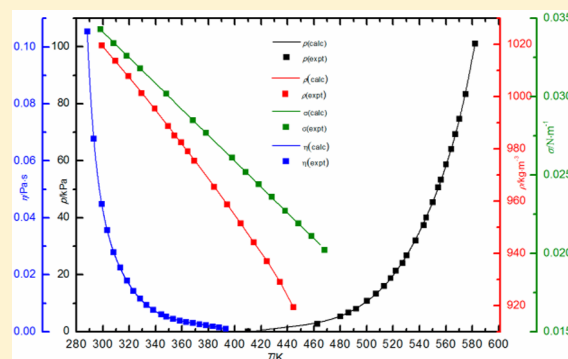


Vapor Pressure, Density, Viscosity, and Surface Tension of Tetrapropylene Glycol

Elena M. Fendu* and Florin Oprea

Petroleum Processing and Environmental Engineering Department, Universitatea Petrol-Gaze, Bd. București, 39 No., 100680, Ploiești, Romania

ABSTRACT: The properties of 2-(2-[2-(2-hydroxypropoxy)propoxy]propan-1-ol (tetrapropylene glycol, TePG) were measured as a function of temperature: vapor pressure (409.85 to 582.15) K, density (298.15 to 443.15) K, viscosity (288.15 to 393.15) K, and surface tension (298.15 to 468.15) K. The obtained data for tetrapropylene glycol were regressed using correlations from simulation software. We are developing a new industrial process to separate propylene glycols mixtures, obtained via the hydrolysis of 1,2-epoxy propane. One component of the mixture is not defined in the database of the simulation program. For synthesis and simulation of this process, it is necessary to have (1) the component TePG defined as a chemical structure, (2) the physical and transport properties of this component, and (3) vapor–liquid equilibrium data for the components of the mixture.



1. INTRODUCTION

A mixture of propylene glycol oligomers is obtained under elevated temperature and pressure by hydrolysis of 1,2-epoxy propane (propylene oxide, PO), a chemical intermediate whose global consumption exceeds 4.5 million tonnes/year.¹ This mixture is separated by distillation, to produce 1,2-propanediol (monopropylene glycol, further abbreviated as MPG) + 1-(2-hydroxypropoxy)propan-2-ol (dipropylene glycol, further abbreviated as DPG) preponderant in mixtures of isomers + 2-[2-(2-hydroxypropoxy)propoxy]propan-1-ol (tripropylene glycol, further abbreviated as TPG) preponderant in mixtures of isomers + 2-(2-[2-(2-hydroxypropoxy)propoxy]propan-1-ol (tetrapropylene glycol, further abbreviated as TePG) preponderant in mixtures of isomers + poly(propylene glycol) (further abbreviated as PPG). All propylene glycols have numerous applications, including use as antimicrobials, preservatives, antifreeze, as functional fluids, detergents, paints, textile industry, plasticizers, for obtaining unsaturated polyester resins, cosmetics, pharmaceuticals, food, humectants, and dehydrating agents.² We are developing a new industrial process to separate the propylene glycols mixture in order to obtain tetrapropylene glycol.³ The synthesis and simulation of this new industrial process requires good thermodynamic models (physical and transport properties and vapor–liquid equilibrium data). We can only obtain parameters of these models by using accurate vapor–liquid equilibrium experimental data for binary systems involved (water–propylene glycols and between propylene glycols). Because TePG is missing in the simulation software database, we had to define TePG as a new component (chemical structure). Additionally, we had to define physical and transport properties of the component. This paper reports experimental data for TePG: vapor pressure, liquid density, liquid dynamic

viscosity, and surface tension. The regression of experimental data for these properties has been performed using the most appropriate correlation of simulation software PRO/II.⁴ To validate the measurements we are comparing the tetrapropylene glycol properties with the same properties of propylene glycols (MPG, DPG, and TPG) available in the literature⁴ and with several experimental data sets determined for tripropylene glycol. Figure 1 shows the chemical structures for monopropylene glycol, dipropylene glycol, tripropylene glycol, and tetrapropylene glycol.

