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Modelling and Experiments on a Quartz Crystal Resonator Sensor for Conductivity Measurements of Low-Concentration Ionic Solutions

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Abstract: The monitoring and control of the electrical parameters of ionic solutions have different applications in industry and are particularly important in medical and biochemical studies, where ionic solutions are commonly used. This paper analyzes the effect of the conductivity of a low-concentration ionic solution on the resonant response of a Quartz Crystal Resonator (QCR) sensor with asymmetric electrodes put in contact with the solution. A model of the QCR sensor is presented, and experimental results obtained with a 6-MHz sensor exposed to low-concentration solutions of NaCl in water are reported. The model and experimental results are in very good agreement throughout the explored conductivity range between around 10 and 200 mS/m.

Keywords: Conductivity, low-concentration ionic solution, Quartz Crystal Resonator (QCR) sensor, impedance analysis.

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1. Introduction

Quartz Crystal Resonator (QCR) sensors can operate in contact with a liquid due to the comparatively low losses associated with the vibration of the crystal in Thickness Shear Mode (TSM) [1].

Usually, in the applications that use QCR sensors in liquid, the influence of the conductivity and the dielectric properties of the solution are parasitic contributions that can interfere with the measurement [2]. This aspect is of particular importance in chemical and biomedical applications, where ionic solutions are commonly used [3-4].

These parasitic effects can be avoided by eliminating any direct interface between the solution and the bare quartz crystal. This is accomplished by making the electrode in contact with the solution large enough to cover the whole crystal area exposed to the liquid. This produces a screening action that prevents the electric field from penetrating into the solution, therefore the electrical characteristics of the liquid are not influential.

There are cases where it would be interesting to monitor the electric properties of the solution. In such cases, it is possible to use a sensor configuration in which the electrode area is smaller than the area exposed to the solution, therefore generating an electrode-liquid-quartz electrical path that goes in parallel with the sensor itself [5].

This paper focuses on the modelling of a QCR sensor with asymmetric electrodes in contact with a liquid and demonstrates that, by considering an appropriate sensor characteristic frequency, i.e. the frequency at maximum conductance, an analytical relationship with the liquid conductivity can be derived that agrees well with experimental results.

2. Sensor Configuration

The sensor configuration used in this study is an AT-cut quartz crystal with the electrodes shapes shown in Figure 1. This quartz crystal has a smaller disc electrode (A) that occupies partially one face, and a larger disc electrode (B) that completely covers the other face. The linked semicircular patterns on the small-electrode face serve to route electrical contacts out of the crystal.

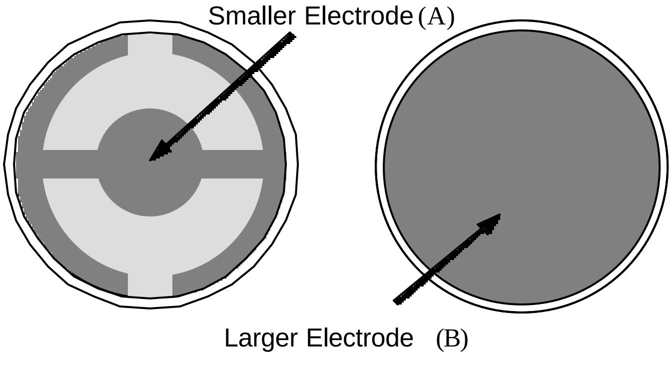

Fig. 1. Quartz crystal with asymmetric electrode configuration.

The crystal is intended to operate in a measurement cell as shown in Figure 2. It can be observed that the upper crystal face in contact with the liquid is the one with the smaller electrode A, while the lower face is that with the larger electrode B.

The asymmetric electrode configuration causes a fringing electric field E to penetrate into the liquid, determining a probing action that can be exploited to sense liquid properties.

