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Thermodynamic Properties of D- and L-Tartaric Acid in Aqueous and Ethanol Solution at 298.15 K

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Enthalpies of mixing and excess volumes of D- and L-tartaric acid in aqueous and ethanol solution were measured at 298.15 K. Excess enthalpies of mixing were observed for three different concentrations of aqueous and ethanol solutions of tartaric acid. Enthalpies of mixing were exothermic for all the concentrations in both solvents. Enthalpic stabilization on mixing was increased with decreasing concentration of tartaric acid for both solvents. Excess volumes of D- and L-tartaric acid in aqueous solution showed volume contraction on mixing, but those of D- and L-tartaric acid in ethanol solution showed volume expansion on mixing. Volume contractions of aqueous solution were increased with decreasing concentration of tartaric acid.

Introduction

Molecular interactions such as solute−solute and solute−solvent interactions influence the processes and reactions that take place in the solution, especially intermolecular interactions due to the stereospecific structure of molecules. Takagi et al.[1] measured the enthalpies of mixing of enantiomers of tartaric acid with threonine first and then alanine.[2] In recent years, some investigators have studied the problem of chiral interaction in aqueous and other solvents.[3−6] Previous calorimetric work in our laboratory includes measurement of enthalpies of mixing for binary liquid chiral systems,[7−9] in order to understand the chiral discrimination and structural properties of the liquids in the mixture.

To understand the structure and behavior of tartaric acid in the solution, enthalpies of mixing of aqueous and ethanol solution of D- and L- tartaric acid have been measured at 298.15 K for a large range of mole fraction of heterochiral concentration. Dilution experiments were also performed.

Experimental Section

D- and L-tartaric acid (Tokyo Kasei and Kishida Kagaku) were used without further purification. Purified water was used for the preparation of the aqueous solutions.[10] Ethanol(Kishida Kagaku, Special grade) was dried by storage over molecular sieves 4A freshly activated. The excess enthalpies of mixing and dilution were measured by a microcalorimeter (Thermometic AB, Järfälla, Sweden) with a 0.8 mL mixing vessel at 298.15 K.[11,12] Solutions of one enantiomer were placed in the stainless steel mixing vessel. The calorimeter syringe was filled with ca. 500 μL of solution of other enantiomers and placed in a pump. A cannula from the syringe was carefully inserted into the sample stage. Samples (20 to 100 μL) were injected into the vessel by use of gastight syringes (Hamilton 1710N or 1725N) fitted with a stainless steel cannula (0.2 mm i.d., 0.45 mm o.d., 1 m length). Once assembled, the sample was left for about 2 h to equilibrate thermally. When it reached equilibrium temperature, the injection experiments were initiated. Details of the calorimetric procedure have been described previously.[12,13] The enthalpies of solution of propane-1-ol in water was used as a test reaction[14] for the calorimetric measurements. Also other reliability test of the microcalorimeter system and the procedures used had been performed using a binary mixture of (1 − x)1,4-dimethybenzene + x1,3-dimethylbenzene and (1 − x)1,4-dimethybenzene and x1,2-dimethylbenzene as reported previously[15] for exothermic and endothermic system. Densities of the samples measured by a vibrating-tube densimeter (Anton Paar D60) at (298.15 ± 0.001) K are also listed in Table 1. The details of densitometric procedures were the same as those described previously.[15]

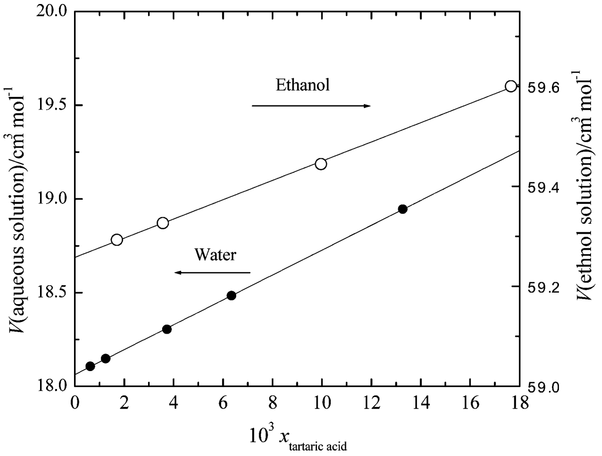

Figure 1. Molar volumes of aqueous and ethanol solution of tartaric acid at 298.15 K.

