

**Synthesis and Characterization of Mixed Ligands of Dithizone
and Schiff Base Complexes with Selected Metal Ions.**

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Abstract

Salicylaldehyde was react with 4-amino-2,3-dimethyl-1-phenyl-3-pyrazoline-5-on to produce the Schiff base ligand 2,3-dimethyl-1-phenyl-4-salicylidene-3-pyrazoline-5-on (L). The prepared ligand was identified by Microelemental Analysis, and FT.IR, UV-Vis spectroscopic techniques. A new complexes of Fe(III),Co(II),Ni(II),Cu(II),Ce(III) and Pb(II) with mixed ligands of dithizone (DTZ) and Schiff base were prepared in aqueous ethanol with a 2:2:1 M:L:DTZ. The prepared complexes were characterized using flame atomic absorption, (C.H.N) Analysis, FT.IR and UV-Vis spectroscopic methods as well as magnetic susceptibility and conductivity measurements. In addition biological activity of the ligands and complexes against two selected type of bacteria were also examined. Some of the complexes exhibit good bacterial activities. From the obtained data the octahedral structure was suggested for all prepared complexes.

Keyword: - Schiff base, complexes, antipyrene Schiff base.

Introduction

Schiff bases are a class of important compounds in medical and pharmaceutical field. They show biological activities including antibacterial⁽¹⁻⁴⁾, antifungal, anticancer⁽⁵⁻⁷⁾ and herbicidal activities⁽⁸⁾. Furthermore, Schiff bases are utilized as starting materials in the synthesis of industrial⁽⁹⁻¹¹⁾ and biological compounds^(12,13). Metal complexes of Schiff bases are extensively studied due to synthetic flexibility, selectively toward a variety of metal atoms⁽¹⁴⁾. They are found useful in catalysis in medicine as antibiotics and anti-inflammatory agents and in the industry as anticorrosion⁽¹⁵⁻¹⁹⁾. Literature is abundant on physico-chemical proportion of various symmetrical Schiff bases and chelates Schiff bases with their pyridine, 2,2'-bipyridine and 1,10-phenanthroline adducts⁽²⁰⁻²²⁾. The present paper reports the synthesis and characterization of new Fe(III),Co(II),Ni(II),Cu(II),Ce(III) and Pb(II) complexes with mixed ligands of dithizone and Schiff base.

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Experimental

Instrumentation

UV-Vis spectra were recorded on a (Shimadzu UV-160 A) Ultra Violet-Visible Spectrophotometer. I.R-spectra were taken on a (Shimadzu, FTIR-8400 S) Fourier Transform Infrared. Spectrophotometer (4000-400) cm^{-1} with samples prepared as KBr discs. Atomic Absorption was obtained by using a (Shimadzu A.A-160A) Atomic Absorption / Flame Emission Spectrophotometer. Microelemental analysis (C.H.N) was performed in AL-al- Bayt University, Jordan by using (Euro Vector EA 3000 A Elemental Analyser). Conductivities were measured for 10^{-3}M of complexes in DMF at 25°C by using (Philips PW- Digital Conductimeter). Magnetic susceptibilities were performed by using (Brucker Magnet B.M.6) instrument at 25°C . Melting points were obtained by using (Melting Point Apparatus).

Materials

The following chemicals were used as received from suppliers; $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Pb}(\text{NO}_3)_2$ (Merck); Salicylaldehyde, KOH, 4-amino-2,3-dimethyl-1-phenyl-3-pyrazoline-5-on, DMF, dithizone and acetic acid (B.D.H). All chemicals were of analytical grade used as suppliers without further purification.

Study of Biological Activity

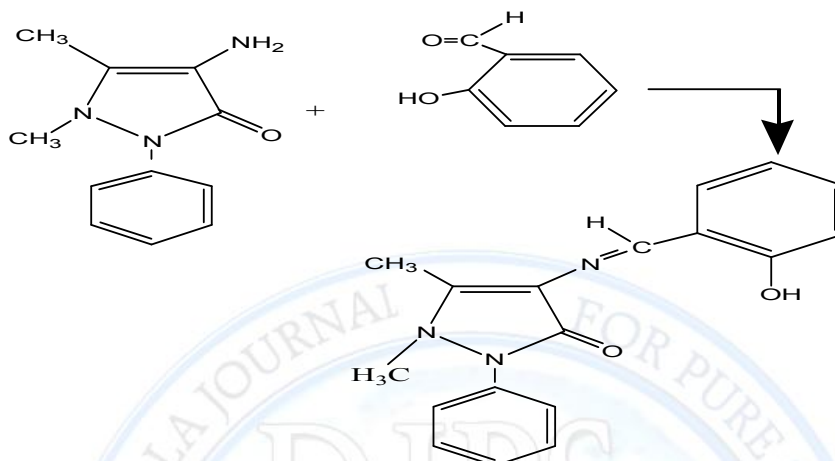
Two selected types of bacteria were used, including Escherichia Coli (E. Coli) as Gram Negative Bacteria and Staphylococcus Aureus (Staph. Aureus) as Gram Positive Bacteria in Nutrient Agar medium, using (DMF) as a solvent and a control, the concentration of the compounds in this exposure was (10^{-3}M) by using disc sensitivity test. This method involves the exposure of the zone inhibition toward the diffusion of micro-organism on agar plate. The plates were incubated for 24hr. at 37°C .

