



**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH  
TECHNOLOGY**

**Association Thermodynamic Parameters (Conductometrically) for Nano Cobalt Sulfate in  
Mixed EtOH–H<sub>2</sub>O Solvents at Different Temperatures**

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**Abstracts**

The molar conductance for nano cobalt sulfate (CoSO<sub>4</sub>) in different percentages of ethanol (EtOH) and water were measured at 298.15, 303.15, 308.15 and 313.15K. From the molar conductance for nano CoSO<sub>4</sub>, the solvation parameters like, activity coefficient, association constant, free energy of association, enthalpy of association and entropy of association were estimated. All these solvation parameters were discussed.

**Keywords:** Thermodynamics, molar conductance, nano cobalt sulfate, free energy, enthalpy, entropy of association, mixed EtOH – H<sub>2</sub>O solvents.

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**Introduction**

Cobalt sulfate hepta hydrate is essential for most higher forms of life, but more than a few milligrams each day is harmful. Rarely have poisoning resulted from cobalt compound. Upon inhalation of salts, there is some evidence for carcinogenicity. Cobalt sulfate is used in storage batteries and electroplating baths, sympathetic inks, and as an additive to soils and animal feeds. For these purposes, cobalt sulfate is produced by treating cobalt oxides with sulfuric acid[1]. Our purpose is to try to estimate different concentrations of nano copper sulfate to get rid from body and environment and to view the different thermodynamics for nano cobalt sulfate.

**Experimental**

**Materials**

CoSO<sub>4</sub> from Al Nasr chemicals Co. was used without purification. Ethanol of the type Adwic was used directly without purification, Double-distilled water was used throughout this study.

**Preparation of nano CoSO<sub>4</sub>**

CoSO<sub>4</sub> of the type Adwic was milled by ball - mill. The ball – mill was a retsch MM2000 swing mill with 10 cm<sup>3</sup> stainless steel, double – walled tube. Two stainless steel balls of 12 mm diameter and 7 gm weight for each were used. Ball-milling was performed at 20225 Hz for half an hour at room temperature (without circulating liquid and the temperature did not rise above 30°C).

**Results and discussion**

**X-ray diffraction**

The X-ray diffraction of nano cobalt sulfate in Fig. (1) shows that it has about 100% of the structure is CoSO<sub>4</sub>. The axial ratio of a: b: c is 2.163: 1.000: 1.683. The crystal system is monoclinic, the cell dimensions are: a = 14.04, b = 0.46, c = 10.03. The forms are (0 0 1) (1 1 0). The cleavage is {0 0 1} perfect, {1 1 0} indistinct. P compared to Vedic-CMOS.

Position	Area	Crystal Size L(nm)	Microstrain	RMS Strain(%)
19.94101	1.196818	53	0.1	0.1
20.30598	1.687939	30.9	0.1	0.1
21.77233	0.9821675	45.6	0.1	0.1
22.17584	0.7086378	51.6	0.1	0.1
23.35417	11.22402	3	0.1	0.1
24.95448	1.506016	40.8	0.1	0.1
25.83244	1.090416	32.2	0.1	0.1
27.79621	0.4827081	63.5	0.1	0.1
29.28784	8.172743	3	0.1	0.1
30.24901	0.7089157	75.3	0.1	0.1
30.72258	0.5178185	89.8	0.1	0.1
31.50839	0.6151297	76.8	0.1	0.1
32.53265	7.899448	3	0.1	0.1

Table (1): The crystal size of nano cobalt sulfate.

