The Audio Frequency Conductance and thermodynamic studies of Some Metal Glutarate salts in Aqueous Medium at Different Temperatures

(Part II: Zinc, Nickel and Cobalt glutarates)

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Abstract __The electrical conductances at audio frequency of aqueous solutions of zinc, nickel and cobalt glutarates have been measured at four temperatures in the temperature range 298.15K to 313.15K. The limiting molar conductances (Λo), association constants (KA) and the closest distances of approach (a) were calculated using the complete and modified forms of Fouss-Hsia (F/H) and Pitts (P). Quantitative results showed that these salts do not behave as "strong" electrolytes, and that their dissociations are far from complete. The abnormally law conductances of these electrolytes are not due to the ion pair formation. The standard thermodynamic functions (ΔHo, ΔGo, ΔSo) for association reactions as well as Walden product have been evaluated.

Index Terms_ glutarate salts, conductance measurement, minimization technique, thermodynamic parameters.

1 INTRODUCTION

Following our investigation of association reactions of of 2:2 electrolytes (salts of dicarboxylic acids) in aqueous medium [1-3], we investigate the conductance behavior of Zinc, Nickel and Cobalt glutarate in the same medium at the temperature range from 298.15K to 313.15K. Information on ion-ion and ion-solvent interactions can be obtained from conductivity measurements. The limiting molar conductivity (Λo), association constant (KA) and the closest distance of approach (a) were determined using the theoretical conductance equations of the complete and modified forms of Fouss-Hsia [4,5] (F/H) and Pitts (P)[6,7]. From the temperature dependent of the association constant, thermodynamic quantities ΔHo, ΔGo and ΔSo were evaluated. Also, Walden product (Λo η) was derived and discussed.

2 EXPERIMENTAL

Conductance measurements were made, as before [2,3], at a range of temperatures between 298.15-313.15 K. All experimental techniques were described as before [3]. The cell constant (as determined by standard solutions of KCl) was 0.1662295 cm^{-1}.

All three studied salts were prepared by chemical and electrochemical methods[3]. A comparison by those two methods with respect to CHN analysis indicated that the electrochemical one is the most perfect as it as most specific method. So, such method was chooses here as new technique for the first time after the success of employing it during the preparation of both Mg and Cu glutarates[3].

As mentioned before, each metal (zinc, nickel or cobalt) was fused carefully in a proporiate furnace, followed by pouring them separately into a well done cavities already made before. The electrolysis of glutaric acid was began, by using the same specified metal as both cathode and anode under a constant applied potential of 2V. An initial current of 3A was displayed immediately, which started to decrease gradually until a steady value was obtained leaving a neutral solution. This solution of the metal glutarate was evaporated to dryness and a pure salt was collected. The suggested mechanism is :

$\mathbf{M = M^{+2} + 2e}$ (oxidation at anode)

$\mathbf{2H^{+} + 2e = H_2}$ (reduction at cathode)

$$\underset{\text{glutaric acid}}{HOOC\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}COOH} + \underset{\text{metal(Mg or Cu)}}{M} \longrightarrow (OOC\text{-}CH_2CH_2CH_2\text{-}COO)M + H_2$$

