DOI 10.5162/16dss2022/P03

Electrical Conductivity Sensor with Open-Source Hardware for the Microfluidic Determination of Reaction Parameters

Robin Dinter[1], Lennart Helwes[1], Marcel Pillath[1], Norbert Kockmann[1]

[1]TU Dortmund University, Department of Chemical and Biochemical Engineering, Laboratory of Equipment Design, Dortmund/Germany
Contact: robin.dinter@tu-dortmund.de

Introduction

Due to the miniaturization of equipment in microprocess engineering and flow chemistry, the miniaturization of sensors and analytical devices for automated inline process control is also necessary [1]. For example, electrolytic conductivity is suitable as a measured variable for the quantitative determination of ion concentration and reaction progress in microfluidics [2]. In this work, an electrical conductivity measuring device was developed and verified using only open-source hardware for low-cost microfluidic determination of reaction parameters. The first section focuses on open-source electrical circuit design. Next, the newly developed electrical conductivity flow sensor with temperature compensation is verified and compared to a conventional reference batch conductometer. The detailed microfluidic application of the electrical conductivity flow sensor is not the main scope of this work and can be found in another study by Dinter *et al.* [3].

Development of the Measuring Devices

The newly developed electrical conductivity flow sensor consists of two measurement devices, as shown in Fig. 1.

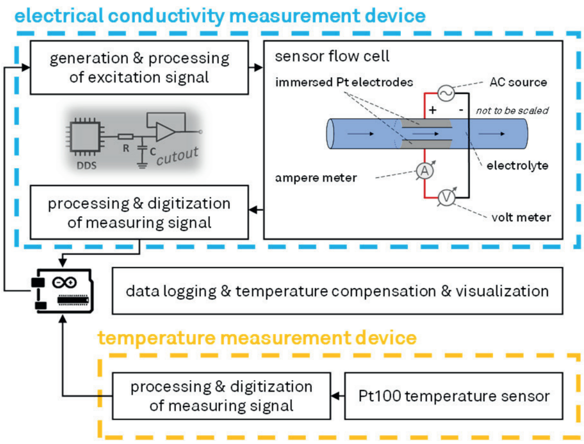

Fig. 1: Block diagram of the newly developed flow sensor for measuring temperature-compensated electrical conductivity using open-source hardware.

The first device is the electrical conductivity measurement device, which consists of the electrical circuit and the sensor flow cell. The self-designed electrical circuit generates the sensor's alternating current (AC) excitation signal with a defined frequency and amplitude, measuring the impedance in the sensor flow cell. The self-developed sensor flow cell consists of two Pt electrodes directly immersed in the electrolyte solution to create a stable electric field between the two electrodes. The inner channel diameter of the flow cell is 1.6 mm. After the impedance measurement in the flow cell, the electrical circuit digitalizes and processes the measured signal to a micro-controller-board (MCB).

The measured impedance indirectly represents the electrical conductance $G\,[S]$. Thus, the electrical conductivity $\kappa\,[S\,m^{-1}]$ is derived from the electrical conductance in combination with the cell constant $K\,[1\,m^{-1}]$ (Equ. 1).

$$\kappa\left[\frac{S}{m}\right] = G\,[S]\cdot K\left[\frac{1}{m}\right] \qquad \textbf{Equ. 1}$$

The quotient of the electrode distance $l\,[m]$ and the electrode surface A [m^2] is the cell constant K of the sensor flow cell, which is selected to $1\,cm^{-1}$. This enables precise measurement of electrolytic solutions between $10\,\mu S\,cm^{-1}$ and $20\,mS\,cm^{-1}$ according to the literature. [4, 5].

In addition to the electrical conductivity measurement device, a device for measuring temperature was introduced to compensate for the influence of temperature on electrical conductivity. The temperature dependence on electrical conductivity is usually non-linear since the relationship between temperature and electrical conductivity is established indirectly via viscosity. For comparing the measured electrical conductivities at different temperatures, the measurements are related to a reference temperature of 25 °C. Nevertheless, in practice, there is a linear temperature compensation for many solutions, where only the slope of the linear function needs to be adjusted, as shown in Equ. 2. [6]

$$\kappa_{25} = \frac{\kappa}{1+\Theta_C\cdot(T-25)} \qquad \textbf{Equ. 2}$$

The compensated electrical conductivity κ_{25} is calculated by the measured values for the electrical conductivity κ and temperature T. The factor Θ_C is the electrical conductivity's temperature coefficient, which is determined experimentally, as shown in the Verification and Application section.