Synthesis of Schiff base (L)⁽²³⁾

(0.26ml, 1mmole) of salicylaldehyde was added to ethanolic solution of (0.5g, 1mmole) of 4-amino-2,3-dimethyl-1-phenyl-3-pyrazoline-5-on. The solution mixture was stirred and refluxed for 2 hours, yellow crystalline precipitate observed. The resulting precipitate was filtered off, recrystallized from menthol and dried at 50°C . The preparation method of the ligand (L) is represented in scheme-1.

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Schem-1) Preparation Method of the Ligand (L)(

Preparation of Metal Complexes (general procedure)

An aqueous solution of the metal salts containing 0.404g, 0.291g, 0.290g, 0.242g, 0.434g and 0.331g $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Pb}(\text{NO}_3)_2$ (2mmole) respectively was added gradually with stirring to ethanolic KOH solution (0.5g, 2mmol) of 2,3-dimethyl-1-phenyl-4-salicylidene-3-pyrazoline-5-on, an ethanolic solution of (0.20g, 1mmole) dithizone was added to the mixture in each case by using stichiometric amount (2:2:1) Metal:ligand:dithizone molar ratio. The mixture was refluxed with constant stirring for about 1 hour. The mixture was cooled at room temperature dark precipitate was formed, filtered and recrystillized from methanol.

Results and Discussion

The synthesized ligand (L) was characterized by FT.IR, UV-Vis, and (C.H.N) analysis. The solid complexes were prepared by reaction of alcoholic solution of the ligand with the aqueous solution of the metal ions and ethanolic solution of dithizone (DTZ) in a (M:L:DTZ) of (2:2:1). The (C.H.N) analysis with metal contents of these complexes were in good agreements with the calculated values (Table-1) includes the physical properties and elemental analysis. The molar conductance in DMF (10^{-3}M) indicated the electrolyte type⁽²⁴⁾

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ratio (1:2) and (1:4) in case of Fe(III) and Ce(III) complexes, the data were recorded in (Table-2).

The effective magnetic moments (Table-2) of the complexes lie in the range (1.93-5.72) B.M. This value refers to a paramagnetic (high spin) which has been reported for most octahedral geometry. In case of Pb(II) complex because of filled-d orbital, therefore the magnetic moment ($\mu=0$) is diamagnetic⁽²⁵⁾.

The UV-Vis spectra data for the free ligands and all metal complexes are listed in (Table-2). The UV-Vis spectrum of the ligand (L) (Fig-1) spectrum of the shows three peaks at 256 nm, 340 nm and 362 nm assigned to $(\pi - \pi^*)$ and $(n - \pi^*)$ electronic transitions⁽²⁶⁾. The electronic spectrum of dithizone (Fig-2) shows three peaks at 282 nm, 348 nm and 474 nm attributed to $(\pi - \pi^*)$ and $(n - \pi^*)$ electronic transitions respectively⁽²⁷⁾. The electronic spectrum of Fe(III) complex gave absorption peak at 251 nm related to charge transfer (C.T). Other peak at 380 nm was assigned to ${}^6A_{1g} \rightarrow {}^4T_{2g(D)}$ ⁽²⁸⁾. The spectrum of Co(II) complex showed peak at 288 nm due to charge transfer. Other three peaks at 346 nm, 494 nm and 570 nm were found to be caused by (d-d) electronic transition type ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$, ${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$ and ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$ respectively⁽²⁹⁾. The spectrum of Ni(II) complex appeared absorption peak at 223 nm was related to charge transfer, then other three peaks at 468 nm, 620 nm and 864 nm were assigned to electronic transition type ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(P)}$, ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(F)}$ and ${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)}$ respectively⁽³⁰⁾. The spectrum of Cu(II) complex gave absorption peak at 320 nm due to charge transfer. The peak at 440 nm was caused by electronic transition⁽³¹⁾ ${}^2E_g \rightarrow {}^2T_{2g}$. The spectrum of Ce(III) complex showed two absorption peaks at 320 nm and 480 nm were assigned to (f-f) transition between the metal and ligands⁽³²⁾. The spectrum of Pb(II) complex (Fig-3) showed absorption peak at 311 nm due to charge transfer. The absence of absorption peaks in the visible region indicated no (d-d) electronic transition happened, this is a good result for octahedral complex.⁽³³⁾

