

# Thermophysical Properties of Glycols and Glymes

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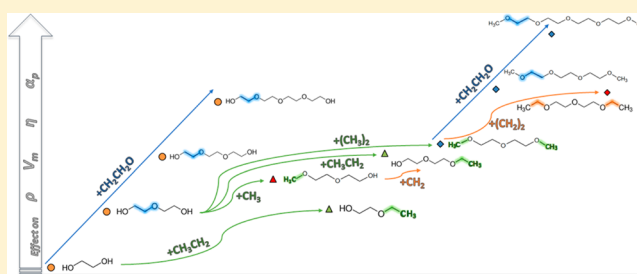
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## Supporting Information

**ABSTRACT:** Experimental data for density, viscosity, refractive index and sound speed of 11 glycols and glymes were measured in the temperature range between (283.15 and 373.15) K and at atmospheric pressure. The compounds evaluated include ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol (TriEG), tetraethylene glycol (TeEG), ethylene glycol ethyl ether (EGEE), diethylene glycol methyl ether (DEGME), diethylene glycol dimethyl ether (DEGDME), diethylene glycol ethyl ether (DEGEE), diethylene glycol diethyl ether (DEGDDE), triethylene glycol dimethyl ether (TriEGDME), and tetraethylene glycol dimethyl ether (TeEGDME). Additionally, derivative properties, such isobaric thermal expansion coefficient and isentropic compressibilities were also estimated and discussed. The compounds evaluated were selected to evaluate the impact of molecular structure changes on their thermophysical properties. Effects like the increase in the number of ethoxy groups, shown to lead to an increase of the density, molar volume, viscosity, and refractive index, or the loss of the hydroxyl groups through the substitution of the hydroxyl groups hydrogen by a methyl or ethyl group, shown to lead to a significant decrease on the density, viscosity, and sound speed, are evaluated and discussed.



## 1. INTRODUCTION

Glycols are organic compounds primarily used as raw material in the polymer industry because of their high boiling points, hygroscopicity, noncorrosiveness, freezing point depression, lubricating, and plasticizing and solvent properties. Additionally, their alcohol end groups makes them popular intermediates in the preparation of numerous esters. Among the glycols, ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol (TriEG), and tetraethylene glycol (TeEG) are the first four members of a homologous series of diols commonly used. The EG hygroscopicity is ideal for use in fibers treatment, paper, adhesives, printing inks, leather, and cellophane. Furthermore, EG is used as precursor for polyester fibers, polyethylene terephthalate plastics, coolants in automobile antifreeze, and resins. Diethylene glycol is an important chemical intermediate in the manufacture of unsaturated polyester resins, polyurethanes, and plasticizers. Moreover, both DEG and TriEG are used in natural gas processing as dehydration agents. TriEG is also used on air conditioning systems as dehumidifiers, as a vinyl plasticizer, or as an intermediate in the manufacture of polyester resins and polyols. On the other hand, TeEG is used in polyester resins, as a plasticizer, as a chemical intermediate, as a solvent in the production of inks and dyes, as a process solvent in hydrocarbon purification processes, and as a coupling agent in the production of textile lubricants and formulations.<sup>1</sup>

Glycol ethers or glymes on the other hand, are aprotic, saturated polyethers that present high solvency, high stability in strong bases, and moderate stability in acid solutions. Glymes efficiently solvate cations and can increase anion reactivity, thus increasing both selectivity and reaction rates. Furthermore, glymes are used as reaction solvents, in closed loop applications such as gas scrubbing, and in refrigeration systems. The higher molecular weight glymes beginning with diethylene glycol diethyl ether (DEGDDE) are suitable for emissive applications such as in coatings, inks, adhesives, and cleaning compounds.

The constant development of new technologies and the demand for new products, compel the industry to continuously optimize existing processes. Moreover, this development and optimization usually rely on process simulators that require accurate description of the thermophysical properties of the compounds present. Nonetheless, for the majority of the glycols and glymes, the thermophysical properties are not fully and accurately characterized. Thus, the characterization of these compounds' thermophysical properties, like density, viscosity,

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**Table 1. Chemical Structure, Compound Description, CAS Number, Molecular Weight, Water Mass Fraction Content, Mass Fraction Purity and Supplier of the Studied Glycols and Glymes**

Compound	Chemical structure
Ethylene glycol (EG) (CAS:107-21-1 ; Mw = 62.07 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 7.9·10 <sup>-4a</sup> ; wt% = 99.5% <sup>b</sup> ) acquired from Fluka	
Diethylene glycol (DEG) (CAS:111-46-6 ; Mw = 106.12 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.6·10 <sup>-3a</sup> ; wt% = ≥99% <sup>b</sup> ) acquired from Sigma Aldrich	
Triethylene glycol (TriEG) (CAS:112-27-6 ; Mw = 150.17 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.2·10 <sup>-3a</sup> ; wt% = ≥99% <sup>b</sup> ) acquired from Sigma Aldrich	
Tetraethylene glycol (TeEG) (CAS:112-60-7 ; Mw = 194.23 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.9·10 <sup>-3a</sup> ; wt% = 99% <sup>b</sup> ) acquired from Sigma Aldrich	
Ethylene glycol ethyl ether (EGEE) (CAS:110-80-5 ; Mw = 90.12 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 4.1·10 <sup>-3a</sup> ; wt% = 99% <sup>b</sup> ) acquired from Panreac	
Diethylene glycol methyl ether (DEGME) (CAS:111-77-3 ; Mw = 120.15 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.5·10 <sup>-3a</sup> ; wt% = 99% <sup>b</sup> ) acquired from Acos Organic	
Diethylene glycol dimethyl ether (DEGDME) (CAS:111-96-6 ; Mw = 134.17 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.6·10 <sup>-2a</sup> ; wt% = 99.5% <sup>b</sup> ) acquired from Sigma Aldrich	
Diethylene glycol ethyl ether (DEGEE) (CAS:111-90-0 ; Mw = 134.17 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 2.9·10 <sup>-4a</sup> ; wt% = ≥98% <sup>b</sup> ) acquired from Acros Organic	
Diethylene glycol diethyl ether (DEGDDE) (CAS:112-36-7 ; Mw = 162.23 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 6.6·10 <sup>-4a</sup> ; wt% = ≥98% <sup>b</sup> ) acquired from Fluka	
Triethylene glycol dimethyl ether (TriEGDME) (CAS:112-49-2 ; Mw = 178.23 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.5·10 <sup>-4a</sup> ; wt% = 99% <sup>b</sup> ) acquired from Fluka	
Tetraethylene glycol dimethyl ether (TeEGDME) (CAS:143-24-8 ; Mw = 222.28 g mol <sup>-1</sup> ; H <sub>2</sub> O wt = 1.7·10 <sup>-4a</sup> ; wt% = ≥99% <sup>b</sup> ) acquired from Fluka	

<sup>a</sup>after moderate temperature and vacuum drying procedure. <sup>b</sup>as reported by the supplier.

speed of sound, and refractive index, is of key relevance if one aims at their accurate characterization, or the development/optimization of theoretical models, correlations, or equations of state (EoS). Aiming to overcome these limitations, a set of compounds were selected, namely, ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol (TriEG), tetraethylene glycol (TeEG), ethylene glycol ethyl ether (EGEE), diethylene glycol methyl ether (DEGME), diethylene glycol dimethyl ether (DEGDME), diethylene glycol ethyl ether (DEGEE), diethylene glycol diethyl ether (DEGDDE), triethylene glycol dimethyl ether (TriEGDME), and tetraethylene glycol dimethyl ether (TeEGDME), in order to fully characterize their thermo-physical properties on the (283.15 to 373.15) K temperature range and at atmospheric pressure. Apart from their properties characterization, the set of compounds selected allows the study of the effect of the molecule chain length, and therefore the increase of the number of ether groups, and the effect of replacing the hydrogen(s) of the glycols hydroxyl group(s)

with a methyl or with an ethyl group on the compounds properties.

## 2. EXPERIMENTAL SECTION

**2.1. Materials.** Eleven glycols and glymes were studied in this work, namely, ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol (TriEG), tetraethylene glycol (TeEG), ethylene glycol ethyl ether (EGEE), diethylene glycol methyl ether (DEGME), diethylene glycol dimethyl ether (DEGDME), diethylene glycol ethyl ether (DEGEE), diethylene glycol diethyl ether (DEGDDE), triethylene glycol dimethyl ether (TriEGDME), and tetraethylene glycol dimethyl ether (TeEGDME). The compounds chemical structures, purities, suppliers, and corresponding designations are presented in Table 1.

It is well established that even small amounts of water and other impurities have a great impact in the compound's properties, especially on transport properties like viscosity.

Therefore, in order to remove traces of water and volatile compounds, individual samples of each glycol were dried at moderate temperature ( $\approx 323$  K), vacuum ( $\approx 0.1$  Pa), and under continuous stirring for a minimum of 48 h prior to the measurements. The only exceptions were the diethylene glycol dimethyl ether (DEGDME) and ethylene glycol ethyl ether (EGEE) that were too volatile for this treatment and would distill under these conditions. For these compounds, a purification by distillation was carried. From a sample of 25 mL, an initial fraction of about 5 mL was discarded in order to remove water and solvents. The remaining 15 mL of compound was further distilled and the distillate used; the remaining compound was also discarded. Before each measurement, the compound was further distilled (discarding small initial fractions) to remove traces of water adsorbed during the compound manipulation. The purity of each glycol was checked by  $^1\text{H}$  and  $^{13}\text{C}$  NMR, both before and after the measurements, to ensure that no degradation occurred. The final water content, after the drying step and immediately before the measurements, was determined with a Metrohm 831 Karl Fischer coulometer (using the Hydranal–Coulomat AG from Riedel-de Haën as analyte). The average water content, the molecular weight, and the mass purity of each glycol are presented in Table 1.

