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Thermodynamic Functions, Solubility and Density of Catechol in Pure Water, Methanol and Their Binary Solvent Mixtures at 293.15 to 313.15K Temperatures

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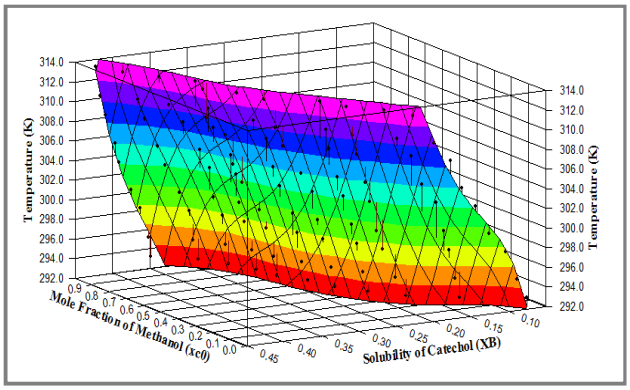
ABSTRACT

Solubility of catechol in water, methanol and in water-methanol binary mixtures have been experimentally measured using a gravimetric method at temperatures 293.15, 295.15, 298.15, 300.15, 303.15, 305.15, 308.15, 310.15 and 313.15K. Catechol solubility values are correlated with temperature by using the Apelblat equation. The combined nearly ideal binary solvent (NIBS)-Redlich-Kister equation is used to fit experimental solubility data in mixed solvents at constant temperature. Thermodynamic functions including ΔH^0_{soln}, ΔG^0_{soln}, and ΔS^0_{soln} of catechol in different solvents are obtained from the modified van't Hoff equation.

Graphical Abstract



Keywords: Catechol, Solubility, Density, Apelblat equation.

INTRODUCTION

Catechol naturally occurs in fruits and vegetables in small amounts, it is one of the main natural phenols in argan oil [1]. It is also found in *Agaricus bisporus* [2]. It is also a component of castoreum, a substance from castors, used in perfumery.

Solubility data is required for selection of proper solvent and design an optimized crystallization process, solubility of catechol in pure solvents for some temperatures are available [3, 4]. However there is no data available on solubility of catechol in water-methanol for the complete binary composition range. In this paper the systematic study of solubility and density of catechol in water+methanol binary solvents over the entire composition range from 0 to 1 mole fraction, at temperatures 293.15 to 313.15K is reported. The thermodynamic functions for saturated catechol solution are calculated using modified van't Hoff equation.

MATERIALS AND METHODS

Material: Triple distilled water was used in all experiments. Other chemicals was supplied by

Chemical Name	Supplier Name	Percentage purity	Standard
Catechol	Sigma-Aldrich co.	≥99%	Reagent Grade
Methanol	Merck,Darmstadt, Germany.	≥99.8%	G.R.

Apparatus and Procedure: The solubility of catechol was measured using an apparatus similar to that described in the literature [5, 6]. In this work, an excess amount of catechol was added to the binary solvents mixtures prepared by weight (Shimadzu, Auxzzo) with an uncertainty of ± 0.1 mg in a specially designed 100 mL double jacketed flask. Water was circulated at constant temperature in jacket between the outer and inner walls of the flask. The temperature of the circulating water was controlled by auto temperature control thermostat within (± 0.1) K. The solution was continuously stirred using a magnetic stirrer for about 1 h so that equilibrium is assured and no further solute dissolved, and the temperature of solution is same as that of circulating water the stirrer was switched off and the solution was allowed to stand for 1 h. Then a fixed quantity of the supernatant liquid was withdrawn from the flask in a weighing bottle with the help of pipette which is hotter than the solution. The weight of this sample was taken and kept in an oven at 343 K until the whole solvent was evaporated and the residue was completely dry. This was confirmed by weighing two or three times until a constant weight was obtained after keeping the sample in an oven for another 30 min every time. The solubility has been calculated using weight of solute and weight of solution. Each experimental value of solubility is an average of at least three different measurements and the standard uncertainty of the experimental mole fraction solubility(x_B), value is ± 0.003. The mole fraction solubility(x_B), initial the mole fraction of methanol/ethanol/1-propanol (x_C^0), were calculated using usual equations [7]. The standard uncertainty for x_C^0 is 0.0002. Densities were determined using a 15 cm^3 bicapillary pycnometer as described earlier. For calibration of pycnometer triply distilled and degassed water with a density of 0.99705 $g{\cdot}cm^{-3}$ at 298.15 K was used. The pycnometer filled with air bubble free experimental liquids was kept in a transparent walled thermostat (maintained at constant temperature ± 0.1 K) for (10 to 15) min to attain thermal equilibrium. The heights of the liquid levels in the two arms were measured with the help of a traveling microscope, which could read to 0.01 *mm*. The estimated standard uncertainty of the density measurements of the solvent and binary mixtures was 10 $kg{\cdot}m^{-3}$ [8-11].

