



Short Communication

Synthesis, characterization of Schiff base Pyrazolone compound

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Abstract

Research in chemistry-synthesis of novel Schiff base ligands and its metal complex many research area and pharmaceuticals. Mainly pyrazolone derivative are novel ligands synthesis, characterization and application in various research area and many documented. Schiff base ligands and transition metal complex show good activity in research application in area of inorganic chemistry, pharmaceuticals and coordination chemistry as well as medicinal chemistry. Due to so many large scale compounds structure variability and highly biological activity importance in our main interest for research for novel synthesis, inorganic metal complexes and coordination transition-metal series with novel Schiff base pyrazole ligands.

Keywords: Schiff base pyrazolone, Pyrazolone compound, Schiff base, Metal complex, Schiff base ligand.

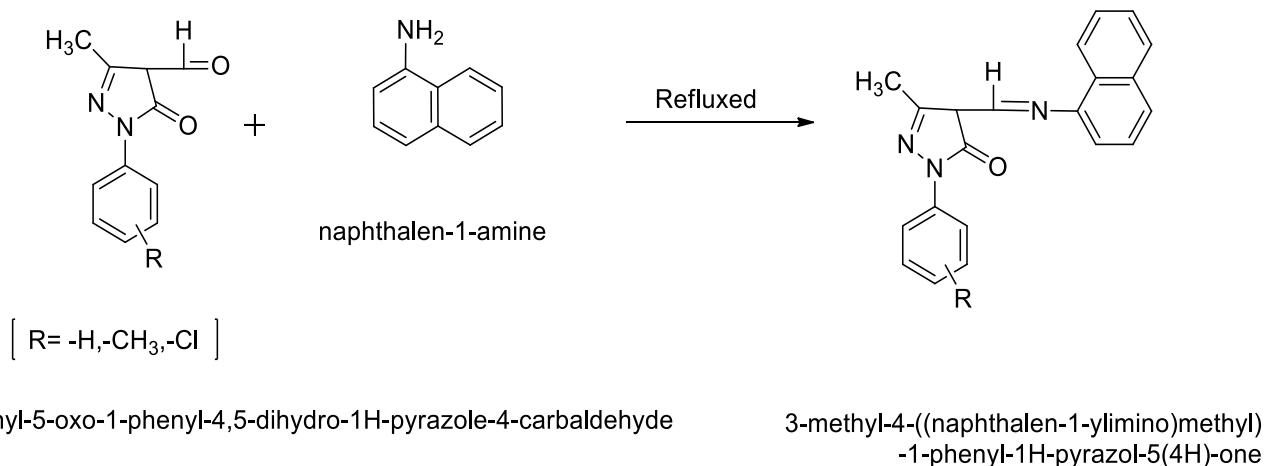
Introduction

Research in chemistry-Synthesis of novel Schiff base pyrazolone ligands and its metal complexes has many research areas. Schiff base Pyrazolone ligands have a huge area for synthesis various compounds and having highly biological active structure. Manly Schiff base pyrazolone derivative are 3-methyl-5-oxo-1-phenyl-4,5-dihydro-1H-pyrazole-4-carbaldehyde which synthesis, Characterization and biological application are well documented. Due to its rich biological activity and effective of broad spectrum antibacterial activity, antipyretic activity and antitumor, anti-tuberculosis, anticonvulsant and antidepressant activities, Schiff base pyrazolone and its metal complexes have found huge research area as well as in coordination chemistry, metal complexes inorganic chemistry and medicinal chemistry received considerable attention.

Materials and methods

All Chemical have laboratory grade. Purification of all solvent then used for synthesis of compounds. Ethylenediamine (sigma-aldrich), 1-phenyl-3-methyl-2-pyrazoline-5-one (Merk), O-phenylenediamine (Merk), ZnCl₂.6H₂O (sigma-aldrich).

Synthesis of ligands: 1mmol of 3-methyl-5-oxo-1-phenyl-4,5-dihydro-1H-pyrazole-4-carbaldehyde, (R=-H)H₂L₁ was dissolved in methanol solvent. This solution slightly heated then added in to a solution of 1mmol of 1-aminonaphthalene in 50ml. This reaction mixture was refluxed 4-5 hours. After cool the solution. After cooling solution crystalline compound take separated. The compound are carefully purified with filtration then residue washed with diluted methanol and water then dry in oven. Above same method (R=-CH₃) H₂L² and (R=-Cl) H₂L³.



Scheme-1: Synthesis of ligands.