In order to study the binding mode of the ligand with the metal ions, a comparison was made for the FT-IR spectra of the free ligands and those of the prepared complexes and the data was tabulated in (Table-3). The IR spectrum of the ligand (L) (Fig-4) exhibited broad band at 3290 cm^{-1} was assigned to $\nu(\text{OH})$ stretching frequency, absent this band in the spectra of all complexes, which indicated deprotonation and involvement of the enol O in chelation⁽³⁴⁾. The bands at 1655 cm^{-1} and 1593 cm^{-1} in the ligand spectrum ascribed to $\nu(\text{C}=\text{O})$ and $\nu(\text{C}=\text{N})$ stretching vibrations respectively,^(35,36) suffered a great change to lower frequency was also observed on complexation (Fig-5) with metal ion. The presence of coordination water in the spectra of all complexes⁽³⁷⁾ were suggested by the very broad absorption around $(3414-3430) \text{ cm}^{-1}$. The new bands observed at $(560-340) \text{ cm}^{-1}$ are tentatively assigned to $\nu(\text{M}-\text{O})$, $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{S})$ (Metal-Ligands) stretching bands⁽³⁸⁻⁴⁰⁾.

The IR spectrum of dithizone (Fig-6) appeared band at 3379 cm^{-1} due to the stretching mode of $\nu(\text{NH})$ ⁽⁴¹⁾, on complexation a shifting with change in shape were observed from this band, while increasing in intensity were noticed. The significant may be a result of

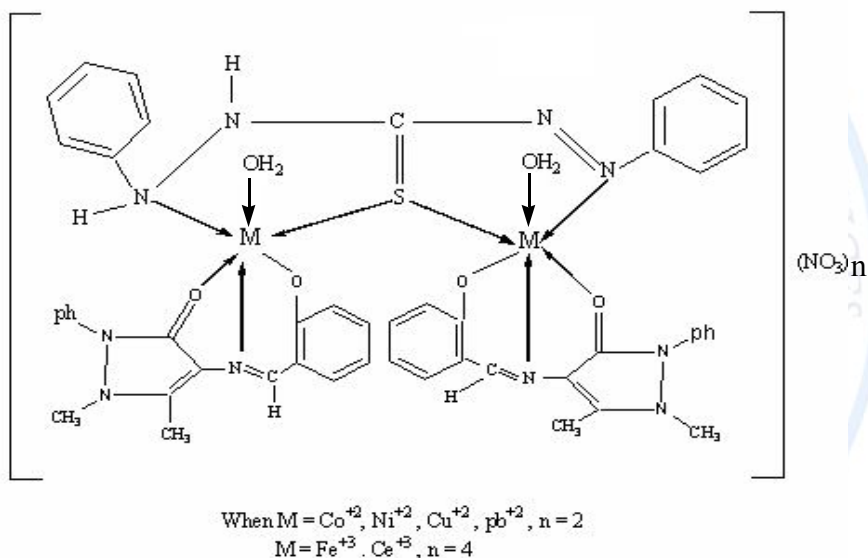
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coordination with metal ion. The band at 1438 cm^{-1} due to $\nu(\text{N}=\text{N})$ stretching vibration⁽⁴²⁾, suffered a great change to higher frequency was also observed on complexation with metal ion. The spectrum of dithizone characteristic band at 891 cm^{-1} which was assigned to $\nu(\text{C}=\text{S})$ stretching⁽⁴³⁾, this band was shifted to lower frequency in the spectra of the complexes.

Finally, the biological activities of the ligands and their complexes have also tested against selected type of bacteria. The results show that the Co(II) and Cu(II) complexes as relatively strong activating capacity. Whereas, all the parents ligands possessed no activity towards the same specimen of bacteria. The data was recorded in (Table-4).

According to the results obtained and spectral analysis an octahedral structure has been suggested to these complexes.



