

High Precision Tuning Fork Sensor for Liquid Property Measurements

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Abstract— Application of piezoelectric flexural mechanical resonators such as tuning forks to accurate measurements of liquid physical properties is discussed. It was shown earlier that liquid properties such as viscosity, density and dielectric constant can be obtained by measuring the resonator AC impedance within certain frequency range and fitting it to the resonator equivalent circuit model [1]. Error sources for the liquid property measurements and their influence on the measured value are investigated. It is shown experimentally that the reproducibility of the viscosity and density measurements using this technique can meet and often exceed the one delivered by the well established analytical instrumentation. It is also demonstrated here that better performance is resulting from the use of the whole impedance curve over a frequency range, which produces better statistics and natural averaging of the noise.

I. INTRODUCTION

We have previously shown [1] that the complex impedance of a flexural resonator in a liquid environment could be represented by the equivalent circuit shown on Fig.1.

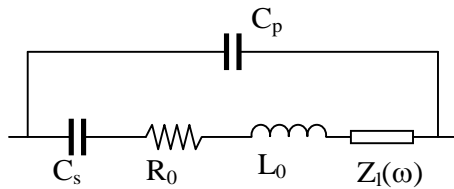


Fig. 1

The equivalent parameters C_s , R_0 , L_0 represent respectively the mechanical compliance, loss and inertia of the resonator in vacuum. An additional contribution to the impedance $Z_l(\omega)$ from the surrounding liquid is given by following relationship: $Z_l(\omega) = Ai\omega\rho + B\sqrt{\omega\rho\eta}(1+i)$, where ω is the operation frequency, ρ is the liquid density, η is the liquid viscosity, A and B are the geometry factors that depend only on the resonator geometry and mode of oscillation.

C_p is the electrical capacity of the resonator electrodes that is affected by the electrical properties of the surrounding liquid due to the fringing field. The changes in C_p can be represented by the following relationship:

$C_p(\epsilon) = C_p(1) + (\epsilon - 1)\partial C_p / \partial \epsilon$, where $C_p(1)$ is the electrodes capacitance in vacuum, ϵ is liquid permittivity, and

$\partial C_p / \partial \epsilon$ is the sensitivity to changes in the electrical properties of the environment.

It is evident from the equivalent circuit that the liquid property-dependent impedance component $Z_l(\omega)$ is not directly accessible for the measurement, therefore it is necessary to know the values of other equivalent circuit components to be able to extract the value of the component of interest. It is usually done by measuring the resonator response in vacuum, where $Z_l(\omega) = 0$ and extracting these values using standard techniques. Geometry factors $\partial C_p / \partial \epsilon$, A and B are calibrated by submerging the resonator in a liquid with known properties and fitting the measured resonator response to the equivalent circuit varying $\partial C_p / \partial \epsilon$, A and B as free parameters. Measuring the properties of an unknown liquid is done by fitting the measured resonator response to the equivalent circuit varying ϵ , ρ and η as free parameters[2].

II. PRELIMINARY DISCUSSION

The impedance of the flexural resonator depends on the frequency and following parameters: vacuum parameters C_s , R_0 , L_0 ; geometry factors $\partial C_p / \partial \epsilon$, A , B and liquid parameters of interest ϵ , ρ and η . Once the vacuum parameters and geometry factors are calibrated, the impedance of the resonator depends only on frequency and the three liquid properties that are found by measuring the resonator impedance in the unknown liquid. To be able to calculate the three unknown parameters the impedance value has to be measured at several different frequencies. In practice it is easier to measure the absolute value of the complex impedance, so from now on we will use $Z(\omega)$ to depict the absolute value of the complex resonator impedance.