The MCB logs all measurements and sends the measured impedances and temperatures via serial communication to a computer for further calculation, compensation, and visualization using the Jupyter Lab software project (Version: 3.3.2) with Python (Version: 3.10.6, Delaware, USA). In the following subsections, the installed components and electrical circuit design of the measurement devices are explained in detail using only open-source hardware.

DOI 10.5162/16dss2022/P03

Electrical Conductivity Measuring Device

The electrical circuit for the electrical conductivity measuring device is designed as follows. First, a programmable sine wave generator (AD9833, Analog Devices, Norwood, USA) is actuated via a microcontroller (Atmega3288, Microchip Technology, Chandler, USA) to generate the AC excitation signal for the sensor (Fig. 2).

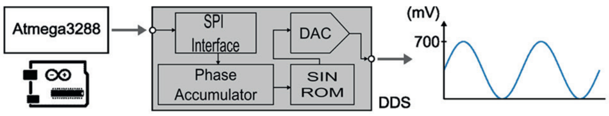

Fig. 2: Generation of the AC excitation signal with a sine wave generator including the internal structure.

Here, the programmable sine wave generator, also called a direct digital synthesis chip (DDS), digitally generates a sine wave and converts it into an analog signal. The internal circuitry consists of a phase accumulator, a SIN ROM and a digital-analog-converter (DAC). The advantages of the DDS are that the signal frequency can be arbitrarily set over a wide range ($0.1\,Hz - 12.5\,MHz$) and with a high resolution ($0.004\,Hz$) [7]. However, AC with a specific frequency and voltage amplitude is needed to measure the electrolytic conductivity adequately. In the Verification and Application section, the dependence of the electrical conductivity on the frequency is experimentally investigated and the operating frequency is selected. The voltage amplitude was set to $2.4\,V$ because it must be within the operation range of the ADC ($0 - 4\,V$) and provide a sufficient safety margin against overvoltage. The output of the DDS is an analog sine wave with a defined frequency with a voltage range from $0 - 700\,mV$. It does not have sufficient amplitude nor change polarity as required for the application [7]. Therefore, it needs to be processed first by a high-pass filter and conditioned next by a non-inverting operational amplifier (OpAmp).

The DDS's output signal is a superposition of two signals of different frequencies, an AC excitation signal with a specific frequency and a direct current (DC) offset. It is essential to remove the DC offset to avoid electrolysis inside the sensor flow cell. Therefore, a high-pass filter is applied, consisting of a capacitor and a resistor, as shown in Fig. 3.

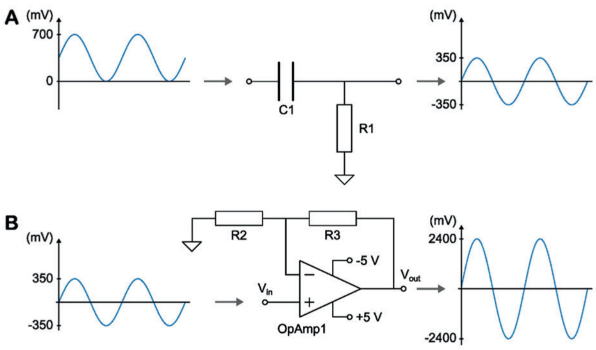

Fig. 3: Processing and conditioning of the AC excitation signal. A) High-pass filter circuit for filtering the DC offset. B) OpAmp for amplifying the filtered signal.

The capacitor is a frequency-dependent resistor that attenuates low frequencies by the high reactance for passing higher frequency voltages. After the high-pass filter removes certain frequencies of the signal, the output signal is a sine wave signal with an amplitude of $-350\,mV$ to $350\,mV$, which is further conditioned by an OpAmp (AD8055, Analog Devices, Norwood, USA), amplifying the signal to the required signal strength and specific voltage amplitude of $2.4\,V$ (Fig. 3).

After conditioning, the AC excitation signal is passed to a voltage divider measuring the impedance of the sensor flow cell (Fig. 4).

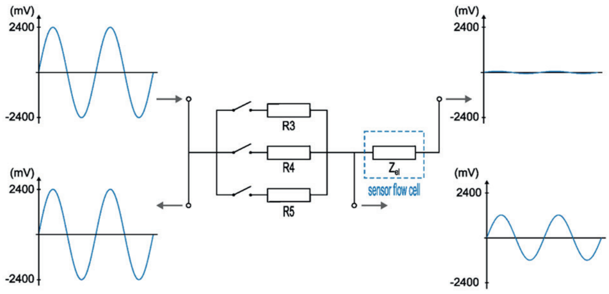

Fig. 4: Processing of the measured impedance by a voltage divider with input and output signals of the sensor flow cell and voltage divider.

