

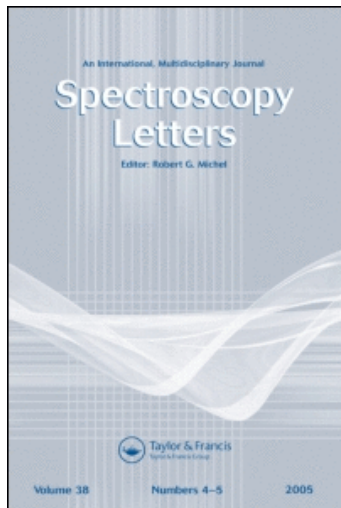
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Coordination Chemistry of Thenoyltrifluoroacetone 1- Synthesis and Characterization of In^{3+} - thenoyltrifluoroacetone Complex

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Abstract

In^{3+} -thenoyltrifluoroacetone $\text{In}^{3+}(\text{TTA})_3$ Complex has been prepared by reaction of $\text{In}(\text{SO}_4)_3 \cdot 2\text{H}_2\text{O}$ and thenoyltrifluoroacetone ligand in presence of acetate buffer. The complex has been investigated by conventional chemical and physical methods of analysis. Microanalytical data of the investigated complex showed the formula of the complex to be $\text{In}(\text{TTA})_3$ formula. Trials to prepare mixed ligand complexes starting with $\text{In}(\text{TTA})_3$ using neutral co-ligand were unsuccessful. The complex is soluble in ethanol and could be of potential use in clinical studies.

Introduction

The coordination chemistry of Indium in different oxidation states has attracted interest because of its possible relevance to radiopharmaceuticals and nuclear medicine [1]. In-oxine compound for instance is proved for the labeling of leukocytes (white blood cells) which are used for imaging sites of infection or inflammation [1]. Thenoyl trifluoroacetone ligand has been studied extensively in the chelation solvent extraction of lanthanides and transition metal ions [2-5]. Few attention however has

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been paid to isolation and characterization of these chelate compounds in the solid state using conventional chemical and physical methods of analysis.

In the present investigation we synthesized and characterized a neutral In^{3+} -thenoyltrifluoroacetone complex of potential use in clinical studies.

Experimental

Abbreviations HTTA = 3-(2-Thenoyl)-1,1,1-trifluoroacetone

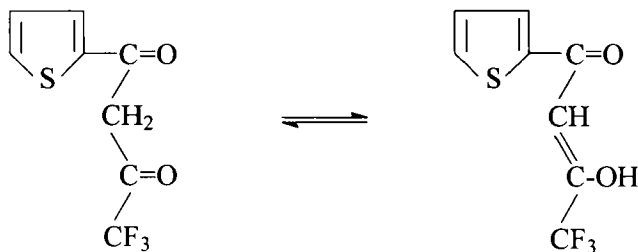
All chemicals used were of analytical grade. IR spectra were recorded in KBr Disk on a Perkin Elmer spectrophotometer Model 883. UV-Vis. Spectra (in $\text{C}_2\text{H}_5\text{OH}$) were measured on a Perkin Elmer Spectrophotometer Lampd 5. ^1H NMR were measured in DMSO on a FT-NMR GNM-Ex 400 MHz. Elemental analysis were carried out on Perkin Elmer 2400 CHN elemental Analyzer. X-ray powder diffraction was measured on Siemens D5000 using Cu tube operated at 40Kv, 35 mA.

Preparation of $\text{In}(\text{TFA})_3$

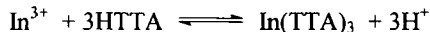
To a solution of (2.90 mmol) of $\text{In}(\text{SO}_4)_3 \cdot 2\text{H}_2\text{O}$ in acetate buffer medium (pH=5.5-6) was added ethanolic HTTA solution (30ml, 7.20 mmol). The reaction mixture was refluxed for about 3 hours. The solid product was separated, washed several times with ethanol. Slow evaporation of hot ethanolic solution of complex gave a white-red solid (1.40 g, 45%), m.p: 160°C (uncorrected), IR $\nu(\text{C}=\text{O})$ 1675 cm^{-1} . Analytical data [Found C, 37.49; H, 1.46 Calc. for $\text{InC}_{24}\text{H}_{12}\text{O}_6\text{S}_3\text{F}_9$ C, 37.02; H, 1.55].