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References

- 1- Karia, F.D and Parsania, P.H., **Asian.J.Chem.**, 11(1999)991.
- 2- More, P.C., Bhalvankar, R.B and Pattar, S.C., **Indian.Chem.Soc.**, 78(2001)474.
- 3- Baseer, M.A., Jadhav, V.D., Phule, R.M., Archana, Y. and Vibhute, Y.B., **Orient.J.Chem.**, 16(2000)553.
- 4- Pandeya, S.N., Sriram, D., Nath, G and Declereq, E., **Ifarmaco.**, 54(1999)624.
- 5- Desai, S.B., Desai, P.B and Desai, K.R., **Hetrocycl.Comm.**, 7(2001)83
- 6- Samadhiya, S and Halve, A., **Orient.J.Chem.**, 17(2001)119.
- 7- Aydogan, F., Ocal, N., Turgut, Z and Yolacan, C., **Bull.Korean.Chem.Soc.**, 22(2001)476.
- 8- Pathak, P., Jolly, V.S and Sharma, K.P., **Orient.J.Chem.**, 16(2000)161.
- 9- Taggi, A.E., Hafez, A.M., Wack, H., Young, B., Ferraris, D and Lectka, T., **J.Am.Chem.Soc.**, 124(2002)6626.
- 10- Hansongnern, K., Tempiam, S., Liou, J.C., Laio, F.L and Lu, T.H., **Anal.Sci.**, 19(2003)13.
- 11- Croot, P.L and Johansson, M., **Electroanalysis.**, 12(2000)565.
- 12- Choi, D., Lee, S.K., Chung, T.D and Kim, H., **Electroanalysis.**, 12(2000)477.
- 13- Halve, A and Goyal, A., **Orient.J.Chem.**, 12(2001)87.
- 14- Spinu, C and Kriza, A., **Acta.Chim.Slov.**, 47(2000)179.
- 15- Sun, B., Chen, J., Hu, J and Li, X., **J.Chin.Chem.Lett.**, 12(2001)1043.
- 16- Boghae, D.M and Mohebi, S., **Terahedron.**, 58(2002)5357.
- 17- Liu, J., Wu, B., Zhang, B and Liu, Y., **Turk.J.Chem.**, 30(2006)41.
- 18- Budakoti, A., Abid, M and Azam, A., **Eur.J.Med.Chem.**, 41(2006)63.
- 19- Jin, V.X., Tan, S.I., and Ranford, J.D., **Inorg.Chim.Acta.**, 358(2005)677.
- 20- Chohan, Z.H., **Synth.React.Inorg.Met.Org.Chem.**, 3(2001)1.
- 21- Thangadurai, T.D and Natarajan, K., **Trans.Met.Chem.**, 26(2001)500.
- 22- Oswole, A.A., Woods, J.A.O and Odunola, O.A., **Synth.React.Inorg.Met.Org.Chem.**, 33(2003)167.
- 23- AL- Hamdani, A.A.S., **J.Um-Salama. Sci.**, 2(2005)2.
- 24- Geary, W.J., **Coord.Chem.Rev.**, 7(1971)110.
- 25- Khuble, R.C and Singh, R.P., **Indian.J.Chem.**, 22A(1982)214.
- 26- Sonme, M and Sekerci, M., **Polish.J.Chem.**, 76(2002)907-914.
- 27- Suzuki, T., Shiotsuki, M., Wada, K., Kondo, T and Mitsudo, T., **Organo.Metal.Lics.**, 18(1999)18.
- 28- Daniel, E., Seema, C., Melvyn, R.C., Kim, M.K and Janet, R.M., **Inorg.Chem.**, 40(2001)1591-1596.
- 29- Sandhu, S.S., Jaswal, J.S and Sandhu, G.K., **Indian.J.Chem.**, 35(1996)218-222.
- 30- Cezar, S and Angela, K., **Acta.Chim.Slov.**, 47(2000)179-185.
- 31- Chakravarty, A.R., Nreddy, A.P., Santra, B.K and Thomas, A.M., **J.Indian.Chem.Sci.**, 114(2002)391-401.
- 32- Lever, A.B.P., "Inorganic Spectroscopy", Elsevier Publishing Company, London, New York (1968).
- 33- Daniel, E., Seema, C., Melryn, R and Kim, M.K., **Inorg.Chem.**, 40(2001)7.
- 34- Yildiz, E and Boztepe, H., **Turk.J.Chem.**, 26(2002)897-903.

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- 35- Osowole, A. A., Kolawole, G. A and Fagade, O. E., **Synth.React.Inorg.Met.Org.Chem and Nano-Met Chem.**,35(2005)829.
- 36- Osowole, A. A., Kolawole, G. A and Fagade, O. E., **Int.J.Chem.**,15(2005)237.
- 37- Sengupta, S. K., Pandey, O. P., Srivastava, B. K and Sharma, V. K., **Trans.Met.Chem.**,23(1998)349.
- 38- El-Saied, F. A., El-Bahnasawy, R. M., Abdel-Azeem, A. K and El-Sawaf, A. K., **Polyhedron**,13(1994)1781.
- 39- Sharma, V. K and Sengupta, S. K., **Metal-Organic and Nano-Metal Chemistry**,23(1993)401.
- 40- Thangadurai, T. D and Natarajan, K., **Indian.J.Chem.**,41(2002)741.
- 41- Maradiya, H. R., **Turk.J.Chem.**,25(2001)441-450.
- 42- Ray, U., Banerjee, D., Mostafa, G., Lu, T. H and Sinha, C., **New.J.Chem.**,28(2004)1437-1442.
- 43- Abou-Melha, K. S and Faruk, H., **J.Iran.Chem.Soc.**,5(2008)122-134.