2. EXPERIMENTAL SECTION

2.1. Materials. Tetrapropylene glycol used for the experimental determination of physical and transport properties is obtained from a mixture of higher propylene glycols available from Dow Chemical Company. This mixture contains 0.98 mass fraction TPG and TePG according to the certificate of analysis. We have experimentally determined the true boiling points curve (Figure 2) using a high efficiency column (DX Sulzer lab packing, 30 mm inner diameter and 1.2 m height). We identified at 0.04 kPa tetrapropylene glycol in the distillation range (36 to 89) % volume at (422.65 to 423.15) K, as shown in Figure 2. This fraction was separately collected and purified again using the same Sulzer column and subsequently dried. The composition of the final fraction of TePG, separated in the second step was analyzed with the Clarus 500 instrument using an Elite WAX (0.32 mm × 60 m × 0.25 mm) column. Owing to the difficult

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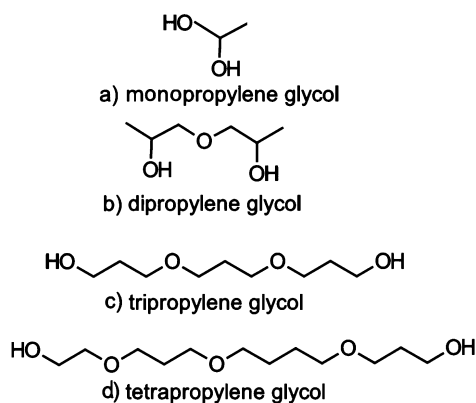


Figure 1. Chemical structure for (a) monopropylene glycol, (b) dipropylene glycol, (c) tripropylene glycol, and (d) tetrapropylene glycol.

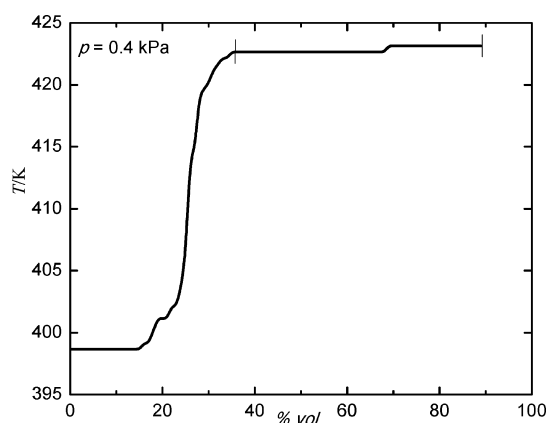


Figure 2. True boiling point curve for higher propylene glycols mixture that correlates boiling temperature T with percentage volume % vol at pressure p of 0.4 kPa. The curve between the vertical bars denotes the tetrapropylene glycol fraction.

identification of TePG isomers, we decided to use in our experimental measurements a mixture of the isomers in the proportion resulting from reaction. Further we will use the nomenclature tetrapropylene glycol or the abbreviation TePG for the isomers mixture with molar mass $250 \text{ g}\cdot\text{mol}^{-1}$, normal boiling temperature experimentally determined 582.2 K , and chemical formula $\text{C}_{12}\text{H}_{26}\text{O}_5$. Descriptions of the TePG and TPG (also used in experiment) samples are presented in Table 1.

The pressure was measured using a DPI 705 sensor with the measuring range between 0 and $1.01 \cdot 10^5 \text{ Pa}$ and the temperature was measured with VWR International, LLC, NIST traceable digital thermometers ($\pm 0.05 \%$ accuracy and 0.001 K resolution).