Results and Discussion

Thenoyl trifluoroacetone (HTTA) has the following tautomeric form



metal complexation reaction was carried out according to the following equation:



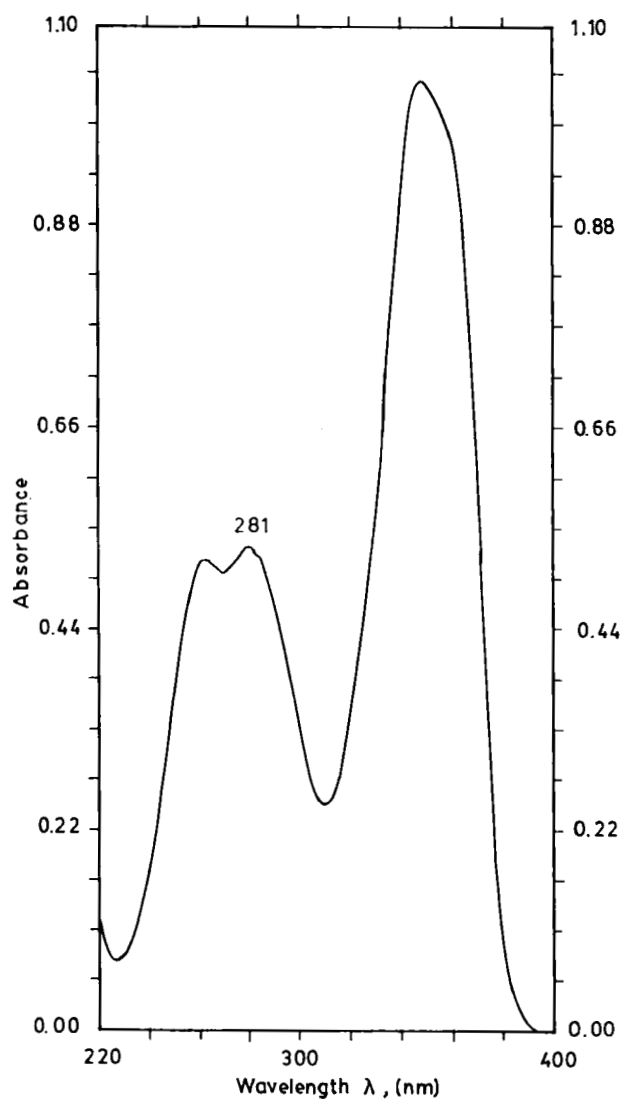


Fig. (1) UV-Vis. spectrum of $\text{In}(\text{TTA})_3$ Complex

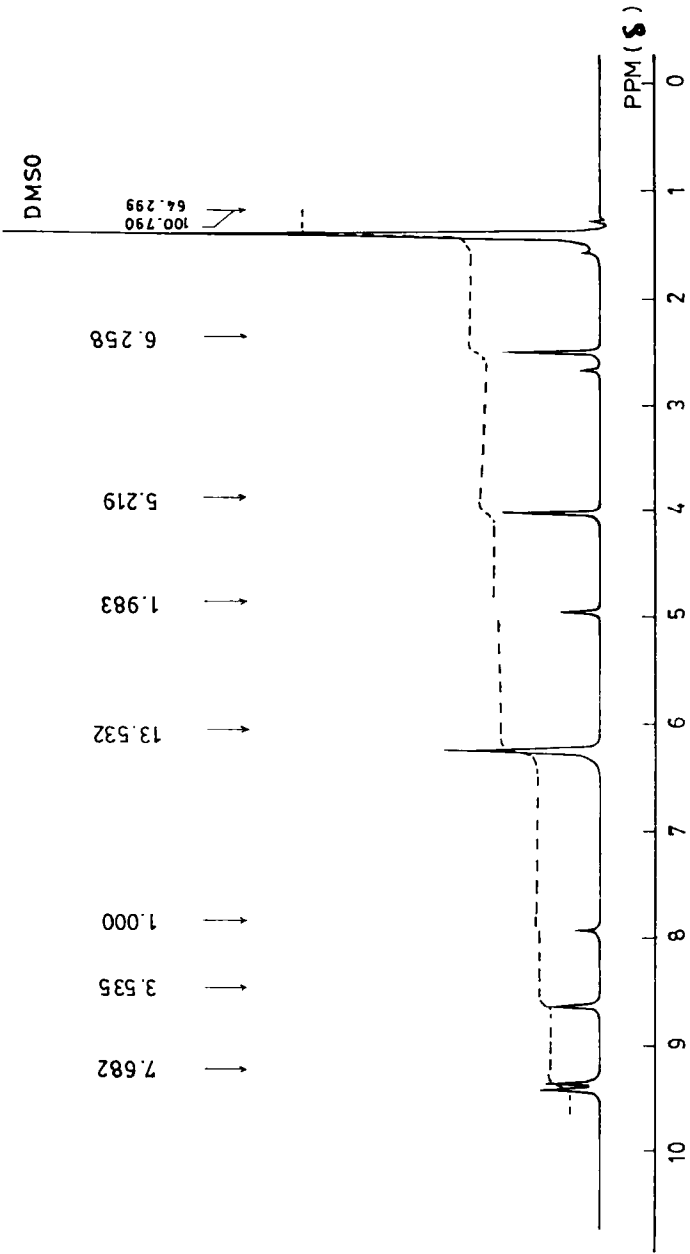


Fig. (2) ¹H NMR (DMSO) of the investigated Complex

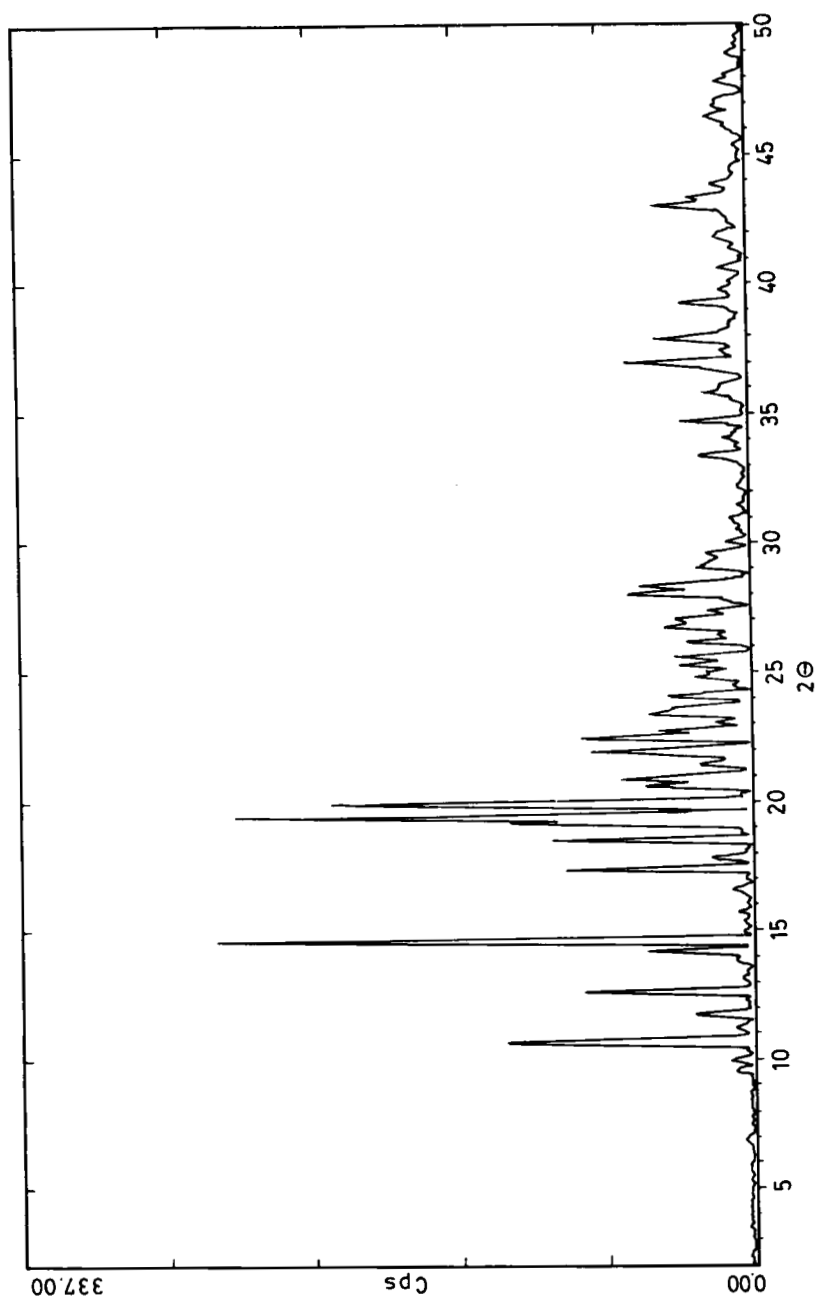
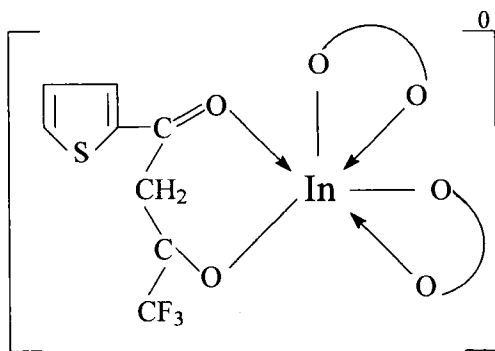
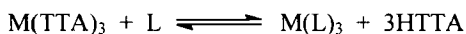


Fig. (3) X-ray diffraction powder pattern of In (TTA)₃

The light red compound of In^{3+} -TTA was characterized by conventional chemical and physical methods of analysis. The ethanolic solution of the complex exhibits two absorption maxima at 348nm and 281 nm Fig (1) attributed to d-d transition and charge transfer