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A Novel Acoustic Resonator for Speed of Sound Measurement in Dense Organic Vapours

Bertrand Mercier(✉), Nitish B. Chandrasekaran, and Piero Colonna

Propulsion & Power, Aerospace Engineering, Delft University of Technology, Delft, The Netherlands
{b.h.mercier,n.b.chandrasekaran,p.colonna}@tudelft.nl

Abstract. Speed of sound measurements are currently of the utmost importance for the development of thermodynamic models of fluids. A need for accurate thermodynamic models was identified for siloxanes, because of their use as working fluid in organic Rankine cycle power systems and because of the scientific interest in non ideal and non classical gas dynamics. The initiative of designing and realizing a novel acoustic resonator capable of performing speed of sound measurements in dense vapors at temperatures up to 400 °C stemmed from these considerations. Preliminary and unique experimental results are presented for siloxane D_6 (dodecamethylcyclohexasiloxane) for two isotherms at 346 °C and 348 °C and at pressures ranging form 5.5 bar to 6 bar. These measurements demonstrate for the first time the possibility of measuring the speed of sound of dense organic vapor at very high temperature. An initial comparison with speed of sound estimations provided by current thermodynamic models shows that the calculated values are approximately 6% higher.

Keywords: Siloxane · D_6 · Speed of sound · Acoustic resonator

1 Introduction

The accurate estimation of thermodynamic properties of fluids is often an essential requirement for engineering developments. The properties of most commonly used working fluids are currently formulated in terms of the Helmholtz energy if a sufficient number of reliable experimental data is available, and an outstanding precision can be achieved. Speed of sound measurements are particularly important because they can be highly accurate and are reach of thermodynamic information, as they can be used to correlate volumetric and caloric properties [8].

Innovation in organic Rankine cycle power systems technology largely depends also on the possibility of adopting novel working fluids, which better suit the various technological and non-technological requirements. Sufficiently accurate thermodynamic models are mandatory and their development depends

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on the availability of accurate measurements [1]. Furthermore, in recent years, significant steps forward were made in the niche field of non ideal compressible fluid dynamics (NICFD), all resting on the possibility of relying on accurate fluid thermodynamic models.

These applications demand for the estimation of properties at high reduced temperature and pressure in the dense vapor thermodynamic region, and experimental data in these conditions are not available, especially for complex organic molecules, given the challenges of performing measurements at high temperature.

The idea of conceiving and realizing a high-temperature resonator to measure the speed of sound of organic vapors in the dense vapor phase is motivated by these needs.

A comprehensive review of methods and rigs for speed of sound measurements commonly used to obtain data for the development of thermodynamic property models of fluids can be found in Ref. [6]. The speed of sound can be obtained either by measuring the travel time of a pressure perturbation along a known distance, or by detecting the induced resonance which builds up in cavities for a given acoustic wave number. Errors in the order of 0.01% can be achieved with both techniques [4,7,10].

Here, a speed of sound measurement apparatus designed to be operated at temperatures as high as 400 °C and pressure up to 10 bar is presented for the first time, together with its initial commissioning. Special attention is given to the design of the acoustic actuation system, which is particularly challenging at high temperature. A brief description of the operating procedures is also provided. Preliminary results related to the speed of sound of D_6 in a thermodynamic region exhibiting strong dense gas effects are also treated.

2 The Organic Vapor Acoustic Resonator (OVAR)

All types of acoustic resonators consist of a cavity filled with the fluid of interest and are fitted with at least two systems for generating and measuring acoustic waves. Here, the cavity serves the purpose of promoting a modal behaviour of the wave propagation, which is then used to determine the speed of sound from the resonant frequencies associated with the modes. For instance, considering the OVAR, which is a rectangular cuboid of length l_0 and square section of length h_0, the resonant frequencies are given as a function of the speed of sound c by

$$f_{k,m,n} = \frac{c}{2}\sqrt{\left(\frac{k}{l_0}\right)^2 + \left(\frac{m}{h_0}\right)^2 + \left(\frac{n}{h_0}\right)^2} \tag{1}$$

where k, m and n are integers corresponding respectively to the mode numbers in the longitudinal and the two radial directions. If l_0 is significantly larger than h_0, this relation can be simplified as

$$f_k = k\frac{c}{2l_0} \tag{2}$$

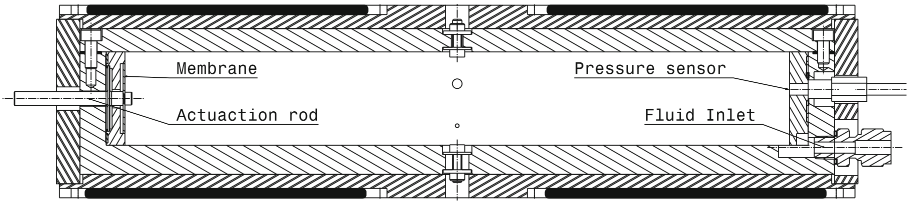

Fig. 1. CAD drawing of the longitudinal section of the resonator.

for values of k such that the frequency of the longitudinal mode is significantly smaller than that of the first radial mode. The dimensions of OVAR are $l_0 = 283$ mm and $h_0 = 40$ mm, which allows to use Eq. (1) for $k \leq 6$. The frequency $f_{7,0,0}$ is indeed only 1.2% lower than $f_{0,1,0}$, which means that both modes may overlap.