2.2. Apparatus and Procedure. Tetrapropylene glycol vapor pressure is experimentally determined using a total pressure apparatus built in our laboratory. This static apparatus of our laboratory is shown schematically in Figure 3. In this figure the numbers in brackets represent the parts of the apparatus. A

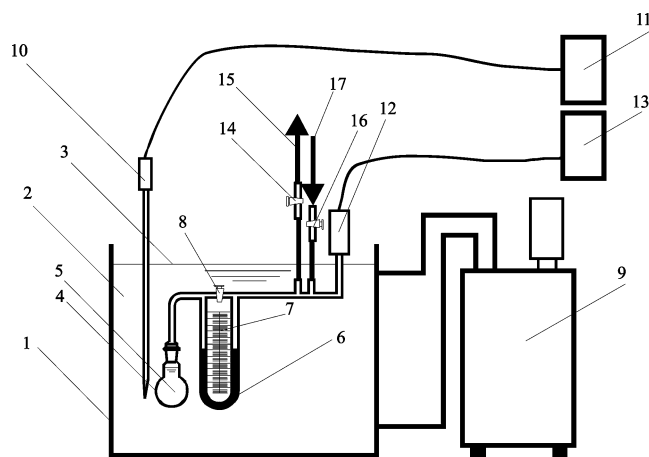


Figure 3. Operation scheme of static apparatus used to determine tetrapropylene glycol vapor pressure. 1, transparent glass-walled bath; 2, oil; 3, oil level in thermostatic bath; 4, equilibrium cell; 5, sample of TePG; 6, U-shaped tube with mercury; 7, graduated scale; 8, coupling valve; 9, constant temperature bath (cold reservoir source); 10, temperature sensor; 11, digital thermometer; 12, pressure sensor; 13, digital pressure indicator; 14, admission valve; 15, vacuum; 16, air valve; 17, air.

sample of TePG (5) is introduced into the equilibrium cell (4). The cell is connected to a U-shaped tube (6) containing a manometric liquid, mercury. The degassing of the sample using liquid nitrogen and a vacuum pump was a standard procedure, before the vapor pressure measurement. The solution was frozen near the liquid nitrogen temperature, and degassed using the vacuum pump to remove any gaseous impurities for 20 min.⁵ This step was performed when coupling valve (8) was open. After degassing, coupling valve (8) is closed and the equilibrium cell and the U-shaped tube are placed in transparent glass-walled bath (1) with thermostatic oil (2), where the desired temperature was maintained within $\pm 0.05 \text{ K}$. By heating the TePG sample, vapors are emitted that cause the oscillation of the level in the U-shaped tube. When level of the mercury remains constant (pressure of the system did not change for 30 min), air is introduced in the system through a valve (16) until the level of the mercury in the tube is the same in the two branches as it can be seen on a graduated scale (7). At this point the vapor pressure and temperature are measured, respectively, using a pressure sensor (12) and digital pressure indicator (13) and a temperature sensor (10) and digital thermometer (11). Three replicates were performed for each state condition. The standard uncertainty⁶ for temperature was $\pm 0.01 \text{ K}$ and the relative standard uncertainty⁶ for vapor pressure was 0.01.

Densities of TePG are measured using a calibrated glass pycnometer having a bulb volume of 10 cm^3 .⁷ The volume of the pycnometer was calibrated as a function of temperature using distilled, deionized, and degassed water at various temperatures. The pycnometer filled with liquid was kept in a thermostatically controlled oil bath (maintained constant to $\pm 0.05 \text{ K}$) for (20 to 25) min to reach thermal equilibrium. The density measure-

Table 1. Chemical Sample Description for Tripropylene Glycol and Tetrapropylene Glycol

chemical name	source	initial mass fraction purity	purification method	final mass fraction purity	analysis method
tripropylene glycol	Dow Chemical	0.9993	none		
tetrapropylene glycol	Dow Chemical	0.7551	distillation	0.994	GC ^a

^aGas–liquid chromatography.

ments were carried out at various temperatures. Each experimental density value was an average of at least three measurements. The estimated uncertainties⁶ for temperature and density were ± 0.01 K and ± 0.14 kg·m⁻³, respectively.

