NEPHELOMETRIC STUDY OF KINETICS OF INORGANICCOMPOUND INAQUEOUSSOLUTION

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Abstract:

The present study based on the kinetic measurements of precipitation reactions of Nickel Sulphat,and Oxalic acid. In this study nephelometer is used for studying precipitation reactions. Study shows variation in precipitate formation with time. The effect of concentration on the rate of reaction were investigated, it is observed that reaction follows first order kinetics. Precipitate formation of Nickel sulphat and Oxalic acid is carried out at different temperatures, experiment were also performed to study effect of salt on precipitation. The thermodynamic parameters $\Delta H, \Delta S, \Delta G$,Ea were calculated using Arrhenius equation.

Key Words: Nickel Sulphate,Oxalic Acid, Precipitation reaction, nephelometer, Kinetics

Introduction

The solubility of an ionic salt in pure water depends on the solubility product constant alone, provided that only cations and anions of salts are in solution.[1] one of the oldest methods of chemical separation is fractional precipitation.[2] this involves the addition of a reagent which precipitates most of metal ion and leaves another mostly in solution. Many ions which form insoluble salts can be determined by titration, if a suitable method of estimating the equivalence point is available.[3]the most convenient method is potentiometric, but chemical indicators are available for a number of cases

We have given the combined application of various methods of investigation[4] (e.g. nephelometry, conductivity measurements, electron microscopy, isotope exchange, paper

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chromatography, co-precipitation etc) lead to refined insightinto the course of precipitation reaction

The precipitation reaction for Nickelsulphat, was previously carried outusing nephelometry[5]. It measures the scattering of light radiation by dispersed phase.
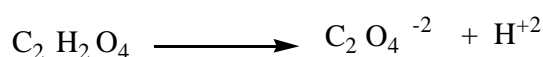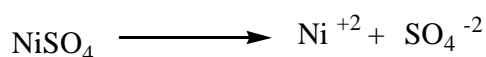
Experimental:

The chemicals used for the present investigations were of S.D.Fine Chemicals Ltd and used without further purification. Double distilled water was used as solvent. A (CL 52D) Elico make nephelometer was calibrated using farmazine solution.,which was prepared by using Hydrazine Sulphate and Hexamethylentetra amine .equal volume of these solution were mixed and and kept for 48 hours, this solution was used for calibration of nephelometre . Effect of variation of concentration of concentration of nickel sulphate, effect of radiation and effect of temperature, effect of salt was studied. A known amount of nickel sulphate was mixed with known concentration of Oxalic acid and the appearance of turbidity was measured by using using nephelometer.

Results and Discussion:

Part-I   **Reaction between Nickel Sulphate and $C_2 H_2 O_4$**

For the present study, we have taken a definite amount of solid Nickel sulphate , which is treated with Oxalic acid solution. The solution becomes turbid, due to the formation of Nickel Oxalate. The reaction takes sufficient times; therefore it can be successfully studied by using nephelometry.

It is possible that $NiSO_4$get dissociate in solution

$$NiSO_4 \longrightarrow Ni^{+2} + SO_4^{-2}$$

$$C_2 H_2 O_4 \longrightarrow C_2 O_4^{-2} + H^{+2}$$

$$Ni^{+2} + C_2 O_4^{-2} \longrightarrow NiC_2 O_4$$

In the present investigation, we monitored the turbidity obtained due to product formation. Although reaction order is different, we maintained pseudo first order conditions. So the different order reactions exhibit different functional forms for the time dependence of reactant concentrations.

It was observed that NTU reading is slow initially for few seconds thanincreases up to 2 to 3 min and then slightly decreases for two to three readings and smoothly decreases. for lower concentration of Nickel Sulphate. The curve shows sudden increase in NTU reading for 1 to 2min then slightly towards constant than smooth decrease in reading is shown for higher concentration of Nickel Sulphate . When 500 mg of Nickel Sulphate was dissolved in Oxalic Acid. A smooth curve was observed

