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Implementation of 3ω Method for a Wide Range Measurements of Thermal Conductivity of Liquids

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Abstract

Thermal conductivity is an important thermophysical property of materials. The advancements in material sciences and manufacturing techniques have led to the development of various techniques for thermal conductivity measurements. Among these methods, the AC hot wire (3ω) method is a prominent option due to its advantages such as insensitivity to black body radiation and minimal sample requirement. Even though this measurement method has been quite popular in thermal conductivity measurements of thin films for several decades and is gathering attention for the measurements of liquids, there are no studies where the hardware of the measurement methodology is depicted as the method is quite complicated. In this study, a custom-made 3ω setup is presented and its performance in a wide range of thermal conductivity measurements is evaluated by investigating the thermal conductivity of base fluids such as water, ethanol, methanol, ethylene glycol (EG), and mineral oil. In addition, the relative thermal conductivity of the base fluids is compared with the literature. The proposed setup shows that the maximum error rates were obtained as in a range of 0.5 to 5% depending on the base fluid.

Keywords: 3ω method; hot wire method; relative thermal conductivity.

1. Introduction

Thermal conductivity is an important thermophysical property of the materials. The advancements in material sciences and manufacturing techniques have led to development of various techniques for thermal conductivity measurements. Among these methods, AC hot wire (3ω) method is a prominent option due to its advantages such as, insensitivity to black body radiation and minimal sample requirement (Cahill, 1990).

In 3ω method, a metal wire, which is used as both a heat source and a thermal sensor, is inserted into the sample. Then, an alternating current is applied at the frequency of ω which results in heat dissipation with 2ω frequency. This periodical input causes a temperature distribution at the same frequency with a phase difference as an output. Due to the temperature dependence resistivity of metals, an oscillating resistance deviation is added to the resistance of the hot wire. Eventually, an observable voltage drop at 3ω is obtained on sensor terminals. The thermal conductivity of the sample can be obtained by using the amplitude and frequency relationship of 3ω harmonic (Turgut et al., 2008) (Figure 1).

3ω thermal conductivity measurement method has been popular for several decades for the thermal characterization of solids and thin films (Su et al, 2016). Only a few attempts have been made for measurement of fluids in the last two decades (Hoffmann et al., 2016; Karthik et al., 2012; Oh et al., 2008; Turgut et al., 2008; Wang et al., 2007; Wu et al., 2009). Although the reliability of 3ω method is proved experimentally by several research groups, any test device that implements the method is not commercially available.

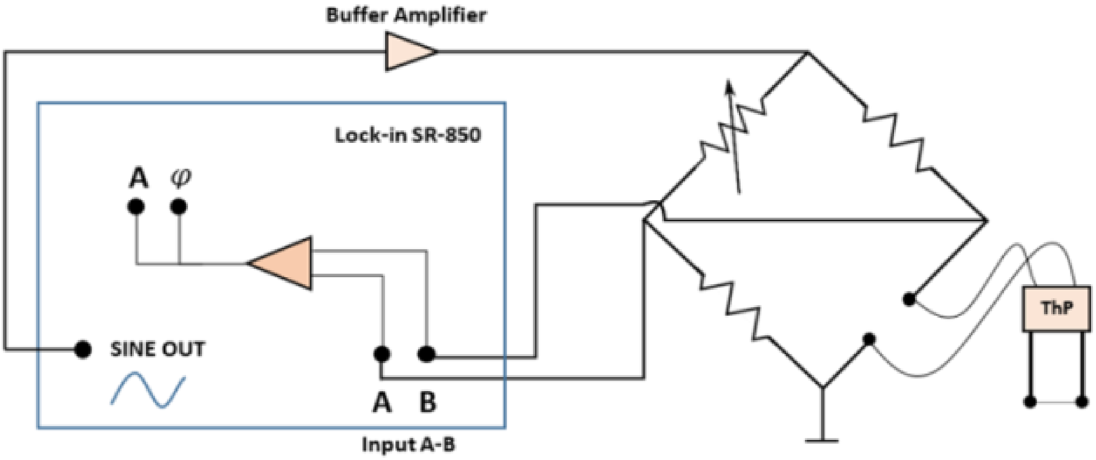

Figure 1. 3ω measurement setup (Turgut et al., 2008)