The liquid dynamic viscosity of TePG (η) was obtained by multiplying the experimental determined kinematic viscosity (ν) by the measured density (ρ). The kinematic viscosity of TePG was performed with two commercial capillary viscometers of Ubbelohde type which have capillary diameters of 0.78 mm and 0.88 mm.⁹ Double distilled, deionized, and degassed water was used for the calibration.¹⁰ The flow time was determined with a digital stopwatch capable of measuring time within ± 0.01 s. Measurements were accomplished in transparent glass-walled water and glycerin baths, depending on the desired temperature, with a thermal stability of ± 0.01 K. Each measurement was repeated at least five times, and the results were averaged. The kinematic viscosity (ν) was calculated from the following equation:

$$\nu = k \cdot t \quad (1)$$

where ν is the kinematic viscosity, t is the flow time of the liquid in the viscometer, and k is the calibration viscometer constants provided by the manufacturer that has values of 0.1193 for range of temperature (288.15 to 333.15) K and 0.02853 for range (338.15 to 393.15) K. The relative standard uncertainty⁶ for dynamic viscosity was 0.01.

Surface tension of TePG was determined using a Traube stalagmometer by drop method, where the number of drops of the liquid and water were measured at constant temperature.¹¹ The measurements were done in three replicates, and the average values are reported. We estimate the uncertainties⁶ in our measurements to be within ± 0.01 K for temperature and ± 0.0001 N·m⁻¹ for surface tension.

3. RESULTS AND DISCUSSION

Experimental data for TePG properties were regressed using the most appropriate correlation of simulation software. Correlations of experimental data used in regression were chosen taking into account the correlations available in the simulation software for MPG, DPG, and TPG properties.⁴

3.1. Vapor Pressure. The results for the vapor pressure measurements of tetrapropylene glycol for temperatures from (409.85 to 582.15) K are presented in Table 2. The vapor

Table 2. Experimental Values of Vapor Pressure p for Tetrapropylene Glycol at Different Temperatures T^a

T/K	p/kPa	T/K	p/kPa
409.85	0.01	537.15	32.05
462.55	2.68	543.15	37.42
480.15	5.34	545.15	40.02
486.15	6.66	550.15	45.37
492.15	8.02	554.15	50.69
500.15	10.72	556.15	53.36
507.15	13.36	560.15	58.74
513.15	16.05	564.15	64.02
518.15	18.79	567.15	69.28
522.15	21.34	570.15	74.63
527.15	24.02	575.15	83.34
530.15	26.71	582.15	101.02

^aStandard uncertainty u is $u(T) = 0.01$ K and relative standard uncertainty u_r is $u_r(p) = 0.01$.

pressure of tetrapropylene glycol was measured at higher temperatures, compared to other properties presented, because at lower temperatures than these the vapor pressure has very low values and is very difficult to obtain. The vapor pressure experimental data were regressed. In Figure 4 are presented the

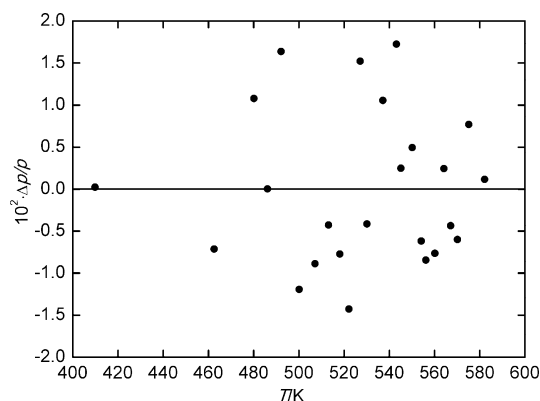


Figure 4. Relative deviations $\Delta p/p = \{p(\text{calc}) - p(\text{expt})\}/p(\text{calc})$ of the calculated vapor pressures $p(\text{calc})$ with eq 2 from those determined experimentally $p(\text{expt})$ for tetrapropylene glycol.

relative deviations $\Delta p/p$ between the calculated vapor pressures $p(\text{calc})$ and those experimentally determined $p(\text{expt})$. Equation 2 represents the specific correlation for vapor pressure of TePG as function of temperature written with the values of the coefficients obtained from the experimental data regression.

$$\ln(p/kPa) = 1.7975 \cdot 10^5 - \frac{6.4082 \cdot 10^6}{T} - 28739 \ln T + 0.079537 \cdot T^2 - 6.7778 \cdot 10^{-5} \cdot T^3 \quad (2)$$

