INTELLIGENT MEASUREMENT INSTRUMENT FOR DETERMINATION OF HF ADMITTANCE OF LIQUIDS

J. Božičević, M. Stamać* and V. Medved**

Faculty of Technology,
University of Zagreb, Yugoslavia

Summary

A computerized instrument has been developed for measurement of HF admittance of small quantities of liquids, to be applied in chemical analysis. The measurement cell is the element of an adjustable, parallel resonant circuit. A specially designed control unit with a microcomputer automatically adjusts the circuit in resonance and calculates admittance of measured liquid.

Introduction

There are many ways to detect zones of different kinds of liquids moving in capillary tubes, based either on thermal, optical, electrical or some other property of the substances. One approach consists in measuring high frequency electrical properties [13, 14, 15]. The present method is directed toward determining properties of small amounts of liquids, less than 100 μl, and detecting microamounts of liquids, less than 1 μl, for separation in capillary tubes. With this purpose, a possibility has been investigated for applying HF methods of measurement regarding liquid parameters; conductivity, permittivity, admittance and several types of measurement cells have been investigated, together with the construction of an original microcell [2, 3, 4].

At higher frequencies liquids are neither "pure" conductors nor dielectrics, but are characterized by complex admittance, a function of measured frequency [1, 9]. This property has been investigated and explored for various measurement purposes [10, 11]. The subject of this work is an original method for measuring admittance of liquids at high frequencies and an intelligent measurement instrument developed through the application of this method.

In the following, first the principle of an original measurement method will be described. Then general requirements for automatic detection and a

* Institute of Civil Engineering, University of Zagreb, Yugoslavia.
** Faculty of Physical Culture, University of Zagreb, Yugoslavia.

control system acting on input parameter changes, are exposed. Convenient mathematical approximations will be stated. Finally, the technical construction of a suitable instrument, built arround a microprocessor, acting as a measuring and control unit, will be described.

Basic measuring principle

The initial idea of applying a compensating measurement method in resonance circuit was in connection with measuring the HF conductivity of liquids having negligible dielectric properties [5]. Circuit adjustment was provided through automatic compensation of changes in the capacitive component of the measurement cell, which in turn was a function of the changing dielectric properties of the measured liquid. But to realize a more precise measurement and characterization of the liquid, the value of permittivity of these dominantly conducting liquids was not to be neglected. Thus a resonance circuit was developed according to Figure 1, where changes in complex admittance of the measurement cell are compensated by simultaneous adjustment of the capacitance and conductance of the circuit. As it can be seen from the picture, adjustment is realized using a controllable resistor — a FET transistor and a controllable condenser — the "varicap" diode.

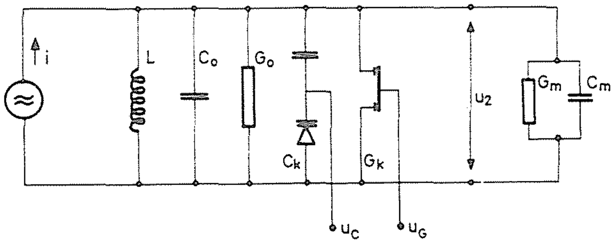

Fig. 1. Adjustable parallel resonant circuit

Such an approach to solve a measurement problem of admittance in small liquid amounts enabled to surmount the inadequacies of standard methods and a fully automatized measurement process has been achieved. A fundamental advantage of this method consists in a constancy of influences of nonlinearities and parasitic impedances throughout the measurement. For a better comparison, it is useful to underline the basic deficiencies of standard methods: the *U—I* method does not produce accurate enough data and because of the nonlinearity of the (G_m, Y_m) function it is difficult to estimate separately the values of conductance and permittivity. Bridge methods are prone to disturbances because the voltage supply and the measuring device do not have the same voltage reference. This holds also for Schering's bridge, the most suitable for the measurements concerned. The substitution method applied on an oscillating circuit in resonance did not produce satisfactory results. During adjustments, the real part of admittance is changing and influences the circuit stability.