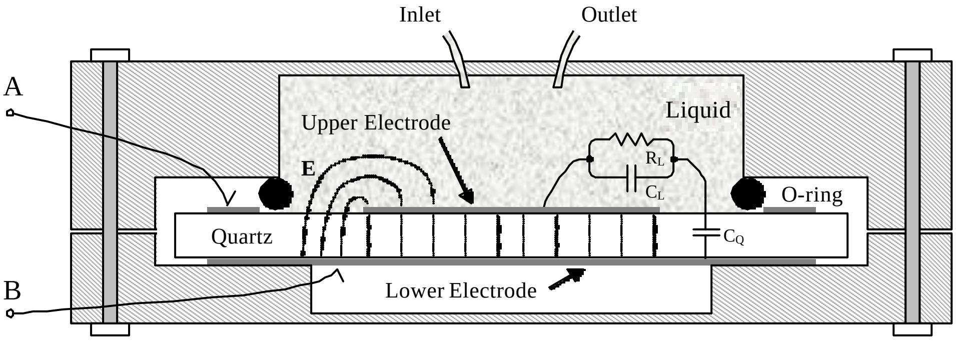

Fig. 2. QCR sensor mounted in the measurement cell.

The impedance of the liquid is represented by the parallel of $R_L = 1/(k\,\sigma)$ and $C_L = k\varepsilon$, where s and e are the liquid conductivity and dielectric permittivity respectively, and k is an appropriate form factor having the units of linear length. The capacitance $C_Q = \varepsilon_Q A_Q / t$ represents the contribution of the outside region of bare crystal, where ε_Q is the quartz permittivity, A_Q is the area of the portion of the upper face falling inside the measurement cell and uncovered by the electrode A, and t is the crystal thickness.

When the liquid is an ionic solution, primarily the conductivity σ becomes a function of the ionic concentration, while, in particular with water low-concentration solutions, the permittivity ε is affected to a lower extent. Increasing s produces the effect of widening of the effective area of the upper electrode, with the consequent variation of the sensor electric admittance [2, 5-6]. The variation of the sensor admittance around resonance can be therefore exploited to measure the liquid conductivity.

3. Theoretical Model

Figures 3a) and 3b) respectively show QCR sensors with equal and asymmetric electrodes with the upper face in contact with a liquid and with the corresponding equivalent circuits valid around the crystal fundamental resonance.

The circuit of Figure 3a) is the classic Butterworth-Van Dyke (BVD) model, where L, C and R represent the equivalents of motional mass, compliance and losses, and C_0 is the electrical capacitance of the crystal.

The circuit of Figure 3b) is an extension of the BVD model where the components R_L, C_L and C_Q, previously described in section 2, have been included.

