Excess Molar Volumes and Viscosities for Binary Mixtures of Propylene Glycol Monomethyl Ether with Methacrylic Acid, Benzyl Methacrylate, and 2-Hydroxyethyl Methacrylate at (298.15, 308.15, and 318.15) K

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Densities and viscosities at (298.15, 308.15, and 318.15) K are presented as a function of mixture composition for the binary mixtures of propylene glycol monomethyl ether with methacrylic acid, benzyl methacrylate, and 2-hydroxyethyl methacrylate. From the experimental data, the excess molar volumes and viscosity deviations have been calculated. These results have been correlated with the Redlich–Kister type polynomial to derive the coefficients and standard deviation. McAllister's three-body and four-body interaction models were also used to correlate the kinematic viscosities. These model's parameters were treated to be temperature-dependent.

Introduction

The color filter is one of the essential parts of a color liquid crystal display, and the pigment-dispersed color resist (PDCR) is the most important material for manufacture of this part. We are interested in investigating the photolithography processes, transport properties, and storage stability of new color resists. The thermophysical properties of a binary mixture such as density and viscosity are useful in the design of many types of process and transport equipment in chemical industries. Methacrylic acid (MAA), benzyl methacrylate (BzMA), 2-hydroxyethyl methacrylate (2-HEMA), and propylene glycol monomethyl ether (PGME) are the key compounds in the manufacturing of the pigment-dispersed color resist industries. A series of density and viscosity measurements have been made recently in our laboratory.¹⁻³ This paper reports on the binary systems composed of propylene glycol monomethyl ether with MAA, BzMA, and 2-HEMA at (298.15, 308.15, and 318.15) K and over the entire composition range. No literature data were found at comparable conditions. From the new experimental data, the excess molar volumes $V^{\rm E}$ and viscosity deviations $\delta \eta$ have been calculated. These results have been correlated with the Redlich-Kister type polynomial to derive the coefficients and standard deviation. McAllister's three-body and four-body interaction models were also used to correlate the kinematic viscosities. In this paper, these model's parameters were also treated to be temperature-dependent.

Experimental Section

MAA (mass fraction 99 %) was obtained from Sigma-Aldrich. 2-HEMA (mass fraction 98 %) was supplied by Acros Organics. BzMA (mass fraction 98 %) was purchased from Showa Chemical Co. Ltd. Propylene glycol monomethyl ether (mass fraction 99 %) was obtained from Fluka Chemicals (Germany). The purities of these substances were checked with gas chromatography before use. All reagents were used without further purification.

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Figure 1. Densities ρ and viscosities η of propylene glycol monomethyl ether at different temperatures.

Table 1. Densities ρ and Viscosities η of Propylene Glycol Monomethyl Ether at Different Temperatures

	ρ/g•0	cm ⁻³	η/mPa•s		
T/K	exptl	lit. ⁵	exptl	lit. ⁵	
298.15	0.9159	0.9165	1.707	1.694	
308.15	0.9071	0.9069	1.306	1.329	
318.15	0.8969	0.8971	1.069	1.078	

Density Measurement. The densities were determined with a pycnometer having a nominal internal volume of 10 cm³. The internal volume of the pycnometer was calibrated with pure water⁴ at each temperature. The sample mixture was prepared by mass with an uncertainty of \pm 0.0001 in mole fraction. To minimize evaporation during the sample preparation, the less volatile component was charged first. Three loaded pycnometers were immersed in a thermostatic bath (Neslab GP-500), which was controlled to within \pm 0.03 K. A precision digital thermometer (model 1560, Hart Scientific) with a thermistor probe was used to read the temperature with an uncertainty of \pm 0.015 K. The mixture densities were obtained by averaging the results from these three replications. The uncertainty of reported densities was estimated to be less than \pm 0.1 %. The sample compositions were frequently checked with a gas

Table 2.	Density	p and	Viscosity n	for	Propylene	Glycol	Monometh	vl Ether	(1)) + (MAA	(2)	
					/			4					

