MĀPAN-Journal of Metrology Society of India (March 2020) 35(1):19–24
https://doi.org/10.1007/s12647-019-00331-x

ORIGINAL PAPER

Accuracy Estimation of Propagation Velocity in Variable Path Ultrasonic Interferometer for Liquids

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Received: 15 April 2019 / Accepted: 09 July 2019 / Published online: 30 July 2019

Abstract: In this article, we describe the effects and the degree to which they cause error in the measurement of propagation velocity. Various effects include nonlinearity in path measurement, temperature stability and number of maxima or minima consideration. Double-distilled water was used as a sample to estimate various effects. Finally, it has been concluded that the variable frequency approach may be preferred for better accuracy. It has also been observed that the digital frequency selection is more precise than mechanical distance variation.

Keywords: Interferometer; Measurement error; Ultrasonic velocity

1. Introduction

Two ultrasonic interferometric techniques exist for the estimation of propagation velocity in liquids. The first is variable path and other is variable frequency. Variable path ultrasonic interferometer is widely used as a low cost tool for estimation of ultrasonic propagation velocity. It utilizes the superposition of ultrasonic waves by varying ultrasonic path of propagating waves. Standing waves are formed due to interference of waves, and the wavelength corresponding to these standing waves inside liquid is measured for the estimation of velocity. One of the critical aspects in the variable path interferometer is the degree of accuracy of wavelength measurement. The accuracy is also affected by the accuracy of the frequency of excitation source. The ultrasonic waves in the frequency range 0.5 MHz to 100 MHz are important for velocity measurement and liquid characterization purpose [1, 2]. The major advantage of ultrasound velocity measurement is that most chemical phenomenon, reaction and physical properties of materials affect ultrasonic propagation parameters [3–5]. The ultrasound velocity is used for calculating other thermodynamic properties. The ultrasonic propagation velocity is important for the calculation of the physio-chemical and thermodynamic properties of liquids, fluids and polymers [6]. To estimate these properties accurately and precisely, accurate measurement of propagation velocity is needed [7–9]. The propagation velocity is used to study density, viscosity, Gibb's free energy and deviation in thermo-acoustic parameter of liquids. The propagation velocity is also useful for the study of intermolecular interaction of binary mixture of liquids [10–12]. The binary organic mixture solutions find applications in chemical, food and drug industry. In biomolecular investigations, small changes in velocity play vital role [13–18].

The ultrasonic variable path interferometer is extensively used instrument for velocity measurement. The standing waves are generated in liquid due to interference of propagating waves. By changing the path length of ultrasound, corresponding wavelength of the standing wave is measured. In the other interferometric method, the path of ultrasound is kept constant and the frequency of the radio frequency (RF) source is changed so that maxima and minima are detected [19]. With the advent of direct digital synthesis (DDS) technology, it is possible to vary the ultrasound source frequency at small steps (Hz) to precisely change the wavelength in the sample [20]. The variation in the radio frequency is generally preferred within the 3 dB bandwidth of the ultrasonic transducers.

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In this article, various effects contributing the significant errors in the measurement of velocity have been estimated using double-distilled water as sample. The various effects, such as effect of deviation in the resonant frequency of resistance capacitance (RC) circuit-based RF generator, deviation in temperature of ultrasonic cell, effect of hysteresis and backlash in variable path, nonlinearity in the micrometer screw gauge distance measurement and effect of number of nodes and antinodes under consideration, are described in detail. All effects in velocity measurement have been considered for its amount of contribution in error or precision. The double-distilled water was used as experimental liquid in ultrasonic measurement, and obtained results are compared with the literature [21].

2. Ultrasonic Propagation Velocity: Principle and Method

A piezoelectric transducer, when excited with external radio frequency source, generates continuous ultrasonic waves. The piezoelectric transducer vibrates at the RF frequency, and ultrasonic waves are generated in the liquid in contact with transducer. The ultrasonic waves in the medium of propagation are related with the equation:

$$c = f \cdot \lambda \tag{1}$$

where c is the propagation velocity of sound (ms^{-1}), f is the frequency (Hz) and λ is the wavelength (m).

