

Solubility of Tris(hydroxymethyl)aminomethane in Water + 1-Propanol Mixtures at Various Temperatures

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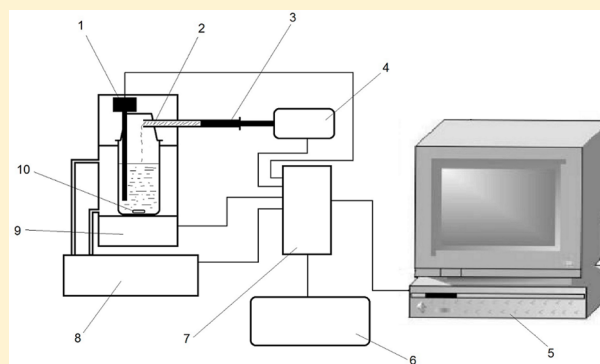
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ABSTRACT: Solubility of tris(hydroxymethyl)aminomethane (TRIS) in various mass fraction compositions of water + 1-propanol solvent mixtures at (293.2, 298.2, 303.2, 308.2, and 313.2) K was measured using a laser monitoring technique. The generated data were mathematically represented using the Jouyban–Acree model. The correlated mole fraction solubilities are in good agreement with the corresponding experimental values as documented by an overall mean percentage deviation of 2.3 %. Densities of TRIS saturated solutions in water and 1-propanol at various temperatures were measured and employed to predict the density of TRIS saturated solutions in their binary solvent mixtures using a well-established method.



INTRODUCTION

Buffering agents dissolved in mixed solvents are used as mobile phases and/or background electrolytes in analytical separation methods such as high performance liquid chromatography (HPLC) or capillary electrophoresis (CE) where the low solubility of the buffering agents at higher concentrations of the organic solvent may cause practical problems. The addition of organic solvents to aqueous solutions may improve the solubility of the analyte, the resolution of the peaks of various analytes, or determine other relevant analytical parameters like pK_a values, partition coefficients, or electrophoretic and electroosmotic mobilities.¹

Tris(hydroxymethyl)aminomethane (TRIS), the chemical abstract service number of 77-86-1 and acid dissociation constant (pK_a) of 8.1, is a common buffering agent in chemical and biochemical areas. It possesses good solubility in water; however, in many applications, organic solvents are added to the aqueous solutions to alter some other parameters. The addition of organic solvents to the aqueous solutions causes some solubility problems for TRIS, and the solubility data of TRIS in water + organic solvent mixtures are required in practice. The available solubility data of TRIS in various solvent mixtures is reviewed in a recent publication along with a report of TRIS solubility in water + methanol mixtures at 293.2 K to 313.2 K.² A quick survey on the published HPLC and CE methods for pharmaceutical analysis showed that TRIS buffer

has been used in water +1-propanol (1-PrOH) with considerable frequency.³

Owing to practical importance and to continue our systematic investigations on the solubility of solutes in solvent mixtures, the solubility of TRIS in aqueous mixtures of 1-PrOH at different temperatures is measured using a lab-made setup. This setup has been validated using measured solubilities of acetaminophen (as a nonelectrolyte) and TRIS (as an electrolyte) and their comparison with previously reported data taken from the literature.^{2,4} To provide a predictive tool for the calculation of the solubility of TRIS at any composition of the binary solvent mixture and temperatures, the data are fitted to the Jouyban–Acree model and its combined version with the van't Hoff equation.

EXPERIMENTAL SECTION

Materials. TRIS (with the stated purity of 0.999 in mass fraction) was purchased from Merck (Germany). 1-PrOH (0.999 mass fraction purity) was purchased from Scharlau (Barcelona, Spain). Double distilled water was used for the preparation of the solutions. All reagents were used as received

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Table 1. List of the Used Materials

material	purity	unit	company	country
TRIS (CAS no. 77-86-1)	0.999	mass fraction	Merck	Germany
1-PrOH	0.999	mass fraction	Scharlau	Spain
water	conductivity < 1.5	$\mu\text{S}\cdot\text{cm}^{-1}$	lab made	