In this paper, a custom-made electronic hardware for 3ω measurements is presented. The performance of the developed system is investigated in a wide range thermal conductivity measurements of base fluids. The wide range measurement in this study refers to the thermal conductivity change between water (~0.6 w/mK) and mineral oil (~0.1 W/mK) which calculated as 5 times. The compatible results with previous measurements in the literature for mineral oil (Koruk et al., 2017), water (Çengel & Ghajar, 2014), in addition ethanol, methanol and ethylene glycol samples (Lide, 2004) promise possible use of the developed setup.

2. The 3ω Method

The theoretical background of the 3ω method is briefly explained as they are previously explained in more detail by Turgut et al. (2008) and Chirtoc & Henry (2008). The temperature amplitude $T(2\omega,\phi)$ is given in Eq. (1).

$$T(2\omega,\phi) = \frac{\dot{q}}{2\pi k_s l}\left(ln\frac{\mu_s}{1.2594r} - i\frac{\pi}{4}\right) \tag{1}$$

where ϕ is the phase angle (radian), $\dot{q}$ is the rate of heat transfer (W), k_s is the thermal conductivity of sample (W/mK), l is half of the length of the wire (m), μ_s is the thermal diffusion length (m), and r is the radius of the wire (m). The second harmonic is obtained by the rate of the heat transfer amplitude. The dimensionless impedance F factor is presented in Eq. (2) (Turgut et al., 2008).

$$F(2\omega) = \frac{Z_s}{Z_p} = \frac{z_s/2\pi rl}{l/(\pi r^2 k_p)} = \frac{k_p r}{2l^2} z_s(2\omega) \tag{2}$$

where z_s (m^2K/W) is the specific thermal impedance of the interface, k_p is the thermal conductivity of probe (W/mK), and Z_p is the thermal resistance of the half-length wire in the axial direction, considering the end supports as an infinite heat sink.

The term $V(3\omega, \phi)$ depending on the 3ω is generated by the mixing of excitation current at ω with the resistance change at 2ω and it is given in Eq. (3);

$$V(3\omega, \phi) = \frac{I_0 R_0}{2} r_{el} T(2\omega, \phi) \tag{3}$$

Where I_0 is the current through the wire (A), R_0 is the resistance of wire (Ω), and r_{el} is the temperature coefficient of the resistance (1/K).

With Eq. (1), Eq. (2), Eq. (3) is transformed into Eq. (4);

$$V(3\omega, \phi) = \left(\frac{Il}{\pi r^2}\right)^3 C_M F(2\omega) \tag{4}$$

where I is the current (A) and C_M is the figure of merit. To determinate the relative thermal conductivity, the imaginary parts of Eq. (5) are proportioned for water and the sample. The proportion of the imaginary parts is presented in Eq. (6);

$$V(3\omega, \phi) = \mathrm{Re}(3\omega, \phi) + i\mathrm{Im}(3\omega, \phi) \tag{5}$$

$$\frac{k_s}{k_w} = \frac{\mathrm{Im}[V_w(3\omega, \phi)]}{\mathrm{Im}[V_s(3\omega, \phi)]} \tag{6}$$

where k_w is the thermal conductivity of water (W/mK).

The relationship between the theoretical results and the measurements in the 3ω method is also given in Figure 2 as a block diagram. The developed embedded hardware was designed as three components that are the main controller and signal generator, signal conditioning and lock-in amplifier stages. The components are shown in Figure 3 for performing the measurement of the thermal conductivity of fluids.

Two sinusoidal signals are generated which are synchronous with each other in the setup. One of the generated sinusoidal signals is to excite the probe and the other one is the reference signal needed to perform the measurement. The key principle of measurement method is measuring of 3rd harmonic of modulated sinusoidal wave which is effected with relation between heating wire and excited liquid. In this way, sinusoidal wave at 3f frequency is generated as a measurement reference signal. Theoretical and experimental studies have determined that nickel wire probes provide the most appropriate signal (0.5 V amplitude and 0.5-2 Hz frequency range) for measurement (Turgut et al., 2008). In this regards, 1 Hz sinusoidal signals for exciting the probe and 3 Hz sinusoidal signals for the reference are generated in this unit. A 32-bit microcontroller with 12-bit DAC resolution is used to perform this operation.