The voltage divider measures the voltages before and after the reference resistor. It is possible to switch between three reference resistors for a variable and extended measuring range. The $20\,\Omega$ resistor (R6) offers a measuring range of $10\,mS - 200\,mS$ and the $2000\,\Omega$ resistor (R4) has a range of $100\,\mu S - 2\,mS$. In this work, the $200\,\Omega$ resistor (R5) was used, resulting in measured electrical conductance in the range of $1\,mS - 20\,mS$. All resistors are precision resistors with tolerances of less than $0.1\,\%$.

After the voltage divider, the resulting measurement signals are processed, as shown in Fig. 5.

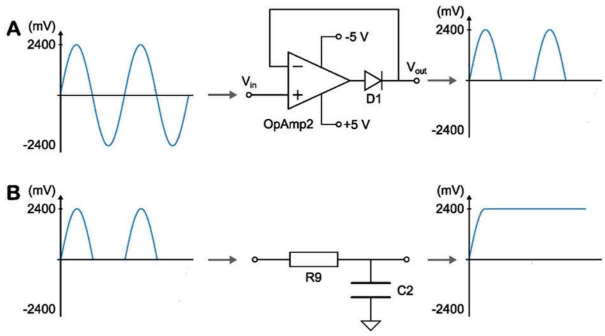

Fig. 5: Processing of the measuring signal. A) Half wave rectifier for filtering the negative half waves. B) Low-pass filter for filtering the rectified measurement signal into a representative DC measuring signal.

The measurement values must be processed and the negative voltage segments must be removed since negative voltages destroy the analog-digital converter (ADC) for digitization. This is done with a half-wave rectifier (Fig. 5) consisting of an OpAmp (AD8055, Analog Devices, Norwood,

DOI 10.5162/16dss2022/P03

USA) and a diode (1N4148, Olitech Electronics, SheungWan, HK). The diode allows the electrical current to pass in only one direction and removes the negative parts of the AC excitation signal. The resulting signals are sine waves containing only the positive parts. Next, a low-pass filter converts the resulting half-waves into a representative DC signal for digitization (Fig. 5). This attenuates the high frequencies and allows lower voltage frequencies to pass.

A low-cost, high-resolution ADC (model: ADS1115, Texas Instruments, Dallas, USA) is used to digitize the DC signal, as shown in Fig. 6.

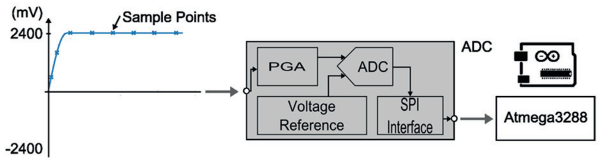

Fig. 6: Digitization of the DC signal with an ADC including the internal structure of the ADC.

The ADC samples the voltage values at specific points in time, which correspond to the sample rate of the ADC (860 samples per second at a resolution of $16\ bits$). As a result, the maximum quantization error is about $1.5\ \mu S$. Finally, the values are transmitted to the external computer via the MCU and calculated into electrical conductivities.

To verify the newly designed electrical circuit with open-source components, all electronic components are simulated using circuit simulation software (LTSpice, Analog Devices, Norwood, USA), as shown in Fig. 7.

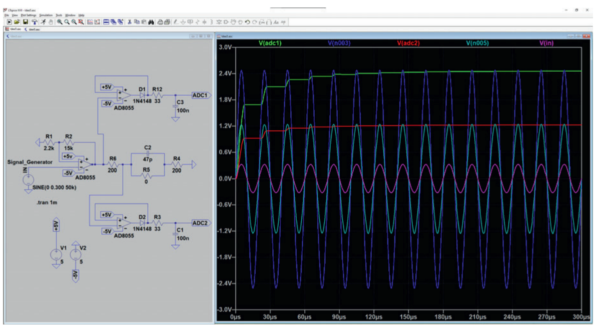

Fig. 7: Simulation of the newly designed electrical circuit for the electrical conductivity measurement device.

The electrical circuit simulation results correctly reflect the theoretical voltage curves shown in the previous figures and are electrically checked for the final application.

