

Diffusion Coefficients in Some Ternary Systems

Jaroslav Bulička* and Jaroslav Procházka

*Institute of Chemical Process Fundamentals, Czechoslovak Academy of Sciences,
165 02 Prague 6-Suchbát, Czechoslovakia*

Diffusion coefficients of some solutes were determined in partially miscible, mutually saturated solvents at 20 and 30 °C. The systems were toluene–acetone–water, 1-butanol–succinic acid–water, and MIBK–acetic acid–water. Concentration of the solutes ranged between 0 and 5% by mass. The diffusion coefficients were also determined in the binary system 1-butanol–water at 20 °C for concentrations up to saturation. The diaphragm method was employed for measurement. A linear relationship between diffusivity and concentration and constancy of the group $D\mu/T$ have been found in the indicated ranges.

The aim of this work was to determine concentration and temperature dependences of the diffusion coefficients of the solutes in both liquid phases of the following ternary systems frequently used for the study of liquid–liquid extraction: (i) toluene–acetone–water, (ii) 1-butanol–succinic acid–water, and (iii) methyl isobutyl ketone (MIBK)–acetic acid–water. The attention was focused on the low concentration range of the solute (up to 5% mass). For the system (ii) exhibiting a marked mutual solubility of the solvents, the diffusivity of 1-butanol in water and of water in 1-butanol was measured at 20 °C in the whole concentration range.

The literature on diffusion coefficients in these systems (2–4, 8–11, 15) is relatively scarce. They mostly concern pure binary systems while the measurements of this work were performed with mutually saturated solvents to approximate the situation commonly encountered in liquid–liquid extraction. As found by Anderson (2), mutual solubility may lead in nonaqueous solvents to considerable deviations. Part of the data in the literature were obtained at different temperatures and/or for infinitely diluted solutions.

The Chemicals Used

Twice redistilled water was used for measurement. The other chemicals were analytical grade products (by Lachema). Acetone, 1-butanol, and methyl isobutyl ketone were redistilled in an efficient column; toluene was redistilled in the same manner with some methanol added. Physical properties characterizing purity of the chemicals together with the tabulated values are shown in Table I.

Experimental Method

Diffusivities were measured by the diaphragm method in the modification proposed by Stokes (18). The apparatus as well as the experimental method were adapted according to Samohýl's (17) experience. The diffusion cell (Figure 1) was a cylindrical glass vessel 30 mm i.d. with a horizontal diaphragm made of sintered glass S 4. The thickness of the diaphragm was 1.6 mm and the pore diameter ranged between 7 and 15 μ . The diaphragm divided the cell into two compartments of equal volume (about 30 cm³). The bottom compartment was sealed by a Teflon stopper while the upper compartment was vented to the atmosphere through a capillary in a ground-in stopper. The ground-in stopper was carefully finished and used without grease to prevent contamination of the measured solvents.

The content of each compartment of the cell was stirred by a polyethylene-coated iron rod driven by magnets rotating outside the cell. The cells were placed in two water baths housing three

cells each. The present temperature was held constant by an ultrathermostat within 0.1 °C. The speed of revolution of the impellers was 120 min⁻¹. The solutions used for measurement were degassed. Degassing was achieved by boiling the solutions for 0.5 h under a reflux condenser.

Filling of the cell with solutions followed this procedure: The bottom compartment was first filled to about three-quarters with pure solvent and sealed. The upper compartment was vacuumed and the diaphragm heated by a stream of warm air to prevent freezing of the liquid within the pores.

After a thorough evaporation of the liquid trapped in the diaphragm the solvent in the bottom compartment began gradually to boil and the vapors displaced the air present also within the diaphragm. After about 2 min the cell was put up-side-down in order to fill the diaphragm with the solvent. Two operations were then carried out following quickly one after the other. First, the space above the solvent was devacuumed by removing the Teflon stopper causing a rapid filtration through the diaphragm. The bottom compartment of the cell was then vented to the atmosphere in such a way as to keep a layer of the solvent constantly covering the diaphragm. The bottom compartment of the cell (held now still up) was then filled with the measured solution, and the pores of the diaphragm were rinsed with the solution. The rinsing was speeded up by vacuum. Care was taken to keep the diaphragm constantly covered with a layer of the solution. After a fivefold rinsing, a sample was drawn for analysis, the bottom compartment containing the impeller was completely filled with the solution sealed with the Teflon stopper, and the cell was put back into working position. The upper compartment was filled with the same solution and the cell was placed into the thermostated bath. There it was kept for about an hour while stirring the solutions in both compartments.