Table(1):- Physical Properties and Elemental Analysis of the Ligand and It's Complexes.

| Compounds | Color | M.P °C | Yield % | Analysis Calc (Found) | | | |
|---|------------|--------|---------|-----------------------|------------------|----------------|------------------|
| | | | | M% | C% | H% | N% |
| Ligand(L) | Yellow | 198 | 86 | - | 70.35 (70.02) | 5.53 (4.97) | 13.68 (12.57) |
| Dithizone(DTZ) | Black | - | - | - | - | - | - |
| [Fe ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | Deep Brown | 211 | 73 | 10.28 (9.91) | 46.51 (46.06) | 3.48 (3.12) | 11.07 (10.51) |
| [Co ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | Brown | 139 | 68 | 10.29 (9.88) | 51.30 (51.11) | 3.83 (2.91) | 12.21 (11.68) |
| [Ni ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | Purple | 239 | 85 | 10.21 (9.92) | 51.35 (50.94) | 3.84 (3.23) | 12.22 (11.53) |

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| | | | | | | | |
|--|-------------|-----|----|------------------|------------------|----------------|------------------|
| $[\text{Cu}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_2$ | Deep Purple | 137 | 71 | 11.07 (10.62) | 50.86 (49.97) | 3.80 (3.07) | 12.11 (11.81) |
| $[\text{Ce}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_4$ | Black | 171 | 63 | 19.55 (18.51) | 41.06 (40.83) | 3.07 (2.97) | 9.77 (8.47) |
| $[\text{Pb}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_2$ | Black | 201 | 77 | 28.71 (27.97) | 40.77 (39.68) | 3.05 (2.88) | 9.70 (8.17) |

Table(2):- UV-Vis, Magnetic Susceptibility and Conductance Measurements Data.

| Compounds | λ_{max} (nm) | ABS | Wave number (cm^{-1}) | ϵ_{max} ($\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) | Λ_{m} ($\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$) in DMF(10^{-3}M) | μ_{eff} (B.M) |
|--|--------------------------------|-------|--|--|---|-----------------------------|
| Ligand(L) | 256 | 1.365 | 39062 | 1365 | - | - |
| | 340 | 2.265 | 29411 | 2265 | | |
| | 362 | 1.354 | 27624 | 1354 | | |
| Dithizone(DTZ) | 282 | 0.128 | 35460 | 128 | - | - |
| | 348 | 1.628 | 28735 | 1628 | | |
| | 474 | 1.176 | 21097 | 1176 | | |
| $[\text{Fe}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_4$ | 251 | 0.523 | 39840 | 523 | 263 | 5.72 |
| | 380 | 0.835 | 26315 | 835 | | |
| $[\text{Co}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_2$ | 288 | 1.523 | 34722 | 1523 | 124 | 4.75 |
| | 346 | 1.926 | 28901 | 1926 | | |
| | 494 | 0.681 | 20242 | 681 | | |
| | 570 | 0.466 | 17543 | 466 | | |
| $[\text{Ni}_2(\text{L})_2\text{DTZ}(\text{H}_2\text{O})_2](\text{NO}_3)_2$ | 223 | 0.555 | 44843 | 555 | 157 | 3.08 |
| | 468 | 1.616 | 21367 | 1616 | | |

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| | | | | | | |
|---|-----|-------|-------|------|-----|------|
| | 620 | 0.273 | 16129 | 273 | | |
| | 864 | 0.185 | 11574 | 185 | | |
| [Cu ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | 320 | 1.816 | 31250 | 1816 | 162 | 1.93 |
| | 440 | 0.881 | 22727 | 881 | | |
| [Ce ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | 320 | 1.831 | 31250 | 1831 | 275 | 2.47 |
| | 480 | 1.500 | 20833 | 1500 | | |
| [Pb ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | 311 | 1.827 | 32154 | 1827 | 138 | Dia |

Table(3):- The Main Frequencies of the Ligands and It's Complexes(cm⁻¹).

| Compounds | v(OH) | v(NH) | v(C=O) | v(N=N) | v(M-O) | v(M-S) |
|---|--------------------------|----------|-----------------------|-----------------------|------------------|--------|
| | + v(H ₂ O) | | + v(C=N) | + v(C=S) | + v(M-N) | |
| Ligand(L) | 3290 br. | - | 1655 sh. 1593 s. | - | - | - |
| Dithizone(DTZ) | - | 3379 m. | - | 1438 sh. 891 s. | - | - |
| [Fe ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | - 3430 br. | 3215 br. | 1637 sho. 1566 m. | 1489 sh. 875 s. | 549 w. 442 w. | 372 w. |
| [Co ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - 3417 br. | 3240 br. | 1604 m. 1527 m. | 1488 sho. 833 sho. | 509 w. 416 w. | 340 w. |
| [Ni ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - 3420 br. | 3280 sh. | 1610 sh. 1525 v.s. | 1494 sho. 875 sho. | 554 w. 460 w. | 340 w. |
| [Cu ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - | 3371 br. | 1585 sh. | 1465 s. | 551 w. | 395 w. |