Results and discussion

Characterization of H₂L¹, H₂L², H₂L³

Ligand (H₂L¹): Yield: 81%, melting point: 159-161°C,

¹H NMR, (DMSO) (400 MHz, MS): d 2.34 (s,3H), 7.17-7.20 (t,1H), 7.43-8.11 (m,1H), 8.90-8.93 (d,1H), 12.39-12.42 (d,1H)

IR spectra (KBr,cm⁻¹): 3749, 3063, 1651(C=O), 1497(C=N_{cyclic}), 1350, 1281, 748)

EI-MS m/z: ----- M+ (calculated = 327)

Ligand (H₂L²): Yield 83%, melting point 154-158°C

¹H NMR (DMSO), (400 MHz, MS) : d 2.34 (s,3H), 2.35(s,3H), 7.24-7.26 (d,2H), 7.62-7.69 (m,2H), 7.73-7.78 (m,1H), 7.86-7.89 (d,1H), 7.90-7.92 (m,3H), 8.05-8.11 (m,2H), 8.89-8.91 (d,1H), 12.41-12.44 (d,1H)

IR spectra (KBr, cm⁻¹): 3742, 3055, 1651(C=O), 1512(C=N_{cyclic}), 1342, 1288, 771)

EI-MS m/z: ----- M+ (calculated = 341)

Ligand (H₂L³): Yield: 82%, melting point 173-175°C

¹H NMR (DMSO), (400 MHz, MS): d 2.31(s,3H), 7.46-8.07(m, 11H), 8.87-8.91(d,1H),12.28-12.31(d,1H).

IR spectra (KBr, cm⁻¹): 3742, 3063, 1651(C=O), 1481(C=N_{cyclic}), 1404, 1296, 764)

EI-MS m/z: ----- M+ (calculated = 361)

¹H-NMR Spectra: The ¹H NMR characterization spectral data of H₂L¹, H₂L² and H₂L³ in DMSO-d₆ at 25°C temperature. Ligands H₂L¹ and H₂L³ having two singlet with six protons integration near the δ 2.34ppm and δ 2.78ppm of CH₃ group connect to pyrazolone phenyl ring. Ligands of H₂L¹, H₂L² have triplet signal of ~ 1.5ppm present of methyl and proton of ethylenediamine. We get a sharp singlet at δ~8.90ppm, observed that group is aldehyde proton for all synthesis novel Schiff base ligands. We got a phenyl multiplet ring signal in the range for δ~7.33-7.97ppm. From the NMR reading and observed all data those ligands shows its keto-enol tautomerism.

Infrared Spectra: A Spectra of FT-IR summarized various frequency that characteristic IR spectra for the Schiff base ligands show spectral band region at broad area 3400-3700cm⁻¹ its show the generally free ν(OH). Mostly range we observed in spectra region 3100-3700cm⁻¹ due to present of ν(OH) in intermolecular of H-bonding. Its show that a keto-enol tautomeric form present in compound in solid state. Next frequency is a phenyl ring show ν(CH) at 3050cm⁻¹. ν(CH) at 3100cm⁻¹ and ν(C=C) at 1550cm⁻¹. A band range ~ 1650cm⁻¹, ~ 1590cm⁻¹, ~ 1310cm⁻¹. A spectra region between 288-3000cm⁻¹ present due to ν(C-H). A strong band of Schiff base ligand present δ(O-H) from 100-1280cm⁻¹.