In the second stage the upper compartment was repeatedly rinsed and filled with the other thermostated solution. The rinsing was repeated five times always removing the solution from the compartment carefully to prevent convective flows through the pores. On completion of the rinsing a sample was drawn for analysis and the upper compartment was filled with the other solution and sealed with the stopper provided with the capillary. The measurement was commenced by turning on the impellers. Having completed the experiment the solutions in both compartments were again sampled for analysis.

The above procedure ensures, in accord with the theoretical analysis of Mills (12), that at the onset of diffusion the concentration within the diaphragm equals the concentration of the bottom solution giving rise to a concentration jump at the upper edge of the diaphragm.

The formula (eq 1) was used to calculate the diaphragm cell integral diffusion coefficient, D_i , which depends not only on the total range of concentration but also on the duration of experiment and, through the boundary conditions, on the type of apparatus.

$$D_i = - \frac{1}{\beta \Delta t} \ln (\Delta c_t / \Delta c_0) \quad (1)$$

where β is a diaphragm constant. Its value was assessed by measuring the rate of diffusion of KCl from 0.5 N solution into pure water.

The value of the diaphragm cell integral diffusion coefficient, D_i , used for the calculation was taken equal to 1.84×10^{-5} (cm² s⁻¹) as given by Wolf et al. (21). The average error of the cali-

Table I. Physical Properties of Chemicals Used

	ρ (g cm ⁻³)		n	
	Exptl	Lit. ^a	Exptl	Lit. ^a
Acetone	0.7906	0.79053–0.79082	1.3592	1.3588–1.35911
1-Butanol	0.8095	0.80961–0.80970	1.3993	1.3992
Acetic acid	1.0498	1.0491–1.04926	1.3720	1.3716–1.3721
MIBK	0.8009	0.8007–0.8008	1.3958	1.3958
Toluene	0.8668	0.86683–0.8670	1.4969	1.4966–1.49693

^aData from ref 7 and 19.

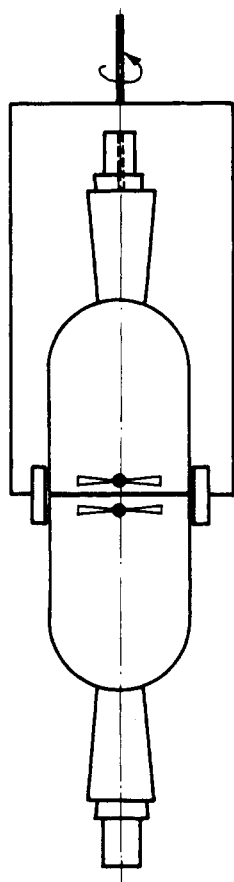


Figure 1. Magnetically stirred diaphragm cell.

bration was less than $\pm 0.2\%$. The whole program of measurement was interspersed with periodic calibration tests. The deviations remained within the above indicated limits.

The obtained values of the diaphragm integral diffusion coefficient, D_i , were processed to give the differential values, D , assuming that the latter is a linear function of concentration. Considering further the equal volumes of both compartments one can write, as shown by Gordon (5), that

$$D_i = D(\bar{c}); \quad \bar{c} = (c_0' + c_0'' + c_1' + c_1'')/4 \quad (2)$$

The measurements in all ternary systems were carried out in the concentration range between 0 and 5% by mass. The concentration ranges, for which individual values of D_i were obtained, are approximately determined by the initial concentration difference of the solutions in both compartments, which was about 1–1.5%. The duration of the experiments was estimated from the relation for the optimum time of measurement (16)

$$\theta = 1.2/D \quad (3)$$

with the exception of diffusion of succinic acid and water in 1-

butanol where, owing to the higher viscosity of the solvent, the optimum duration of the experiment was too large.

Analytical Methods

The concentration of KCl in water was determined by potentiometric titration with water solution of AgNO₃. The standard error of the analysis was $\pm 0.1\%$ rel.