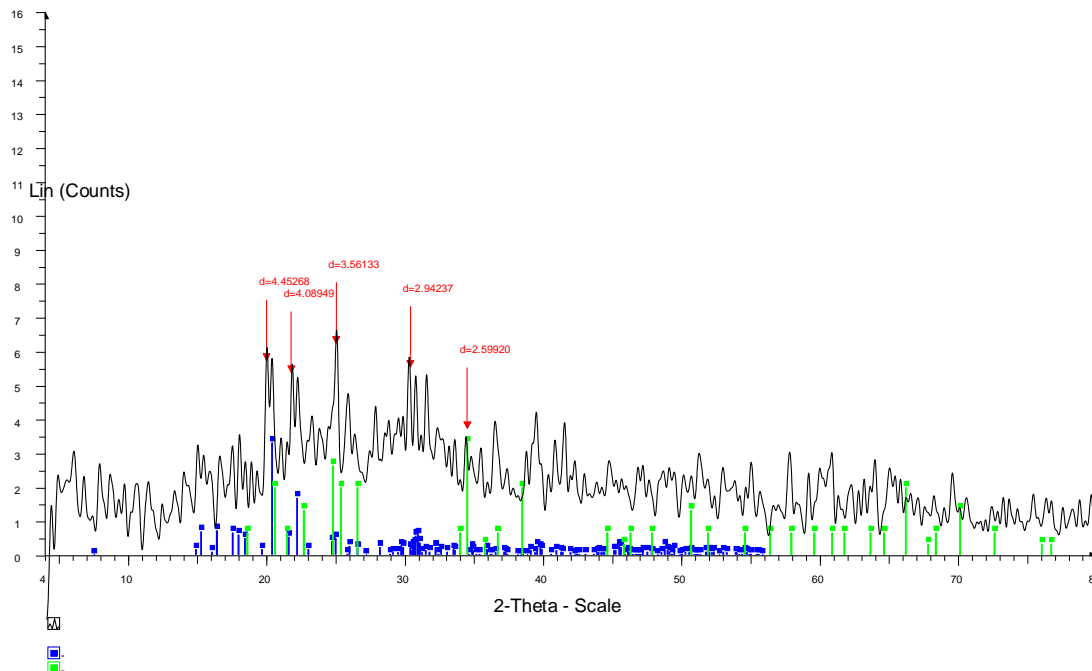
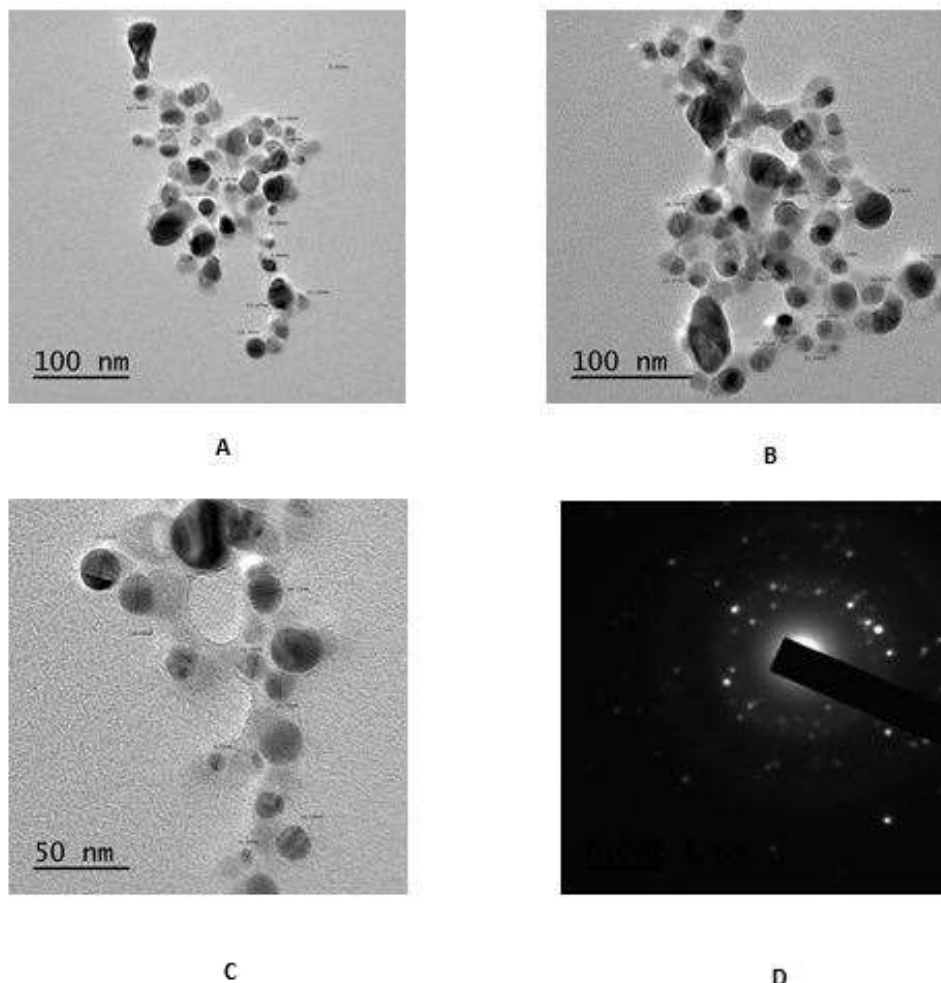


Fig. (1) X-ray diffraction of nano cobalt sulfate

**TEM Images**

All images measured by using JEOL HRTEM – JEM 2100 (JAPAN) show that TEM of  $\text{CoSO}_4$  obtained in ethanol are irregular spheres in the form of cylinders Fig. (2). The diameter in the range of 3 - 89 nm. The small sizes in the range between 3, 40 to 45 nm are collected to give bigger sizes till 89 nm. These different sizes were proved also by x- ray diffraction which gave crystal sizes in the same order .The non homogeneity in sizes for nano cobalt sulfate need controlling during the primary preparation of the samples.



