



Speeds of Sound in *n*-Hexane and *n*-Heptane at Temperatures from (233.33 to 353.21) K and Pressures up to 20 MPa

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Abstract

The speed of sound in high-purity *n*-hexane and *n*-heptane was experimentally studied utilizing the double-path length pulse-echo technique. Measurements with each alkane were carried out at temperatures from (233 to 353) K with pressures up to 20 MPa. Considering the uncertainty contributions from temperature, pressure, path-length calibration, pulse timing and sample purity, the relative expanded combined uncertainty ($k=2$) in the speed of sound in *n*-hexane ranges from (0.012 to 0.042) % over the investigated ranges of pressure and temperature; for *n*-heptane, the uncertainty varies from (0.014 to 0.018) %. The sound speed data measured in *n*-hexane were among the data used for the development of a new fundamental equation of state, which is, however, not described in this work. The experimental data of *n*-heptane can be considered appropriate for modeling purposes and validation of existing equations of state.

Keywords *n*-Heptane · *n*-Hexane · Pulse-echo technique · Speed of sound

1 Introduction

The alkane *n*-hexane is currently being investigated as a possible working fluid for ORC-processes in geothermal power plants or in the field of waste heat recovery since the high critical temperature could lead to a better efficiency [1, 2]. Furthermore, it is relevant as a fuel component in the process chain of biodiesel [3] and in food processing technology for the extraction of vegetable oils [4, 5]. Additionally, *n*-hexane is relevant for a variety of industrial applications and is used as an extractant or reaction medium in polymerization [6]. As a constituent of natural gas, there

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is also a special interest in the thermodynamic properties of *n*-hexane for mixture models of such fuels.

The alkane *n*-heptane is mainly used as an extractant and solvent in the chemical industry and for vitamin [7] or vegetable oil extraction [8] in the food industry. It is also used in the extraction of bitumen from oil sands [9], the treatment of catalysts [10] and in polymerization processes [11]. Moreover, *n*-hexane is employed as a standard zero point for the octane number of engine fuels [12] and, thus, for the octane rating of petrol or rather the synthesis of knock resistant fuels [13].

A fundamental equation of state for *n*-hexane and *n*-heptane widely used in technical applications was developed by Span and Wagner in 2003 [14]. The aim of their work was to represent 15 non-polar and weakly polar substances with a generalized form of an equation of state so that it remains valid for an entire group of substances. This approach made it possible to describe similar substances for which only few measurement data were available. However, this model is not intended to serve as a reference equation for substances with a sufficient database. The equation for *n*-hexane by Span and Wagner [14] was developed without specific fitting of any speed of sound data, and only one data set by Muringer et al. [15] was used for the development of the equation for *n*-heptane [14]. As a result, an uncertainty of 2 % for calculated sound speeds in the homogeneous liquid phase of *n*-heptane was reported. Against this background, new fundamental equations of state for *n*-hexane and *n*-heptane are under development at Ruhr University Bochum. For the development of the new equation for *n*-hexane by Thol et al. [16], as already implemented in REFPROP 10.0 [18] and TREND 4.0 [19], selected data points in the liquid phase of the speed of sound data sets by Daridon et al. [20] and Khasanshin et al. [21] were considered as primary data as well as the speed of sound data measured within the scope of the present work. The objective of our present study was to improve the data situation for the speed of sound in *n*-hexane at lower temperatures and pressures and to provide a consistent as well as reliable data set for modeling purposes.

For the development of the new fundamental equation of state for *n*-heptane, Thol et al. [17] used the data sets of Daridon et al. [22] and Muringer et al. [15] as well as selected data points of the speed of sound measurements by Baumhögger et al. [23] as primary data in the homogeneous liquid phase. The speed of sound data of *n*-heptane measured in the present work were not yet used in the development process of this new equation, which is also already implemented in REFPROP 10.0 [18] and TREND 4.0 [19]. However, our new data can still be used for validation of this equation as an independent and reliable data set or for further improvement in the future. Speed of sound data of other alkanes [*n*-pentane (Scholz et al. [24]) and *n*-hexane (this work)] measured with the same experimental setup and according to the same metrological protocols have already been used for the development of new fundamental equations of state by Thol et al. [16, 25]; therefore, we are confident that our results of speed of sound measurements in *n*-heptane are particularly suitable for the sake of validation or improvement of new models.

The (p , T) state points we investigated within the scope of the present work and further data from literature are presented in pressure versus temperature phase diagrams (Fig. 1) for *n*-hexane and *n*-heptane, respectively. Additionally, the available experimental (p , c , T) data from literature are summarized in Table 1 for *n*-hexane

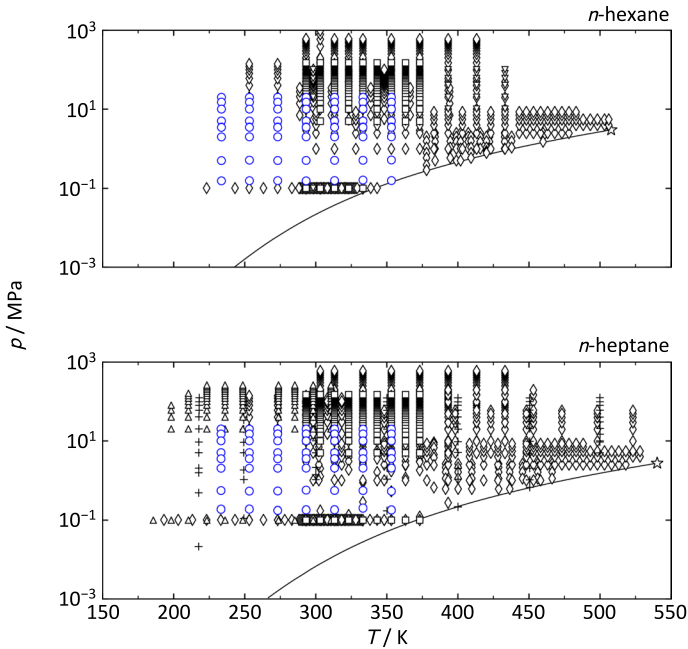


Fig. 1 Pressure vs. temperature phase diagram for *n*-hexane and *n*-heptane showing the (p, T) state points investigated in the present work and a selection of experimental (p, c, T) data from literature in the liquid phase. \circ this work; \triangle Muringer et al. [15]; ∇ Khasanshin et al. [21]; \square Daridon et al. [20, 22]; $+$ Baumhögger et al. [23]; \diamond further data from literature (see Table 1 and Table 2, respectively); —, phase boundary and \star , critical point, calculated with the equations of state by Thol et al. [16, 17]

and in Table 2 for *n*-heptane, stating the number of points and ranges of temperature and pressure.