The determination of succinic acid in water and 1-butanol was carried out by titration with sodium hydroxide using phenolphthalein. Prior to the titration the organic phase was diluted by a sufficient amount of water to homogenize the sample. The standard error of the determination was ± 0.3 and 0.5% rel, respectively.

Acetic acid in water was titrated by sodium hydroxide solution using phenolphthalein. The standard error of the determination was $\pm 0.3\%$ rel.

The determination of acetic acid in MIBK was carried out by potentiometric titration with sodium methoxide in pyridine-methanol solution. The standard error of determination was $\pm 0.5\%$ rel.

Acetone in water and organic phase was determined by refractometry using a Zeiss-Jena immersion refractometer with a thermostated prism. Weighted samples were used for calibration. The standard error of determination was $\pm 0.3\%$ rel.

The determinations of 1-butanol in water and water in 1-butanol were both carried out also by refractometry with the thermostated prism after calibration with weighted samples. The standard error of determination was $\pm 0.3\%$ rel.

Error Analysis

The estimate of the error of the determination of diffusivity is based on the fact that the duration of an experiment is sufficiently long to be determined with relatively high precision. Accordingly, the error of diffusivity stems only from the accuracy of chemical analysis. On writing eq 1 in a somewhat different form we obtain:

$$D_i = -\frac{1}{\beta \Delta t} \ln \frac{c_1 - c_2}{c_3 - c_4} \quad (4)$$

The concentrations in both compartments were determined both at the beginning and at the end of the experiment. Thus we may write:

$$dD_i = -\frac{1}{\beta \Delta t} \frac{c_3 - c_4}{c_1 - c_2} (dc_1 - dc_2) - \frac{(c_1 - c_2)dc_3}{(c_3 - c_4)^2} + \frac{(c_1 - c_2)dc_4}{(c_3 - c_4)^2} \quad (5)$$

A maximum error occurs if the errors of chemical analyses combine. Because in each experiment we have $c_1 > c_3 > c_4 > c_2$ the error of diffusivity is at maximum if

$$\text{sgn } dc_1 = \text{sgn } dc_4 \neq \text{sgn } dc_3 = \text{sgn } dc_2 \quad (6)$$

Because the concentrations were determined with constant absolute error, for an estimate of the maximum error we may write that

Table II. Diffusivity of Acetone in the Toluene–Acetone–Water System

Temp (°C)	Water phase		Organic phase		
	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	
20	0.59	1.14	0.72	2.70	
	0.92	1.14	0.82	2.71	
	1.14	1.13	0.83	2.73	
	1.44	1.11	0.83	2.80	
	2.06	1.08	1.13	2.76	
	2.95	1.09	1.22	2.70	
	3.45	1.07	2.02	2.78	
	3.70	1.04	3.25	2.65	
	4.84	1.01	3.26	2.66	
	5.96	1.01	3.27	2.60	
			4.32	2.57	
			4.32	2.59	
			4.36	2.64	
			4.37	2.51	
	$a = 1.154 \times 10^{-5}$ $b = -2.67 \times 10^{-7}$ Standard rel error of regression = 0.4%	$a = 2.79 \times 10^{-5}$ $b = -4.63 \times 10^{-7}$ Standard rel error of regression = 0.4%			
30	0.75	1.44	0.73	3.06	
	1.22	1.44	0.87	3.09	
	2.90	1.37	0.88	3.17	
	3.31	1.34	0.89	3.16	
	4.10	1.31	0.99	3.09	
			3.94	2.95	
			4.10	2.97	
			4.13	2.99	
			4.53	2.95	
			4.55	2.99	
		$a = 1.477 \times 10^{-5}$ $b = -3.94 \times 10^{-7}$ Standard rel error of regression = 0.35%	$a = 3.15 \times 10^{-5}$ $b = -4.16 \times 10^{-7}$ Standard rel error of regression = 0.35%		

$$dc_1 = dc_4 = -dc_3 = -dc_2 = dc \quad (7)$$

For the relative error of diffusivity we thus get:

$$\frac{dD_i}{D_i} = 2dc \frac{(c_3 - c_4)^2 + c_1 - c_2}{(c_1 - c_2)(c_3 - c_4) \ln \frac{c_1 - c_2}{c_3 - c_4}} \quad (8)$$

The magnitude of the expression in eq 8 increases with the growing value of the difference ($c_1 - c_2$) and decreasing value of the difference ($c_3 - c_4$). In the experiments involved these differences took the following values: ($c_1 - c_2$) ≤ 1.5 and ($c_3 - c_4$) > 0.3. The mean square error of the concentration was 0.01% by mass. With these values the maximum error of diffusivity amounts to 10%.