Results and Discussions

Experimental results of densities and molar volumes are listed in Table 1, and shown in Figure 1. Molar volumes of solutions of D- and L-tartaric acids measured were determined with the smoothing equation:

$$V/\mathrm{cm}^3\cdot\mathrm{mol}^{-1} = a + bx \qquad (1)$$

where x is the mole fraction of tartaric acid. Molar volumes of aqueous and ethanol solution of D-tartaric acid and L-tartaric acid increased linearly with increasing concentration of tartaric

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Table 1. Densities and Molar Volumes of Aqueous and Ethanol Solution of D- and L-Tartaric Acids at 298.15 K

solution wt %	$10^4 x(\mathrm{D})$	ρ g·cm^{-3}	V cm^3·mol^{-1}	solution wt %	$10^4 x(\mathrm{L})$	ρ g·cm^{-3}	V cm^3·mol^{-1}
			Aqueous Solution				
0.52017	6.2718	0.999491	18.1056	0.51684	6.2315	0.999343	18.1063
1.0292	12.466	1.00167	18.1478	1.0839	13.134	1.00187	18.1530
3.1137	37.253	1.01060	18.3115	2.9594	36.468	1.01028	18.3069
5.0506	63.437	1.01986	18.4842	5.0599	63.559	1.01990	18.4851
10.078	132.73	1.04336	18.9451	10.086	132.85	1.04337	18.9464
			Ethanol Solution				
0.54724	17.018	0.787203	59.2915	0.55337	17.209	0.787245	59.3005
1.14079	35.623	0.789980	59.3268	1.0249	34.412	0.789816	59.3292
3.14623	99.635	0.799516	59.4492	3.1374	99.350	0.799446	59.4389
5.42243	176.50	0.810830	59.6005	5.4892	176.76	0.810866	59.6022

Table 2. Best Fit Coefficients of Equation 1 and Standard Deviation of the Fits

	a	b	s_f cm^3·mol^{-1}	$V_2(x \rightarrow 1)^a$ cm^3·mol^{-1}
		Aqueous Solution		
D-tartaric acid	18.0643	66.3331	0.0008	84.39
L-tartaric acid	18.0653	66.2721	0.0010	84.34
		Ethanol Solution		
D-tartaric acid	59.2575	19.3778	0.0017	78.64
L-tartaric acid	59.2563	19.5169	0.0009	78.77

[a] $V_2(x \rightarrow 1)$, partial molar volume of tartaric acid at $x = 1$.

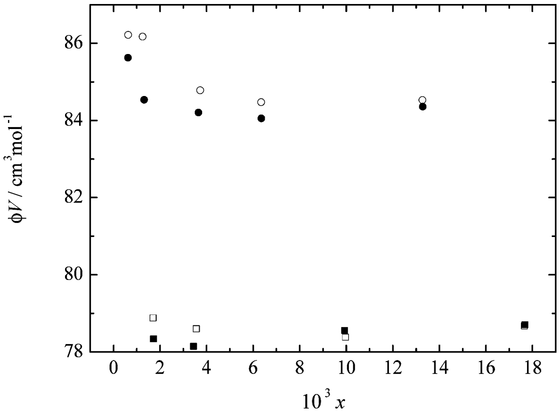

Figure 2. Apparent molar volumes of tartaric acids: ○, D-tartaric acid in aqueous solution; ●, L-tartaric acid in aqueous solution; □, D-tartaric acid in ethanol solution; ■, L-tartaric acid in ethanol solution.

