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Physico-chemical analysis on SN (ii) and PB (ii) complexes

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Abstract

In this paper the current examination was under taken to with a view analyze the conceivable complex arrangement of Sn(II) and Pb(II) metal salts of O–Nitrophenol, 2-4 dinitrophenol, 1-Nitroso-2-napthol and Hydroxy quinoline with thiosalicylic corrosive utilizing total ethanol as dissolvable. Coordination locales in our subsequent ligand are Oxygen and Sulfur particles and go about as a bidentate twofold confronted ligand. The main aim in the present investigation the preparation of Ligands, methods of metals (Sn and Pb) as well as theory of IR Spectro & copy.

Keywords: examination, complexes, theory, arrangement

Introduction

After 1925, there were many evidences regarding the complex formation of s-block alkali and alkaline earth metals despite the absence of d- orbitals in them. The above facts enabled us to investigate the complexing abilities of p-block metals. We are especially interested with the preparation and characterisation of mixed ligand complexes of tin and lead metals in lower oxidation state e.g +2 state.

The present investigation was under taken to with a view examine the possible complex formation of Sn(II) and Pb(II) metal salts of O–Nitrophenol, 2-4 dinitrophenol, 1-Nitroso-2-napthol & Hydroxy quinoline with thiosalicylic acid using absolute ethanol as solvent. Coordination sites in our second ligand are Oxygen and Sulphur atoms and act as a bidentate double faced ligand.

Material and Method

The ligand 8-mercapto quinoline (8HSQ) used in the complex formation has been synthesized by the method given below- Quinoline (50 gm) was added over 10 minutes to 40 gm sulphuric acid and the solution slowly poured into 200gm oleum with stirring below 100°C. The mixture was heated at 160°C for 4 hours, cooled and poured into 1 kg ice to precipitate quinoline 8- suplhuric acid. It was washed and dried. The product was treated in a porcelin cup with 20% caustic soda in small portions. The solution was evaporated and dried at 160°C for 8-10 hours to yield the sodium salt of quinoline -8- sulphuric acid. The salt was powdered and mixed with equal amount of powdered phosphorous pentachloride. The mixture heated in an oil bath at 80–100^{0}C. unitl melted and then at 140-145^{0}C unitil the liquid metal did not tend to solidify and longer. It look about 40-60 minutes. The melt was rapidly poured into a mortar and was ground as soon as the melt began to set water -8- sulphonly chloride further ground under water to leach out NaCl and $POCl_3$. Quinoline -8- sulphonyl chloride was dissolved in 250 ml concentrated hydrochloric acid and the mixture poured into a $SnCl_2$, solution to give the stannous salt of 8 - mercapto quinoline. It was decomposed by making a thin paste with water adding 926 gm sodium tartarate and 55.6 gm caustic soda. Strong evolution of heat and change in colour was observed. Final decomposition was obtained by boiling the mixture, cooling and filtering to give the sodium salt of 8 - mercapto quinoline.

Sodium salt of 8 - mercapto quinoline in one litre of water was neutralized with HCL to pH 5.5 -6.0. The bright red dihydrate of 8- mercapto quinoline began to precipitate at pH 8. After standing over night the precipitate at pH 8. After standing over night the precipitate of dihydrate was separated from tin and other metals by dissolving it in concentrated ammonia solution and filtering. The filtrate was neutralised with HCL to precipitate the dihydrate of 8 - mercapto quinoline. It was filtered, washed with water and dried in an vacuum desiccator. The authenticity of the compound was confirming by its elemental analysis, melting point determination and infrared spectroscopy. The compound was recrystallised in alcohol. The yield of the product was about 70% and melted at 58% C.

Metal Chelates of Sn (H) and Pb (II) chelates of o-nitrophenol (ONP)

95% alcoholic solution of n-nitrophenol and suspension of lead acetate or stannous chloride in 95% ethanol were mixed together in 2:1 prportion. The mixture was refluxed on magnetic hot plate with constant stirring for 30 -45 minutes. Aclear solution was obtained which was made alkaline with ammonium hydroxide Deep orange and brown coloured chelates of Lead and Tin respectively were separated. It was filtered washed ethanol and finally dried in an electric oven and 100^{0}C.

Sn $(ONP)_2$

Found: Cr= 36.50; H =2.05; N=7.15; Sn=30.21%
Calcultaed: C=36.413; H=2.02; N =7.09; Sn=30.07%

Pb $(ONP)_2$

Found: C=29.90; h = 1.70; N=5.85; Pb =42.92%
Calcultaed: C=29.81; H = 1.65; N = 5.79; Pb 42.85 %

Preparation of Sn (11) and Pb (II) chelates of 2,4 dinitro phenol (DNP)

To the suspension of lead acetate or stannous chloride in 95% Ethanol., 2-4 dinitro phenol was added in 1:2 mole ratio. The resulting mixture was refluxed on magnetic hot plate for 20-30 minutes. A clear solution was obtained in which ammonium hydroxide was added to make the solution alkaline. Light orange and brown coloured precipitate of Lead and Tin adducts respectively were obtained which were filtered, washed with ethanol and finally dried in an electric oven at 100°C.