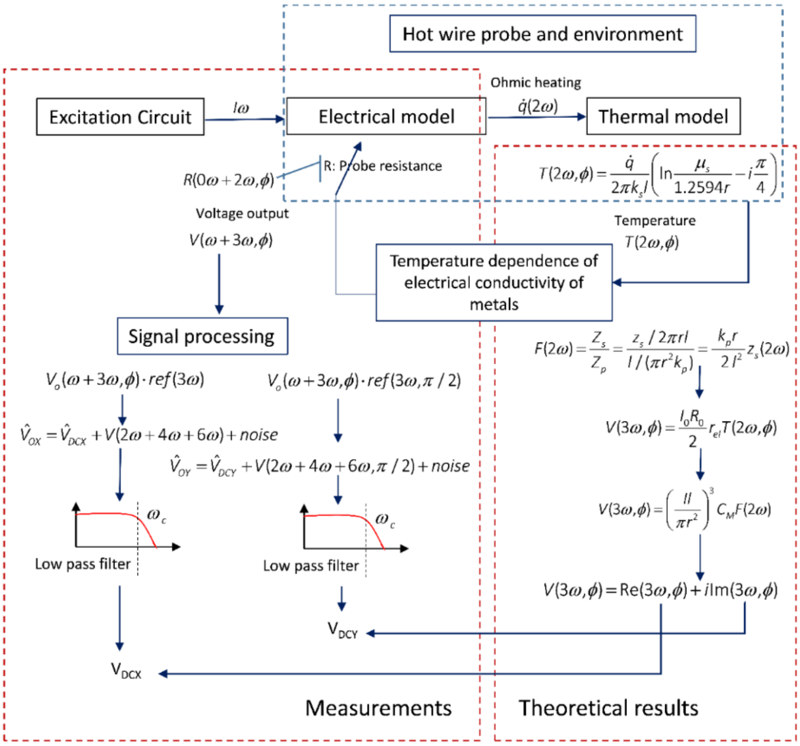

Figure 2. Theoretical and physical explanation of 3ω method

The signals are processed through the 2nd order Butterworth low-pass filters to eliminate switching noise on the digitally generated sinusoidal waves for providing pure sinusoidal signals to lock-in amplifier. Then, the signal is amplified by the preamplifier to provide the required amplitude at the experiment. After that, the signal is amplified by the current amplifier in order to provide sufficient current to excite the wire for sensing temperature change around the wire related with thermal conductivity of the liquid.

The temperature around the probe in the sample form by the effect of the signal, with the resistance of the probe varies proportionally and the Wheatstone bridge is used to detect this change (Figure 1). Since the resistance change affects the voltage balance at the Wheatstone bridge outputs, on both legs of the bridge is sent to the lock-in amplifier for the signal measurement. Before this process, for each measurement output voltage of Wheatstone bridge should be balanced to cancel the ω signal by auto zeroing unit Ateş et al., 2016).

Since the signal difference from the Wheatstone bridge is quite small, this signal is amplified with a gain of 95 dB with the instrumentation amplifier, and then the modulated signal is sent to the lock-in unit. The lock-in amplifier is a measurement device based a phase sensitive detection which developed to detect very small AC signals about a few nanovolts in noise. The signal component at a certain frequency and phase in the signal to be measured by this method can be measured according to the frequency of the reference signal. With this method, undesirable signal components and noises can be filtered better than band pass filters tuned very high Q factor.