Figure 1 shows a section view of the resonator. The cavity is machined from a block of stainless steel 316L, and closed on the top by a plate, also of stainless steel. The acoustic waves are generated by a 30 mm diameter membrane (labelled in the drawing), which is driven by a piezoelectric stack (Thorlabs PK3JUAP1) by means of a rod, at a frequency up 600 Hz. A high frequency pressure sensor capable of sustaining temperatures as high as 500 °C (Kulite XTEH-10L-190SM-300PSI-A) is fitted on the opposite side of the cavity. The fluid is injected through a small circular duct, labelled as fluid inlet in Fig. 1, with the help of a volumetric hand pump. The inlet pipe is also equipped with a pressure sensor (Druk Unik 5000) which is located in an environment at ambient temperature.

Heating is provided by four electric pads, in black in Fig. 1, delivering 50 W each and mounted on a 10 mm thick aluminium shell ensuring a sufficient temperature homogeneity all along the resonator. The temperature of each pad is measured and controlled within ±0.1 °C up to 400 °C with the help of four thermocouples inserted in the aluminium shell near the center of the corresponding pad. In addition, the fluid temperature temperature is measured by a K-type thermocouple of diameter 0.7 mm that protrudes into the resonator together with a NI-9210 thermometer. Both devices were calibrated for temperatures ranging between 175 °C and 375 °C. The thermal insulation is guaranteed by a 45 mm thick casing of ceramic wool (not shown in Fig. 1).

3 Application

3.1 Experimental Procedure

The resonator is operated by setting the thermodynamic state of the fluid along isotherms. The first step consists in heating the apparatus up to the set point, while vacuum is maintained in the cavity. The fluid is then injected using the hand pump. Once all the fluid is vaporised inside the cavity, a sequence is run to determine the frequency response of the resonator in order to calculated the

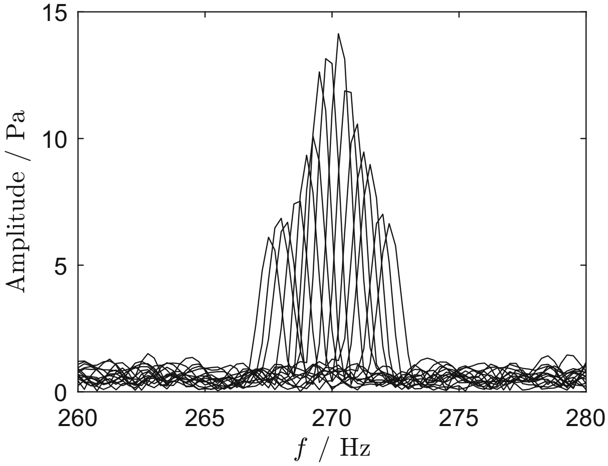

Fig. 2. Mode $k = 2$ acoustic resonance.

speed of sound by means of Eq. (2). The membrane is excited with a sinusoidal motion at a given frequency, while the acoustic pressure at the opposite end of the cavity is simultaneously recorded by the high frequency pressure sensor. This operation is repeated by shifting the excitation frequency across the frequency range that contains the resonance frequency of either mode $k = 2$ or $k = 3$. After a sequence, the fluid density is changed by injecting or remove some fluid.

For all measurement points, the fluid temperature inside the cavity is measured together with the pressure inside the inlet pipe. The density is determined by estimating the amount of fluid injected from the hand pump into the cavity, given that the volume V occupied by the fluid is known. Therefore, for any measurement point, the temperature, density, pressure and speed of sound are available.

3.2 Post Processing Procedure

The resonance frequency of the cavity is determined by post processing the series of acoustic signals recorded for all individual test points with varying excitation frequencies. The Fourier transform of each signal is computed to determine the amplitude of the acoustic signal at the prescribed frequency. Figure 2 depicts the pressure amplitude vs frequency recorded for an exemplary measurement point. In this example, the fluid was D_6 at a temperature T = 346 °C and a pressure of 5 bar. The excitation frequency was varied from 267 Hz to 272 Hz with steps of 0.33 Hz. A well defined peak is observed in the frequency response of the resonator, which indicates that a resonance has been captured. The resonance frequency f_0 is determined by fitting the top part of the peak with a Lorentzian function. The speed of sound is then deduced from this frequency using Eq. (2),

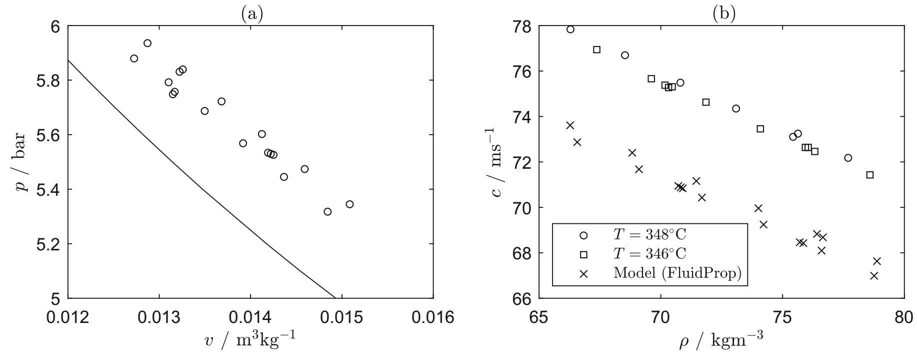

Fig. 3. Measurement in the Pv thermodynamic plane (a), and corresponding speed of sound (b).