Furthermore, we obtained additional experimental points for TPG vapor pressure for comparison with PRO II database.⁴ Thus, our experimental procedure and the apparatus for the vapor pressure measurements was verified. The experimental vapor pressures and vapor pressure curve traced with eq 2 of TePG, the experimental points of TPG, and vapor pressure curves for others propylene glycols as MPG, DPG, and TPG from PRO II database,⁴ are presented in the Figure 5. The vapor pressure of propylene glycols increases with the increase in temperature and decreases with increase in molar mass at fixed temperature. The low vapor pressures of propylene glycols compared with those of water, situate the propylene glycols in the class of high boiling point liquids. The low volatility of propylene glycols reduces their tendency to evaporate and leads to their use as plasticizers, antifreeze, and hygroscopic agents.

3.2. Density. Table 3 contains experimental values of densities for tetrapropylene glycol determined in the range from (298.15 to 443.15) K. After the experimental data regression, according to PRO II software, the relative deviations $\Delta \rho/\rho$ of the calculated liquid densities $\rho(\text{calc})$ from those determined experimentally $\rho(\text{expt})$ were determined and are presented in Figure 6. The specific correlation of tetrapropylene glycol density as function of temperature is given by eq 3.

$$\rho/\text{kg} \cdot \text{m}^{-3} = 1041.2 - 1.3992 \cdot T + 0.036660 \cdot T^2 - 0.00080351 \cdot T^3 \quad (3)$$

Figure 7 shows the DPG and the TPG curves of densities as function of temperature, data available in PRO II database, in comparison with a few experimental points of TPG density and

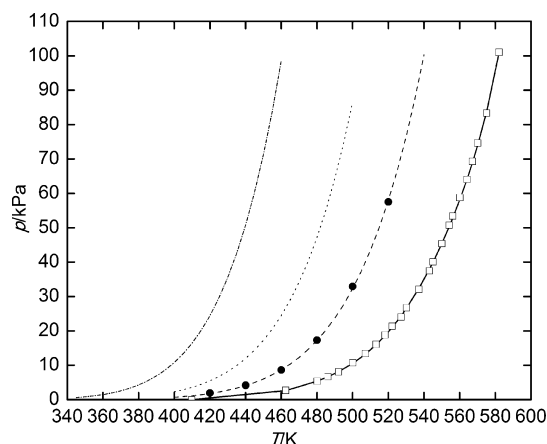


Figure 5. Vapor pressure curves of propylene glycols. —, 1,2-propanediol vapor pressure curve from PRO II database; ···, dipropylene glycol vapor pressure curve from PRO II database; — — —, tripropylene glycol vapor pressure curve from PRO II database; ●, experimental vapor pressures of TPG; □, experimental vapor pressures of TePG; — · — · —, vapor pressure curve for TePG traced with regress eq 2.

Table 3. Experimental Values of Liquid Density ρ at Temperature T and Pressure $p = 0.1$ MPa for Tetrapropylene Glycol^a

T/K	$\rho/\text{kg m}^{-3}$	T/K	$\rho/\text{kg m}^{-3}$
298.15	1019.6	368.15	975.5
308.15	1013.7	383.15	965.4
318.15	1007.9	393.15	958.7
328.15	1001.3	403.15	951.4
338.15	995.4	413.15	944.3
348.15	988.8	423.15	937.0
353.15	985.1	433.15	928.9
358.15	982.5	443.15	919.4
363.15	979.0		

^aStandard uncertainties u are $u(T) = 0.01$ K, $u(p) = 10$ kPa, and the combined expanded uncertainty U_c is $U_c(\rho) = 0.14 \text{ kg} \cdot \text{m}^{-3}$ (0.95 level of confidence).