2 Experimental Section

2.1 Apparatus Description

The measurements reported in this work were carried out with a speed of sound apparatus where the double-path pulse-echo technique is applied similar to the design of Meier and Kabelac [166]; thus far, our apparatus has been used for investigating the sound speed in several liquids [24, 167, 168]. Inside a cylindrical measuring cell, an x-cut quartz-crystal is used as a piezoelectric disc transducer, which is mounted perpendicular to the axis of the cell. The transducer is located axially off-center between two polished stainless steel reflectors that close the cell at each end. This results in two different path lengths of $L_1 = 20$ mm and $L_2 = 30$ mm. To conduct a measurement, a waveform generator (Agilent, model 3322A) generates a 30-cycle sinusoidal burst at a carrier frequency of 8 MHz,

Table 1 Review of speed of sound measurements in *n*-hexane

Author	Year	Points	<i>T</i> /K	<i>p</i> /MPa	Phase ^a
Allegra et al. [26]	1970	8	303.14	0.1–981	Liquid
Alonso et al. [27]	2011	7	283–314	0.101325	Liquid
Aminabhavi et al. [28]	1994	5	298–319	0.101325	Liquid
Badalyan et al. [29]	1971	52	303–334	0.1–118	Liquid
Ball and Trusler [30]	2001	82	298–374	0.1–101	Liquid
Basu et al. [31]	2013	6	298–324	0.101325	Liquid
Boelhouwer [32]	1967	40	253–334	0.1–141	Liquid
Bohidar [33]	1988	9	293.14	0.1–83	Liquid
Bolotnikov et al. [34]	2005	17	293–374	0.0–1	SATL
Cerdeirina et al. [35]	2001	42	288–330	0.101325	Liquid
Cholpan et al. [36]	1981	6	223–344	0.101325	Liquid
Cominges et al. [37]	2001	4	288–309	0.101325	Liquid
Daridon et al. [20]	1998	275	293–374	0.1–151	Liquid
Dmitriev [38]	1984	5	302.99	0.1–201	Liquid
Eden and Richardson [39]	1960	6	293.14	0.1–99	Liquid
Ermakov and Ismagilov [40]	1976	24	377–438	0.3–3	Liquid
Gayol et al. [41]	2007	14	288–321	0.101325	Liquid
Gepert and Ernst [42]	2003	5	293–314	0.101325	Liquid
Golik et al. [43]	1987	5	302.99	0.1–99	Liquid
Ismagilov and Ermakov [44]	1982	42	383–460	0.3–3	Liquid/SATL
Kagramanyan and Badalyan [45]	1978	42	303–374	0.1–203	Liquid/gas
Kannappan et al. [46]	2002	5	293–314	0.101325	Liquid
Khasanshin and Shchemelev [47]	2001	20	298–434	0.1–50	Liquid
Khasanshin et al. [21]	2008	48	298–434	0.1–101	Liquid
Kireev and Otpushchennikov [48]	1974	13	183–304	0.0–1	SATL
Kiryakov et al. [49]	1971	42	303–314	0.1–197	Liquid
Kling et al. [50]	1953	23	293–374	0.1–50	Liquid
Mascato et al. [51]	2007	5	288–309	0.101325	Liquid
Melikhov et al. [52]	1991	119	293–414	10.0–601	Liquid
Melnikov et al. [53]	1988	10	153–434	0.0–1	SATL
Neruchev et al. [54]	1969	11	293–494	0.0–3	SATL
Neruchev et al. [55]	2005	54	293–508	0.0–4	SATL/SATV
Orge et al. [56]	1999	4	303–319	0.101325	Liquid
Rodriguez et al. [57]	2003	4	293–314	0.101325	Liquid
Rolling and Vogt [58]	1960	28	288–395	0.1–35	Liquid
Scholz et al. (<i>this work</i>)	2020	56	233–354	0.2–21	Liquid
Verveiko et al. [59]	1986	26	293–324	0.1–608	Liquid
Verveiko et al. [60]	1991	16	293–324	0.1–601	Liquid
Zheng et al. [61]	2016	179	300–507	0.0–9	Liquid SATL/SATV
Zotov et al. [62]	1975	10	173–474	0.0–2	SATL

^aSATL or SATV denotes saturated liquid states or saturated vapor states, respectively

Table 2 Review of speed of sound measurements in *n*-heptane

Author	Year	Points	<i>T</i> /K	<i>p</i> /MPa	Phase ^a
Aicart et al. [63]	1983	3	298.14	0.101325	Liquid
Akamatsu et al. [64]	1987	1	298.14	0.101325	Liquid
Alonso et al. [27]	2011	7	283–314	0.101325	Liquid
Alonso et al. [65]	2013	3	293–304	0.1	Liquid
Aminabhavi and Gopalakrishna [66]	1995	1	298.15	1	Liquid
Aminabhavi et al. [28]	1994	5	298–319	0.101325	Liquid
Aralaguppi et al. [67]	1991	1	298.15	0.101325	Liquid
Aralaguppi et al. [68]	1999	1	298.15	0.101325	Liquid
Badalyan et al. [29]	1971	91	303–364	0.1–118	Liquid
Bahadur and Sastry [69]	2003	1	308.15	0.101325	Liquid
Baragi et al. [70]	2006	1	298.15	0.101325	Liquid
Basu et al. [31]	2013	6	298–324	0.101325	Liquid
Baumhögger [23]	2017	83	217–501	0.0–126	Liquid
Benson et al. [71]	1981	1	298.14	0.101325	Liquid
Blanco et al. [72]	2013	1	298.15	0.101325	Liquid
Blanco et al. [73]	2013	15	288–324	0.101325	Liquid
Boelhouwer [32]	1967	60	253–454	0.1–141	Liquid
Calvar et al. [74]	2009	1	313.15	0.101325	Liquid
Canosa et al. [75]	1999	3	288–299	0.101325	Liquid
Cerdeirina et al. [35]	2001	46	288–334	0.101325	Liquid
Cholpan et al. [36]	1983	11	223–324	0.101325	Liquid
Chorazewski and Tkaczyk [76]	2006	5	292–315	0.101325	Liquid
Chorazewski et al. [77]	2010	5	292–314	0.101325	Liquid
Choudary et al. [78]	1986	2	303–314	0.101325	Liquid
Danusso and Fadigati [79]	1953	1	303.13	0.101325	Liquid
Daridon et al. [22]	1999	279	293–374	0.1–151	Liquid
Dey et al. [80]	2009	1	298.15	0.101325	Liquid
Dharmaraju et al. [81]	1981	1	303.14	0.101325	Liquid
Dzida and Cempa [82]	2008	48	292–319	0.1–102	Liquid
Dzida and Ernst [83]	2003	59	293–319	0.1–122	Liquid
Dzida and Goralski [84]	2006	6	293–319	0.101325	Liquid
Dzida and Waleczek [85]	2010	48	292–319	0.1–102	Liquid
Dzida et al. [86]	2005	90	293–319	0.1–93	Liquid
Edgar and Calingaert [87]	1929	1	293.14	0.101325	Liquid
Freyer et al. [88]	1929	7	273–324	0.101325	Liquid
Garkusha et al. [89]	1988	2	293–334	0.101325	Liquid
Gayol [90]	2012	1	298.15	0.101325	Liquid
Gayol et al. [41]	2007	15	288–324	0.101325	Liquid
Gayol et al. [91]	2010	1	298.15	0.101325	Liquid
Golik and Ivanova [92]	1962	6	293–344	0.101325	Liquid
Golik et al. [93]	1982	68	313–454	0.1–197	Gas/liquid
Hasanov [94]	2012	73	298–524	0.1–59	Liquid
Ismagilov and Ermakov [44]	1982	48	391–467	0.1–3	Gas/liquid