The ultrasonic interferometers may be implemented by one of the following two methods:

2.1. Fixed Frequency Variable Path Interferometer

The continuous RF excitation is applied to the transducer at one end and movable reflector parallel to transducer kept at the other end. The reflector movement and measurement is carried out using a micrometer screw gauge. During micrometer screw gauge movement, the maxima or minima of current meter are detected [22]. The separation d between adjacent nodes or antinodes will be half-wavelength. So, the ultrasonic propagation velocity in the sample is given by:

$$c = 2d \cdot f \tag{2}$$

For better estimation of wavelength, more number of nodes or antinodes are considered and average is preferred.

2.2. Fixed-Path Variable Frequency Interferometer

In this technique, the separation between the transducer and reflector is fixed and the frequency is swept within bandwidth of the ultrasonic transducer. Due to interference of ultrasound, the position of nodes and antinodes get shifted at different frequencies. The frequency difference between two consecutive resonance peaks Δf is proportional to ultrasonic velocity and is related by [19]:

$$c = 2\Delta f \cdot D \tag{3}$$

where D is the separation between transducer and reflector.

3. Experimental Setup

The ultrasonic propagation velocity measurement was carried out in double-distilled water. The temperature of liquid sample was controlled and maintained between 5 and 35 °C with stability of better than ± 0.1 °C and step resolution of 0.01 °C. The ultrasonic interferometer excitation and detection device was used to generate the desired frequency of excitation. Frequency of 2 MHz, 6 MHz and 10 MHz was also utilized in order to study the behavior at harmonics. Figure 1 shows the simplified block diagram of variable path ultrasonic interferometer (PICO model: BL02). The basic design includes the piezoelectric disk mounted at the base of cylindrical double-walled sample holder. The reflector is used at opposite end and moves in parallel to the transducer. The water is circulated though the outer jacket of sample holder to maintain the desired temperature of the sample by using temperature controller (Julabo F12-ME).

4. Results and Discussion

Various factors that affect the ultrasonic propagation velocity measurement and the degree of their influence have been studied and analyzed:

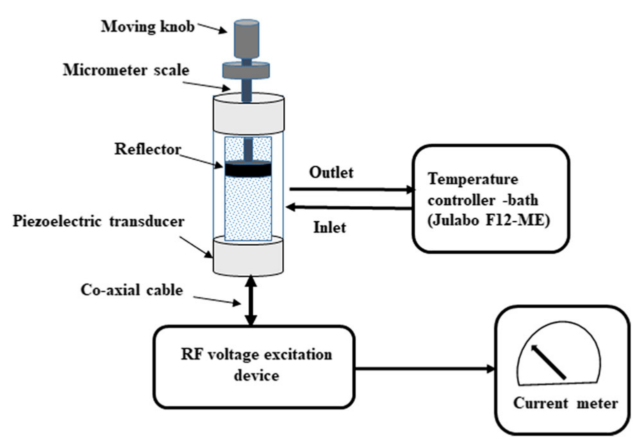

Fig. 1 Block diagram of an ultrasonic interferometer with temperature controller to maintain the sample temperature within ± 0.1 °C

