Speeds of Sound in Methanol at Temperatures from 233.33 to 353.21 K at Pressures up to 20 MPa

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Abstract
We report experimental speeds of sound in methanol. Measurements were conducted at temperatures from 233 to 353 K with pressures up to 20 MPa using the double-path length pulse-echo technique. The relative expanded combined uncertainty (k = 2) in measurement was estimated to vary from 0.012 to 0.014%, considering contributions from temperature, pressure, path length calibration, pulse timing, and purity of the sample. Experimental speeds of sound gained in the scope of this work were compared with the equation of state by de Reuck and Craven, as well as with further data from literature.

Keywords Methanol · Pulse-echo technique · Speed of sound

1 Introduction
The power-to-methanol technology is considered more and more important with increasing focus on the challenge of storing energy from volatile renewable sources. In such processes, hydrogen descending from electrolysis operated with excess renewable energy is converted to methanol with CO₂ captured from, e.g., industrial combustion gases [1–3]. At times of insufficient supply of power from renewable sources, stored liquid methanol can be used as fuel to cover the excess demand of electricity. Design and engineering of such processes employ thermophysical property data gained from fundamental equations of state. The currently used fundamental equation of state of methanol was developed by de Reuck and Craven [4]. Since some shortcomings of this equation of state are well known, Thol and Span [5] currently work on a new equation of state. Since such approaches are empirical models, their accuracy is directly related to the quality of the employed experimental data.

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Even though several experimental \((p, c, T)\) data sets of methanol are available in the literature, only very few authors provide comprehensive data sets over larger ranges of temperature and especially pressure (see Table 1). In addition, not all of the available data are consistent, which necessitated further speed of sound measurements to clarify the existing data situation. Temperatures of the isotherms studied in this work have been selected partly to allow for direct comparison with other data sets. In a \((p,T)\) diagram, Fig. 1 shows the state points of speed of sound measurements carried out in the scope of this work as well as of further data available from literature.

2 Experimental Section

2.1 Apparatus Description

Speed of sound measurements in methanol were conducted employing the double-path length pulse-echo technique with an apparatus according to the design of Meier and Kabelac [58]. The experimental set-up has been used for speed of sound measurements of several liquids in our previous works [59–62]; thus, it is only explained in brief at this point. A detailed description with illustrations of the measuring cell and a schematic drawing of the entire measuring system can be taken from the work of Gedanitz et al. [63]. The actual acoustic sensor consists of an \(x\)-cut quartz crystal as transducer, located axially off-center between two polished stainless steel reflectors, resulting in two different path lengths \(L_1 = 20\ \text{mm}\) and \(L_2 = 30\ \text{mm}\). If the piezoelectric disc transducer is excited by a tone burst (here a 30-cycle sinusoidal burst at 8 MHz) of a waveform generator (Agilent, model 3322A), ultrasonic pulses propagate into the surrounding fluid and are reflected at both ends of the measuring cell. The returning wave fronts excite the transducer and the resulting electronic signals are captured by a digital oscilloscope (Agilent, model MS6032A). These signals are referred to as echoes. Due to the unequal path lengths, the second echo is detected delayed by the time difference \(\Delta t_{\text{echo}}\). The speed of sound can be determined according to Eq. (1), incorporating a correction \(\tau\) according to Harris [64] to account for diffraction:

\[
c = \frac{2(L_2 - L_1)}{\Delta t_{\text{echo}} + \tau}.
\]

The waveform of the two resulting echoes is averaged from 16 consecutive pulses and a bandpass filter based on a Fast Fourier Transform with a bandwidth of 1.6 MHz is applied, to enhance the signal-to-noise ratio. Based on these processed data, the time difference \(\Delta t_{\text{echo}}\) is computed according to the algorithm described by Dubberke et al. [65].