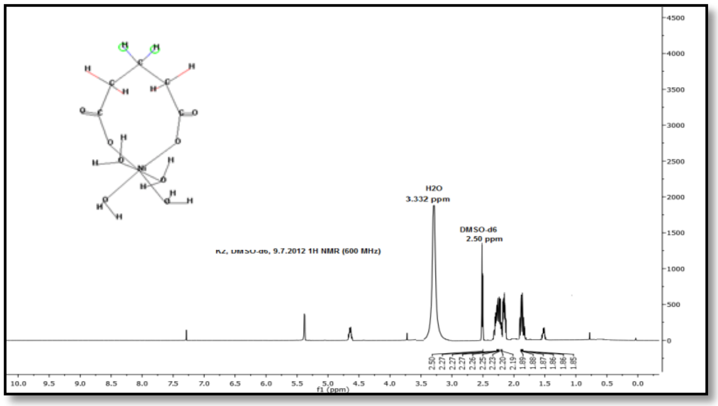

Figure 1. 1H NMR spectra for Ni- glutarate

The product of Zinc glutarate was anhydrated white crystalline powder. Elemental analysis confirmed the composition with experimental/calculated values (mass percent): C 31.021/30.701, H 4.704/4.098. The FT-IR spectra exhibits the following absorption bands (KBr pellet, υ/cm-1), stretching frequencies corresponding to the carboxylate ion υ(CO2-) and υ(C-O) at 1587, 1557, 1411, 1404, 1359 and 1303cm-1; the 1207 cm-1 absorption band is associated with υ(C-C), the following absorptions with metal-oxygen bonding and with υ(C-H) 1006, 918, 833, 761, 682, 561, 505 and 414 cm-1.

A light green crystal of nickel glutarate as a tri-hydrated was obtained as a final product. Elemental analysis indicated the composition with experimental / calculated values (mass percent): C 24.862/ 24.706, H 4.797 / 4.942. FT-IR spectra exhibits the following absorption bands (KBr pellet, υ/cm-1); υ(O-H) bands at 3545, 3334cm-1; stretching frequencies corresponding to the carboxylate ion υ(CO2-) and υ(C-O) at 1566, 1461, 1402, 1321 and 1247 cm-1; the 1157 cm-1 absorption band is associated with υ(C-C) and following absorptions are with metal-oxygen bonding and with υ(C-H) 1029, 898, 829 and 655cm-1 [6,7]. The H-NMR δH1 = 1.85 and δH2 = 2.20–2.27 ppm. the 1H NMR was recorded in DMSO-d6 solvent.

The geometrical structures of zinc, nickel and cobalt glutarates were confirmed by using the computer program Hyperchem 8, with the aid of using PM3 method. This method is mainly concerned with the computation of the dimensions of the chemical bonds as well as their energies. The recommended structures shown in Figure (2) were approved due to their maximum stabilities upon which CHN analysis were matched and taken into consideration.

CoC5H6O4.4H2 NiC5H6O4.3H2O ZnC5H6O4

Figure 2. The geometrical structures of glutarate salts

All stock solutions were prepared by weight, and all measurements were done using the weight dilution technique. Figure (3) illustrates an example of FT-IR.

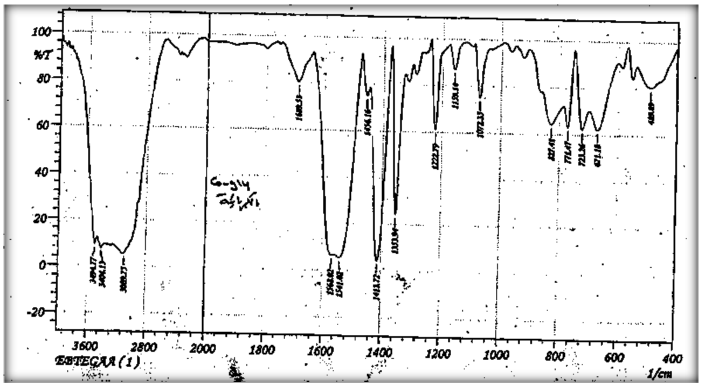

Figure 3. FT-IR spectra for Co- glutarate

3 Results and Discussion

The measured equivalent conductances (Λ) and the corresponding concentrations C in mol dm-3 at four different temperatures are given in tables 1-3. These data were analysed, using the complete and modified forms of both F/H and P equations. A minimisation technique has been used in terms of KA, Λo and a. The full details are given in part I[3].