4.1. Effect of Deviation in the Resonant Frequency of RC Circuit-Based RF Generator

The oscillator used to excite the piezoelectric disk in the simplest form may use resistor and capacitor feedback circuit to define frequency. In continuous wave excitation, transducer vibrates at the excitation frequency. Whereas, in broadband excitation it vibrates at their resonant frequency. The tuning of the RC may deviate from that of the resonance of the transducer and the resonance frequency if considered for estimation of velocity, it will cause error [23]. For instance, commercially available RC oscillator frequency may deviate maximum 2% as specified in the literature [24]. Therefore, for 2 MHz oscillator excitation corresponds to 40 kHz deviation in frequency. For half-wavelength in water at 25 °C corresponding shift in wavelength becomes ± 14.66 µm and propagation velocity shift to 1496.727 ± 29.38 ms^{-1}. Also due to temperature, there is variation in RC oscillator frequency. The temperature stability of ± 40 ppm/ °C (± 80 Hz) in RC oscillator contributes deviation of 0.05986 ms^{-1} $°C^{-1}$ in velocity. For estimation of this effect, room temperature (27 °C) was used as reference temperature at which it is considered that there is no deviation in excitation frequency. Figure 2 shows estimation of deviation in propagation velocity with frequency deviation due to temperature variation from 5 to 45 °C. When the frequency becomes major concern in measurement of propagation velocity, the excitation frequency may be either calibrated or known up to higher degree of the accuracy.

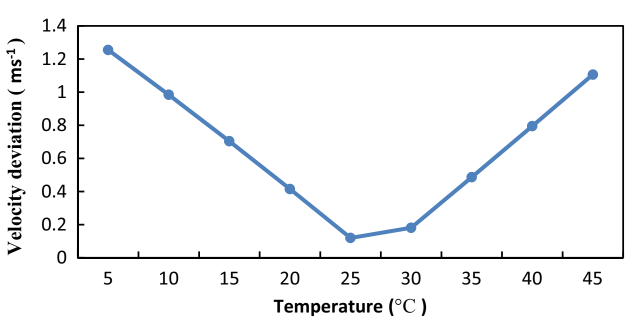

Fig. 2 Deviation in ultrasonic propagation velocity due to frequency drift of oscillator with temperature range 5 °C to 45 °C

4.2. Effect of Number of Maxima and Minima Consideration

Number of half-wavelengths (n) detected for the estimation of propagation velocity largely affect the measurement. The precision of wavelength detection is confined by ratio of the resolution of micrometer scale and number of wavelength counts [25]. For example for $n = 50$, using 10 µm screw gauge resolution, the precision of wavelength detection becomes 0.2 µm. This in turn measures the propagation velocity with ± 0.8 ms^{-1}. In order to study the effect of the number of maxima or minima considered, entire experiment was performed at fixed temperature 25 °C. The ultrasonic propagation velocity was measured for different n counts. The experiment was repeated for three different frequencies, and each experiment was repeated for 10 times for estimation of standard deviation. Table 1 shows the effect of n counts on the standard deviation in propagation velocity measurement at various frequencies. From Table 1, it is obvious that the wavelength detection is limited by resolution of micrometer. Using micrometer with better resolution improves measurement. It is observed that more precise measurement is obtained for higher value of n. The experimental results reveal that higher n counts must be preferred at relatively higher frequencies due to its associated lower wavelengths. Figure 3 prominently indicates the dependence of standard deviation in the velocity measurement as a function of n. Higher n values give more precise value of velocity. The estimated precision at 2 MHz is 1498.04 ± 0.13 ms^{-1}.

Table 1 The propagation velocity with standard deviation measured for different n values

Counts 'n'	Temp = 25 °C					
	Propagation velocity (ms^{-1})			Standard deviation		
Frequency	2 MHz	6 MHz	10 MHz	2 MHz	6 MHz	10 MHz
1	1504.00	1533.60	1468.00	21.9089	5.366563	64.18723
10	1497.60	1498.08	1496.00	2.019901	2.762607	6.164414
20	1498.48	1498.00	1496.80	0.867179	2.629068	2.167948
30	1497.39	1498.68	1498.13	0.851555	3.464102	1.095384
40	1497.86	1498.26	1497.70	0.487852	1.59311	1.151086
50	1498.04	1498.54	1497.36	0.420793	1.596496	1.374045
60	1498.33	1498.00	1496.99	0.385093	1.632993	0.942575