**2.2. Density, Viscosity, and Speed of Sound.** Two approaches were used to determine the compound's density. One using a SVM3000 rotational Stabinger viscometer–densimeter and other using a DSA 5000 M vibrating tube densimeter and ultrasound speed meter both from Anton Paar. Using the automated SVM3000 Anton Paar rotational Stabinger viscometer–densimeter, density ( $\rho$ ) and dynamic viscosity ( $\eta$ ) were determined in the temperature range from (283.15 to 373.15) K and at atmospheric pressure ( $0.1 \pm 0.01$  MPa) within a standard uncertainty on the density of (0.5, 1 and 2)  $\text{kg}\cdot\text{m}^{-3}$  for the samples of 99.5%, 99% and 98% purity, respectively, and 2 % on the dynamic viscosity. The standard uncertainty on the temperature is 0.02 K. Further details regarding the use of the equipment and methodologies for the determination of densities and viscosities can be found elsewhere.<sup>2</sup> As will be shown and discussed below, some compounds present very low viscosity values. Thus, in order to experimentally determine these viscosities, the SVM3000 viscometer–densimeter was calibrated for low viscosities by using *n*-octane as the reference fluid and validated using other low viscosity compounds. Within this low viscosity calibration (0.360 to 1.271) mPa·s the dynamic viscosity standard uncertainty obtained was 2.8 %.

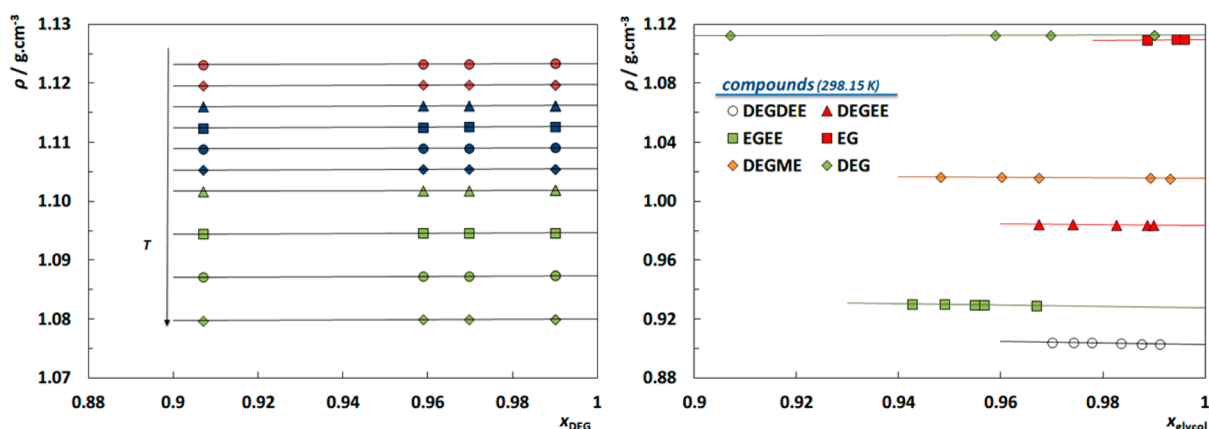
Using the DSA 5000 M vibrating tube densimeter and ultrasound speed meter (ultrasound transducer frequency of 3 MHz),

density and speed of sound were measured at atmospheric pressure and in the (283.15 to 343.15) K temperature range. The standard uncertainty on the density of this equipment is (0.5, 1 and 2)  $\text{kg}\cdot\text{m}^{-3}$  for the samples of 99.5%, 99% and 98% purity, respectively, and  $1.0\text{ m}\cdot\text{s}^{-1}$  on the speed of sound. Before each measurement, all samples were heated in an oven, under nitrogen, at the maximum experimental temperature. Then, for the same single sample, a complete series of measurements were made, decreasing the temperature from (343.15 to 283.15) K in decrements of 10 K until 313.15 K and in decrements of 5 K from therein downward. At each temperature, three to seven data readings were taken and some measurements were repeated with a new injection, allowing asserting an estimate for the repeatability and reproducibility for density values lower than 0.0005 % and 0.004 %, respectively, and for speed of sound of 0.01 % and 0.07 %, respectively. The standard uncertainty on the temperature is 0.01 K. Further details regarding the use of the equipment, calibration, and methodologies for the determination of densities and speed of sound can be found elsewhere.<sup>3</sup>

**2.3. Refractive Index.** Measurements of refractive index ( $n_D$ ) were performed at 589.3 nm using an automated Abbemat 500 Anton Paar refractometer. Refractive index measurements were carried out in the temperature range from (283.15 to 353.15) K and at atmospheric pressure. The Abbemat 500 Anton Paar refractometer uses reflected light to measure the refractive index, where the sample on the top of the measuring prism is irradiated from different angles by a light-emitting diode (LED). The standard uncertainty in temperature is 0.05 K, and  $2 \times 10^{-4}$   $n_D$  on the refractive index.

### 3. RESULTS AND DISCUSSIONS

**3.1. Density.** The density measurements were carried out at atmospheric pressure and in the temperature range from (283.15 to 373.15) K using the SVM3000 Anton Paar rotational Stabinger viscometer–densimeter and in the (283.15 to 343.15) K temperature range using the DSA 5000 M vibrating tube densimeter and ultrasound speed meter. Even though both equipment use the same technique to determine the density two different approaches were adopted in the determination of the compounds densities. Although for the SVM3000, all the compounds were purified as described above, for the DSA 5000 M measurements, the densities of EG, DEG, DEGDME, EGEE, DEGEE, and DEGDDEE were determined by extrapolating the compound aqueous mixtures densities, with known compositions, using a linear fit, as depicted in Figure 1.



**Figure 1.** Density as a function of DEG (left) and glycol (right) mole fractions. The solid lines represent the linear fit to the experimental data and the extrapolation to water mole fractions down to zero.

These solutions were prepared using high-purity water from a Milli-Q system from Millipore and kept in special tight flasks with Teflon caps to prevent evaporation and contamination by contact with air. The aqueous mixtures compositions and densities are reported in Table S1 in the Supporting Information. The standard uncertainty in solution composition, expressed in mole fraction, was estimated to be less than 0.001 and the standard uncertainty of the extrapolated densities of the pure compounds, based on the quality of the fits and calculated with the error propagation method, were found to range from (1.1 to 0.04)  $\text{kg}\cdot\text{m}^{-3}$  for EG and DEGEE, respectively (the complete list of the extrapolated densities expanded uncertainties are reported in Table S3 in the Supporting Information). The densities for the remaining four compounds were determined through their measurements after the drying procedure following the same procedure adopted for the SVM3000.

Using the two approaches, only small average relative deviations

$$\%AD = \sum_{i=1}^N \frac{\rho_{\text{DSA5000M}} - \rho_{\text{SVM3000}}}{\rho_{\text{SVM3000}}} \cdot 100 \quad (1)$$

of  $-0.14\%$  ( $\Delta\rho_{\text{max}} = -0.0014 \text{ g}\cdot\text{cm}^{-3}$ ) were observed between the two equipment and methodologies, as depicted in Figure 2.

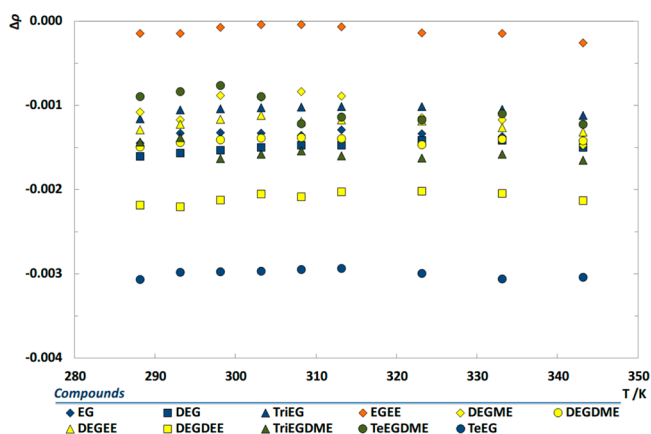


Figure 2. Density deviations (density values used in  $\text{g}\cdot\text{cm}^{-3}$ ) between the SVM3000 and the DSA 5000 M results as a function of temperature, for the studied compounds.

Considering the small deviations observed between the two equipment and methodologies adopted and the fact that the density measurements obtained using the SVM3000 density-viscometer were performed using the pure ILs, the results obtained from the SVM3000 will be used this point onward as the experimental density data for the discussion and literature comparison.

Inherently to their industrial interest, density values for these compounds are widely available in the literature (a summary of all the literature density data used in this work is provided in Table S5 in the Supporting Information).<sup>4–55</sup> Overall, small and nonsystematic relative deviations between the data determined here and that available in the literature is observed; nonetheless, some discrepancies among different authors are found, as depicted in Figure 3. Percentage absolute average deviations, %AAD, of 0.21 % and 0.17 % for the glycols and the remaining compounds, respectively, are observed. Moreover, the percentage absolute average deviations found among the literature data is larger than those observed between the two approaches used.

The density values, for the studied compounds, are reported in Table 2 and depicted in Figure 4. As depicted in Figures 4 and 5, and commonly observed for other common organic compounds, the glycols' densities increase with the number and molar density of the ethoxy groups.

The substitution of one hydrogen in one of the hydroxyl groups by a  $\text{CH}_3$  leads to an important decrease on the density, as seen in Figure 5 for the case of DEG–EGME and TriEG–DEGME. Moreover, the substitution of both hydrogens on the diols hydroxyl groups, by either an ethyl or methyl group, leads also to a decrease on the densities denoting the impact of the loss of hydrogen bonding capability on the bulk organization and compactness. Nonetheless, the substitution of both hydroxyl hydrogens, by  $\text{CH}_3$  groups, does not lead to a decrease proportionally larger to that observed for only one substitution.

