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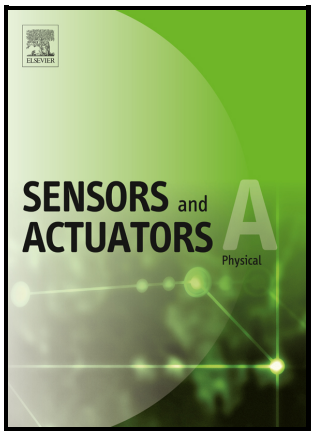

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Implementation and calibration of a phase detector circuit as a QCM sensor to characterize fluid properties

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***Abstract**:*

In this work, a quartz resonator or QCM ~~microbalance~~ sensor system based on a phase detector circuit is implemented. Until now, the phase detection method in QCM sensors has been used for mass measurement applications. In this work, the system is characterized for its use in applications to measure the properties of a liquid (density and viscosity). In a first part, the implementation of the system is described. Once implemented, it is calibrated with solutions of increasing viscosities, obtaining the sensitivity equation and the resolution of the output voltage to changes over the square root of the density-viscosity product. Finally, the results are compared with those obtained by the authors in previous works using oscillator circuits and a network analyzer as electronic measurement interfaces.

***Keywords**:*

Phase Detection, QCM Sensors, Liquid Medium, Quartz Resonator, Sensitivity, Resolution.

1. Introduction

Quartz crystal microbalance (QCM) is an ultrasensitive mass sensor based on a piezoelectric resonator, usually AT-cut quartz, in which a thickness-shear-mode (TSM) acoustic wave is propagated. Under specific conditions, the resonant frequency varies proportionally with a rigid mass layer deposited on its surface [1]. In 1985, Kanazawa and Gordon [2] demonstrated that the resonant frequency of QCM sensors, f_s, is also sensitive to changes in the density (ρ) and viscosity (η) of a solution in contact with the sensor (1).

$$\Delta f_s = -f_0^{\frac{3}{2}} \cdot \sqrt{\frac{1}{\pi C_{66} \rho_q}} \Delta\sqrt{\rho\eta} \tag{1}$$

where:

- Δf_s is the series resonant frequency shift (Hz);
- f_0 is the fundamental series resonance frequency of the crystal without charge (Hz);

- $\overline{c_{66}}$ is the elastic constant of the AT-cut quartz for the transverse thickness mode (2,947 × 10^{11} g/(cm.s^2));
- ρ_q is the quartz density (2.651 g/cm^3);
- $\Delta\sqrt{\rho\eta}$ are changes in the square root of the product of the density and the viscosity of the liquid.

To analyze the QCM resonator as an electronic component within a circuit, Martin and Granstraff [3] have proposed the modification of the electromechanical model of BVD (Butterworth Van-Dyke) shown in Figure 1. The mass added to the surface of the quartz resonator contributes by increasing the value of the inductance L_{Q0} of the unloaded Butterworth-Van Dyke circuit by a value equal to L_{mass} (2), which depends on mass density per surface unit, ρ_S. The liquid medium characterized by the liquid density viscosity product, $\rho\eta$, influences an increase in L_{Q0} and R_{Q0} by an amount equal to L_{liq} (3) and R_{liq} (4) respectively.

$$L_{mass} = \frac{2\omega_o L_{Q0}\rho_s}{N\pi}\sqrt{\left(\frac{1}{\bar{c_{66}}\rho_q}\right)} \tag{2}$$

$$L_{liq} = \frac{\omega_0 L_{Q0}}{N\pi}\sqrt{\left(\frac{2\rho_L\eta_L}{\omega_0\bar{c_{66}}\rho_q}\right)} \tag{3}$$

$$R_{liq} = \frac{\omega_0 L_{Q0}}{N\pi}\sqrt{\left(\frac{2\omega_0\rho_L\eta_L}{\bar{c_{66}}\rho_q}\right)} \tag{4}$$

The presence of the fluid and the additive mass alter the series resonance frequency, f_s, (5), which is the magnitude of measurement usually used in the capture process, such as:

$$\Delta f_s \approx -\left\{\frac{2\cdot f_0^2}{\sqrt{\overline{c_{66}}\cdot\rho_q}}\cdot\Delta\rho_s + f_0^{\frac{3}{2}}\cdot\sqrt{\frac{1}{\pi C_{66}\rho_q}}\Delta\sqrt{\rho\eta}\right\} \tag{5}$$

This microbalance sensor is widely used to study the evolution of processes by observing the associated mass changes, but it can also be used to characterize the physical properties of a fluid. In the latter case, in addition to the expected changes in the resonant frequency, f_s, there are also variations in the series resistance, R, (6) versus the parameters which characterize the liquid properties:

$$\Delta R \;= 4\cdot L_{Q0}\cdot {f_0}^{3/2}\cdot\sqrt{\frac{\pi}{\overline{c_{66}}\cdot\rho_q}}\cdot\Delta\sqrt{\rho\eta} \tag{6}$$

where L_{Q0} is the motional inductance of the quartz without charge.