Table 2 (continued)

Author	Year	Points	T/K	p/MPa	Phase ^a
Junquera et al. [95]	1988	1	298.14	0.101325	Liquid
Kimura et al. [96]	1983	1	298.14	0.101325	Liquid
Kimura et al. [97]	2013	1	298.15	0.101325	Liquid
Kireev and Otpushchennikov [48]	1974	12	193–304	0.101325	Liquid
Kiryakov et al. [98]	1974	54	303–394	0.1–11	Liquid
Kiryakov and Otpushchenniko [99]	1971	123	303–394	0.1–203	Liquid
Kiyohara and Benson [100]	1979	1	298.14	0.101325	Liquid
Kling et al. [50]	1953	23	293–374	0.1–50	Liquid
Kumaran et al. [101]	1984	1	298.14	0.101325	Liquid
Luning Prak et al. [102]	2014	10	293–339	0.101325	Liquid
Malakondaia et al. [103]	1978	2	298–314	0.101325	Liquid
Marino et al. [104]	2001	6	278–299	0.101325	Liquid
Marks [105]	1955	1	273.15	0.101325	Liquid
Martinez-Banos et al. [106]	2013	5	293–314	0.101325	Liquid
Melnikov et al. [53]	1988	11	193–524	0.101325	Gas/liquid
Muringer et al. [15]	1985	113	185–311	0.1–264	Liquid
Naidu and Naidu [107]	1981	1	303.14	0.101325	Liquid
Nakai et al. [108]	1991	1	298.15	0.101325	Liquid
Nath [109]	1996	1	298.15	0.101325	Liquid
Nath [110]	1997	1	293.15	0.101325	Liquid
Nath [111]	1998	1	303.15	0.101325	Liquid
Nath [112]	2002	2	288–299	0.101325	Liquid
Natt and Baccaredd [113]	1948	1	293.14	0.101325	Liquid
Nayak et al. [114]	2001	1	298.15	0.101325	Liquid
Nayeem et al. [115]	2016	3	308–319	0.101325	Liquid
Neruchev et al. [54]	1969	10	293–474	0.101325	Gas/liquid
Neruchev et al. [55]	2005	83	213–554	0.0–3	SATL/SATV
Nishikawa et al. [116]	1998	1	298.15	0.101325	Liquid
Ohomuro et al. [117]	1987	1	298.14	0.101325	Liquid
Orge et al. [118]	1997	1	298.15	0.101325	Liquid
Orge et al. [56]	1999	3	303–314	0.101325	Liquid
Oswal and Maisuria [119]	2002	1	303.15	0.101325	Liquid
Pandey et al. [120]	1994	1	298.15	0.101325	Liquid
Papaioannou et al. [121]	1991	1	298.15	0.101325	Liquid
Pardo et al. [122]	2002	8	288–309	0.101325	Liquid
Parthasarath [123]	1935	1	296.14	0.101325	Liquid
Pereiro and Rodriguez [124]	2007	3	293–304	0.101325	Liquid
Pereiro and Rodriguez [125]	2008	3	293–304	0.101325	Liquid
Povey et al. [126]	2003	1	293.15	0.101325	Liquid
Prakash et al. [127]	1981	1	303.14	0.101325	Liquid
Prasad et al. [128]	1978	1	296.14	0.101325	Liquid
Przybyla et al. [129]	2011	5	292–314	0.101325	Liquid
Rai et al. [130]	1989	1	298.14	0.101325	Liquid
Raikar et al. [131]	1993	1	298.15	0.101325	Liquid

Table 2 (continued)

Author	Year	Points	T/K	p/MPa	Phase ^a
Rajagopal and Subrahmanyam [132]	1978	3	298–324	0.101325	Liquid
Rajendran [133]	1996	1	298.15	0.101325	Liquid
Rama Rao [134]	1940	6	273–324	0.101325	Liquid
Rao and Reddy [135]	1988	1	298.14	0.101325	Liquid
Rathnam et al. [136]	2010	3	303–314	0.101325	Liquid
Rathnam et al. [137]	2012	3	303–314	0.101325	Liquid
Reddy et al. [138]	2013	1	303.15	0.101325	Liquid
Rodriguez et al. [139]	1999	3	288–299	0.101325	Liquid
Rodriguez et al. [57]	2003	4	293–314	0.101325	Liquid
Rosario Rajkuma et al. [140]	1984	1	303.15	0.101325	Liquid
Sachdeva and Nanda [141]	1981	13	293–354	0.101325	Liquid
Sastry and Dave [142]	1996	1	303.15	0.101325	Liquid
Sastry and Raj [143]	1996	3	298–314	0.101325	Liquid
Sastry and Valand [144]	1996	2	298–309	0.101325	Liquid
Sastry [145]	1997	2	303–314	0.101325	Liquid
Sastry et al. [146]	1999	3	298–319	0.101325	Liquid
Schaafts [147]	1944	1	293.14	0.101325	Liquid
Scholz et al. (<i>this work</i>)	2020	54	233–353	0.15–20	Liquid
Sharma et al. [148]	2008	1	298.15	0.101325	Liquid
Sharma et al. [149]	2008	1	308.15	0.101325	Liquid
Siwach et al. [150]	2010	1	308.15	0.101325	Liquid
Sperkach et al. [151]	1979	7	223–324	0.101325	Liquid
Sreenivasulu and Naidu [152]	1979	2	303–314	0.101325	Liquid
Sugiura and Ogawa [153]	2009	1	298.15	0.101325	Liquid
Tamura et al. [154]	1983	1	298.14	0.101325	Liquid
Tamura et al. [155]	1984	2	293–304	0.101325	Liquid
Tamura et al. [156]	1994	1	298.15	0.101325	Liquid
Tardajos et al. [157]	1986	1	298.14	0.101325	Liquid
Tourino et al. [158]	2004	1	298.15	0.101325	Liquid
Treszczanowicz et al. [159]	1982	1	298.14	0.101325	Liquid
Tutov and Otpushennikov [160]	1966	1	293.14	0.101325	Liquid
Utter and Kling [161]	1948	1	290.14	0.101325	Liquid
Wilson and Richards [162]	1931	3	298–324	0.101325	Liquid
Wilson et al. [163]	1991	1	293.15	0.101325	Liquid
Zheng et al. [61]	2016	189	302–537	0.0–9	Liquid/ SATL/SATV
Zotov et al. [164]	1968	6	293–344	0.101325	Liquid
Zotov et al. [62]	1975	10	193–514	0.101325	Gas/liquid
Zotov et al. [165]	1995	219	193–540	0.0–601	Liquid/ SATL/SATV