In order to detect the 3ω signal, an analog demodulator is used as a phase sensitive detector in the developed system for measurement. The signal modulated with 3 Hz sinusoidal generated due to the heating effect on the 1 Hz carrier signal is multiplied with the reference signal at 3 Hz by the demodulator. The output of the demodulator is the product of four sinusoidal waves and noise. These sinusoidal waves are generated with sum and difference frequency of signal and reference by multiplication of them. If the frequency of the modulated signal is equal to reference frequency, the output signal will be a DC signal as the frequency difference is zero. If output signal of the demodulator is filtered by 3rd order Butterworth low pass filter, undesired signals and noise rejected thus output signal of the filter is proportional to the modulated sinusoidal wave amplitude as can be seen in Figure 2. The signal at duplicated frequencies are filtered by 3rd order a Butterworth low-pass filter demodulator output. Since the imaginary part ratios are important for thermal conductivity measurement, the phase of the reference signal is shifted by 90 degrees during the measurement process, so that the voltage at the lock-in output directly gives the value of the imager part.

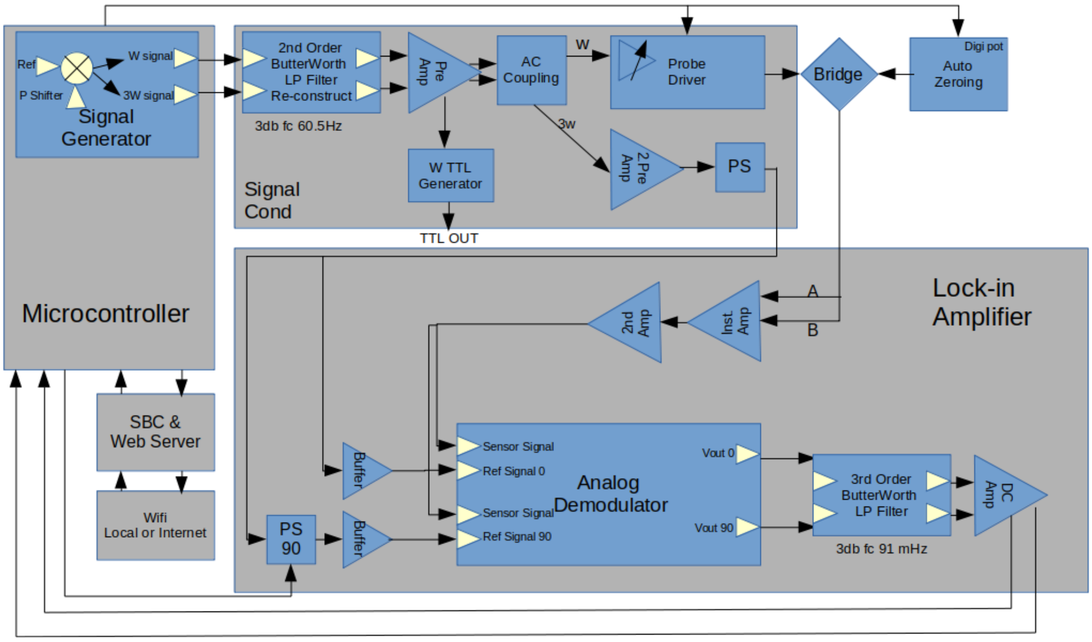

Figure 3. The block diagram of the hardware

Imaginary voltage signal in lock-in amplifier output is logged as a real time at 62 Hz sampling frequency rate by 12-bit ADC of microcontroller. Since the thermal conductivity value is determined to relative, the reference liquid and the sample liquid are measured separately and the data of the each measurement are recorded individually. When the testing process time is ended, embedded software analyze gathered data of two different liquids. The average values are calculated with using intelligent algorithm to finding steady-state arrays. The ratio of the thermal conductivity is calculated relatively by the average obtained values of the sample and the reference liquid.

3. Results and Discussions

The imaginary part of the signal related to the thermal characteristic of the fluid in the measurement process is logged from the output of the lock-in amplifier. The setup is designed as an application specific electronic hardware for providing basic measurement output previously mentioned as the working principle. It is waited for 15 seconds to get the setup reached for the steady state conditions. Then the data is logged for a time period of 64 seconds with totally 500 number of samples.

In order to observe the repeatability and wide range performance of this specifically designed measuring system, the experiments were carried out for the base fluids. In a wide range measurements, mineral oil (0.120 W/mK) (Koruk et al., 2017) and water (0.598 W/mK) (Cengel & Ghajar, 2014) were chosen as samples. In addition, ethanol (0.169 W/mK), methanol (0.200 W/mK) and ethylene glycol (0.256 W/mK) samples, which have reliable values (Lide, 2004) in the literature, were also used.