Table 1. Experimental conductance data for Zn- glutarate

295.15K		**303.15K**		**308.15K**		**313.15K**	
Conc. $\times10^{-4}$	**Λ**	**Conc. $\times10^{-4}$**	**Λ**	**Conc. $\times10^{-4}$**	**Λ**	**Conc. $\times10^{-4}$**	**Λ**
3.89129	94.850	8.60823	98.571	5.71190	107.023	2.01178	131.543
7.30307	89.233	9.29738	97.522	9.19080	104.470	4.51771	125.177
8.75139	86.812	10.10465	96.311	10.90324	103.329	4.94783	124.206
9.36278	86.469	10.70978	95.526	11.71388	101.855	5.49567	122.109
9.99229	86.013	11.42150	94.667	12.48630	102.210	6.09165	120.941
10.57576	85.197	11.85630	93.999	13.14776	100.861	7.40413	118.024
11.78171	83.531	12.27824	93.477	13.83398	100.063	8.12779	116.717
12.20615	83.350	12.86722	93.074	14.50153	99.469	8.72219	116.389
12.65160	83.044	13.35277	92.802	15.10713	99.333	9.52157	114.910
13.16061	82.357	13.80420	92.777			10.24931	113.644
13.50842	82.083	14.20320	91.926			10.96092	112.332
14.35796	81.278	14.57803	91.843			11.72611	111.381
14.69043	81.136						
15.03671	80.925						
15.37439	80.770						

Λ (ohm-1cm2 equiv-1); Conc. = concentration (mol dm-3)

TABLE 2. EXPERIMENTAL CONDUCTANCE DATA FOR NI- GLUTARATE

298.15K		303.15K		308.15K		313.15K	
Conc.×10^{-4}	**Λ**	**Conc.×10^{-4}**	**Λ**	**Conc.×10^{-4}**	**Λ**	**Conc.×10^{-4}**	**Λ**
1.59454	103.207	4.63681	102.173	2.02348	120.967	6.23650	126.741
3.20412	98.831	5.27346	100.555	6.05811	110.306	6.98751	124.895
7.33934	91.955	6.26285	99.533	7.65726	110.033	7.91475	122.865
7.68653	91.911	7.34480	97.319	11.86118	102.306	9.39759	121.167
8.92839	90.298	7.70484	97.625	14.02009	101.077	11.12046	118.090
9.23596	89.990	8.83701	95.934	15.14341	100.440	11.89387	116.700
9.65433	89.534	9.33047	95.314	15.75532	99.176	12.68540	115.315
10.03247	88.645	10.69247	94.056	16.24661	99.247	13.43004	115.110
10.78052	87.891	11.20137	93.493	16.81985	98.335	14.17776	113.729
11.18447	87.689	11.80682	92.922	17.37245	98.556		
11.22476	87.644	12.27413	92.770	17.79595	98.079		
11.81684	87.216	12.64309	92.692	18.24557	97.484		
12.08491	86.657	13.24352	91.628	18.52062	97.383		

Λ(ohm1m2 equiv1); Conc. = concentration (mol dm-3)

TABLE 3. EXPERIMENTAL CONDUCTANCE DATA FOR CO- GLUTARATE

298.15K		303.15K		308.15K		313.15K	
Conc.×10^{-4}	**Λ**	**Conc. ×10^{-4}**	**Λ**	**Conc. ×10^{-4}**	**Λ**	**Conc. ×10^{-4}**	**Λ**
1.67154	102.480	3.04719	102.748	3.54551	110.085	1.88307	126.896
2.07230	101.512	4.93263	100.375	4.53380	108.009	2.53523	126.054
2.55628	99.850	7.49754	96.744	5.52657	107.019	3.41099	124.149
3.01497	99.821	9.02576	95.097	7.97790	103.306	4.05469	122.289
3.90771	98.711	9.78000	94.562	8.85908	102.413	5.00160	121.558
4.71893	97.594	11.29355	92.928	9.69587	101.289	5.60638	120.305
5.14520	96.455	11.87889	92.547	10.54856	99.405	8.50810	115.224
6.02111	94.156	12.58811	91.952	11.19579	98.853	9.32360	114.952
6.35206	93.438	12.98450	91.708	11.97983	98.629	10.00914	112.891
6.90821	93.134	13.91007	90.984	12.89504	97.430	10.85765	112.490
7.22534	93.073	14.44434	90.495	13.52004	96.614	11.64404	112.030
7.44512	92.558	15.09984	89.869	14.12722	96.580	12.32463	111.239
7.66112	92.444			14.71106	95.572	12.93717	111.112
7.95323	92.392					13.63790	110.248
8.23457	92.264						