RESULTS AND DISCUSSION

Solubility: Table 1 shows the experimental and calculated (using Apelblat equation) values of solubility (x_B) of catechol at 293.15 to 313.15K in water, methanol and water+methanol respectively. The density of each saturated solution is also reported. Variation of solubility with x_C^0 is visually shown in figure 1.

The solubility of catechol in all solvents increases with temperature. At the same temperature, the solubility trend in solvent is methanol >water + methanol>water. This trend implies that solubility of catechol increases with increasing with mole fraction of methanol, it is prefer to dissolve more in methanol than water. The solubility of catechol in water-methanol mixture with x_C^0 increases with

increases in x_B upto x_C^0=1. This implies that there is strong dipole-dipole interaction between solute and solvent molecules.

Table 1. Experimental $x_{B(exp.)}$ and Calculated $x_{B(cal.)}$ Values of Mole Fraction Solubility and Density (ρ) of Catechol for Various Initial Mole Fractions, (x_C^0), of Methanol at Temperatures (293.15 to 313.15) K and Pressure 101.32 *kPa*[a].

T/(K)	x_C^0	$x_{B(exp.)}$	$x_{B(cal.)}$	*RD*	$\rho \cdot 10^{-3}$/ kg·m^{-3}	T/(K)	x_C^0	$x_{B(exp.)}$	$x_{B(cal.)}$	*RD*	$\rho \cdot 10^{-3}$/ kg·m^{-3}
	0.0000	0.0752	0.0749	0.0048	1.0732		0.0000	0.1621	0.1618	0.0018	1.1158
	0.1000	0.1426	0.1423	0.0025	1.0986		0.1000	0.1926	0.1899	0.0144	1.1206
	0.2000	0.1907	0.1922	0.0078	1.1021		0.2000	0.2461	0.2496	0.0139	1.1166
	0.3011	0.2188	0.2177	0.0053	1.0989		0.3011	0.2601	0.2596	0.0018	1.1112
	0.4000	0.2399	0.2393	0.0024	1.0957		0.4000	0.2858	0.2879	0.0074	1.1057
293.15	0.5054	0.2518	0.2528	0.0038	1.0907	305.65	0.5054	0.2992	0.2977	0.0049	1.0995
	0.5999	0.2716	0.2682	0.0124	1.0854		0.5999	0.3309	0.3277	0.0097	1.0946
	0.7012	0.2762	0.2774	0.0042	1.0795		0.7012	0.3108	0.3165	0.0183	1.0875
	0.8021	0.2903	0.2898	0.0017	1.0746		0.8021	0.3286	0.3314	0.0085	1.0811
	0.9002	0.3076	0.3057	0.0062	1.0691		0.9002	0.3631	0.3616	0.0042	1.0762
	1.0000	0.3484	0.3447	0.0108	1.0647		1.0000	0.3963	0.3969	0.0016	1.0709
	0.0000	0.0888	0.0895	0.0078	1.0828		0.0000	0.1781	0.1823	0.0239	1.1224
	0.1000	0.1488	0.1484	0.0026	1.1027		0.1000	0.2070	0.2057	0.0065	1.1231
	0.2000	0.2050	0.2054	0.0022	1.1042		0.2000	0.2573	0.2576	0.0012	1.1196
	0.3011	0.2255	0.2261	0.0026	1.1013		0.3011	0.2714	0.2680	0.0125	1.1137
	0.4000	0.2451	0.2461	0.0043	1.0969		0.4000	0.3102	0.3026	0.0244	1.1080