^aSATL or SATV denotes saturated liquid states or saturated vapor states, respectively

modulated by a half-cycle \sin^2 function that excites the transducer. The ultrasonic pulses originating from the excited transducer propagate into the fluid into both directions until they reach the two reflectors and are returned to the transducer. Due to the unequal path lengths, one of the two returning echoes arrives delayed by the time difference Δt_{echo} . The electronic signal generated at the transducer is captured with a digital oscilloscope (Agilent, model MS6032A), which averages the waveforms of 16 consecutive pulses. The averaged waveform is treated with a bandpass filter, being based on a Fast-Fourier-Transform with a bandwidth of 1.6 MHz, to enhance the signal-to-noise ratio. Ultimately, the speed of sound c is determined by the following equation

$$c = \frac{2(L_2 - L_1)}{\Delta t_{\text{echo}} + \tau} \tag{1}$$

where the time difference Δt_{echo} between the two echoes is computed from the averaged waveform according to an algorithm by Dubberke et al. [169]. Equation 1 includes the time difference between the ideal and the real case of the wave propagation. The correction of these diffraction effects follows a method described by Harris [170] with values of $\tau/\Delta t_{\text{echo}}$ varying between (0.0016 and 0.0048) %. Since the nominal difference in the path lengths is influenced by the variation of temperature and pressure due to thermal expansion and pressure deformation, the actual path length difference can be determined by the following equation:

$$\Delta L(T, p) = \Delta L_0 \left[1 + \alpha(T - T_0) - \frac{\beta}{3}(p - p_0) \right] \tag{2}$$

In the work we report here, ΔL_0 is the path-length difference at the reference conditions of $T_0=293.15$ K and $p_0=0.1$ MPa. The mean value of the isobaric expansivity α at pressure p_0 was determined by integration of values for the differential thermal expansion of stainless steel 1.4571 over the temperature interval $[T_0, T]$, according to Eq. 3:

$$10^6 \alpha / \text{K}^{-1} = \sum_0^4 \frac{a_i}{(i + 1)} \cdot \left(\frac{T - T_0}{T_0} \right)^i \tag{3}$$

Here, the coefficients a_i as given in Table 3 are fixed values given by Meier [171], except a_0 which is obtained from calibration (see Sect. 2.2). The mean compressibility β of the cell over the pressure interval $[p_0, p]$ at the

Table 3 Coefficients of Eq. 3 for the mean thermal expansivity α and coefficients of Eq. 4 for the mean isothermal compressibility β of the stainless steel 1.4571 cell

i	0	1	2	3	4
a_i	8.36800	5.08059	- 3.74480	1.86720	- 0.34148
b_i	219.720	- 0.080	-	-	-

temperature T is obtained from material-property data [172] as Young's modulus E and Poisson's ratio $\nu=0.3$ according to the following Eq. 4 with the coefficients b_i given in Table 3:

$$\beta = (1 - 2\nu)/E = (1 - 2\nu) \left[\sum_0^1 b_i(T/K)^i \right]^{-1} \quad (4)$$

The acoustic sensor of the speed of sound apparatus described above was housed in a stainless-steel pressure vessel that was completely immersed into a calibration bath thermostat (Fluke, model 7060). By using an ethylene glycol and water mixture, the temperature range from (233 to 353) K was realized. Temperature measurement was carried out with a long stem 25 Ω standard platinum resistance thermometer (SPRT, Rosemount Aerospace, model 162CE) in conjunction with a direct current thermometry bridge (Isotech, model TTI-2). With a calibration on ITS-90, a standard uncertainty in temperature measurement of $u(T)=0.004$ K was achieved. The pressure was measured using two vibrating-quartz-crystal pressure transducers (Paroscientific, models 1000-500A and 1000-6 K), covering different ranges of pressure. A differential pressure indicator (Rosemount, model 3051) was employed to separate the pressure measuring circuit from the measuring cell, thus, preventing the pressure transducers from getting in contact with the liquid sample, but with nitrogen instead. With a second circulation bath thermostat, the pressure transducers and the differential pressure indicator were adjusted to a constant temperature in order to maintain a standard uncertainty of $u(p)=0.0024$ MPa.

2.2 Calibration

While the time difference between the two echoes (Eq. 1) is measured at each state point, the actual difference in the path lengths is accurately calibrated only once prior to a complete series of measurements. The path length difference ΔL_0 at the reference state and the coefficient a_0 for the calculation of the mean isothermal expansion in Eq. 3 are determined from calibration measurements in high-purity water (see Table 4). In the present work, the calibration remains the same as presented in our previous work [24], where speed of sound measurements were carried out at nine state points with ambient pressure and temperatures of $T=(274.15, 278.15, 283.15, 293.15, 303.15, 313.15, 333.15, 343.15$ and $353.15)$ K. The parameters ΔL_0 and a_0 were regressed using Eqs. 1 to 3. Comparing the results of the calibration in water with values calculated from the IAPWS-95 equation of state by Wagner and Pruß [173], our experimental speed of sound data is reproduced within the uncertainty of the equation of state, which was reported to be 0.005 % in the temperature range of our calibration. Furthermore, reference data by Del Grosso and Mader [174] and Fujii and Masui [175] are in a good agreement with our speed of sound measurements in water.

Table 4 Description of the used chemical samples water, *n*-hexane and *n*-heptane, where *w* is mass fraction

Chemical name	CAS number	Source	Purity as supplied	Purification method	Final purity	Analytical method ^a
Water	7732-18	Sigma-Aldrich	0.999997	Freeze–thaw		
<i>n</i> -Hexane	110-54-3	Sigma-Aldrich	> 0.9900	Freeze–thaw	<i>w</i> = 0.9973	GC, KF ^b
<i>n</i> -Heptane	142-82-5	Honeywell	> 0.9980	Freeze–thaw	<i>w</i> = 0.9984	GC, KF ^c

^aGC is gas chromatography and KF is Karl-Fisher titration.

^bH₂O < 0.010 mass %

^cH₂O < 0.005 mass %

2.3 Experimental Material

A detailed characterization of all substances used in this work can be found in Table 4. High-purity water with a stated purity of 99.9997 % by Sigma-Aldrich was used for the calibration. After the water had been decanted into a stainless-steel bottle, the sample was degassed by repeated cycles of freezing in liquid nitrogen and simultaneous evacuation. Due to their hygroscopic behavior, the two sample materials *n*-hexane and *n*-heptane were decanted within the dry inert nitrogen atmosphere of a glove box and then degassed in the same way. For both materials, a coulometric Karl Fischer titration was carried out by the supplier (Sigma-Aldrich or Honeywell, respectively), in which the water content was specified to be not more than 0.01 mass % for *n*-hexane or 0.005 mass % for the anhydrous *n*-heptane, respectively. The actual purity of the *n*-hexane sample was investigated with gas chromatography to be at least 99.73 mass %, using a flame ionization detector. No further analysis was carried out for *n*-heptane, since a purity of at least 99.84 mass % had already been certified by gas chromatography, conducted by the supplier.

2.4 Experimental Procedure

The experimental procedure applied for the speed of sound measurements presented in this work is the same for both substances. At first, the apparatus was thoroughly cleaned, and the measuring cell was evacuated to remove any residual contamination from previous studies. Before the cell was filled with the sample, the bath thermostat was set to the lowest temperature of 233 K, and the sample cylinder containing either *n*-hexane or *n*-heptane was heated to increase its vapor pressure. By taking advantage of the pressure and temperature gradient, the sample was then transferred into the measuring cell. Next, the pressure was increased to the highest pressure of 20 MPa with the aid of the hand pump being part of the sample manifold, and the system was left to equilibrate before measurements began. Speed of sound measurements for each sample were carried out along isotherms over a temperature range from (233 to 353) K, starting at $T = 233$ K and $p = 20$ MPa and were continued along isotherms with decreasing pressure.