*Fig.2(a-d) : TEM for nano cobalt sulfate*

**Conductometric measurements**

In 5 ml of the  $\text{CoSO}_4$  solution ( $1.0 \times 10^{-3}$  M) was placed in the titration cell, thermostatic at the preset temperature and the conductance of the solution was measured after the solution reached thermal equilibrium. Then, a known amount of solvent was added in a stepwise manner using a calibrated micropipette. The conductance of the solution was measured after each addition until the desired constant reading was achieved. The specific conductance values were recorded using conductivity bridge HANNA, HI 8819N with a cell constant equal to 1. The conductometer was conducted with a thermostat of the type the Kottermann 4130 ultra thermostat. The temperature was adjusted at 298.15, 303.15 and 308.15 and 313.15 K.

**Association and triple ion association constants:**

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The molar conductance ( $\Lambda_m$ ) values were calculated using equation (1) [2-21]:

$$\Lambda_m = \frac{(K_s - K_{solv})K_{cell} \times 1000}{C} \quad (1)$$

Where  $K_s$  and  $K_{solv}$  are the specific conductance of the solution and the solvent, respectively ;  $K_{cell}$  is the cell constant and  $C$  is the molar concentration of the  $CuSO_4$  solution.

By drawing the relation between molar conductance ( $\Lambda_{O,m}$ ) and  $\sqrt{C_m}$ , straight lines were obtained from which we extrapolate the line to zero salt concentration to evaluate the limiting equivalent conductance.

The association constant  $K_A$  was calculated by using equation (2). The experimental data for conductance measurements were analyzed using Fuoss-Shedlovsky extrapolation techniques which follows equation [22-39]: -

$$\frac{I}{\Lambda_m S(Z)} = \frac{I}{\Lambda_o} + \left( \frac{K_A}{\Lambda_o^2} \right) (C \Lambda_m \gamma_{\pm}^2 S(Z)) \quad \dots\dots\dots (2)$$

$$\text{Where } S(Z) = 1 + Z + Z^2/2 + Z^3/8 + \dots\dots\dots \text{ etc.} \quad \dots\dots\dots(3)$$

$$\text{And } Z = \frac{S(\Lambda_m C)^{1/2}}{\Lambda_o^{3/2}} \quad \dots\dots\dots(4)$$

The value of ( $\Lambda_o$ ) was used to calculate the Onsager slope ( $S$ ) from the equation:

$$S = a\Lambda_o + b \quad \dots\dots\dots (5)$$

$$\text{Where } a = 8.2 \times 10^5 / (\epsilon T)^{3/2} \quad \dots\dots\dots(6)$$

$$b = 82.4/\eta (\epsilon T)^{1/2} \quad \dots\dots\dots (7)$$

Where ( $\epsilon$ ) is the dielectric constant of the solvent, ( $\eta_o$ ) is the viscosity of the solvent and ( $T$ ) is the temperature. Using the values of ( $\epsilon$ ) and ( $\eta_o$ ), the value of ( $S$ ) were easily estimated. Using the data of ( $\Lambda_m$ ),  $S(Z)$  and ( $\Lambda_o$ ), the values of degree of dissociation ( $\alpha$ ) were calculated by using the following equation:

$$(\alpha) = \frac{\Lambda_m S(Z)}{\Lambda_o} \quad \dots\dots\dots(8)$$

Using these ( $\alpha$ ) and ( $\epsilon$ ) values, the mean activity coefficients ( $\gamma_{\pm}$ ) were evaluated by means of equation (9).

$$\text{And } \log \gamma_{\pm} = - \frac{Z_+ Z_- A \sqrt{I}}{I + B r^o \sqrt{I}} \quad \dots\dots\dots(9)$$

Where  $Z_+$ ,  $Z_-$  are the charges of ions in solutions  $A$ ,  $B$  are the Debye-Hückel constant.

$$A = 1.824 \times 10^6 (\epsilon T)^{-3/2} ; B = 50.29 \times 10^8 (\epsilon T)^{-1/2}$$

And ( $r^o$ ) is the solvated radius.