Molar volumes,  $V_m$ , were calculated and are reported in Tables 3 and 4 and depicted in Figure 6 as a function of temperature. As shown, the glycols and glymes present a small temperature dependency and increase in the order  $\text{EG} < \text{DEG} \approx \text{EGEE} < \text{DEGME} < \text{TriEG} \approx \text{DEGEE} < \text{DEGDME} < \text{TeEG} < \text{DEGDDEE} \approx \text{TriEGDME} < \text{TeEGDME}$ .

For the diols (EG, DEG, TriEG, and TeEG) and glycol dimethyl ethers (DEGDME, TriEGDME, and TeEGDME) the

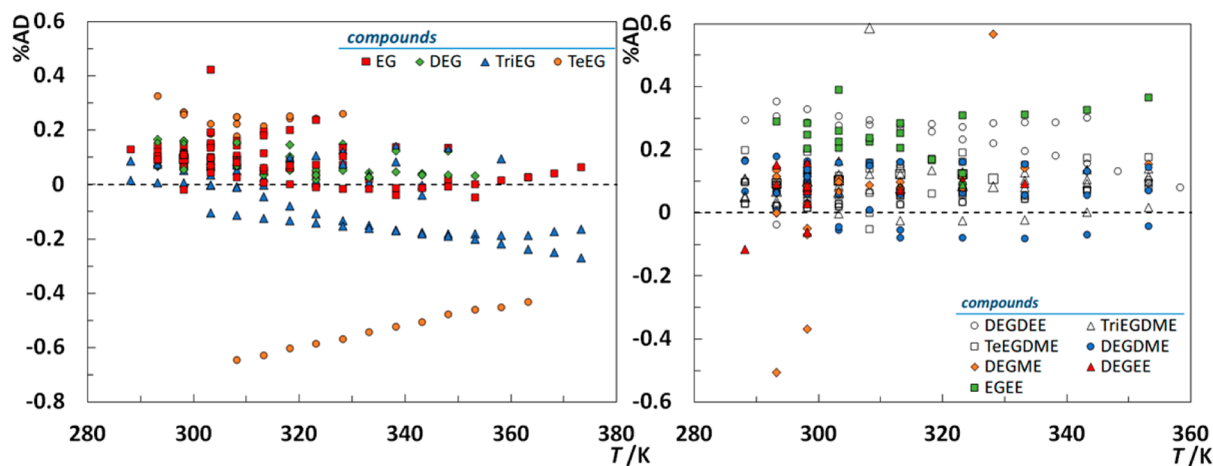


Figure 3. Density relative deviations between the literature<sup>4–55</sup> and the experimental density data, obtained through the SVM3000 densimeter, as a function of temperature.





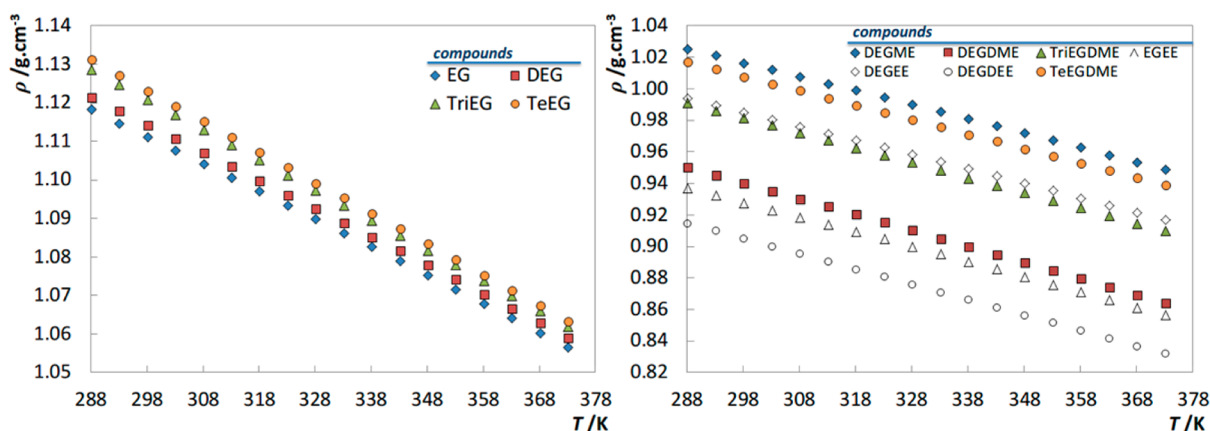


Figure 4. Density as a function of temperature for the studied glycols.

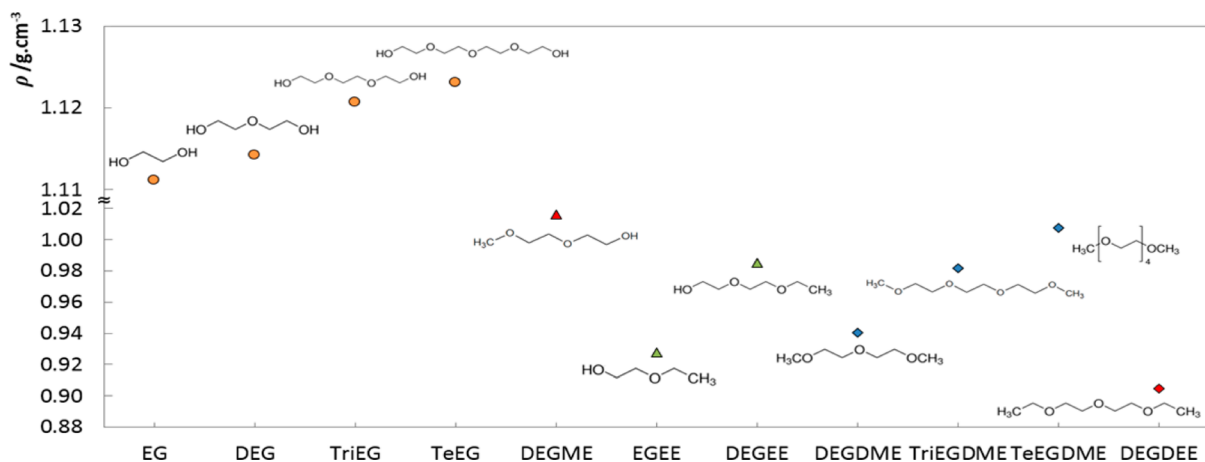


Figure 5. Density of the studied glycols and glymes at 298 K.

Table 3. Molar Volumes,  $V_m$ , Viscosity,  $\eta$ , and Refractive Index,  $n_D$ , as a Function of Temperature and at Atmospheric Pressure,  $(0.10 \pm 0.01)$  MPa, for the Glycols<sup>a</sup>

$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$
K	$\text{cm}^3 \cdot \text{mol}^{-1}$	mPa·s		K	$\text{cm}^3 \cdot \text{mol}^{-1}$	mPa·s		K	$\text{cm}^3 \cdot \text{mol}^{-1}$	mPa·s		K	$\text{cm}^3 \cdot \text{mol}^{-1}$	mPa·s
EG														
283.15			1.43461	308.15	56.22	11.577		333.15	57.14	5.197	1.41955	358.15	58.13	2.835
288.15	55.51	26.801	1.43312	313.15	56.40	9.627	1.42562	338.15	57.33	4.555	1.41655	363.15	58.34	2.552
293.15	55.69	21.197	1.43167	318.15	56.58	8.182		343.15	57.53	4.010	1.41655	368.15	58.55	2.306
298.15	55.86	17.134	1.43000	323.15	56.77	6.980	1.42260	348.15	57.73	3.552	1.41351	373.15	58.76	2.102
303.15	56.04	13.998	1.42855	328.15	56.96	6.007		353.15	57.93	3.161				
DEG														
283.15			1.45019	308.15	95.86	18.180		333.15	97.47	7.570	1.43424	358.15	99.15	3.944
288.15	94.63	47.142	1.44860	313.15	96.18	14.852	1.44068	338.15	97.79	6.561	1.43105	363.15	99.49	3.527
293.15	94.94	36.020	1.44702	318.15	96.50	12.3993		343.15	98.12	5.718	1.43105	368.15	99.85	3.169
298.15	95.24	28.233	1.44540	323.15	96.82	10.419	1.43745	348.15	98.46	5.019	1.42786	373.15	100.21	2.871
303.15	95.55	22.478	1.44379	328.15	97.14	8.849		353.15	98.80	4.429				
TriEG														
283.15			1.45944	308.15	134.94	23.599		333.15	137.34	9.528	1.44293	358.15	139.85	4.874
288.15	133.06	63.57	1.45774	313.15	135.41	19.158	1.44955	338.15	137.83	8.221	1.43956	363.15	140.37	4.346
293.15	133.53	48.05	1.45610	318.15	135.89	15.863		343.15	138.33	7.137	1.43956	368.15	140.89	3.896
298.15	134.00	37.27	1.45447	323.15	136.37	13.252	1.44626	348.15	138.83	6.243	1.43622	373.15	141.42	3.515
303.15	134.46	29.41	1.45283	328.15	136.85	11.192		353.15	139.33	5.487				
TeEG														
283.15			1.46333	308.15	174.18	28.464		333.15	177.33	11.367	1.44633	358.15	180.63	5.762
288.15	171.70	79.710	1.46169	313.15	174.81	23.158	1.45319	338.15	177.98	9.784	1.43947	363.15	181.30	5.127
293.15	172.33	59.678	1.45999	318.15	175.42	19.080		343.15	178.64	8.480	1.44288	368.15	181.98	4.585
298.15	172.94	45.862	1.45827	323.15	176.06	15.887	1.44976	348.15	179.28	7.405	1.43947	373.15	182.68	4.116
303.15	173.56	35.923	1.45661	328.15	176.70	13.380		353.15	179.94	6.491				

<sup>a</sup>Standard uncertainty in the temperature,  $u(T)$ , is 0.02 K and 0.01 K for the SVM300 and DSA 5000 M measurements, respectively, in the viscosity,  $u(\eta)$ , is 2 % and in the refractive index,  $u(n_D)$ , is  $2 \cdot 10^{-4}$ ; the combined standard uncertainty in the molar volume,  $u_c(V_m)$ , is  $1.10 \cdot 10^{-2} \text{ cm}^3 \cdot \text{mol}^{-1}$ .

