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Synthesis and spectral studies of Fe (II) and Co (II) complexes with cloxacillin

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Abstract

The complexes of Fe (II) and Co (II) with β -Lactam-cloxacillin have been reported. The structures of these newly synthesized complexes were substantiated by elemental analysis, molar conductivities, infrared, electronic absorption spectra and magnetic moment measurements. On the basis of these analyses it has been confirmed that direct reaction of metal salts with cloxacillin-suspension leads to formation of complexes.

Keywords: Transition metals, antibiotic, cloxacillin and, spectral analysis

Introduction

Cloxacillin possesses the β -lactam ring structure, which is used for the chemotherapy of bacterial infections as antibiotic. In addition to a broad spectrum of antibacterial action, two properties contribute to the unequaled importance of β -lactam antibiotics in chemotherapy. The uniquely lethal antibacterial action of cloxacillin has been attributed to a selective inhibition of bacterial cell wall synthesis [1] specially the basic mechanism involved is inhibition of the biosynthesis of the peptidoglycan that provides strength and rigidity to the cell wall of bacteria.

Cloxacillin is a fine, colourless, crystalline powder that is sparingly soluble in water. It is available in a variety of oral dosage forms [2-5].

Experimental

Complexes of cloxacillin Fe (II) and Co (II) ions

All chemicals such as cloxacillin, Fe (II) chloride and Co (II) chloride, methyl alcohol, ethyl alcohol, n-pentane, acetone, DMF and DMSO used in this study were of analytical grade. These chemicals were purchased from CDH or Merck.

Preparation of solution

The 0.01 M aqueous solution of ligand cloxacillin, was taken in a dry and clean conical flask containing 50 ml of redistilled water. The flask was fitted with cork and stirred vigorously with glass rod for two hours. To make it clear solution it was again vigorously stirred with an electrical stirrer for three hours. But I failed to get its clear solution. So its suspension was used as such for complexation.

A 0.01 M solution of metal salts were prepared by dissolving appropriate amount of metal salt in 10 ml of redistilled water in a dry and clean conical flask. The solution was filtered to remove any insoluble residue.

Preparation of complexes

The complexes were prepared by the slow addition of 10 ml of 0.01 M metal salt solution to the magnetically stirred 50 ml of 0.01 M of ligand cloxacillin suspension at room temperature. As the amount of metal cation was increased cloxacillin dissolved. The solution was attained coloured syrupy state. The concentration of final solution gave rise to colour precipitates, which were filtered and washed with distilled water, ethanol and ether regularly and repeatedly three times. The precipitates were dried in a desiccator over calcium chloride, and then solid complexes were obtained. Solubility tests of these complexes were performed. The thin layer chromatography of this complex was performed by using Aluminium plates coated with silica gel-60GF254 as the stationary phase. The solvent system was consisted of butyl acetate glacial acetic acid, methanol, water (5:2.5:2.5:1, V/V) as mobile phase. Its melting point was determined using open capillary tube in kjeldal flask filled with concentrated sulphuric acid.

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The elemental analysis of the complex was carried out at Central Drug Research Institute (CDRI), Lucknow. The

elemental analysis of the complex is given as:

Elemental analysis

	% Of metal ion Cal/(found)	% Of C Cal/(found)	% Of H Cal/(found)	% Of N Cal/(found)
[Fe(clox)Cl(H ₂ O) ₃]	9.63/(9.43)	39.32/(39.08)	3.97/(3.92)	7.24/(7.11)
[Co(clox) ₂ (H ₂ O) ₂]	6.11/(6.02)	47.31/(47.13)	3.53/(3.51)	8.71/(8.58)

Complexes of cloxacillin

The complexes of cloxacillin, with lanthanide metal and transition metal (II) ions (Fe²⁺ and Co²⁺) are anhydrous as evident from analytical, spectral studies. All these complexes are quite stable at room temperature. The complexes are generally soluble in common organic solvents such as alcohol, benzene, DMF, DMSO but partially soluble in diethyl ether, and in water.

