SPECIFIC VOLUME AND VISCOSITY OF ETHANOL-WATER MIXTURES UNDER HIGH PRESSURE

BY YOSHIYUKI TANAKA, TAKESHI YAMAMOTO, YOSHIHISA SATOMI, HIRONOBU KUBOTA AND TADASHI MAKITA

The specific volume and the viscosity of ethanol-water mixtures at 25°C (298.15K) and 50°C (323.15K) have been measured under pressures up to 3200 and 800 bar (10^8Pa), respectively. The measurements were performed by a modified Adams piezometer and a falling-cylinder viscometer. The maximum uncertainties are estimated to be 0.05% for the specific volume and 2% for the viscosity.

The specific volume of the pure components and their mixtures is found to decrease monotonously with increasing pressure. The results obtained are compared with several sets of literature values. The numerical data at each temperature and composition are correlated satisfactorily as a function of pressure by both polynomial and the Tait equations. It is also found that a definite minimum appears on the isothermal compressibility versus composition isobars, arising from the complex interactions between hydrogen-bonded water and alcohol molecules.

The viscosity of pure ethanol and mixtures is found to increase almost linearly with increasing pressure, whereas that of water is nearly independent of pressure in these experimental conditions. The viscosity isotherms can be formulated by a quadratic equation of pressure within the experimental error. As for the composition dependence of the viscosity, a distinct maximum appears near 0.3 mole fraction of ethanol on all isobars at both experimental temperatures.

Introduction

The physico-chemical properties of aqueous solutions of alcohols are of interest in many fields of science. The solutions often show some anomalies in various physical properties such as partial molar volume, compressibility, velocity of sound, sound absorption, viscosity and so on, which have been yet inadequately understood. Such properties of mixtures in a wide region of temperature and pressure are important both in chemical engineering designs and in theoretical investigations of excess thermodynamic properties. However there have been only a few measurements on these properties, especially under high pressure.

The present investigation was undertaken to provide extensive and accurate P-1-T and viscosity data under high pressure for ethanol-water mixtures. Numerical data have been determined at 25°C and 50°C under pressures up to 3200 bar for specific volume and 800 bar for viscosity, employing a modified Adams piezometer and a falling-cylinder viscometer. Empirical correlation formulas have also been presented for both properties from the experimental results.
Specific Volume and Viscosity of Ethanol-Water Mixtures under High Pressure

**Experimental**

**P-V-T measurements**

The specific volume of the ethanol-water mixtures has been measured by a modified Adams piezometer\(^1\) similar to that used by Newitt et al.\(^2\) The piezometer is made of Pyrex glass. The schematic diagram is given in Fig. 1 and the typical dimensions are summarized in Table 1. The volumes of the piezometer and the capillary stem were determined by weighing them filled with distilled water or mercury. The piezometer was set in a high pressure vessel equipped with a pair of optical windows which enable us to observe and control precisely the mercury level in the capillary stem. The high pressure vessel was immersed in a thermostat bath controlled within ±0.01°C. The pressure was measured by Heise Bourdon gauges calibrated against a pressure balance. The uncertainty in pressure measurements is estimated to be less than 0.1%.

The mercury trapped in the piezometer was washed with ethanol and weighed after drying.

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Table 1 Dimensions of Instruments used

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Length at 25°C</th>
<th>Outer diameter</th>
<th>Inner diameter</th>
<th>Total volume</th>
<th>Volume of capillary</th>
</tr>
</thead>
<tbody>
<tr>
<td>Piezometer</td>
<td></td>
<td></td>
<td></td>
<td>18.3215 cm³</td>
<td></td>
</tr>
<tr>
<td></td>
<td>16.7 cm</td>
<td>1.6 cm</td>
<td>1.4 cm</td>
<td>0.03616 cm³</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Length at 29°C</th>
<th>Outer diameter</th>
<th>Inner diameter</th>
<th>Total volume</th>
<th>Volume of capillary</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscometer</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glass tube</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15.5 cm</td>
<td>1.0869 cm</td>
<td>0.8072 cm</td>
<td>3.570 cm</td>
<td>3.5278 g</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.7607 cm</td>
</tr>
</tbody>
</table>

The specific volume of the sample liquid was calculated by the following equation:

\[
v = \frac{V_o (1 - k_a) - V_e (1 - k_e) - (W_{te}/\rho_{te})}{V_o \rho_o}
\]

where

- \( v \): specific volume of the liquid at \( P \) bar in \( \text{cm}^3/\text{g} \).  
- \( V_o \): inner volume of the piezometer at 1 bar in \( \text{cm}^3 \).  
- \( V_e \): inner volume of the capillary at 1 bar in \( \text{cm}^3 \).  
- \( W_{te} \): weight of mercury trapped in g.  
- \( \rho_o \): density of the liquid at 1 bar in g·cm\(^{-3}\).  
- \( \rho_{te} \): density of mercury at \( P \) bar in g·cm\(^{-3}\).  
- \( k_a \): compression of Pyrex glass at \( P \) bar.