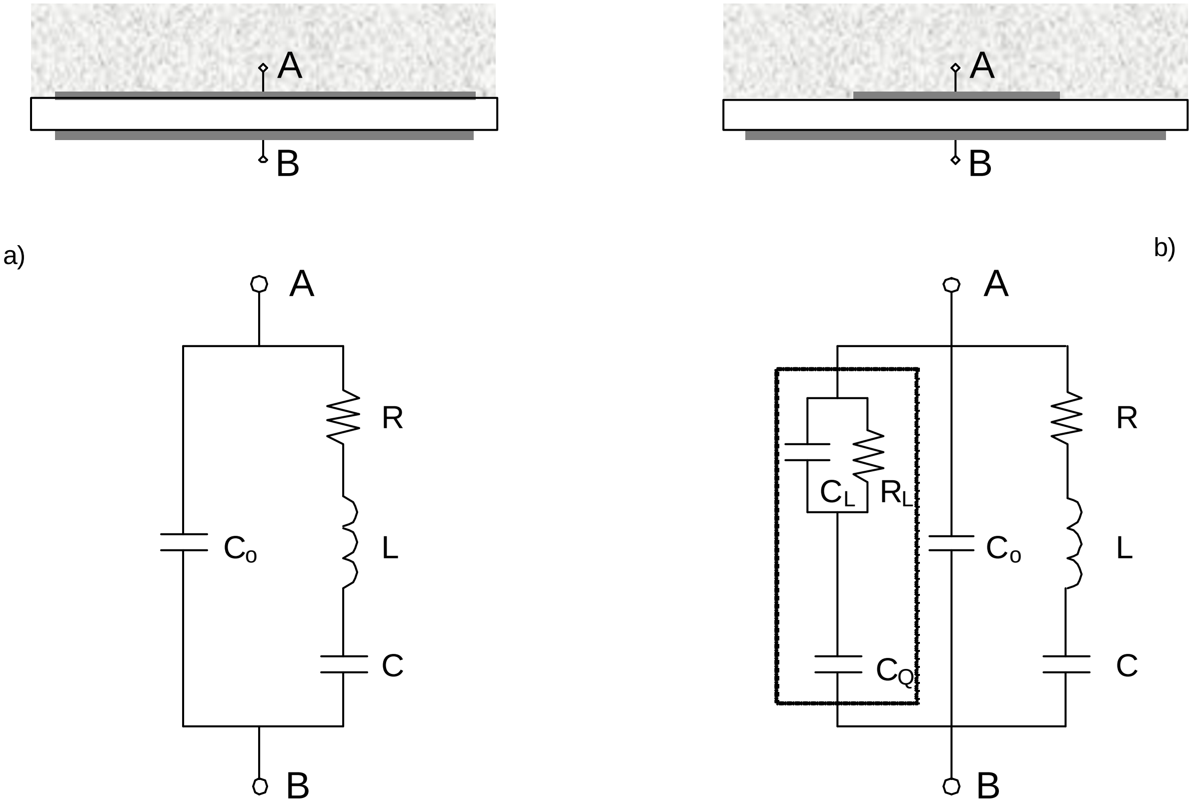

Fig. 3. QCR sensor with equal (a) and asymmetric (b) electrode configurations with one face in contact with a liquid and corresponding equivalent electric circuits.

For the classical BVD model of Figure 3a), the admittance $Y(\omega)$ measured between terminals A and B is given by:

$$Y(\omega) = Y_m(\omega) + j\omega C_0 \tag{1}$$

The motional component $Y_m(\omega)$ is:

$$Y_m(\omega) = \frac{1}{R + \frac{1}{j\omega C} + j\omega L} \tag{2}$$

For the extended BVD model of Figure 3b), the admittance $Y(\omega)$ is now given by:

$$Y(\omega) = Y_m(\omega) + j\omega C_0 + Y_L(\omega)\,, \tag{3}$$

where $Y_L(\omega)$ is caused by the fringing field and has the following expression:

$$Y_L(\omega) = \frac{1}{\frac{R_L}{1 + j\omega C_L R_L} + \frac{1}{j\omega C_Q}} \tag{4}$$

The resonant angular frequency of the motional arm $\boldsymbol{w}_S = (1/LC)^{1/2}$, called the series resonant frequency, is the primary parameter that is commonly measured with QCR sensors in general and for in-liquid analysis in particular. This is because w_S is related to the mass uptake in a coated crystal, to the viscoelastic properties of the coating, and also to the density-viscosity product of the contacting liquid [1]. The resistance R is also important to quantify losses [1].

Considering the BVD model of Figure 3a), the series resonant frequency ω_S can be conveniently measured as the frequency where the conductance $G(\omega)$, i.e. the real part of admittance $Y(\omega)$, is maximum. This can be easily done with an impedance analyzer.

In the case of Figure 3b), with the extended BVD model, the presence of $Y_L(\omega)$ does not modify the series resonant frequency w_S. However, depending on which other characteristic frequency of the sensor is monitored, either for reasons of convenience or due to the intrinsic operation of the oscillator or different electronic equipment used, there might be significant variations caused by Y_L. This is the case, for instance, with the parallel resonant frequency and the zero-phase frequency that are both affected by Y_L in a complicated way difficult to be expressed in analytical form [5-6].

As an alternative, we propose to extend the method of the maximum-conductance frequency to the case of the presence of Y_L by further deepening into the analysis of the model of Figure 3b).
The expression of the conductance $G(\omega)$ is:

$$G(\omega) = G_m(\omega) + G_L(\omega) = \frac{\omega^2 RC^2}{(1-\omega^2 LC)^2 + \omega^2 R^2 C^2} + \frac{\omega^2 R_L {C_Q}^2}{1 + \omega^2 {R_L}^2 (C_L + C_Q)^2} \tag{5}$$

The frequency-dependent term $G_L(\omega)$ causes the frequency ω_G for which $G(\omega)$ is maximum to be in principle different from w_S. However, G_L becomes constant for R_L either equals to zero or infinity, i.e. for a liquid either insulating or perfectly conductive. In both cases it follows that $w_G = w_S$.