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| | | | | | | |
|---|----------|----------|-----------|----------|--------|--------|
| | 3414 br. | | 1523 sho. | 879 m. | 455 w. | |
| [Ce ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | - | 3244 sh. | 1604 s. | 1489 s. | 560 w. | 374 w. |
| | 3425 br. | | 1563 sho. | 852 w. | 440 w. | |
| [Pb ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - | 3217 s. | 1593 s. | 1492 sh. | 547 w. | 385 w. |
| | 3425 br. | | 1523 sh. | 883 m. | 482 w. | |

br =broad,sh=sharp,sho=shoulder,v.s= very strong, s= strong, br= broad, w=weak.

Table(4):- Diameters (mm) of Deactivation of Bacteria for the Ligands and its Complexes

| Compounds | Staphylococcus | Escherichia |
|---|----------------|-------------|
| | Aurous | Coli |
| Ligand(L) | - | - |
| Dithizone(DTZ) | - | - |
| [Fe ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | + | + |
| [Co ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | + | +++ |
| [Ni ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - | + |
| [Cu ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | +++ | +++ |
| [Ce ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₄ | + | + |
| [Pb ₂ (L) ₂ DTZ(H ₂ O) ₂](NO ₃) ₂ | - | + |

(-) = No inhibition.

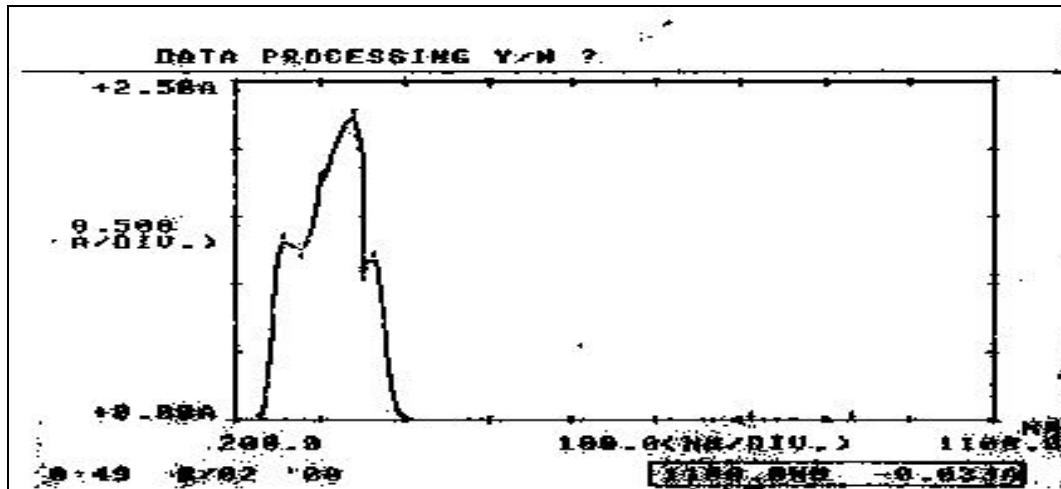
(+) =Inhibition diameter (6-8) mm.

(++) =Inhibition diameter (8-10) mm.

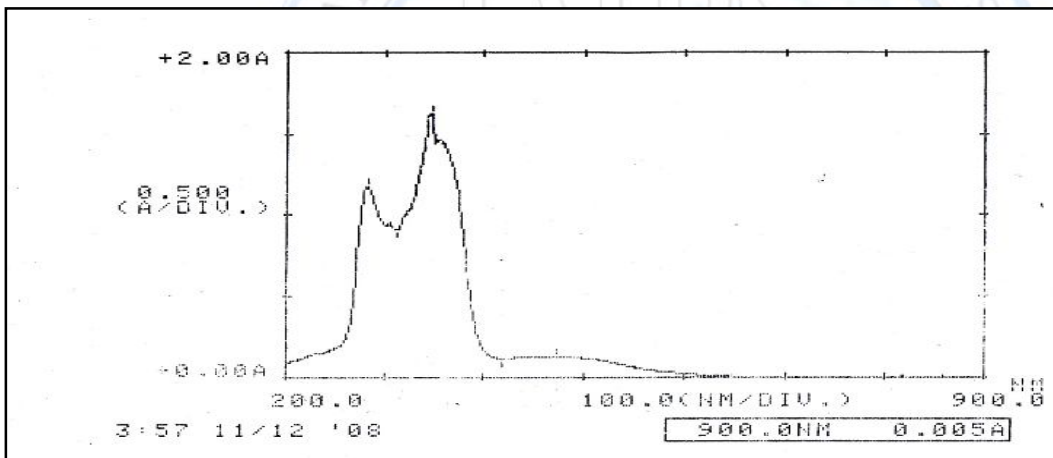
(+++)=Inhibition diameter (10-20) mm.