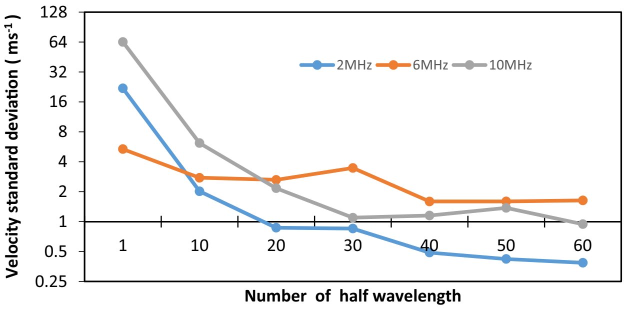

Fig. 3 Standard deviation in propagation velocity for different number of half-wavelength measurement at temperature 25 °C

4.3. Effect of Deviation of Sample Temperature from Temperature Bath

The ultrasonic liquid cell temperature stability and accuracy also contributes in the measurement of propagation velocity. The ultrasonic cell temperature fluctuation plays a key role to decide the precision of velocity measurement. Usually, it is considered that temperature coefficient of propagation velocity is linear in liquids, however, practically resulting in nonlinear variation [13]. Generally, the velocity measurement with low-volume liquid having provision of adjustable distance is thermally unstable due to temperature difference in cell and temperature controller [7]. From the literature, it has been observed that the temperature deviation of ultrasonic cell increases from the set controller temperature when measured at elevated temperature from the ambient temperature. This is particularly due to external effects of environment, which tries to pull the temperature of cell to the ambient. This effect becomes more prominent as the difference in temperature increases. This temperature difference introduces an error in propagation velocity.

For the estimation of such effect in the propagation velocity, measurements have been carried out at $n = 50$ with excitation frequency of 2 MHz. Figure 4a shows the effect of temperature difference on ultrasonic velocity measurement. From Fig. 4a, it clearly indicates that the measured ultrasonic velocity deviation from the literature became larger at lower temperatures from ambient temperature 27 °C. Figure 4b depicts the deviations in measured propagation velocity quantitatively. It was observed that maximum deviation of 4.6 ms^{-1} occurred at 5 °C which is indicative of temperature difference of 1 °C between sample and set temperature.

Moreover, high-voltage (more than 100 V) excitation applied to ultrasonic transducer may produce excessive heat in the liquid and liquid property may change which effectively cause error in velocity measurement [22].

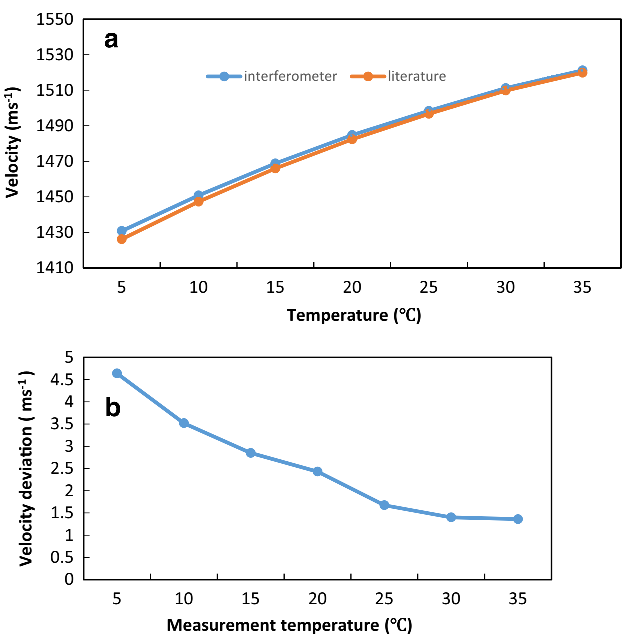

Fig. 4 **a** Ultrasonic prorogation velocity in water at different temperatures. **b** Deviation of measured ultrasonic propagation velocity in water with reference to the literature at various temperatures