The use of the quartz resonator in the feedback network of an electronic oscillator circuit makes it possible to determine the changes in the resonant frequency, f_s, from the variations in the oscillation frequency of the associated electronic circuit. The low cost of the oscillators and the possibility of "continuous" real-time monitoring make the oscillators a good choice for most QCM sensor applications. However, oscillation phase variations produce oscillation frequency instabilities, decreasing the resolution of the sensor [4]. In order to reduce frequency fluctuations, Arnau *et al.* [5] have proposed a

new measurement technique. It consists of exciting the crystal with a very stable fixed frequency and measuring the phase changes of the sensor. In second reference coming from the Arnau group [6], they have experimentally verified the validity of this technique for the measurement of mass deposited on the surface of the resonator in contact with a liquid.

In our work, a phase detection system for QCM resonators was implemented and calibrated in order to characterize the variations in the properties of a liquid. This system is similar to those proposed by Arnau. In a first part, the implemented electronic system is described. Subsequently, the performed calibration and the found sensitivity equation are described. By this way, the resolution of the system is estimated. Finally, the results are compared with those obtained by an active measurement with oscillators and by a passive measurement with a network analyzer.

2. Sensor system design

The sensor system is inspired by the block diagram proposed by Montagut *et al.* in references [5,6] to develop electronic interfaces for phase detection measurement in QCM sensors. Figure 2 shows the implemented electronic sensor system. The circuit is made up of two branches: one of them contains the QCM resonator and the other a parallel RC network (R_6 - C_{11}) that simulates an uncharged quartz at the resonant frequency. Both branches will be excited by a signal with a fixed frequency close to that of resonance. The phase changes caused by the magnitude to be measured (mass changes or liquid properties changes) in the QCM resonator will give rise to a phase difference between the output signals of both branches, VINA and VINB in Figure 2, proportional to the variations of the magnitude to be measured. If both signals are brought to a phase detector, an output voltage can be obtained that is proportional to the phase shift and, therefore, to changes in the magnitude to be measured.

As a QCM resonator, a 9MHz crystal from Inficon (AT-cut, 25ºC, nº149272-1) was used. As the QCM resonator will be used submerged in liquid medium, the equivalent BVD circuit of the crystal was characterized in both air and distilled water, using an Agilent E5070B network analyzer. Table 1 summarizes the obtained results. The values obtained for R and C_p in distilled water were used to choose the values of R_6 and C_{11}.

The IC AD9913 Direct Digital Synthesizer (DDS) from Analog Devices was chosen to generate the excitation frequency. The AD9913/PCBZ Evaluation Board was used with an external 25MHz crystal as device clock oscillator and a PLL 2x multiplier to obtain a frequency around crystal resonance (9MHz). Its short-term stability was determined by calculating the Allan deviation [7]. Figure 3 shows the obtained results. For a normalization of the frequency stability measurements in the time domain, the IEEE has proposed a two-sample deviation called Allan deviation. It is denoted by σ (τ) and corresponds to equation (7),

$$\sigma(\tau) = \sqrt{\frac{1}{2} < (y_{k+1} - y_k)^2 >} \tag{7}$$

where y_k represents the relative frequency variation, defined as $y_k = \Delta f_k / f_0$, Δf_k represents the frequency fluctuation or frequency noise of the signal, and f_0 is the nominal frequency. The fractional

frequencies, $y = \Delta f/f$, are measured over the time interval, τ. The Allan deviation is calculated with respect to the averaging time, τ, as the noise is a function of the averaging time (also called "measurement time" or "integration time"). A maximum noise $\sigma_{max} = 2{,}56.10^{-8}$ was found for an integration time of 1s ($\sigma_{max} f_0 = 0{,}23Hz$).

The IC AD8302 from Analog Devices was used as phase detector, the same one used by Montagut *et al* [6]. Before implementing the system, the relationship between the phase shift at the detector inputs and the output voltage was experimentally determined for a working frequency of 9MHz. Calibration was performed using the manufacturer's recommended settings on the data sheet. It is the same configuration used in the sensor system of Figure 2, from VINA and VINB to the right.

The VINA and VINB inputs were generated using a prototype designed by Televes based on FPGA where a dual output DDS with programmable phase shift is implemented. Two sinusoidal signals synchronized in frequency (9 MHz) with increasing phase shifts from 0º to 180º with 5º increments were applied. The phase shift between the input signals to the AD8302 (pins 2 and 6) and the output voltage (pin 9) were simultaneously measured using an Agilent 53132A frequency counter and an Agilent 34461A multimeter, respectively. A reference voltage (pin 11) of 1.836 V was measured with a handheld multimeter. Figure 4 shows the obtained results. By linear regression between 0º and 140º, the equation (7) was obtained:

$$V_{phase} = -0{,}0106 \mid \Delta\varphi \mid +1{,}792, \ (0^o<|\Delta\varphi|<140^o) \tag{7}$$

In the branches connected to the phase detector inputs (QCM branch and R_6-C_{11} branch that simulates the behavior of the QCM resonator in water), the voltage buffers were implemented using low-noise operational amplifiers OPA656 from Texas Instrument. The output signals of the two branches have a phase shift of 90 ° in order to optimize the operation of the phase detector.

In addition to the output proportional to the phase difference (VPHASE), the AD8302 incorporates an amplitude detector on pin 13. The VMAG output provides a voltage proportional to the difference between the amplitudes. In this case, the manufacturer indicates that the linear response is obtained around 0dBs.