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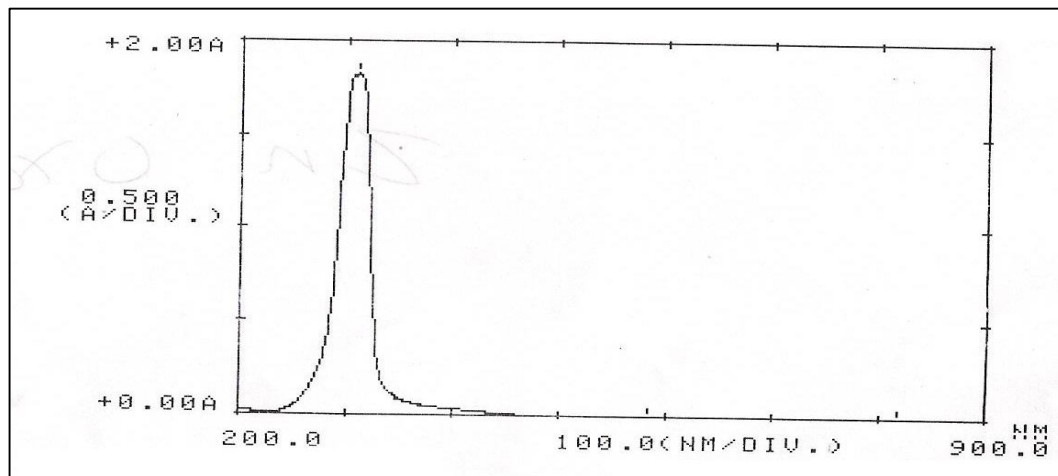
Fig(1):-UV-Vis Spectrum of Schiff base (L).



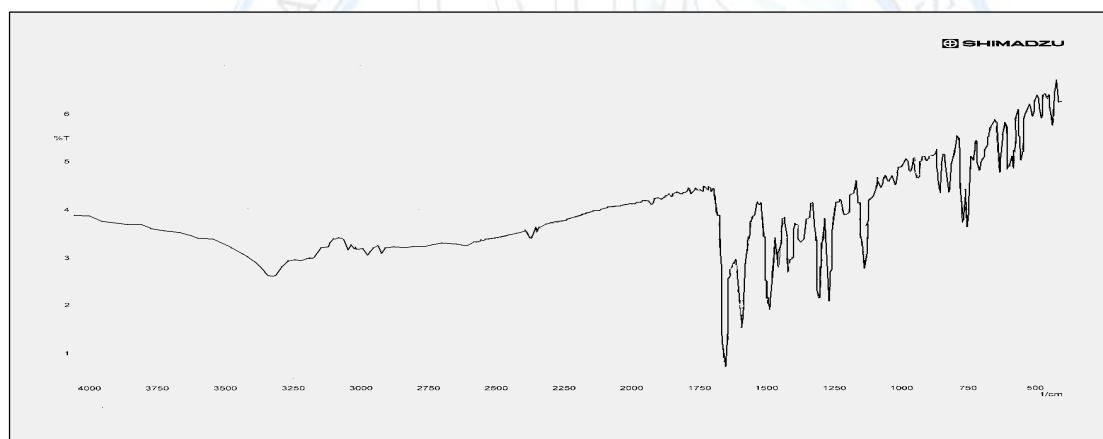
Fig(2):- UV-Vis Spectrum of Dithizone.