295.65	0.5054	0.2602	0.2589	0.0048	1.0923	308.15	0.5054	0.3075	0.3114	0.0128	1.1011
	0.5999	0.2764	0.2781	0.0062	1.0869		0.5999	0.3410	0.3429	0.0058	1.0952
	0.7012	0.2824	0.2827	0.0011	1.0812		0.7012	0.3340	0.3285	0.0164	1.0886
	0.8021	0.2942	0.2937	0.0017	1.0753		0.8021	0.3505	0.3470	0.0098	1.0821
	0.9002	0.3155	0.3147	0.0024	1.0697		0.9002	0.3799	0.3763	0.0094	1.0777
	1.0000	0.3521	0.3556	0.0098	1.0653		1.0000	0.4097	0.4066	0.0074	1.0730
	0.0000	0.1036	0.1057	0.0201	1.0911		0.0000	0.2003	0.2032	0.0144	1.1280
	0.1000	0.1563	0.1560	0.0018	1.1072		0.1000	0.2267	0.2243	0.0105	1.1275
	0.2000	0.2222	0.2179	0.0194	1.1081		0.2000	0.2634	0.2643	0.0034	1.1224
	0.3011	0.2338	0.2345	0.0030	1.1049		0.3011	0.2754	0.2763	0.0030	1.1158
	0.4000	0.2563	0.2543	0.0081	1.0999		0.4000	0.3200	0.3193	0.0022	1.1093
298.15	0.5054	0.2668	0.2664	0.0015	1.0937	310.65	0.5054	0.3251	0.3271	0.0062	1.1026
	0.5999	0.2858	0.2890	0.0111	1.0885		0.5999	0.3660	0.3595	0.0178	1.0973
	0.7012	0.2919	0.2892	0.0094	1.0826		0.7012	0.3404	0.3420	0.0049	1.0903
	0.8021	0.2978	0.2997	0.0063	1.0767		0.8021	0.3706	0.3656	0.0137	1.0853
	0.9002	0.3197	0.3248	0.0159	1.0713		0.9002	0.3889	0.3924	0.0091	1.0798
	1.0000	0.3632	0.3663	0.0085	1.0662		1.0000	0.4166	0.4160	0.0016	1.0752
	0.0000	0.1263	0.1232	0.0242	1.0997		0.0000	0.2280	0.2241	0.0171	1.1328
	0.1000	0.1633	0.1653	0.0123	1.1137		0.1000	0.2432	0.2462	0.0127	1.1303
	0.2000	0.2302	0.2296	0.0025	1.1112		0.2000	0.2715	0.2695	0.0075	1.1254
	0.3011	0.2410	0.2429	0.0077	1.1062		0.3011	0.2832	0.2845	0.0046	1.1184
	0.4000	0.2635	0.2639	0.0015	1.1016		0.4000	0.3350	0.3382	0.0093	1.1122
300.65	0.5054	0.2725	0.2753	0.0100	1.0963	313.15	0.5054	0.3475	0.3448	0.0078	1.1060
	0.5999	0.2954	0.3008	0.0182	1.0907		0.5999	0.3717	0.3774	0.0153	1.0988
	0.7012	0.2995	0.2969	0.0087	1.0842		0.7012	0.3579	0.3573	0.0018	1.0930
	0.8021	0.3102	0.3079	0.0074	1.0789		0.8021	0.3829	0.3873	0.0115	1.0871
	0.9002	0.3329	0.3359	0.0089	1.0731		0.9002	0.4095	0.4099	0.0010	1.0811
	1.0000	0.3753	0.3768	0.0038	1.0678		1.0000	0.4224	0.4250	0.0062	1.0764
	0.0000	0.1445	0.1420	0.0172	1.1089						
	0.1000	0.1741	0.1765	0.0138	1.1167		0.5999	0.3187	0.3137	0.0158	1.0916
	0.2000	0.2399	0.2402	0.0012	1.1146		0.7012	0.3035	0.3060	0.0082	1.0854
303.15	0.3011	0.2516	0.2513	0.0012	1.1089	303.15	0.8021	0.3158	0.3184	0.0083	1.0803
	0.4000	0.2710	0.2751	0.0152	1.1041		0.9002	0.3525	0.3481	0.0123	1.0743
	0.5054	0.2895	0.2857	0.0134	1.0977		1.0000	0.3908	0.3870	0.0098	1.0696