4.4. Effect of Nonlinearity in Distance Measurement

In order to change the distance in ultrasonic interferometer, a micrometer screw gauge is commonly used. It is highly expected that the pitch of micrometer screw gauge is linear. However, the scale of micrometer may include significant nonlinearity. The nonlinearity becomes more vital if the measurement is being carried out at relatively higher frequencies. For instance, at 6 MHz and 25 °C temperature, half-wavelength becomes 124.7 μm. The nonlinearity of the order of few tens of microns contributes significantly in the error in ultrasonic velocity measurement. In order to study the nonlinearity in the distance measurement, care was taken to avoid backlash error and the screw gauge was moved with uniform step in forward and backward

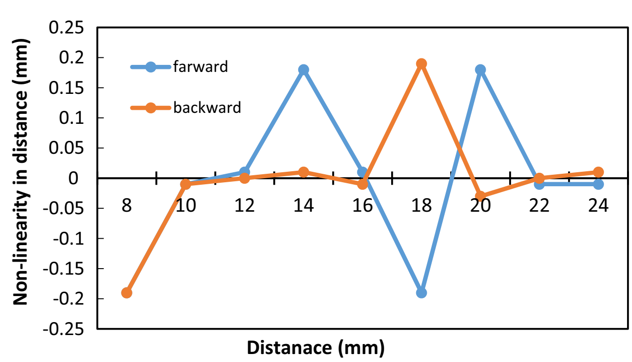

Fig. 5 Nonlinearity effect associated with ultrasonic path measurement at 25 °C

direction. The same distance was compared with a well-established pulse-echo method (Olympus, EPOCH 1000) having measurement resolution of 10 µm. Figure 5 depicts the nonlinearity effect present in the screw gauge-based measurement setup in our laboratory at 25 °C. In case, if the number of half-wavelengths was considered 50 at 10 MHz nonlinearity of ± 190 µm affects the measurement dramatically as half-wave is only 74.8 µm. In this case, the $\lambda/2$ estimation will deviate more than 5% even by adopting best-preferred value of n in the setup.

4.5. Effect of Hysteresis and Backlash Error in Reflector Movement

The distance movement system along with the nonlinearity may also include significant hysteresis with backlash. The backlash may cause undesirable problems in wavelength detection and precise positioning. The backlash error is introduced due to manufacturing errors and friction forces. The scale of micrometer does not fix at exactly same position during reverse direction and produce backlash in path length [26]. Figure 6 shows the effect of backlash in distance measurement. For small value of distance, percentage of backlash error is more if offset error is order of backlash error. It affects the accuracy of distance measurement. The wrong distance measurement due to hysteresis ultimately results in inaccurate value of propagation velocity. For the estimation of hysteresis associated with the movement system within 2 mm to 24 mm, measurements were carried out in both forward and backward direction and were compared with well-established pulse-echo approach. During measurements of total 22 mm, distance was measured as 22.43 mm and 22.51 mm. The measured backlash error in the total distance movement of the 22 mm was found ± 0.170 mm and corresponding error in velocity propagation was 1496.727 ± 13.6 ms^{-1}.

In order to estimate the overall contribution of various effects in the measurement of ultrasonic velocity in interferometer with variable path, all the above affecting parameters were considered. Table 2 shows the sources of the errors in the propagation velocity of water with their contribution. The individual parameters contributing in the propagation velocity is estimated. The type of uncertainty is mentioned. It is obvious from Table 2 that the frequency deviation of the excitation signal plays vital role in estimation of ultrasonic propagation velocity. The propagation velocity measurement may be greatly improved with precisely known (calibrated) source or by using the crystal oscillator. Although the overall uncertainty estimated is 2.422%, apart from frequency accuracy distance measurement also greatly influence the propagation velocity [27]. The precision of the measurement is largely improved by considering more number of half-wavelengths, which is clearly indicated in Table 2.

