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HIGH IMPEDANCE MEASURING OF ANTI-CORROSION COATINGS

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Abstract: The paper deals with problems of very high impedance ($|Z|$<10 GW) measurements in a wide frequency range (10^{-5}¸10^{6} Hz). The measurements are aimed to identify electrical parameters of different anti-corrosion coatings. The construction of the measuring system designed for monitoring the anti-corrosion coatings effectiveness on various types of technical objects is presented. The circuit solutions analysed refer, in particular, to the input adapter, the measuring signal generator, and the phase-sensitive detector. The test results of the whole measuring path are shown and some parameters determining low and high band of the system frequency range are pointed out.
Keywords: impedance measurement, equivalent circuit, anti-corrosion coatings

1 INTRODUCTION

The high impedance measurements under discussion are focused on physico-chemical objects, especially on identification of electrical parameters of various types of anti-corrosion coatings. The problem has some practical and economical meaning because of early detection of some coating defects and undercoating rusting. A continuous monitoring of the anti-corrosion coating condition ensures reliability and safety of technical objects (e.g. pipelines, bridges, fuel tanks, steel structures).

The main objective of the coating measurements is an analysis of very high impedance (of an order of GΩ) in a wide frequency range (10^{-5}–10^{6} Hz) assuming the measuring signal amplitude on a level of 10mV-100mV due to nonlinearity of the object under test [1, 2, 3]. Nowadays, the instruments and the techniques used for electrochemical impedance spectroscopy (EIS) allow to measure parameters of relatively low impedance (<10 MΩ). On the other hand, modern anti-corrosion coatings (e.g. rubber or organic linings) represent impedance exceeding 10 GΩ, which causes serious measuring difficulties. An additional disadvantage of the currently in use impedance spectroscopy instrumentation is a high complexity of the equipment to be used, as a rule, in laboratory experiments [4].

In view of the presented facts, the authors have developed a new project aimed to elaborate a field-worthy low-cost microsystem for measuring parameters of very high impedance anti-corrosion coatings intended for continuous monitoring of the anti-corrosion protection performance. The paper presents the results of testing, realised by a laboratory measuring system. The results are necessary for a correct design of a final version of microsystem.

2 ELECTROCHEMICAL BACKGROUND

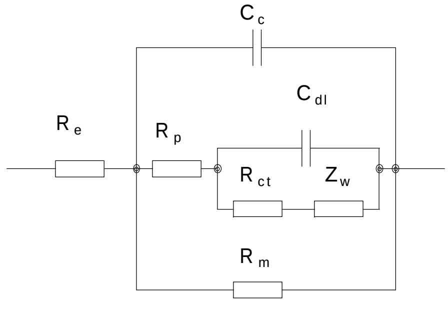

Figure 1. Equivalent circuit for coated metal, where: R_e - the electrolyte resistance; R_p - the resistance of the electrolyte in pores; R_{ct} - the charge transfer resistance; R_m - the resistance of the coating material; C_c - the capacitance of the coating; C_{dl} - the double layer capacitance; Z_w - the diffusion (Warburg) impedance.

Modelling of any electrochemical system is complicated and coated metals are at the extreme of complexity [3]. There are several models that occur during the life of a coating and the most general one is shown in Fig. 1. During the life of a coating, an equivalent model can change due to evaluation of the electrolyte-coating-metal system caused by such processes as moisture penetration, onset of corrosion, and break up of the coating. When the coating is new and effective, R_p, C_{dl}, R_{ct} and Z_w are not present in the model. As the electrolyte penetrates the coating, R_p comes into play. When corrosion starts, R_{ct} and C_{dl} become operable. When the corrosion rate becomes high, diffusion impedance Z_w is also present. If the corrosion rate decreases, Z_w becomes unnecessary, but if the coating breaks up, C_c, R_m, R_p become inoperable and the system acts like a bare metal.

An analysis of the presented parameters with an equivalent circuit is possible on the basis of impedance spectrum of anti-corrosion coating. Fig. 2 presents exemplary curves for impedance modulus and phase of 0,8mm thick silocsiran lining manufactured by Elf Atochem USA at different stages of corrosion (after 2, 24, 240 hours of coating exposure in 70% H_2SO_4 at 80°C) [5]. The graph shows that an identification of equivalent circuit parameters requires measurements to be done in a range from mHz to MHz and in a low frequency range a very high impedance is noted ($|Z_x|\approx 1G\Omega$).