[a]Standard uncertainties in u are u(T) = 0.1 K, u(x_C^0) = 0.0002, u(x_B) = 0.003, and u(ρ) = 10 kg·m^{-3}.
The relative uncertainty in pressure ur(p) = 0.05.

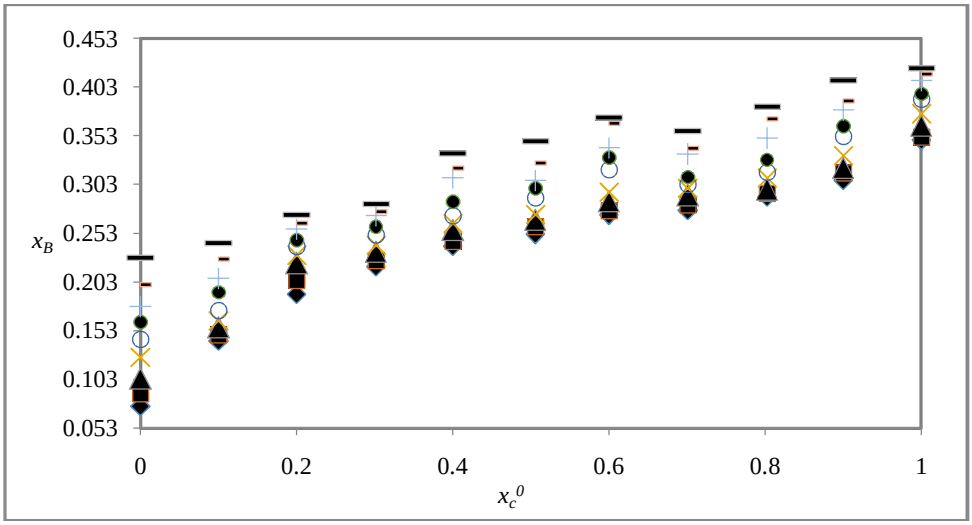


Figure 1. Mole Fraction Solubility (x_B) Variation with Initial Mole Fraction (x_C^0) of Methanol at Various Temperatures (◆T=293.15 K, ■T=295.65 K; ▲T=298.15 K; ×T=300.65 K; ○T=303.15 K;●T=305.65 K; +T=308.15 K; -T=310.65 K and ▬T=313.15 K

Apelblat Model: Between the different methods, the modified semi-empirical Apelblat model (eq 1) is a suitable way to correlate solubility data against temperature [12, 13].The equation is based on solid-liquid equilibrium theory provide excellent agreement between experimental and calculated values of solubility [14].

