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Research Article

**Preparation, Analytical and Spectral characterization
of Cr(III) Complex With 2,4-Thiazolidinedione and
Benzoate ion**

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ABSTRACT

Complex of Cr(III) with 2,4-thiazolidinedione (TZD) as a primary ligand and benzoate ion (BEN) as a secondary ligand has been synthesized under microwave irradiation and characterized by micro analytical data, metal estimation, molar conductance, magnetic moment, UV-Visible, IR and Far-IR spectral studies. Composition and non-electrolytic nature (1:0 type) of the complex was confirmed by the micro analytical, metal estimation and molar conductance values. Probable geometry of the complex was arrived from the magnetic moment and UV-Visible spectral data. IR and Far-IR spectra of the complex confirm the TZD coordination through nitrogen atom whereas benzoate ion coordinates through the oxygen atom.

Key words: 2,4-thiazolidinedione, Benzoate, Cr(III) complex, Microwave.

INTRODUCTION

2,4-thiazolidinedione belongs to a pharmacologically important class of heterocyclic compounds used for the treatment of type-2 diabetes.¹⁻² TZD derivatives lower the plasma glucose levels by acting as ligands for α -peroxylase proliferators-activated receptors.³⁻⁴ In addition, this class of heterocyclic compounds possesses various other biological activities such as antihyperglycemic, antimicrobial, anti-inflammatory, anticonvulsant and insecticidal, etc.,⁵⁻⁷ TZDs are also known for lowering the blood pressure and thereby reducing chances of heart failure and micro-albuminuria in patients with type-2 diabetes.⁸⁻⁹ A survey of literature reveals that metal complexes of many drugs have been found to be more effective than the drug alone.¹⁰⁻¹¹ Therefore much attention is given to the use of TZD due to its high complexing nature with essential metals. On the other hand,

microwave irradiation now a day is an accepted tool for accelerating the organic and inorganic reactions it leads to high reaction selectivity and utilization of minimum amount of solvents: It is an eco-friendly technique.¹²⁻¹³ This paper deals with the microwave assisted preparation and spectral characterization of Cr(III) using TZD and benzoate ion as ligands.

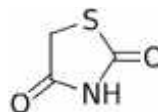


Figure-1
2, 4-thaizolidinedione

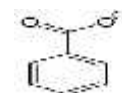


Figure - 2
Benzoate ion

EXPERIMENTAL:

1) Materials

2,4-thiazolidinedione (Merck), sodium benzoate,

chromium nitrate, DMSO, DMF, methanol, ethanol were of AnalaR grade, and used as such without further purification.

2) Method

a) Preparation of Cr(III) complex

Chromium nitrate 1.00g (2.50 mmol) dissolved in methanol, 2,4-thiazolidinedione 0.90 g (7.47 mmol) in methanol was added in drops with constant stirring; The mixture was irradiated in a microwave oven for about 10 seconds. Then sodium benzoate 1.08 g (7.50 mmol) in ethanol was added to the above solution and the whole mixture was irradiated in a microwave oven for 10 seconds. The precipitated green coloured complex was filtered, washed with ethanol and dried.

b) Instrumental techniques

The elemental analysis of the complexes was carried out by using (Thermo Finnegan make, Flash EA1112 Series Instrument) CHNS (O) analyzer. The electrical conductivity measurements were conducted using 10^{-3} M solutions of the metal complexes in acetonitrile with Systronic Conductivity Bridge 304 at 30°C. The UV-Visible spectra of Cr(III) complex was recorded on Varian, Cary 5000 model UV Spectrophotometer. The IR spectra of the ligand and complex were recorded on a Perkin Elmer, Spectrum RX-I, FT IR spectrometer in $4000-400\text{ cm}^{-1}$ range with KBr pellet technique. The Far-IR Spectrum of the complex was recorded by Bruker 3000, FT IR Spectrometer.

