Available online at www.pelagiaresearchlibrary.com

Pelagia Research Library

Der ChemicaSinica, 2015, 6(2):1-4

Der Chemica Sinica
Pelagia Research Library
ISSN: 0976-8505
CODEN (USA) CSHIA5

Synthesis and evaluation of Zn (II) Imiquimod complex

Shruti S. Sarwade*, W.N. Jadhav, B.C. Khade and S.R. Bhusare.

Department of Chemistry, Dnyanopasak Science College, Parbhani-431401.
Swami Ramanand Teerth Marathwada University Nanded, Maharashtra, India.

ABSTRACT:

New complex of Zn (II) Imiquimod of the composition has been synthesized by the interaction of $ZnCl_2.4H_2O$ with said ligand in ethanol medium. The complex so obtained has been characterized on the basis of molar conductance, magnetic measurement, IR, UV and thermogravimetric analyses.

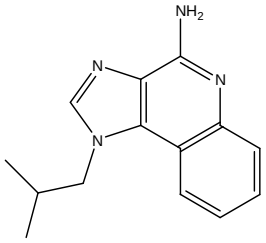
Keywords: Zn complex, Imiquimod, IR, UV, TG/DTA.

INTRODUCTION

Metal co-ordination to biologically active molecule can be employed as a strategy to enhance their activity and overcome resistance, for instance, metal complexes of thiosemicarbazones can be more active than the free ligand, or can be employed as a vehicle for activation of the ligand as the cytotoxic agent.

It has also been reported that metal complexes as a chemotherapeutic drugs has become a vibrant and growing area of research in recent time and some of the metal based drugs are already in the market for treating various diseases and aliments.

Imiquimod is a novel synthetic compound that is member of the imidazoquinolone family of drugs. This class of drugs is unique because it has properties of topical immune response modifiers and stimulators. Imiquimod resembles a nucleoside analog and is known for its potent induction of endogenous antiviral pro- inflammatory mediators [1]. Imiquimod formulated as a5% (w/w) cream is approves by US Food and Drugs Administration for treating external genital and perianal warts[2].



3-(2-methylpropyl)-3,5,8-triazatricyclo[7.4.0.02,6]trideca-1(9),2(6),4,7,10,12-hexaen-7-amine

Fig. 1 Structure of Imiquimod

MATERIALS AND METHODS

Materials:
All chemicals used in this study were of analytical grade. They included Imiquimod (Imi) of the formula $C_{14}H_{16}N_4$, 240.30.42g/molandmetal salt $ZnCl_2.4H_2O$. These chemicals were purchased from Merch or Aldrich. The organic solvent ethanol was purchased from BDH and used without further purification.

Synthesis of Imiquimod Zinccomplex.
Complex was prepared by dissolving pure Imiquimod (0.1mM, 0.24 gm.) in ethanol and adding solution to ethanolic zinc chloride solution (0.1mM, 0.13gm). The reaction mixture was refluxed for 2hrs. The mixture was filtered .The complex was dried in oven. White powder is obtained.

RESULTS AND DISCUSSION

The complex was found to be non-hygroscopic solid with white colour. The complex is soluble in DMSO and DMF. Conductivity of the complex, at room temperature suggests its non- electrolytic nature. The analytical data of the metal complex showed 1:2 stoichiometry of (M: L).

Table 1: Analytical data of ligand/complex:

Sr.no.	Composition of complex	colour	Yield %	M.P(^{0}C)	Magnetic moment (B.M)	Molar conductance Ω $^{-1}$cm^{2}mol^{-1}
1	$C_{14}H_{16}N_4$	yellow	-	191	-	-
2	$Zn(C_{14}H_{16}N_4)_2Cl_2$	white	72	195	diamagnetic	10.64

UV Visible spectra and magnetic moment:
Absorption bands of ligand and its metal complex showed the complex gave strong absorption bands at438nm, 330nm and 271nm. UV/ Visible spectra of this ligand and its complex has been interpreted in terms of charge transfer transitions from the metal to the antibonding orbital of the ligand and of the ∏-∏* transition. These bands undergo hypsochromic shifts in the metal complex due to complexation ions of the ligand [3].

The Zn (II) complex is diamagnetic, as expected as the metal ion has d^{10} configuration [4].

Eleni K. et. al synthesized complexes of antibacterial agent pipemidic acid, they reported UV bands of Zn(II) at 330nm, 369nm and 425nm(shoulder) attributed to charge transfer bands and having octahedral geometry.

Infrared spectra:
The IR spectra of the free ligand and its metal complex were carried out in the range of 4000-400 cm^{-1}. The assignments have been carried out based on the literature values obtained for similar structure compounds.