transition respectively. Relevant IR band for free ligand which provide structural evidence for the mode of attachment of the ligand to Indium is 1654 cm^{-1} $\nu(\text{C}=\text{O})$. This band was observed at higher frequency (1675 cm^{-1}) in the spectrum of the complex indicating that the coordination has been done via oxygen atoms. The weak band at 457 cm^{-1} can be assigned to In-O stretching. These results were supported by ^1H NMR spectrum of the investigated complex (Fig 2) which indicate that only one proton was involved in the coordination and the ligand act as a bidentate OO donor. The microanalytical data of the compound correspond to the chemical formula $\text{In}(\text{TTA})_3(\text{H}_2\text{O})_2$. X-ray powder diffraction pattern of the investigated complex is shown in (Fig 3). A trial to prepare a single crystals for X-ray crystals structure studies was unsuccessful. On the bases of the foregoing discussion the following structure is plausible in which In^{3+} has coordination number 6.



For preparation of mixed ligand complexes of Indium starting with $\text{In}(\text{TTA})_3$ as precursor and using thioacetamide, thiourea, L-glutamic acid as co-ligands were unsuccessful and non-synergistic reaction has been occurred according to the following equation



where L is the co-ligand involved in the reactions.

In conclusion; Indium in oxidation state (+3) forms neutral complex with thenoyl trifluoroacetone which has the chemical formula $\text{In}(\text{TTA})_3$. The complex is soluble in ethanol and could be of potential use in clinical studies.

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