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# "Synthesis & Characterization of Complexes of Some New N'-(1-(4-hydroxy-2-oxo-2H-chromen-3-yl) ethylidene-4-methoxybenzohydrazide with Zn (II) &Cd (II) Metals

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**Abstract:** ***We report synthesis of complexes of some new N'-(1-(4-hydroxy-2-oxo-2H-chromen-3-yl) ethylidene4methoxybenzohydrazide With Zn (II) & Cd (II) Metals. Schiff's base ligand N'-(1-(4-hydroxy-2-oxo-2H-chromen-3-yl) ethylidene-4-methoxybenzohydrazide synthesized by condensing3 acetyl-4-hydroxy-2H-chromen-2-one with 4 methoxybenzohydrazide. The complexes were synthesized and characterized by elemental analysis, IR, Electronic spectra, molar conductance, TGA and powder XRD.***

**Keywords:** 3 acetyl-4-hydroxy-2H-chromen-2-one, 4 methoxybenzohydrazide, Schiff's base Zn (II) and Cd (II) metal complexes

## 1. Introduction

Schiff's bases are important in the progress of chemistry of coordination compound. In recent years, metal complexes prepared from Schiff's bases have been examined to a great extent as they possess their wide range of applications in numerous scientific areas due to physical and chemical properties. Schiff's base complexes are concentrated to larger extent in study of their catalytic activity in homogeneous and heterogeneous reactions. Schiff bases are very important due to their modified structures, a wide spectrum of biological and industrial application [1-2]. They possess pharmacological activities such as antimicrobial agents [3-7], antidepressant agents [8], antiviral agents [9], anticancer agents [10], fungicidal agents [11], bactericidal agents [12], cytotoxic agents [13], herbicidal agents [14], insecticidal agents [15], antioxidants agents [16], and anti-proliferative agents [17]. ] 2H-chromen-2-one is also named as 2H-1-Benzopyran-2-one or Coumarin. Coumarins also possess medicinal properties such as anti-bacterial [18], anti-fungal [19], anti-filarial [20] anti-HIV activities [21], anti-coagulants [22], antioxidant [23], antiulcerogenic [24], anti-inflammatory properties [25], selective coronary vasodilators [26] and possess antitumor properties [27]. They are known for stimulating changes in cell growth and also intercellular communication mechanisms [28].

## 2. Material and Methods

The solution conductivities of the metal complexes in DMSO were measured on digital conductivity bridge at room temperature. The conductivity cell with platinized platinum electrode with a cell constant 1.001 $cm^{-1}$ was used. The concentration of the solution was around 10-4M. The molar conductance values were interpreted with the help of data given in the literature [29, 30]. Gouy's method was used for the measurement of magnetic susceptibility of the compounds at room temperature. Electronic spectra in this solvent were recorded on SHIMADZU-UV-1601 UV/visible double beam spectrophotometer in the region 200-800 nm using quartz tubes of 1 cm path length. Infrared spectra of the ligands and metal complexes were taken as KBr pellets on Shimadzu spectrometer and some Infrared spectra were recorded as KBr pellets on FTIR-4100 spectrophotometer.

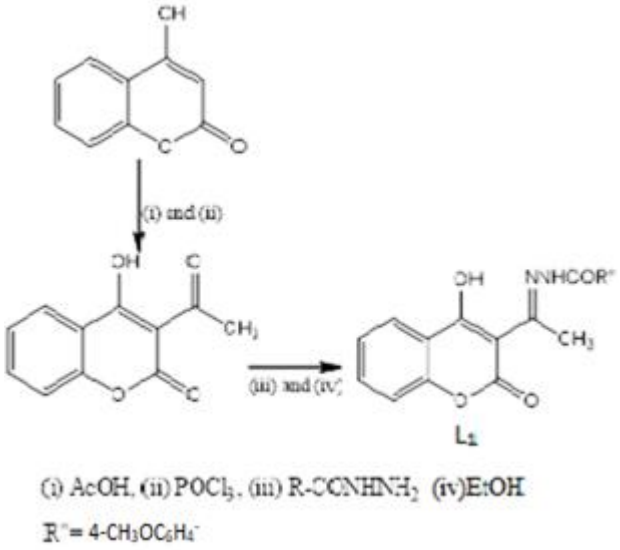
**Analytical Methods:**
Chemicals of good quality (AR) grade were used throughout the experimental work. Solvents: Methyl alcohol and Ethyl alcohol and chloroform were used for synthesis of Schiff base and their transition metal complexes. The solvents were purified by distilling over dry calcium oxide. All other solvents the synthesized Schiff base was soluble in methanol and their complexes with Zn (II) & Cd (II) were soluble in DMSO. Metal chlorides were used as received from S. D. fine chemicals.