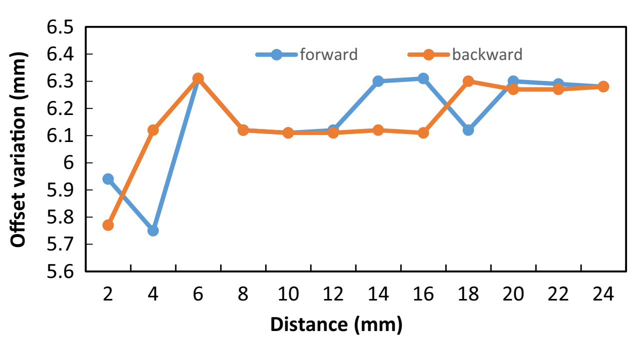

Fig. 6 Offset variation with change in ultrasonic path measurement

5. Conclusions

The ultrasonic interferometer is widely used for the study of physical and biochemical organic liquids. Therefore, the accurate and precise velocity estimation in ultrasonic interferometer is required. It clear that once the frequency of the excitation, detection and the separation between the transducer and reflector is known to high degree of accuracy, overall measurement of ultrasonic velocity improves drastically [28]. From Eq. (3), it may be inferred that fixed-path variable frequency interferometer system may prove to be relatively more accurate over the variable distance as

Table 2 Accuracy due to errors in the propagation velocity of water at 2 MHz and temperature (25 °C)

S. no.	Affecting parameters	Wavelength deviation (µm)	Wave velocity (ms^{-1})	Uncertainty (%)	Uncertainty type
1.	Deviation in the resonant frequency of RC circuit (Max 2%)	±14.660	1496.727 ± 29.38	2.0	B
2.	Deviation of ultrasonic cell temperature	±0.509	1498.4 ± 1.637	0.109	B
4.	Backlash in distance (n = 50)	±3.400	1496.727 ± 13.6	0.908	B
5.	Nonlinearity in distance (n = 50)	±3.800	1496.727 ± 15.2	1.015	B
6.	For n = 50, counts	±0.0319	1498.04 ± 0.133	0.008	A
7.			Total uncertainty	2.422	

the distance kept constant and frequency may be controlled more precisely and accurately. With the advent of direct digital synthesis (DDS) technology-based signal generator, it is possible to have flexibility to adjust the frequency with 1 Hz step with an accuracy of about 50 ppm levels [20].

Acknowledgements The authors would like to thank the Director, CSIR-National physical laboratory, for providing the necessary facilities to carry out above work. The authors (Sahil Sharma) also thank to University Grant Commission (UGC) New Delhi, India, for providing the Research fellowships.

References

[1] F. Eggers and T. Funck, Ultrasonic measurements with milliliter liquid samples in the 0.5–100 MHz range, Rev. Sci. Instrum., **44** (1973) 969–977.

[2] S. Rajagopalan, S. Sharma and P. Dubey, Measurement of ultrasonic velocity with improved accuracy in pulse echo setup, Rev. Sci. Instrum., **78** (2007) 085104.

[3] U. Kaatze, T. O. Hushcha and F. Eggers, Ultrasonic broadband spectrometry of liquids a research tool in pure and applied chemistry and chemical physics, J. Solut. Chem., **29** (2000) 299–368.

[4] S. Yadav, A. Zafer, A. Kumar, N. Sharma and D. Aswal, Role of national pressure and vacuum metrology in indian industrial growth and their global metrological equivalence, MAPAN, **33** (2018) 347–359.

[5] A. Kumar, V. N. Thakur, A. Zafer, N. Sharma, S. Yadav and D. Aswal, Contributions of national standards on the growth of barometric pressure and vacuum industries, MAPAN, **34** (2019) 13–17.

[6] I. Perepechko, Low-temperature properties of polymers, First edition Pergamon, London (1980).

[7] A. Sarvazyan, Development of methods of precise ultrasonic measurements in small volumes of liquids, Ultrasonics, **20** (1982) 151–154.

[8] M. I. Aralaguppi, T. M. Aminabhavi, R. H. Balundgi and S. S. Joshi, Thermodynamic interactions in mixtures of bromoform with hydrocarbons, J. Phys. Chem., **95** (1991) 5299–5308.

[9] B. Marwein and S. Bhat, Thermodynamic study of molecular interactions in ternary liquid systems, Thermochim. Acta, **118** (1987) 277–285.