For an intermediate conductivity, a conservative approximation can be derived by assuming that R_L has the value for which the slope of $G_L(\omega)$ is maximum at $w = w_S$. This occurs for:

$$R_L (C_L + C_Q) = \frac{\sqrt{3}}{3\omega_s} \tag{6}$$

In this worst-case condition, an overestimated expression of ω_G can be derived from Eq.(5) resulting in:

$$\omega_G \cong \omega_s \left(1 + \frac{9 R_L {C_Q}^2}{64 RC^2 Q^4} \right), \tag{7}$$

where $Q = \omega_S L/R$ is the motional quality factor at series resonance.

In Eq.(7) the values of R_L and R can be comparable, and C_Q can be two orders of magnitude larger than C. Under these conditions, even for low values of Q in the order of few hundreds, the difference between w_G and w_S is negligibly small.

Therefore, it can be concluded that, irrespectively of the conductivity of the liquid, the angular frequency ω_G where the conductance $G(\omega)$ of Eq.(5) has a maximum is essentially equal to the motional series resonant frequency w_S. As a consequence, measuring the former is a convenient method to determine the latter. Thereafter, it will be assumed that $w_G = w_S$.

As a first consequence, given that ω_S is not dependent on Y_L, it is expected that the measured ω_G is not affected by the liquid conductivity s.

The real part G and the imaginary part B of the quartz crystal admittance at ω_S are given by:

$$G(\omega_s) = G_m(\omega_s) + G_L(\omega_s) = \frac{1}{R} + \frac{\omega_s^{\,2} C_Q^{\,2} R_L}{1 + \omega_s^{\,2} (C_L + C_Q)^2 R_L^{\,2}} \tag{8}$$

$$B(\omega_s) = B_m(\omega_s) + B_L(\omega_s) = \omega_s C_0 + \frac{\omega_s C_Q (1 + \omega_s^2 (C_L + C_Q) C_L R_L^{\,2})}{1 + \omega_s^2 (C_L + C_Q)^2 R_L^{\,2}} \tag{9}$$

For high values of the conductivity s the resistance R_L tends to zero, therefore $G_L(\omega_S)$ and $B_L(\omega_S)$ respectively become:

$$\lim_{R_L \to 0} G_L = 0 \tag{10}$$

$$\lim_{R_L \to 0} B_L = \omega_s C_Q \tag{11}$$

On the other hand, when σ tends to zero R_L diverges and $G_L(\omega_S)$ and $B_L(\omega_S)$ respectively become:

$$\lim_{R_L \to \infty} G_L = 0 \tag{12}$$

$$\lim_{R_L \to \infty} B_L = \frac{\omega_s C_Q C_L}{C_Q + C_L} \tag{13}$$

On the basis of the above equations, it is expected that, as a function of the liquid conductivity s, $G(\omega_S)$ shows a non-monotonic trend with a maximum at the value that makes $R_L = 1/[\omega_S(C_Q + C_L)]$, while $B(\omega_S)$ shows a monotonic behavior.

Summarizing, the liquid conductivity σ is expected to respectively produce no effect on the series resonant frequency ω_S, a non-monotonic trend with an intermediate maximum for the conductance $G(\omega_S)$, and a monotonic rising trend for the susceptance $B(\omega_S)$.

4. Experimental Apparatus

The used sensor is a 6 MHz AT-cut quartz crystal (Inficon 008-010-G10) with a configuration of the electrodes as shown in Figure 1. The diameters of the crystal, upper electrode (A) and lower electrode (B) are respectively 14 mm, 7 mm and 13.5 mm. The crystal was mounted in a measurement cell purposely made with the same configuration shown in Figure 2.

A block diagram of the complete experimental set-up is shown in Figure 4.

To obtain reference values of liquid conductivity, solutions of sodium chloride in deionized water were prepared in concentrations from 0.06% to 0.1% in weight. The corresponding values of the conductivity of the solutions have been calculated using the Kohlrausch law [7] obtaining values in the range between 13 mS/m and 205 mS/m.