General concept of measurement system

The basis of our measuring instrument is the circuit L, C_0, G_0 parallelly oscillating in resonance, according to Figure 1. It is connected to a current source $i_0 = I_0 \sin \omega t$, so the voltage in the circuit is $u_2 = U_2 \sin \omega t$, in phase with the current, i.e. $\varphi = 0$. Parallel to the basic circuit, the measurement cell having an admittance (G_m, C_m) — the subject of measurement, and a controllable admittance (G_k, C_k), are connected. Such a resonance circuit forms part of the intelligent measurement system with block diagram shown in Figure 2.

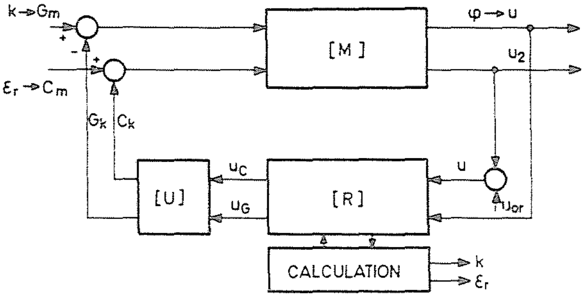

Fig. 2. Measurement and control system

A change in the measured admittance (G_m, C_m) puts the circuit out of resonance, resulting in phase $\varphi \neq 0$ and changing voltage u_2. This is information for the control unit which, besides providing control function, also calculates conductivity and permittivity of the liquid. Control voltages u_g and u_c from the control unit are acting on the FET transistor and the varicap diode, and adjust the compensating admittance (G_k, C_k) to a value that the sum of measured and compensating admittance changes equals zero. When this is reached, the oscillating circuit is in resonance again and its behavior is determined only by G_0 and C_0: $\varphi=0$ and $u=u_2-u_{0_r}=0$.

The measuring circuit is characterized by

$$U=\frac{I_0}{\sqrt{(G_0+\Delta G)^2+(\omega\cdot\Delta C)^2}}-U_0, \qquad \varphi=\operatorname{arc\,tg}\frac{\omega\Delta C}{G_0+\Delta G},$$

$$\Delta G=G_m-G_k, \quad \Delta C=C_m-C_k\,.$$

Using the Jaccobian matrix, these nonlinear relationships become a linear matrix

$$[M]=\begin{bmatrix} -\dfrac{I_0 G}{N_1} & -\dfrac{I_0\omega^2\Delta C}{N_1} \\ -\dfrac{\omega\Delta C}{N} & \dfrac{\omega G}{N} \end{bmatrix}$$

with $N=G^2+(\omega\Delta C)^2$, and $N_1=N^{3/2}$.

So, these relationships can be represented as

$$\begin{bmatrix} U \\ \varphi \end{bmatrix}=[M]\begin{bmatrix} \Delta G \\ \Delta C \end{bmatrix}.$$

With the circuit being adjusted, a steady state is reached and $\Delta G=0$ and $\Delta C=0$, so the matrix $[M]$ becomes $[M]_0$

$$[M]_0=\begin{bmatrix} -\dfrac{I_0}{G^2} & 0 \\ 0 & \dfrac{\omega}{G} \end{bmatrix}=\frac{1}{G}\begin{bmatrix} -\dfrac{I_0}{G} & 0 \\ 0 & \omega \end{bmatrix}$$

Thus in the vicinity of a steady state, the behaviour of the measurement circuit is approximately described by:

$$\begin{bmatrix} U \\ \varphi \end{bmatrix}=\frac{1}{G}\begin{bmatrix} -\dfrac{I_0}{G} & 0 \\ 0 & \omega \end{bmatrix}\begin{bmatrix} \Delta G \\ \Delta C \end{bmatrix}.$$

From a theoretic control point of view, it is a multivariable process, with two output and four input variables ($\Delta G=G_m-G_k$, $\Delta C=C_m-C_k$). The aim of control has to be analyzed using the control theory of multivariable processes. Linearization of the measurement circuit equation followed by an adequate choice of control unit characteristics, enables to simplify the control task and apply the classical theory.