3. Experimental results and discussion

3.1. Calibration, sensitivity and resolution

To study the sensitivity of the sensor system to changes in the square root of the density-viscosity product of a liquid, solutions of glycerol 99% extra pure (Scharlau GL0027) in water with increasing viscosities were used. The temperature of the solutions was kept constant at 25°C during the experiments using a Polyscience Programmable Temperature Controller. Using an Anton Paar DMA-35 densimeter, the density and temperature of the solutions were measured during the tests. The viscosity was determined using the Bosart and Mason tables [8]. Table 2 summarizes the density and viscosity of each of the solutions used at a temperature of 25 ° C. The test frequency was chosen by studying the frequency

response of the system with the QCM resonator immersed in each mixture. The DDS was programmed to provide increasing frequencies, with an increase of 1 Hz every millisecond. The DDS output frequency and output voltage (VPHASE) were monitored. Throughout the experiment the temperature was kept constant. Figure 5 shows the results obtained for each solution and the chosen test frequency (f_t = 9.0000044 MHz) in order to optimize the detection of variations in the density-viscosity product of the liquid.

Subsequently, the calibration of the sensor was carried out in order to determine its sensitivity equation. For a fixed test frequency, f_t,, the output voltage was monitored as a function of time, with a sampling interval of 1s for 5 min per solution, and the mean value and the standard deviation of the output voltage were determined for each mixture.

Table 3 summarizes the obtained values. With the mean values, the sensitivity curve of the sensor system (8) was determined, which relates the variation of the output voltage with the variation of the square root of the density-viscosity product, where A_1, A_2 and A_3 are the experimental sensitivity coefficients for a reference solution $\sqrt{\rho\eta}_0 = 1{,}0534\ \cancel{3}\sqrt{\boldsymbol{g/cm^3 \cdot cp}}$ and where V_0 = 0,64063 V is the output voltage for $\sqrt{\rho\eta}_0$.

$$\frac{\Delta V[V]}{V_0} = A_3\left(\Delta\sqrt{\rho\eta}\right)^3 + A_2(\Delta\sqrt{\rho\eta})^2 + A_1\left(\Delta\sqrt{\rho\eta}\right) \quad (8)$$

$$A_1 = 2{,}07 \cdot 10^{-1}/\left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)$$

$$A_2 = -5{,}22 \cdot 10^{-2}/\left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)^2$$

$$A_2 = 4{,}58 \cdot 10^{-3}/\left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)^3$$

Figure 6 graphically shows the curve together with the experimental data. The coefficient of determination of the fitting is R^2 = 0.9954. On the other hand, regarding the standard deviation of the voltage samples (table 3), it does not increase for high $\Delta\sqrt{(\rho\eta)}$ values (noise does not increase). The worst case value is of the order of 0,5mV for a 8% of glycerol in water.

Regarding the resolution of the phase detection system, it can be estimated by making a piecewise linear approximation of the curve in Figure 6 and using the worst-case standard deviation determined for the voltage samples in the different glycerol solutions during calibration. If the linear approximation in two sections shown in Figure 7 is made, a sensitivity of $99{,}6\ mV/\sqrt{g/cm^3 \cdot cp}$ is obtained for variations in the liquid less than $1{,}39\sqrt{g/cm^3 \cdot cp}$ (R^2 = 0.9725) and of $18\ mV/\sqrt{g/cm^3 \cdot cp}$ for variations greater than $1{,}39\sqrt{g/cm^3 \cdot cp}$ and up to $5{,}62\sqrt{g/cm^3 \cdot cp}$ (R^2 = 0.9313). Therefore, using the worst case standard deviation of 0.5 mV, it can be estimated that the implemented system has a resolution of the order of $2.8 \cdot 10^{-2}\sqrt{g/cm^3 \cdot cp}$ to changes in the product density by viscosity of the liquid greater than $1{,}39\sqrt{g/cm^3 \cdot cp}$ and of $0{,}5 \cdot 10^{-2}\sqrt{g/cm^3 \cdot cp}$ for changes less than that value.

Although the sensitivity and resolution to changes in fluid properties deteriorate for higher values of the density-viscosity product, the system allows real-time measurements with high viscosities, for which the oscillators present operating problems.

3.2. Comparison with two other QCM measurement methods: oscillators and network analyzer

In [~~8~~9] and [~~9~~10] the authors characterized an Active Bridge Oscillator (ABO) oscillator and a network analyzer, respectively, for use as electronic interfaces for measuring the square root of the density-viscosity product of a liquid in contact with a QCM resonator. In both cases, a 9MHz Inficon resonator similar to the one in the present work was used. It should be noted, as the first aspect to highlight in the comparison, that the frequency sensitivity curves against changes in the square root of the density-viscosity product of the liquid obtained with both the network analyzer and the ABO oscillator are linear. However, the phase detector curve is a polynomial function of order three.

On the other hand, the network analyzer allows passive measurements in highly viscous media where the oscillators have performance problems due to non-compliance with the Barkhausen conditions. However, the phase detection method also allows measurements in more dampening media than oscillators, as it does not have the performance issues of oscillators with low-quality factor resonators.