		$ ho/g\cdot cm^{-3}$		η/mPa•s				
<i>x</i> ₁	T = 298.15 K	T = 308.15 K	T = 318.15 K	T = 298.15 K	T = 308.15 K	T = 318.15 K		
0.0	1.0095	0.9994	0.9892	1.267	1.078	0.945		
0.1000	1.0009	0.9918	0.9824	1.376	1.163	1.002		
0.2000	0.9920	0.9832	0.9742	1.659	1.372	1.157		
0.3000	0.9835	0.9749	0.9661	1.982	1.577	1.312		
0.4000	0.9753	0.9665	0.9575	2.124	1.702	1.394		
0.4999	0.9666	0.9578	0.9489	2.233	1.762	1.429		
0.6000	0.9575	0.9487	0.9402	2.255	1.770	1.432		
0.7000	0.9473	0.9386	0.9303	2.140	1.685	1.361		
0.8001	0.9369	0.9283	0.9199	1.969	1.554	1.258		
0.9000	0.9262	0.9178	0.9091	1.809	1.431	1.165		
1.0	0.9159	0.9071	0.8969	1.707	1.306	1.069		

Table 3. Density ρ and Viscosity η for Propylene Glycol Monomethyl Ether (1) + BzMA (2)

		$ ho/g\cdot cm^{-3}$		η/mPa•s				
x_1	T = 298.15 K	T = 308.15 K	T = 318.15 K	T = 298.15 K	T = 308.15 K	T = 318.15 K		
0.0	1.0347	1.0258	1.0170	2.302	1.885	1.580		
0.1000	1.0268	1.0181	1.0095	2.194	1.806	1.520		
0.2000	1.0186	1.0101	1.0014	2.090	1.716	1.441		
0.3000	1.0098	1.0014	0.9925	1.990	1.628	1.364		
0.4000	1.0002	0.9918	0.9828	1.895	1.538	1.291		
0.5000	0.9897	0.9813	0.9722	1.800	1.465	1.228		
0.6000	0.9781	0.9697	0.9605	1.731	1.406	1.166		
0.7000	0.9652	0.9568	0.9475	1.677	1.354	1.115		
0.8000	0.9509	0.9423	0.9327	1.633	1.312	1.084		
0.9000	0.9346	0.9259	0.9160	1.640	1.308	1.077		
1.0	0.9159	0.9071	0.8969	1.707	1.306	1.069		

Table 4. Density ρ and Viscosity η for Propylene Glycol Monomethyl Ether (1) + 2-HEMA (2)

		$\rho/g \cdot cm^{-3}$		η/mPa•s				
x_1	T = 298.15 K	T = 308.15 K	T = 318.15 K	T = 298.15 K	T = 308.15 K	T = 318.15 K		
0.0	1.0671	1.0577	1.0486	5.784	4.194	3.181		
0.1000	1.0552	1.0462	1.0378	5.120	3.747	2.869		
0.2000	1.0428	1.0341	1.0255	4.607	3.408	2.623		
0.3001	1.0299	1.0212	1.0126	4.130	3.065	2.373		
0.4000	1.0163	1.0075	0.9989	3.656	2.757	2.148		
0.5000	1.0017	0.9930	0.9844	3.256	2.477	1.947		
0.6000	0.9862	0.9776	0.9690	2.869	2.208	1.753		
0.7000	0.9698	0.9615	0.9528	2.496	1.944	1.559		
0.8000	0.9527	0.9445	0.9355	2.208	1.730	1.395		
0.9000	0.9348	0.9266	0.9173	1.933	1.528	1.237		
1.0	0.9159	0.9071	0.8969	1.707	1.306	1.069		

chromatography at the end of measurements to verify that there was no change in the compositions.

Viscosity Measurement. The kinematic viscosities ν were measured using Cannon-Fenske routine viscometers (size 75, supplied by Cannon Instrument Co.). The viscometer was placed in a thermostatic water bath (TV-4000, TAMSON), in which the temperature was regulated to within \pm 0.01 K. An electronic stop watch was used to measure the flow times. Triplicates or more measurements of flow times were reproducible within \pm 0.2 % or less. The kinematic viscosities $\nu/\text{cm}^2 \cdot \text{s}^{-1}$ were obtained from the relation

$$\nu = kt \tag{1}$$

where $k/\text{cm}^{2}\text{s}^{-2}$ is the capillary constant of viscometer and t/s is the flow time. The viscometer was calibrated with doubledistilled water at each working temperature, and the capillary constant at each specific temperature was determined by averaging 10 calibration runs. The uncertainty of viscosity measurements was estimated to within ± 1.0 %, and the values of absolute viscosities η/mPa ·s were calculated by using the equation of $\eta = \rho \nu$.