The values of \( k_a \) and \( \rho_{te} \) were cited from the results of Adams\(^3\) and Grindley et al.\(^3\) respectively.

The densities of mixtures under the atmospheric pressure were determined experimentally by a pycnometer and partly cited from the handbook.\(^4\)

**Viscosity measurements**

The viscosity has been measured by a falling-cylinder viscometer. The viscosity was determined principally based on the Stokes law on a rigid sphere falling in an infinite homogeneous fluid. The details of the theoretical basis are described by Swift et al.\(^5\-7\).

The schematic diagram of the viscometer employed is given in Fig. 2. The apparatus consists of a precisely bored Pyrex glass tube equipped coaxially in a high pressure vessel and a glass cylind-

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6) J. Lohrenz, G. W. Swift and F. Kurata, ibid., 6, 547 (1960)
7) J. Lohrenz and F. Kurata, ibid., 8, 190 (1962)
rical plummet with hemispherical ends. The instrument dimensions are listed in Table 1. A sample liquid was introduced into both sides of the glass tube through a fine flexible pipe made of stainless steel. The plummet is provided with four small projecting lugs at each end of the cylindrical part, which act as a guide to keep the plummet concentric when it falls. The falling time of the plummet was determined within ±0.1 ms by an electronic time-interval counter with the aids of a He-Ne gas laser beam passed through a pair of optical windows and a phototransistor. The viscometer could be rotated on a horizontal axis in order to return the plummet to its starting position. The temperature of the sample was maintained constant within ±0.05°C by circulating a thermostatic fluid through the jacket around the pressure vessel and measured by a thermister device. The pressure was measured by a Bourdon gauge with the same accuracy as the case of $P-V-T$ measurements. The falling time ranged from 5 to 20 seconds according to the change of the viscosity of samples and was measured about 20 times at each experimental condition. The mean reproducibility of the falling time is within 0.5%. The arithmetic mean values were taken as the final values.

Due to the geometric effect of the plummet and the wall effect of the glass tube, the Stokes law is not valid strictly for a falling-body other than a rigid sphere in an infinite homogeneous fluid. Therefore, the calibration of the present viscometer is required with fluids of known viscosity. The basic equation of the falling-body viscometer is as follows:

$$\eta = K(\rho_d - \rho)l$$  \hspace{1cm} (2)

where

- $\eta$: viscosity of a liquid at $P$ bar in $10^{-8}$Pa·s,
- $\rho$: density of a liquid at $P$ bar in g·cm$^{-3}$,
- $\rho_d$: density of the plummet at $P$ bar in g·cm$^{-3}$,
- $l$: falling time in s.

The instrument constant $K$ and its change with both temperature and pressure were determined based on the experimental viscosity values under the atmospheric pressure obtained by an Ostwald viscometer and the standard viscosity values of water correlated by the International Association for the Properties of Steam.

The densities of mixtures were calculated from Eq. (3) obtained in the present work. When the resistance factor is plotted against the Reynolds number in logarithmic coordinates, the calibration curve is found to be a straight line with a slope of about $-1$. This means that each measurement was carried out in a laminar flow region with lower Reynolds numbers than 0.5 throughout the experimental condition.

Materials

Extra pure ethanol was obtained from Wako Pure Chemical Industries, Ltd. The reported purity is more than 99.5% in volume. Ethanol and water were purified several times by the fractional distillation. The mixtures of ethanol and water were prepared by weighing, using an analytical balance with a sensitivity of ±0.1mg. Therefore their composition, mole fraction of ethanol, should

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be substantially accurate within 0.01 %.