**Table 4.** Molar Volumes,  $V_m$ , Viscosity,  $\eta$ , and Refractive Index,  $n_D$ , as a Function of Temperature and at Atmospheric Pressure, (0.10 ± 0.01) MPa, for the Glymes<sup>a</sup>

$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$	$n_D$	$T$	$V_m$	$\eta$
K	cm <sup>3</sup> ·mol <sup>-1</sup>	mPa·s		K	cm <sup>3</sup> ·mol <sup>-1</sup>	mPa·s		K	cm <sup>3</sup> ·mol <sup>-1</sup>	mPa·s		K	cm <sup>3</sup> ·mol <sup>-1</sup>	mPa·s
DEGDME														
283.15			1.41249	308.15	144.24	0.879		333.15	148.25	0.633	1.39050	358.15	152.57	0.486
288.15	141.20	1.161	1.41020	313.15	145.02	0.825	1.39921	338.15	149.09	0.599		363.15	153.48	0.464
293.15	141.95	1.081	1.40802	318.15	145.81	0.767		343.15	149.94	0.566	1.38623	368.15	154.40	0.444
298.15	142.70	1.011	1.40579	323.15	146.60	0.718	1.39490	348.15	150.80	0.537		373.15	155.33	0.441
303.15	143.47	0.942	1.40359	328.15	147.42	0.674		353.15	151.67	0.505	1.38178			
DEGDDE														
283.15			1.41658	308.15	181.26	1.067		333.15	186.32	0.744	1.39382	358.15	191.69	0.556
288.15	177.42	1.483	1.41428	313.15	182.26	0.9874	1.40296	338.15	187.35	0.699		363.15	192.81	0.526
293.15	178.35	1.353	1.41202	318.15	183.25	0.916		343.15	188.42	0.658	1.38929	368.15	193.94	0.498
298.15	179.32	1.254	1.40978	323.15	184.27	0.852	1.39838	348.15	189.50	0.621		373.15	195.08	0.470
303.15	180.30	1.156	1.40749	328.15	185.28	0.795		353.15	190.59	0.587	1.38474			
TriEGDME														
283.15			1.42712	308.15	183.38	1.623		333.15	187.99	1.064	1.40579	358.15	192.85	0.801
288.15	179.89	2.414	1.42498	313.15	184.27	1.439	1.41434	338.15	188.94	1.026		363.15	193.85	0.756
293.15	180.76	2.162	1.42284	318.15	185.19	1.373		343.15	189.89	0.962	1.40153	368.15	194.89	0.715
298.15	181.59	1.956	1.42071	323.15	186.10	1.270	1.41005	348.15	190.87	0.903		373.15	195.92	0.650
303.15	182.48	1.777	1.41862	328.15	187.04	1.179		353.15	191.85	0.814	1.39723			
TeEGDME														
283.15			1.43599	308.15	222.55	2.660		333.15	227.84	1.677	1.41578	358.15	233.32	1.188
288.15	218.56	4.337	1.43414	313.15	223.60	2.389	1.42373	338.15	228.92	1.566		363.15	234.45	1.115
293.15	219.58	3.846	1.43196	318.15	224.64	2.183		343.15	229.98	1.456	1.41174	368.15	235.59	1.047
298.15	220.60	3.335	1.42992	323.15	225.69	1.995	1.41973	348.15	231.08	1.358		373.15	236.75	0.978
303.15	221.59	2.967	1.42777	328.15	226.77	1.832		353.15	232.19	1.250	1.40773			
EGEE														
283.15			1.41490	308.15	98.12	1.503		333.15	100.68	0.962	1.39271	358.15	103.47	0.666
288.15	96.20	2.314	1.41250	313.15	98.61	1.365	1.40180	338.15	101.21	0.895		363.15	104.05	0.623
293.15	96.66	2.066	1.41024	318.15	99.11	1.247		343.15	101.77	0.829	1.38849	368.15	104.64	0.584
298.15	97.14	1.845	1.40803	323.15	99.62	1.143	1.39691	348.15	102.33	0.770		373.15	105.24	0.542
303.15	97.63	1.661	1.40613	328.15	100.14	1.051		353.15	102.90	0.725	1.38380			
DEGME														
283.15			1.42914	308.15	119.27	2.666		333.15	121.94	1.606	1.41010	358.15	124.83	1.094
288.15	117.19	4.514	1.42723	313.15	119.79	2.373	1.41733	338.15	122.48	1.489		363.15	125.43	1.019
293.15	117.68	3.963	1.42498	318.15	120.29	2.148		343.15	123.04	1.372	1.40635	368.15	126.06	0.950
298.15	118.22	3.403	1.42390	323.15	120.84	1.946	1.41368	348.15	123.64	1.268		373.15	126.67	0.877
303.15	118.74	3.000	1.42174	328.15	121.39	1.772		353.15	124.22	1.155	1.40241			
DEGEE														
283.15			1.43110	308.15	137.47	2.933		333.15	140.71	1.718	1.41082	358.15	144.16	1.153
288.15	134.99	5.157	1.42916	313.15	138.11	2.592	1.41887	338.15	141.38	1.583		363.15	144.88	1.072
293.15	135.61	4.466	1.42711	318.15	138.75	2.331		343.15	142.07	1.456	1.40683	368.15	145.62	0.997
298.15	136.23	3.811	1.42501	323.15	139.40	2.099	1.41483	348.15	142.75	1.343		373.15	146.35	0.921
303.15	136.85	3.328	1.42290	328.15	140.04	1.901		353.15	143.45	1.225	1.40276			

<sup>a</sup>Standard uncertainties in temperature,  $u(T)$ , is 0.02 K and 0.01 K for the SVM300 and DSA 5000 M measurements, respectively, in density,  $u(\eta)$ , is 2 % and in refractive index,  $u(n_D)$ , is  $2 \cdot 10^{-4}$ ; the combined standard uncertainty is  $u_c(V_m) = 1.10^{-2} \text{ cm}^3 \cdot \text{mol}^{-1}$ .

compounds' molar volume increases with the molar volume of the ethoxy group increase in the order of  $38.8 \text{ cm}^3 \cdot \text{mol}^{-1}$ . Furthermore, the substitution of a hydrogen on one of the diols' hydroxyl groups by an ethyl group—for the case of EG and EGEE as well as DEG and DEGEE—present an increase of  $40.5 \text{ cm}^3 \cdot \text{mol}^{-1}$  that corresponds to the sum of the  $\text{CH}_3$  and  $\text{CH}_2$  groups volumes, as observed for the case of DEG and DEGME ( $\Delta V_m = 22.6 \text{ cm}^3 \cdot \text{mol}^{-1}$ ) and DEGME and DEGEE ( $\Delta V_m = 17.8 \text{ cm}^3 \cdot \text{mol}^{-1}$ ). These observations confirm the molar volumes characteristic additivity.

**Thermal Expansion Coefficient.** The isobaric thermal expansion coefficients,  $\alpha_p$ , of the studied compounds, which reflect the volumetric changes with temperature, were calculated with the following equation

$$\alpha_p = \frac{1}{V} \left( \frac{\partial V}{\partial T} \right)_p = -\frac{1}{\rho} \left( \frac{\partial \rho}{\partial T} \right)_p \quad (2)$$

where  $\rho$  is the density,  $T$  is the temperature, and  $p$  is a fixed pressure. In the temperature range covered, the density of all the compounds studied can be described using a quartic equation of the type

$$\rho / \text{kg} \cdot \text{m}^{-3} = A + B \cdot T + C \cdot T^2 + D \cdot T^3 + E \cdot T^4 \quad (3)$$

The appropriate number coefficients (4 or 5) was optimized using the  $F$ -test statistical analysis and are reported in Table S7 in Supporting Information together with the standard deviations.