Table 2. Experimental Mole Fraction Solubility^a of Tris(hydroxymethyl)aminomethane (TRIS) in Binary Mixtures of Water (1) + 1-PrOH (2) at Various Temperatures

x_1^0	293.2 K	SD	298.2 K	SD	303.2 K	SD	308.2 K	SD	313.2 K	SD
1.00	0.0831	0.0003	0.0920	0.0001	0.1013	0.0001	0.1099	0.0013	0.1228	0.0001
0.97	0.0826	0.0003	0.0907	0.0001	0.0990	0.0003	0.1079	0.0001	0.1196	0.0009
0.93	0.0796	0.0017	0.0868	0.0013	0.0976	0.0024	0.1061	0.0007	0.1187	0.0016
0.89	0.0763	0.0022	0.0828	0.0024	0.0931	0.0020	0.1035	0.0026	0.1158	0.0031
0.83	0.0682	0.0019	0.0756	0.0021	0.0853	0.0013	0.0970	0.0013	0.1100	0.0008
0.77	0.0585	0.0009	0.0675	0.0007	0.0767	0.0001	0.0867	0.0009	0.0982	0.0008
0.69	0.0503	0.0008	0.0594	0.0006	0.0660	0.0011	0.0752	0.0012	0.0858	0.0014
0.59	0.0401	0.0009	0.0446	0.0008	0.0516	0.0013	0.0592	0.0006	0.0687	0.0023
0.45	0.0246	0.0011	0.0286	0.0003	0.0331	0.0006	0.0399	0.0005	0.0460	0.0013
0.27	0.0138	0.0007	0.0175	0.0001	0.0202	0.0005	0.0238	0.0003	0.0278	0.0006
0.00	0.0038	0.0002	0.0050	0.0002	0.0063	0.0001	0.0072	0.0001	0.0088	0.0001

^aThe relative standard uncertainty for the solubilities is 1.7% or $u_r(x) = 0.017$, the standard uncertainty for temperature is 0.1 K and the measurements were made at atmospheric pressure. Data are the mean of three measurements. SD: standard deviation. x_1^0 is the mole fractions of water in the absence of TRIS.

from the company without further purification. Table 1 lists the details of materials used in this work.

Instrumentation. The thermodynamic solubility of a solute could be determined employing various techniques which are summarized in a recent work.⁵ The most common method is the shake-flask method of Higuchi and Connors which requires several time-consuming steps.⁶ The synthetic method⁷ is an alternative common method for the determination of thermodynamic solubility of a solute. It is based on the disappearance of the solid solute from the solution as monitored by a laser beam. TRIS solubility in water +1-PrOH mixtures is determined using a lab-made setup based on a synthetic method which is described in earlier works.^{2,4,8} Briefly the filled syringe with TRIS powder was weighed using an electronic balance (Sartorius, Germany) with an uncertainty of 0.01 g before and after saturation of the solution, and the mass difference determines the mass of solute added to saturate the solution. It should be noted that the total masses of the solvent mixtures were 150 g and those of the solute + syringe varied between 0.10 g and 4.00 g. The saturated concentration of the solution is calculated using the mass of the solvent added to the dissolution vessel. After reaching the experimental temperature, the setup adds a small amount of TRIS to a given mass of the solvent and/or solvent mixture, and the contents were stirred continuously at a constant temperature with the uncertainty of ± 0.1 K. As the particles of TRIS are dissolved, the signals indicating the number of particles decreases gradually and reaches the minimum value when TRIS is completely dissolved. Then another mass of TRIS is dispensed to the vessel, and the procedure is repeated until the laser beam can not return to the minimum value, which means the last added powder could not be dissolved. This cycle was checked several times by the software, and then the system was stopped and the total amount of the added drug was recorded and used to calculate the solubility value. The signals for neat solvents are considered as the minimum intensity of the signals detected by

the photo converter. Further details of the setup have been provided in earlier works.^{2,4}

Calculations. The average of at least triplicate measurements was used to calculate the mole fraction solubility of TRIS. The solvents 1 and 2 masses (m_1 and m_2) were divided to their molar masses (MW_1 and MW_2), and then the mole fractions of solvents 1 and 2 in the absence of the solute (x_1^0 and x_2^0) were computed using

$$x_i^0 = \frac{m_i/MW_i}{(m_1/MW_1 + m_2/MW_2)} \quad (1)$$

The saturated mole fraction solubility of TRIS ($x_{m,T}$) in different compositions of the binary solvents at various temperatures was calculated using

$$x_{m,T} = \frac{m_{\text{TRIS}}/MW_{\text{TRIS}}}{(m_{\text{TRIS}}/MW_{\text{TRIS}} + m_1/MW_1 + m_2/MW_2)} \quad (2)$$

where m_{TRIS} and MW_{TRIS} are the mass of added TRIS to the solution and molar mass of TRIS ($121.14 \text{ g}\cdot\text{mol}^{-1}$), respectively. Evaluation of the accuracy of the calculated data was performed by computing the mean percentage deviations (MPD) between the calculated and experimental solubilities according to

$$\text{MPD} = \frac{100}{N} \sum \left(\frac{x_{m,T}^{\text{calculated}} - x_{m,T}^{\text{experimental}}}{x_{m,T}^{\text{experimental}}} \right) \quad (3)$$

where N is the number of data points in each set.