Λ (ohm-1cm2 equiv-1); Conc. = concentration (mol dm-3)

The results of the minimisation technique for the best fit values of the three parameters KA, Λo and <u>a</u> are listed in tables (4-6) together with the diffusion coefficients (Dsalt), Walden products and the corresponding values of the standard deviation (σ) .

TABLE 4. BEST FIT RESULTS FOR ZN-GLUTARATE

Temp.	Parameter*	F-H complete	F-H modified	Pitts complete	Pitts modified
298.15 K	$\Lambda_\circ$	112.5	110.7	105.56	110.2
	K_A	78.5	38.5	268	60
	a	0.46	0.46	0.6	0.34
	$D_{salt} \times 10^{-5}$	1.497	1.473	1.405	1.467
	σ	0.075	0.091	0.154	0.164
	$\Lambda_\circ \eta$	1.00226	0.98623	0.94043	0.98177
303.15 K	$\Lambda_\circ$	121.7	118.9	118.8	121.2
	K_A	88	104	277	72
	a	0.79	1.7	0.4	0.91
	$D_{salt} \times 10^{-5}$	1.647	1.609	1.608	1.640
	σ	0.436	0.453	0.288	0.299
	$\Lambda_\circ \eta$	0.97141	0.94906	0.94826	0.96742
308.15 K	$\Lambda_\circ$	124.7	129.7	128	132.9
	K_A	99	104	281	73
	a	1.21	1.35	0.4	0.7
	$D_{salt} \times 10^{-5}$	1.757	1.784	1.761	1.828
	σ	0.685	0.161	0.426	0.406
	$\Lambda_\circ \eta$	0.92302	0.93747	0.92500	0.9606
313.18 K	$\Lambda_\circ$	147.8	147.9	143.2	146.6
	K_A	86	138	284	92
	a	0.7	1.4	1.1	1
	$D_{salt} \times 10^{-5}$	2.066	2.067	2.002	2.049
	σ	0.471	0.379	0.268	0.328
	$\Lambda_\circ \eta$	0.96661	0.96727	0.93652	0.95876

**Preferred Value*

Units of : Λ□ ohm-1 cm2 equiv-1 ;KA dm3 mol-1; a nm; Dsalt cm2 s-1; σ unitless; Λ□□ ohm-1 cm2 equiv -1 p

Standard thermodynamics quantities for the association reaction are obtained from the temperature dependence of the association constant KA. The standard enthalpy change (ΔHo) was determined from the slope of log KA versus 1/T, while the standard Gibbs energy (ΔGo) and entropy (ΔSo) changes were calculated using the well known relations[10]:- -RTlnKA= ΔGo = ΔHo -T ΔSo .

As in part (I), it was noted that the minimised Λo and a values for the complete and modified Fouss-Hisa equations were almost greater than those values obtained using the complete and modified forms of Pitts. The reason for this was mentioned[11,12].