## 3. Experimental

**General procedure for synthesis of Schiff'sbases:**
Aryl hydrazides are synthesized by treating aryl ester with hydrazine hydrate as per reported procedure [31].3-acetyl-4-hydroxy-2H-chromen-2-one is prepared as per previous reported method [32]. The imines $L_1$ were prepared by adding 3-acetyl-4-hydroxy-chromen-2-one (0.01mole) and 4 methoxybenzohydrazide (0.01 moles each) in ethanol (50 ml) and refluxing the mixture for 4 hrs. After cooling, the product was crystallized from ethanol. The purity of the ligands was checked by M. P. and TLC.

**Procedure for synthesis of Metal complexes:**
0.02 moles (for Zinc and Cadmium complexes) of ligand $L_1$ was taken in round bottomed flask containing 30ml of methanol and refluxed for few minutes.0.01 moles of metal salt dissolved in 20ml of methanol was added drop wise in hot solution of ligand. The contents were refluxed for two

hours. Solution was cooled and precipitation was not found. Ten percent alcoholic ammonia solution was added drop wise with stirring till precipitation was observed. The pH of precipitation for each complex was noted.




**Result and Discussion**

Characterization of synthesized ligand $L_1$: N'-(1-(4-hydroxy-2-oxo-2H-chromen-3-yl) ethylidene)-4-methoxybenzohydrazide ($L_1$):

Color: yellow; Yield: 82%; M. P.: 186°C; IR (KBr, $cm^{-1}$): 3500-2650 (3460, 3270) (broad Phenolic $V_{OH}$& $V_{NH}$), 1700 ($V_{C=0}$) of lactone, 1670 ($V_{C=0}$) of aryl hydroxide, 1620 ($V_{C=N}$) of mine.1558 & 1498 aromatic ($V_{C=C}$), 1350 ($V_{C=0}$) Phenolic OH.).

C, H, N % for $C_{19}H_{16}N_2O_4$. Analytical: C 67.78, H 4.72, and N 8.52; Calculated: C 67.85, H 4.79, and N 8.33.

## 4. Result and Discussion

On the basis of elemental analysis, metal ligand ratio and thermo gravimetric analysis molecular formulae of the complexes are assigned in table no.1. Complexes possess different colors than ligand, insoluble in ethanol, chloroform and acetone where as they are sparingly soluble in DMSO /DMF. They decompose at relatively higher temperature (>270°C) indicating good thermal stability at normal conditions.

**Table 1:** Physical and analytical data

| Compound | Molecular formula Formula Wt PH range of precipitation Color M. P. °C M: L ratio 7.5-8 Green 234 1: 2 | Formula Wt PH range of precipitation Color M. P. °C M: L ratio | PH range of precipitation Color M. P. °C M: L ratio | Color | M. P. °C | Sol$^n$ Cond. $\mu_v$ | M: L ratio |
|---|---|---|---|---|---|---|---|
| [Zn ($L_1$) Cl] [Cu (C12H10N3O4) 2] 584.00 7.5-8 Dark Green 192 1: 2 | [Zn ($C_{19}H_{14}N_2O_5$) Cl] | 452.20 | 7.0-7.5 | Yellowish white | >300 | 22.18 | 1: 1 |
| [Cd ($L_1$) Cl] | [Cd ($C_{19}H_{14}N_2O_5$) Cl] | 499.20 | 7.5-8 | White | >300 | 24.16 | 1: 1 |

| Compound | M. F. | Elemental analysis % found (calculated) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | C | H | N | S | Cl | M | |
| | | | | | | | Zn | Cd |
| [Zn (L1) Cl] | [Zn ($C_{19}H_{14}N_2O_5$) Cl] | 50.42 (50.47) | 3. 10 (3.34) | 6.19 (6.20) | - - | 7.83 (7.84) | 14.41 (14.46) | |
| [Cd (L1) Cl] | [Cd ($C_{19}H_{14}N_2O_5$) Cl] | 45.65 (45, 71) | 3.01 (3.03) | 5.58 (5.61) | - - | 7.07 (7.10) | | 22.48 (22.52) |

**Infrared spectra:**

Infrared spectral study of metal complexes was recorded for examination of bonding pattern in the synthesized complexes. The assignments to each bonding mode are supported by literature values. Important absorption bands are presented in Table No 2. The comparison of IR spectral data of all the complexes and corresponding ligands helps in concluding the bonding pattern of each complex.