$Sn (DNP)_2$

Found: C= 2935; H= 1.28; N= 11.60; Sn= 24.55%

Calculated: C= 29.70; H = 123; N =11.55; Sn=24.49%

$PB (DNP)_2$

Found: C=25.18; H= 1.08; N=9.79; Pb = 3620%

Calculated: C=25.13; 11=1.05; N=9.77; Pb=36.12%

To the suspension of lead acetate or stannous chioride in 95% ethanol. The solution of 1-nitroso-2-napthol was added in 1:2 proportion. The resulting, mixture was refluxed on the magnetic hot plate with contant stirring of ammonium hydroxide to the solution. Brown and ash coloured precipitates of lead and Tin Chelates respectively were obtained. It was filtered, washed with ethanol and finally dried in an electric oven at 100^0C.

$SN (8HQ)_2$

Found: C=53.16; H =2.99; N=6:92; Sn = 29.24%

Calculated: C=53.11; H=2.95; N=6.88; Sn=29.18

$PB (8HQ)_2$

Found: C=43.68; H=2.46; N=5.66; Pb=48.90%

Calculated: C=43.63; H=2.42; N=5.65; Pb=41.82%

Method of Analysis

Estimation of Metals: Lead The complex was decomposed by above method 60 ml Pb^2 ion solution was treated with dilute solution of sulphuric acid (1 percent). The precipitate of Lead sulphate obtained was washed with rectified spirit and dried in oven until a contant weight is obtained.

Since 303.25gm $Pb\ 5O_4$. containes 207.19 gm Pb.1gm of Pb SO_4 contains 207.19/ 303.25gm Pb=0.6829 gm Pb.

Estimation of Non Metals - Carbon, Hydrogen & nitrogen

Results were obtained from micro-analytical laboratory, Regional Spphisticated Instrumentation Centre, C.DR.I. Lucknow.

Physical Measurement

Melting Points determined on electrical Tempo T -1150 melting point apparatus. The infrared spectral results were obtained from Chemical Laboratory, CDRI Lucknow. It was recorded on Perkin - Elmer model No. 577 spectrophotometer in KBr disc.

The Theory of Infrared Spectroscopy

In order to understand the theory of IR absorption spectroscopy, one has to understand the phenomenon of vibrational rotational spectra. Let us consider a diatomic molecule associated with a dipole moment.

The vibratory motion of the nuclei of diatomic molecule may be similar to the vibration of a linear harmonic oscillator. In such a oscillator, the force tending to restore an atom to its original state is proportional to the displacement of the vibratory S- atom from the original position (Hooke's Law). Suppose the bond between the two nuclei of a diatomic molecule is distorted from its equilibrium length r_e to a new length r. Then the restoring forces on each atom of the diatomic molecule will be given by -

$$m_1\ d^2r_1 / dt^2 = - K (r - r_e) \qquad (1)$$

$$m_1\ d^2r_2 /dt^2 = K (r-r_e). \qquad (2)$$

Where K is the propotionality contant and known as force contant; it is registered as a measure of the stiffness of the bond; r_1 and r_2. are the position of atoms 1 and 2 relative to the centre of the gravity of the molecule. We know

$$r_1=m_2r/m_1 +m_2 \qquad (3)$$

$$\text{and } r_2=m_2r/m_1+m_2 \qquad (4)$$

Where m_1 and m_2 are the masses of two atoms a vibrating diatomic molecule. On substituting equation (3) in (1) we get

$$(m_1m_2/m_1+m_2)\ d^2r/dt^2=-K(r-r_e) \qquad (5)$$

As re is a contant, its differentiation with respect to time t will be zero, i.e

$$d^2r/dt^2=d2\ (r–r_e)/dt^2 \qquad (6)$$

Substitution of the above equation in equation (5) yields

$$(m_1m_2/m_1+m_2)\ d_2(r–re)/dt_2=-K(r-r_e) \qquad (7)$$

$$\text{Put } r–r_e = X \text{ and } m_1m_2/m_1+m_2 = u \qquad (8)$$

On substituting equation (8) in (7) we get

$$ud^2x/dt^2= – Kx \qquad (9)$$

$$\text{or } d^2x/dt^2 + kx/u=0 \qquad (10)$$

$$\text{or } d^2x/dt^2 + W^2x=0 \qquad (11)$$

$$\text{Where } W^2 = K/u \text{ or } W= (K/u)^{1/2} \qquad (12)$$

Equation (10) is the expression of a simple harmonic motion with frequency of vibration (v) of follows:

$$V=w/2\ (22/7). = (1 /2)\ (22 /7)\ (K/u)^{1/2} \qquad (13)$$

Where K is a force constant expressed in dynes per cm and u is the reduced mass of the system (Eq.8)

Results and Discussion

We have synthesized and characterized a number of mixed ligand complexes of Sn (II) and Pb (II) metal salts of O-nitrophenol (ONP), 2,4- dinitrophenol (DNP), 1-n itroso-2-naphthol (1N2N) and 8-hydroxy quinoline (8HQ) with thiosalicylic acid. They have the general formula ML_2HL, where M= Sn (II) or Pb (II), L= deprotonated ONP, DNP, 1N2N or 8HQ, Thiosyl acid.

Conclusion

Our usual method of synthesizing these adducts to take asuspension of the Sn (II) or Pb(II) metal salt (ML_2) in absolute alcohol in a conical flask and then to add second ligand (HL) in a ratio of 2: 1, then stirring the mixture till all the solids went into the solution. The content were warmed for about two hours, but in few cases the complexation completed within an hour. On cooling the resulting solution, the adduct separated which was filtered, washed with absolute alcohol and dried in an electric in an electric oven at 80°C.

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