$$lnx_{B=} A + \frac{B}{T} + ClnT \qquad ...(1)$$

A, *B*, and *C* are the model parameters and T is temperature in Kelvin. *A* and *B* represent the non-idealities of the solutions in terms of the variation of activity coefficients, *C* reflects to the effect of temperature on the enthalpy of fusion [15]. *A*, *B*, and *C* parameters are determined using non-linear least square fitting [16]. Solubility values of catechol in water, methanol, and their mixtures are calculated by eq 1. Relative deviation (*RD)* [17] is calculated using eq 2.

$$RD = \frac{x_B^{exp.} - x_B^{cal.}}{x_B^{exp.}} \qquad ...(2)$$

The data of experimental mole fraction solubility, calculated solubility and *RD* in monosolvent (water, methanol) and water-methanol mixtures are listed in table 1. The values of parameters *A*, *B*, *C* along with co-relation coefficient (R^2) are listed in table 2.

Table 2. Model Parameters and Correlation Coefficient of the Apelblat Equation

Solvents	Mole fraction x_C^0	Parameters			R^2
		A	B	C	
Methanol	0.0000	926.5154	-46178.9730	-135.8253	0.9971
	0.1000	-827.1516	35101.5677	124.1861	0.9959
	0.2000	599.0465	-28415.8039	-88.6800	0.9914
	0.3011	66.3771	-4103.3377	-9.4891	0.9941
	0.4000	-481.4889	20317.1737	72.3069	0.9876
	0.5054	-466.2955	19771.2563	69.9698	0.9913
	0.5999	-239.8662	9436.8040	36.3265	0.9811
	0.7012	-396.9150	16867.5593	59.5164	0.9812
	0.8021	-684.8053	29715.8102	102.4876	0.9895
	0.9002	-261.4324	10602.1830	39.4461	0.9884
	1.0000	95.8429	-5186.3584	-13.9448	0.9842

NIBS-Redlich-Kister Model: The solubility data at constant temperature is fitted into combined NIBS-Redlich-Kister model [18-21].

$$lnx_B = x_C^0 lnx_1 + x_A^0 lnx_2 + x_C^0 x_A^0 \sum_{i=0}^{3} M_i (x_C^0 - x_A^0)^i \quad ...(3)$$

Where x_A^0 is initial mole fraction of water and x_1,x_2 are solubilities of catechol in pure methanol and water, respectively. M_i is curve fit parameters (four parameter equation). All values of M_i along with R^2 value are listed in table 3. The values of R^2 are close to unity shows that NIBS-Redlich-Kister model is very well applicable for this solubility data.

Table 3. NIBS-Redlich-Kister model parameters

T/K	Range of x_C^0	M_0	M_1	M_2	M_3	R^2
Water + Methanol + Catechol						
293.15	0.1-0.9	1.769	-1.901	1.662	-1.996	0.999
295.15	0.1-0.9	1.554	-1.935	1.147	-0.856	0.995
298.15	0.1-0.9	1.378	-1.960	0.478	-0.212	0.973
300.15	0.1-0.9	1.064	-1.766	-0.229	0.867	0.909
303.15	0.1-0.4	3.2561	21.371	58.267	43.915	1.000
	0.5-0.9	0.7609	2.0196	12.226	10.575	0.987
305.65	0.1-0.4	2.6961	16.433	42.87	31.37	1.000
	0.5-0.9	0.644	2.0208	-11.745	10.34	0.954
308.15	0.10-0.90	0.670	-1.126	-0.330	1.016	0.871
310.65	0.1-0.4	2.7414	16.344	36.282	24.075	1.000
	0.5-0.9	0.4784	1.5596	-7.564	6.2413	0.711
313.15	0.1-0.4	2.6617	16.345	33.99	22.085	1.000
	0.5-0.9	0.4503	0.8352	-6.1381	6.1838	0.820

Thermodynamics Functions of Dissolution: According to the van't Hoff equation, the standard molar enthalpy change of solution ΔH^0_{soln} is generally obtained from the slope of the $ln\ x_B$ vs $1/T$ plot. Average temperature T_{mean} is introduced to obtain a single value of ΔG^0_{soln} and ΔS^0_{soln} in the temperature range studied.