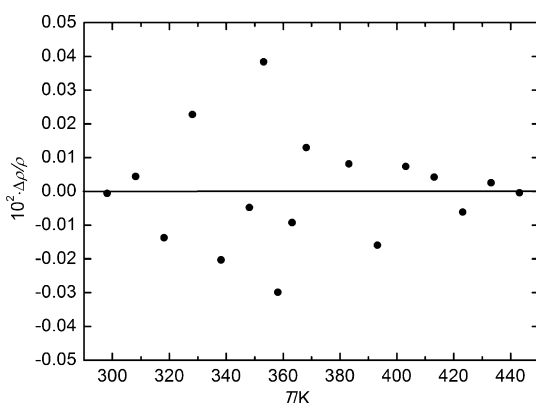


Figure 6. Relative deviations $\Delta\rho/\rho = \{\rho(\text{calc}) - \rho(\text{expt})\}/\rho(\text{calc})$ of the calculated liquid densities $\rho(\text{calc})$ with eq 3 as a function of temperature from those determined experimentally $\rho(\text{expt})$ for tetrapropylene glycol.

with experimental and calculated values of TePG density. As it can be seen in this figure the density decreases with the increase in temperature for propylene glycols and increases with the increase in molar mass at fixed temperature. At a constant

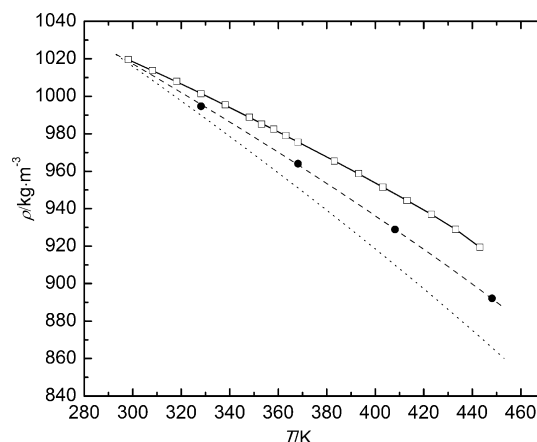


Figure 7. Variation of liquid density ρ with temperature T for higher propylene glycols: ···, dipropylene glycol from PRO II database; — — —, tripropylene glycol from PRO II database; — · — · —, tetrapropylene glycol from PRO II database; ●, experimental liquid density of TPG; □, experimental liquid density of TePG; —, liquid density curve for TePG traced with regress eq 3.

temperature, it can be observed that the differences in density between these three propylene glycols are roughly equal. This is due to the difference of chemical structure of DPG, TPG, and TePG, more specifically assignment of a propoxy group in this homologue series.

3.3. Viscosity. Table 4 contains the experimental values of viscosities of tetrapropylene glycol determined in the range from

Table 4. Experimental Values of Liquid Dynamic Viscosity η at Temperature T and Pressure $p = 0.1$ MPa for Tetrapropylene Glycol^a

T/K	$\eta/\text{Pa} \cdot \text{s}$	T/K	$\eta/\text{Pa} \cdot \text{s}$
288.15	0.10544	344.15	0.00604
293.15	0.06781	348.15	0.00522
299.15	0.04489	353.15	0.00442
303.15	0.03566	358.15	0.00384
308.15	0.02785	363.15	0.00338
313.15	0.02248	368.15	0.00300
318.15	0.01797	373.15	0.00262
323.15	0.01432	378.15	0.00228
328.15	0.01158	383.15	0.00190
333.15	0.00939	388.15	0.00148
338.15	0.00765	393.15	0.00102

^aStandard uncertainties u are $u(T) = 0.01$ K, $u(p) = 10$ kPa, and the relative standard uncertainty u_r is $u_r(\eta) = 0.01$.

(288.15 to 393.15) K. Figure 8 shows the relative deviations $\Delta\eta/\eta$ of the liquid dynamic viscosities $\eta(\text{calc})$ calculated with eq 4 as a function of temperature from those experimentally determined $\eta(\text{expt})$.

$$\ln(\eta/\text{Pa} \cdot \text{s}) = -2.2932 \cdot 10^5 + \frac{5.6583 \cdot 10^6}{T} + 39443 \ln T - 0.26295 \cdot T^2 + 0.00034739 \cdot T^3 \quad (4)$$

Experimental and calculated data sets of TePG viscosities are compared with viscosities data sets for DPG and TPG available in the literature⁴ and with several experimental values for TPG (Figure 9). For temperatures less than 310 K the dynamic viscosities are different, for temperatures from (315 to 350) K the TePG dynamic viscosity values are roughly equal to TPG