The advantage of the passive method using a network analyzer is that it allows to extract effectively the parameters of the electrical model of the resonator (extended BVD model). The QCM resonator is measured in isolation and no external circuit influences the electrical behavior of the sensor. However, it presents difficulties when it comes to following the evolution of experiments in real time, since it requires a relatively long measurement time to obtain each sample.

The main advantage of the phase method over passive measurement is the sampling rate. As with oscillators, a real-time measurement of the variations of the magnitude to be measured can be obtained. On the other hand, as in the case of oscillators, the circuits can be relatively simple and inexpensive compared to the equipment associated with passive measurement, which is more expensive and large, making them more suitable for in-situ or remote measurements.

Regarding the resolution to changes in the liquid, in [~~9~~10] the values for the Miller and ABO oscillator topologies determined by the authors in previous works are collected. It can be seen that the resolution of the oscillators at 9 MHz (ABO: $12{,}4 \cdot 10^{-4}\sqrt{g/cm^3 \cdot cp}$; Miller: $4{,}6 \cdot 10^{-4}\sqrt{g/cm^3 \cdot cp}$) is better than the resolution of the phase detection system ($50 \cdot 10^{-4}\sqrt{g/cm^3 \cdot cp}$) in the most favorable range.

4. Summary and conclusions

This paper presents the implementation and calibration of a phase detection circuit as a QCM sensor for measuring the properties of a liquid (density and viscosity) using a 9 MHz QCM resonator. The direct digital synthesizer AD9913 was chosen from Analog Devices to generate the system test frequency. The AD9913 / PCBZ evaluation board was used with an external 25 MHz crystal as the device clock oscillator and a 2x PLL multiplier to obtain a frequency around QCM resonance (9 MHz). Its short-term stability

was determined by calculating the Allan deviation, obtaining adequate stability. An AD8302 from Analog Devices was used as phase detector. Before implementing the system, the relationship between the phase shift at the detector inputs and the output voltage was experimentally determined. The excitation frequency was chosen by studying the frequency response of the system in a resonant environment, with the QCM sensor immersed in glycerol solutions of increasing viscosities. Subsequently, the sensitivity equation of the output voltage to changes in $\Delta\sqrt{\rho\eta}$ of the liquid in contact with the QCM resonator was determined, finding that the system follows a third-order polynomial curve. To validate the operation of the circuit, the output voltage was converted into phase shift using the experimental equation obtained for the AD8302 IC. Using the equivalent electrical model of the QCM sensor loaded with the liquid, the expected theoretical phase shift for each mixture was determined, verifying that the sensor system works correctly. In addition, the resolution of the phase detection circuit was estimated, finding a value of $0{,}5 \cdot 10^{-2}\sqrt{g/cm^3 \cdot cp}$ for liquids with viscosities lower than 5.3 cp and $2{,}8 \cdot 10^{-2}\sqrt{g/cm^3 \cdot cp}$ for liquids between 5.3 cp and 37 cp.

The results were compared with those obtained by the authors in previous works using oscillator circuits and a network analyzer as electronic measurement interfaces, finding that the phase detection method is a method to be considered when it is intended to work in real time, in highly damping media, where the QCM resonator quality factor is very low and the oscillators exhibit performance problems. Although the resolution found to the changes in the liquid ($\Delta\sqrt{\rho\eta}$) for the phase detector circuit is less than the resolution of the oscillators, the circuit is able to withstand higher damping.

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Author Biographies

Jacobo Costas Costas received his M.S. degree in telecommunications engineering in the University of Vigo in 2015, and is currently studying a master’s degree in microelectronics in the University of Sevilla. Nowadays he works as an HW development engineer, in INDRA SISTEMAS S.A., particularly in FPGA development for Defense and Transport projects. He is author of A Dedicated Hardware Implementation for Biometric Recognition Based on Finger Veins (2019).

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Hubert Perrot received an Engineering degree in Chemistry in 1986 (Université de Lyon) and a PhD in Microelectronics in 1990 (Ecole Centrale de Lyon). He involved during this period in the development of chemical sensors based on chemically modified ISFET. After a period of post-doctoral fellow with A. Barraud (Centre d'Etudes Nucléaires de Saclay), he became Research Scientist in Electrochemistry at Paris (CNRS, Sorbonne University). He is now a Research Director (CNRS), his current research is focused on instrumental developments of quartz crystal microbalance for biosensors and fuel cells.

Ana María Cao y Paz received her M.Sc. degree in mechanical-electrical engineering and Ph.D. degree from the Anahuac University, México, and the University of Vigo, Spain, in 1997 and 2010 respectively. Since 2001 she has developed different research activities at the University of Vigo. She is currently an Associate Professor in the Department of Electronic Technology. She has authored over 45 journal and conference papers. Her research interests include quartz crystal microbalance sensors and fiber optic sensors.

José Fariña received the M.Sc. and Ph.D. degrees in electrical engineering from the University of Santiago de Compostela, Spain, in 1984 and 1989, respectively. He is currently an Associated Professor with the Department of Electronic Technology, University of Vigo, Spain. He has authored more than 110 journal and conference articles and holds several Spanish, European, and U.S. patents. His current research interests include development of electronic oscillators for quartz crystal microbalance sensor and the implementation of complex control and intelligent sensors in embedded platforms.