Table 3. Excess Volumes of Aqueous Solution and Ethanol Solutionof Tartaric Acid at 298.15 K[a]

f_D	$10^4\ V^E$ cm^3·mol^{-1}	f_D	$10^4\ V^E$ cm^3·mol^{-1}	f_D	$10^3\ V^E$ cm^3·mol^{-1}
aqueous solution				ethanol solution	
9.806 %		5.0553 %		5.023 %	
0.1006	−3.7	0.1008	−4.7	0.1002	2.36
0.2007	−7.0	0.1997	−8.9	0.2014	6.43
0.3006	−8.4	0.2996	−11.8	0.3019	8.06
0.4006	−9.8	0.4003	−13.5	0.4002	8.98
0.5010	−10.6	0.4994	−13.9	0.5008	9.15
0.6010	−10.1	0.5998	−14.3	0.7005	7.87
0.7009	−8.4	0.6994	−13.2	0.8010	6.15
0.7993	−5.4	0.7997	−9.6	0.9005	3.71
0.8967	−2.4	0.8988	−5.0		

[a] Mole fraction of pseudo-two-component system of D-tartaric acid and L-tartaric acid: $f_D = n_{\text{D-tartaric acid}}/(n_{\text{D-tartaric acid}} + n_{\text{L-tartaric acid}})$.

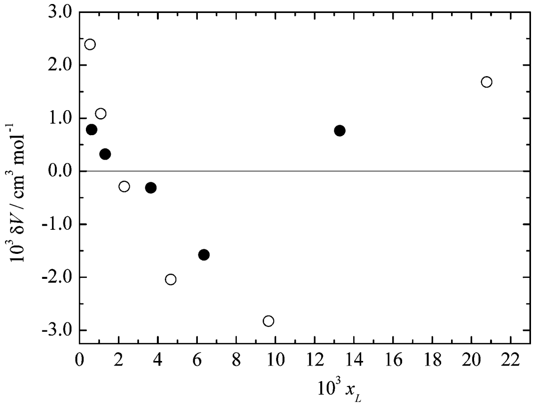

Figure 3. Deviation plots of molar volumes for aqueous solution of L-tartaric acid at 298.15 K. $\delta V = V - V$ (eq 1 with coefficients in Table 2). ●, present work; ○, Patterson and Woolley.[16]

acid as shown in Figure 1. Those correlation coefficients were more than 0.9999. Molar volumes of aqueous solution of L-tartaric acids are shown in Figure 2 and are in good agreement with the data of Patterson and Woolley.[16]

When molar volumes of solution are extrapolated to $x = 1$ by eq 1 with coefficients in Table 2, the values show partial molar volumes of tartaric acid in liquid state. Volumes of tartaric acid calculated by eq 1 in water were 7 % larger than those of one in ethanol solution as listed in 4th column of Table 2. Apparent molar volume φV of tartaric acid was also calculated by eq 2 and is shown in Figure 3:

$$\varphi V/\mathrm{cm^3 \cdot mol^{-1}} = (V - V_1^* \times n_1)/n_2 \tag{2}$$

where V, V_1^*, and n_i are molar volume of solution, volume of pure solvent, mole of component 1, respectively. Apparent molar volumes of tartaric acids were almost constant in water and ethanol as shown in Figure 3. But those of D- and L-tartaric acids in water were also 7 % larger than those in ethanol. The partial molar volumes of tartaric acids and apparent molar volumes of tartaric acids in water and ethanol were almost the same value for each solution. That means excess volumes of mixtures of water or ethanol + hypothetical liquid tartaric acids at 298.15 K were very small.