All used electronic components and prices [7] (last accessed: Feb 10th, 2022) are listed in Tab. 1. It is shown that the total hardware costs of the open-source electrical components are less than 50 €.

Tab. 1: Electronic components and prices [7] of the newly designed open-source electrical circuit for electrical conductivity measurements.

Component	Label	Price / €
Arduino Nano	MCU	13.00
ADS1115	ADC	14.00
AD9833	DDS	10.00
AD8055	OpAmp1, 2, 3	8.45
Resistor / $15\ k\Omega$	R1, R2, R7, R8	0.60
Resistor / $2.2\ k\Omega$	R3	0.15
Resistor / $100\ \Omega$	R9,10	0.16
Resistor / $20\ \Omega$	R6	0.25
Resistor / $200\ \Omega$	R5	0.43
Resistor / $2k\ \Omega$	R4	0.20
Capacitor / $100\ nF$	C1, C2, C3	0.21
1N4148 diode	D1, D2	0.04
Jumper & boards		2.31
		49.80

After the electrical circuit for the electrical conductivity measuring device was correctly designed, the temperature measuring device for temperature-compensation is briefly described in the following.

Temperature Measuring Device

The additional temperature measuring flow device was developed in parallel with the measuring device for electrical conductivity, as shown in Fig. 1. Both flow sensors are connected in series to compensate for the temperature dependence on electrical conductivity and enable temperature-compensated measurements at different temperatures. The temperature measuring device consists of a Pt100 resistance thermometer (Pt100A 10/10, Electronic Sensor, Heilbronn, GER) immersed in a flow cell. A resistance-to-digital converter (RTDC) (AX3185, Maxim Integrated, San Jose, USA) is used for processing and digitalizing the measured temperatures.

Verification and Application

The newly developed measuring flow device has a modular design and measures temperature-compensated electrical conductivities $< 10\ mS\ cm^{-1}$ of flowing electrolyte solutions in a microchannel. In this work, the focus is on verifying the electrical design circuit for the conductivity sensor and the validity of the application. First, the operating frequency of the AC excitation signal was investigated and the linear temperature compensation was tested. Next, the newly developed flow sensor was compared with a conventional reference batch conductometer to verify the suitability of the flow sensor for the microfluidic applications. A detailed overview of the microfluidic application is given in another study by Dinter *et al.* [3].

Operating Frequency of the AC Excitation Signal

For microfluidic application and testing of the sensor, a suitable value for the operating frequency of the applied AC excitation signal must first be determined. It is important because the measured electrical conductivity depends on the applied AC frequency due to the electrodes' capacitive influence on the electrolyte's measured electrical conductance. The dependence of the electrical conductivity on the frequency was examined experimentally, as shown in Fig. 8.

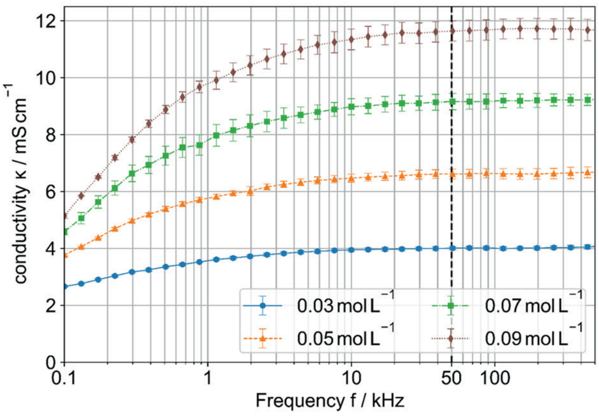

Fig. 8: Dependence of the electrical conductivity on the applied AC excitation signal frequency at a voltage amplitude of $2.4\ V$. KCl solutions with varying concentrations between $0.03\ mol\ L^{-1}\ -\ 0.09\ mol\ L^{-1}$.

Here, the measured electrical conductivity for four potassium chloride (KCl) solutions with different concentrations between $0.03\ mol\ L^{-1}\ -\ 0.09\ mol\ L^{-1}$ is shown as a function of the applied AC frequency in kHz. The frequency varies from $100\ Hz\ -\ 500\ kHz$ to find a possible operating frequency of the newly developed flow sensor. All curves rise with increasing frequency from $100\ Hz\ -\ 500\ kHz$ until they reach a plateau and remain stable for all frequencies tested. At frequencies below $10\ kHz$, the influence of polarization effects on the electrodes results in measured electrical conductivities lower than above $10\ kHz$. In addition, at low concentrations, KCl forms a plateau at a lower frequency of $10\ kHz$, while at higher concentrations, it starts to form a plateau at frequencies higher than $30\ kHz$. This is due the increased electrode polarization effect with larger electrical conductive electrolytes. It is concluded that the more electrical conductive the measured electrolyte, the higher the frequency of the applied AC excitation signal must be. Since the results do not indicate that the use of a frequency of $50\ kHz$ or higher has a negative effect on the measurement of electrolytes with lower electrical conductivity, the operating frequency of the AC excitation signal was fixed to $50\ kHz$ in this work.