The new bands in the regions 544 $cm^{-1}$ and 430 $cm^{-1}$ observed in the complex spectra may be assigned to stretching of M–O and M–N bond respectively [33-35].

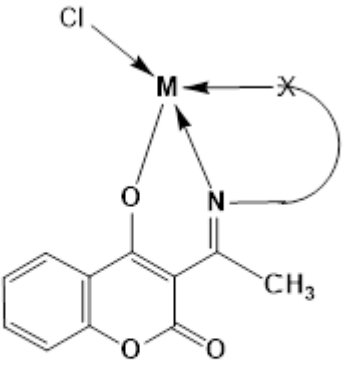
**Table 2:** Infrared Absorption Frequencies ($cm^{-1}$) N'-(1-(4-hydroxy-2-oxo-2H-chromen-3-yl) ethylidene)-4 methoxybenzohydrazide ($L_1$):

| S. No | Ligand/ Complex | Bond vibrational modes (stretching – v) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Lactone | Hydrazide | Azomethine | Enolic | New Peaks | |
| | | (C=O) | (C=O) | (C=N) | (C=O) | M-O | M-N |
| 1 | L1 | 1708 | 1678 | 1610 | 1360 | --- | --- |
| 2 | Zn (L1) Cl | 1702 | 1655 | 1579 | 1355 | 544 | 465 |
| 3 | Cd (L1) Cl | 1707 | 1660 | 1580 | 1357 | 519 | 430 |

On the basis of elemental analysis, conductivity, magnetic susceptibility the Zinc (II) and Cd (II) complexes of present work may be assigned as monomeric structure with tetrahedral geometry.

## 5. Conclusion

Hence on the basis of elemental analysis, IR spectra, conductivity measurement data, following tetrahedral structures are proposed for Zn (II) and Cd (II) complexes.




Monomeric tetrahedral Structure of Zn(II) and Cd(II)Complexes with Ligand $L_1$ Where X= O

## References

[1] Kumar, S., Niranjan, M. S., Chaluvaraju,, K. C., Jamakhandi, C. M. and Kadadevar D., *Journal of Current Pharmaceutical Research*, **1,** 39-42, (**2010).**

[2] Singh, U. K., Pandeya, S. N., Sethia S. K., Pandey, M., Singh A., Garg A. and Kumar, P., *Int. J. of Pharm. Sci. and Drug Res.*, **2 (3),** 216-218, (**2010).**

[3] Kratky M., Sova J. V., Volkova M., Buchta V., Trejtnar F. S. and Rikova J., *European Journal of Medicinal Chemistry*, **50**, 433-440, (**2012**).

[4] Suresh, R., Kamalakkannan, D., Ranganathan, K., Arulkumaran, R., Sundararajan, R., Sakthinathan, S. P., Vijayakumar, S., Sathiyamoorthi K., Mala, V., Vanangamudi, G., Thirumurthy, K., Mayavel P., and Thirunarayanan G., *SpectrochimActa A MolBiomolSpectrosc*, **101,** 239–248, (**2013**).

[5] Vijesh, A. M., Isloor A. M., Shetty P., Sundershan S. and Fun H. K., *European Journal of Medicinal Chemistry*, **62,** 410-415, (**2013**).

[6] Pudota, P. T., Purohit R. S. M. and Pujar G. V., *Journal of Applied Chemical Research*, **7**, 7-18, (**2013**).

[7] Hussain, Z., Yousif E., Altaie A., *Organic and Medicinal Chemistry Letters*, **4**: 1, (**2014**).

[8] Asha, B. T., Rabindra K. N., Lata P. K. and Sunil C. H., *Arabian Journal of Chemistry*, Synthesis and biological evaluation of Schiff's bases and 2-azetidinones of isonocotinylhydrazone as potential antidepressant and nootropic agents, (**2011);** doi: 10.1016/j. arabjc.2011.02.015.

[9] Shaoyong, K., Yanhong, W., Ziwen, Y., Kaimei, W., Ying L. and Liqiao S., *Bioorganic & Medicinal Chemistry Letters*, **23**, 5131–5134, (**2013).**

[10] Monteiro, A., Gonçalves, L. M. and Maria, M. M., *European Journal of Medicinal Chemistry*, **79**, 266-272, (**2014**).

[11] Isloor, A. M., Kalluraya B. and Shetty P., *European Journal of Medicinal Chemistry*, **44,** 3784-3787, (**2009**).

[12] Wang, X., Yin J., Shi L., Zhang G. and Song B. *European Journal of Medicinal Chemistry*, **77**, 65-74, (**2014**).