RESULTS AND DISCUSSION

Table 2 lists the experimental mole fraction solubility of TRIS in various mole fractions of water (1) + 1-PrOH (2) mixtures at (293.2, 298.2, 303.2, 308.2, and 313.2) K. There are good agreements between measured solubility data of TRIS in aqueous solutions at various temperatures and those reported in the literature. This comparison was made in an earlier work.² As expected, the solubility of TRIS in each solvent composition

and also in the monosolvents increased with increasing temperature. The solubility data of TRIS in each solvent system at various temperatures (x_T) could be mathematically represented using the van't Hoff equation⁹

$$\ln x_T = A + \frac{B}{T} \quad (4)$$

where A and B are the model constants calculated using a least-square method. Table 3 lists the model constant and the MPD

Table 3. Model Constants for van't Hoff Equation for Various Mole Fractions of Water (x_1^0) and the Obtained Mean Percentage Deviation (MPD) for Back-Calculated Solubilities Using eq 4

x_1^0	A	B	MPD
1.00	3.515	-1759.978	0.5
0.97	3.231	-1679.231	0.5
0.93	3.721	-1834.986	0.8
0.89	4.031	-1940.103	1.0
0.83	4.849	-2212.741	0.9
0.77	5.209	-2357.897	0.2
0.69	5.187	-2394.180	0.9
0.59	5.280	-2496.087	1.3
0.45	6.224	-2914.011	1.1
0.27	6.417	-3129.026	1.4
0.00	7.224	-3740.048	3.2
overall			1.1

values for the back-calculated data using eq 4. Figure 1 shows the van't Hoff plot of the measured solubilities in the

investigated solvent systems at different temperatures. There are very good linear relationships between $\ln x_T$ and $1/T$ as expected for the temperature range in this study.

Using the combined nearly ideal binary solvent/Redlich–Kister equation,¹⁰ the solubility of TRIS in various compositions of water (1) + 1-PrOH (2) mixtures at a given temperature could be represented as

$$\ln x_m = x_1^0 \ln x_1 + x_2^0 \ln x_2 + x_1^0 x_2^0 \left[\sum_{i=0}^2 S_i (x_1^0 - x_2^0)^i \right] \quad (5)$$

where x_m , x_1 , and x_2 are the mole fraction solubility of TRIS in the solvent mixtures, the monosolvents 1 and 2 at a given temperature, and S_i are the constants of the model calculated using a no-intercept least-square analysis. Table 4 lists the numerical values of the model constants of eq 5 and the obtained MPD values for the back-calculated solubility data.

Table 4. Model Constants of eq 5^a and the MPD Values for Back-Calculated Solubilities in Water +1-PrOH Mixtures at Various Temperatures (K)

T/K	S_0	MPD
293.2	2.169	2.3
298.2	2.016	2.2
303.2	1.949	2.2
308.2	2.159	1.8
313.2	2.119	1.7
overall		2.0

^a S_1 and S_2 values are not statistically significant ($p > 0.10$).

