Fluid Phase Equilibria 418 (2016) 175–180

Contents lists available at ScienceDirect

Fluid Phase Equilibria

journal homepage: www.elsevier.com/locate/fluid

Speed of sound measurements in deuterium oxide (D_2O) over the temperature range from (278.2 to 353.2) K at pressures up to 20 MPa

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ARTICLE INFO

Article history:
Received 29 August 2015
Received in revised form
18 October 2015
Accepted 20 October 2015
Available online 22 October 2015

Keywords:
Double-path length
Heavy water
Measurements
Pulse-echo technique
Speed of sound

ABSTRACT

We report speed of sound measurements in heavy water (deuterium oxide, D_2O) over the temperature range from (278.2 to 353.2) K with pressures up to 20 MPa. The double-path length pulse-echo technique was applied. A calibration with ordinary (light) water at temperatures between $T = (274.15$ and $353.15)$ K and at ambient pressure was carried out. After adjusting the numerical value of the acoustic path length, our calibration data agreed with values calculated from the reference equation of state for water within the uncertainty of the equation of 0.005%. For the measurements reported here, the relative combined expanded uncertainty ($k = 2$) in speed of sound is 0.011%. Comparisons of our experimental data with values calculated from the reference equation for heavy water and to a preliminary version of a newly developed equation of state for heavy water are presented. Relative deviations are within the uncertainties stated for the equations, which are 1.0% below and 0.5% above $T = 350$ K for the reference equation (estimation based on comparisons to literature data) and 0.08% for the newly developed equation of state (stated by the author). For the reference equation, relative deviations ranged from (-0.32 to 0.62)%, and for the newly developed equation relative deviations of (-0.07 to 0.02)% were observed. Our data will be used for further improvement of the new equation of state, which is expected to become the new reference for properties of heavy water.

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1. Introduction

Accurate thermophysical properties are most conveniently calculated with fundamental equations of state, and experimental sound speed data are particularly useful for developing these equations. Against this background we investigated the (p, c, T) behavior of heavy water (deuterium oxide, D_2O). Measurements were carried out along six isotherms at $T = (278.21$, 283.21, 293.20, 313.20, 333.20 and $353.20)$ K with pressures up to 20 MPa. We estimate the relative combined expanded uncertainty ($k = 2$) in speed of sound to be 0.011% for the present measurements. Comparisons of the measured sound speeds with values calculated from the IAPS84 formulation by Hill et al. [1] as implemented in the NIST REFPROP database version 9.1 by Lemmon et al. [2] are shown. This equation was adopted as the international standard for the properties of heavy water by the International Association for the Properties of Water and Steam, IAPWS, in 1984. Hill et al. [1] do not provide an uncertainty estimate for their equation with regard to speed of sound calculations. Nevertheless, based on comparisons to available literature data, the uncertainty in liquid phase speed of sound can be estimated to be 1.0% below and 0.5% above $T = 350$ K. Since these values are relatively large, compared to our own experimental uncertainty, we also show comparisons to a preliminary version of a newly developed equation of state for heavy water by Herrig [3]. In addition, comparisons to experimental speed of sound data from the literature are shown.

2. Experimental section

The speed of sound apparatus we used for the measurements reported here is based on the design of Meier and Kabelac [4] and was set up by Gedanitz et al. [5] for accurate speed of sound measurements over the temperature range from (253.15–353.15) K with pressures up to 30 MPa. Recent modifications of the sample manifold of the apparatus limit measurements to pressures of 20 MPa. The instrument applied the double-path length pulse-echo technique. This technique is advantageous because changes in the signal, *e.g.*, caused by time delays in the measurement electronics or in the leads, are the same for the emitted and the received signal.

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http://dx.doi.org/10.1016/j.fluid.2015.10.034
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Therefore, possible changes are compensated for to first order. In contrast to previous publications (*cf.* Gedanitz et al. [5] and Wegge et al. [6]), the present speed of sound measurements were not carried out utilizing the phase-comparison technique as introduced by Muringer et al. [7] and Kortbeek et al. [8], and modified by Meier and Kabelac [4]. Instead a single-burst method was applied, as proposed by Ball and Trusler [9], Benedetto et al. [10], and most recently by Dubberke et al. [11].

2.1. Apparatus description

The speed of sound instrument used in the present work was described in detail by Gedanitz et al. [5] and by Wegge et al. [6]. Briefly, our acoustic sensor was made up of a piezoelectric X-cut quartz crystal (*o.d.* 15 mm) installed between two polished stainless steel reflectors. The sensor was incorporated in a stainless steel pressure vessel. A glass feed-through was installed in the closure of the vessel to connect the sensor inside the vessel and the electronic devices outside. For temperature control the pressure vessel was immersed in a calibration bath thermostat (Fluke, type: 7060, USA).

The temperature of the pressure vessel was measured with a long stem 25 Ω standard platinum resistance thermometer (SPRT, Rosemount Aerospace, type: 162CE, USA) calibrated on ITS-90 and a direct current thermometry bridge (Isotech, type: TTI-2, UK). The expanded uncertainty ($k = 2$) in temperature measurement was 8 mK. Pressures were measured with vibrating-quartz-crystal-type pressure transducers (Paroscientific, USA). Depending on the pressure range, one of two transducers, with maximum pressures of 3.45 MPa and 41.4 MPa, was used. The transducers (as well as the entire pressure measurement circuit) were thermostated to $T = 318.15$ K to minimize the effects of variations in ambient temperature. We used a differential pressure indicator (Rosemount, type: 3051, USA) to separate the sample liquid in the sample manifold from the pressure transducers in the pressure measurement circuit. The expanded uncertainty ($k = 2$) in pressure measurement was 1.22 kPa for pressures below 2.50 MPa and 4.92 kPa for pressures up to 20 MPa.