When the imaginary voltage values of the measured reference fluid and the sample fluid whose thermal conductivity is to be determined are proportioned according to Eq. 6, thermal conductivity change rates are calculated for each sample depending on the reference fluid. For all samples, water measurement was carried out as a reference sample in accordance with the working systematic and calculations were made according to the voltage values of the water. In order to facilitate traceability, two measurement results representing the general behavior are presented in the graphics. Indices 1 and 2 were used for each measurement result.

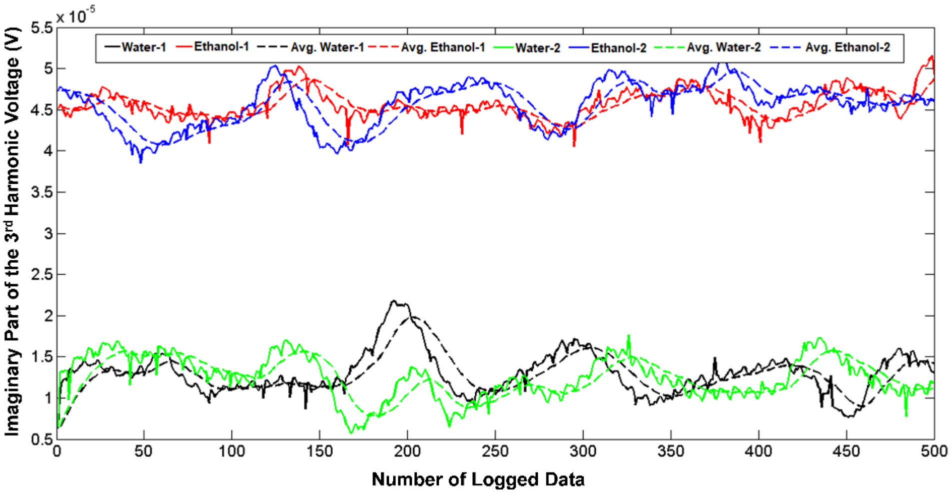

Figure 4. Imaginary part of the voltage signals of the 3rd harmonic of the ethanol sample

In Figure 4, the voltage values obtained for ethanol-1, ethanol-2 and water-1 and water-2 used as reference fluids are shown. The averages of the voltage values obtained were calculated as 45.575 and 45.582 µV for ethanol-1 and ethanol-2, respectively, and 12.802 and 12.339 µV for water-1 and water-2, respectively. When the acquired voltage values are proportioned according to the expression in Eq. 6, the relative thermal conductivity values for ethanol-1 and ethanol-2 were calculated as 0.281 and 0.271, respectively. Relative thermal conductivity is obtained as 0.275 when literature values of ethanol and water are used. Accordingly, it is seen that the relative thermal conductivity measurements for ethanol-1 and ethanol-2 can be measured with a maximum error of ± 2.2%. Voltage values for methanol, ethylene glycol (EG), and mineral oil samples are shown in Figure 5, Figure 6, and Figure 7, respectively.

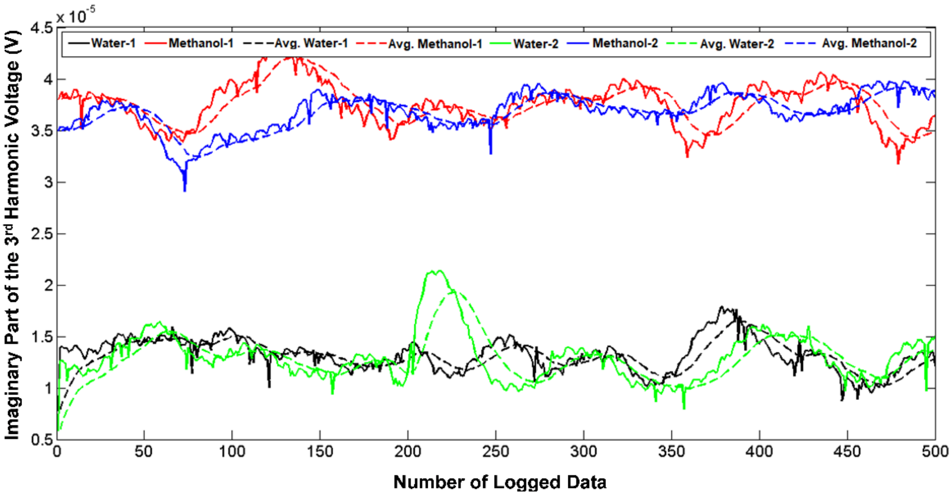

Figure 5. Imaginary part voltage signals of the 3rd harmonic of the methanol sample

