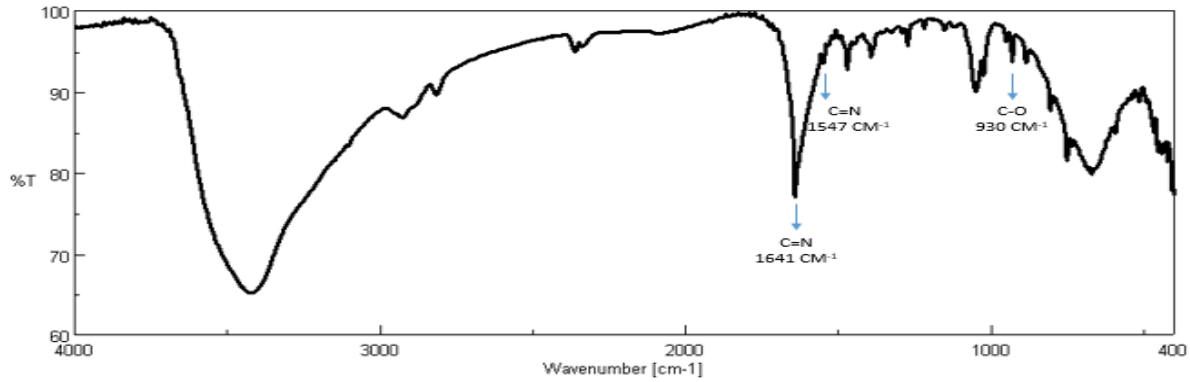

Figure 6. FT-IR absorption spectra of $[Ni_2LCl_4]$

Table 3.

Conductivity and melting point of the ligand and complex:

Compounds	M.P (°C)	Color	Yield (%)	Conductivity µS	ῡ(C=N) cm^{-1}	ῡ(C-O) cm^{-1}
L	110°C	Yellow	40.202	**0**	1575	949
$[Ni_2LCl_4]$	>250 °C	Green	52.02	70	1547	930

4. Conclusion:

The synthesise of a new ligand (L) di(Furanyle Methylene Hydrazone) Piperazine (DFMHP) was carried by condensation of 1,4 di formyle piperazine with hydrazine mono hydrate ,Then add furfural to getting ligand (L). Then the reaction of this ligand with Nickle(ll) ion were carried out using metal Chloride salt by the (1:2) molar ration respectively conduced . $[Ni_2LCl_4]$

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