To measure the speed of sound, the piezoelectric quartz transducer was electrically excited at its resonance frequency of 8 MHz by a 30-cycle sinusoidal burst, which was further modulated by a half-cycle $\sin^2$-function. This produced ultrasonic pulses, which were propagated into the fluid in both directions. The quartz crystal was used as emitter and receiver, and the sound was partially reflected at both ends of the ultrasonic sensor with the path length of $L_1 = 20$ mm in one direction and $L_2 = 30$ mm in the other direction. Hence, echoes reflected by the different reflectors arrive at the quartz crystal with a time difference Δt_{echo}. Utilizing the modified single-burst method by Dubberke et al. [11], the first two echoes were downloaded from the memory of the digital oscilloscope (Agilent Technologies, type: MSO6032A, USA) for further processing. To enhance the signal-to-noise ratio a band-pass filter based on Fast-Fourier-Transformation with a filter-width of ±20% of the quartz' resonance frequency was applied to the raw data. Zero padding in the frequency domain improved the time resolution of the echoes. Based on the processed data, the characteristic time difference Δt_{echo} between the first and second echo was computed; see Dubberke et al. [11] for a detailed description. With this measuring principle, the determination of the time difference Δt_{echo} is no longer subject to the interpretation of the experimenter as was the case for previous measurements with the present apparatus (*cf.* [5] and [6]). Therefore, the substance specific allowance for the interpretation of Δt_{echo}, which was taken into account within the uncertainty analysis by Wegge et al. [6], can be neglected and, consequently, the overall uncertainty of the measurements was reduced.

The speed of sound in the fluid was determined by

$$c = \frac{2(L_2 - \mathrm{L}_1)}{\Delta t_{\mathrm{echo}}}. \tag{1}$$

The relation above describes only ideal wave propagation; diffraction effects had to be taken into account to evaluate the time differences between the ideal and the real case. Thus, a correction was applied to the measured time difference, following the method described by Harris [12]. The correction was smaller than 0.01% for the present measurements. The difference in path length $\Delta L = (L_2 - L_1)$ is fixed during a series of measurements, except for changes due to thermal expansion and compression under pressure. Since direct methods (*i.e.*, measurement by a coordinate measuring machine) to determine ΔL did not yield reproducible results, we obtained it by calibration with purified, ordinary (light) water, which is discussed in detail elsewhere (see Ref. [6]). Briefly, we performed measurements in high-purity (light) water at ten state points at $T =$ (274.15, 278.15, 283.15, 293.15, 303.15, 313.15, 333.15, 343.15 and 353.15) K and ambient pressure. In Fig. 1 the results of our calibration measurements are shown. The relative deviations of the experimental speeds of sound from values calculated with the IAPWS95 formulation by Wagner and Pruß [13] are plotted versus temperature. This equation was adopted as international standard for the properties of water and steam for scientific and general use by the IAPWS in 1995. The uncertainty of the equation for the specific region considered here was estimated by Wagner and Pruß to be 0.005%. The deviation plot includes speed of sound data measured in the present work as well as experimental data reported by Del Grosso and Mader [14] and Fujii and Masui [15]. After calibrating the difference in path length ΔL, the relative deviations of our experimental data and of the reference data show a similar trend compared to the IAPWS95 formulation [13]. All experimental data are represented within the uncertainty of the equation of state.

2.2. Experimental procedures

Measurements were carried out along isotherms. The heavy water sample was filled into a stainless steel sample cylinder (see Section 2.3), which was connected to the sample manifold. To remove any remaining ordinary (light) water, the whole system was evacuated for several days with the set-point temperature of the bath thermostat set to the highest possible temperature (353.15 K). Moreover, the system was flushed with nitrogen several times and, subsequently, evacuated again for a duration of one day. Afterwards, the set-point temperature of the bath thermostat was set to

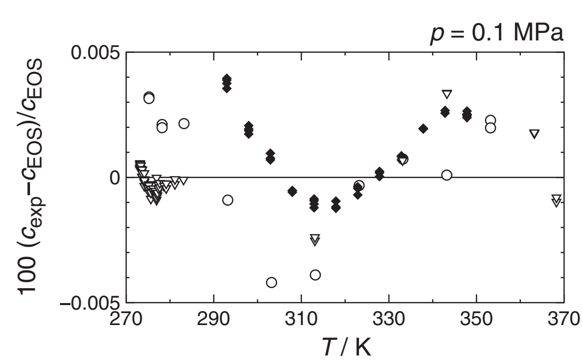

Fig. 1. Results of the calibration of the speed of sound sensor in ordinary (light) water. Relative deviations of experimental speeds of sound c_{exp} from values c_{EOS} calculated with the EOS of Wagner and Pruß [13] are plotted versus temperature T. ○, this work; ▽, Del Grosso and Mader [14]; ◆, Fujii and Masui [15]. The uncertainty of the equation of state (zero line) is ±0.005%.

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