The calculated thermal expansion coefficients for the compounds studied are reported in Table S8 in the Supporting

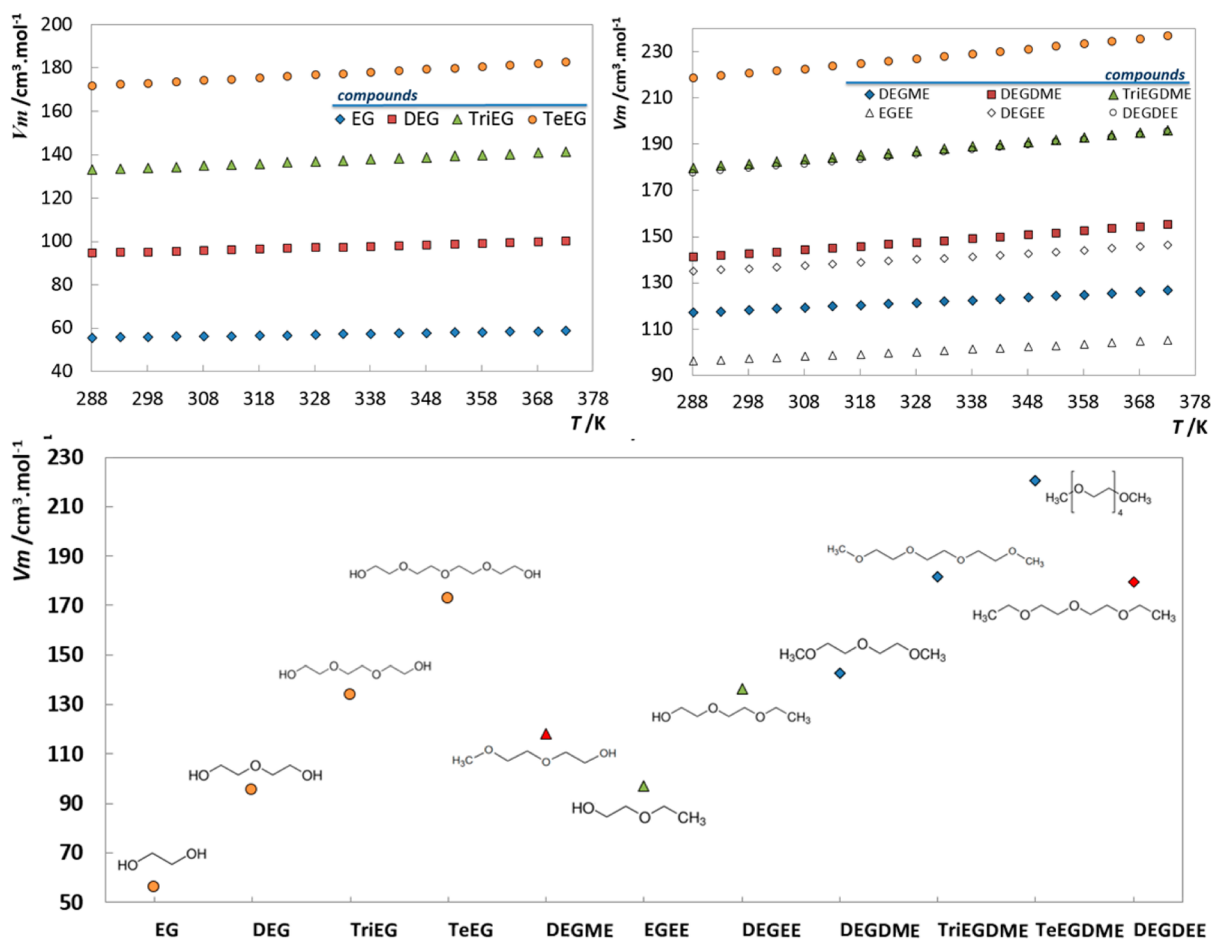


Figure 6. Molar volume as a function of temperature (top) and at 298.15 K (bottom) for the studied compounds.

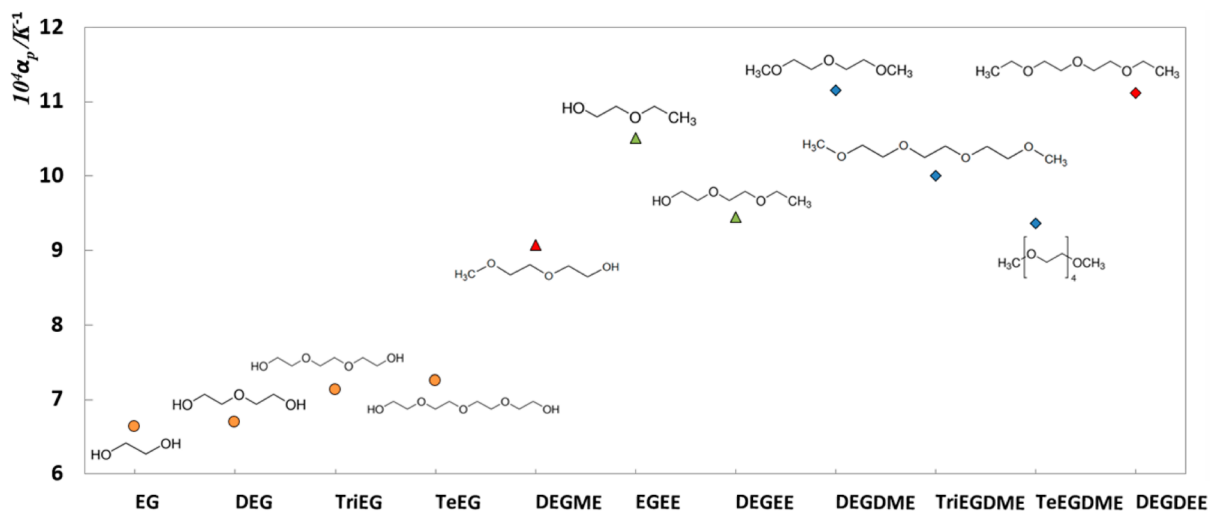


Figure 7. Thermal expansion coefficients of the studied compounds at 298 K.

Information and depicted in Figure 7, where it can be seen that the change in the density of glycols and glymes is small as the temperature increases, which denotes the weak thermal expansion of these compounds. The results also indicate that for the diols, the thermal expansion increases with the number of ethoxy groups, whereas for the glymes, the thermal expansion decreases with the number of ethoxy groups. Moreover, the substitution of the hydrogens in the diols terminal hydroxyl groups leads to an increase on the thermal expansion. In fact, the thermal expansion

increases with the number of hydrogen substitutions, as depicted in Figure 7. This behavior denotes structures more rigid and optimally packed for the compounds with one and then two terminal OH groups. Furthermore, one can also observe, with no surprise, that this effect is more significant for the shorter glycols.

**3.2. Viscosity.** The viscosity measurements were carried out at atmospheric pressure and in the (288.15 to 373.15) K using the SVM3000 Anton Paar rotational Stabinger viscometer–densimeter, as reported in Tables 3 and 4 and depicted in Figure 8.



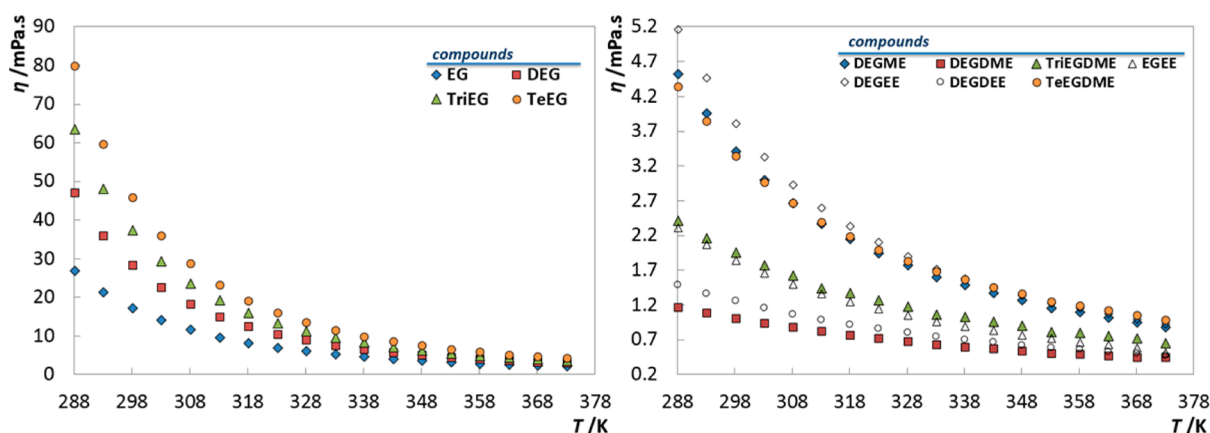


Figure 8. Viscosity as a function of temperature for the studied compounds.

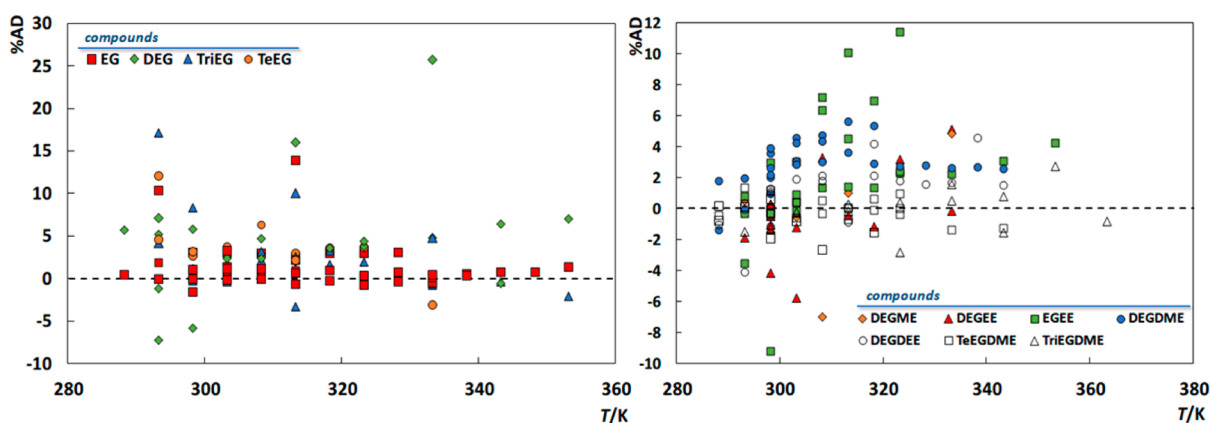


Figure 9. Viscosity relative deviations between the literature<sup>7,9–12,16,18–21,28,32,34,35,38,40,42,43,55–71</sup> and the experimental viscosity data.