The important IR frequencies (in KBr) of the ligand, and the metal complex with tentative assignments are given.

The band around 3400-3100 cm^{-1} is assigned to *ν* N-H and is supported by the presence of *d*(NH_2) deformation bands around 1600-1500 cm^{-1}.The *ν* N-H vibration frequency is displaced in the complex .The movement of the band 3334 cm^{-1} to lower wave number suggests the involvement of the amino nitrogen in the coordination to the metal ions. The *ν* C=N 1529cm^{-1} in Imiquimod is shifted to 1510cm^{-1} in the complex.

From the above discussion it can be said that Imiquimod coordinates to Zn(II) through amine NH_2 group nitrogen and -C=N group of thiazole [5,6].

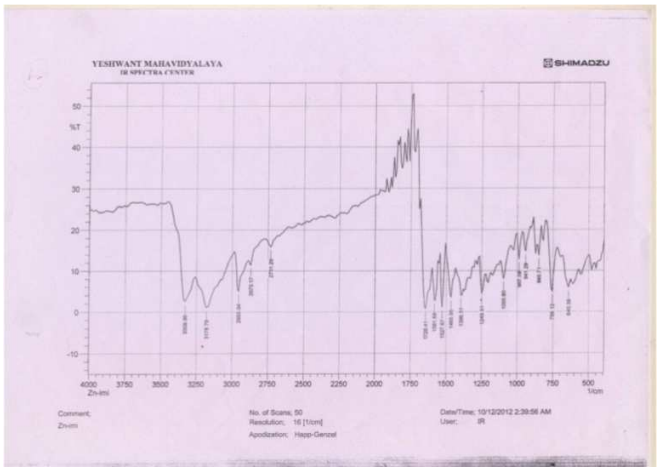


Fig. 2 IR Spectra of complex.

THERMOGRAVIMETRIC ANALYSIS:
TGA and its derivative thermogram are shown. The elimination of rest of the part occurs finally.

The TG curve shows single step decomposition within temperature range 295-526^{0}C. The major portion of Imiquimod ligand is eliminated in the first stage degradation. ZnO is suggested as the metallic residue. The step is accompanied by two endothermic and one exothermic peak.

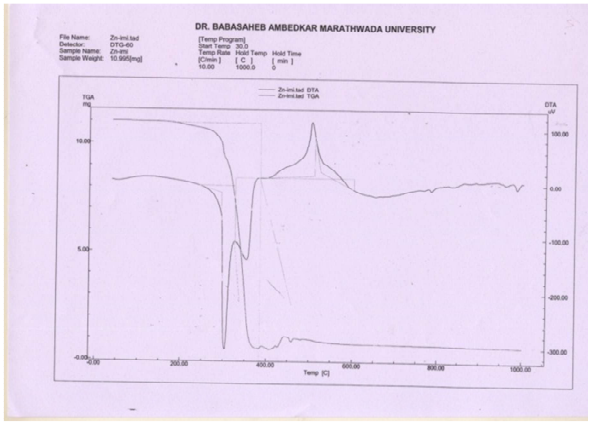


Fig. 3 TG/ DTA graph of complex.

CONCLUSION

Hence on the basis of elemental analysis, IR, UV and thermogravimetric study following structure of complex is proposed.

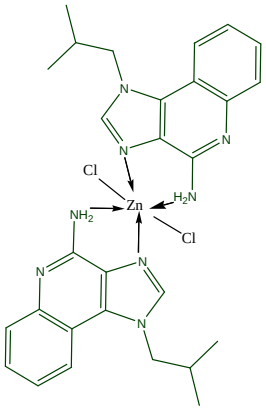


Fig. 4 Structure of complex.

REFERENCES

[1] Daniel De Paula, Carolina Azenha Martins and Maria Vitoria Lopes Badra Bentley, *Biomed.Chromatogr*.**2008**, 22: 1416-1423.
[2] Bo Zhao, Yu-ZhiRong, Xiao-HuaHuanga and Jing-Shan Shen, *Bioorganic & Medicinal Chemistry Letters* 17, **2007,** 4942–4946.
[3] Eleni K. Efthimiadou, YiannisSanakis, Nikos Katsaros, Alexandra Karaliota and George Psomas, *polyhedron*, **2007**, 26, 1148-1158.
[4] Salman M. Saadeh, *Arabian Journal of Chemistry*, **2013**, 6, 191–196.
[5] 5.Gehad G. Mohamed, M.A. Badawy, M.M Omar, M.M. Nassar, A.B. Kamel, *SpectrochimicaActa part* A 77 (**2010**) 77-781.
[6] Bamigboye M.O., Obaleye J.A Lawal M. and Aluko O.M. , *Chemistry and Material Research*, **2012**,vol 2, No. 3.