The dependence of controlled variables (G_k, C_k) from control variables (U_g, U_c) is characterized by

$$\begin{bmatrix} G_k \\ C_k \end{bmatrix} = [U] \cdot \begin{bmatrix} U_g \\ U_c \end{bmatrix}.$$

The elements of matrix $[U]$ are given with characteristics of the FET transistor and the varicap diode. They are nonlinear and have to be linearized by adding a nonlinearity characterized by matrix $[C]$ with elements determined in a way that the dependence (G_k, C_k) from (U_g, U_c) be linear.
The control unit is characterized by

$$\begin{bmatrix} U_g \\ U_c \end{bmatrix} = [R] \begin{bmatrix} U \\ \varphi \end{bmatrix},$$

$[R]$ being the characteristic matrix. By substitution equations of all parts of the circuit into one, we obtain a matrix which characterizes the automatic, controlled measurement system:

$$[S] = [R]\,[M]_0\,[U]\,[C]$$

$$\begin{bmatrix} U_g \\ U_c \end{bmatrix} = [R] \begin{bmatrix} U \\ \varphi \end{bmatrix} = [R]\,[M]_0 \begin{bmatrix} \Delta G \\ \Delta C \end{bmatrix} = [R]\,[M]_0 \begin{bmatrix} G_m \\ C_m \end{bmatrix} -$$

$$-[R]\,[M]_0\,[U]\,[C] \begin{bmatrix} U_g \\ U_c \end{bmatrix}.$$

Since in steady state the matrices $[M]_0$ and $[U]$ are diagonal, one can suppose that matrix $[R]$ may also be diagonal. This also gives the matrix $[S]$ in a diagonal form so the system can be treated as an autonomous multivariable system and analyzed like two, formally separated, control circuits — controllers. Synthesis can be accomplished supposing the PID behavior of regulators.

Construction of the measurement and control system

When attempting to realize control circuits one additionally has to solve the problem of calculating measured conductivity and permittivity. Therefore, the control unit is designed using a Motorola MC 6800 microprocessor to perform control operations as well as to calculate conductivity and permittivity of the investigated liquid.

The structure of our measurement system is represented in Figure 3. The output quantities of the measurement circuit: voltage u_2 and its phase angle φ_2 are being compared with the reference values u_{0r} and φ_1, resulting in generating error voltages. These two voltages enter an analog-to-digital conversion process and are fed to the computer. Using these data, the computer calculates control voltages and generates control signals to actual control elements; the FET transistor and the varicap diode.

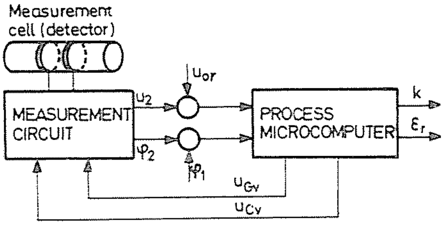

Fig. 3. Structure of measurement instrument

Calculated values of control voltages are also input data for calculating conductivity and permittivity. The final calculated values can be plotted as functions of time.

When calibrating the instrument, calibration reference is provided using the combination of a parallel connected precise resistor and capacitor. This combination is parallel connected to the RLC circuit, with an empty measurement cell. After calibration, the measured quantities can be calculated from the control voltage values.

The influence of system parameters on system stability is studied separately. Parameter values are estimated according to basic conditions of the stability of a discrete system [8].

All commands are entered into the system via keyboard. Commands and data entered, can also be echoed to the display.

The main program is written in PASCAL and time critical routines in the assembler.

The software is designed to provide following operations; start and system calibration, resonance control and calculation of conductivity and permittivity. When starting to work the system sets the oscillating circuit into resonance. This is provided by feedback of the control program. Then the system calibration operation starts. The values of control voltages corresponding to initial resonance conditions are the data for the system calibration program. It calculates input-output characteristics of the measurement circuit, taking into account characteristics of final control elements, too, and stores them in the memory in a tabulated form. Now, the instrument is ready for continuous resonance control, detection and measurement.