In the functional form we have a system of equations:

$$Z_{\omega_n} = Z(\omega_n, \epsilon, \rho, \eta), \quad (1)$$

where Z_{ω_n} is the impedance absolute value measured at a frequency ω_n . Obviously, depending on properties of the $Z(\omega)$ function, it is necessary to have at least three measured values to be able to solve this system of equations for the unknown ϵ , ρ and η . Since the measured value of Z_{ω_n} always includes some error ΔZ , the accuracy of the solution may strongly depend on the choice of the frequency points ω_n at which the impedance values were measured. Any measurement error and the resulting errors in the parameters can be related in the following manner. Assuming that the impedance error

magnitude is considerably less than the impedance value, it can be represented accurately enough by a Taylor series retaining only linear terms and neglecting all higher order terms. In this case:

$$\Delta Z \approx \frac{\partial Z}{\partial \varepsilon} \Delta \varepsilon + \frac{\partial Z}{\partial \rho} \Delta \rho + \frac{\partial Z}{\partial \eta} \Delta \eta,$$

where ΔZ and all derivatives are functions of ω and all other parameters described above. The inverse value of the appropriate partial derivative is the corresponding parameter sensitivity to the error in the impedance.

The frequencies at which the impedance measurements are performed are usually quite accurate due to availability of high quality AC sources. Nevertheless, if contribution of frequency error $\Delta \omega$ needs to be considered, one more term has to be added to the expression above: $(\partial Z / \partial \omega) \Delta \omega$.

III. CASE STUDY

To illustrate the sensitivity of the liquid parameters measured with flexural resonator to the error in the impedance measurements and the choice of frequencies let's consider a typical 32.768 KHz quartz tuning fork. The equivalent parameters of such a fork are shown in Table I. C_p value includes the capacitance of the mounting, typical value for the fork itself is 1.5 pF

TABLE I. PARAMETERS OF A TYPICAL QUARTZ TUNING FORK

Vacuum	Value	Geometry	Value
C_p , pF	4.92	$dC_p/d\varepsilon$, pF	0.171
L_s , H	6723	A, $\Omega \cdot \text{cm}^3 \cdot \text{s} \cdot \text{g}^{-1}$	1643
R_s , Ω	41740	B, $\Omega \cdot \text{cm}^{3/2} \cdot \text{s}^{1/2} \cdot (\text{g} \cdot \text{cP})^{-1/2}$	42679
C_s , pF	0.0351		

The sensitivity of corresponding liquid parameters to the error in the impedance measurement has been calculated for this particular tuning fork and a liquid with dielectric constant of 3, density of 0.79 g/ccm and viscosity of 10 centipoise for a frequency range around the resonance, Fig. 2.

The error sensitivity of the dielectric constant measurements is a relatively smooth function of frequency, while the sensitivity of both density and viscosity turn into infinity at two particular frequency points. Even a small error in the impedance measurements near these frequencies would be highly amplified. Meanwhile choosing frequencies between these singularity points where the sensitivity curve approaches x-axis would result in nearly minimal achievable measurement errors. Finding such optimal measurement frequencies is problematic in practice since the location of these sensitivity singularities depends on that very density and viscosity of the unknown liquid we are trying to measure.

The most obvious way of dealing with such difficulties would be to over-define the system of functional equations (1) by adding more impedance measurements from a frequency range wide enough to ensure the presence of optimal