Uncertainty of experimental results

The numerical data of the specific volume and the viscosity obtained contain a definite uncertainty resulting from several sources of experimental errors. The main sources and their portions in the final values are estimated as follows:

(Specific volume measurement)

\[
\begin{array}{|l|c|}
\hline
\text{Error source} & \text{Uncertainty contributing to } \nu \\
\hline
\text{Temperature} & \pm 0.001\% \\
\text{Pressure} & \pm 0.01\% \\
\text{Composition of mixture} & \pm 0.01\% \\
\text{Piezometer volume} & \pm 0.01\% \\
\hline
\end{array}
\]

(Viscosity Measurement)

\[
\begin{array}{|l|c|}
\hline
\text{Error source} & \text{Uncertainty contributing to } \gamma \\
\hline
\text{Temperature} & \pm 0.1\% \\
\text{Pressure} & \pm 0.01\% \\
\text{Composition of mixture} & \pm 0.02\% \\
\text{Falling time} & \pm 0.5\% \\
\text{Density} & \pm 0.01\% \\
\text{Instrument constant, } K & \pm 1.0\% \\
\hline
\end{array}
\]

Taking into account the above estimations, the uncertainties probable in the present measurements may be less than 0.05 % for the specific volume data and 2.0 % for the viscosity data. This fact has been verified satisfactorily by the comparison with the reliable data of pure components by other authors, as partly described below.

Results and Discussions

P-V-T data

Table 2 lists the measured P-V-T relations for ethanol-water mixtures at 25 °C and 50 °C under pressures up to 3200 bar. X is the mole fraction of ethanol in the mixtures. The results are also plotted in Figs. 3 and 4 together with the previously published data for pure water and mixtures for comparison. The specific volume decreases monotonously with increasing pressure throughout the experimental conditions at each composition of mixtures. At 25 °C, the present results agree quite well with the values given by Kell \textit{et al.}\textsuperscript{9} and Grindley \textit{et al.}\textsuperscript{9} for pure water up to 3000 bar and those of Moesveld\textsuperscript{10} for mixtures up to 1500 bar. However, some discrepancies between the present work and the recent report of Yusa \textit{et al.}\textsuperscript{11} are found to be up


\textsuperscript{10} A. L. Moesveld, \textit{Zeit. Phys. Chem.}, 105, 450 (1923)


\* This report was published recently after the completion of experimental part of this study.
### Table 2 The Specific Volume of Ethanol-Water Mixtures

<table>
<thead>
<tr>
<th>Component</th>
<th>Volume (cm³/g)</th>
<th>Pressure (bar)</th>
<th>Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>0.100</td>
<td>0.100</td>
<td>0.100</td>
</tr>
<tr>
<td>Water</td>
<td>0.100</td>
<td>0.100</td>
<td>0.100</td>
</tr>
<tr>
<td>Mixtures</td>
<td>0.100</td>
<td>0.100</td>
<td>0.100</td>
</tr>
</tbody>
</table>

**Note:** The table contains detailed data for the specific volume of ethanol-water mixtures under high pressure conditions.
to 0.9% at high ethanol mole fractions and high pressures. As for 50°C, the present results for pure water are in good agreement with the values of Kell et al.\textsuperscript{32} and Grindley et al.\textsuperscript{31} However, there exists no data on mixtures available in literature for direct comparison with the present work.
Specific Volume and Viscosity of Ethanol-Water Mixtures under High Pressure

Table 4 Coefficients of the Tait Equation

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>X</th>
<th>R (bar)</th>
<th>C (-)</th>
<th>Ave. Dev. (%)</th>
<th>Max. Dev. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>0.00</td>
<td>2754</td>
<td>0.2911</td>
<td>0.04</td>
<td>0.07</td>
</tr>
<tr>
<td></td>
<td>0.10</td>
<td>3124</td>
<td>0.2989</td>
<td>0.04</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>0.20</td>
<td>2268</td>
<td>0.2511</td>
<td>0.05</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>0.40</td>
<td>1552</td>
<td>0.2308</td>
<td>0.04</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>0.60</td>
<td>1157</td>
<td>0.2179</td>
<td>0.07</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>0.80</td>
<td>919</td>
<td>0.2179</td>
<td>0.03</td>
<td>0.08</td>
</tr>
<tr>
<td></td>
<td>1.00</td>
<td>778</td>
<td>0.2065</td>
<td>0.06</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>0.00</td>
<td>3321</td>
<td>0.3241</td>
<td>0.04</td>
<td>0.08</td>
</tr>
<tr>
<td></td>
<td>0.10</td>
<td>2952</td>
<td>0.2887</td>
<td>0.03</td>
<td>0.11</td>
</tr>
<tr>
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<td>0.30</td>
<td>2404</td>
<td>0.2792</td>
<td>0.04</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>0.60</td>
<td>1410</td>
<td>0.2686</td>
<td>0.07</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>0.80</td>
<td>921</td>
<td>0.2282</td>
<td>0.05</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>1.00</td>
<td>790</td>
<td>0.2278</td>
<td>0.08</td>
<td>0.17</td>
</tr>
</tbody>
</table>