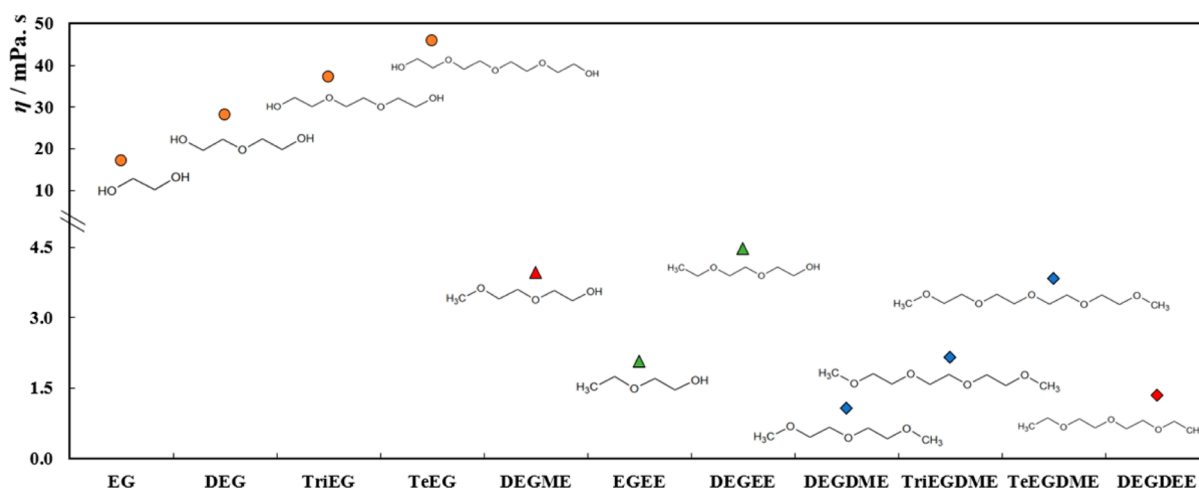


Figure 10. Viscosity of the studied compounds at 298 K.

For the viscosity, fewer data is available in the literature<sup>7,9–12,16,18–21,28,32,34,35,38,40,42,43,55–71</sup> compared with that for density (a summary of all the literature viscosity data used in this work is provided in Table S5 in the Supporting Information). As depicted in Figure 9, viscosity data for the same compound presents some deviations among the authors, with our data presenting absolute average deviations of 7.4 % toward the literature values. Moreover, the viscosity data determined here are coherent with almost all the data available, but larger deviations are observed toward the data of Kinart et al.<sup>72</sup> for

TriEG and DEG, Kumagai et al.<sup>56</sup> for DEG, Islam et al.<sup>73</sup> and Miller et al.<sup>74</sup> for EGEE, and Pal et al.<sup>67</sup> for DEGME. By other hand, small deviations toward Corradini et al.<sup>12</sup> (−0.02 %) or Ge et al.<sup>60</sup> (0.67 %) for EG and Mesquita et al.<sup>23</sup> (−0.34 %) for DEG and TriEG can be found.

As depicted in Figure 10, the loss of the hydroxyl groups, through the substitution of the hydroxyl groups' hydrogen by a methyl, leads to a significant decrease in the viscosity. Furthermore, the substitution by an ethyl, instead of methyl group, leads to a slightly lower decrease in the viscosity than that

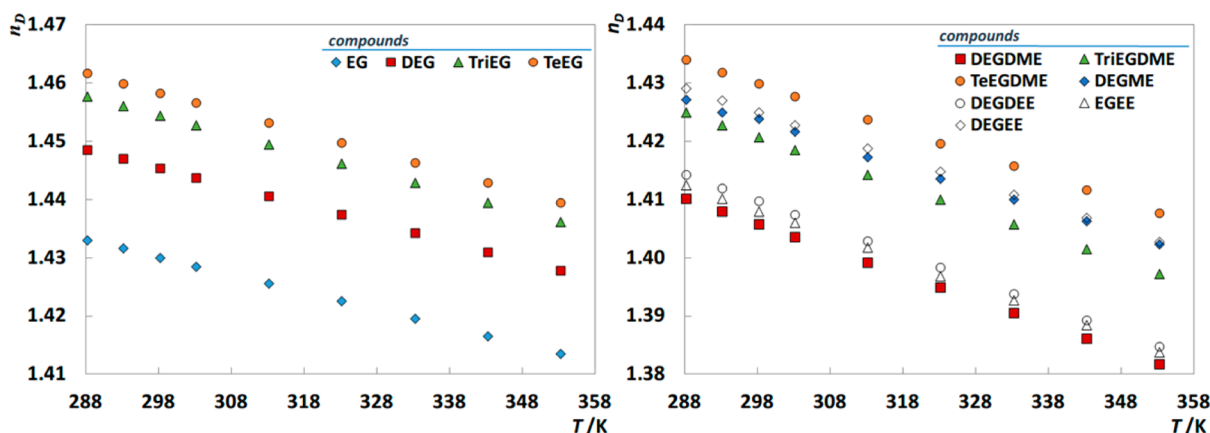


Figure 11. Refractive index as a function of temperature for the studied compounds.

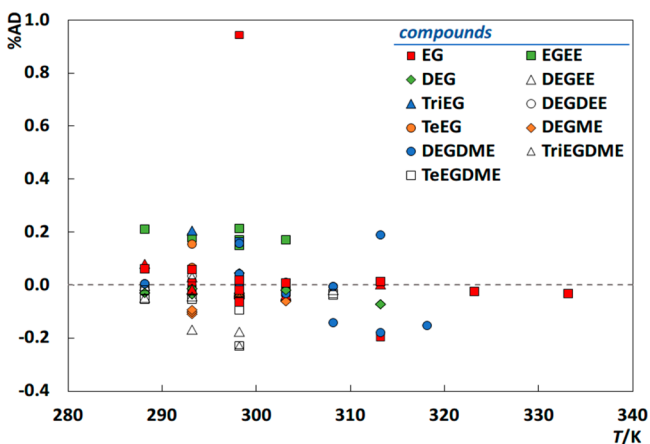


Figure 12. Refractive index relative deviations, between experimental and literature<sup>7,8,16,17,19,28,30,31,33,34,41,48,52,53,65,71,74–87</sup> data, as a function of temperature for the studied compounds.

observed for the methyl substitution, as seen for the pairs DEGME–DEGEE and DEGDME–DEGDDE. Moreover, the increment of ethoxy groups in the compound leads to an increase of the viscosity.

**3.3. Refractive Index.** The refractive index is an optical property of materials and specifies the dielectric response of a fluid to an electrical field induced by electromagnetic waves. The

experimental refractive indices of the investigated compounds are shown in Figure 11 and reported in Tables 3 and 4. The refractive indices were measured in the (283.15 to 353.15) K temperature range, at atmospheric pressure, upward and downward on the temperature with no hysteresis effects observed.

The relative deviations between the data measure in this work and that reported in literature<sup>7,8,16,17,19,28,30,31,33,34,41,48,52,53,65,71,74–87</sup> are depicted in Figure 12 (a summary of all the literature refractive index data used in this work is provided in Table S5 in the Supporting Information). As shown, small and no systematic deviations (%AAD = 0.10 %) were identified.

As depicted in Figure 13, the increase of ethoxy groups in the compound leads to an increase of the refractive index. Nonetheless, contrary to that observed for the molar volume behavior this increase is not constant but seems to present an asymptotic tendency toward a constant value. This behavior seems even more evident if the data for the diols is plotted with the refractive indices of polyethylene glycols<sup>88</sup> with different molecular weights, as depicted in Figure S1 in Supporting Information. On the other hand, the loss of the hydroxy groups, through the substitution of the hydroxyl groups' hydrogen by a methyl, leads to a decrease of the refractive index. Furthermore, similar to what is observed for the molar volumes' behavior, the substitution by an ethyl, instead of a methyl group, leads to a slightly lower decrease, as seen for the pairs DEGME–DEGEE and DEGDME–DEGDDE, for which an increment of a CH<sub>2</sub>

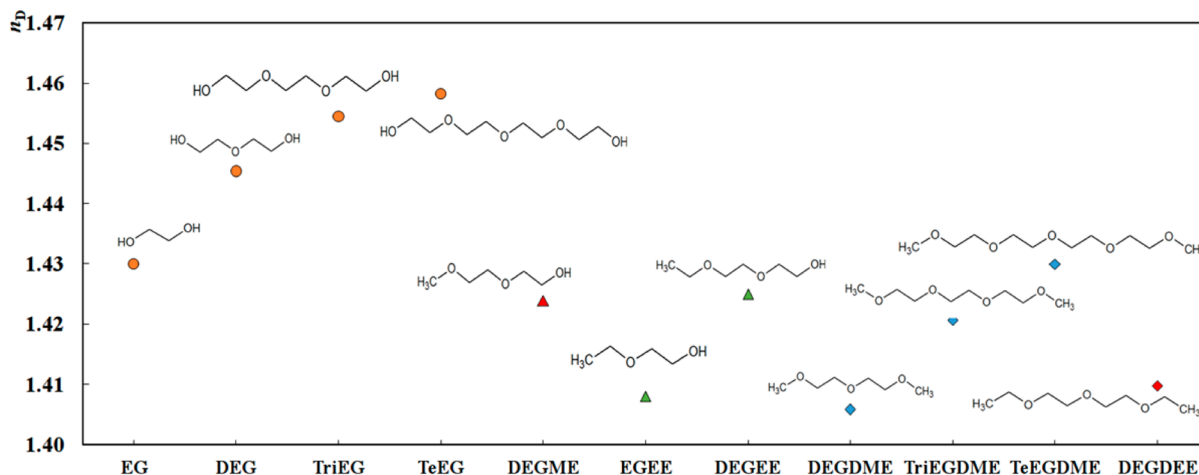


Figure 13. Refractive index of the studied compounds at 298.15 K.

group, in the methyl terminal groups, leads to a small increase of the refractive index.