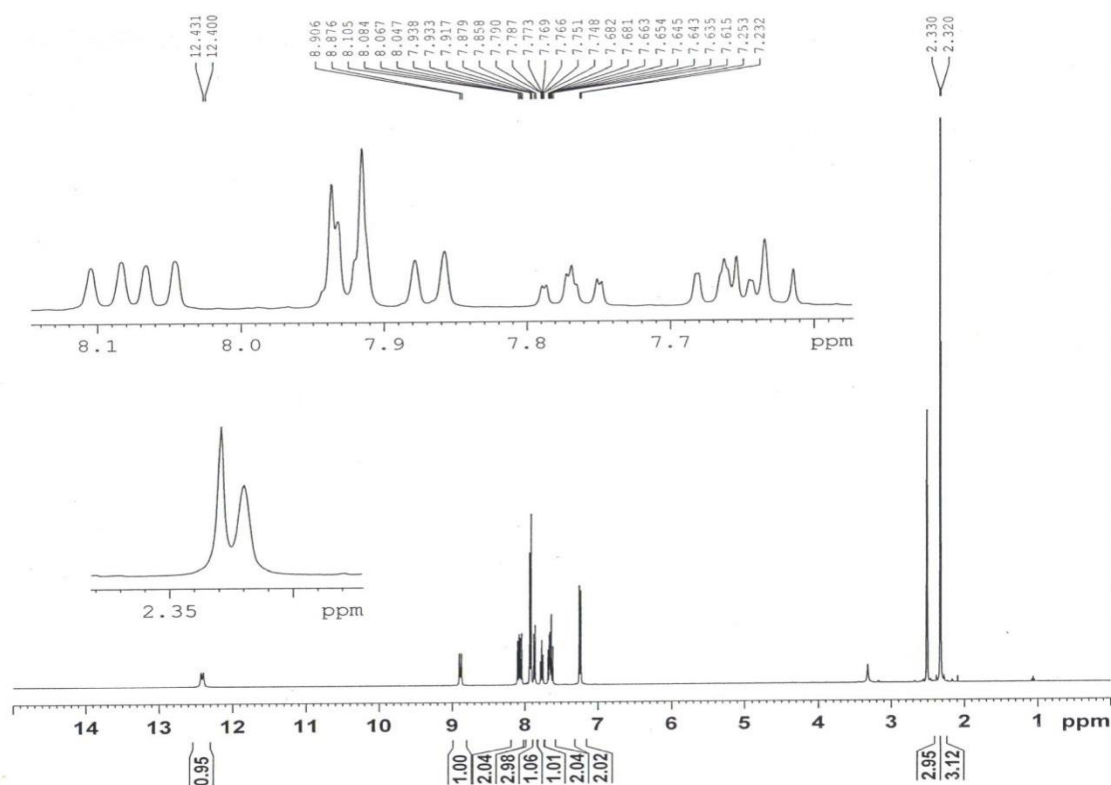


Figure-1: ¹H NMR H₂L².

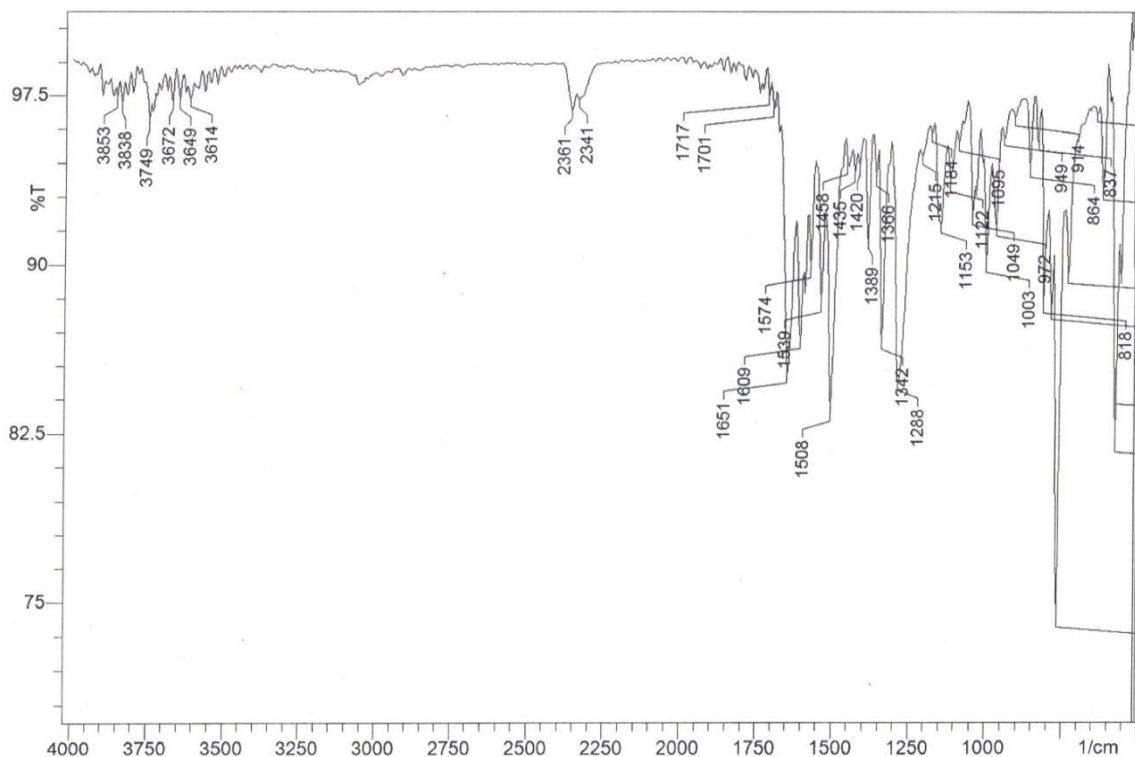


Figure-2: Infrared date H₂L³.

Conclusion

The novel coordination compounds of Schiff base pyrazolone give good biological activities as well as its metal complexes are proposed various applications in various fields of chemistry and pharmaceuticals area. A coordination of Schiff base with metal compounds are confirmed from NMR and FT-IR. Its biological activity of Schiff base compounds against antifungal and antibacterial make them more suitable in cells and tissues. Schiff base pyrazolone also have antioxidant properties because present high lipophilicity and down electron withdrawing capacity and high polarizability increase.

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