Excess volumes of mixing V^E were determined by eq 3, listed in Table 3, and shown in Figure 4:

$$V^E/\mathrm{cm^3 \cdot mol^{-1}} = V - (V(D)(1 - f_L) + V(L)f_L) \tag{3}$$

where f_L is the mole fraction of L-tartaric acid in pseudo-two-component systems of L- and D-tartaric acids: $f_L = n_{\text{L-tartaric acid}}/(n_{\text{D-tartaric acid}} + n_{\text{L-tartaric acid}})$. $V(D)$ and $V(L)$ are the molar volume of pure D- and L-enantiomer solution, respectively. Excess volumes of aqueous solution were very small but negative. Aqueous solutions of D- and L-tartaric acid mixture were more favorable packing than pure aqueous solution of D- and L-tartaric acid. However, excess volumes of ethanol solution were positive over the whole range of mole fractions. The

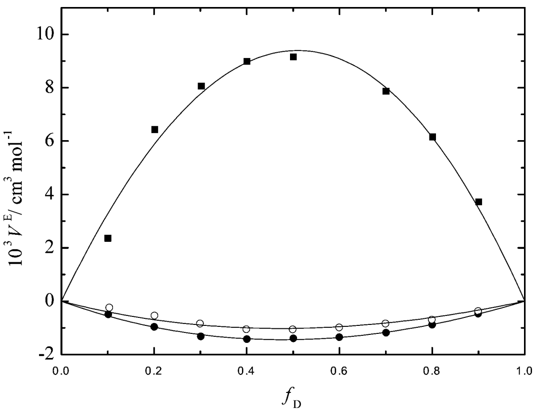

Figure 4. Excess volume of aqueous and ethanol solution of D-tartaric acid + L-tartaric acid: ●, 5 % aqueous solution of tartaric acid; ○, 10 % aqueous solution of tartaric acid; ■, 5 % ethanol solution of tartaric acid. $f_D = n_{L\text{-tartaric acid}}/(n_{D\text{-tartaric acid}} + n_{L\text{-tartaric acid}})$.

ethanol solutions of D- and L-tartaric acid mixtures were more unfavorable packing than pure ethanol solution of D- and L-tartaric acid. The volume change of mixing at equimolar composition of aqueous solution and ethanol solution at 5 % were 0.001 % and 0.01 % of ideal mixing, respectively.

The experimental results for enthalpies of mixing of D- and L-tartaric acid of different concentration aqueous and ethanol solutions are listed in Table 4 and plotted in Figure 5 and in Figure 6. Excess enthalpies of mixing of aqueous solution of D- and L-tartaric acid were very small, but all excess enthalpies measured concentration that showed exothermic enthalpy change. The mixing process of the aqueous solution of D- and L-tartaric acid created a stabilization of heterochiral contacts as compared to homochiral ones over the whole range of composition at 298.15 K. Enthalpy changes on mixing decreased with increasing the concentration of tartaric acid in solutions as shown in Figure 5. In the aqueous solution of tartaric acid, three states on mixing might be changed. One is the ionization of tartaric acid, the second is the solvation of tartaric acid, and the third is the formation of heterochiral contacts. Enthalpy of ionization and enthalpy of solvation were endothermic and exothermic reactions, respectively. The ionization of tartaric acid on mixing in the higher concentration was lees than in the lower concentration as in the Ostwald dilution law for such a week acid of carbolic acids. The effect of the enthalpy of ionization of tartaric acid on enthalpy of mixing for higher concentration was less than that of lower concentration. In the lower concentration solution, enthalpy of solvation of tartaric acid predominated over enthalpy of ionization of tartaric acid and vice versa. The enthalpy change is less negative for concentrated aqueous solution than dilute solution of tartaric acid. Minimum excess enthalpies at $f_D = 0.5$ are listed in Table 5. The enthalpies of mixing at minimum values increased with increasing concentration of tartaric acid as shown in Figure 4. This result might be consistent with the result of Takagi and Amaya[2] where 15.6 % (w/w) aqueous solution showed slightly endothermic enthalpy change. The concentration was three time higher than our highest one of 5 % (w/w). Excess enthalpies of mixing of ethanol solution of D- and L-tartaric acid were also very small, and all enthalpies showed exothermic enthalpy change as such as as aqueous solution. But excess enthalpies of ethanol solution were 55 % to 77 % less stabilized on mixing than those of aqueous solution at the same concentrations of tartaric acids. Also positive excess volumes of mixing lead to unstabilization or less stabilization on mixing process and vice versa. Stabilization on mixing of heterochiral solution of aqueous and ethanol might involve stabilization from volume changes on mixing of aqueous and ethanol solution.