NiSO4 Vs N C2H2O4 0(O.A)
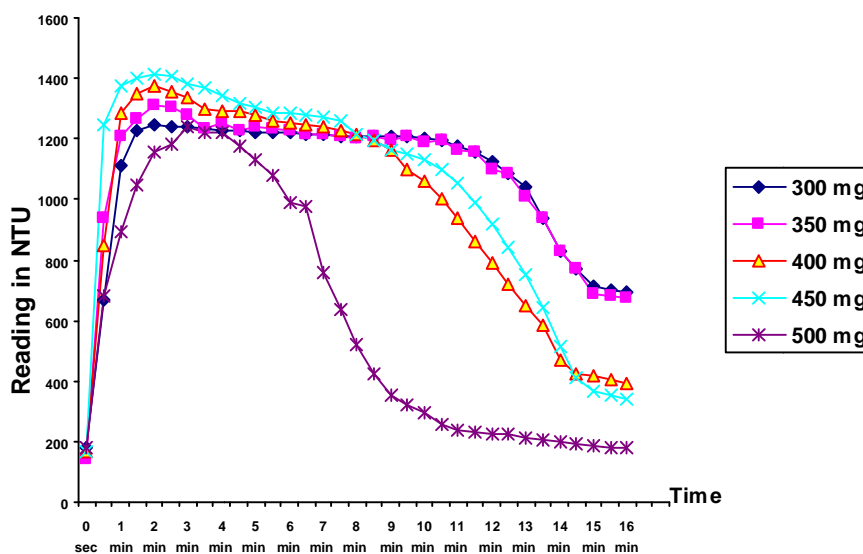
Rate constants were determined (Table 1). It is observed that with the increasing the concentration of Nickel sulphate the rate constant increases but decreasing the concentration Oxalic acid increases the rate constant. The data suggests that when the concentration of Oxalic acid and Nickel Sulphate are in the same range 10^{-2} M. rate constant is Maximum.

Table No.1 Variation of rate constant (k Sec-1) with [NiSO$_4$]

[H$_2$C$_2$O$_4$] (M)	[NiSO$_4$] 19.38x 10^{-2}M k Sec^{-1}	[NiSO$_4$] 38.76 x 10^{-2}M kSec^{-1}	[NiSO$_4$] 58.14 x 10^{-2}M k Sec^{-1}
0.1	9.5 x 10^{-3}	12.3 x 10^{-3}	22.4 x 10^{-3}
0.05	7.3 x 10^{-3}	12.8 x 10^{-3}	12.0 x 10^{-3}
0.025	5.8x 10^{-3}	8.9 x 10^{-3}	10.1 x 10^{-3}

To check the effect of added salt on precipitation, we added KCl, The result reveals that at [C$_2$H$_2$O$_4$] = 0.1 M and [NiSO$_4$] =38.76 x 10^{-2}M, 58.14 x 10^{-2} M and 77.52. It hasbeen observed that there is   increase in rate constant with increasing concentration of KCl is observed.

Variation in rate constant with KCl is shown in (Table No. 2)

Table No. 2 Effect of Salt [KCl] On Rate Constant (k sec-1).

[KCl] (M)	[C$_2$H$_2$O$_4$] = 0.1 M [NiSO$_4$] 38.76 x 10^{-2}M	[C$_2$H$_2$O$_4$] = 0.1 M [NiSO$_4$] 58.14 x 10^{-2}M	[C$_2$H$_2$O$_4$] = 0.1 M [NiSO$_4$] 77.52 x 10^{-2}M
0.0	12.3 x 10^{-2}	24.4 x 10^{-2}	20.2 x 10^{-2}
0.1	1.22x 10^{-2}	9.1 x 10^{-3}	3.2 x 10^{-3}
0.2	8.2 x 10^{-3}	11.2 x 10^{-3}	10.12 x 10^{-3}
0.3	8.3 x 10^{-3}	12.4 x 10^{-3}	11.19 x 10^{-2}

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Table No. 2 Effect of Tempreture On Rate Constant (k sec-1).

Temp	$[C_2H_2O_4] = 0.1$ M [NiSO$_4$] 19.38×10^{-2}M	$[C_2H_2O_4] = 0.1$ M [NiSO$_4$] 38.76×10^{-2}M	$[C_2H_2O_4] = 0.1$ M [NiSO$_4$] 58.14×10^{-2}M
293^0 k	3.5×10^{-3}	3.91×10^{-3}	10.2×10^{-3}
299^0 k	2.6×10^{-3}	4.9×10^{-3}	12.5×10^{-3}
303^0 k	10.5×10^{-3}	13.5×10^{-3}	20.3×10^{-3}

From the values it has been observed that there is increase in rate constant with the increase in temperature.

From the values of rate constant activation energy of reaction were calculated by using Arrhenius equation i.e. $\qquad k = Ae^{-E/RT}$