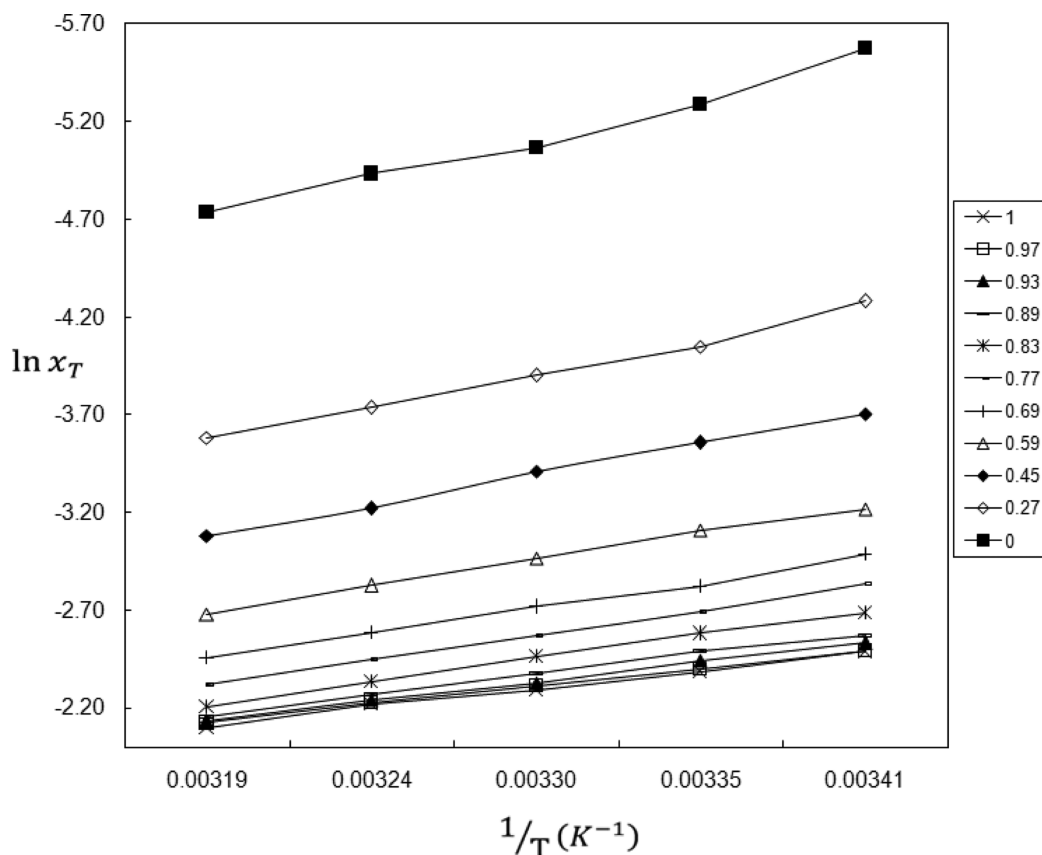


Figure 1. Logarithm of the experimental mole fraction solubility of TRIS in water + 1-PrOH mixtures as a function of temperature.

The model has been extended to represent both solvent composition and temperature effects on solubility as¹¹

$$\ln x_{m,T} = x_1^0 \ln x_{1,T} + x_2^0 \ln x_{2,T} + \frac{x_1^0 x_2^0}{T} \left[\sum_{i=0}^2 J_i (x_1^0 - x_2^0)^i \right] \quad (6)$$

where $x_{m,T}$ is the mole fraction solubility in the solvent mixtures at absolute temperature T , $x_{1,T}$ and $x_{2,T}$ denote the mole fraction solubility of TRIS in the monosolvents 1 and 2, respectively, and J_i are the constants (expressed in K unit) of the model computed by a regression analysis. The model has been used to represent other physicochemical properties and renamed the Jouyban–Acree model.¹² Equation 6 is able to predict the solubility of a solute in mixed solvents at various T once its coefficients are obtained from a training process. The only required input data are the $x_{1,T}$ and $x_{2,T}$ values. The solubility data at all temperatures were fitted to eq 6 and the obtained model is

$$\ln x_{m,T} = x_1^0 \ln x_{1,T} + x_2^0 \ln x_{2,T} + \frac{x_1^0 x_2^0}{T} [624.281 + 37.824(x_1^0 - x_2^0)] \quad (7)$$

which correlated the solubility data with the correlation coefficient of 0.996 which is a significant correlation with $p < 0.0005$. The obtained MPD value for the back-calculated solubility data was $2.3 \pm 2.0\%$ ($N = 55$). As it has been shown in earlier works, eq 6 could be trained using solubility data at 298 K and the solubility at other temperatures could be predicted employing the experimental solubility data in the monosolvents at other temperatures.^{2,13–15} The MPD for the predicted solubility data was $2.7 \pm 2.3\%$ ($N = 36$), obtained by employing experimental values of $x_{1,T}$ and $x_{2,T}$ at any temperature of interest.

The Jouyban–Acree model was combined with the van't Hoff model as^{2,16,17}

$$\ln x_{m,T} = x_1^0 \left(A_1 + \frac{B_1}{T} \right) + x_2^0 \left(A_2 + \frac{B_2}{T} \right) + \frac{x_1^0 x_2^0}{T} \left[\sum_{i=0}^2 J_i (x_1^0 - x_2^0)^i \right] \quad (8)$$

to provide a more versatile predictive equation where no further experimental data is required after the training process of the model. The trained model from combining trained van't Hoff equations using $x_1^0 = 0.00$ and $x_1^0 = 1.00$ and the J terms from eq 7 is

$$\ln x_{m,T} = x_1^0 \left(3.515 - \frac{1759.978}{T} \right) + x_2^0 \left(7.224 - \frac{3740.048}{T} \right) + \frac{x_1^0 x_2^0}{T} [624.281 + 37.824(x_1^0 - x_2^0)] \quad (9)$$

which back-calculates the solubility data with the MPD of $2.3 \pm 2.0\%$ ($N = 55$). Equation 8 could be trained using the solubility data at two temperatures (the lowest and highest temperatures of interest) and then be used to predict the solubility of other temperatures and solvent compositions using the interpolation technique.² The obtained MPD value for this predictive analysis is $2.2 \pm 2.4\%$ ($N = 33$). The main advantage of eq 9 over eq 7 is

that it does not require any experimental data for predicting the solubility in binary solvents at various temperatures.