The acoustic sensor was housed in a stainless steel pressure vessel and allows for speed of sound measurements up to 20 MPa. Two vibrating-quartz crystal pressure transducers (Paroscientific, models 1000-500A and 1000-6 K) and a differential pressure indicator (Rosemount, model 3051) are utilized to measure the pressure at
| Author | Year | Points | $T$ (K) | $p$ (MPa) | Phase\(^a\) |
|--------|------|--------|---------|-----------|-------------|
| Abraham et al. [8] | 2000 | 3 | 303–313 | 0.101325 | Liquid |
| Abraham et al. [9] | 1997 | 3 | 303–313 | 0.101325 | Liquid |
| Allam and Lee [10] | 1996 | 2 | 298 | 0.101325 | Liquid |
| Aminabhavi et al. [11] | 1993 | 3 | 298–308 | 0.101325 | Liquid |
| Aminabhavi and Banerjee [12] | 1998 | 1 | 298 | 0.101325 | Liquid |
| Aminabhavi and Patil [13] | 1999 | 1 | 298 | 0.101325 | Liquid |
| Arce et al. [14] | 2004 | 1 | 283–334 | 0.101325 | Liquid |
| Arce et al. [15] | 2006 | 1 | 298–314 | 0.101325 | Liquid |
| Artemchenko [16] | 1963 | 6 | 283–333 | 0.101325 | Liquid |
| Bahadur and Deenadayalu [17] | 2011 | 4 | 298–303 | 0.101325 | Liquid |
| Bakshi et al. [18] | 1996 | 1 | 280–361 | 0.101325 | Liquid |
| Bohidar [19] | 1988 | 9 | 293 | 0.1–83 | Liquid |
| Carnevale and Litovitz [20] | 1955 | 5 | 303 | 0.098–196 | Liquid |
| Casas et al. [21] | 2001 | 1 | 298 | 0.101325 | Liquid |
| Chauhan et al. [22] | 1995 | 3 | 298–318 | 0.101325 | Liquid |
| Danusso and Fadigati [23] | 1953 | 4 | 293–323 | 0.101325 | liquid |
| Dávila et al. [7] | 2016 | 42 | 253–353 | 0.1–30 | liquid |
| Dávila and Trusler [24] | 2009 | 56 | 298–343 | 0.091–60 | Liquid |
| Eden and Richardson [25] | 1960 | 3 | 293 | 0.1–98 | Liquid |
| Emery and Gasse [26] | 1979 | 1 | 273 | 0.101325 | Liquid |
| Fort and Moore [27] | 1965 | 1 | 298 | 0.101325 | Liquid |
| Freyer et al. [28] | 1929 | 6 | 273–323 | 0.101325 | Liquid |
| Gadaibaev and Shakhparonov [29] | 1973 | 4 | 181–213 | 0.101325 | Liquid |
| George et al. [30] | 2002 | 2 | 308–319 | 0.101325 | liquid |
| Gill et al. [31] | 1993 | 1 | 298 | 0.101325 | Liquid |
| Golik et al. [32] | 1958 | 4 | 293–323 | 0.101325 | Liquid |
| Gonzalez et al. [33] | 2004 | 3 | 293–303 | 0.101325 | Liquid |
| Govindarajan et al. [34] | 2003 | 5 | 293–313 | 0.101325 | Liquid |
| Iglesias et al. [35] | 1998 | 1 | 298 | 0.101325 | Liquid |
| Iglesias et al. [36] | 1998 | 1 | 298 | 0.101325 | Liquid |
| Iloukhani and Rostami [37] | 2003 | 1 | 303 | 0.101325 | Liquid |
| Jatkar [38] | 1939 | 2 | 370–408 | 0.101325 | Liquid |
| Javed [6] | 2019 | 63 | 220–500 | 0.1–124 | liquid |
| Kumar et al. [39] | 1981 | 1 | 298 | 0.101325 | Liquid |
| Marino et al. [40] | 2000 | 1 | 298 | 0.101325 | Liquid |
| Moses Ezhil Raj et al. [41] | 2009 | 5 | 303–323 | 0.101325 | liquid |
| Ogawa and Murakami [42] | 1987 | 1 | 298 | 0.101325 | Liquid |
| Ohmori et al. [43] | 2001 | 32 | 513–540 | 4–50 | SCR |
| Orge et al. [44] | 1997 | 1 | 298 | 0.101325 | Liquid |
| Parthasarathy et al. [45] | 1953 | 2 | 300 | 0.101325 | Liquid |
| Pereira et al. [46] | 2002 | 5 | 293–333 | 0.101325 | Liquid |
| Pereira et al. [47] | 2003 | 5 | 293–333 | 0.101325 | Liquid |
a standard uncertainty of \( u(p) = 0.0024 \) MPa. The entire pressure vessel is immersed into a calibration bath thermostat (Fluke, model 7060), realizing speed of sound measurements at temperatures from 233 to 353 K. The temperature is measured with a long-stem 25 Ω standard platinum resistance thermometer (SPRT, Rosemount Aerospace, model 162CE), calibrated on ITS-90 and read with a direct current thermometry bridge (Isotech, model TTI-2), stating a standard uncertainty in temperature measurement of \( u(T) = 0.004 \) K. With measurements over the stated temperature and pressure range, thermal expansion and pressure deformation can affect the actual path length, which is considered according to Eq. (2):

