



# Viscosity and density measurements of compressed liquid dimethyl adipate using oscillating body techniques



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## ABSTRACT

The article reports viscosity measurements of compressed liquid dimethyl adipate obtained with a vibrating wire sensor. The vibrating wire instrument was operated in the forced, or steady-state, mode of oscillation. The viscosity measurements were carried out up to 20 MPa and at temperatures from (293 to 358) K. The required density of the liquid sample was obtained using a vibrating U-tube densimeter, model DMA HP, from Anton Paar GmbH. The measurements were performed in the temperature range (293–358) K and at pressures up to 25 MPa.

The viscosity results were correlated with density, using a modified hard-spheres scheme. The root mean square deviation of the data from the correlation is less than 0.2%. The expanded uncertainty of the present viscosity results is estimated as  $\pm 1\%$  at a 95% confidence level.

Independent viscosity measurements were performed, at atmospheric pressure, using an Ubbelohde capillary in order to compare with the vibrating wire results, extrapolated by means of the above mentioned correlation. The two data sets agree within  $\pm 0.6\%$ , well within the mutual uncertainty of the experimental methods. No literature data could be found for the viscosity of dimethyl adipate at pressures above 0.1 MPa. As a consequence, the present viscosity results could only be compared upon extrapolation of the vibrating wire data to 0.1 MPa. The present extrapolated vibrating-wire results have a good agreement with the literature data at temperatures around 293 K. However, the absolute deviations of the literature results increase steadily with increasing temperature up to around +0.04 mPa s or +2.0%. For higher temperatures up to 358 K the absolute deviations are nearly constant, around +0.05 mPa s. The maximum relative deviation to literature data in the whole range of the present viscosity measurements was +4.2% at 358 K. The density results obtained in the present work agree with the literature data within  $\pm 0.1\%$  in the temperature and pressure ranges of the measurements.

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## 1. Introduction

Adipates are claimed to be “green” solvents for a wide range of applications in the chemical and polymer, industries. They are used as solvents, plasticizers, lubricants, hydraulic fluids, gear and automotive engine oils, low temperature greases and fuel additives [1–10]. They are stable, have low toxicity, low vapour pressure and are commercially available at reasonably low cost [1–10].

As plasticizers they are alternatives to some phthalates [4,5,8], which may possibly have undesirable effects [3,8]. As novel

solvents, they have the potential to replace conventional organic solvents [11], with high volatility and high toxicity [3,7]. Diesters with small chain substitutes, like dimethyl adipate, have viscosity and density close to those of water. As fuel additives they can be injected directly on automotive engines and simultaneously decrease both NO<sub>x</sub> and smoke emissions [3,6,12].

Applications of these liquids in industrial processes require the knowledge of their thermophysical properties, like viscosity and density. Recently, simultaneous measurements of viscosity and density of compressed liquid diethyl adipate were obtained with a vibrating wire technique by Meng et al. [10]. Comuñas et al. [2], published density data of compressed liquid dimethyl, diethyl and diisobutyl adipates, using an Anton Paar DMA HPM densimeter. The latter authors have also reported viscosity measurements of the same adipates [2], at atmospheric pressure, obtained with an Anton Paar Stabinger SVM 3000 viscometer.

Notwithstanding the numerous potential applications of dimethyl adipate, no viscosity data have been found in the literature for pressures above 0.1 MPa. In the present work measurements of

*Abbreviations:* HFC, hydrofluorocarbons; KF, Karl-Fischer; NMR, nuclear magnetic resonance; PTB, Physikalisch-Technische Bundesanstalt; PTFE, politetrafluoroethylene; SI, International System of Units.

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the viscosity and of the density of compressed dimethyl adipate were performed in the range of (293–358) K and at pressures up to 20 MPa. The viscosity measurements were carried out using a vibrating wire technique in the forced, or steady-state, oscillation mode of operation. The density measurements were carried out with an Anton Paar DMA HP densimeter.

The results for both properties are compared with independent measurements performed in the present work and with the available literature data, which, for the viscosity, is limited to results at atmospheric pressure.

### 1.1. Vibrating wire viscosity measurements

The vibrating wire instrument for measuring viscosity used in the present work is a new version of the equipment which has been validated by Pádua et al. [13] and used to study 2,2,4-trimethylpentane [14], 1,1,1,2-tetrafluoroethane (HFC 134a) [15], cyclohexane [13], toluene [16] and mixtures of poly(ethyleneglycols) saturated with supercritical carbon dioxide [17], over wide ranges of temperature and pressure. The initial instrumentation and method used in those works, is discussed fully in Ref. [18]. The vibrating wire sensor consists of an axially tensioned metallic wire that is subject to a magnetic field. An alternating current of variable frequency is applied, in a range of frequencies around the resonance frequency. As a consequence, the wire undergoes transverse oscillations that induce an electromotive force across the wire [18]. The analysis of the signal in the frequency domain (resonance curve) enables the determination of the viscosity of the fluid, through the description of the hydrodynamic effects of the fluid on the oscillation parameters of the wire [19].

The procedure to determine the viscosity has undergone some recent improvements [20,21]. Shortly, the new procedure is based on the treatment of the complex voltage measured at the wire ends, and not only of its modulus.