[13] Kolundz, B., Markovic, V., Stanojkovic, T., Joksovic L., Matic I., Todorovic, N., MarijanaNikolic, M., Milan, D. and Joksovic, M. D., *Bioorganic & Medicinal Chemistry Letters* **24,** 65–71, (**2014**).

[14] DeMilo A. B., Redfern R. E., *Journal of Agricultural and Food Chemistry*, **27,** 760-762, (**1979**).

[15] Gumrukcuoglu, N., Sokmen B. B., Ugras S., Ugras H. I. and Yanardag R., *Journal of Enzyme Inhibition and Medicinal Chemistry*, **28**, 89-94, (**2013**).

[16] Sztanke, K., Maziarka A., Osinka A. and Sztanke M., Bioorganic *& Medicinal Chemistry*, **21**, 3648-3666, (**2013**).

[17] Mohana, K. N., Mallesha L., *Journal of Fluorine Chemistry*, **156,** 15-20, (**2013**).

[18] Simone, M. de Souza, Franco, Delle M., and ArturSmaniaJr. Z., Naturforsch.6**0c,** 693-700 (**2005**) and Kayser O., and Kolodziej H., *Z. Naturforsch*.5**4c**, 169-174 (**1999**).

[19] Cristina, M., Simone, M. D., Claudia, G., Franco, D., Elza, F. A., Smania, and Artur (jr) S. *Z. Naturforsch.6***3c**, 21-28 (**2008**).

[20] Tripathy, R. P., Tripathy, R., Bhaduri, A. P., Singh, S. N., Chatterjee, R. K., and Murthy, P. K., *ActaTropica***, 76**, 101-106, (**2000**).

[21] Uchiumi, F., Hatano, T., Ito H., Yoshida, T., and Tanuma, S. I., *Antiviral Res.5***8**, 89-98, (**2003**).

[22] Pineo, G., and Hull, R. D., *Hematol. Oncol. Clin. North America* **17**, 201-216, (**2003**).

[23] Tyagy, Y. K., Kumar, A., Raj, H. G., Vohra, P., Gupta, G., Kumari, R., Kumar, P., and Gupta, R. K., *Eur. J. Med. Chem.4***0**, 413-420, (**2005**).

[24] Bighetti, A. E., Antonio, M. A., Kohn, L. K., Rehder, V. L. G., and Foglio, M. A., *Phytomedicine***, 12**, 72D77, (**2005).**

[25] Kontogiorgis, C. and Hadjipavlou-Litina, D., *J. Enz. Inhib. Med. Chem.1***8**, 63-69, (**2003**).

[26] Dongmo, A. B., Azebaze, A. G., Nguelefack, T. B., Ouahouo, B. M., Sontia, B., Meyer, M., Nkengfack, A. E., Kamanyi, A., and Vierling, W., *J. Ethnopharmacol*., **111 (2),** 329-34, (**2007**).

[27] Tong, L., Huang, T. Y., Liang, M., Wu, P., Liang, N. C., and Li., J. L. *Chinese Pharmaceutical Bull*., **10**, 105–109, (**1994**).

[28] Tyagi J. S., Paruthi, H. K., Taneja A. D., and Prasad, C. R. G., *Chem. Era*, **11**, 22, (**1975**).

[29] Sorenson, J. R. J. and Singel, H., "Metal ions in biological systems", Marcel Dekkar, New York, p.77, (**1982**).

[30] Dresner, J., RCA, Rev, **30,** 322, (**1969).**

[31] B. S Furniss BS; V Rogers PWG Smith; AR Tatchell, Vogel's text book of practical organic chemistry, 5$^{th}$Ed$^{n}$ (Longman scientific and Technical, Essex England), **1989**, pp1269-70.

[32] Thaisrivong s. s., Tomich, paulKeith., D water paugh., Kong-Teck Chong., W. Jeffrey Howe., Chin-Ping Yang., Joseph, W. Strohbath., Steve, R. T., Jamps, P. Mcurath., J. Med. Chem.37, (20), 3200-3204, (1994).

[33] Rahangdale, M., Pethe, G., Yaul A., and Aswar, A., Research J. of Pharma., Bio. And Chem. Sci., 2 (3), 341-348, (2011). Rao, N. R., Venkateshwar, P. R. Reddy, G. V. and Ganorkar, M. C., Indian J. Chem., 26A (10), 887 (1987).

[34] Rao, N. R., Venkateshwar, P. R. Reddy, G. V. and Ganorkar, M. C., Indian J. Chem., 26A (10), 887 (1987).

[35] Aguiari, A., Bullita, E., Casellato, U., Guerriero, P., Tamburini, S., Vigato, P. A. and Russo, U., Inorganic Chim. Acta, 219 (1-2), 135-146, (1994).