System application and conclusions

The instrument has been used for measurements of electrical properties of liquids, at frequencies of about 1 MHz, in various static microcells. Ranges of measured conductivities and permittivities were (0—200 μS) and (0.01—5), respectively. The accuracy is better than 0.5%. Experiments are conducted with the aim to detect various separated zones of substances in a capillary column.

It is hoped that the instrument described demonstrates how the classically difficult measurement task can be overcome by computerized measurement.

References

1. BERCIK, J.: Vodivostne a dielektricke merania v chemickej analyze. (Conductive and Dielectric Measurements in Chemical Analysis) SVTL, Bratislava 1962.
2. BOŽIČEVIĆ, J. et al.: Electr. Letters *7*, 688 (1971).
3. BOŽIČEVIĆ, J.: Elektrotehnika *15*, 432 (1972).
4. BOŽIČEVIĆ, J.—EVERETS, F.: SE Patent 355, 959, 16 Mar 1973.
5. BOŽIČEVIĆ, J.: Internal note, TF Zagreb 1972.
6. BOŽIČEVIĆ, J.—STAMAĆ, M.: Computerized High-frequency Liquid Conductivity Measurement, pp. 291—293 in Proceedings of IMEKO Symposium on Computerized Measurement, Dubrovnik 1981.
7. BOŽIČEVIĆ, J.—STAMAĆ, M.: Kompenzacijska metoda mjerenja VF admitancije kapljevina, (Compensation Method of Measurement of HF admittance of Liquids) pp. 627—633 in JUKEM 82-X Yugoslav Symposium on Measurements and Measuring Equipment Budva 1982.
8. BOŽIČEVIĆ, J.—STAMAĆ, M.: An Approach to Resonance Control of the parallel RLC Measurement circuit with Variable R and C elements (in preparation).
9. OEHME, F.: Chemische Analysen durch Messung von Dielektrizitätskonstanten, (Chemical Analyses by Measurement of Permittivity) Leipzig 1953.
10. OEHME, F.: Dielektrische Messmethoden, Zweite Aufl., Verlag chemie, (Dielectrical Measuring Methods) Weinheim 1965.
11. PUNGOR, E.: Oscillometry and Conductometry, Pergamon Press, New York 1965.
12. STAMAĆ, M.: Design of a Microprocessor-controlled Instrument for Measurement of Conductivity and Permittivity (in preparation).
13. USIKOV, S. V.: Metodi isledovanja elektrofizičeskih svojstva i procesov perenosa v elektrolitah i dielektrikah, (Methods of Determination of Electrophysical Properties and Transmission Processes in Electrolytes and Dielectries) Himija, Moskva 1967.

14. Usikov, S. V.: Visokočastotnije metodi izmerenija provodimosti i dielektričeskoj pronicaemosti rastvorov beskontaktnim sposobom, (High Frequency Methods of Contactless Measurement of conductivity and Permittivity of Solutions) Izv. Akad. Nauk SSSR, Leningrad 1961.
15. Zarinskij, V. A.—Ermakov, V. I.: Visokočastotnij himičeskij analiz, (High Frequency Chemical Analysis) Nauka, Moskva 1970.
16. Stamać, M.: Mjerenje visokofrekvencijske admitancije kapljevine (Measurement of High Frequency Admittance of Liquids) Master's Thesis, Faculty of Technology, University of Zagreb, Zagreb 1981.

Prof. Dr. Juraj Božičević, Faculty of Technology, University of Zagreb, Piorettieva 6, 41000 Zagreb, Yugoslavia
Marin Stamać, Institute of Civil Engineering, University of Zagreb, J. Rakuše 1, 41000 Zagreb, Yugoslavia
Vladimir Medved, Faculty for Physical Culture, University of Zagreb, Horvaćanski zavoj 15, 41000 Zagreb, Yugoslavia