Results

The results of measurements are summarized in Tables II–V. The parameters of the following linear correlation were determined by the least-square method for each phase and temperature of the examined systems

$$D = a + bc \quad (9)$$

Numerical values of these parameters are also shown in the above tables together with the appropriate standard error. The scatter of the data in individual series is primarily due to the error of the corresponding analytical method. The analyses were usually repeated three times; in the case of the titration of acetic acid in MIBK five times.

Table III. Diffusivity of Succinic Acid in the 1-Butanol–Succinic Acid–Water System

Temp (°C)	Water phase		Organic phase		
	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	
20	0.69	0.57	0.43	0.24	
	0.86	0.55	0.59	0.23	
	0.90	0.57	0.61	0.23	
	1.02	0.56	0.86	0.22	
	1.84	0.55	1.40	0.21	
	2.17	0.54	2.44	0.21	
	2.56	0.52	2.96	0.21	
	3.66	0.52	3.71	0.23	
	4.07	0.48	3.76	0.23	
	4.42	0.49	4.75	0.22	
	4.47	0.48	4.85	0.21	
	5.21	0.47	5.56	0.21	
		$a = 5.84 \times 10^{-6}$ $b = -2.18 \times 10^{-7}$ Standard rel error of regression = 0.55%	$a = 2.29 \times 10^{-6}$ $b = -3.07 \times 10^{-8}$ Standard rel error of regression = 1.35%		
	30	0.68	0.82	0.72	0.34
0.88		0.79	1.74	0.32	
0.99		0.78	2.24	0.32	
2.69		0.77	2.91	0.33	
2.96		0.76	3.44	0.32	
2.97		0.75	3.80	0.33	
4.32		0.73	4.74	0.31	
		$a = 8.18 \times 10^{-6}$ $b = -2.05 \times 10^{-7}$ Standard rel error of regression = 0.60%	$a = 3.41 \times 10^{-6}$ $b = -4.99 \times 10^{-8}$ Standard rel error of regression = 1.09%		

Table IV. Diffusivity of Acetic Acid in the MIBK–Acetic Acid–Water System

Temp (°C)	Water phase		Organic phase		
	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	
20	0.58	1.04	0.71	1.44	
	0.74	1.04	0.72	1.41	
	1.04	1.03	0.97	1.44	
	1.71	1.04	1.04	1.40	
	2.34	0.99	1.05	1.40	
	3.05	1.00	1.07	1.42	
	3.79	0.95	3.64	1.41	
	3.85	0.96	4.01	1.40	
	5.03	0.90	4.19	1.44	
	5.16	0.91	4.34	1.41	
			4.46	1.43	
			5.16	1.43	
		$a = 1.067 \times 10^{-5}$ $b = -2.96 \times 10^{-7}$ Standard rel error of regression = 0.5%	$a = 1.421 \times 10^{-5}$ $b = 1.09 \times 10^{-9}$ Standard rel error of regression = 0.4%		
	30	0.69	1.29	0.71	1.77
1.12		1.28	0.93	1.78	
1.74		1.26	1.03	1.76	
2.06		1.28	3.27	1.74	
2.42		1.24	3.53	1.71	
2.89		1.22	3.69	1.73	
2.99		1.22	4.39	1.70	
3.39		1.23	4.40	1.70	
4.10		1.20	4.55	1.75	
4.74		1.17	4.75	1.72	
4.98		1.20	4.82	1.67	
		$a = 1.312 \times 10^{-5}$ $b = -2.71 \times 10^{-7}$ Standard rel error of regression = 0.3%	$a = 1.786 \times 10^{-5}$ $b = -1.73 \times 10^{-7}$ Standard rel error of regression = 0.4%		