A gain here, it is obvious from the minimisation technique that the parameter Λo had the greatest effect on the values of S2 during the variation of the three parameters Λo, KA and a due to the fact that Λo is the leading term in all conductance equations. As shown in of values. The increase of Λo with an increase of temperature is due to the decrease of solvent viscosity, while the increase in KA with increase in temperature is due to the decrease in dielectric constant which leads to the stabilization of ion-pair. The same trend was found by others[13,14].

table (5) and (6), each system gave a reasonable best set of parameters at each temperature. It is clear that both Λo and KA showed an expected trend with temperature, and this trend for a was quite irregular and covered a broader range With respect to the association constants given in tables(4-6), the same behavior as for our previous glutarate salts[3], it can be anticipated that zinc, nickel and cobalt glutarates do not behave as "strong" electrolytes, and that their dissociation are far from complete. Furthermore, it can be generally predicated, for all three studied salts, that the degree of dissociation is changed from one to another.

Once more again, the three studied salts are more highly dissociated than succinate, oxalates and malonates due to the less degree of their donar property[3].

TABLE 5. BEST FIT RESULTS FOR NI-GLUTARATE

Temp.	**Parameter***	**F-H complete**	**F-H modified**	**Pitts complete**	**Pitts modified**
298.15 K	$\Lambda_\circ$	112.9	114.2	109	112.8
	K_A	28	50	251	40
	$\underline{a}$	0.45	0.5	0.65	0.33
	$D_{salt} \times 10^{-5}$	1.503	1.520	1.451	1.501
	σ	0.192	0.246	0.265	0.185
	$\Lambda_\circ\eta$	1.0058	1.01741	0.97711	1.00494
303.15 K	$\Lambda_\circ$	120.2	120.3	115.2	119.1
	K_A	42	62	259	48
	$\underline{a}$	0.58	0.71	0.3	0.55
	$D_{salt} \times 10^{-5}$	1.627	1.628	1.559	1.612
	σ	0.228	0.286	0.359	0.229
	$\Lambda_\circ\eta$	0.95.944	0.96023	0.91953	0.95065
308.15 K	$\Lambda_\circ$	134	135.2	128.6	133.7
	K_A	56	65	269	58
	$\underline{a}$	0.61	0.66	0.4	0.6
	$D_{salt} \times 10^{-5}$	1.845	1.860	1.769	1.839
	σ	0.296	0.307	0.369	0.276
	$\Lambda_\circ\eta$	0.96855	0.97723	0.92952	0.96638
313.18 K	$\Lambda_\circ$	157.7	158	151.3	152.5
	K_A	71	84	307	81
	$\underline{a}$	0.5	0.6	0.6	0.92
	$D_{salt} \times 10^{-5}$	2.204	2.209	2.112	2.132
	σ	0.313	0.317	0.336	0.309
	$\Lambda_\circ\eta$	1.03136	1.03332	0.98950	0.99735

**Preferred Value*

Units of : $\Lambda_\circ$ *ohm-1 cm2 equiv-1 ;KA dm3 mol-1; a nm; Dsalt cm2 s-1;* σ *unitless;* $\Lambda_\circ\eta$ *ohm-1 cm2 equiv -1 p*

In figures (4-6), curves of molar conductance versus square root of concentration at four temperatures have been obtained for the three glutarate salts. For these electrolytes, the measured conductivity was abnormally small. Abnormally weak salts usually form auto-complexes readily, as is shown by Vosburgh and Beckman's solubility measurements[15] for zinc and cadmium oxalates and by Ives and Riley's conductivity measurements[16] for copper malonate. In this part, with the exception of zinc glutarate, however, the experimental conductances give no evidence of this.