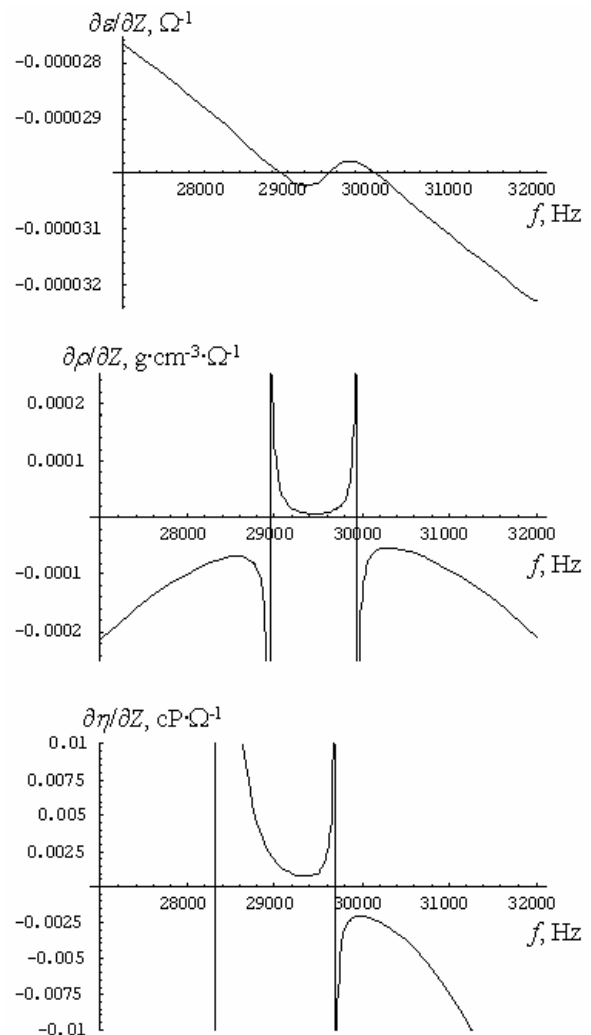


Fig. 2

measurement ranges within the ensemble and to apply statistical data reduction techniques.

IV. CURVE FITTING USING LEAST SQUARE METHOD

For solving such over-defined problem least square method is the most straightforward [3]. The error in the impedance measurement ΔZ and the error in the measured parameter Δx (x represents dielectric constant, density or viscosity) can be related as follows:

$$\int_{f_0}^{f_1} (Z + \Delta Z - Z(x + \Delta x))^2 df \xrightarrow{x} \min,$$

where Z represents "ideal" (error-free) experimental curve, $Z(x)$ represents model of the impedance as a function of liquid parameter x , which can be found with infinite accuracy, so that $Z = Z(x)$. With the errors ΔZ and Δx this integral will be of some finite positive value.

Assuming that the error Δx is small, the expression above can be written in the following way:

$$\int_{f_0}^{f_1} \left(Z + \Delta Z - Z(x) - \frac{\partial Z}{\partial x} \Delta x + o(\Delta x^2) \right)^2 df \xrightarrow{x} \min$$

Neglecting the second order and higher order terms of Δx and taking into account that $Z = Z(x)$ it can be re-written as follows:

$$\int_{f_0}^{f_1} \left(\Delta Z - \frac{\partial Z}{\partial x} \Delta x \right)^2 df \xrightarrow{x} \min$$

The derivative of the integral above by x should be zero at the minimum:

$$\frac{\partial}{\partial x} \int_{f_0}^{f_1} \left(\Delta Z - \frac{\partial Z}{\partial x} \Delta x \right)^2 df = 0;$$

$$\frac{\partial}{\partial x} \int_{f_0}^{f_1} \left(\Delta Z - \frac{\partial Z}{\partial x} \Delta x \right)^2 df =$$

$$= \int_{f_0}^{f_1} \left(-2\Delta Z \Delta x \frac{\partial^2 Z}{\partial x^2} + 2\Delta x^2 \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} \right) df = 0,$$

$$\Delta x \neq 0 \Rightarrow \int_{f_0}^{f_1} \left(-\Delta Z \frac{\partial^2 Z}{\partial x^2} + \Delta x \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} \right) df = 0$$

The last integral equation provides the relationship between error in the impedance measurement and error in liquid property.