Using the values of association constant ( $K_A$ ), the values of the dissociation constant ( $K_D$ ) were easily calculated by means of the following equation:

$$K_D = 1/K_A \quad \dots\dots\dots (10)$$

The values of the triple ion association constant ( $K_3$ ) were calculated by using the equation (11).

$$\frac{\Lambda_m C^{1/2}}{(1 - \frac{\Lambda_m}{\Lambda_o})^{1/2}} = \frac{\Lambda_o}{(K_A)^{1/2}} + \frac{\lambda_3^o C}{K_3 (K_A)^{1/2}} (1 - \frac{\Lambda_m}{\Lambda_o}) \quad \dots\dots\dots (11)$$

Equation (11) was derived by Fuoss and using Walden approximation ( $\Lambda_o = 3 \lambda_o$ ). The values of ( $\eta_o$ ,  $\Lambda_o$ ,  $\Lambda_m$ ,  $C$ ,  $S$ ,  $Z$ ,  $S(Z)$ ,  $\gamma_{\pm}$ ,  $K_A$ ,  $K_D$ ,  $\alpha$  and  $K_3$  for the solutions of  $10^{-3}$  M concentration were calculated and are reported in Tables (1-4) for nano cobalt sulfate in mixed EtOH-H<sub>2</sub>O solvents at different temperatures.

**Thermodynamic Parameters of association Nano Cobalt Sulfate Solutions:**

The Gibbs of free energy change of association ( $\Delta G_A$ ) of different concentration of the metal salt solutions in pure MeOH and also in H<sub>2</sub>O were calculated [40-53] from the association constant ( $K_A$ ) by using equation (12) [45-53]

$$\Delta G_A = - 2.303 RT \log K_A \quad \dots\dots\dots (12)$$

Where R is the gas constant and T is the absolute temperature.

The enthalpy change of association ( $\Delta H_A$ ) for the metal salts was calculated for each type from the association constants by using Van't Hoff equation (13) [25-53]:

$$\frac{d \ln K}{dT} = \frac{\Delta H_A^0}{RT^2} \quad \dots\dots\dots (13)$$

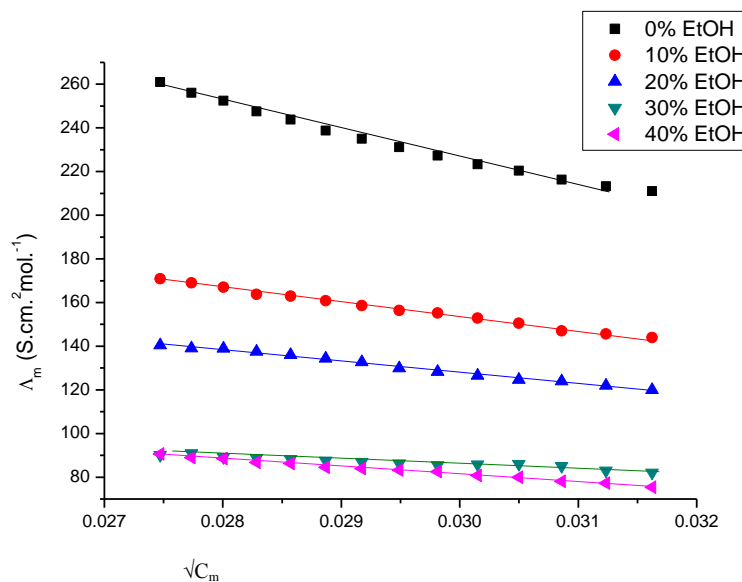
By drawing the relation between  $\log K_A$  and  $1/T$  giving straight line with slope  $(-\Delta H_A/2.303R)$  as shown in Fig.( 5,6).

The entropies of association ( $\Delta S_A$ ) for the electrolytes were calculated by the use of Gibbs-Helmholtz equation (14).

$$\Delta G_A = \Delta H_A - T\Delta S_A \quad \dots\dots\dots (14)$$

The calculated values of ( $\Delta H_A$ ) and ( $\Delta S_A$ ) for the metal salts are presented in Table (6).

The limiting equivalent conductance ( $\Lambda_o$ ) increase as the temperature increased and dissociation degree decrease and then increase as the temperature increased indicating higher solvation process.