![Figure 1](https://example.com/fig1.png)

**Fig. 1** Selection of experimental \((p, c, T)\) data of methanol from literature and \((p, T)\) state points investigated in the present work shown in a \((p, T)\) phase diagram. ⬤, this work; ▼, Javed et al. [6]; ◊, Davila et al. [7]; ×, further data from literature; ———, phase boundary and ☆, critical point as calculated with the equation of state of de Reuck and Craven [4] (Color figure online)
where $\Delta L_0$ is the path length difference at the reference temperature $T_0=293.15$ K and the reference pressure $p_0=0.1$ MPa. The mean value of the isobaric expansivity $\alpha$ at reference pressure $p_0$ was received from values for the differential thermal expansion of stainless steel 1.4571 according to Eq. (3):

$$10^6\alpha/K^{-1} = \sum_{i=0}^{4} \frac{a_i}{(i+1)} \cdot \left( \frac{T-T_0}{T_0} \right)^i.$$  

While the coefficients $a_i$ for $i \geq 1$ are values given by Meier [66] according to Table 2, $a_0$ and the path length difference $\Delta L_0$ given in Eq. (2) are determined from calibration measurements with high-purity water. Here, the calibration remains the same as presented in our previous work [60], where measurements at $T=274.15, 278.15, 283.15, 293.15, 303.15, 313.15, 323.15, 333.15, 343.15,\text{ and }353.15$ K and ambient pressure were conducted. The calibration was evaluated by comparison with the IAPWS-95 equation of state by Wagner and Prüß [67] with the experimental speeds of sound being reported within the uncertainty of the equation of state of 0.005%.

In Eq. (2), $\beta$ describes the mean compressibility of the cell at temperature $T$ and is determined from material properties [68] according to Eq. (4):

$$\beta = (1-2\nu)/E = (1-2\nu) \left[ \sum_{i=0}^{4} b_i(T/K)^i \right]^{-1},$$

where $E$ is Young’s modulus and $\nu=0.3$ is Poisson’s ratio with the employed coefficients $b_i$ given in Table 2.

### 2.2 Experimental Material

The samples used in the present work are specified in Table 3. High-purity water with a purity of 99.9997 mol% was employed for the calibration. The purity of the used methanol was found to be 99.9300 mol%, investigated by gas chromatographical analysis, as stated by the supplier. Additionally, a mass fraction of water of less than 0.001%, known from coulometric Karl Fischer titration, was stated. In order to avoid contact with the humidity of ambient air, the methanol sample was transferred

| $a_i$ | $b_i$ (GPa) |
|------|-------------|
| 8.36800 | 219.720 |
| 5.08059 | $-0.080$ |
| $-3.74480$ | -- |
| 1.86720 | -- |
| $-0.34148$ | -- |
to a stainless steel bottle within the dry inert argon atmosphere of a glove box that was manufactured in-house. Prior to use, the sample was degassed by repeated freeze–pump–thaw cycles, as described in our previous works [59–62].

### 2.3 Experimental Procedure

The experimental procedure applied to conduct the speed of sound measurements in the scope of this work was adapted from previous works [59, 60]. The stainless steel bottle containing the sample of methanol was attached to the evacuated speed of sound apparatus and carefully heated. Simultaneously, the temperature of the bath thermostat was set to the lowest possible temperature of 233 K, thus taking advantage of the pressure and temperature gradient during the filling process. A connected hand pump was utilized to increase the pressure up to 20 MPa and the system was left to equilibrate prior to the first measurement. Measurements were subsequently carried out along isotherms with decreasing pressure. From isotherm to isotherm, the temperature of the measuring cell was increased overnight.