For each temperature and composition the specific volume data are correlated as a function of pressure by the following quartic equation:

\[ v = A_1 + A_2 P + A_3 P^2 + A_4 P^3 + A_5 P^4 \tag{3} \]

and the Tait equation:

\[ \frac{v - v_0}{v_0} = C \log \left( \frac{B + P}{B + 1} \right) \tag{4} \]

where \( v_0 \) is the specific volume at the atmospheric pressure and \( P \) the pressure in bar. The empirical coefficients were determined by the least squares method as given in Tables 3 and 4 together with the average and the maximum deviations of the experimental data from the formulas. Although the polynomial equation gives a better fit to the data in general, the isothermal compressibility

\[ \gamma_T = -\frac{1}{v} \left( \frac{\partial v}{\partial P} \right)_T \tag{5} \]

is calculated by the Tait equation because the polynomial equation sometimes gives unexpected behaviors by the differentiation. The functional dependences of the isothermal compressibility on composition are shown in Figs. 5 and 6 at 25°C and 50°C, respectively. It is found that a definite minimum exists near \( x = 0.1 \) on the lower pressure isobars than 2000 bar. As well known in the case of pure water, the isothermal compressibility has a minimum near 50°C on the isobars lower than 2000 bar. This anomaly could be seen in the range of low ethanol composition, and disappears with increasing composition near \( x = 0.2 \), where the isothermal compressibility is independent of temperature under each pressure. Furthermore, it seems that another inflection might occur at higher mole fraction of ethanol near \( x = 0.7 \) on the low pressure isobars. (Concerning this second inflection, more detailed experimental results will be reported in the near future.) Yusa et al. have found the similar
phenomenon in his isothermal compression versus composition isobars. These anomaly would be attributed to complex interactions between the hydrogen-bonded water and the bifunctional nature of alcohol molecules.\textsuperscript{12,13}

**Viscosity**

The viscosity of ethanol-water mixtures has been measured at 25\textdegree{} and 50\textdegree{}C up to 800 bar. The raw data are tabulated in Table 5. Values of the viscosity obtained at the atmospheric pressure are compared as a function of composition to other data available in literature\textsuperscript{14-22} in Fig. 7. The vis-

\begin{thebibliography}{99}
\bibitem{12} F. Franks and D. J. G. Ives, *Quart. Rev.*, 20, 1 (1956)
\bibitem{13} G. Ne\textquoteright{}methy and H. A. Scheraga, *J. Chem. Phys.*, 36, 3382 (1962); *ibid.*, 36, 3401 (1962)
\bibitem{14} J. Traube, *Ber.*, 19, 871 (1886)
\bibitem{15} A. E. Dunstan, *Z. Phys. Chem.*, 49, 500 (1904)
\bibitem{17} W. Herz and E. Martin, *Z. anorg. allgem. Chem.*, 132, 41 (1924)
\bibitem{20} R. L. Kay, *J. Solution Chem.*, 5, 57 (1976)
\bibitem{21} E. L. Lederer, *Koll. Rein.*, 34, 270 (1952)
\end{thebibliography}
Table 5  The Viscosity of Ethanol-Water Mixtures (10^{-3} Pa·s=cP)

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>0.00</th>
<th>0.20</th>
<th>0.30</th>
<th>0.40</th>
</tr>
</thead>
<tbody>
<tr>
<td>P (bar)</td>
<td>25</td>
<td>50</td>
<td>25</td>
<td>50</td>
</tr>
<tr>
<td>1.0</td>
<td>2.335</td>
<td>1.080</td>
<td>2.314</td>
<td>1.108</td>
</tr>
<tr>
<td>9.8</td>
<td>2.400</td>
<td>1.134</td>
<td>2.407</td>
<td>1.155</td>
</tr>
<tr>
<td>19.6</td>
<td>2.461</td>
<td>1.156</td>
<td>2.481</td>
<td>1.168</td>
</tr>
<tr>
<td>29.4</td>
<td>2.500</td>
<td>1.175</td>
<td>2.559</td>
<td>1.211</td>
</tr>
<tr>
<td>39.2</td>
<td>2.567</td>
<td>1.185</td>
<td>2.638</td>
<td>1.249</td>
</tr>
<tr>
<td>49.0</td>
<td>2.602</td>
<td>1.200</td>
<td>2.703</td>
<td>1.275</td>
</tr>
<tr>
<td>58.9</td>
<td>2.666</td>
<td>1.232</td>
<td>2.771</td>
<td>1.341</td>
</tr>
<tr>
<td>68.6</td>
<td>2.702</td>
<td>2.845</td>
<td>2.779</td>
<td></td>
</tr>
<tr>
<td>78.5</td>
<td>2.771</td>
<td>2.939</td>
<td>2.884</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 7  Composition dependence of the viscosity of ethanol-water mixtures at 25° and 30°C at the atmospheric pressure