**Fig. (3): The relation between molar conductance ( $\Lambda_m$ ) and  $\sqrt{C_m}$  of nano CoSO<sub>4</sub> in ethanol-H<sub>2</sub>O at 298.15 K.**

Table (2): The values of viscosity ( $\eta_o$ ), limiting molar conductance ( $\Lambda_o$ ), molar conductance ( $\Lambda_m$ ), Walden product ( $\Lambda_o \eta_o$ ), fluidity ratio ( $R_x$ ), Fuoss-Shedlovsky parameters (S, Z and S(z)), activity coefficient ( $\gamma_{\pm}$ ), association constant ( $K_A$ ), dissociation constant ( $K_D$ ), degree of dissociation ( $\alpha$ ), triple ion association constant ( $K_3$ ), Gibbs free energy of association ( $\Delta G_A$ ) for nano CoSO<sub>4</sub> in ( ETOH-H<sub>2</sub>O) at 298.15 K.

VOL.% OF EtOH	$10^2 \eta_o$ (poise)	$\Lambda_o$	$\Lambda_m$	$\Lambda_o \eta_o$	S	Z	S(z)	$\gamma_{\pm}$	$K_A$	$10^3 K_D$	$\alpha$	$10^5 K_3$	$\Delta G_A$
0%	0.8921	566.64	208.3	5.054	190.717	0.0062	1.0063	0.9154	5782.70	0.173	0.3699	16.8	-21.47
10%	0.8977	350.14	147	3.143	142.424	0.0081	1.0081	0.9082	4107.71	0.243	0.4232	13.6	-20.62
20%	0.9042	288.10	123.9	2.605	129.447	0.0090	1.0091	0.9051	3860.86	0.259	0.4339	13	-20.47
30%	0.9118	161.20	84.3	1.978	107.741	0.0118	1.0149	0.8898	3823.668	0.348	0.4438	12.5	-19.61
40%	0.9209	154.79	76.5	1.4254	100.118	0.0134	1.0135	0.8784	3329.732	0.434	0.4606	11.7	-19.47

$\Lambda_o$  in (S cm<sup>2</sup>.mol<sup>-1</sup>),  $\Lambda_m$  in (S cm<sup>2</sup>.mol<sup>-1</sup>) and  $\Delta G_A$  in (kJ mol<sup>-1</sup>).

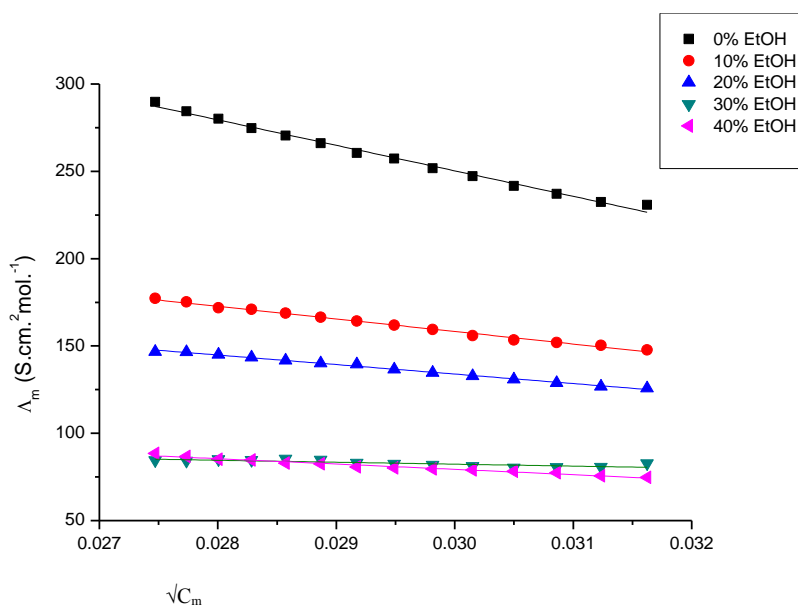


Fig. (4): The relation between molar conductance ( $\Lambda_m$ ) and  $\sqrt{C}$  of nano  $CoSO_4$  in ethanol- $H_2O$  mixed solvent at 303.15 K.

Table (3): The values of viscosity ( $\eta_o$ ), limiting molar conductance ( $\Lambda_o$ ), molar conductance ( $\Lambda_m$ ), Walden product ( $\Lambda_o \eta_o$ ), fluidity ratio ( $R_x$ ), Fuoss-Shedlovsky parameters (S, Z and S(z)), activity coefficient ( $\gamma_{\pm}$ ), association constant ( $K_A$ ), dissociation constant ( $K_D$ ), degree of dissociation ( $\alpha$ ), triple ion association constant ( $K_3$ ), Gibbs free energy of association ( $\Delta G_A$ ) for nano  $CoSO_4$  in (EtOH- $H_2O$ ) at 303.15 K.