2.5 Uncertainty Analysis

In order to obtain a complete working equation for the estimation of the uncertainty of the speed of sound in *n*-hexane and *n*-heptane (see Eq. 5), ΔL in Eq. 1 was substituted by the relation shown in Eq. 2. Additionally, Eq. 1 was formulated for the measurement in the calibration fluid, taking c_0 , Δt_0 and τ_0 into account and was further solved for ΔL_0 , allowing to eliminate this quantity by substitution in the previously developed equation.

$$c(T, p) = c_0 \frac{(\Delta t_0 + \tau_0)}{(\Delta t + \tau)} \left[1 + \alpha(T - T_0) - \frac{\beta}{3}(p - p_0) \right] \quad (5)$$

The square of the standard uncertainty of the speed of sound is then given according to Eq. 6, with the small correction terms of the diffraction τ neglected for the case of the uncertainty analysis only.

$$\begin{aligned} u^2(c) &= \left[\left(\frac{\partial c}{\partial c_0} \right) u(c_0) \right]^2 + \left[\left(\frac{\partial c}{\partial \Delta t_0} \right) u(\Delta t_0) \right]^2 + \left[\left(\frac{\partial c}{\partial \Delta t} \right) u(\Delta t) \right]^2 + \left[\left(\frac{\partial c}{\partial \alpha} \right) u(\alpha) \right]^2 + \left[\left(\frac{\partial c}{\partial \beta} \right) u(\beta) \right]^2 \\ &= \left[\left(\frac{c}{c_0} \right) u(c_0) \right]^2 + \left[\left(\frac{c}{\Delta t_0} \right) u(\Delta t_0) \right]^2 + \left[\left(\frac{c}{\Delta t} \right) u(\Delta t) \right]^2 + \left[\left(\frac{c_0 \Delta t_0 (T - T_0)}{\Delta t} \right) u(\alpha) \right]^2 + \left[\left(\frac{-c_0 \Delta t_0 (p - p_0)}{3 \Delta t} \right) u(\beta) \right]^2 \end{aligned} \quad (6)$$

The expanded combined uncertainty $U(c)$ with a coverage factor k is then given by Eq. 7:

$$U(c) = k \sqrt{u^2(c) + [(\partial c / \partial p)_T u(p)]^2 + [(\partial c / \partial T)_p u(T)]^2 + u^2(c(w))} \quad (7)$$

No corrections were made for the presence of impurities, therefore, Eq. 6 includes a term $u(c(w))$, which considers the contribution of impurities in the sample to the standard uncertainty of the speed of sound. The influence was quantified by a sensitivity analysis, carried out by using the respective mixture models as available in NIST's REFPROP database 10.0 [18]. Impurities such as water or the respective isomers were investigated, and to be conservative, the largest influence was considered. This was approximately 64 % of the expanded uncertainty for *n*-hexane and about 45 % of the expanded uncertainty for *n*-heptane, at the state point presented in Table 5 or Table 6, respectively.

For both series of measurements, the respective median state point was chosen to exemplify the uncertainty budget for the speed of sound, which has been estimated for each state point individually. The expanded uncertainty of the speed of sound ($k=2$) in *n*-hexane $c=1139 \text{ m s}^{-1}$ at $T=293.20 \text{ K}$ and $p=5 \text{ MPa}$ is 0.219 m s^{-1} , as presented in detail in Table 5, which corresponds to a relative expanded combined uncertainty of 0.019 %. Over the entire (p, T) region investigated, $U(c)$ varies between (0.152 and 0.350) m s^{-1} , i.e., the relative expanded combined uncertainty ranges from (0.012 to 0.042) %, with the contamination by 2-methylpentane being the dominant factor.

Table 5 Uncertainty budget for speed of sound measurements conducted in *n*-hexane at the median state point, where T is temperature, p is pressure, c is sound speed, Δt is time difference, α is the mean isobaric expansivity, β is the mean isothermal compressibility, w is mass fraction and subscript 0 refers to calibration measurements in pure water

Quantity	Value	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution
c_0	1482.52 m s ⁻¹	0.07 m s ⁻¹	0.768	0.057 m s ⁻¹
Δt_0	13.32385 μ s	0.00017 μ s	86 m s ⁻¹ · μ s ⁻¹	0.015 m s ⁻¹
Δt	17.33781 μ s	0.00022 μ s	66 m s ⁻¹ · μ s ⁻¹	0.015 m s ⁻¹
α	8.37×10^{-6} K ⁻¹	4.18×10^{-7} K ⁻¹	4.88×10^{-1} m s ⁻¹ K	0.002×10^{-4} m s ⁻¹
β	2.04×10^{-6} MPa ⁻¹	1.02×10^{-7} MPa ⁻¹	1.87×10^3 m s ⁻¹ ·MPa	0.019×10^{-2} m s ⁻¹
T	293.202 K	0.004 K	4.12 m s ⁻¹ K ⁻¹	0.016 m s ⁻¹
p	5.0238 MPa	0.0024 MPa	7.48 m s ⁻¹ ·MPa ⁻¹	0.018 m s ⁻¹
$c(w)$	1139.256 m s ⁻¹	0.088 m s ⁻¹	1	0.088 m s ⁻¹
Combined expanded uncertainty ($k=2$):				0.219 m s ⁻¹

Sensitivity coefficients associated with temperature and pressure were estimated from the equation of state from Thol et al. [16]

Table 6 Uncertainty budget for speed of sound measurements conducted in *n*-heptane at the median state point, where T is temperature, p is pressure, c is sound speed, Δt is time difference, α is the mean isobaric expansivity, β is the mean isothermal compressibility, w is mass fraction and subscript 0 refers to calibration measurements in pure water

Quantity	Value	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution
c_0	1482.52 m s ⁻¹	0.07 m s ⁻¹	0.801	0.059 m s ⁻¹
Δt_0	13.32385 μ s	0.00017 μ s	89 m s ⁻¹ · μ s ⁻¹	0.015 m s ⁻¹
Δt	16.64000 μ s	0.00024 μ s	71 m s ⁻¹ · μ s ⁻¹	0.015 m s ⁻¹
α	8.37×10^{-6} K ⁻¹	4.18×10^{-7} K ⁻¹	1.08 m s ⁻¹ K	0.005×10^{-4} m s ⁻¹
β	2.04×10^{-6} MPa ⁻¹	1.02×10^{-7} MPa ⁻¹	1.96×10^3 m s ⁻¹ ·MPa	0.020×10^{-2} m s ⁻¹
T	293.203 K	0.004 K	4.03 m s ⁻¹ K ⁻¹	0.016 m s ⁻¹
p	5.0548 MPa	0.0024 MPa	6.81 m s ⁻¹ MPa ⁻¹	0.016 m s ⁻¹
$c(w)$	1187.028 m s ⁻¹	0.061 m s ⁻¹	1	0.061 m s ⁻¹
Combined expanded uncertainty ($k=2$):				0.181 m s ⁻¹

Sensitivity coefficients associated with temperature and pressure were estimated from the equation of state from Thol et al. [17]

The speed of sound in *n*-heptane $c = 1187$ m s⁻¹ at $T = 293.20$ K and $p = 5$ MPa has an expanded uncertainty ($k = 2$) of 0.181 m s⁻¹ as shown in Table 6, which corresponds to a relative expanded combined uncertainty of 0.015% . We find that $U(c)$ varies between (0.157 and 0.217) m s⁻¹ with the relative expanded combined uncertainty being roughly constant between (0.014 and 0.018)%. For the speed of sound data measured in *n*-heptane, the contribution from impurities and from calibration turned out to be the dominant factors.