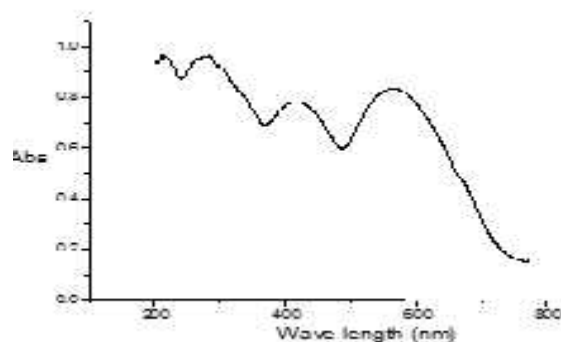


Figure – 3
UV-Visible spectrum of Cr (III) complex

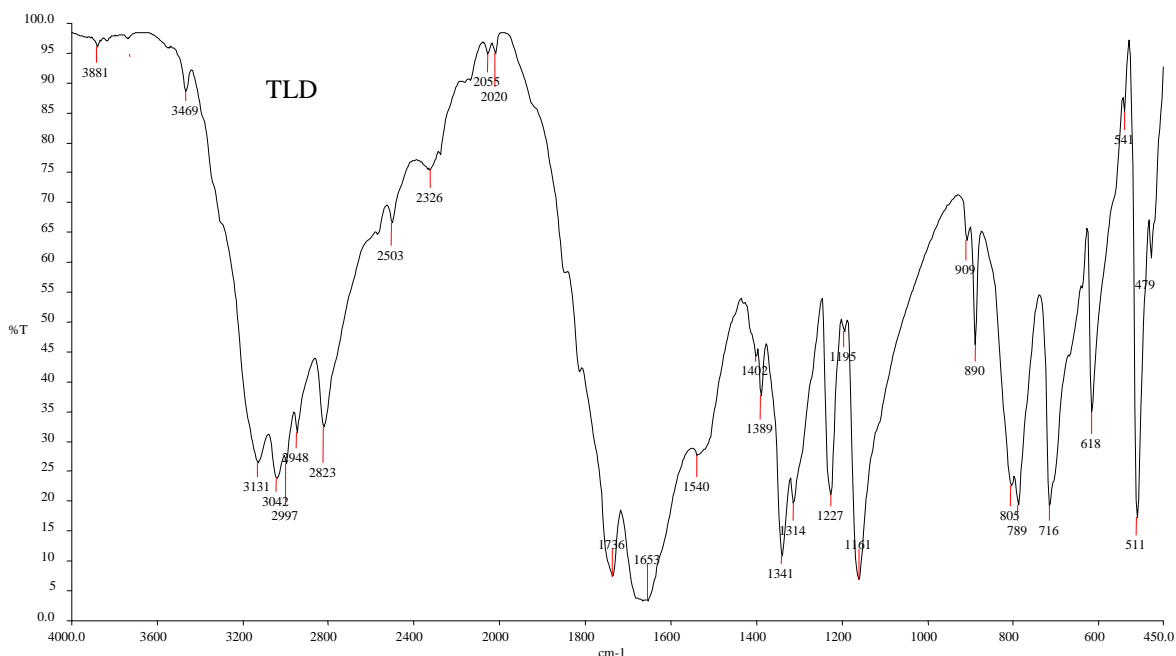


Figure-4
IR spectrum of 2,4-thiazolidinedione

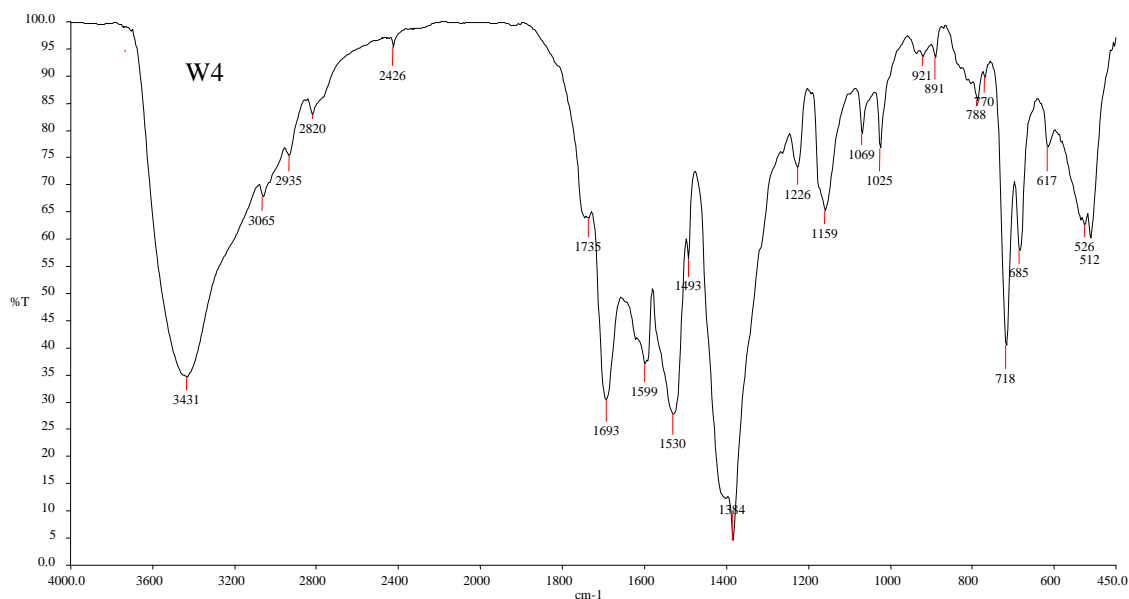


Figure-5
IR spectrum of $[\text{Cr}(\text{BEN})_3(\text{TZD})_3]$

Table-1
Analytical data of $[\text{Cr}(\text{BEN})_3(\text{TZD})_3]$

Complex	% of elements						Molar conductance ($\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$)	Yield (%)
	C	H	N	O	S	M		
$[\text{Cr}(\text{Benz})_3(\text{TZD})_3]$	46.99	4.60	5.48	25.06	12.05	6.78	90.30	65

RESULTS AND DISCUSSION

Analytical data

The prepared complex was green in colour with the yield of about 65%. The elemental analytical data were in good agreement with the calculated values. From the results of elemental analysis and metal estimation, the formula of the complex was given as $[\text{Cr}(\text{BEN})_3(\text{TZD})_3]$. The molar conductance value ($90.3 \text{ ohm}^{-1}\text{cm}^2\text{mol}^{-1}$) indicates the non electrolytic (1:0 type) nature of the complex.¹⁴

Magnetic moment and UV-Visible spectra:

The magnetic moment value of Cr(III) complex 3.50 BM, which confirm its octahedral geometry. The complex exhibits three λ_{max} values at 582 nm (λ_1), 420 nm (λ_2) and 276 nm (λ_3) which corresponds to ${}^4\text{T}_{1g}$, ${}^4\text{A}_{2g}$, ${}^4\text{T}_{1g}(\text{F})$, ${}^4\text{A}_{2g}$ and ${}^4\text{T}_{1g}(\text{P})$, ${}^4\text{A}_{2g}$. These transitions also confirm the distorted octahedral geometry of the complex.¹⁵

IR and Far-IR spectra¹⁶⁻¹⁷

The IR spectra give the information about the functional groups present in the ligand which are entering into the coordination sphere. The IR spectrum of free TZD showed (C=O) at 3437cm^{-1}

, (N-H) at 3452cm^{-1} and (C-N) at 1286cm^{-1} . The (N-H) stretching frequency gets broadened in the complex at 3431cm^{-1} . The benzoate ion shows the asymmetric and symmetric stretching vibrations of carboxylate group in the region $1352\text{-}1550\text{cm}^{-1}$ which get broadened in the complex ($1535\text{-}1599\text{cm}^{-1}$). These observations indicate that the ligands which have entered into the coordination sphere of the Cr(III) complex. In far-IR spectrum, the M-N coordination of TZD at 507cm^{-1} and M-O coordination of benzoate ion at 419cm^{-1} confirm the ligating atoms of both the ligands.

CONCLUSION

Cr(III) complex with TZD and benzoate ion was prepared under microwave irradiation and characterized by various physico-chemical and spectral methods. The molecular formula of the complex is $[\text{Cr}(\text{BEN})_3(\text{TZD})_3]$. The probable geometry of the complex is distorted octahedral.

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