VOL.% OF EtOH	$10^2 \eta_o$ (poise)	$\Lambda_o$	$\Lambda_m$	$\Lambda_o \eta_o$	S	Z	S(z)	$\gamma_{\pm}$	$K_A$	$10^3 K_D$	$\alpha$	$10^5 K_3$	$\Delta G_A$
0%	0.800	688.23	237.027	5.506	228.10	0.0059	1.0060	0.9170	0.0068	0.15	0.3464	18.5	-22.25
10%	0.803	374.04	151.977	3.006	156.60	0.0082	1.008	0.9085	0.0045	0.22	0.4097	14.3	-21.19
20%	0.808	306.60	128.877	2.477	142.38	0.009	1.0093	0.9049	0.0041	0.24	0.4242	13.5	-20.97
30%	0.813	154.90	80.577	1.859	106.32	0.0103	1.0114	0.8917	0.0022	0.45	0.52822	11.8	-20.44
40%	0.819	171.79	77.427	1.407	102.90	0.0136	1.0136	0.885	0.0019	0.51	0.5868	8.8	-19.50

$\Lambda_o$  in (S cm<sup>2</sup>.mol<sup>-1</sup>),  $\Lambda_m$  in (S cm<sup>2</sup>.mol<sup>-1</sup>) and  $\Delta G_A$  in (kJ mol<sup>-1</sup>).

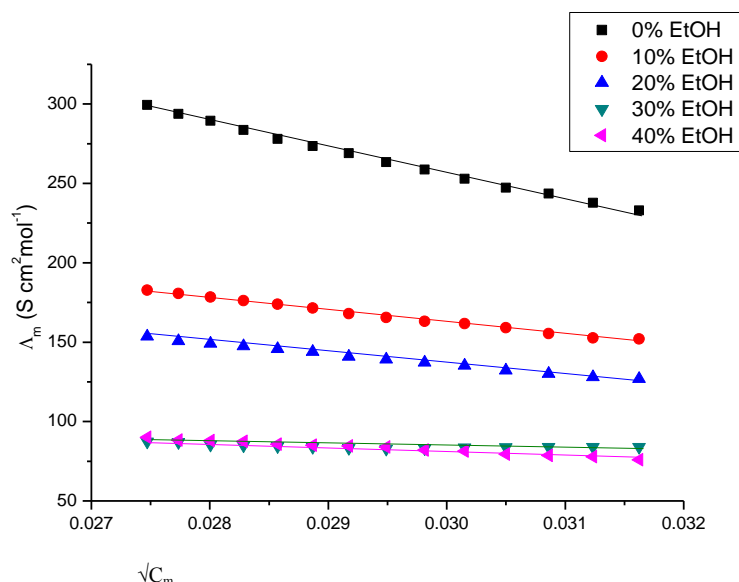


Fig. (5): The relation between molar conductance ( $\Lambda_m$ ) and  $\sqrt{C}$  of nano  $CoSO_4$  in ethanol- $H_2O$  at 308.15 K.

Table (4) : The values of viscosity ( $\eta_o$ ), limiting molar conductance ( $\Lambda_o$ ), molar conductance ( $\Lambda_m$ ), Walden product ( $\Lambda_o \eta_o$ ), fluidity ratio ( $R_x$ ), Fuoss-Shedlovsky parameters (S, Z and S(z)), activity coefficient ( $\gamma_{\pm}$ ), association constant ( $K_A$ ), dissociation constant ( $K_D$ ), degree of dissociation ( $\alpha$ ), triple ion association constant ( $K_3$ ), Gibbs free energy of association ( $\Delta G_A$ ) for nano  $CoSO_4$  in ( ETOH- $H_2O$ ) at 308.15 K.

VOL.% OF EtOH	$10^2 \eta_o$ (poise)	$\Lambda_o$	$\Lambda_m$	$\Lambda_o \eta_o$	S	Z	S(z)	$\gamma_{\pm}$	$K_A$	$10^3 K_D$	$\alpha$	$10^5 K_3$	$\Delta G_A$
0%	0.7222	736.34	243.556	5.3184	249.71	0.0060	1.0060	0.9173	7538.60	0.133	0.3327	19.6	-22.87
10%	0.7272	395.22	155.358	2.87404	170.68	0.0083	1.0083	0.9085	4898.71	0.204	0.3963	15.1	-21.77
20%	0.7329	332.20	130.158	2.43469	156.99	0.0099	1.0091	0.9069	4749.21	0.202	0.3995	15.4	-21.79
30%	0.7397	196.67	83.958	1.75888	114.95	0.0165	1.0166	0.8886	4043.66	0.289	0.5448	11.23	-19.5
40%	0.7478	179.60	78.708	1.34345	102.45	0.0193	1.0194	0.8654	3693.46	0.370	0.5887	9.43	-19.05