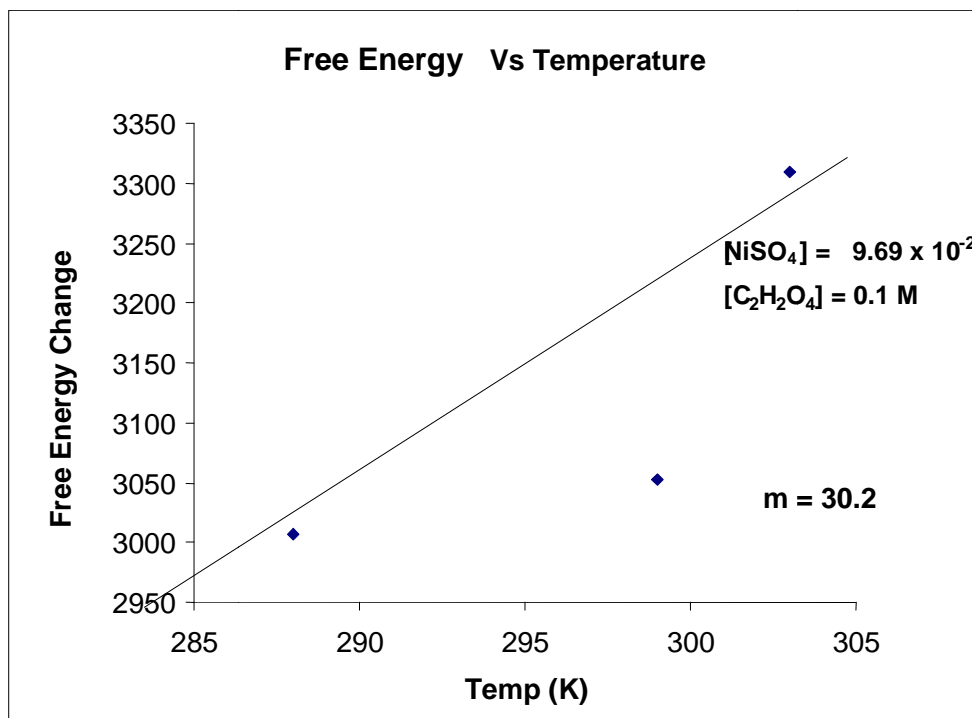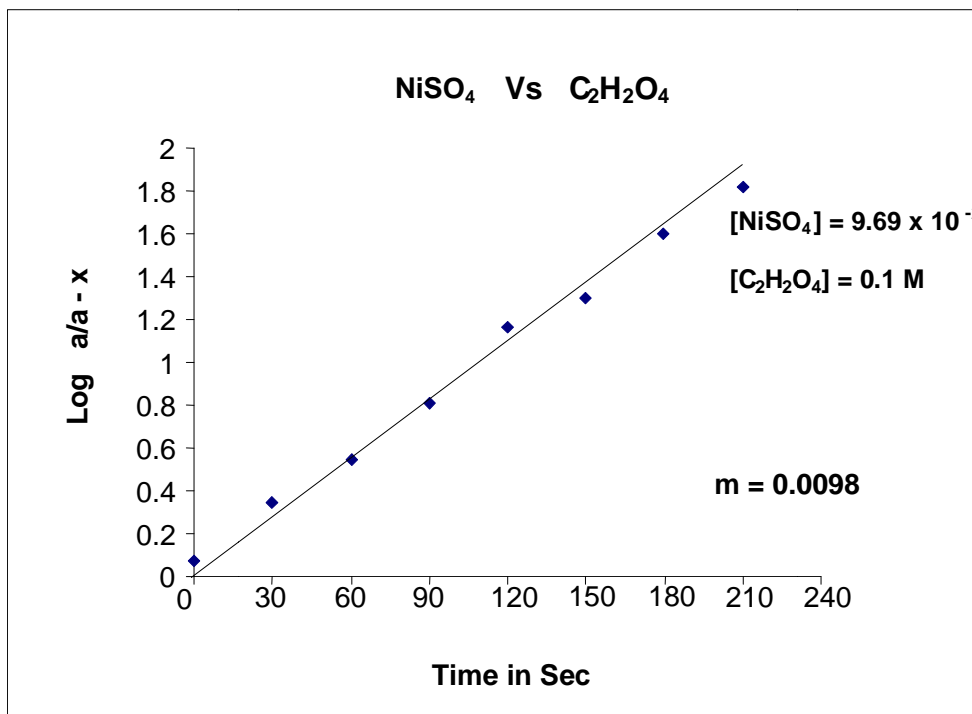Free energy, ΔG* is calculated by using equation

$$\Delta G^* = -RT\ln K$$

Free energy values at different temperature were calculated and graph of ΔG* Vs Temperature was plotted which gives straight line, slope of this line is used to calculate ΔS* values i.e. entropy change, intercept of this line gives values of ΔH*.

Ea* = 34.82×10^2 KJ mol^{-1} $\qquad$ ΔG* = 30.51×10^2 KJ mol^{-1}

ΔH* = 29.75×10^2 KJ mol^{-1} $\qquad$ ΔS* = -30.2 KJ mol^{-1}

$NiSO_4$ Vs $C_2H_2O_4$

$[NiSO_4] = 9.69 \times 10^{-2}$

$[C_2H_2O_4] = 0.1$ M

m = 0.0098

Free Energy Vs Temperature

$[NiSO_4] = 9.69 \times 10^{-2}$

$[C_2H_2O_4] = 0.1$ M

m = 30.2

$\Delta S = 30.2$ KJmol-1 $\Delta H = 29.75 \times 10^{2}$ KJmol-1

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