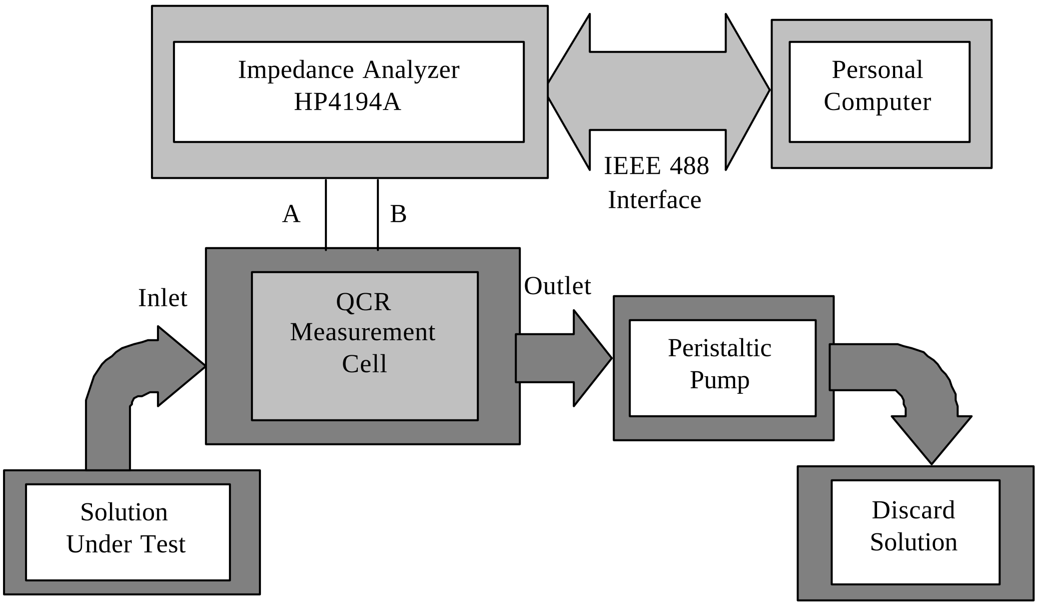

Fig. 4. Block diagram of the experimental set-up.

It is important to point out that the extremely low concentrations used produce negligible variations of the density and viscosity of water [5], therefore no effect is expected on the motional admittance Y_m, and on w_S in particular, caused by acoustic-mechanical action.

The QCR sensor, through the terminals *A* and *B*, was connected to an impedance analyzer (Hewlett-Packard HP4194A) interfaced to a personal computer via an IEEE 488 interface. The impedance analyzer was programmed to measure $G(\omega)$ and $B(\omega)$ around resonance, and extract the values of w_S, $G(\omega_S)$ and $B(\omega_S)$.

The test solutions were fluxed in the measurement cell by means of a peristaltic pump (Watson Marlow SciQ-401) at the flow rate of 2 µl/s. A picture of the cell and the pump is shown in Figure 5.

5. Experimental Results

Figure 6 shows the measured trends of $f_s = \omega_S/2\pi$, $G(f_s)$, and $B(f_s)$ versus time in response to solutions of different conductivity fluxed in the sensor cell.

It can be observed that, as expected, the series resonant frequency f_s do not change. In addition, in agreement with Eq.(8) and Eq.(9), $G(f_s)$ and $B(f_s)$ show trends that are respectively non-monotonic and monotonic versus the conductivity.

Fig. 5. Measurement cell and peristaltic pump.

By inserting the expressions $R_L = 1/(k\sigma)$ and $C_L = k\varepsilon$ into Eq.(8) and Eq.(9), the following equations are obtained:

$$G(\omega_s) = \frac{1}{R} + \frac{\omega_s^{\,2} C_Q^{\,2}\, 1/k\sigma}{1+\omega_s^{\,2}(k\varepsilon + C_Q)^2 (1/k\sigma)^2} \tag{14}$$

$$B(\omega_s) = \omega_s C_0 + \frac{\omega_s C_Q \left(1+\omega_s^2 (k\varepsilon + C_Q)\varepsilon/k\sigma^2\right)}{1+\omega_s^2 (k\varepsilon+C_Q)^2 (1/k\sigma)^2} \tag{15}$$