In general both errors ΔZ and Δx are functions of the frequency at which the impedance measurement is taken. The "average" frequency-independent liquid property error can be

$$\text{defined as } \Delta x \int_{f_0}^{f_1} \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df = \int_{f_0}^{f_1} \Delta x(f) \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df .$$

The impedance error ΔZ is almost always frequency-dependent (random noise, frequency-dependent gain, non-linearity, etc.), unless it is an offset that can easily be subtracted from the data or adjusted close to zero. Nevertheless, the "average" frequency-independent impedance error can be formally defined as

$$\Delta Z \int_{f_0}^{f_1} \frac{\partial^2 Z}{\partial x^2} df = \int_{f_0}^{f_1} \Delta Z(f) \frac{\partial^2 Z}{\partial x^2} df .$$

In this case the relationship between the average errors can be written as follows:

$$\Delta x \int_{f_0}^{f_1} \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df = \Delta Z \int_{f_0}^{f_1} \frac{\partial^2 Z}{\partial x^2} df .$$

For the values listed in the table above and a frequency range of 27-32 kHz the relationship between the average errors is calculated:

$$\Delta Z = -33442 \cdot \Delta \varepsilon, \quad \Delta Z = 59939 \cdot \Delta \rho, \quad \Delta Z = 9121 \cdot \Delta \eta$$

This means that the absolute average error of ~3.3 k Ω in impedance will cause an error of ~0.1 in the measured dielectric constant. By the same token, an absolute average error of ~6 k Ω in impedance will cause ~0.01g/ccm error in density and an impedance error of ~900 Ω will cause a viscosity error of ~0.1 cP.

V. PROPAGATION OF CALIBRATION ERRORS

The approach described above allows calculation of the influence of errors developed in the course of sensor calibration in vacuum and in a known liquid standard. In

vacuum $\int_{f_0}^{f_1} \Delta x_v \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df = \int_{f_0}^{f_1} \Delta Z \frac{\partial^2 Z}{\partial x_v^2} df$, where ΔZ is the

error at given frequency point f and Δx_v – error in one of the equivalent circuit element values x_v : C_s , L , R or C_p ; subscript "v" in the integration limits depicts the frequency range in vacuum mode. As it was done before average (frequency-independent) error Δx_v of a parameter value is defined as

$$\text{follows: } \Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df = \int_{f_0}^{f_1} \Delta x_v(f) \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df .$$

The cumulative error in impedance produced by errors in the vacuum parameters can be represented by the following

sum: $\Delta Z = \sum_{v=1}^4 \frac{\partial Z}{\partial x_v} \Delta x_v$, and, therefore, the error in the

measured parameter produced by errors in vacuum calibration can be written as follows:

$$\int_{f_0}^{f_1} \Delta x \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df = \sum_{v=1}^4 \left(\Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df \right) .$$

Similarly, all errors in liquid-calibrated parameters ΔA , ΔB , $\Delta(dC_p/d\varepsilon)$ acquired during a liquid calibration procedure produce overall error in measured parameter that can be calculated as follows:

$$\int_{f_0}^{f_1} \Delta x \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df = \sum_{i=1}^3 \left(\Delta x_i \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_i} \frac{\partial^2 Z}{\partial x_i^2} df \right), \quad (2)$$

where and Δx_i is one of the three parameters itemized above.

In practice, vacuum calibration precedes liquid calibration, and all errors acquired during vacuum calibration propagate through the process into errors in the liquid calibration parameters. Assuming for simplicity that the frequency sweep is same as in the measurement mode, the relationship between errors in liquid calibration parameters and errors in vacuum calibration parameters can be written as follows:

$$\int_{f_0}^{f_1} \Delta x_i \frac{\partial Z}{\partial x_i} \frac{\partial^2 Z}{\partial x_i^2} df = \sum_{v=1}^4 \left(\Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df \right) .$$