Table 4. Excess Enthalpies of Mixing of Different Concentration (1 − f_D) Tartaric Acid and f_D Tartaric Acid Aqueous and Ethanol Solution at 298.15 K[a]

f_D	H^E J·mol^{-1}	f_D	H^E J·mol^{-1}	f_D	H^E J·mol^{-1}
(1 − f_D) L-Tartaric Acid + f_D Tartaric Acid in Aqueous Solution at 0.50911 wt %					
0.0891	−70.05	0.4063	−198.6	0.6882	−180.7
0.1092	−78.76	0.4239	−213.1	0.6941	−195.9
0.1635	−118.3	0.4388	−211.0	0.7340	−166.4
0.1970	−122.6	0.4680	−218.1	0.7516	−173.6
0.2268	−134.3	0.4943	−227.0	0.7863	−146.9
0.2690	−157.0	0.5508	−224.0	0.8194	−139.8
0.2811	−150.3	0.5798	−222.1	0.8466	−111.0
0.3283	−173.4	0.6119	−214.7	0.9008	−97.79
0.3697	−188.0	0.6478	−204.0	0.9169	−70.61
0.3801	−198.0				
(1 − f_D) Tartaric Acid + f_D Tartaric Acid in Aqueous Solution at 1.0107 wt %					
0.07652	−26.73	0.4272	−118.4	0.6360	−105.6
0.0823	−44.33	0.4465	−120.4	0.6466	−110.8
0.1422	−45.95	0.4531	−124.0	0.6958	−96.00
0.1520	−65.23	0.4726	−133.0	0.7531	−79.76
0.2119	−79.38	0.5381	−131.9	0.7775	−79.09
0.2639	−89.61	0.5665	−128.6	0.8206	−62.18
0.3094	−97.95	0.5672	−125.8	0.8398	−48.57
0.3497	−106.9	0.5996	−120.8	0.9015	−34.72
0.3855	−111.3	0.6039	−123.5	0.9129	−28.29
0.4176	−116.1				
(1 − f_D) Tartaric Acid + f_D Tartaric Acid in Aqueous Solution at 5.0045 wt %					
0.1172	−9.182	0.3990	−26.56	0.6481	−22.93
0.1189	−9.400	0.4029	−27.41	0.6805	−21.65
0.2098	−14.28	0.4434	−28.13	0.7106	−20.03
0.2125	−14.32	0.5127	−29.05	0.7396	−18.67
0.2849	−19.16	0.5511	−28.37	0.7864	−14.24
0.2882	−19.49	0.5868	−26.56	0.8099	−14.26
0.3469	−22.76	0.5956	−26.76	0.8805	−9.299
0.3506	−23.92	0.6302	−24.17	0.8950	−8.452
(1 − f_D) Tartaric Acid + f_D Tartaric Acid in Ethanol Solution 0.55149 wt %					
0.1194	−33.68	0.4169	−156.9	0.6954	−130.4
0.1251	−33.87	0.4486	−171.1	0.7137	−122.7
0.2133	−71.99	0.4618	−175.7	0.7740	−102.1
0.2224	−78.46	0.487	−191.1	0.7890	−91.73
0.2892	−106.2	0.4946	−193.4	0.7912	−89.73
0.3002	−109.8	0.5165	−194.1	0.8726	−60.75
0.3516	−130.2	0.5548	−184.1	0.8820	−53.28
0.3639	−136.8	0.5993	−173.9	0.8834	−59.27
0.4040	−154.6	0.6515	−154.8		
(1 − f_D) Tartaric Acid + f_D Tartaric Acid in Ethanol Solution at 1.2857 wt %					
0.1207	−17.44	0.4374	−72.19	0.6904	−47.67
0.2058	−30.58	0.4552	−76.80	0.7255	−46.16
0.2179	−35.20	0.4900	−81.90	0.7483	−41.72
0.2799	−45.71	0.5081	−83.53	0.7833	−39.61
0.2947	−49.68	0.5465	−70.29	0.7986	−32.99
0.3414	−55.22	0.5692	−72.07	0.8168	−29.85
0.3578	−60.60	0.5912	−64.09	0.8785	−17.71
0.3931	−65.30	0.6438	−56.48	0.8992	−13.13
0.4105	−69.98	0.6647	−55.19		
(1 − f_D) Tartaric Acid + f_D Tartaric Acid in Ethanol Solution at 5.1144 wt %					
0.08624	−3.526	0.4302	−14.85	0.6374	−14.12
0.1588	−6.223	0.4593	−15.72	0.6784	−12.89
0.2207	−8.525	0.4855	−16.41	0.7250	−11.47
0.2741	−10.12	0.5133	−16.48	0.7785	−10.01
0.3206	−11.55	0.5396	−16.06	0.8406	−6.968
0.3615	−12.29	0.5686	−15.65	0.9134	−3.970
0.3978	−13.86	0.6011	−15.07		