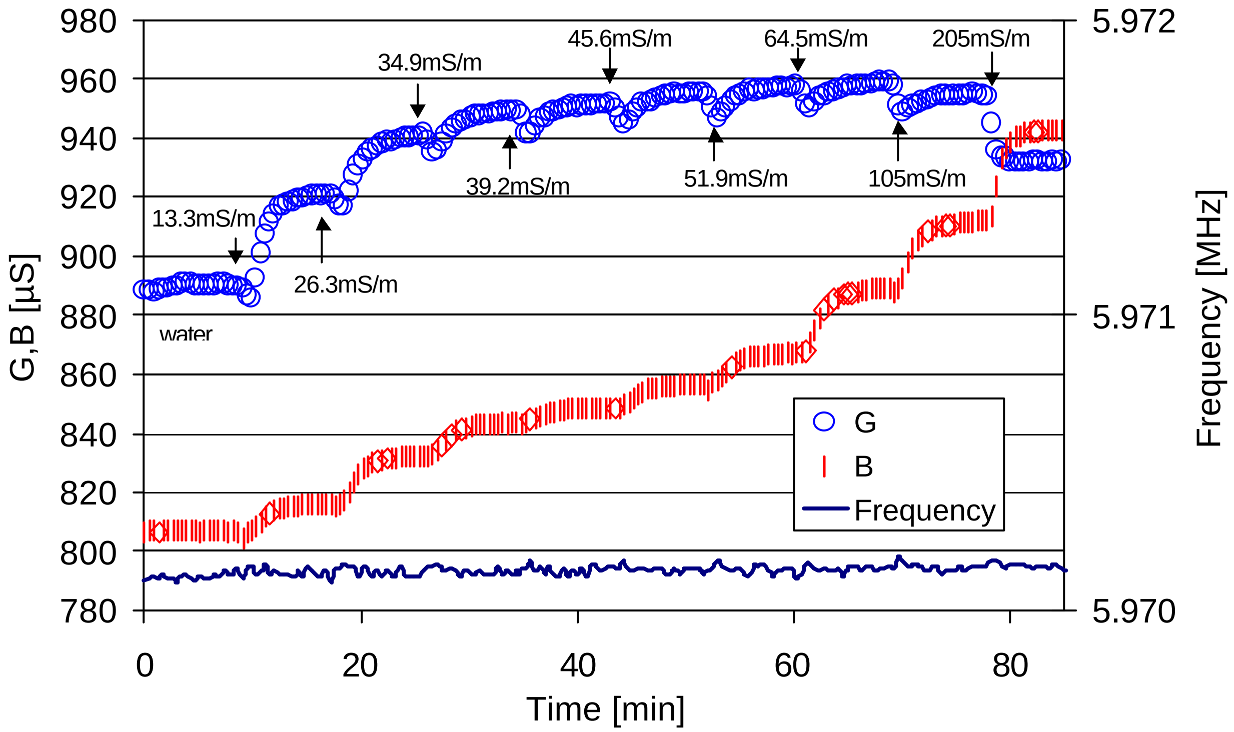

Fig. 6. Measured f_s, $G(f_s)$, and $B(f_s)$ versus time under exposure to NaCl solutions of different conductivity.

The parameters of the equivalent model of Figure 3 have been measured for the sensor exposed to deionized water, therefore in conditions of negligible conductivity that reduce Y_L to the series of C_L and C_Q. The following values have been obtained: $R = 1.1$ kΩ, $C = 13.4$ fF, $L = 53$ mH, and $C_0+C_LC_Q/(C_L+ C_Q) = 21$ pF. In addition, from the crystal and cell dimensions ($A_Q = 35$ mm^2, $t = 0.278$ mm) and the relative permittivity of quartz ($\varepsilon_Q/\varepsilon_0 = 4.55$), it has been calculated $C_Q = 5$ pF.

The relative permittivity of water ($\varepsilon/\varepsilon_0$) has been assumed equal to 78.5. Eq.(14) and Eq.(15) now contain all known quantities, either measured or calculated, apart from the unknown form factor k, which can be used as a fitting parameter for the experimental data measured at the reference values of the conductivity σ.

Figure 7 shows the results of the fitting process, where the points are the experimental data and the curves are plots of Eq.(14) and Eq.(15). It can be observed that an excellent agreement is obtained. This is especially true for the conductance G, while for the susceptance B the fitting is less accurate. The obtained best-fit value of k is 4.54 mm, that is a consistent figure considering the crystal and cell dimensions.