$$T_{mean} \frac{n}{\sum_{i-1}^{n}\left(\frac{1}{T}\right)} \quad ... (4)$$

Where n is the number of experimental points. In the present work, T_{mean}=302.98 K and the temperature range is (293.15 to 313.15) K in both pure solvents and binary solvent mixtures. Heat capacity of the solution can be assumed as constant. Hence values of ΔH^0_{soln} are derived using eq 5.

$$\Delta H^0_{sol} = -R\left(\frac{\partial lnx_B}{\partial 1/T}\right) - R\left[\frac{\partial lnx_B}{\partial\left(\frac{1}{T}-\frac{1}{T_{mean}}\right)}\right] \quad \ldots (5)$$

The lnx_B Vs 10000 ($1/T - 1/T\ mean$) plot of different solutions including pure solvents and binary solvent mixtures are displayed in figures 2. From these figures, it can be seen that a trend of increasing solubility with temperature is observed. The slope and the intercept for each solvent are listed in table 4. Thus the modified van't Hoff equation can be thought to be fit to calculate the enthalpy change of solution. The standard molar Gibbs energy change for the solution process ΔG^0_{soln}, can be calculated in the way similar to Krug *et al* [22] as

$$\Delta G^0_{sol} = -RT \times intercept \quad ... (6)$$

In which the intercept used is that obtained in plots of lnx_B as a function of ($1/T - 1/T\ mean$). The standard molar entropy change ΔS^0_{soln} is obtained from

$$\Delta S^0_{Sol} = \frac{\Delta H^0_{Sol} - \Delta H^0_{Sol}}{T_{mean}} \quad \ldots(7)$$

Both ΔG^0_{soln} and ΔS^0_{soln} pertain to the mean temperature T_{mean}=302.92 K. The results are shown in table 5, together with $\%\zeta H$ and $\%\zeta TS$. It is worthy to note that relative contribution of enthalpy $\%\zeta H$ and $\%\zeta\, TS$ which are defined as

$$\%\zeta_H = \frac{\Delta H^0_{Sol}}{|\Delta H^0_{Sol}| + |T\Delta S^0_{Sol}|} X100 \quad \ldots(8)$$

$$\%\zeta_{TS} = \frac{|T\Delta S^0_{Sol}|}{|\Delta H^0_{Sol}| + |T\Delta S^0_{Sol}|} X100 \quad \ldots(9)$$

can be simply used to calculate the main contributors of enthalpy or entropy to ΔG°_{soln} [23]. The values of ΔH° and ΔS° for all solutions are positive indicating the solution process as endothermic. The contribution of enthalpy to positive molar Gibbs energy is more as compared to entropy for all solutions. Density values are used to calculate excess molar functions [24].

Table 4. Slope (m) and Intercept (c) of the lnx_B vs. $10000(1/T - 1/T_{mean})$ Plot along with R^2

Water + Methanol			
x_c^0	m	c	R^2
0.0000	-5032	-1.982	0.992
0.1000	-2518	-1.706	0.977
0.2000	-1551	-1.446	0.968
0.3011	-1228	-1.383	0.995
0.4000	-1587	-1.274	0.974
0.5054	-1425	-1.237	0.974
0.5999	-1567	-1.151	0.981
0.7012	-1162	-1.170	0.965
0.8021	-1331	-1.121	0.947
0.9002	-1347	-1.046	0.984
1.0000	-961.1	-0.952	0.986