[a] $f(L) = n_{L\text{-tartaric acid}}/(n_{D\text{-tartaric acid}} + n_{L\text{-tartaric acid}})$.

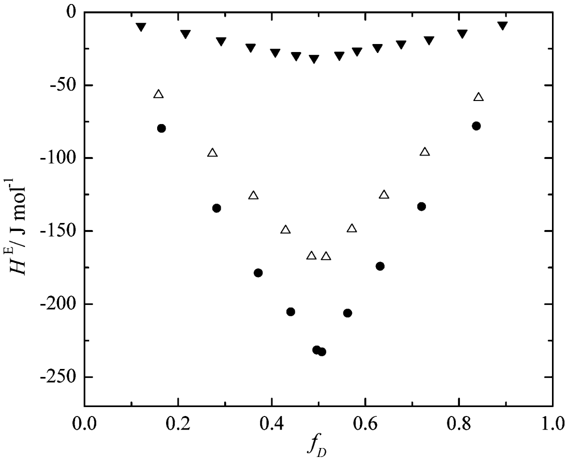

Figure 5. Comparison of the enthalpies of mixing of $(1 - f_{\rm D})$ tartaric acid + $f_{\rm D}$ tartaric acid aqueous solution at different concentration aqueous solutions: ●, 0.5 %; ▲, 1 %; ▼, 5 %.

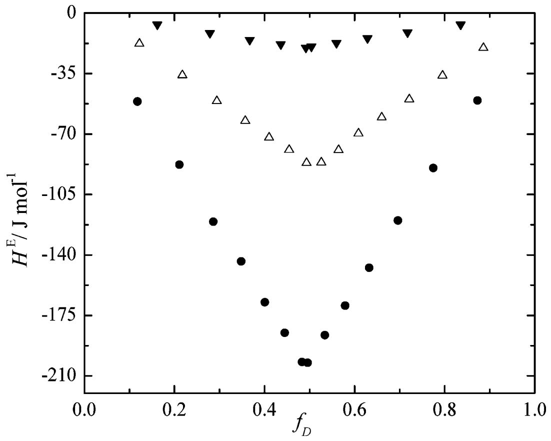

Figure 6. Comparison of the enthalpies of mixing of $(1 - f_{\rm D})$ tartaric acid + $f_{\rm D}$ tartaric acid ethanol solution at different concentration ethanol solutions: ●, 0.5 %; △, 1 %; ▼, 5 %.