In the present apparatus the tungsten wire is tensioned by a weight. However, the authors decided not to use the possibility to determine the density of the samples, by resort to a description of the effect of the buoyancy force acting on the weight on the oscillation characteristics of the vibrating wire sensor [13,18]. The complementary density data necessary to compute the viscosity from the vibrating wire raw data was obtained independently by resort to Anton Paar vibrating U-tube densimeters, which are thought to ensure a higher accuracy under the present circumstances.

The electronic instrumentation and the acquisition of the raw data used in the present work, are similar to those described by Diogo et al. [20,21]. In particular, the complex voltage at the wire ends, resulting from the forced wire oscillations is given, according to the hydrodynamic model, as a function of the frequency,  $\omega$ , by [20]

$$\mathbf{V}_H = \frac{Y}{\omega(\beta' + 2\Delta_0) + i\omega(1 + \beta) + (\omega_0^2/i\omega)} \quad (1)$$

where  $Y$  is a constant dependent on the current intensity, the magnetic flux, and the mass per unit length of the wire;  $\omega_0$  is the wire resonance frequency in the total absence of damping;  $\Delta_0$  is the internal damping of the wire [13]. The parameters  $\beta$  and  $\beta'$  account for the added mass due to the acceleration of the fluid and the viscous damping due to the fluid motion, respectively [13,18]. The viscosity,  $\eta$ , and the density,  $\rho$ , of the fluid are related to  $\beta$  and  $\beta'$  through relations published by Retsina et al. [19]. The experimental complex voltage was taken to be the sum of  $\mathbf{V}_H$ , as given by Eq. (1) with additional contributions which are aimed to incorporate all the distorting perturbations that may affect

**Table 1**

Characterization of the sample of toluene, used in the calibration of the vibrating wire sensor, and of the sample of dimethyl adipate, used in the density and viscosity measurements.

Liquid	Source of sample	Water content (mg kg <sup>-1</sup> )	Purity (mass fraction)
Toluene	Riedel-de Haën	22	99.7% <sup>b</sup>
Dimethyl adipate	Sigma Aldrich	<sup>a</sup>	99% <sup>b</sup>

<sup>a</sup> The water content of the sample of dimethyl adipate before and after the density and viscosity measurements are reported in Tables 3, 5 and 6, respectively.

<sup>b</sup> Mass fraction purity reported by the supplier.

the frequency response of the vibrating wire sensor as described earlier [20].

$$\mathbf{V}_{AC} = \mathbf{I} \left( i\omega L_2 + \left[ \frac{1}{R_1 + i\omega L_1} + i\omega C_1 \right]^{-1} \right) + i\mathbf{b} + \mathbf{V}_H \quad (2)$$

## 2. Materials and methods

### 2.1. Materials

The toluene used on the calibration of the vibrating wire sensor, was acquired from Riedel-de Haën with a purity of 99.7% and the dimethyl adipate was provided by Sigma Aldrich with 99% purity. The purity of both compounds was verified by <sup>1</sup>H and <sup>13</sup>C nuclear magnetic resonance spectrometry (NMR), and no contamination was detected other than water. Both liquids were dried with molecular sieves from Sigma Aldrich with 0.4 nm porosity and no further purification was performed. Before introduction in the vibrating wire measurement cells, both liquids were filtered with a porous ceramic filter, with (16–40)  $\mu\text{m}$  porosity. For both the capillary viscosity and the density measurements, a PTFE 25 mm diameter membrane filter with 0.45  $\mu\text{m}$  porosity was used. The water content was monitored, before and after all the viscosity and density measurements, using a Karl-Fischer 831 KF Coulometer from Metrohm. Both liquids were also degassed before the vibrating wire measurements by using helium. Table 1 shows the characterization of the sample of toluene, used in the calibration of the vibrating wire sensor, and of the sample of dimethyl adipate, used in the density and viscosity measurements.

### 2.2. Measurement of the viscosity using the vibrating wire technique

The viscosity measurements have been carried out using a vibrating-wire sensor with a tungsten wire with a nominal radius of 50  $\mu\text{m}$ , supplied by Goodfellow (U.K.), tensioned by an aluminium sinker with a mass of about 0.172 kg. The sensor parameters are shown in Table 2. The wire radius has been determined by calibration with liquid toluene at 293.03 K and 0.1 MPa by using viscosity

**Table 2**

Parameters of the vibrating wire sensor at  $T_{\text{ref}}$ .

	$T_{\text{ref}}$ (K)		
Tungsten wire			
Radius <sup>a</sup>	293.03	$R_s$ (m)	$50.02 \times 10^{-6}$
Density <sup>b</sup> , [24]	293.15	$\rho_s$ (kg m <sup>-3</sup> )	19230
Sinker			
Mass		$M_w$ (kg)	0.1719116
Resonance under vacuum			
Frequency <sup>c</sup>	293.1	$\omega_1$ (rad s <sup>-1</sup> )	7403.5
Internal damping coefficient <sup>c</sup>	293.1	$\Delta_0$	$4.18 \times 10^{-5}$

<sup>a</sup> By calibration,

<sup>b</sup> From the literature,

<sup>c</sup> Measured

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