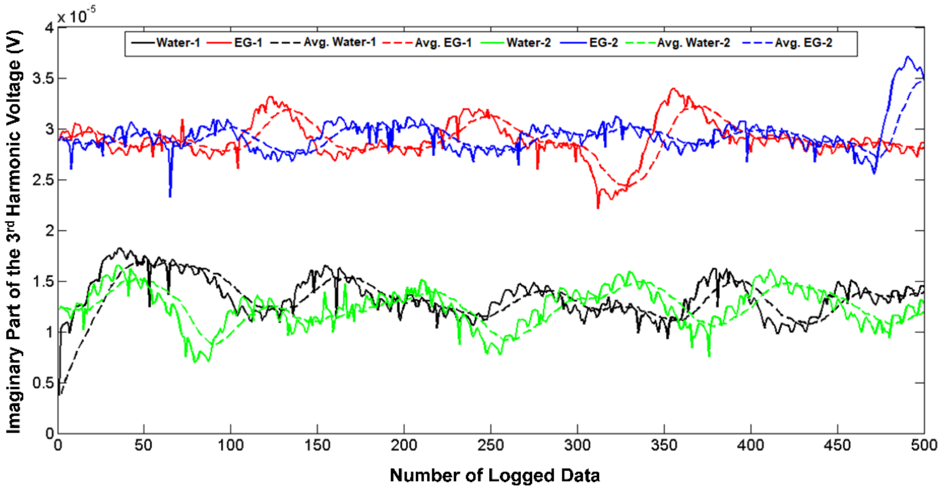

Figure 6. Imaginary part voltage signals of the 3rd harmonic of the ethylene glycol (EG) sample

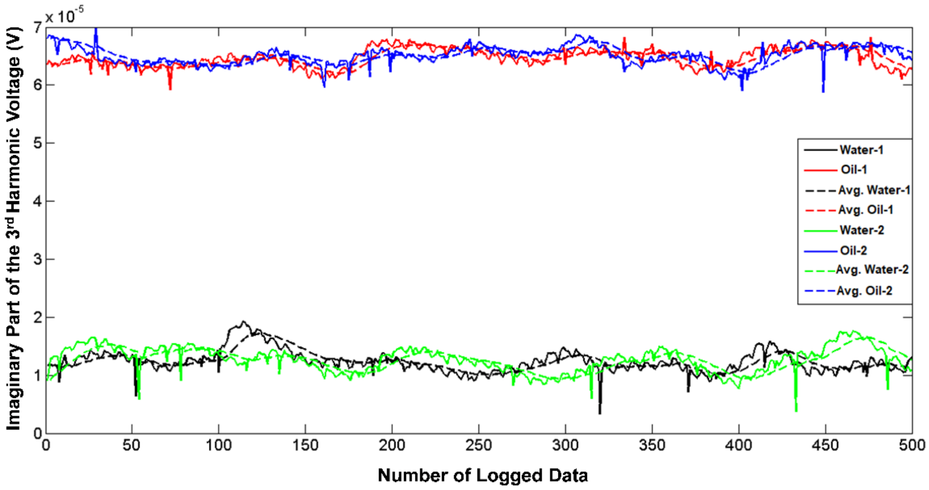

Figure 7. Imaginary part voltage signals of the 3rd harmonic of the mineral oil sample

According to the acquired voltage values, the average voltage values for each sample are given in Table 1. The relative thermal conductivity values calculated based on the average voltage values and their error rates according to the literature values are presented in Table 2.