As it was done before, average (frequency-independent) error Δx_i of a liquid-calibrated parameter value is defined as follows:

$$\Delta x_i \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_i} \frac{\partial^2 Z}{\partial x_i^2} df = \int_{f_0}^{f_1} \Delta x_i(f) \frac{\partial Z}{\partial x_i} \frac{\partial^2 Z}{\partial x_i^2} df \text{ and}$$

$$\Delta x_i = \frac{\sum_{v=1}^4 \left(\Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_v^2} df \right)}{\int_{f_0}^{f_1} \frac{\partial Z}{\partial x_i} \frac{\partial^2 Z}{\partial x_i^2} df}$$

Δx_i in the formula (2) should be substituted by this expression to find the cumulative error in the measured parameter, which gives the final expression below:

$$\int_{f_0}^{f_1} \Delta x \frac{\partial Z}{\partial x} \frac{\partial^2 Z}{\partial x^2} df = \sum_{l=1}^3 \left(\Delta x_l \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_l} \frac{\partial^2 Z}{\partial x^2} df \right) +$$

$$+ \sum_{l=1}^3 \left(\frac{\int_{f_0}^{f_1} \frac{\partial Z}{\partial x_l} \frac{\partial^2 Z}{\partial x^2} df}{\int_{f_0}^{f_1} \frac{\partial Z}{\partial x_l} \frac{\partial^2 Z}{\partial x_l^2} df} \sum_{v=1}^4 \left(\Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x_l^2} df \right) \right) +$$

$$+ \sum_{v=1}^4 \left(\Delta x_v \int_{f_0}^{f_1} \frac{\partial Z}{\partial x_v} \frac{\partial^2 Z}{\partial x^2} df \right)$$

These 3 terms are, again:

1. The error introduced in the measured parameter by error acquired during calibration in liquid
2. The error introduced in the measured parameter by the error in liquid parameter resulting originally from the error in vacuum calibration
3. The error in the measured parameter introduced by error in vacuum calibration.

VI. EXPERIMENTAL MEASUREMENTS OF REPRODUCIBILITY

An experimental investigation of the measurement reproducibility has been performed with the sensor assembly shown on Fig. 3 using a Cannon™ CT-1000 Constant Temperature Bath. The quartz tuning fork is mounted at the top end of the extension PCB to provide better temperature equilibration, which is very important for high-precision viscosity measurements.



Fig. 3

The measurements were performed on a set of Cannon™ Viscosity Standards - mineral oils of well characterized density and viscosity. Five test tubes containing oil samples of different density and viscosity were equilibrated in the constant

temperature bath at 50°C for 4 hours. The sensor probe was cleaned in HPLC-grade hexanes, dried by nitrogen flow, introduced into the sample and equilibrated for 30 minutes. The sensor impedance curve consisting of 201 points was then acquired within 27-32 KHz frequency range and processed using least square routine. The calculated values of density and viscosity were recorded. After cleaning and drying, the sensor probe was introduced into second sample, equilibrated and measurements taken. After the last sample was measured, the process was repeated 9 more times. The value of the percent peak-to-peak scatter in the measured density and viscosity was calculated for each sample. The results are shown in Table II.

TABLE II. MEASUREMENT-TO-MEASUREMENT RESULTS SCATTER IN EACH CALIBRATION STANDARD AT 50°C

<i>Cannon™ standard</i>	<i>Density, g/ccm</i>	<i>Scatter, %</i>	<i>Viscosity, cP</i>	<i>Scatter, %</i>
S3	0.8403	0.144	2.008	0.352
S6	0.8559	0.196	3.757	0.407
S20	0.8458	0.064	10.65	0.187
N35	0.8492	0.041	18.25	0.298
S60	0.8566	0.098	29.72	0.256

These results demonstrate that the repeatability of the described technique is substantially better than 1% for a wide range of liquid viscosity. This repeatability approaches and in some cases exceeds the performance of well-established analytical methods as defined by ASTM standards, D445 in particular [4]. The relatively small size of this sensor allows for considerably smaller sample volume, faster thermal equilibration, shorter measurement cycle times, and convenient cleaning compared to rotary and capillary viscometers.

ACKNOWLEDGMENT

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