Table V. Diffusivity in the 1-Butanol–Water System

Temp (°C)	Water phase		Organic phase	
	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)	<i>c</i> (% by mass)	$10^5 D$ (cm ² s ⁻¹)
20	0.89	0.82	1.96	0.45
	1.30	0.79	3.78	0.42
	1.42	0.81	3.88	0.42
	1.84	0.78	7.43	0.34
	2.48	0.74	7.44	0.34
	2.56	0.77	10.90	0.30
	3.79	0.71	13.29	0.25
	4.20	0.71	15.90	0.22
	5.18	0.66		
	5.52	0.63		
	6.15	0.61		
	$a = 8.56 \times 10^{-6}$		$a = 4.82 \times 10^{-6}$	
	$b = -3.85 \times 10^{-7}$		$b = -1.69 \times 10^{-7}$	
Standard rel error of regression = 0.6%		Standard rel error of regression = 1%		

Discussion

The data obtained at 20 and 30 °C can be compared using the relation

$$\lim_{c \rightarrow 0} \frac{D(c)\mu(c)}{T} = \left(\frac{D\mu}{T} \right)_0 = \text{constant} \quad (10)$$

which is also a part of the correlation of Wilke and Chang (20). Assuming that this relation applies also to nonzero concentrations and assuming further linearity of the $D(c)$ and $\mu(c)$ functions in the intervals covered by the experiments, the results can be correlated by a quadratic equation as

$$D\mu/T = (D\mu/T)_0 + c(p + qc) \quad (11)$$

The constants of this equation for the investigated systems are summarized in Table VI. The appropriate linear functions $\mu(c)$ were obtained by linearization of the regression equations from the data of other authors (1, 6, 13, 14). Table VI shows for comparison also the $(D\mu/T)_0$ values computed from the relation of Wilke and Chang. The association factors for the water and the organic phase were taken equal respectively to 2.6 and 1.0. In comparison with the correlations according to eq 9 the mean deviation of eq 11 is greater excepting diffusion of acetone in toluene. Nevertheless, the deviation of the latter relation still remains acceptable. A comparison with Tables II–V shows the variability of the $D\mu/T$ values with concentration (given in Table VI in the column designated "range") to be in the water phase substantially lower than that of D . No significant difference exists in the organic phase. As may be apparent from Tables II–V, the dependence of $D(c)$ in a number of cases is very strong even over the narrow interval involved. The change scarcely drops below 10%; in the case of diffusion of water in 1-butanol measured up to the saturation the change exceeds 100%. A comparison of the values of $(D\mu/T)_0$ from this study with those

computed from the correlation of Wilke and Chang indicates that the results estimated from this correlation for the 1-butanol–succinic acid–water and the MIBK–acetic acid–water systems are systematically higher. A feasible explanation may be the presence of the other solvent in the solution in our experiments.

A comparison of the data obtained in this work for the 1-butanol–water system with those from the literature (3, 4, 10, 15) displays a generally good agreement. Exceptional disagreement is shown in the diffusion coefficients of water in 1-butanol measured by Randall et al. (15). These data, however, appear to disagree also with those of Emanuel and Olander (4).

Conclusion

The diffusion coefficients were measured by the diaphragm method in the binary system water–1-butanol and the ternary systems toluene–acetone–water, 1-butanol–succinic acid–water and MIBK–acetic acid–water. In all cases excepting diffusion of acetic acid in MIBK at 20 °C, the diffusion coefficient decreased with increasing concentration of the diffusing solute. The effect of concentration can be expressed by a linear function with an error mostly below 1%.

The effect of temperature was satisfactorily accounted for in terms of $D\mu/T$, the water phase concentration dependence of this quantity being substantially weaker than that of the diffusivity alone. A quadratic expression was used for this correlation but the quadratic term has been found to be of little significance in the range of concentrations studied.

Glossary

<i>a</i>	constant in eq 9
<i>b</i>	constant in eq 9
<i>c</i>	concentration
<i>c</i> ₁ , <i>c</i> ₂	initial concentration in lower and upper compartment, respectively
<i>c</i> ₃ , <i>c</i> ₄	final concentration in lower and upper compartment, respectively
\bar{c}	mean concentration defined by eq 2
Δc	concentration difference on both sides of the diaphragm at a given time instant
<i>D</i>	differential diffusion coefficient
<i>D</i> _i	diaphragm integral diffusion coefficient
<i>n</i>	refractive index
<i>p</i>	constant in eq 11
<i>q</i>	constant in eq 11
Δt	duration of measurement
<i>T</i>	absolute temperature
β	constant of diaphragm cell
θ	optimum time of duration of experiment
μ	dynamic viscosity
ρ	density
σ	standard error
<i>0</i> , <i>f</i>	initial and final value
' , ''	top and bottom compartment of diaphragm cell