**3.4. Sound Velocity.** The sound velocity measurements were carried out at atmospheric pressure and in the (283.15 to 343.15) K temperature range using the DSA 5000 M vibrating tube densimeter and ultrasound speed meter from Anton Paar, as reported in Table 5 and depicted in Figure 14. The same procedure adopted for the EG, DEG, DEGME, EGEE, DEGREE, and DEGDEE density evaluation, of determining the pure density through the extrapolation of aqueous mixture densities down to water mole fractions of zero, was also followed for the sound velocities. The aqueous mixtures' compositions and sound speed are reported in Table S2 in the Supporting Information. The standard uncertainties of the extrapolated sound speeds, of the pure compounds, based on the quality of the fits and calculated with the error propagation method are reported in Table S4 in the Supporting Information.

Experimental sound velocities for the compounds studied are much scarcer than those available for the other properties evaluated. Furthermore, small deviations (%AAD of 0.3 %) are found between the experimental data determined here and those reported in the literature,<sup>7–9,18,26,31,55,82,89–103</sup> as depicted in Figure 15 (a summary of all the literature sound speed data used in this work is provided in Table S5 in the Supporting Information). The highest deviations observed are against data reported by George et al.<sup>8</sup> for EG, DEG, and TriEG and for the temperatures above 328 K and seem related to the inconsistent behavior observed within the authors' data.<sup>8</sup>

As depicted in Figures 14 and 16, the increase of the ethoxy groups leads to an increase of the sound speed for the glymes (DEGDME, TriEGDME, and TeEGDME), whereas for the glycols (EG, DEG, TriEG, and TeEG), the behavior observed does not follow a monotonic trend, with DEG presenting smaller sound speeds than all the other glycols. Despite being exceptional, this behavior has been reported by others.<sup>8,18,26,93,94</sup> Nonetheless, if DEG is removed from the series and PEG400 added, the glycols' sound speed seems to decrease with the increase of the molecular weight, as depicted in Figure S2 in Supporting Information.

On the other hand, the loss of the hydroxyl groups, and therefore the loss of the capability to establish hydrogen bonds, leads to a significant decrease of the speed of sound. Similar to that observed for density and viscosity, this decrease is more relevant for the case of the loss of one hydroxyl group, through the substitution of the hydroxyl group's hydrogen by a methyl or ethyl group, than for the case of the loss of both hydroxyl groups.

**Isentropic Compressibilities.** Because there was no evidence of ultrasonic absorption at the working frequency (3 MHz) of the sound speed analyzer,  $u$  can be considered a pure thermodynamic property.<sup>104</sup> Therefore, isentropic compressibilities,  $k_s$ , reported in Table 5 and depicted in Figure 17, can be estimated by combining  $u$  and  $\rho$  through the Newton–Laplace equation

$$k_s = \frac{1}{\rho u^2} \quad (4)$$

As depicted in Figure 17, the glycols (EG, DEG, TriEG, and TeEG) present similar isentropic compressibilities, whereas the glymes (DEGDME, TriEGDME, and TeEGDME) present a decrease on the isentropic compressibilities as the number of ethoxy groups increase. Moreover, the loss of the hydroxyl groups, due to the substitution of the hydroxyl group hydrogen

Table 5. Sound Speed,  $u$ , and Isentropic Compressibility,  $k_s$ , as a Function of Temperature and at Atmospheric Pressure,  $(0.10 \pm 0.01)$  MPa, for the Compounds Studied<sup>a</sup>

T	EG		DEG		TriEG		TeEG		DEGME		EGEE		DEGREE		DEGDME		TriEGDME		TeEGDME		DEGDEE		
	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	$u$	$10^{10} k_s$	
283.15	1689.9	3.126	1615.9	3.409	1658.7	3.213	1647.6	3.254	1467.7	4.513	1360.6	5.740	1428.9	4.913	1340.7	5.834	1401.4	5.122	1439.7	4.727	1303.4	6.419	6.419
288.15	1678.3	3.179	1603.6	3.473	1642.8	3.287	1630.6	3.334	1450.2	4.642	1342.0	5.928	1410.5	5.064	1320.0	6.050	1381.4	5.297	1420.3	4.879	1283.2	6.658	6.658
293.15	1666.5	3.234	1591.4	3.538	1627.2	3.362	1614.1	3.414	1432.7	4.777	1323.9	6.121	1392.6	5.218	1299.9	6.271	1361.6	5.478	1401.0	5.037	1263.1	6.908	6.908
298.15	1654.7	3.291	1579.2	3.604	1611.5	3.439	1597.9	3.496	1415.4	4.916	1305.7	6.323	1374.6	5.380	1280.0	6.502	1342.1	5.665	1382.0	5.201	1243.3	7.168	7.168
303.15	1642.9	3.349	1566.9	3.672	1596.2	3.518	1582.0	3.580	1398.1	5.060	1286.7	6.543	1356.8	5.547	1260.1	6.744	1322.8	5.861	1363.1	5.370	1223.4	7.442	7.442
308.15	1631.1	3.408	1554.9	3.741	1581.1	3.598	1566.3	3.665	1380.9	5.210	1268.6	6.765	1339.0	5.721	1240.3	6.998	1303.6	6.064	1344.3	5.547	1203.9	7.727	7.727
313.15	1619.3	3.469	1542.8	3.813	1566.0	3.680	1550.6	3.753	1363.9	5.365	1250.2	7.001	1321.7	5.899	1220.6	7.265	1284.4	6.278	1325.6	5.731	1184.1	8.031	8.031
323.15	1595.5	3.597	1518.8	3.960	1536.3	3.851	1519.8	3.935	1329.7	5.694	1213.8	7.504	1286.9	6.281	1181.7	7.837	1246.9	6.727	1289.1	6.117	1145.1	8.682	8.682
333.15	1571.5	3.733	1494.5	4.117	1506.8	4.032	1489.4	4.127	1296.1	6.049	1178.5	8.045	1252.6	6.693	1143.4	8.465	1209.9	7.217	1253.4	6.532	1107.9	9.378	9.378
343.15	1547.4	3.876	1470.1	4.284	1477.5	4.224	1459.4	4.330	1262.7	6.432	1143.7	8.635	1218.9	7.137	1105.8	9.154	1174.0	7.744	1218.5	6.978	1071.6	10.138	10.138

<sup>a</sup>Standard uncertainty in the temperature,  $u(T)$ , is 0.02 K and 0.01 K for the SVM300 and DSA 5000 M measurements, respectively, in sound speed,  $u_c(u)$ , is  $1 \text{ m}\cdot\text{s}^{-1}$ ; the combined standard uncertainty  $u_c(k_s) = 1.10^{-13} \text{ kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ .

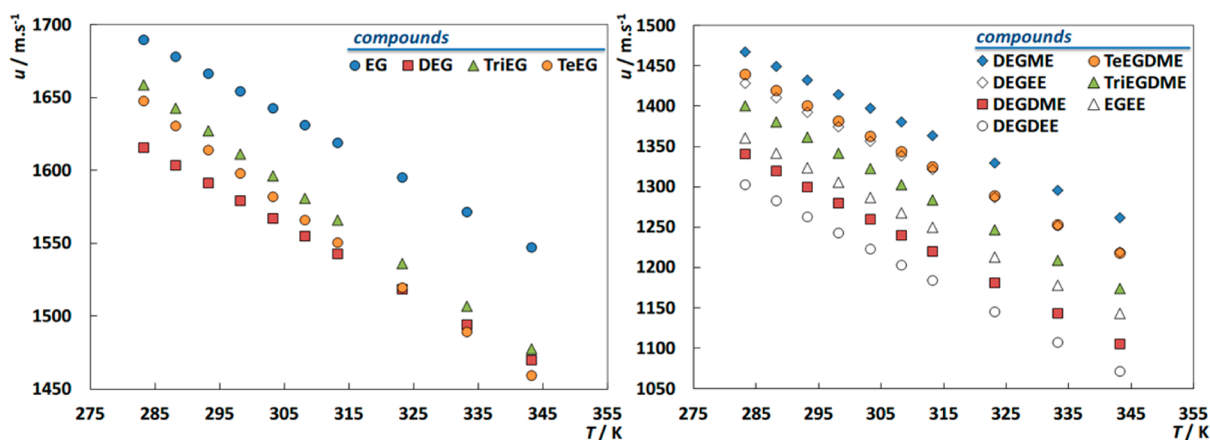


Figure 14. Sound speed as a function of temperature for the studied compounds.

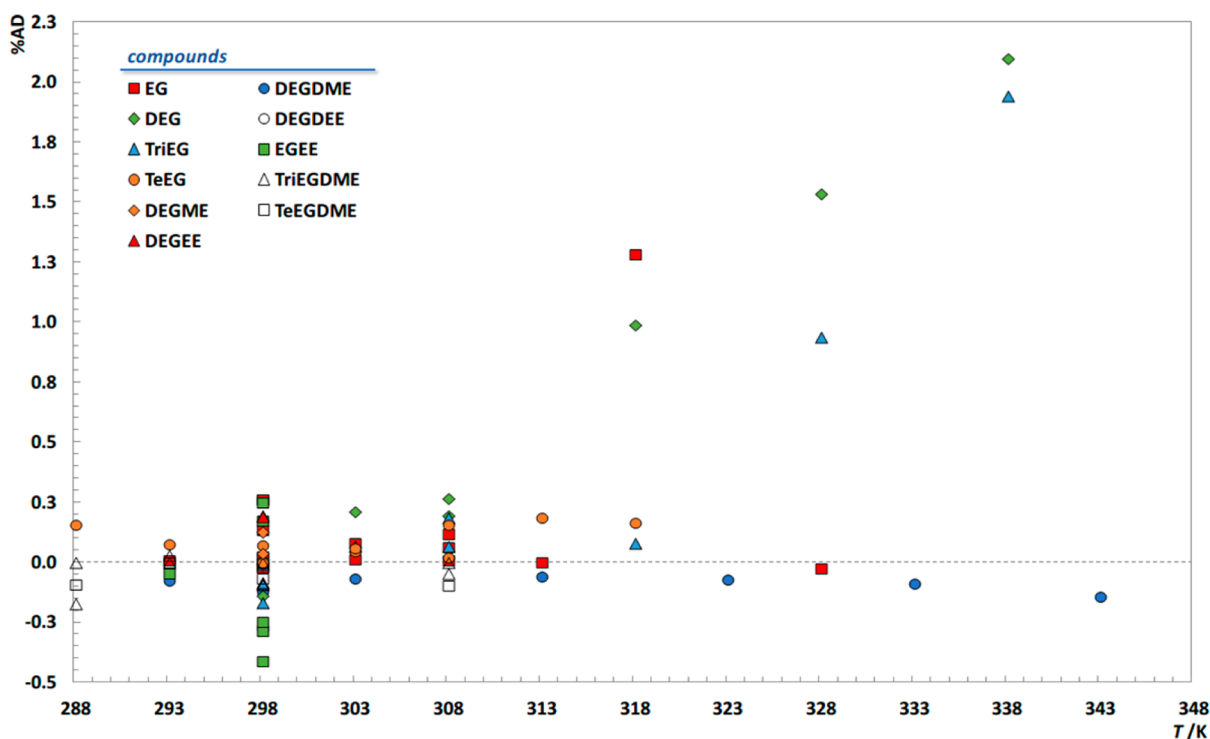


Figure 15. Sound speed percentage average deviations between experimental and literature data.<sup>7–9,18,26,31,55,82,89–103</sup>