Daniel Rose received his bachelor's degree in electrical engineering in 1990 (University of Technology of Troyes). In 1991 he became an aeronautical technician at Nangis Airfield. Between 1999 and 2010 he worked as a CNRS electronics technician at LISE (Sorbonne University, Paris). Since 2010 he is CNRS electronics engineer assistant at LISE (Sorbonne University, Paris). He works in instrumentation related to electrochemistry.

Table 1

Medium	R_Q (Ω)	L_Q (mH)	C_Q (fF)	C_p (pF)	f_S (MHz)	Q
Air	5,5	6,6257	47,263	20,517	8,993714	68075
Distilled water	149,3	6,6288	47,263	28,798	8,991672	2509

Table 2.

Approximate fraction of glycerol by mass (%)	$\boldsymbol{\rho}$ (25℃)($\boldsymbol{g/cm^3}$)	η(25℃) (cp)	$\sqrt{\boldsymbol{\rho\eta}(25℃)}$ $\left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)$
8	1,0161	1,092	1,0534
21	1,0485	1,592	1,2920
39	1,0944	3,052	1,8276
46	1,1131	4,165	2,1531
51	1,1265	5,319	2,4478
59	1,1484	8,322	3,0914
63	1,1593	10,818	3,5413
66	1,1675	13,233	3,9305
72	1,1838	21,240	5,0144
78	1,2000	37,145	6,6765

Table 3

Approximate fraction of glycerol by mass (%)	V_{out} (mV)	
	Average value	Standard deviation
8	640,63	0,489 ~~4,89E-01~~
21	665,74	0,012 ~~1,15E-02~~
39	733,65	0,027 ~~2,72E-02~~
46	752,97	0,015 ~~1,48E-02~~
51	768,31	0,027 ~~2,74E-02~~
59	791,46	0,023 ~~2,25E-02~~
63	803,15	0,031 ~~3,07E-02~~
66	815,78	0,023 ~~2,34E-02~~
72	828,19	0,143 ~~1,43E-01~~
78	849,40	0,005 ~~5,27E-03~~

