Studies of complexes of Rb and Cs metal salts of some organic acids with Bis (8-hydroxy) – 5 – quinolyl) – methane

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Abstract: A lot of complexes of alkali metals with oxine and its derivatives have been studied. The present work deals with the chelating ability of Bis (8-hydroxy-5-quinonyl) methane as a ligand. In this work mixed ligands complexes of Rb and Cs were studied having one of the ligand Bis (8-hydroxy-5-quinonyl) methane. These complexes have the general formula (ML)₂H₂L₁ where M=Rb or Cs, L = deprotonated organic acids such as ortho nitro phenol, DNP, TNP etc. H₂L₁ = Bis (8-hydroxy-5-quinonyl) methane. These complexes were characterized by their elemental analysis, magnetic moment, infrared and electronic spectra. A lot of complexes of alkalimetals were studied with organic ligands. They are highly stable. Sidwick and Brewer studied a lot of complexes of alkali metals with organic ligands having general formula ML – HL. In these complexes central atom combines with oxygen and nitrogen donar atoms of the ligands. A number of mixed ligands complexes of the composition (ML₂)H₂L₁ have been synthesized by Prakash and et-al. Where M = Li, Na or K, L = deprotonated 2,4 – dinitrophenol, 2,4,6 – trinitrophenol. H₂L₁ = Bis (8-hydroxy – 5 – quinolyl) methane. Recently Prakash and Yadav have reported synthesis of four chelate polymers of alkaline earthmetals with Bis (8-hydroxy-5-quinonyl) methane.

Key words: I.R, Electronic spectra, molar conductivity.

I. Experimental

A. Material and methods

All the chemicals used were of AR Grade.

For preparing this ligand 14 – 15 gm. 8 – hydroxyquinoline (oxine) was dissolved with stirring in 50 ml concentrated H₂SO₄, maintaining the temperature 5°C. After that 7.2 ml formaldehyde was then added dropwise with stirring the solution over a period of three hours, after which the temperature of the solution was maintained at 0 to 5°C for 2 hours and then poured in 6 litre of distilled water maintained at room temperature. After 16 hours the solution was filtered and a bright yellow product reported as hydrogen sulphate salt of Bis (8-hydroxy-5-quinonyl) methane was obtained. The yellow precipitate was dissolved in hot distilled water and neutralized with NH₄OH to give the desired compound. On recrystallization from dimethyl formamide, a white amorphous powder was obtained that melts at 23³C.

B. Composition

(Percentage) C – 76 H – 4.70 N – 9.07

(C₃H₅NO₂) Required C – 75.4 H – 4.64 N – 9.26%

Preparation of alkali metal salts of some organic acids:

(a) Preparation of Rb and Cs metal salts of o-nitrophenol M(ONP). Equimolar proportion of metal hydroxide (i.e. RbOH and CsOH) and o-nitrophenol were refluxed in absolute alcohol in a conical flask on a water bath for about 20 – 30 minutes. The solution was filtered out, concentrated and cooled. On cooling salts of Rb and Cs were precipitated out. It was filtered washed with ethanol and dried in electric oven at 80³C. Composition of RbONP

<table>
<thead>
<tr>
<th>Found %</th>
<th>C – 32.16</th>
<th>H – 1.68</th>
<th>N – 6.18 Rb – 38.04</th>
</tr>
</thead>
<tbody>
<tr>
<td>Required (C₃H₅NO₂)Rb</td>
<td>C – 32.22</td>
<td>H – 1.79</td>
<td>N – 6.26 Rb – 38.25</td>
</tr>
<tr>
<td>(CsONP) Found</td>
<td>C – 26.49</td>
<td>H – 1.42</td>
<td>N – 5.06 Cs – 48.85</td>
</tr>
<tr>
<td>Required (C₃H₅NO₂)Cs</td>
<td>C – 26.58</td>
<td>H – 1.47</td>
<td>N – 5.17 Cs – 49.06</td>
</tr>
</tbody>
</table>