Table 1. Measurements uncertainty.

Property	Uncertainty	Device
Temperature	$\pm 0.5\,\mathrm{K}$	k-type Thermocouple + NI–9210
Pressure	± 4 mbar + 0.1%	Druk UNIK 5000–16 bars
Density	$\pm 0.44\,\mathrm{kg/m^3}$	Custom hand pump
Speed of sound	$\pm 0.3\%$	Custom resonator

as

$$c = \frac{2lf_0}{k}, \tag{3}$$

where l is the length of the cavity corrected for the thermal expansion using $l = l_0(1+\alpha T)$, $\alpha \simeq 17^{-6}\ \mathrm{K}^{-1}$ being the thermal expansion coefficient of stainless steel 316L.

3.3 Uncertainty Analysis

Speed of sound values are measured with acoustic resonators with the aim of assessing and improving thermodynamic models. It is thus of major importance to estimate the uncertainties associated with the state variables and with the speed of sound. Currently, no calibration of pressure and temperature sensors have been performed yet, therefore pressure and temperature uncertainty are estimated from the specs of the sensors and related data acquisition system: values are summarised in Table 1. The density is set by means of a hand pump of volume displacement $v_d = 1.060\ \mathrm{cm}^3$ per turn with a resolution of the order of 0.1 turn. This volume is converted into the value of the injected mass under the assumption that the density is accurately known. The density of the liquid in the pump is estimated as the density at 20 °C corrected for the temperature with the derivatives measured for D_5 [9]. At ambient conditions, the error introduced by the resolution is 0.1 g for a liquid density of $\rho = 960$ kg/m^3. In addition, the precise location of the transition from liquid to vapour in the inlet pipe is not known. This adds another 0.1 g of uncertainty to the estimation. After dividing these masses by the volume of the resonator $V = 455$ cm^3, the overall uncertainty for the density is ± 0.44 kg/m^3.

Typical errors affecting speed of sound measurements obtained with a resonator are extensively studied in the literature [3,5]. Among those biases, the most relevant for the OVAR setup arise from neglecting the influence of the fluid viscosity and should be smaller than 0.1%. Another type of error may be attributed to the deviation of the cavity shape form an ideal rectangular box. A finite element analysis of the modal behaviour of the cavity was carried out to provide further insights in this respect. The maximum deviation due to this error affecting the estimation derived from Eq. (3) was found equal to 0.29% for mode $k = 2$. This is a systematic error, which is compensated in the post processing of the measurement data. However, it is safe to use this figure as the speed of sound uncertainty to account for possible other shape imperfections.

3.4 Results

The first preliminary experimental campaign involved measurements of D_6 speeds of sound along the two isotherms at 346 °C and 348 °C, at densities ranging between $\rho = 66$ kg/m^3 and $\rho = 79$ kg/m^3. Figure 3(a) reports measurement points the Pv diagram of D_6. The thermodynamic states of these measurements were chosen such that they are close to the dew line and in a thermodynamic region exhibiting strong dense gas effects, as testified by the local value of the compressibility factor $Z \simeq 0.67$. Figure 3(b) shows the comparison between speed of sound measurements values computed with the multiparameter equation of state model documented in Ref. [2]. The comparison shows a very good agreement in terms of trends, however the model seems to underestimate the speed of sound by approximately 6%. This disagreement is much larger than the values of uncertainties reported in Table 1, therefore the possibility of using the OVAR to refine thermodynamic models of organic fluids is fully demonstrated by this first measurement campaign.

4 Conclusion

A novel resonator for high temperature speed of sound measurements in dense organic vapors was conceived, designed, realized and successfully tested in laboratories of the Propulsion and Power group at Delft University of Technology. Preliminary speed of sound measurements were performed in siloxane D_6 vapour in highly non ideal conditions with an estimated accuracy of ± 0.3%. This set of measurements provides a unique database which is compared with the predictions of what is arguably the best thermodynamic model available for this fluid. Significant discrepancies are observed for the thermodynamic conditions of interest. Further speed of sound measurements will be performed and improvement to the instrument and method implemented in order to provide a sufficient number of fully characterized experimental data aiming at the improvement of thermodynamic models of D_6 and possibly of the accuracy of the estimation of its fundamental derivative of gas dynamics in the region in which it is negative or close to zero. It is foreseeable that the OVAR will also be used with other fluids of interest in organic Rankine cycle technology research and non ideal compressible fluid dynamics.

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