[10] M. K. Praharaj, A. Satapathy, P. Mishra and S. Mishra, Ultrasonic studies of ternary liquid mixtures of NN-dimethylformamide, nitrobenzene, and cyclohexane at different frequencies at 318 K, J. Theor. Appl. Phys., **7** (2013) 23.

[11] S. Fakruddin, M. Pushpalatha, C. Srinivasu and K. Narendra, Excess thermo-acoustical parameters in binary liquid mixture containing n-butanol at different temperatures, Karbala Int. J. Mod. Sci., **1**, (2015) 97–100.

[12] S. F. Babavali, P. Shakira, C. Srinivasu and K. Narendra, Comparative study of theoretical ultrasonic velocities of binary liquid mixtures containing quinoline and mesitylene at temperatures T = (303.15, 308.15, 313.15 and 318.15) K, Karbala Int. J. Mod. Sci., **1** (2015) 172–177.

[13] R. Lagemann, D. McMillan Jr. and W. Woolf, Temperature variation of ultrasonic velocity in liquids, J. Chem. Phys., **17** (1949) 369–373.

[14] S. Parveen, D. Shukla, S. Singh, K. Singh, M. Gupta and J. Shukla, Ultrasonic velocity, density, viscosity and their excess parameters of the binary mixtures of tetrahydrofuran with methanol and o-cresol at varying temperatures, Appl. Acoust., **70** (2009) 507–513.

[15] R. Piccirelli and T. Litovitz, Ultrasonic shear and compressional relaxation in liquid glycerol, J. Acoust. Soc. Am., **29** (1957) 1009–1020.

[16] V. Mohammadi, M. Ghasemi-Varnamkhasti, R. Ebrahimi and M. Abbasvali, Ultrasonic techniques for the milk production industry, Measurement, **58** (2014) 93–102.

[17] T. M. Aminabhavi, S. K. Raikar and R. H. Balundgi, Volumetric, acoustic, optical, and viscometric properties of binary mixtures of 2-methoxyethanol with aliphatic alcohols (C1-C8), Ind. Eng. Chem. Res., **32** (1993) 931–936.

[18] T. M. Aminabhavi and S. K. Raikar, Thermodynamic interactions in binary mixtures of 2-methoxyethanol with alkyl and aryl esters at 298.15, 303.15 and 308.15 K, Collect. Czechoslov. Chem. Commun., **58** (1993) 1761–1776.

[19] D. N. Sinha and G. Kaduchak, Noninvasive determination of sound speed and attenuation in liquids, Exp. Methods Phys. Sci., **39** (2001) 307–333.

[20] S. Sharma, U. K. Mishra, S. Yadav and P. K. Dubey, 2019, Improved ultrasonic interferometer technique for propagation velocity and attenuation measurement in liquids, Rev. Sci. Instrum., **90**, (2019) 045107.

[21] W. Marczak, Water as a standard in the measurements of speed of sound in liquids, J. Acoust. Soc. Am., **102** (1997) 2776–2779.

[22] V. N. Bindal and A. K. Kansal, An Ultrasonic interferometer, (1975) Indian patent No. 136940.

[23] For oscillator, Microchip Technology Inc, DS31002A, (1997) 2, 1–20.

[24] For, Analog Devices, LTC1799 datasheet, resistor set SOT-23 oscillator.

[25] N. Bobroff, Recent advances in displacement measuring interferometry, Meas. Sci. Technol., **4** (1993) 907.

[26] P. Dubey and S. Singh, High resolution vertical movement system for transducer and target separation in primary ultrasonic power measurement setup, Measurement, **76** (2015) 201–208.

[27] V. R. Meyer, Measurement uncertainty, J. Chromatogr. A, **1158** (2007) 15–24.

[28] P. K. Dubey and S. Sharma, Improved ultrasonic interferometer excitation and detection device for velocity and attenuation, Measurement (2017) Indian patent filed No. 201711036499.