### 2.4 Uncertainty Analysis

The combined expanded uncertainty in speed of sound was estimated according to our previous studies [59, 60], where the approach is explained in detail, by developing the following term with consideration of Eqs. (1) and (2):

$$ c(T, p) = c_0 \frac{(\Delta t_0 + \tau_0)}{(\Delta t + \tau)} \left[ 1 + a(T - T_0) - \frac{\beta}{3}(p - p_0) \right]. $$

(5)

The square of the standard uncertainty of the speed of sound is then described by Eq. (6):

$$ u^2(c) = \left( \left( \frac{dc}{dc_0} \right) a(c_0) \right)^2 + \left( \left( \frac{dc}{d\Delta t} \right) a(\Delta t_0) \right)^2 + \left( \left( \frac{dc}{da} \right) a(a) \right)^2 + \left( \left( \frac{dc}{dp} \right) a(p) \right)^2 $$

$$ = \left( \left( \frac{e}{e_0} \right) a(e_0) \right)^2 + \left( \left( \frac{e}{\Delta e_0} \right) a(\Delta e_0) \right)^2 + \left( \left( \frac{e}{\Delta t} \right) a(\Delta t) \right)^2 + \left( \frac{c_0\Delta t_0(T - T_0)}{\Delta t} \right)^2 a(a) + \left( \frac{-c_0\Delta t_0(p - p_0)}{3\Delta t} \right)^2 a(p). $$

(6)
Further contributions resulting from the measurement of temperature and pressure as well as from the presence of impurities are considered within the determination of the expanded combined uncertainty $U(c)$ according to Eq. (7) with the expansion factor $k=2$:

$$U(c) = k \sqrt{u^2(c) + \left[(\partial c/\partial p) T u(p)\right]^2 + \left[(\partial c/\partial T) p u(T)\right]^2 + u^2(c(w))}. \tag{7}$$

The respective derivatives were determined by sensitivity analysis, employing the equation of state of de Reuck and Craven [4]. The impact of potential impurities was investigated using mixture models, implemented in TREND 4.0 [69]. Water and ethanol were investigated as the impurities with the largest influence. The result for the combined expanded uncertainty $U(c)$ for the speed of sound measurement in methanol at $T=293.20$ K and $p=5$ MPa is exemplified in Table 4 and was estimated to be 0.134 m·s$^{-1}$. Among all conducted measurements, $U(c)$ varies between 0.126 and 0.176 m·s$^{-1}$, while the relative expanded combined uncertainty remains almost constant with values from 0.012 to 0.014%.

### 3 Results and Discussion

The speed of sound was measured at seven or eight state points on each of nine isotherms, leading to a $(p, c, T)$ data set comprising 65 measuring points (see Table 5). Absolute experimental speeds of sound are plotted versus pressure in Fig. 2, clarifying internal consistency throughout the entire $(p,T)$ region and showing a decreasing speed of sound with increasing temperature. In the present work, speeds of

| Quantity | Value | Standard uncertainty | Sensitivity coefficient | Uncertainty contribution |
|----------|-------|----------------------|-------------------------|-------------------------|
| $c_0$    | 1482.52 m·s$^{-1}$ | 0.07 m·s$^{-1}$ | 0.774 | 0.057 m·s$^{-1}$ |
| $\Delta t_0$ | 13.32385 µs | 0.00017 µs | 86 m·s$^{-1}$·µs$^{-1}$ | 0.015 m·s$^{-1}$ |
| $\Delta t$ | 17.11560 µs | 0.00022 µs | 67 m·s$^{-1}$·µs$^{-1}$ | 0.015 m·s$^{-1}$ |
| $\alpha$ | $8.37 \times 10^{-6}$ K$^{-1}$ | $4.18 \times 10^{-7}$ K$^{-1}$ | $4.59 \times 10^{-4}$ m·s$^{-1}$·K | 0.002 $\times 10^{-4}$ m·s$^{-1}$ |
| $\beta$ | $2.04 \times 10^{-6}$ MPa$^{-1}$ | $1.02 \times 10^{-7}$ MPa$^{-1}$ | $1.88 \times 10^{-3}$ m·s$^{-1}$·MPa | 0.019 $\times 10^{-2}$ m·s$^{-1}$ |
| $T$ | 293.202 K | 0.004 K | 3.14 m·s$^{-1}$·K$^{-1}$ | 0.013 m·s$^{-1}$ |
| $p$ | 5.0166 MPa | 0.0024 MPa | 6.02 m·s$^{-1}$·MPa$^{-1}$ | 0.014 m·s$^{-1}$ |
| $c(w)$ | 1147.612 m·s$^{-1}$ | 0.036 m·s$^{-1}$ | 1 | 0.021 m·s$^{-1}$ |