Table 5. Enthalpies of Mixing of Aqueous and Ethanol Solutions of Chiral Tartaric Acids at $f_{\rm D} = 0.5^{a\prime}$

water		ethanol	
concentration	$H^{\rm E}$ $(f_{\rm D} = 0.5)$	concentration	$H^{\rm E}$ $(f_{\rm D} = 0.5)$
wt %	J·mol^{-1}	wt %	J·mol^{-1}
0.50911	−215.2	0.55149	−159.4
1.0107	−120.6	1.2857	−65.4
5.0045	−25.9	5.1144	−14.8

[a] $f_{\rm D} = n_{\rm L-tartaric\ acid}/(n_{\rm D-tartaric\ acid} + n_{\rm L-tartaric\ acid})$.

Correlation between minimum values of enthalpy of mixing and $m^{1/2}$ are plotted in Figure 7. An almost linear relation was shown for aqueous solution, but ethanol solution was far from a linear relation as shown in Figure 7.

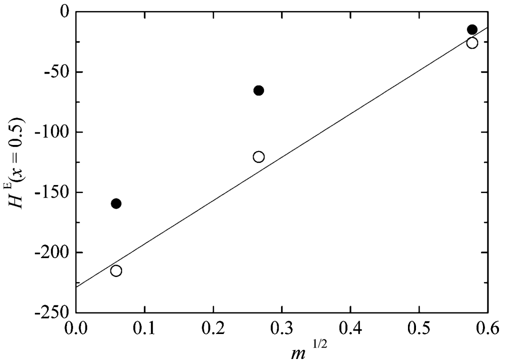

Figure 7. Correlation between excess enthalpies at equimolar concentration and $m^{1/2}$ of tartaric acid solution: ○, aqueous solution; ●, ethanol solution.

Tartaric acids in water dissociate to a carboxyl ion and show low pH as weak acid. Dissociation of weak acid is difficult for tartaric acids in aqueous solution, and it is probably more difficult in ethanol. This means that ionic tartaric acids are solvated with water in a solvation sphere that is strongly interacting with charged parts of carboxylic groups of tartaric acids. Access surface area of tartaric acid and water calculated by HyperChem[17] with a 14 pm water probe was 2574 pm^2 and 236 pm^2, respectively. Geometry optimization of molecular shapes of tartaric acids were carried out by calculations using the with AM1 method. Ratio of accessible surface area (ASA) of tartaric acid and water was 11.3 times. Surface fractions s = ASA(tartaric acid)/{ASA(tartaric acid) + ASA(water)} of solutions measured were 6.911×10^{-4}, 1.367×10^{-2}, and 6.675×10^{-2} for 0.050911, 1.0107, and 5.0045 wt % solution of tartaric acids, respectively. Surface fractions are corresponding to site fractions. Molecules of tartaric acids in aqueous solution may not contact directory, because tartaric acids could be strongly solvated with water. The solutions measured might have enough amounts of water to make the first solvated shell in this work. The waters in the first solvation shell of D- and L-tartaric acids solvated with water (D-TAS1 and L-TAS1, respectively) were strongly oriented to each enantiomer. Since the interaction between solvated D-TAS1 and L-TAS1 that had happened by the dilute system will happen also between D-TAS1 and D-TAS1 and between L-TAS1 and L-TAS1 if the concentration becomes high. It might be considered for decreasing the rate of the contacting between D-TAS1 and L-TAS1 relatively. Then enthalpies of mixing increased with increasing concentration of tartaric acid. When aqueous solutions of each enantiomer were mixed, the configuration of the second shell of tartaric acids changed largely. The concentration dependence of enthalpies of mixing might be explained by two factors. When concentrations of tartaric acid of weak acid in aqueous solution are decreased, dissociation rates of tartaric acids are increased. Contact number between D-TAS1 and L-TAS1 can increase in dilute solution; enthalpic stabilization on mixing increased with decreasing concentration of tartaric acids in aqueous solution. Also numbers of water in the second shell in the solution might be decreased with increasing concentration of tartaric acid. The orientation of water in the second shell might have an effect on enthalpic stabilization on mixing.

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