TABLE 6. BEST FIT RESULTS FOR CO-GLUTARATE

Temp.	**Parameter***	**F-H complete**	**F-H modified**	**Pitts complete**	**Pitts modified**
298.15 K	$\Lambda_\circ$	112	112.1	108.4	111
	K_A	26	82	223	62
	a	0.574	1.24	0.75	1.4
	$D_{salt} \times 10^{-5}$	1.491	1.492	1.443	1.477
	σ	0.312	0.319	0.410	0.387
	$\Lambda_\circ\eta$	0.99781	0.99870	0.96570	0.98890
303.15 K	$\Lambda_\circ$	118.1	116.1	112.4	118.6
	K_A	36	95	228	63
	a	0.63	1.5	0.3	0.73
	$D_{salt} \times 10^{-5}$	1.598	1.571	1.521	1.605
	σ	0.132	0.141	0.416	0.404
	$\Lambda_\circ\eta$	0.94267	0.9267	0.89717	0.94667
308.15 K	$\Lambda_\circ$	127.1	126.6	120.8-121.7	127.6
	K_A	47	99	229-235	67
	a	0.67	1.4	0.5	0.75
	$D_{salt} \times 10^{-5}$	1.748	1.741	1.661-1.674	1.755
	σ	0.360	0.434	0.319	0.277
	$\Lambda_\circ\eta$	0.91869	0.91506	0.87314	0.92229
313.18 K	$\Lambda_\circ$	138.8	142.2	136	144
	K_A	49	107	238	67
	a	0.99	1.4	0.3	0.8
	$D_{salt} \times 10^{-5}$	1.940	1.988	1.901	2.013
	σ	0.720	0.5817	0.241	0.323
	$\Lambda_\circ\eta$	0.90775	0.92998	0.88944	0.94176

**Preferred Value*

Units of : *Λ□ ohm-1 cm2 equiv-1 ;KA dm3 mol-1; a nm; Dsalt cm2 s-1; σ unitless; Λ□□ ohm-1 cm2 equiv -1 p*

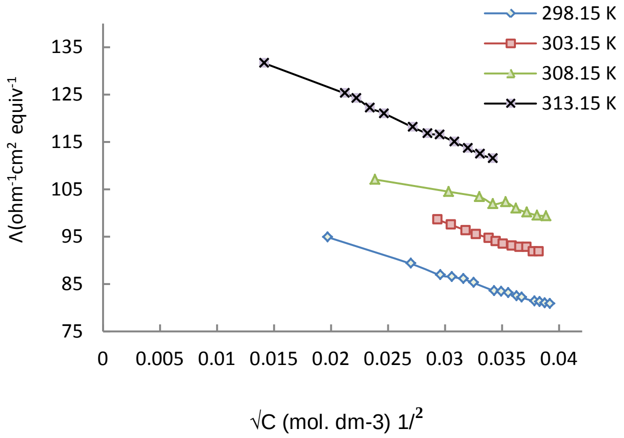

Figure 4. Molar conductance versus square root of concentration for Zn-glutarate at different temperatures

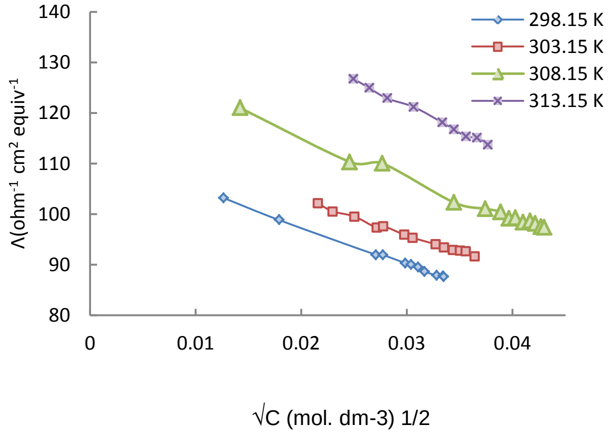

Figure 5.Molar conductance versus square root of concentration for Ni-glutarate at different temperatures

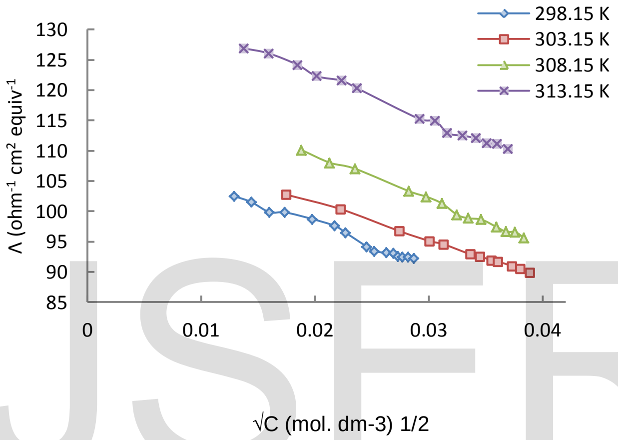

Figure 6. Molar conductance versus square root of concentration for Co-glutarate at different temperatures