Combined expanded uncertainty ($k=2$): 0.134 m·s$^{-1}$
sound in methanol vary from 926 m·s\(^{-1}\) (\(T = 353\) K and \(p = 0.25\) MPa) to 1420 m·s\(^{-1}\) (\(T = 233\) K and \(p = 20\) MPa).

Relative deviations of the experimental speeds of sound in methanol from values calculated with the equation of state of de Reuck and Craven [4] as reference (zero line) are plotted versus pressure in Fig. 3. Relative deviations of the entire data set fall well within the uncertainty of the equation of state, stated by the authors to be

### Table 5: Speeds of sound \(c_{\text{exp}}\) of methanol and expanded uncertainties \(U(c)\) (\(k = 2\)) at temperatures \(T\) and pressures \(p\)

| \(T / K\)     | \(p / \text{MPa}\) | \(c_{\text{exp}} / \text{m·s}^{-1}\) | \(U(c) / \text{m·s}^{-1}\) | \(T / K\)     | \(p / \text{MPa}\) | \(c_{\text{exp}} / \text{m·s}^{-1}\) | \(U(c) / \text{m·s}^{-1}\) |
|---------------|---------------------|--------------------------------------|-----------------------------|---------------|---------------------|--------------------------------------|-----------------------------|
| 233.249       | 0.155               | 1335.959                             | 0.166                       | 300.199       | 10.015              | 1154.058                             | 0.135                       |
| 233.249       | 0.509               | 1337.570                             | 0.166                       | 300.199       | 15.023              | 1181.108                             | 0.137                       |
| 233.249       | 2.011               | 1344.397                             | 0.167                       | 300.198       | 20.012              | 1206.709                             | 0.139                       |
| 233.249       | 5.019               | 1357.677                             | 0.169                       | 313.202       | 0.155               | 1053.364                             | 0.128                       |
| 233.249       | 10.027              | 1379.331                             | 0.171                       | 313.202       | 0.509               | 1055.703                             | 0.128                       |
| 233.249       | 15.025              | 1400.133                             | 0.173                       | 313.202       | 2.009               | 1065.503                             | 0.129                       |
| 233.249       | 19.991              | 1420.070                             | 0.176                       | 313.202       | 5.012               | 1084.413                             | 0.130                       |
| 253.218       | 0.155               | 1260.037                             | 0.151                       | 313.202       | 10.021              | 1114.569                             | 0.132                       |
| 253.218       | 0.507               | 1261.797                             | 0.151                       | 313.202       | 15.020              | 1142.949                             | 0.135                       |
| 253.218       | 2.011               | 1269.270                             | 0.152                       | 313.202       | 20.053              | 1169.991                             | 0.137                       |
| 253.218       | 5.026               | 1283.832                             | 0.153                       | 333.204       | 0.156               | 989.182                              | 0.126                       |
| 253.219       | 10.024              | 1307.299                             | 0.155                       | 333.204       | 0.508               | 991.750                              | 0.126                       |
| 253.218       | 15.027              | 1329.873                             | 0.157                       | 333.204       | 2.009               | 1002.559                             | 0.127                       |
| 253.218       | 20.016              | 1351.505                             | 0.160                       | 333.204       | 5.017               | 1023.307                             | 0.128                       |
| 273.205       | 0.155               | 1187.716                             | 0.140                       | 333.204       | 10.010              | 1055.986                             | 0.131                       |
| 273.205       | 0.506               | 1189.615                             | 0.140                       | 333.204       | 15.028              | 1086.748                             | 0.133                       |
| 273.205       | 2.017               | 1197.773                             | 0.141                       | 333.204       | 20.030              | 1115.589                             | 0.136                       |
| 273.205       | 5.012               | 1213.683                             | 0.142                       | 350.206       | 0.254               | 935.924                              | 0.126                       |
| 273.205       | 10.013              | 1239.253                             | 0.144                       | 350.205       | 0.504               | 937.922                              | 0.126                       |
| 273.205       | 15.019              | 1263.648                             | 0.147                       | 350.205       | 1.005               | 941.877                              | 0.126                       |
| 273.205       | 20.064              | 1287.210                             | 0.149                       | 350.205       | 2.007               | 949.700                              | 0.127                       |
| 293.203       | 0.155               | 1119.236                             | 0.132                       | 350.206       | 5.013               | 972.170                              | 0.128                       |
| 293.203       | 0.509               | 1121.340                             | 0.132                       | 350.205       | 10.021              | 1007.528                             | 0.131                       |
| 293.202       | 2.009               | 1130.279                             | 0.133                       | 350.205       | 15.020              | 1040.294                             | 0.134                       |
| 293.202       | 5.017               | 1147.612                             | 0.134                       | 350.206       | 19.988              | 1070.761                             | 0.137                       |
| 293.202       | 10.013              | 1175.297                             | 0.137                       | 353.203       | 0.254               | 926.000                              | 0.126                       |
| 293.202       | 15.013              | 1201.627                             | 0.139                       | 353.203       | 0.509               | 928.054                              | 0.126                       |
| 293.203       | 20.049              | 1226.850                             | 0.141                       | 353.203       | 2.007               | 940.019                              | 0.127                       |
| 300.199       | 0.155               | 1096.280                             | 0.130                       | 353.202       | 5.011               | 962.872                              | 0.128                       |
| 300.199       | 0.505               | 1098.470                             | 0.131                       | 353.203       | 10.017              | 998.583                              | 0.131                       |
| 300.199       | 1.007               | 1101.570                             | 0.131                       | 353.203       | 15.026              | 1031.849                             | 0.134                       |
| 300.199       | 2.248               | 1109.144                             | 0.131                       | 353.203       | 20.010              | 1062.759                             | 0.137                       |
| 300.199       | 5.018               | 1125.549                             | 0.133                       |                |                     |                                     |                            |