3 Results

The (p, c, T) results of the speed of sound measurements are listed in Table 7 for n -hexane and in Table 8 for n -heptane, respectively, including the combined expanded uncertainty at each particular state point. Sound speeds were measured at eight (T, p) state points along each of the seven isotherms, leading to a total of 56 (p, c, T) data for each alkane. The resulting absolute speed of sound is plotted versus pressure in Fig. 2 for n -hexane and n -heptane, respectively. In both plots, measuring points at the same temperature are connected in order to visualize the course of each isotherm. We note that both measuring sets are internally consistent throughout the whole temperature and pressure range, and the speed of sound increases

Table 7 Speed of sound c_{exp} of n -hexane and expanded uncertainties $U(c)$ ($k=2$) at temperatures T (ITS-90) and pressures p^a

T K	p MPa	c_{exp} m·s ⁻¹	$U(c)$ m·s ⁻¹	T K	p MPa	c_{exp} m·s ⁻¹	$U(c)$ m·s ⁻¹
233.604	20.023	1488.478	0.178	293.202	3.525	1127.765	0.221
233.603	15.042	1463.712	0.176	293.203	2.026	1115.996	0.224
233.602	10.022	1437.662	0.173	293.202	0.510	1103.853	0.227
233.597	5.027	1410.630	0.170	293.202	0.155	1100.970	0.228
233.595	3.515	1402.213	0.170	313.204	20.017	1166.655	0.238
233.597	2.014	1393.760	0.169	313.204	15.028	1131.696	0.247
233.595	0.504	1385.105	0.168	313.204	10.025	1094.142	0.256
233.596	0.155	1383.093	0.168	313.204	5.023	1053.608	0.266
253.311	20.017	1403.043	0.164	313.204	3.520	1040.750	0.270
253.312	15.019	1375.856	0.160	313.204	2.020	1027.518	0.273
253.311	10.019	1347.372	0.157	313.204	0.513	1013.917	0.276
253.311	5.024	1317.441	0.154	313.204	0.157	1010.628	0.276
253.311	3.522	1308.150	0.153	333.206	20.036	1094.445	0.275
253.310	2.024	1298.706	0.152	333.206	15.033	1056.383	0.285
253.311	0.519	1289.072	0.152	333.206	10.041	1015.274	0.295
253.311	0.156	1286.716	0.152	333.206	5.023	970.149	0.306
273.210	20.018	1321.128	0.160	333.206	3.525	955.763	0.310
273.210	15.027	1291.551	0.162	333.206	2.023	940.888	0.313
273.210	10.022	1260.259	0.165	333.206	0.515	925.376	0.317
273.210	5.028	1227.240	0.169	333.206	0.162	921.654	0.318
273.210	3.525	1216.906	0.171	353.206	20.021	1025.309	0.305
273.211	2.024	1206.370	0.173	353.206	15.011	983.892	0.315
273.210	0.507	1195.528	0.174	353.205	10.037	938.990	0.325
273.210	0.157	1192.984	0.175	353.205	5.031	888.794	0.337
293.203	20.017	1242.275	0.196	353.206	3.529	872.505	0.341
293.203	15.030	1210.138	0.202	353.205	2.026	855.549	0.344
293.203	10.026	1175.844	0.210	353.206	0.523	837.830	0.348
293.202	5.024	1139.256	0.218	353.206	0.157	833.386	0.350

^aStandard uncertainties are $u(T)=0.004$ K, $u(p)=0.0024$ MPa

Table 8 Speed of sound c_{exp} of *n*-heptane and expanded uncertainties $U(c)$ ($k=2$) at temperatures T (ITS-90) and pressures p^a

T K	p MPa	c_{exp} m·s ⁻¹	$U(c)$ m·s ⁻¹	T K	p MPa	c_{exp} m·s ⁻¹	$U(c)$ m·s ⁻¹
233.332	20.186	1522.081	0.217	293.203	3.560	1176.442	0.170
233.332	15.032	1497.663	0.216	293.203	2.046	1165.585	0.171
233.331	10.063	1473.185	0.216	293.203	0.568	1154.697	0.171
233.330	5.061	1447.548	0.215	293.203	0.181	1151.826	0.171
233.330	3.531	1439.498	0.216	313.204	20.124	1210.205	0.163
233.330	2.035	1431.525	0.215	313.203	15.061	1177.072	0.162
233.329	0.551	1423.533	0.215	313.204	10.062	1142.321	0.162
233.329	0.188	1421.540	0.215	313.203	5.055	1105.083	0.163
253.230	20.030	1438.961	0.198	313.204	3.566	1093.443	0.163
253.231	15.075	1413.463	0.197	313.204	2.056	1081.377	0.164
253.231	10.053	1386.447	0.197	313.204	0.555	1069.080	0.164
253.231	5.054	1358.339	0.197	313.204	0.182	1065.956	0.163
253.230	3.544	1349.559	0.197	333.206	19.994	1139.241	0.159
253.231	2.031	1340.664	0.197	333.206	15.051	1104.287	0.158
253.230	0.535	1331.728	0.197	333.206	10.048	1066.375	0.158
253.231	0.189	1329.633	0.197	333.206	5.064	1025.538	0.159
273.207	20.103	1359.830	0.183	333.205	3.548	1012.438	0.160
273.207	15.093	1331.886	0.182	333.205	2.047	999.099	0.160
273.207	10.050	1302.324	0.182	333.205	0.553	985.407	0.161
273.207	5.046	1271.427	0.182	333.205	0.200	982.111	0.161
273.207	3.536	1261.760	0.182	353.205	19.989	1072.219	0.157
273.207	2.037	1252.040	0.182	353.205	15.089	1034.843	0.157
273.207	0.539	1242.103	0.182	353.205	10.047	993.248	0.157
273.207	0.175	1239.655	0.182	353.205	5.056	948.182	0.158
293.203	20.112	1283.421	0.171	353.205	3.569	933.837	0.159
293.203	15.082	1253.031	0.170	353.206	2.051	918.749	0.160
293.203	10.066	1220.973	0.170	353.205	0.531	903.052	0.161
293.203	5.055	1187.028	0.170	353.205	0.180	899.379	0.160

^aStandard uncertainties are $u(T)=0.004$ K, $u(p)=0.0024$ MPa

with decreasing temperature and increasing pressure. Speeds of sound in *n*-hexane, measured in the presented work, vary from 833 m s^{-1} ($T=353$ K and $p=0.15$ MPa) to 1489 m s^{-1} at ($T=233$ K and $p=20$ MPa) and from 899 m s^{-1} ($T=353$ K and $p=0.15$ MPa) to 1522 m s^{-1} at ($T=233$ K and $p=20$ MPa) for *n*-heptane.