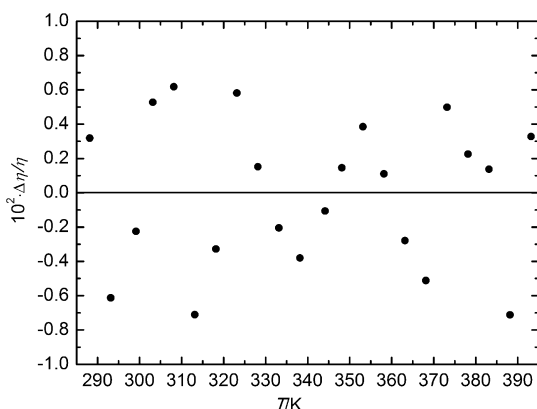


Figure 8. Relative deviations $\Delta\eta/\eta = \{\eta(\text{calc}) - \eta(\text{expt})\}/\eta(\text{calc})$ of the calculated liquid dynamic viscosities with eq 4 $\eta(\text{calc})$ as a function of temperature from those determined experimentally $\eta(\text{expt})$ for tetrapropylene glycol.

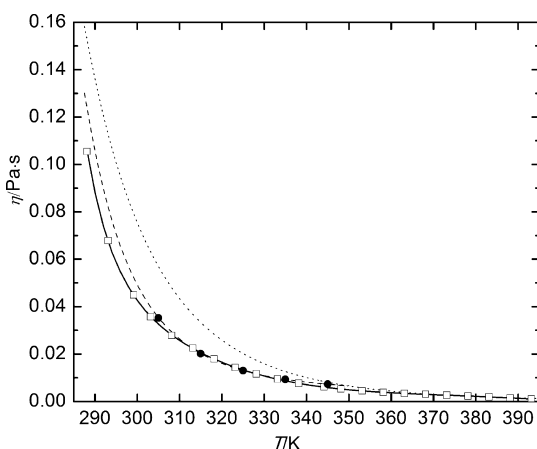


Figure 9. Variation of liquid dynamic viscosities η with temperature T for higher propylene glycols: \cdots , dipropylene glycol from PRO II database; $---$, tripropylene glycol from PRO II database; \bullet , experimental liquid dynamic viscosity of TPG; \square , experimental liquid dynamic viscosity of TePG; $—$, liquid dynamic viscosity curve for TePG traced with regress eq 4.

viscosity values. For temperatures greater than 370 K all the three glycols have almost the same dynamic viscosities. This property is not influenced by the number of propoxy groups existing in the chemical structure of propylene glycol. Propylene glycols flow freely at high temperatures, while at low temperatures their viscosities increase until they eventually set and fail to flow. The variation of viscosity with temperature is necessary for pumping and piping calculations.²

3.4. Surface Tension. The experimental surface tension values of tetrapropylene glycol ranging from (298.15 to 468.15) K are reported in Table 5. The data were regressed and the specific correlation of TePG surface tension as a function of temperature was obtained (eq 5). The relative deviations $\Delta\sigma/\sigma$ of the calculated surface tension $\sigma(\text{calc})$ from those determined experimentally $\sigma(\text{expt})$ are presented in Figure 10.

$$\sigma/\text{N}\cdot\text{m}^{-1} = 0.036355 - 8.2573 \cdot 10^{-5} \cdot T \quad (5)$$

The surface tension of dipropylene, tripropylene, and tetrapropylene glycols against temperature is given in Figure 11. It can be seen that the surface tension decreases with the increase in temperature and increases with the increase in molar

Table 5. Experimental Values of Surface Tension σ at Temperature T and Pressure $p = 0.1$ MPa for Tetrapropylene Glycol^a

T/K	$\sigma/\text{N}\cdot\text{m}^{-1}$
298.15	0.0343
308.15	0.0334
318.15	0.0326
328.15	0.0318
348.15	0.0302
368.15	0.0285
378.15	0.0277
398.15	0.0261
408.15	0.0252
418.15	0.0244
428.15	0.0236
438.15	0.0227
448.15	0.0219
458.15	0.0211
468.15	0.0202

^aStandard uncertainties u are $u(T) = 0.01$ K, $u(p) = 10$ kPa, and the combined expanded uncertainty U_C is $U_C(\sigma) = 0.0001$ N·m⁻¹ (0.95 level of confidence).