- This work, ▽: 11, ◆: 14, ⊙: 15, △: 16, ▲: 17, ■: 18, ▽: 19, ◆: 20, □: 21, ⊙: 22

Viscosity data display a maximum at a composition from 0.2 to 0.3 mole fraction of ethanol at each temperature. Agreement of the data among researchers is comparatively good at 25°C, except the
results reported by Yusa et al.,11) whose data are about 4% higher than others near the maximum. Inconsistency of the data is found to increase at 50°C, where the largest deviation between the data of Sabnis10) and those of Traube14) is about 13% near \( x = 0.2 \).

The viscosity data obtained at high pressures are also compared with those in literatures. The pressure effects on the viscosity are shown in Figs. 8 and 9 together with the results of Abassade et al.22) and Yusa et al.11) for mixtures and those of Golubev et al.23) for pure ethanol. Figs. 8 and 9 include partly interpolated data to the same compositions by a graphical method, because the previous data under pressures available for a direct comparison are limited. It is found that agreement among the four sets of data is rather good at low pressures, but that the discrepancy increases with increasing pressure. A serious inconsistency is found in the results for pure ethanol. In the results of Yusa et

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Specific Volume and Viscosity of Ethanol-Water Mixtures under High Pressure

The pressure coefficient of viscosity, $\left( \frac{\partial \eta}{\partial p} \right)_T$, for ethanol is rather small at 25°C, while the data of Golubev et al. and the present work give an obvious positive pressure dependences. The viscosity of the mixtures increases almost linearly with pressure, whereas that of water is almost independent of pressure in this pressure range.

The viscosity isotherms in this work can be represented by a quadratic equation of pressure.

$$\eta = B_1 + B_2 P + B_3 P^2$$

(6)

where $\eta$ is the viscosity in $10^{-3}$Pa·s (=cP) and $P$ the pressure in bar. The coefficients for each mixture are determined by the least squares method and listed in Table 6 with the average and the maximum deviations.

The composition dependence of the viscosity under high pressures is shown in Fig. 10. Each isobar also exhibits a maximum near the composition $x=0.3$. The maximum shifts slightly to higher ethanol fraction with increasing pressure or temperature.

Conclusion

This paper represents the raw experimental data on the specific volume and the viscosity of ethanol-water mixtures as functions of temperature, pressure and composition. This basic information would be necessary to solve the complicated behavior of aqueous solutions of hydroxy-compounds, which are yet inadequate understood. However, the temperature range is narrow in the present measurements, and more detailed measurements is thought to be needed fully near the composition where the anomaly appears. Furthermore, the relationship between the equilibrium (thermodynamic) properties and the non-equilibrium (transport) properties should be derived, especially as for the appearance of anomalies, as well as in the case of aqueous solutions of other alcohols.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>$x$</th>
<th>$B_1$</th>
<th>$B_2 \times 10^4$</th>
<th>$B_3 \times 10^8$</th>
<th>Ave. Dev. (%)</th>
<th>Max. Dev. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>0.20</td>
<td>2.34083</td>
<td>5.83501</td>
<td>-7.0312</td>
<td>0.23</td>
<td>0.41</td>
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<tr>
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<td>0.30</td>
<td>2.32078</td>
<td>8.15612</td>
<td>-5.517</td>
<td>0.25</td>
<td>0.41</td>
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<td></td>
<td>0.40</td>
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<td>0.37</td>
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</tr>
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<td></td>
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<td>0.36</td>
</tr>
<tr>
<td>50</td>
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<td>3.1000</td>
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<td>1.49</td>
</tr>
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<td>0.50</td>
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<td>2.64408</td>
<td>18.153</td>
<td>0.60</td>
<td>1.13</td>
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<td></td>
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<td>5.21548</td>
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<td>1.55</td>
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<td></td>
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<td>0.85</td>
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<tr>
<td></td>
<td>0.80</td>
<td>0.80963</td>
<td>4.17241</td>
<td>6.664</td>
<td>0.66</td>
<td>1.36</td>
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<tr>
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<td>1.00</td>
<td>0.690264</td>
<td>5.07855</td>
<td>-5.885</td>
<td>0.44</td>
<td>1.12</td>
</tr>
</tbody>
</table>
Fig. 10 Composition dependence of the viscosity of ethanol-water mixtures at 25°C and 50°C under high pressure

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