4 Discussion

In Figs. 3 and 4, relative deviations of experimental speeds of sound in *n*-hexane and *n*-heptane from values calculated with the equations of state of Thol et al. [16, 17] (zero line) are plotted versus pressure along isotherms; the relative deviation of

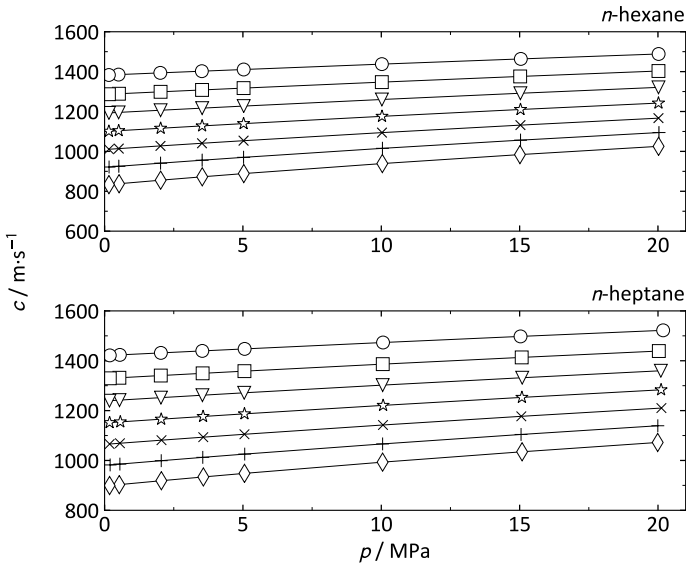


Fig. 2 Results of the speed of sound measurements in *n*-hexane and *n*-heptane. Absolute speeds of sound c_{exp} are plotted versus pressure. \circ 233 K; \square 253 K; ∇ 273 K; \star 293 K; \times 313 K; $+$ 333 K; \diamond 353 K. Lines are shown only to guide the eye

the equation of state by Span and Wagner [14] is furthermore illustrated as a dashed line. Moreover, relative deviations of other relevant speed of sound data from the literature are shown in Figs. 3 and 4.

The data set of *n*-hexane (Fig. 3) obtained in the present work was used in the development of the equation of state of Thol et al. [16] and is represented by the equation within $\pm 0.015\%$ over the whole pressure range for temperatures between (273 and 353) K. These relative deviations are essentially within the estimated uncertainty of our experimental data. At lower temperatures, the experimental speeds of sound were weighted less in the fitting process, and the relative deviation increases up to 0.18% at $T=233$ K, thereby exceeding the experimental uncertainty, which is roughly about 0.012% at these particular state points. Since the calibration with water was carried out starting at $T=274$ K, the necessary extrapolation to $T=233$ K might cause larger relative deviations. The data set by Daridon et al. [20] was also used in the development of the equation of state and is in very good agreement with our reported data within the entire overlapping range of pressure and temperature, starting at $T=293$ K. The data set by Boelhouwer et al. [32] is also consistent with our speed of sound data in the overlapping temperature range from (253 to 333) K and confirms our data particularly at low temperatures; the data set is furthermore agreeing well with the data set by Daridon et al. [20] at higher temperatures and pressures. At $T=(313, 333 \text{ and } 353)$ K and $p=(0.1 \text{ and } 20)$ MPa or (10 and 20) MPa, respectively, a comparison with the data reported by Khasan-shin et al. [21, 47], that was also partly used in the development of the equation of state, is feasible. At all six state points, this data set agrees within (0.04 to 0.08)% with our reported speed of sound data. The data sets by Ball and Trusler [30] and

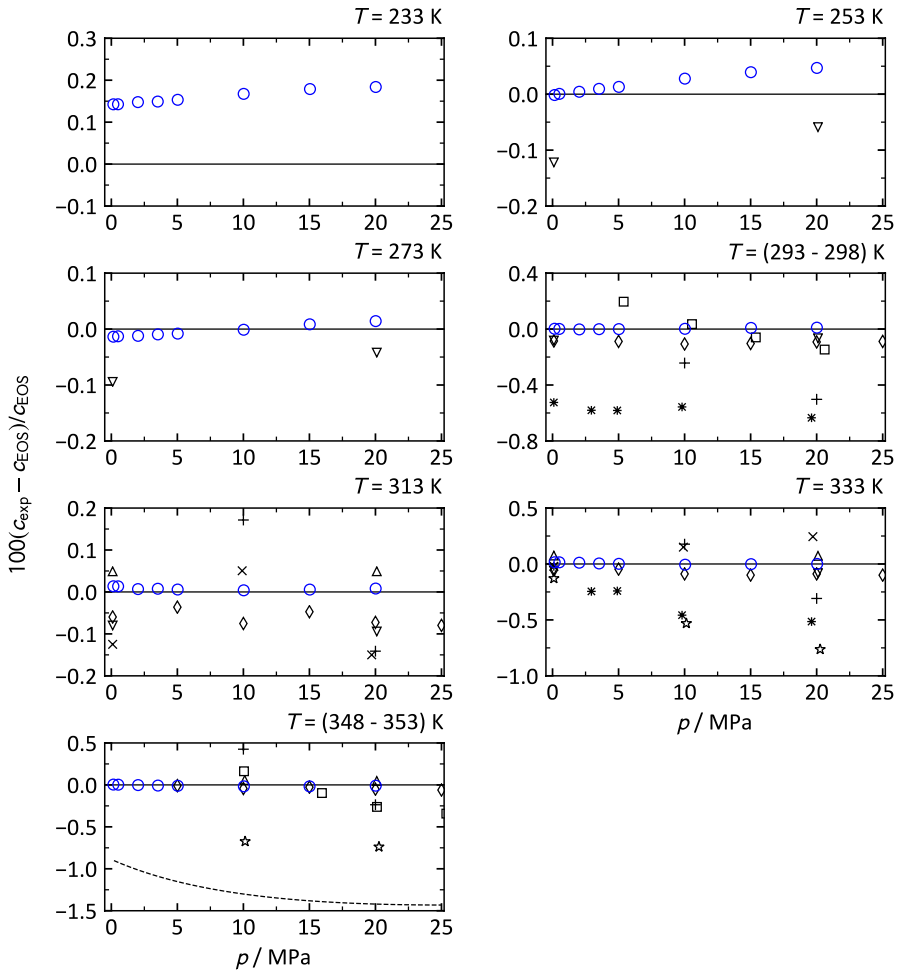


Fig. 3 Results of the speed of sound measurements in *n*-hexane. Relative deviations of the experimental speeds of sound c_{exp} from values c_{EOS} calculated with the equation of state of Thol et al. [16] are plotted versus pressure: \circ this work; \triangle Khasanshin et al. [21]; \diamond Daridon et al. [20]; \square Ball and Trusler [30]; $+$ Melikhov et al. [52]; ∇ , Boelhouwer [32]; \times Badalyan et al. [29]; \star Kagramayan et al. [45]; \ast Kling et al. [50]; --- EOS of Span and Wagner [14]. Please note that different scales for the y-axes are used

Melikhov et al. [52] show a different behavior compared to our data by intersecting the equation of state at pressures between (10 and 15) MPa and with relative deviations of approximately ± 0.25 % or rather ± 0.50 % at the investigated conditions.