C. Process of complexation

Rb or Cs metal salt of organic acid and Bis (8-hydroxy-5-quinonyl) – methane were taken in 2 : 1 (mole) with absolute ethanol in a conical flask. The contents were heated gently with constant stirring till the solid dissolved completely. The conical flask was further heated to get concentrated solution on a sand bath. The clear solution

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was left for cooling, a characteristic coloured crystalline complex was separated out. It was filtered washed and dried in electric oven at 80°C. All the mixed ligand complexes are coloured. They are soluble in polar solvents eg – methanol and insoluble in non polar solvents eg – Benzene, ether etc. They are stable in dry air but decompose on exposure to moisture. They were kept in a dessicator over solid fused anhydrous CaCl₂.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Colour</th>
<th>M.P / Decom. / Trans. Temp</th>
<th>Molar Conductivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bis (8-hydroxy-5-quinolyl) methane (H₂L₁)</td>
<td>colourless (white)</td>
<td>280m</td>
<td>------</td>
</tr>
<tr>
<td>(RbONP)₂H₂L₁</td>
<td>yellow</td>
<td>260t</td>
<td>7.00 ohm-1 cm² mole</td>
</tr>
<tr>
<td>(CsNP)₂H₂L₁</td>
<td>yellow</td>
<td>290t</td>
<td>9.0</td>
</tr>
<tr>
<td>(RbDNP)₂H₂L₁</td>
<td>deep yellow</td>
<td>250 t</td>
<td>9.00</td>
</tr>
<tr>
<td>(CsDND)₂H₂L₁</td>
<td>deep yellow</td>
<td>150m</td>
<td>5.9</td>
</tr>
</tbody>
</table>

Conductivity is taken in methanol solution at 27°C.

II. Results and Discussion

The low values of molar conductivity indicate the covalent nature of complexes. The absorption band of principal interest in the infrared spectra of Bis (8-hydroxy-5-quinolyl) methane (H₂L₁) are 3335, 1580 and 1420 cm⁻¹, the moderately strong band at 3335 cm⁻¹ in the spectrum of H₂L₁ is attributed to the stretching – O – H vibration frequency, while the strong band at 1-420 cm⁻¹ in its spectrum is probably due to the bending – O – H frequency. The absorption band in the region 1580 cm⁻¹ has been assigned to the vibration VC = N group in the quinoline ring. It is evident that the spectrum of the ligand contains a moderately strong band at 3335 cm⁻¹, this band has virtually disappeared (or with its appreciable shifts of about 14 to 94 cm⁻¹) in the complexes, indicating that coordination with oxygen atom of OH group of ligand.

A new broad band of well to medium intensity in the region 2850 – 1950 cm⁻¹ is exhibited by some complexes. This band could be assigned 0 – 17 ---- O/N ---- H ---- 0 ---- absorption and suggests that H-bonding may be regarded to be an essential feature of these complexes. In infrared spectra of the complexes, the 1580 cm⁻¹-band of the ligand (Vc = N) has appeared as split band at 1589 –1610 cm⁻¹. The splitting and shifting of the 1580 cm⁻¹-band of the ligand suggest the coordination of N atom of the C = N group of the quinoline ring with central metal atom.

After complexation, shifting of 1420 cm⁻¹ bending – O – H band of the ligand (H₂L₁) by 10 – 12 cm⁻¹ indicating the chelation of oxygen atom of the – OH group of the ligand. The band in the region 637 – 550 cm⁻¹ in the spectra of all mixed ligand complexes may be assigned to M – 0 band frequency while medium bands in the region 546 – 503 cm⁻¹ is assigned to M – N band frequency. Electronic absorption bands of the complexes are observed in the region 206 – 306 nm and 316 – 360 nm which indicates the π – π* transition and charge transfer.

References