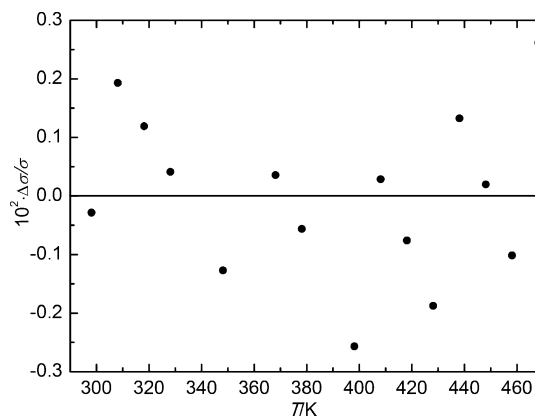


Figure 10. Relative deviations $\Delta\sigma/\sigma = \{\sigma(\text{calc}) - \sigma(\text{expt})\}/\sigma(\text{calc})$ of the calculated surface tension with eq 5 $\sigma(\text{calc})$ as a function of temperature from those determined experimentally $\sigma(\text{expt})$ for tetrapropylene glycol.

mass at fixed temperature for propylene glycols. The variation of surface tension of these propylene glycols with temperature is quasi-linear in the temperature range (298.15 to 468.15) K. Surface tension differences between propylene glycols presented are uniform due to the presence of a new propoxy group in the chemical structure of the respective glycol. Surface tension is an important property when the wetting or penetrating ability of a liquid is considered. Surface tensions of propylene glycols are lower than those of water.²

The temperature range for experimental data of liquid density, liquid dynamic viscosity, and surface tension was from (288.15 to 468.15) K, because these are the temperature limits of the new industrial process to separate the propylene glycols mixture that we are developing.

4. CONCLUSIONS

In this work we presented experimental data for vapor pressure ranging from (409.85 to 582.15) K, the liquid density ranging from (298.15 to 443.15) K, the liquid dynamic viscosity ranging from (288.15 to 393.15) K, and the surface tension ranging from

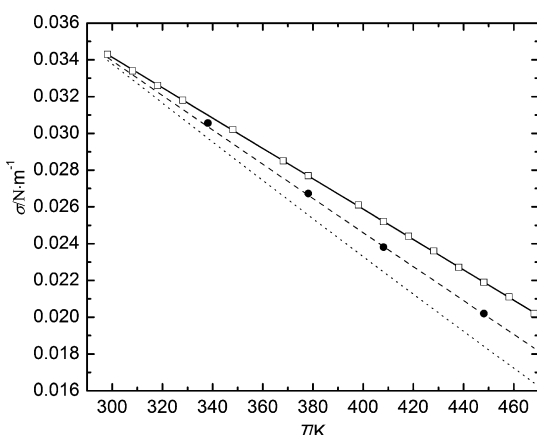


Figure 11. Variation of surface tension σ with temperature T for higher propylene glycols: \cdots , dipropylene glycol from PRO II database; $-\cdots-$, tripropylene glycol from PRO II database; \bullet , experimental surface tension of TPG; \square , experimental surface tension of TePG; $-$, surface tension curve for TePG traced with regress eq 5.

(298.15 to 468.15) K of tetrapropylene glycol. Our physical and transport properties measurements of tetrapropylene glycol were correlated as functions of temperature. The obtained correlations of the TePG properties present the next maximum relative deviations: 1.72% for vapor pressure, 0.0384% for density, 0.712% for dynamic viscosity, and 0.262% for surface tension. Furthermore, the tetrapropylene glycol properties reported in this study are sufficiently accurate for the design of a new industrial process for obtaining TePG from a glycols mixture.

AUTHOR INFORMATION

Corresponding Author

*Tel.: +40244 576 211. Fax: +40244 575847. E-mail: emfendu@upg-ploiesti.ro.

Notes

The authors declare no competing financial interest.

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