The same difficulties resulting from the limited solubilities, as in the case of the four previous glutarate salts mentioned in part[3], were raised here again. Despite that, the values obtained for K_A, Λ_o and **a** for both nickel and cobalt glutarates are satisfactory, providing a particularly another good example of typical incompletely dissociated electrolytes the thermodynamic association constants of which conform to the law of mass action. The same conclusion [3] for the abnormal low conductances of these electrolytes is predicated here as well.

As mentioned in part I [3], no more recent determination of Λ_o at 298.15K for the studied glutarate salts have been found. So, in comparison with ionic conductances at infinite dilution at 298.15K taken from reference [17]:- $\lambda_{o1/2}Zn^{+2}$=52.80, $\lambda_{o1/2}Ni^{+2}$= 49.6 and $\lambda_{o1/2}Co^{+2}$=55.00 together with the value of 58.8[17] and 56.88[18] of glutarate ion, good agreement has been shown for both Nickel and Cobalt glutarates a shown in tables (5 and 6).

The Λ_o value obtained in this part for zinc glutarate is much lower than that given by direct combination of λ_o values of ionic conductances. In conclusion, it appears that the zinc glutarate is capable of showing either marked tendency to auto-complex formation according to the equation:-

$$Zn^{+2}(C_5H_6O_4) + ZnC_5H_6O_4 \leftrightarrow [\, Zn\,(C_5H_6O_4)_2]^{-2}Zn^{+2}$$

as is shown for zinc oxalate [15], or the zinc ions are linked by a bridging glutarate ligand to form some kind of coordination polymer[19].

Data from tables (4-6) show that the Walden product dependence on the temperature is substantially obeyed. The explanation is given before[3].

The standard thermodynamic quantities for the association reaction of metal ion (M^{2+}) and glutarate ions are obtained from the temperature dependence of K_A as given in tables (7-9), while figures (7-9) shown the corresponding plots for all three investigated glutarate salts.

The same interpretations and conclusions are recommended here exactly as given in part I.

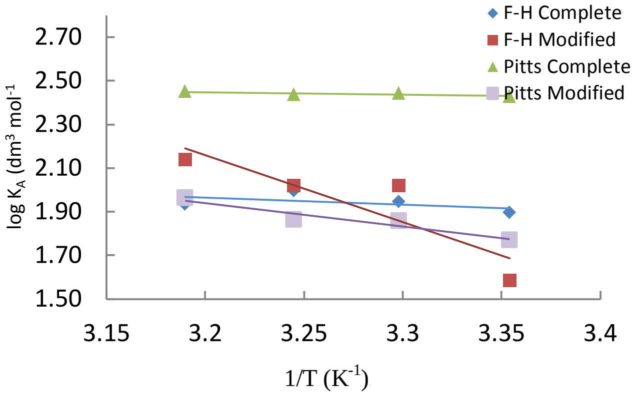

Figure 7. Logarithm of association constant versus inverse temperature for Zn-glutarate

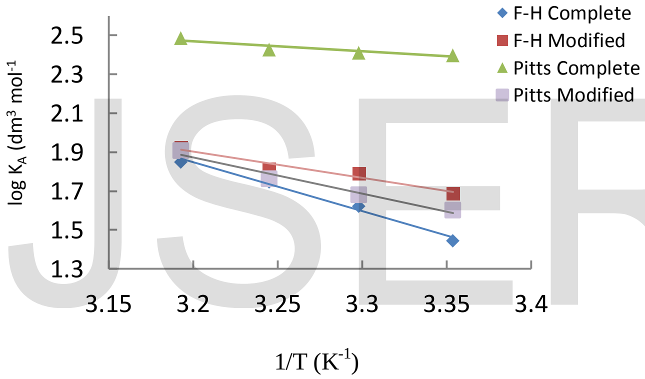

Figure 8. Logarithm of association constant versus inverse temperature for Ni-glutarate