by a methyl or ethyl group, leads to an increase of the  $k_s$ . The behaviors observed are in agreement with the general pattern evidencing higher speeds of sound for compounds whose structures present higher rigidity.

#### 4. CONCLUSIONS

Experimental data for density, viscosity, refractive index, and sound speed of 11 glycols and glymes were measured in the temperature range between (283.15 and 373.15) K and at atmospheric pressure. Additionally, derivative properties, such as isobaric thermal expansion coefficient and isentropic compressibilities, were also estimated and discussed. For the density determination, two different approaches were adopted, whereas for the SVM3000 densimeter, all the compounds went through a purification process, for the DSA 5000 M measurements the densities of EG, DEG, DEGME, EGEE, DEGEE, and DEGDEE were determined by extrapolating aqueous mixtures' densities to the pure compound density.

The compounds evaluated were selected to evaluate the impact of molecular structure changes on their thermophysical properties. The increase of ethoxy groups has shown to lead to an increase of the density, molar volume, viscosity, and refractive index. Furthermore, the loss of the hydroxyl groups, through the substitution of the hydroxyl groups' hydrogen by a methyl group leads to a significant decrease on the density, viscosity, and sound speed. However, the substitution by an ethyl group, instead of methyl, leads to a slightly lower decrease than that observed for the methyl substitution. Moreover, the decrease of the density, viscosity, and speed of sound, observed due to the substitution of both hydrogens on the diols hydroxyl groups by either an ethyl or methyl group, is not proportionally larger to that observed for only one substitution. This behavior although denoting the impact of the loss of hydrogen bonding capability on the bulk organization and compactness, evidence that the hydroxyl groups do not have the same influence.

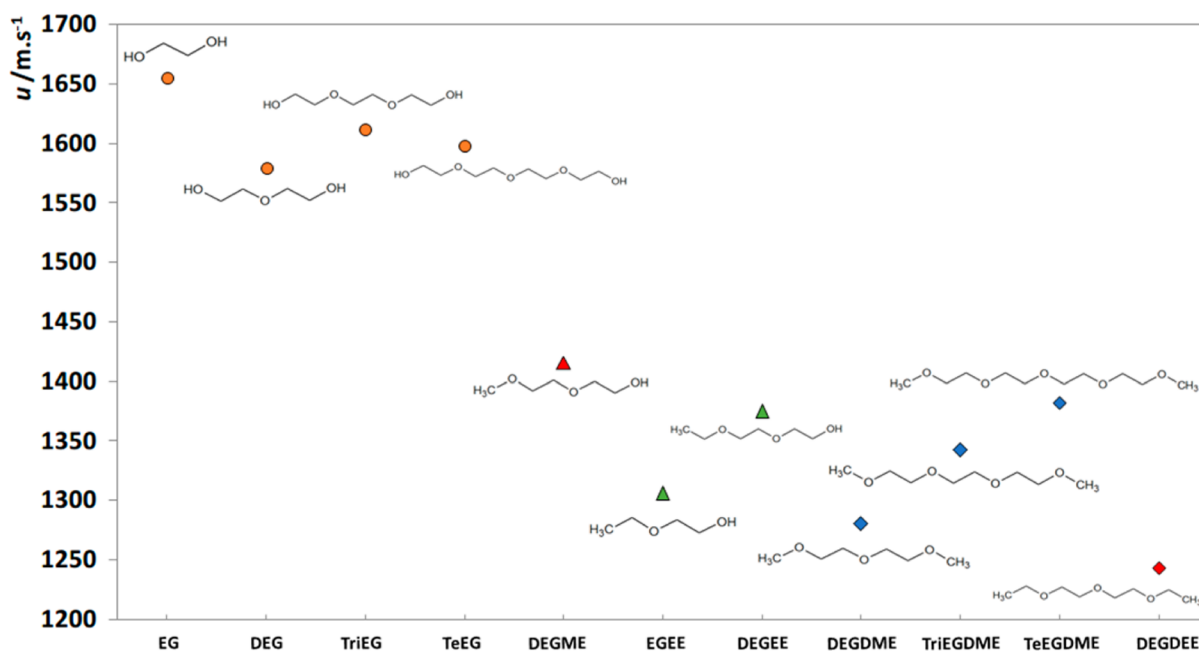


Figure 16. Sound speed of the studied compounds at 298.15 K.

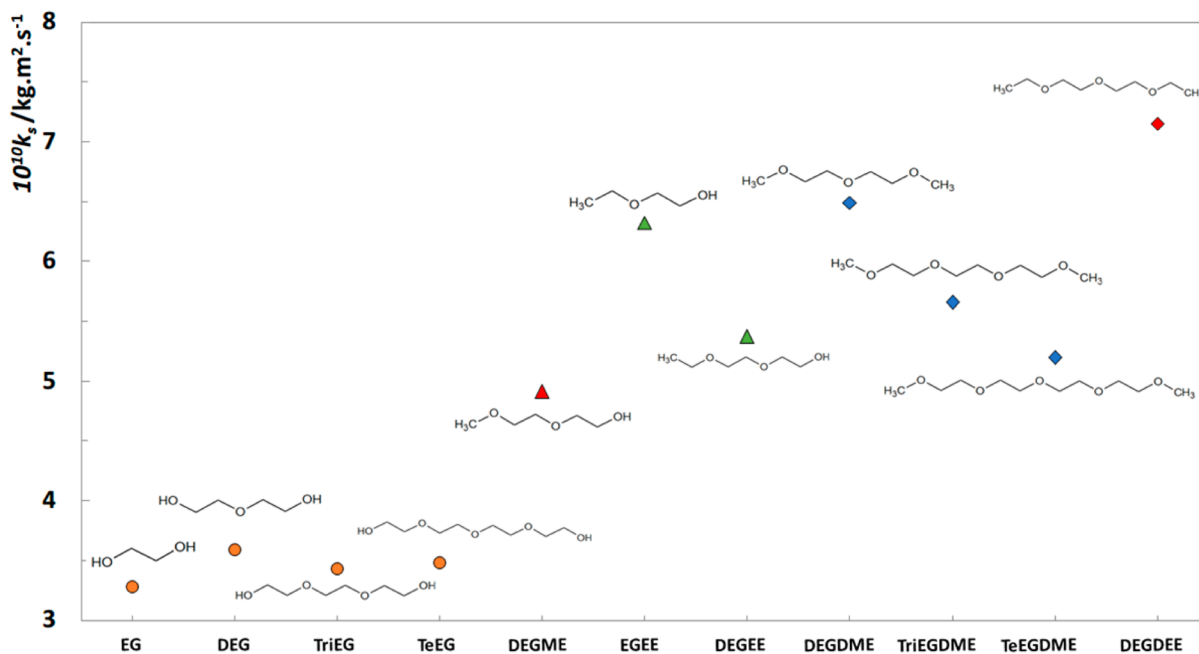


Figure 17. Isentropic compressibilities of the studied compounds at 298.15 K.

On the other hand, the small change in the glycols' and glymes' density as the temperature increases denotes the weak thermal expansion of these compounds. The results also indicate that for the diols, the thermal expansion increases with the number of ethoxy groups, whereas for the glymes, the thermal expansion decreases. Moreover, the substitution of the hydrogens in the diols' terminal hydroxyl groups leads to an increase on the thermal expansion. In fact, the thermal expansion increases with the number of hydrogen substitutions denoting structures more rigid and optimally packed for the compounds with one and then two terminal OH groups. However, similar isentropic compressibilities were observed for the glycols, whereas for the glymes, a decrease on the isentropic compressibilities as the number of ethoxy groups increase is observed. Moreover,

the loss of the hydroxyl groups, due to the substitution of the hydroxyl group hydrogen by a methyl or ethyl group, leads to an increase of the isentropic compressibilities. These behaviors are in agreement with the general pattern evidencing higher speeds of sound for compounds whose structures present higher rigidity.

## ■ ASSOCIATED CONTENT

### § Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.jced.5b00662.

Refractive index plots; sound speed plots; experimental density data for the glycols and glymes aqueous solutions;



experimental sound speed data for the glycols and glymes aqueous solutions; least squares fitting coefficients of the compound's aqueous solutions densities and sound speed; summary of the literature data available for the density, viscosity and sound speed of the studied compounds; and empirical correlations for the viscosity, refractive index and sound speed experimental data. (PDF)

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### Notes

The authors declare no competing financial interest.

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