*Standard uncertainties are \(u(T) = 0.004\) K, \(u(p) = 0.0024\) MPa*
However, a trend can be observed along each isotherm with the relative deviations from the lowest to the highest pressure decreasing between 0.27 and 0.42%. Altogether, relative deviations vary from –0.66% at \( T = 353 \) K and \( p = 15.03 \) MPa to 0.35% at \( T = 253 \) K and \( p = 0.16 \) MPa.

Again, Fig. 4 shows the comparison of the speed of sound measured in the scope of this work with the equation of de Reuck and Craven [4] as reference (zero line). However, the comparison focuses on the individual temperatures and can thus consider further data sets from literature. The data set by Davila et al. [7] is in very good agreement with our reported data and falls within the experimental uncertainty.
at temperatures between 273 and 333 K. At 253 and 353 K both data sets differ by up to 0.04%, slightly exceeding the claimed combined experimental uncertainty. Speeds of sound, gained in the scope of this work, are also in good agreement with the data set by Javed et al. [6] at 350 K, especially at higher pressures, where deviations are within the experimental uncertainty. Comparison with data from Javed et al. [6] at lower temperatures shows a systematical downward offset at 300 K and a systematical upward offset at 244 and 253 K, respectively (see Fig. 4). Further data by Sun et al. [54] and Wilson and Bradley [55] show a upward offset at overlapping temperatures from 273 to 313 or 333 K and at comparable pressures (see Fig. 4).

4 Conclusion

Speeds of sound in high-purity methanol were measured at temperatures from 233 to 353 K and at pressures up to 20 MPa with the overall relative expanded combined uncertainty \( (k=2) \) ranging from 0.012 to 0.014%. The new data deviate from the equation of state of de Reuck and Craven [4] by up to 0.66% but agree with the data set of Davila et al. [7] within 0.014% at temperatures between 273 and 333 K.
and within 0.040% beyond this temperature range. The measurements conducted by Javed et al. [6] are confirmed partly, especially at higher temperatures. On other isothersms significant deviations to the data of Javed et al. [6] are observed.

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