The measured densities and viscosities of propylene glycol monomethyl ether at (298.15, 308.15, and 318.15) K were compared with the literature values, and the results are presented

in Table 1 and in Figure 1. They show that our measurements agree with literature values within the experimental uncertainties.

Results and Discussion

Experimental results for the three binary systems of propylene glycol monomethyl ether with MAA, BzMA, and 2-HEMA are listed in Tables 2 to 4, respectively. Figure 2 shows the variations of the absolute viscosities with the mole fraction of propylene glycol monomethyl ether for these three investigated systems at 308.15 K. In the MAA system, the viscosity increases with the mole fraction of propylene glycol monomethyl ether, reaching a weak maximum at about $x_1 = 0.6$, and then decreasing slightly to the value of pure propylene glycol monomethyl ether. However, the viscosities in the BzMA and 2-HEMA systems decrease monotonically with the mole fraction of propylene glycol monomethyl ether. In this paper, the measured densities and viscosities for pure fluids are also presented as functions of temperature by the following equations, respectively

$$\rho = a_0 + a_1(T/K) \tag{2}$$

$$\eta = b_0 + b_1(T/K) + b_2(T/K)^2$$
(3)

where a_0 , a_1 , b_0 , b_1 , and b_2 are the undetermined parameters.



Figure 2. Viscosity η at 308.15 K: \blacksquare , PGME (1) + MAA (2); \bullet , PGME (1) + BzMA (2); \bullet , PGME (1) + 2-HEMA (2); -, calculated from eq 7; ---, calculated from eq 7 with temperature-dependent parameters of eq 12.

 Table 5. Parameters in Equations 2 and 3 for Density and Viscosity

 Data of Pure Fluids

	a_0	$a_1 \times 10^4$	b_0	b_1	$b_2 \times 10^4$
PGME	1.19997	-9.52	89.5522	-0.5408	8.26
MAA	1.31214	10.20	32.6270	-0.1886	2.80
BzMA	1.29855	-8.85	66.1848	-0.3812	5.60
2-HEMA	1.34284	7.25	318.2490	-1.9081	28.80

Table 6. Correlated Results of Excess Molar Volume V^E

mixture	T/K	A_0	A_1	A_2	σ/cm ³ ·mol [−]
PGME + MAA	298.15	-2.6979	-0.5320	0.9130	0.0301
	308.15	-2.9753	-0.3388	0.2102	0.0266
	318.15	-3.6060	-0.9123	-1.0316	0.0225
PGME + BzMA	298.15	0.8131	-0.3744	0.2153	0.0090
	308.15	0.5311	-0.2284	0.2198	0.0060
	318.15	0.3736	-0.1335	-0.0720	0.0028
PGME + HEMA	298.15	-0.9053	0.0948	0.3838	0.0077
	308.15	-1.1580	-0.0887	-0.2531	0.0129
	318.15	-1.5887	-0.2244	-1.2214	0.0175

Table 7. Correlated Results of Viscosity Deviation $\delta \eta$

mixture	T/K	B_0	B_1	B_2	<i>σ</i> /mPa∙s
PGME + MAA	298.15	3.0874	0.3693	-2.9413	0.0295
	308.15	2.3417	0.4091	-1.8101	0.0199
	318.15	1.7491	0.2798	-1.3855	0.0179
PGME + BzMA	298.15	-0.7907	-0.5215	-0.2815	0.0051
	308.15	-0.5253	-0.2575	0.1350	0.0042
	318.15	-0.4163	-0.2505	0.1652	0.0045
PGME + HEMA	298.15	-1.9512	0.3550	-0.6837	0.0207
	308.15	-1.1037	0.4605	-0.1971	0.0175
	318.15	-0.7200	0.3050	-0.1039	0.0093

The best-fit values of the parameters for pure fluids are given in Table 5. According to eqs 2 and 3 and the parameters of pure fluids in Table 5, the AADs of the calculated densities and viscosities for pure fluids are approximately close to zero.