To convert mole fraction solubility to molar solubility or vice versa, the densities of the solute saturated solutions are required as input data. It has been shown that using a trained version of the Jouyban–Acree model for representing the density of solvent mixtures in the absence of a solute, one may predict the solute saturated density by employing the density values of the saturated solutions in the monosolvents.^{18–23} To provide such a predictive tool for densities of TRIS saturated solutions in water + 1-PrOH mixtures at various temperatures, densities of the solute-free mixtures of water + 1-PrOH mixtures in various mole fraction compositions of the binary mixtures, taken from the literature,²⁴ are fit to the model, and obtained equation is

$$\ln \rho_{m,T} = x_1^0 \ln \rho_{1,T} + x_2^0 \ln \rho_{2,T} + \frac{x_1^0 x_2^0}{T} [-63.714 - 27.129(x_1^0 - x_2^0)] \quad (10)$$

in which $\rho_{m,T}$, $\rho_{1,T}$, and $\rho_{2,T}$ are the densities of the solute-free mixed solvent and the solute-free monosolvents 1 and 2 at the temperature T , respectively. Because of the low TRIS solubilities, the effect of the dissolved solute on the numerical values of the interaction terms of the Jouyban–Acree model for density data is not significant. The terms could be employed to predict the density of TRIS saturated solutions of water + 1-PrOH using

$$\ln \rho_{m,T}^{\text{sat}} = x_1^0 \ln \rho_{1,T}^{\text{sat}} + x_2^0 \ln \rho_{2,T}^{\text{sat}} + \frac{x_1^0 x_2^0}{T} [-63.714 - 27.129(x_1^0 - x_2^0)] \quad (11)$$

in which $\rho_{m,T}^{\text{sat}}$ is the density of TRIS saturated solution of mixed solvent system, $\rho_{1,T}^{\text{sat}}$ and $\rho_{2,T}^{\text{sat}}$ are the density of TRIS saturated solutions of monosolvents 1 and 2 at different temperatures. The measured $\rho_{1,T}^{\text{sat}}$ and $\rho_{2,T}^{\text{sat}}$ values for TRIS saturated solutions of water and 1-PrOH at various temperatures along with their solute free values²⁴ are listed in Table 5. There are decreased

Table 5. Density of TRIS-Saturated Solutions of Water + 1-PrOH Mixtures at Various Temperatures and the Reported Corresponding Solute-Free Density Values (in Parentheses) Taken from a Paper²⁴

T/K	water	1-PrOH
293.2	1.1150 (0.9982)	0.8247 (0.8043)
298.2	1.1420 (0.9971)	0.8227 (0.8002)
303.2	1.1620 (0.9957)	0.8218 (0.7964)
308.2	1.1740 (0.9940)	0.8187 (0.7923)
313.2	1.1880 (0.9922)	0.8180 (0.7885)

patterns for the densities of water and 1-PrOH in the absence of TRIS due to volume expansion of the solvents at higher temperatures. Increased densities for TRIS saturated aqueous solutions at higher temperatures were observed because of the effects of the dissolved TRIS on the mass of the solution. With the use of eq 11, the density of TRIS-saturated solutions of water + 1-PrOH mixtures at various temperatures could be predicted with acceptable accuracies as shown in earlier works.^{18–23}

CONCLUSIONS

We have reported the experimental solubilities of TRIS in water + 1-PrOH mixtures at (293.2, 298.2, 303.2, 308.2, and 313.2) K, and provided a trained version of the Jouyban–Acree model combined with the van't Hoff equation to predict the solubility of TRIS at any composition of the water + 1-PrOH mixtures at a temperature of interest. This work extends the available solubility database of solutes in mixed solvents,²⁵ and also the results could be employed for practical purposes, for example, for crystallization procedures in the chemical industry or for methods like liquid chromatography or capillary electrophoresis. A numerical method was reported for predicting the density of TRIS saturated solutions in water + 1-PrOH mixtures.

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Notes

The authors declare no competing financial interest.

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