Figure 1

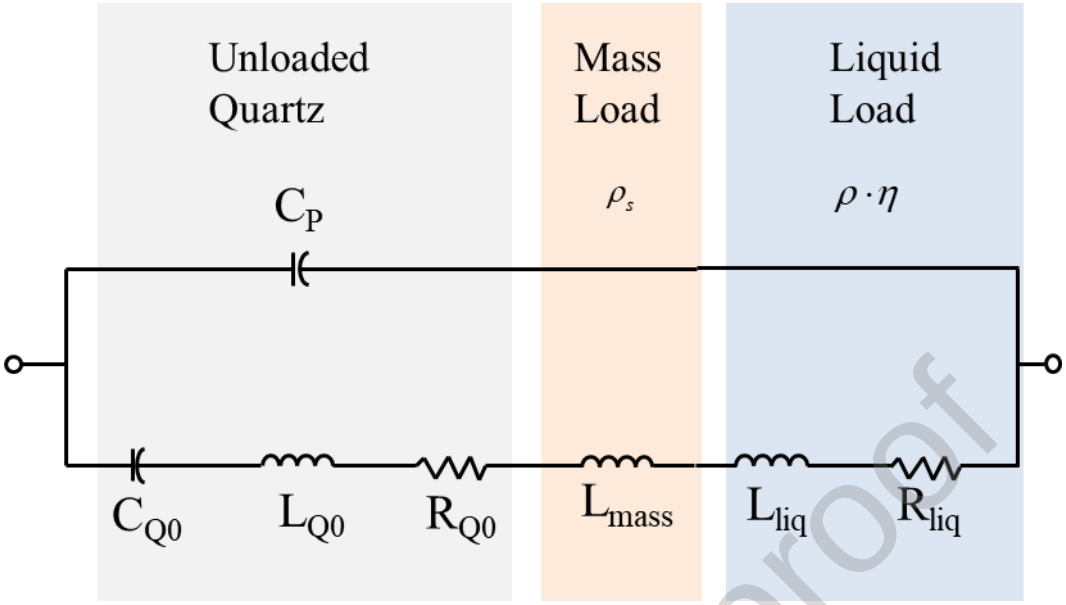


Figure 2

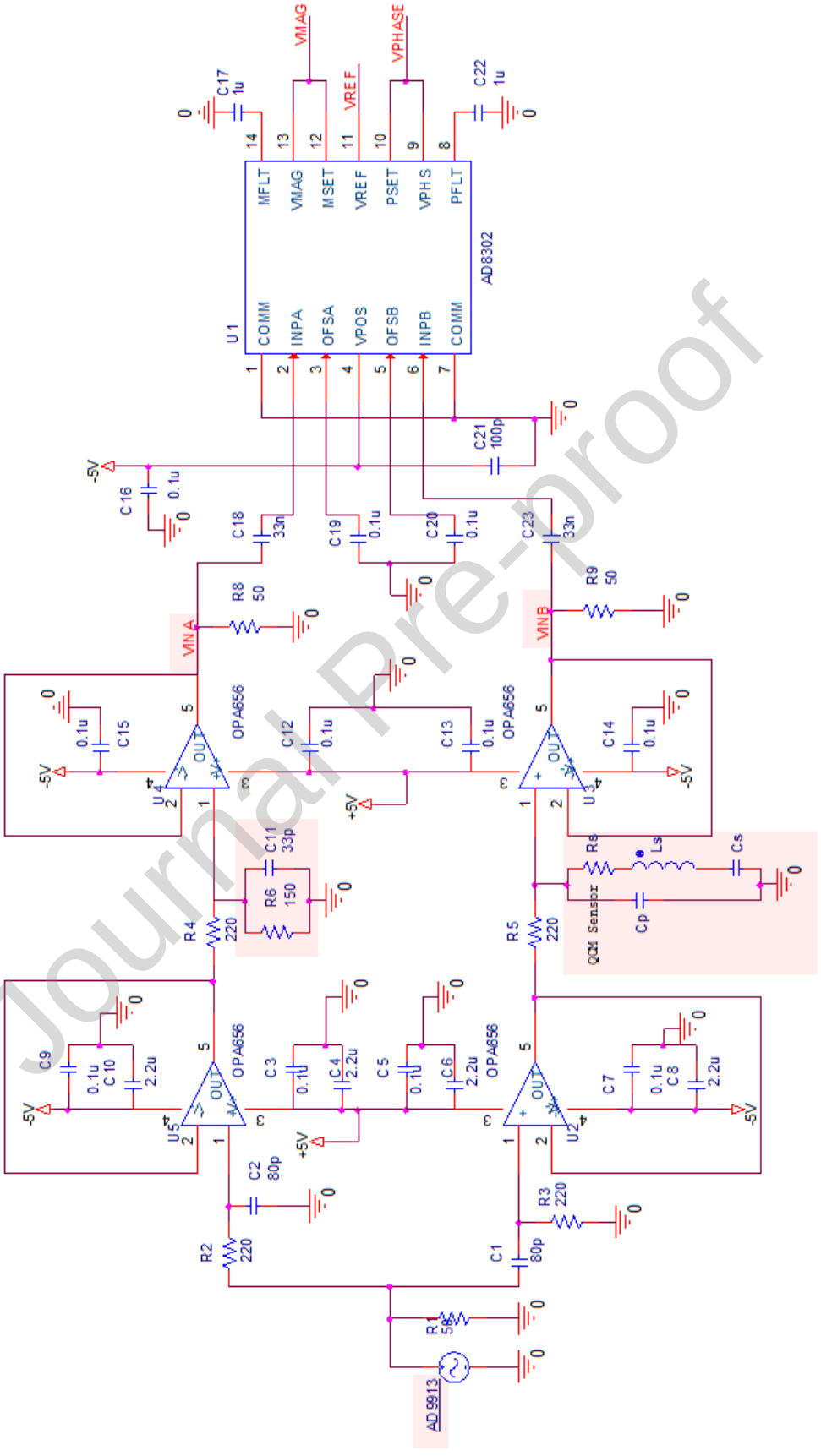


Figure 3

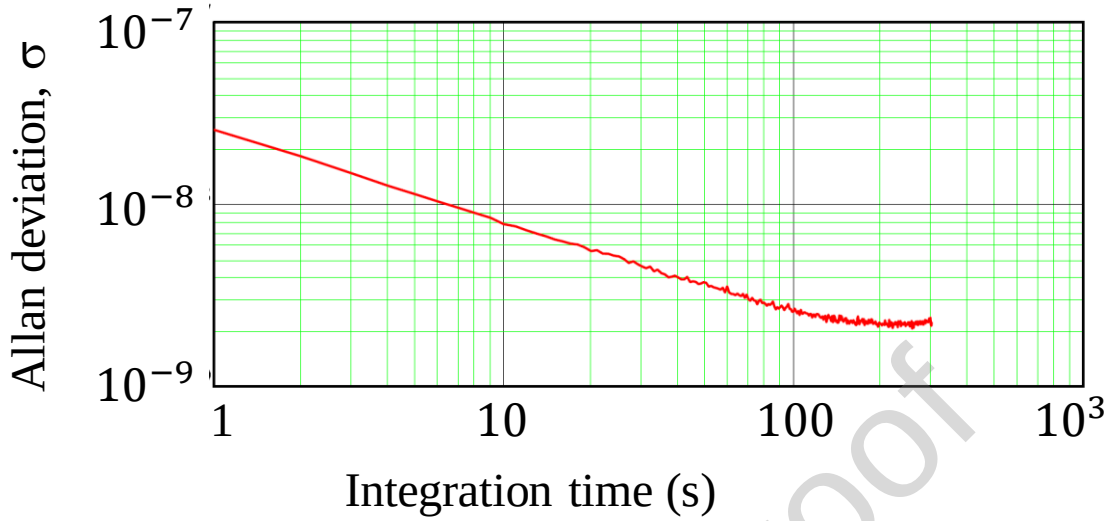


Figure 4

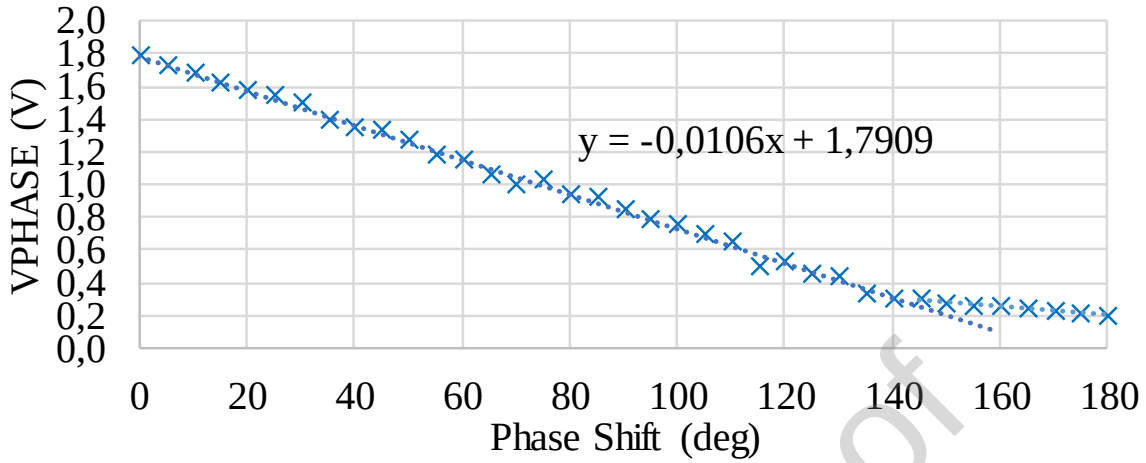


Figure 5

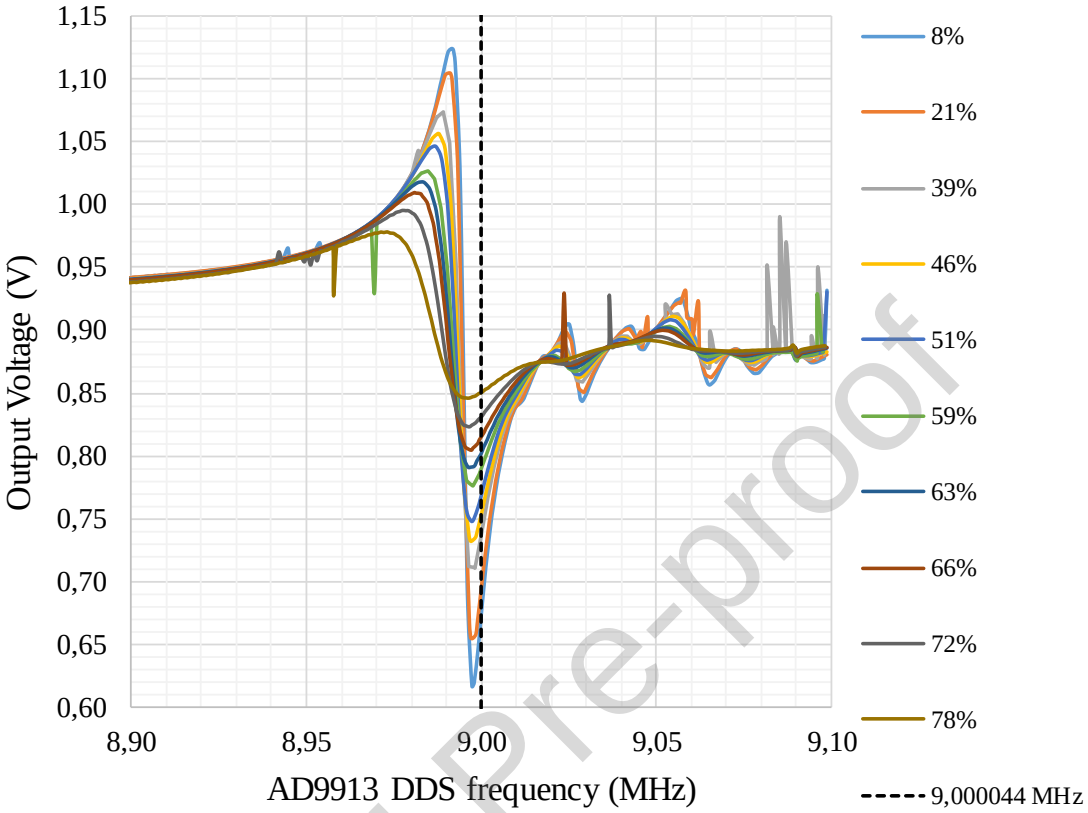


Figure 6

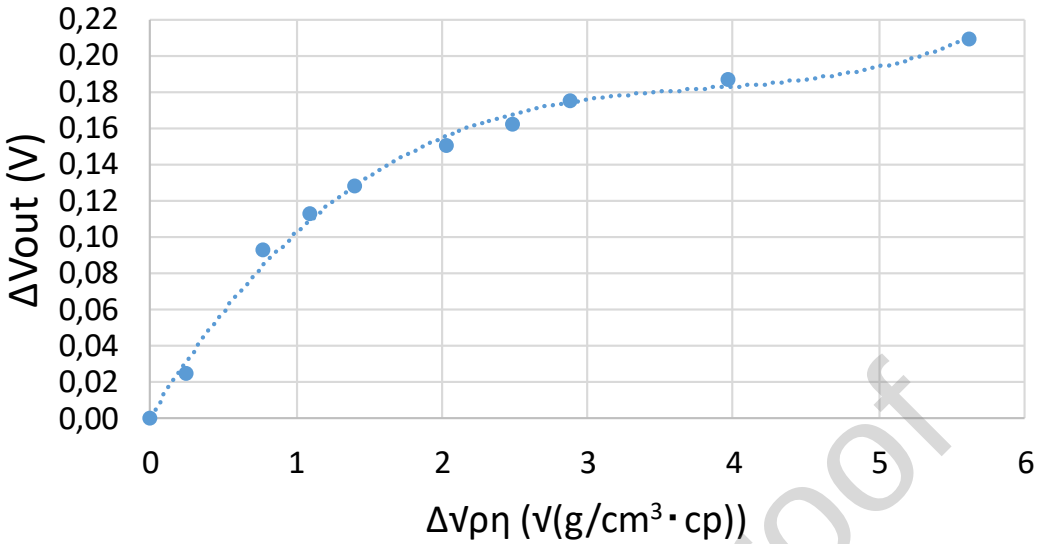
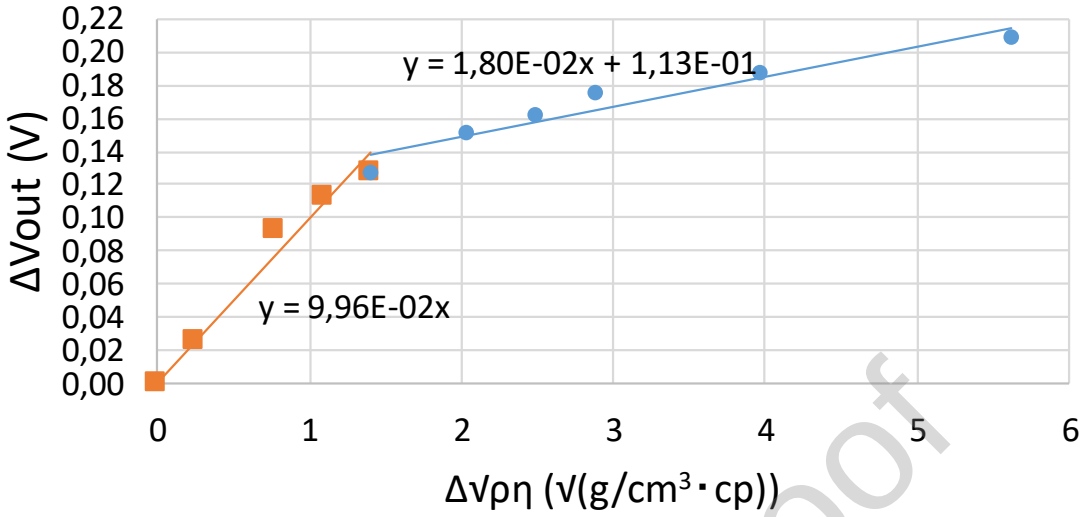


Figure 7