Excess volumes V^{E} and viscosity deviations $\delta \eta$ were calculated from the experimental results by the following equations, respectively

$$V^{\rm E} = V_{\rm M} - (x_1 V_1 + x_2 V_2) \tag{4}$$

$$\delta\eta = \eta_{\rm M} - (x_1\eta_1 + x_2\eta_2) \tag{5}$$

where x_1 and x_2 are the mole fractions, V_1 and V_2 are the molar volumes, and η_1 and η_2 are the viscosities of components 1 and 2, respectively. The subscript M represents mixture properties. The excess volumes and viscosity deviations were correlated



Figure 3. Excess volumes V^{E} at 308.15 K: \blacksquare , PGME (1) + MAA (2); •, PGME (1) + BzMA (2); \blacktriangle , PGME (1) + 2-HEMA (2); -, calculated from eq 6; - - -, calculated from eq 6 with temperature-dependent parameters of eq 11.



Figure 4. Viscosity deviations $\delta\eta$ at 308.15 K: \blacksquare , PGME (1) + MAA (2); \blacklozenge , PGME (1) + BzMA (2); \blacktriangle , PGME (1) + 2-HEMA (2); -, calculated from eq 7; - - , calculated from eq 7 with temperature-dependent parameters of eq 12; · · · , calculated from eq 10.

by a Redlich-Kister type polynomial:6

$$V^{\rm E}/{\rm cm}^3 \cdot {\rm mol}^{-1} = x_1 x_2 \sum_{k=0}^2 A_k (x_1 - x_2)^k$$
 (6)

$$\delta\eta/\text{mPa} \cdot \text{s} = x_1 x_2 \sum_{k=0}^{2} B_k (x_1 - x_2)^k$$
 (7)

The coefficients of A_k and B_k were obtained by fitting the equations to the experimental values with a least-squares method. The correlated results for excess volumes and viscosity deviations are given respectively in Tables 6 and 7, in which the tabulated standard deviation σ was defined as

$$\sigma = \left[\frac{\sum (Y_{\exp} - Y_{cal})^2}{n - p}\right]^{1/2}$$
(8)

where *Y* refers to $V^{\rm E}$ or $\delta\eta$, *n* is the number of data points, and *p* is the number of coefficients. The subscripts exp and cal denote the experimental value and the calculated value, respectively.

		three-body model			four-body model			
mixture	T/K	ν_{12}	ν_{21}	AAD ^a •10 ²	ν_{1112}	ν_{1122}	ν_{2221}	AAD ^a •10 ²
PGME + MAA	298.15	2.5391	2.6498	3.3	1.9274	3.8582	1.6139	1.0
	308.15	2.1005	1.9961	2.6	1.6209	2.8270	1.3726	0.9
	318.15	1.6763	1.6447	2.4	1.3123	2.2717	1.1502	0.9
PGME+BzMA	298.15	1.5796	2.0345	0.5	1.5688	1.8886	2.0149	0.2
	308.15	1.3367	1.6655	0.2	1.3694	1.4502	1.7186	0.2
	318.15	1.1059	1.4255	0.2	1.1229	1.2463	1.4518	0.2
PGME + 2-HEMA	298.15	2.6909	3.9663	0.5	2.3917	3.4550	4.1390	0.3
	308.15	2.2421	2.8918	0.4	2.0107	2.5163	3.1455	0.4
	318.15	1.8049	2.2572	0.3	1.6247	2.0118	2.4299	0.3

Table 8. Correlated Results of McAllister's Models

^{*a*} AAD = $(1/n)\sum_{k=1}^{n} |v_k^{\text{cal}} - v_k^{\text{exp}}|/v_k^{\text{exp}}$.