The speed of sound data from Kling et al. [50] and Kagramayan et al. [45] show a systematic offset from our data in the investigated ranges of pressure and temperature and differ by up to (0.64 or 0.74) %, respectively. The short multiparameter equation of state by Span and Wagner [14] deviates by up to -2.5 % from our speed of sound data of *n*-hexane.

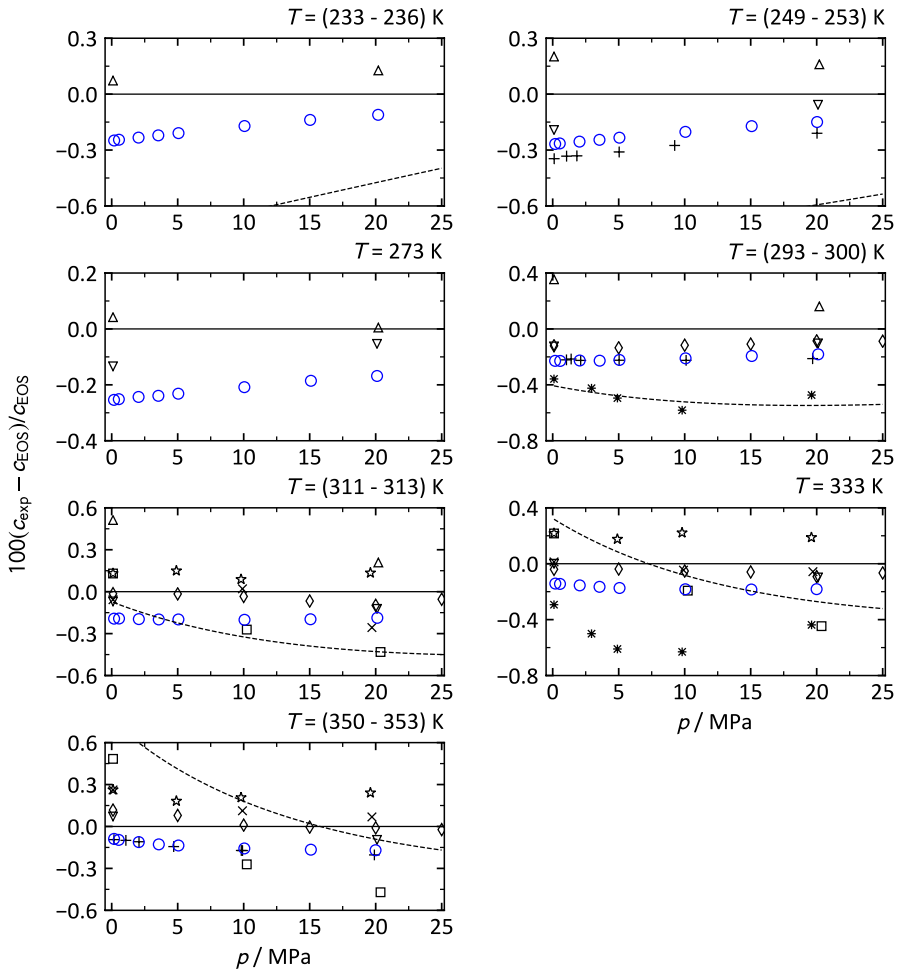


Fig. 4 Results of the speed of sound measurements in *n*-heptane. Relative deviations of the experimental speeds of sound c_{exp} from values c_{EOS} calculated with the equation of state of Thol et al. [17] are plotted versus pressure: \circ this work; Δ Muringer et al. [15]; \diamond Daridon et al. [22]; \square Kiryakov et al. [99]; $+$ Baumhögger [23]; ∇ Boelhouwer [32]; \times Badalyan et al. [29]; \star Golik et al. [93]; \bullet Kling et al. [50]; --- EOS of Span and Wagner [14]. Please note that different scales for the y-axes are used

The speed of sound data of *n*-heptane reported in the scope of this work are presented in Fig. 4 and were not used for the development of the equation of state by Thol et al. [17]. Over the whole temperature and pressure range, an almost systematic offset can be observed with negative relative deviations between -0.26% at $T=253\text{ K}$ and $p=0.18\text{ MPa}$ and -0.09% at $T=353\text{ K}$ and $p=0.18\text{ MPa}$. The trend of each isotherm is somehow comparable with the relative deviations of the model of Span and Wagner [14] from the equation of state by Thol et al. [17]: While the relative deviations are slightly decreasing with increasing pressure at $T=(233\text{ and }253)\text{ K}$ and are almost constant at $T=(273\text{ and }293)\text{ K}$, the trend inverts at higher

temperatures. However, the course of the relative deviations of the Span and Wagner model [14] is changing more significantly with relative deviations of up to 0.75 % at $T=353$ K and $p=0.15$ MPa. In contrast to the results of the speed of sound measurements in *n*-hexane, no inconsistency of the experimental data at $T=233$ K is noticeable.

The speed of sound data reported by Baumhögger et al. [23], which were used in the development of the equation of state by Thol et al. [17], agree with our data within 0.08 % at $T=253$ K and within 0.03 % at $T=(293$ and $353)$ K, over the entire pressure range up to 20 MPa. The data set by Daridon et al. [22] was also used for the development of the equation of state but shows an almost systematic offset of roughly 0.09 % at $T=293$ K that increases with higher temperatures up to 0.22 % at $T=353$ K compared to the data measured in the present work, even though both data sets show a similar trend. The speed of sound data by Muringer et al. [15] were used in the development of the new equation of state as well but are not in good agreement with our data presented here. Just two state points on each isotherm at $p=(0.1$ and $20)$ MPa are overlapping the pressure range of the present work, and the data show a positive offset from our data by (0.17 to 0.71) % at temperatures between (233 and 313) K.

5 Conclusion

We report speeds of sound measured over the temperature range from (233 to 353) K at pressures up to 20 MPa in samples of high-purity *n*-hexane and *n*-heptane. The speed of sound data in *n*-hexane were considered as primary data for the development of the new equation of state by Thol et al. [16]. This work's data agree with the new equation of state within 0.015 % at $T \geq 273$ K and within 0.18 % or better at lower temperatures. The overall relative expanded combined uncertainty ($k=2$) of the *n*-hexane data ranges from (0.012 to 0.042) %. The new speed of sound data of *n*-heptane confirms the data set by Baumhögger et al. [23], which was already used in the development of the new equation of state by Thol et al. [17]. Both data sets agree within 0.08 % at $T=253$ K and 0.03 % at $T=(293$ or $353)$ K, respectively. The overall relative expanded combined uncertainty ($k=2$) of our *n*-heptane data ranges from (0.014 to 0.018) %.

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