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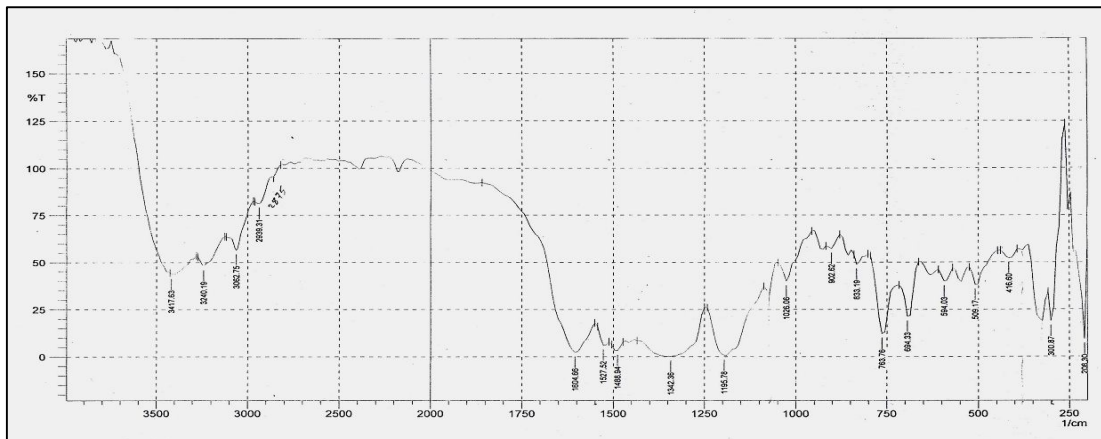
Fig(3):- UV-Vis Spectrum of $[Pb_2(L)_2DTZ(H_2O)_2](NO_3)_2$ Complex.



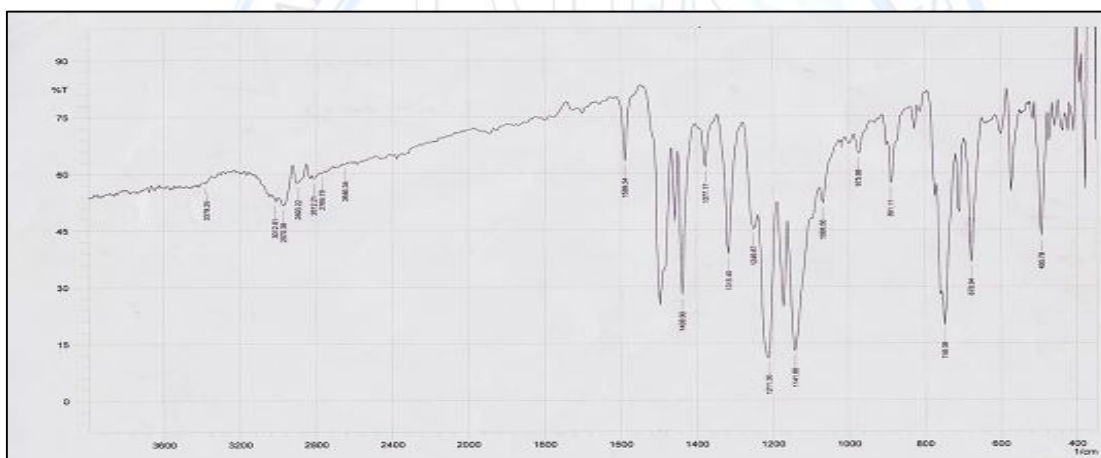
Fig(4):-FTIR Spectrum of Schiff base (L).

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Fig(5):-FTIR Spectrum of $[Co_2(L)_2DTZ(H_2O)_2](NO_3)_2$ Complex.



Fig(6):-FTIR Spectrum of Dithizone.

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تحضير وتشخيص معقدات مختلطة الليكاند للدايثايزون وقاعدة شف مع ايونات بعض
العناصر الفلزية المنتخبة.

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الخلاصة

حضر الليكاند (L) ٣،٢-ثنائي مثيل-١-فنيل-٤-سالسيدين-٣-بايروزولين-٥-اون من تفاعل السلسلديهايد مع ٤-امينو-٣،٢-ثنائي مثيل-١-فنيل-٣-بايروزولين-٥-اون. شخخص الليكاند المحضر بواسطة التحليل الدقيق للعناصر (C.H.N) واطيف الأشعة تحت الحمراء وفوق البنفسجية-المرئية، تم الحصول على معقدات جديدة وذلك من خلال مفاعلة ابونات واطيف الأشعة تحت الحمراء وفوق البنفسجية-المرئية، تم الحصول على معقدات المختلطة للدايثايزون وقاعدة شف وبنسبة مولية (2:2:1) فلز:ليكاند:دايثايزون. شخخصت المعقدات المحضرة بواسطة التحليل الدقيق للعناصر (C.H.N)؛ تقنية الإمتصاص الذري اللهي واطيف الأشعة تحت الحمراء وفوق البنفسجية-المرئية، فضلا عن قياسات الحساسية المغناطيسية والتوصيلية الكهربائية. لقد تمت الإفادة من الليكاندات والمعقدات قيد الدراسة حول إمكانية دراسة الفاعلية البايولوجية ووجد أن لهذه المعقدات قابلية متباينة على قتل الأنواع المنتخبة من البكتريا. ومن النتائج المحصول عليها تم اقتراح الشكل ثماني السطوح للمعقدات المحضرة.

كلمات مفتاحية:- قواعد شف ، قواعد شف لللانتيبيرين قواعد شف ، معقدات