Fable 9. '	Temperature-Dependent	Parameters in the Redlich-	-Kister Type	Polynomial	and the McAlliste	er's Model
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			PGME	+ MAA			
$A_0^0 = 10.9016$	$A_0^1 = -0.0454$	$B_0^0 = 23.0115$	$B_0^1 = -0.0669$	$v_{12}^0 = 15.5100$	$v_{12}^1 = -0.0434$	$v_{1112}^0 = 11.3044$	$\nu_{1112}^1 = -0.0314$
$A_1^0 = 5.2661$	$A_1^1 = -0.0190$	$B_1^0 = 1.7311$	$B_1^1 = -0.0045$	$v_{21}^0 = 17.6785$	$v_{21}^1 = -0.0506$	$v_{2221}^0 = 27.0717$	$v_{1122}^1 = -0.0780$
$A_2^0 = 29.9847$	$A_2^1 = -0.0972$	$B_2^0 = -26.0130$	$B_2^1 = 0.0778$	AAD ^a •10 ²	$^{2} = 3.070$	$v_{2221}^0 = 8.1367$	$v_{2221}^1 = -0.0220$
$\sigma = 0.0368$	cm ³ ·mol ⁻¹	$\sigma = 0.0233$	3 mPa•s			AAD ^a .10 ²	= 1.334
			PGME -	+ BzMA			
$A_0^0 = 7.3435$	$A_0^1 = -0.0220$	$B_0^0 = -6.3467$	$B_0^1 = 0.0187$	$v_{12}^0 = 8.6458$	$v_{12}^1 = -0.0237$	$v_{1112}^0 = 8.1047$	$v_{1112}^1 = -0.0220$
$A_1^0 = -3.9579$	$A_1^1 = 0.0120$	$B_1^0 = -4.5183$	$B_1^1 = 0.0135$	$v_{21}^0 = 11.2951$	$v_{21}^1 = -0.0311$	$v_{2221}^0 = 11.2738$	$v_{1122}^1 = -0.0315$
$A_2^0 = 4.5462$	$A_2^1 = -0.0144$	$B_2^0 = -6.8759$	$B_2^1 = 0.0223$	AAD ^a •10 ²	= 0.678	$v_{2221}^0 = 10.5164$	$v_{2221}^1 = -0.0285$
$\sigma = 0.0078$	cm ³ ·mol ⁻¹	$\sigma = 0.0127$	/ mPa•s			AAD ^a •10 ²	= 0.655
			PGME +	2-HEMA			
$A_0^0 = 9.3134$	$A_0^1 = -0.0342$	$B_0^0 = -20.2275$	$B_0^1 = 0.0616$	$v_{12}^0 = 16.0235$	$v_{12}^1 = -0.0447$	$v_{1112}^0 = 13.9581$	$v_{1112}^1 = -0.0388$
$A_1^0 = 4.8453$	$A_1^1 = -0.0160$	$B_1^0 = 1.1450$	$B_1^1 = -0.0025$	$v_{21}^0 = 29.2202$	$v_{21}^1 = -0.0848$	$v_{2221}^0 = 24.2702$	$v_{1122}^1 = -0.0699$
$A_2^0 = 24.3629$	$A_2^1 = -0.0802$	$B_2^0 = -9.2606$	$B_2^1 = 0.0290$	AAD ^a •10 ²	= 1.023	$v_{2211}^0 = 28.9158$	$v_{2221}^1 = -0.0833$
$\sigma = 0.0172$	cm ³ ·mol ⁻¹	$\sigma = 0.0311$	mPa•s			AAD ^a •10 ²	= 1.133

^{*a*} AAD = $(1/n)\sum_{k=1}^{n} |v_k^{\text{cal}} - v_k^{\text{exp}}|/v_k^{\text{exp}}$.

The variations of $V^{\rm E}$ and δ with the mole fraction of propylene glycol monomethyl ether at 308.15 K are presented in Figures 3 and 4, respectively. Figure 3 shows that the excess molar volumes are negative in propylene glycol monomethyl ether + MAA and + 2-HEMA systems and positive in propylene glycol monomethyl ether + BzMA system over the entire composition range. They imply that volume expansion takes place when propylene glycol monomethyl ether mixes with BzMA and the volume contraction in propylene glycol monomethyl ether + MAA system is greater than propylene glycol monomethyl ether + 2-HEMA system. The above results can be discussed in terms of several effects that may be simply divided into dispersion forces and hydrogen bond contributions. The positive V^{E} values may be attributed to from dispersion forces contribution between propylene glycol monomethyl ether and BzMA molecules. On the other hand, the negative V^{E} values for binary mixtures of propylene glycol monomethyl ether with MAA or 2-HEMA may be attributed to hydrogen bond formation through dipole-dipole interactions between propylene glycol monomethyl ether and MAA or 2-HEMA molecules. Figure 4 illustrates that the viscosity deviations are negative in propylene glycol monomethyl ether + BzMA system as well as in propylene glycol monomethyl ether + 2-HEMA system but positive in propylene glycol monomethyl ether + MAA system over the entire composition range.