Table 1. Average values of voltages for samples of wide range measurement

Sample	**Average Voltage (μV)**
Ethanol-1	45.575
Water-1	12.802
Ethanol-2	45.582
Water-2	12.339
Methanol-1	37.595
Water-1	13.071
Methanol-2	36.725
Water-2	12.793
EG-1	29.182
Water-1	12.370
EG-2	29.349
Water-2	12.505
Mineral Oil-1	64.706
Water-1	12.278
Mineral Oil-2	65.115
Water-2	12.551

As seen in Table 2, the highest error rate in the measurement results for ethanol, methanol, ethylene glycol and mineral oil samples was found in mineral oil with 5%. In methanol and ethylene glycol, the highest error rates were obtained as 3.9% and 0.5%, respectively. It is stated that the results obtained for the ethylene glycol sample are very close to the literature values. According to the obtained relative thermal conductivity results, it is seen that the wide range thermal conductivity measurement performance of the developed system gives results that are quite consistent with the literature.

Table 2. Thermal conductivity ratios for wide range measurement samples

Sample	**Relative Thermal Conductivity (Measurements)**		**Relative Thermal Conductivity (Literature)**	**Error (%)**	
	Min	Max	Average	Min	Max
Ethanol[1]	0.271	0.281	0.275	-1.455	2.182
Methanol[1]	0.348	0.348	0.335	3.881	3.881
EG[1]	0.424	0.426	0.426	-0.470	0.000
Mineral Oil[2]	0.190	0.193	0.200	-5.000	-3.500

[1]*(Lide, 2004)*, [2]*(Koruk et al., 2017)*

4. Conclusions

In this study, a custom-made electronic hardware for 3ω measurements was presented. The performance of the developed system was investigated in wide-range thermal conductivity measurement of base fluids. In wide range measurements, mineral oil and water, which have approximately 5 times difference between thermal conductivity values, were used as reference samples. In addition, ethanol, methanol, and ethylene glycol samples, which have reliable thermal conductivity values in the literature, were also measured.

Following conclusions can be drawn from this study:

- By the improved algorithm, the thermal conductivity ratio of the samples are determined automatically and faster in comparison with Turgut et al., (2008).
- The developed hardware in this system decreases the essential time to achieve steady state condition.
- Thermal conductivity results of the base fluids are obtained by the designed system shows a good agreement with the results for mineral oil, water, in addition ethanol, methanol and ethylene glycol samples.
- The thermal conductivity of base fluids can be measured with a maximum 5% error in the wide range measurement.

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Author Statement

The authors confirm contribution to the paper as follows:
Study conception and design: Ismet Ateş, Alpaslan Turgut, Levent Çetin; data collection: İsmet Ateş, Alper Mete Genç; analysis and interpretation of results: İsmet Ateş, Alpaslan Turgut, Serkan Doğanay; draft manuscript preparation: İsmet Ateş, Alper Mete Genç. All authors reviewed the results and approved the final version of the manuscript.

Conflict of Interest

The authors declare no conflict of interest.

Nomenclature

$\emptyset$	Phase angle (radian)
C_M	Figure of merit
F	Dimensionless impedance
f	Frequency (Hz)
I	Current (A)
I_0	Current through wire (A)
k	Thermal conductivity (W/mK)
k_p	Thermal conductivity of probe (W/mK)
k_s	Thermal conductivity of sample (W/mK)
k_w	Thermal conductivity of water (W/mK)
l	Length of the wire (m)
q	Rate of heat transfer (W)
r	Radius of the wire (m)
r_{el}	Temperature coefficient of the resistance (1/K)
R_0	Resistance of wire (Ω)
T	Temperature (K)
V	Voltage (V)
V_{DCX}	Real part of voltage of 3rd harmonic (V)
V_{DCY}	Imaginary part of voltage of 3rd harmonic (V)

V_{0X}	Real part of the voltage (V)
V_{0Y}	Imaginary part of the voltage (V)
Z_p	The thermal resistance of the half-length wire (m^2 K/W)
Z_s	The thermal impedance of the interface (m^2 K/W)
z_s	Thermal impedance (m^2 K/W)
μ_s	The thermal diffusion length (m)
ω	Omega (rad/s)

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