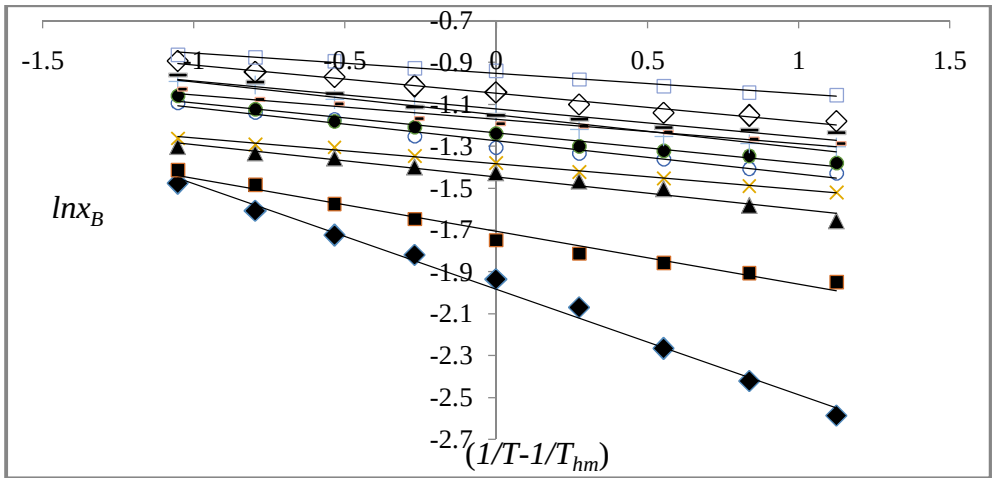


Figure 2. Plot of lnx_B vs. $(1/T - 1/T_{hm})$ for Catechol + Water + Methanol System at various Mole fractions. (◆x_C^0=0.0000; ■ x_C^0=0.1000; ▲ x_C^0=0.2000; × x_C^0=0.3011; ○x_C^0=0.4000; ● x_C^0=0.5054; + x_C^0=0.5999; - x_C^0=0.7012; ▬ x_C^0=0.8021 and◇x_C^0=0.9002; □x$_C^0$=1.0000).

Table 5. Thermodynamic Functions Relative to Solution Process of Catechol at *T mean* = 302.928K

x_C^0	ΔH^0sol/kJ·K^{-1}·mol^{-1}	ΔG^0soln/kJ·K^{-1}·mol^{-1}	ΔS^0soln/KJ·K^{-1}·mol^{-1}	$T\Delta S^0$soln/KJ·K^{-1}·mol^{-1}	%ζH	%ζTS
Water + Methanol						
0.0000	41.8194	4.9954	121.4712	36.8240	53.18	46.82
0.1000	20.8681	4.2998	54.6540	16.5684	55.74	44.26
0.2000	12.8867	3.6445	30.4873	9.2422	58.23	41.77
0.3011	10.1431	3.4857	21.9607	6.6574	60.37	39.63
0.4000	13.1361	3.2110	32.7400	9.9251	56.96	43.04
0.5054	11.8059	3.1177	28.6596	8.6882	57.61	42.39
0.5999	12.9698	2.9010	33.2142	10.0689	56.30	43.70
0.7012	9.6442	2.9489	22.0860	6.6954	59.02	40.98
0.8021	11.0576	2.8254	27.1557	8.2323	57.32	42.68
0.9002	11.1408	2.6363	28.0535	8.5044	56.71	43.29
1.0000	7.9814	2.3994	18.4134	5.5820	58.85	41.15

APPLICATION

Solubility data and Thermodynamic functions including ΔH^0_{soln}, ΔG^0_{soln}, and ΔS^0_{soln} of catechol in water, methanol and their mixture are more useful in field of physical chemistry and chemical engineering calculations involving fluid flow, heat and mass transfer, pharmaceutical industry, agriculture, biology, medicine. Solubility data is required for selection of proper solvent and design an optimized crystallization process.

CONCLUSION

The solubility of catechol is more in methanol than in water and increases with increase in mole fraction of methanol. ΔH^0_{soln} values are higher in water than water+methanol mixture and lowest in methanol indicates the solubility trend in various solvents. Density of solution is depends on solubility and solvent system both.

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