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Synthesis and characterization of a new cobalt complex: Tetramethylammonium difluorochlorocobaltate(II)

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ABSTRACT

A cobalt complex with the tetramethylammonium cation was synthesized and identified. This complex was prepared by reaction of tetramethylammonium chloride with CoF_2 in acetonitrile. Characterization of this complex was made by microanalyses, FT-IR, IR and UV-Visible spectroscopies. The IR spectra reveal the coordination from halides to central cobalt atom and intermolecular hydrogen bonding.

Keywords: Tetramethylammonium halide, Complex Synthesis, difluorochloroCobaltate(II).

INTRODUCTION

In recent years there has been a great deal of interest in the metal halo compounds. This is because of the important prerequisites for a fluorinating agents to be useful is its mildness, versatility, selectivity and operational simplicity. Considering this, development of fluorinating agents based upon higher-valent metal halo derivatives specially transition metals has been the objective of many research laboratories and a host of such reagents derived from transition metal. While halo metals of type MX_2F ($\text{X} = \text{Cl}$) having isolated tetrahedral anions, have been known for some time and are readily accessible by reaction in solution [1-3]. the corresponding cobalt is not found in analogous manner. We had prepared and reported synthesis of a number of $[\text{MoO}_3\text{F}]^-$ anion with tetramethylammonium counter ion previously [4-5]. We have managed to prepare a new fluoro-compound of cobalt that is the analog of the above transition metal compound. trifluorocobaltate(II) have not been synthesized and reported so far. In this paper a direct, simple and one-step method has been used to synthesize this compound. This was two primary incentives for selection of $(\text{CH}_3)_4\text{N}^+$ as the counter ion. Firstly, quaternary ions such as tetramethylammonium are often used as phase transfer catalyst. Secondly, quaternary ions such as tetramethylammonium are used as crystal growing agents.

MATERIALS AND METHODS

(0.23g, 2.37mmol) CoF_2 was taken to the container including a magnet and solved in acetonitrile. The whole solution was mixed at the room temperature for 2 h. Finally the pink compound was precipitated, separated and washed with hexane and diethyl ether. Melting point of this complex is $132.2\text{-}137.2^\circ\text{C}$. IR absorptions (cm^{-1} KBr), $[(\text{CH}_3)_4\text{N}]^+$: 3449(m, br), 3225(w, br), 3015(sh), 2957 (w.br), 2914(w.br), 2784(w.br) 2622(w), 1803(w.br), 1488(ms), 1446(s), 1295(m), 950(ms), 499(w.br) 428(w.br). $[\text{CoF}_2\text{Cl}]^-$: 748(w), 616(w), 350(m).

RESULTS AND DISCUSSION

Preparation for ligand, and This complex reaction of tetra methyl ammonium chloride with CoF_2 in acetonitrile. This complex is quite stable and could be stored without any appreciable change. This complex was characterized by several techniques such as elemental analyze FT-IR, IR,UV electronic spectra, and molar conductance measurements. The elemental analysis data suggest the stoichiometry to be 1:1 [M:L] ratio formation. This complex has sharp melting point at $132.2\text{--}137.2^\circ\text{C}$ that showed the purity of complex. This is insoluble in nonpolar solvents. It has also been proposed that concentration plays a vital role in increasing the degree of inhabitation. The complex of this ligand was obtained from a refluxing mixture of the respective ligand, and cobalt (II) taken in a 1:1:1 molar proportion in acetonitrile. The results are consistent with the proposed mono nuclear formulation. This new method is based on the concept of the reaction of the naked fluoride ion of tetramethylammonium fluoride that is produced by dissociation of $(\text{CH}_3)_4\text{NF}$ in acetonitrile. In spite of the electronic spectra of mono substituted cobaltate ion derivatives (such as CoF_2X^- , where $(\text{X} = \text{Cl})$), which have been measured and calculated, few reports have been found on the electronic spectra of halo compounds. The electronic spectrum shows a transition in acetonitrile at 246 nm, 283nm, 331nm, 383nm. The advantages of the new method are as follows: (a) there is no side product, (b) the reaction is quite fast, (c) only mild conditions are required, and (d) there is an accompanied color change, which provides a visual means for ascertaining the progress of the reaction [6].

Starting materials was obtained from Merck (Berlin, Germany) and was used without further. The UV spectra was taken by Hitachi-320 Perky Elmer Lambda-15 instrument in DMSO solution and IR spectra in the $\tilde{\nu}$ region $400\text{--}4000\text{ cm}^{-1}$ was recorded by Perkin Elmer 783 spectrophotometer by the KBr pellet technique. The conductance of the complex in DMSO solution was measured at room temperature by employing a Systronic 321 conductivity bridge.

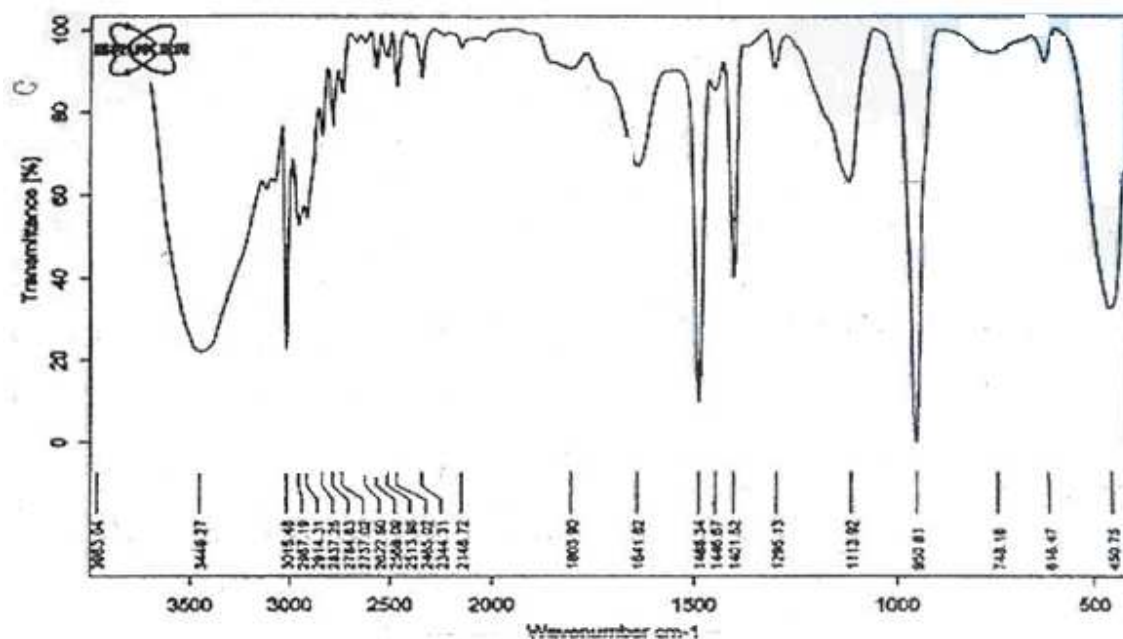


Fig.1. FTIR spectrum of $[(\text{CH}_3)_4\text{N}][\text{CoF}_2\text{Cl}]$

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