The molar conductance values are too low to account for any dissociation. Thus complexes are non-electrolytes.

Spectral Studies transition metal Complexes with cloxacillin

The infrared, ¹H NMR and electronic spectra of the complexes were obtained from external agency C.D.R.I., Lucknow. The IR spectral data and the assignment of the ligand and its metal complexes are given in following table.

Compound	$\nu_{(\text{CN})}$ cyclic	$\nu_{(\text{NH}_2)}$	$\nu_{(\text{CO})}$ β - lactame	$\nu_{(\text{NH})}$ amide	$\nu_{(\text{MO})}$	$\nu_{(\text{MN})}$	$\nu_{(\text{enol})}$
cloxacillin	1325	3290	1770	3050
[Fe(Clox) ₂ (H ₂ O) ₃ Cl]	1380	3295	1585	410	545	1585
[Co(Clox) ₂ (H ₂ O) ₂]	1375	3285	1605	445	610	1580

The infrared spectra of complexes of cloxacillin with transition metal (II) ions and the ligand cloxacillin were recorded in the range of 400–4000 cm⁻¹. The highest frequency of the bands of the ligand at ~3050 cm⁻¹ can be assigned [6] to the stretching ν_{NH} vibration of the –NH group in the ligand cloxacillin. This band is disappear in the all complexes of metal (II) ions indicates that the Complexation [7] of the NH group through metal ions. The frequency at 1770 cm⁻¹ in IR spectra of the ligand due to the carbonyl

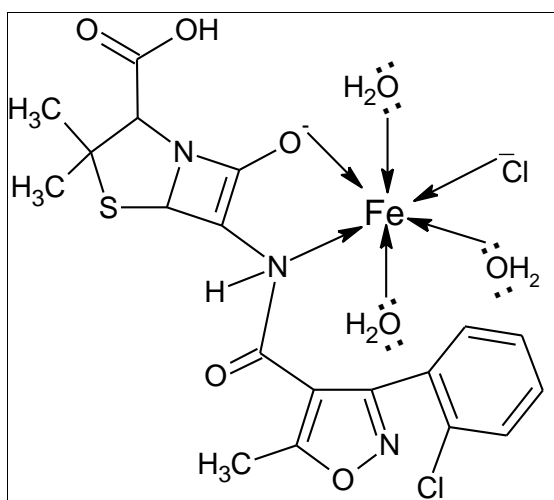
group present in the β -lactam ring reduced to 1585–1605 cm⁻¹ in the metal complexes, which indicates [8] the participation of the group in the complexation.

Thus, on account of infrared spectral properties one can say that cloxacillin acts as bidentate ligand. The electronic spectral data for the solution of these complexes investigated in acetonitrile are recorded in CDRI, Lucknow and presented in the following table.

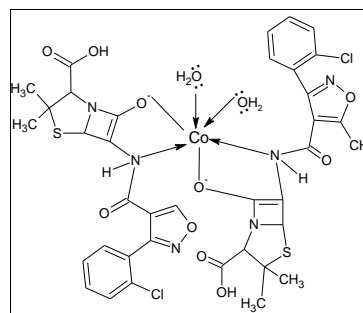
Complexes	Spectral bands	Transitions
[Fe(Clox)(H ₂ O) ₃ Cl]	18575, 22750, 24845, 24900, 25000, 27900, 29540, and 32550.	⁶ A _{1g} , ⁴ T _{1g} , ⁴ T _{2g} (G), ⁴ E _g , ⁴ A _{1g} , ⁴ T _{2g} (D)
[Co(Clox) ₂ (H ₂ O) ₂]	8750, 14525, 25375	³ A _{2g} , ³ T _g , ³ T _{1g} (F), ³ T _{1g} (P).

Conclusion

The complex of Fe (II) with cloxacillin has one chloride, one cloxacillin and three water molecule. So, structures of complexes are six coordinated as below:



The complex of Co (II) with cloxacillin has two cloxacillin and two water molecules. So, structures of complexes are six coordinated as below:



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