Linear Temperature Compensation

After setting the operation frequency, the linear temperature compensation was tested for the newly developed flow sensor to verify the measurement of temperature-compensated electrical conductivities. The following experiment was conducted to investigate the applicability of linear temperature compensation and determine a suitable temperature coefficient.

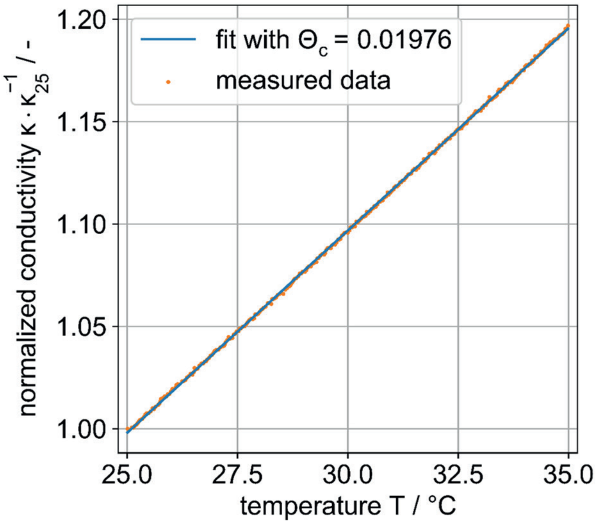

Fig. 9: Measured electrical conductivity linearly fitted ($R^2\ =\ 99.99\ \%$) with a temperature coefficient of $1.976\ \%$ as a function of the temperature, normalized to the measured value at $25\ °C$.

Fig. 9 shows the measured electrical conductivity in a temperature range between $25\ °C$ and $35\ °C$, normalized to the measured electrical conductivity at $25\ °C$ with KCl electrolyte solution of $0.05\ mol\ L^{-1}$. The normalized electrical conductivity increases linearly in the temperature range from $25\ °C$ to $35\ °C$. The measured electrical conductivity value at $35\ °C$ is $19.7\ \%$ higher than the value at $25\ °C$. The coefficient of the temperature compensation θc is the slope of the fitted function. Thus, a slope of 0.01976 results in a temperature coefficient of $1.976\ \%$. This can be compared with a literature value for θc of $1.989\ \%$ between $25\ °C$ and $35\ °C$ [8]. Therefore, the performed linear temperature compensation of electrical conductivity is suitable for $25\ °C$ to $35\ °C$.

Accuracy and Precision

The overall performance of the electrical conductivity flow sensor is evaluated in terms of the accuracy and precision of the measurements. The precision is evaluated by comparing the results of three consecutive measurements of five KCl solutions in the range of $0.01\ mol\ L^{-1}$ and $0.05\ mol\ L^{-1}$. The accuracy is investigated by comparing the measured electrical conductivity values in flow to the measured values of a commercial reference batch conductometer (WTW Multi 9310, Xylem Analytics, Rye Brook, USA). The results are shown as a parity plot in Fig. 10.

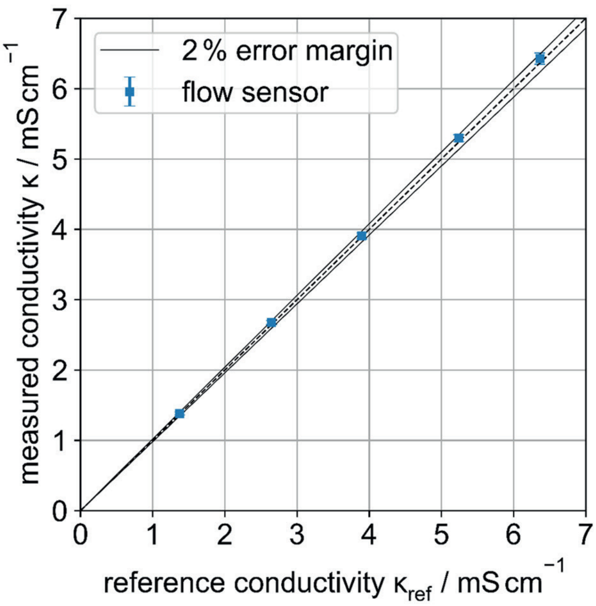

Fig. 10: Parity plot of the measured electrical conductivities with the newly developed flow sensor compared to a commercial reference batch conductometer.