$\Lambda_o$  in (S cm<sup>2</sup>.mol<sup>-1</sup>),  $\Lambda_m$  in (S cm<sup>2</sup>.mol<sup>-1</sup>) and  $\Delta G_A$  in (kJ mol<sup>-1</sup>).

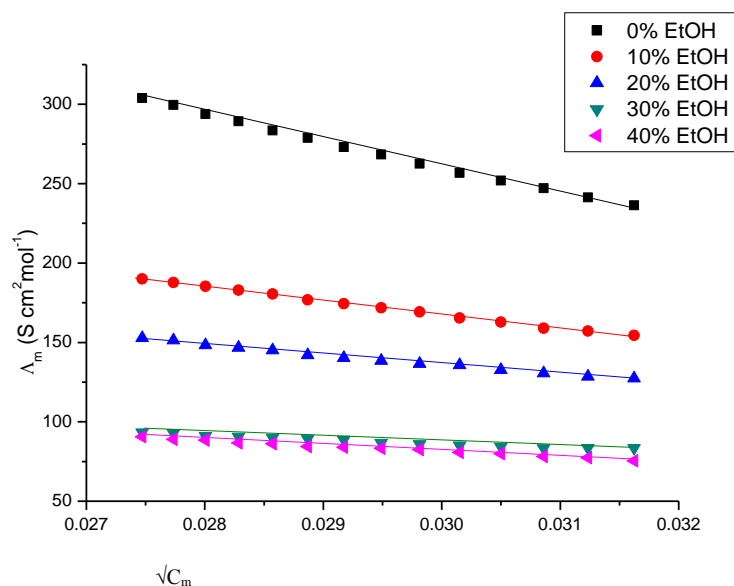


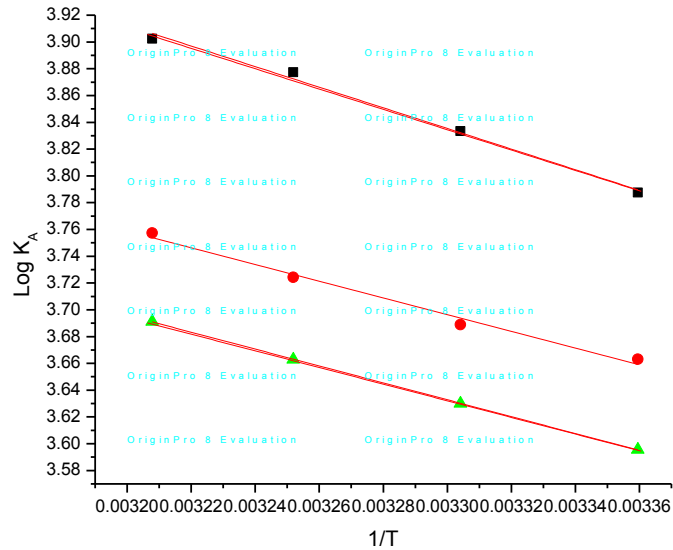
Fig. (6): The relation between molar conductance ( $\Lambda_m$ ) and  $\sqrt{C}$  of nano  $CoSO_4$  in ethanol- $H_2O$  at 313.15 K

Table (5) : The values of viscosity ( $\eta_o$ ), limiting molar conductance ( $\Lambda_o$ ), molar conductance ( $\Lambda_m$ ), Walden product ( $\Lambda_o \eta_o$ ), fluidity ratio ( $R_x$ ), Fuoss-Shedlovsky parameters ( $S$ ,  $Z$  and  $S(z)$ ), activity coefficient ( $\gamma_{\pm}$ ), association constant ( $K_A$ ), dissociation constant ( $K_D$ ), degree of dissociation ( $\alpha$ ), triple ion association constant ( $K_3$ ), Gibbs free energy of association ( $\Delta G_A$ ) for nano  $CoSO_4$  in (  $EtOH-H_2O$ ) at 313.15 K.