CRediT authorship contribution statement

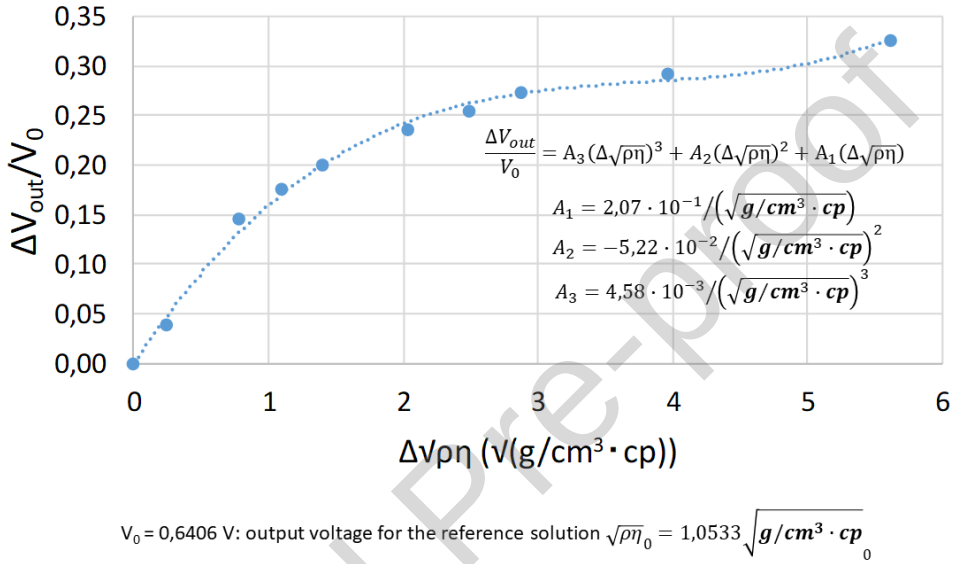
J Costas-Costas, L. Rodriguez-Pardo, H. Hubert, A.M. Cao-Paz, J. Fariña and D. Rose contributed to the design and implementation of the research, to the analysis of the results and to the writing of the manuscript.

Declaration of Competing Interest

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Graphical abstract



New Highlights

- A QCM sensor system based on a phase detector circuit is implemented.
- The system is characterized to measure the properties of a liquid.
- Calibration with solutions of increasing viscosities is carried out.
- Sensitivity equation and resolution are obtained.
- Results are compared with those obtained using oscillators and a network analyzer.