McAllister's multi-body interaction model⁷ was widely used to correlate kinematic viscosity ν data. The three-body McAllister model was defined as

$$\ln(\nu/\text{cm}^{2} \cdot \text{s}^{-1}) = x_{1}^{3} \ln \nu_{1} + 3x_{1}^{2} x_{2} \ln \nu_{12} + 3x_{1} x_{2}^{2} \ln \nu_{21} + x_{2}^{3} \ln \nu_{2} - \ln[x_{1} + x_{2}(M_{2}/M_{2})] + 3x_{1}^{2} x_{2} \ln[(2 + M_{2}/M_{1})/3] + 3x_{1} x_{2}^{2} \ln[(1 + 2M_{2}/M_{1})/3] + x_{2}^{3} \ln(M_{2}/M_{1})$$
(9)

and the four-body McAllister model was given by

$$\ln(\nu/\text{cm}^{2}\cdot\text{s}^{-1}) = x_{1}^{4} \ln\nu_{1} + 4x_{1}^{3}x_{2} \ln\nu_{1112} + 6x_{1}^{2}x_{2}^{2} \ln\nu_{1122} + 4x_{1}x_{2}^{3} \ln\nu_{2221} + x_{2}^{4} \ln\nu_{2} - \ln[x_{1} + x_{2}(M_{2}/M_{1})] + 4x_{1}^{3}x_{2} \ln[(3 + M_{2}/M_{1})/4] + 6x_{1}^{2}x_{2}^{2} \ln[(1 + M_{2}/M_{1})/2] + 4x_{1}x_{2}^{3} \ln[(1 + 3M_{2}/M_{1})/4] + x_{2}^{4} \ln(M_{2}/M_{1})$$
(10)

where ν_{12} , ν_{21} , ν_{1112} , ν_{1122} , and ν_{2221} are model parameters. The calculated results are presented in Table 8. As seen from Table 8, the calculated values of AAD from the McAllister's fourbody interaction model are smaller than those from the three-body model.

The parameters in eqs 6, 7, 9, and 10 were also treated to be temperature-dependent, as given by the following equations, respectively:

$$A_k = A_k^0 + A_k^1 T/K \quad (k = 0 \text{ to } 2)$$
(11)

$$B_k = B_k^0 + B_k^1 T/K$$
 (k = 0 to 2) (12)

$$v_{ij} = v_{ij}^0 + n_{ij}^1 T/K$$
 (*ij* = 12 or 21) (13)

$$v_{iiij} = v_{iiiij}^0 + n_{iiiij}^1 T/K$$
 (*iiij* = 1112 or 2221) (14)

$$\nu_{1122} = \nu_{1122}^0 + \nu_{1122}^1 T/K \tag{15}$$

where A_k^0 , A_k^1 , B_k^0 , B_k^1 , v_{ij}^0 , v_{ij}^1 , v_{iiij}^0 , v_{1iij}^1 , v_{1122}^0 , and v_{1122}^1 are the undetermined parameters. The best-fit values of the parameters together with the standard deviations σ of the calculated excess volumes and viscosity deviations and the average absolute deviations of the calculated kinematic viscosities for propylene glycol monomethyl ether + MAA, propylene glycol monomethyl ether + BzMA, and propylene glycol monomethyl ether + 2-HEMA are given in Table 9. As seen from Tables 6, 7, and 9, the standard deviations of the calculated excess volumes and viscosity deviations are approximately similar, regardless of the parameters treating as temperature-specific or temperaturedependent in the Redlich-Kister type polynomial. However, from Tables 8 and 9, the McAllister's multi-body interaction model with temperature-specific parameters yielded better calculated results than with temperature-dependent parameters did for those three investigated systems. These calculated results are also illustrated in Figures 2 to 4.

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