VOL.% OF EtOH	$10^2 \eta_o$ (poise)	$\Lambda_o$	$\Lambda_m$	$\Lambda_o \eta_o$	S	Z	S(z)	$\gamma_{\pm}$	$K_A$	$10^3 K_D$	$\alpha$	$10^5 K_3$	$\Delta G_A$
0%	0.6527	753.94	247.16	4. 9209	264.94	0.0062	1.0062	0.9165	7716.87	0.13	0.3298	19.8	-23.308
10%	0.6555	432.12	158.95	2.8303	188.55	0.0081	1.0081	0.9109	5802.30	0.172	0.3708	16.8	-22.566
20%	0.6363	334.18	130.609	2.1263	170.79	0.0098	1.0098	0.9055	4987.84	0.2	0.3947	15.2	-22.172
30%	0.661	169.46	83.9535	1.1201	128.19	0.0163	1.0164	0.8916	2647.64	0.378	0.5000	9.98	-20.523
40%	0.682	185.83	78.1	1.2673	132.53	0.1452	1.0143	0.8960	4138.25	0.241	0.4263	13.4	-21.686

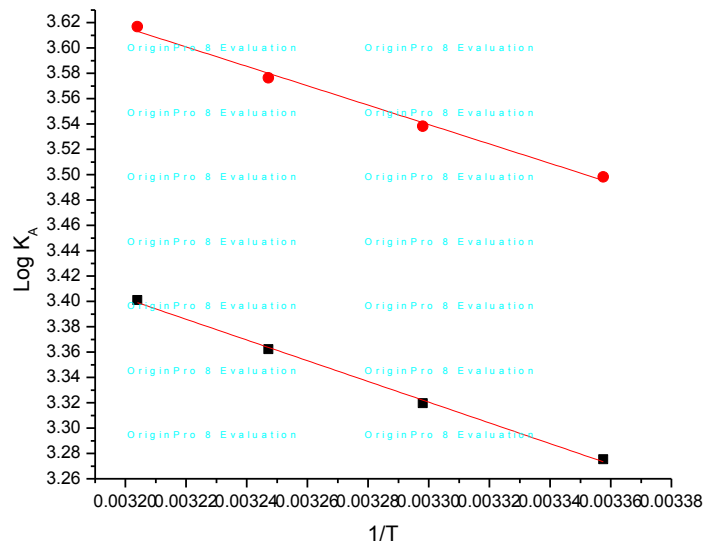
$\Lambda_o$  in ( $S\ cm^2.mol^{-1}$ ),  $\Lambda_m$  in ( $S\ cm^2.mol^{-1}$ ) and  $\Delta G_A$  in ( $kJ\ mol^{-1}$ ).





Series 1 : 0%EtOH – 100% H<sub>2</sub>O  
 Series 2 : 10% EtOH –90% H<sub>2</sub>O Series 3 : 20% EtOH – 80% H<sub>2</sub>O

Fig. (7) : The relation between  $K_A$  and  $1/T$



Series 1 : 30% EtOH – 70% H<sub>2</sub>O  
 Series 2 : 40% EtOH – 60% H<sub>2</sub>O

Fig. (8) : The relation between  $K_A$  and  $1/T$

The calculated values of  $(\Delta H_A)$  and  $(\Delta S_A)$  for nano CuSO<sub>4</sub> were tabulated in Table (6).

Table (6): Enthalpies and entropies of association for nano cobalt sulfate at 313.15K.

$X_s$	$\Delta H_s$ (kJ/mole)	TAS
0.1894	14.8442	38.153
0.2595	17.4128	39.979
0.3528	12.9616	35.134
0.4831	18.8386	39.362
0.6774	11.1296	32.816

It was observed from the data shown in all tables that  $K_A$  decrease with increase of the vol. % of ethanol and decrease also with rise of temperature due to the more association, solvation in ethanol and more solubility at higher temperatures. Dissociation degree ( $\alpha$ ) increase with increase of ethanol percentages due to more solvation and less association. Triple ion ( $K_3$ ) association constants are very small that can be neglected. S (Z) factor is almost equal 1 for divalent salts. Walden products ( $\Lambda_o \eta_o$ ) is not constant but decrease with increase ethanol percentages due to viscosity effect. All the mentioned parameters are decreased with rise of temperature due to increase of the solvation energies favor more solubility and less association.

### Conclusion

This research focused on the study of conductance measurements for nano  $\text{CoSO}_4$ . The stability constants of association were measured by applying the conductometric method at different temperatures. Based on the results, the association constant decrease with increase of EtOH and increase with increasing temperatures indicating increase in association on adding EtOH in solution and decrease by raising temperature due to the increase in the kinetic energy.

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