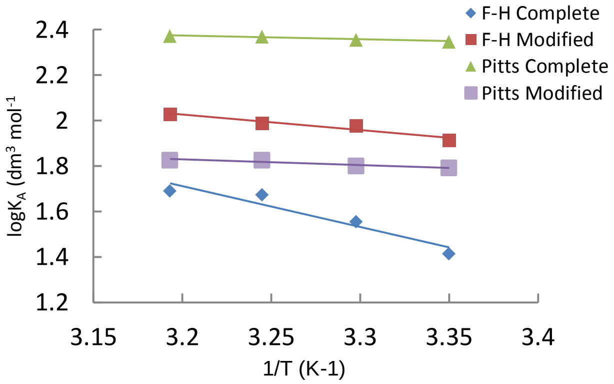

Figure 9. Logarithm of association constant versus inverse temperature for Co-glutarate

Table 7. Thermodynamic data for Zinc glutarate

	T (K)	F-H Complete	F-H Modified	Pitts Complete	Pitts Modified
ΔH°(KJmol^{-1})		6.0064	58.6626	2.155	20.6025
ΔG°(KJ mol^{-1})	298.15	-10.8153	-9.0493	-13.859	-10.1491
	303.15	-11.2896	-11.7056	-13.6729	-10.7788
	308.15	-11.7725	-11.8987	-14.4452	-10.9919
	313.15	-11.5970	-12.8282	-14.7072	-11.7726
ΔS°(J mol^{-1})	298.15	56.4202	227.1068	53.7112	103.1501
	303.15	57.0377	232.1233	52.2114	103.5259
	308.15	57.6956	228.9836	53.8705	102.5377
	313.15	56.2139	228.8957	53.8471	98.2091

Table 8.Thermodynamic data for Nickel glutarate

	T (K)	F-H Complete	F-H Modified	Pitts Complete	Pitts Modified
ΔH°(KJ mol^{-1})		47.8478	25.9391	9.9460	35.6318
ΔG°(KJ mol^{-1})	298.15	-8.2599	-9.6972	-13.6966	-9.1441
	303.15	-9.4204	-10.4019	-14.0054	-9.7569
	308.15	-10.3128	-10.6946	-14.3334	-10.4027
	313.15	-11.0980	-11.5357	-14.9100	-11.4411
ΔS°(J mol^{-1})	298.15	188.1865	119.5245	79.2977	150.179
	303.15	188.9106	119.8781	79.0083	149.7237
	308.15	188.7414	118.8825	78.7909	149.3899
	313.15	188.2353	119.6705	79.3742	150.3205

Table 9. Thermodynamic data for Cobalt glutarate

	T (K)	F-H Complete	F-H Modified	Pitts Complete	Pitts Modified
ΔH°(KJ mol^{-1})		34.4051	13.1801	3.2174	4.7201
ΔG°(KJ mol^{-1})	298.15	-8.0762	-2.2212	-6.0406	-1.6794
	303.15	-9.0319	-2.8304	-6.7929	-1.8770
	308.15	-9.8639	-3.1690	-7.5224	-2.1448
	313.15	-10.1325	-3.4622	-7.7008	-2.1679
ΔS°(J mol^{-1})	298.15	142.4830	51.6563	31.0516	21.4645
	303.15	143.2853	52.8138	33.0211	21.7621
	308.15	143.6606	53.0558	34.8526	22.2777
	313.15	142.2243	53.1448	34.8661	21.9961

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