The angle bisector (dashed line) represents points where the measured values correspond to the reference values. The closer the data points are to the dashed line, the higher the accuracy of the measurements compared to the reference measurements. Here, the measurement results are within a $2\,\%$ error margin compared to the commercially available instrument. In addition to the high accuracy, the precision of the measured values is also high due to the small errors of the measurement. Therefore, the accuracy and precision of the newly developed electrical conductivity flow sensor are entirely acceptable for low-cost determination of electrical conductivity in microfluidic.

Application for Microfluidics

Further studies by Dinter *et al.* [3] successfully show the scope of application of the newly developed electric conductivity sensor. The microfluidic reaction parameters of the saponification reaction of ethyl acetate were accurately determined. However, the temperature-compensated electrical conductivities were directly related to the saponification reaction progress (conversion) and enabled robust and cost-effective in-line process control. In addition, the reaction kinetics were calculated using the measured conversions of the saponification reaction. [3, 9]

Conclusion

In summary, the presented electric conductivity flow sensor using mainly open-source hardware is a cost-effective (hardware costs $< 50\,€$) and successful alternative to commercial measurement devices in the field of microfluidics. The electrical circuit was successfully designed and verified using basic and freely available electrical components such as OpAmps or Diodes for signal processing and open-source break-out boards for signal generation and digitization. The interconnection of the sensor flow cell and the electrical circuit enables the measurement of electrolyte solutions with electrical conductivities $< 10\,mS\,cm^{-1}$ accurately and precisely within a $2\,\%$ error margin. In parallel, the temperature measuring device was integrated to accurately measure the temperature-compensated electrical conductivity in the temperature range from $25\,°C$ to $35\,°C$. The successful application of the developed measuring device for the cost-effective determination of microfluidic reaction parameters is given in further studies by Dinter *et al.* [3].

Further research potential for the electrical circuit exists in modifying the electrode wiring. Instead of the current two-wire measurement, a four-wire measurement allows higher frequencies to determine higher electrical conductivities with lower electrical interference. Additionally, non-linear temperature compensation can improve the accuracy of the electrical conductivity measurement at varying temperatures in a broader temperature range.

References

[1] HÖVING, S., BOBERS, J., KOCKMANN, N.: Open-source multi-purpose sensor for measurements in continuous capillary flow. In: *J. Flow Chem* 12, 185-196 (2022).

[2] TROJANOWICZ, M.: Flow chemistry vs. flow analysis. In: *Talanta* 146, 621-640 (2016).

[3] DINTER, R., HELWES, L., KOCKMANN, N.: Reaction Kinetic Investigation with Open-Source Microfluidic Conductivity Sensor. In: *μFIP conference proceeding* 22CBMS-0001, 23 (2022).

[4] HAMILTON COMPANY, Conductivity Sensor Selection. In https://www.hamiltoncompany.com/process-analytics/conductivity-knowledge/conductivity-sensor-selection (last accessed: 10/27/2022).

[5] ATKINS, P.: Atkins' Physical Chemistry, Oxford University Press, Oxford, 2018.

[6] ANALOG DEVICES: AD9833 EP, Programmable Waveform Generator. In https://www.analog.com/media/en/technicaldocumentation/data-sheets/ad9833.pdf (last accessed: 10/27/2022).

[7] REICHELT ELEKTRONIK: Onlineshop. In https://www.reichelt.de/ (last accessed: 02/10/2022)

[8] RADIOMETER ANALYTICAL SAS: Conductivity Theory and Practice. In https://www.tau.ac.il/~chemlaba/Files/Theoryconductivit.pdf (last accessed: 10/27/2022).

[9] BOROVINSKAYA, E. *et al.*: Experimental Studies of Ethyl Acetate Saponification Using Different Reactor Systems: The Effect of Volume Flow Rate on Reactor Performance and Pressure Drop. In *Appl. Sci.* 9(3), 532 (2019).

Acknowledgments

The authors thank the technician Carsten Schrömges for his support and the German Research Foundation (DFG) for financial support under Grants KO2349/18.