Table VI. Constants of Correlation (6) and a Comparison of $(D\mu/T)_0$ with Wilke and Chang

System	Phase	$10^{10} p$	$10^{11} q$	$(D\mu/T)_0$	σ , %	Range $(D\mu/T)_0$, %	$(D\mu/T)_0^a$
Toluene–acetone–water	w	0.3	−2.7	3.87	3.5	1	3.83
	o	−15.4	4.4	5.55	0.3	13	5.37
1-Butanol–succinic acid–water	w	−1.0	−1.8	2.88	2.9	3	3.03
	o	0.6	−5.3	2.78	3.5	6	3.18
MIBK–acetic acid–water	w	−2.8	−0.9	3.82	3.0	4	4.14
	o	4.1	1.9	3.89	3.0	7	5.82

^a Values computed from the relation of Wilke and Chang (20).

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The Refractive Indices and Densities of Ternary Mixtures of Benzene-Toluene-Xylene

Jelenka Savković-Stevanović* and Dušan Simonović

Faculty of Technology and Metallurgy, Department of Chemical and Metallurgical Engineering, University of Beograd, 11001 Beograd, Yugoslavia

The relationship between the composition of the ternary mixtures of benzene-toluene-xylene and the refractive index, as well as density, was determined at 25 °C.

Relationships between the composition and refractive index, as well as density, are known in literature for the binary mixtures: benzene-toluene, benzene-xylene, and toluene-xylene (6). With the aim to use ternary mixtures of benzene-toluene-xylene as the test mixture for distillation columns, the examination of the relationship between composition and density along with index of refraction was undertaken.

Experimental Section

For preparation of different solutions of the ternary mixture, reagent grade pro analysis (Merck) was used, with the following data:

	Bp at 760 mmHg <i>t</i> , °C	Density $d_{25}^{25} C_4$ g ml ⁻¹	RI $n_{25}^{25} C_D$
Benzene	80.1	0.8779	1.497 10
Toluene	110.6	0.8649	1.493 66
Xylene	138.5	0.8639	1.494 90

This is in good agreement with the data reported in literature (4, 6). The density and refractive index mean deviations are 0.6 and 0.05%, respectively.

The solutions were prepared by weight, the binary mixture benzene-toluene being prepared first in 20 mol % intervals with subsequent addition of xylene. Required precautions were applied to prevent any loss due to evaporation. Individual component weights were determined by a Mettler balance Type H20 with an accuracy of ±0.005%.

Densities and the refractive index of prepared solutions were determined in duplicate at 25 °C. The temperature was controlled within ±0.005 °C by an Ultra-Thermostat. A Zeiss refractometer was used to observe the refractive index with an accuracy of ±0.005%. For the determination of densities Ostwald's pycnometers were used.

Table I. Benzene-Toluene System

Benzene composition, mole fraction	Density, $d_{25}^{25} C_4$, g ml ⁻¹	Refractive index (±0.000 05), $n_{25}^{25} C_D$
0.00	0.8649	1.493 66
0.20	0.8675	1.494 15
0.40	0.8705	1.494 95
0.60	0.8734	1.495 60
0.80	0.8760	1.496 35
1.00	0.8779	1.497 10

Table II. Benzene-Xylene System

Benzene composition, mole fraction	Density, $d_{25}^{25} C_4$, g ml ⁻¹	Refractive index (±0.000 05), $n_{25}^{25} C_D$
0.00	0.8639	1.494 90
0.20	0.8645	1.494 85
0.40	0.8670	1.495 00
0.60	0.8700	1.495 30
0.80	0.8737	1.496 00
1.00	0.8779	1.497 10

Table III. Toluene-Xylene System

Toluene composition, mole fraction	Density, $d_{25}^{25} C_4$, g ml ⁻¹	Refractive index (±0.000 05), $n_{25}^{25} C_D$
0.00	0.8639	1.494 90
0.20	0.8642	1.494 70
0.40	0.8645	1.494 50
0.60	0.8646	1.494 20
0.80	0.8648	1.493 90
1.00	0.8649	1.493 66

Results

The obtained results are presented in Tables I-IV. The density-composition and refractive index-composition curves are shown on Figures 1-3.