The maximum of the conductance is for a conductivity of around 65 mS/m, in agreement with the literature [6]. Below such a value, a monotonic response is obtained and the proposed QCR sensor can be used as a high-sensitivity conductivity meter.

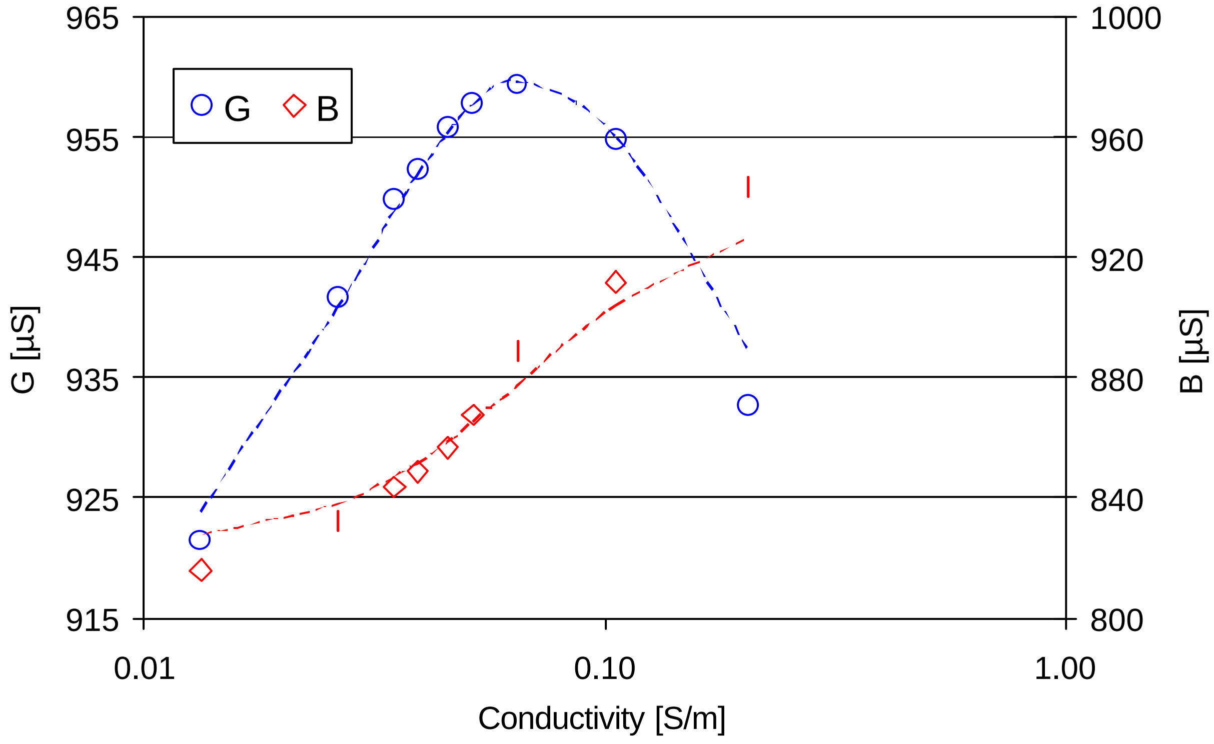

Fig. 7. Result of the fitting between experimental data of $G(f_s)$ and $B(f_s)$ (discrete points) and the theoretical model (dashed curves) as a function of liquid conductivity.

5. Conclusions

In this work the effect produced by the electric conductivity of a low-concentration ionic solution in contact with a QCR sensor with asymmetric electrodes has been studied and modelled.

The experimental results obtained with solutions of NaCl in water have shown that, in agreement with the proposed model, the conductivity of the solution does not produce variations in the series resonance frequency of the sensor. Conversely, because of the electric field fringing effect, there are predictable variations in both the real and imaginary parts of admittance at the series resonance.

The present work contributes to a better understanding of the effects due to the solution in in-liquid applications of QCRs [8-9], and verifies the potential use QCRs with asymmetric electrodes as high-sensitivity conductivity sensors.

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