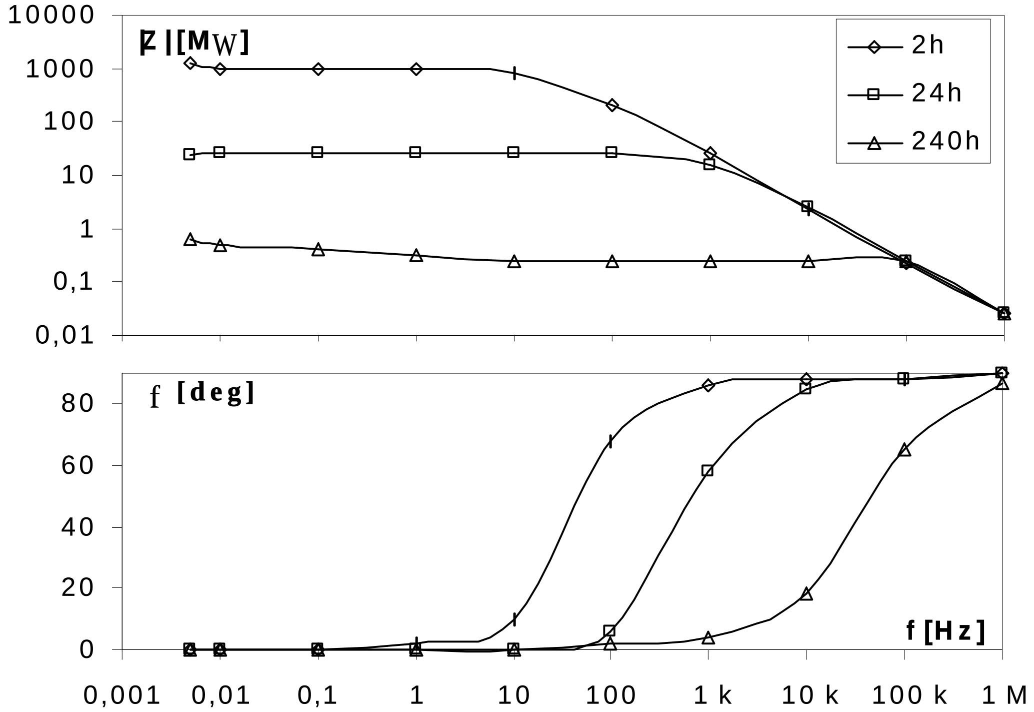

Figure 2. Impedance spectrum of anti-corrosion coating (type: silocsiran manufactured by Elf Atochem USA, thickness 0,8mm) in 70% H_2SO_4 at 80°C.

Utilizing the impedance data, the values for the parameters of an equivalent circuit model can be obtained and it is possible to transform the model parameters to the coating characteristics, namely the moisture content of the coating, delaminations, blisters, and other defects, the presence and rate of corrosion. This allows to assess the current condition of a coating and to predict its performance in the future.

3 PRINCIPLES OF MEASURING SYSTEM

The most essential problems in the measurements of anti-corrosion coatings are combined with measuring high impedance in low frequency range. Due to these facts, the authors have designed and constructed a measuring laboratory system enabling to identify the impedance parameters of two-terminal networks for objects with $|Z_x|<10G\Omega$) in the frequency range from 1mHz to 1 kHz (decade changeable). The system has been based on DAQ card from National Instruments (AT-MIO-16E-1 type) installed in PC computer and input circuitry externally connected to card. The block diagram of the system is presented in Fig. 3.

In the input circuitry two signals are extracted: current proportional (u_1~i_x) and voltage proportional (u_2~u_x) [6, 7]. Current signal u_1 is connected to output of operational amplifier (LMC6084), which works in current-voltage converter configuration described by the following equation:

$$u_1 = -i_x R_R \,, \qquad (1)$$

where: R_R – range reference resistor (1 GΩ, 100 MΩ, 10 MΩ …) placed in feedback loop of OPAMP.

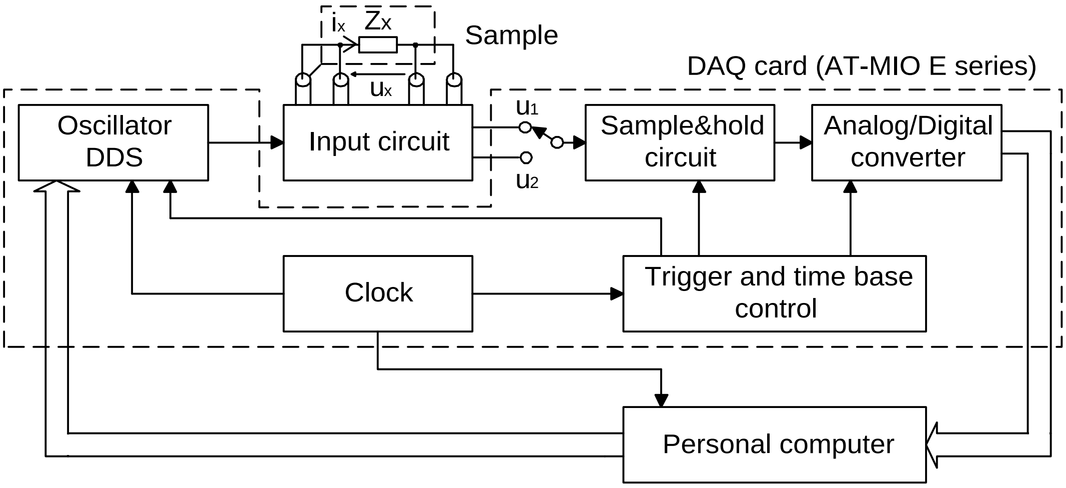

Figure 3. Block diagram of system for high impedance objects measuring.

The voltage proportional signal u_2 is equal to the measuring generator output voltage, so it is obtained from the output of the second operational amplifier of a constant unity gain. To determine the magnitudes as well as the phases of voltages u_1 i u_2 the Discrete Fourier Transformation (DFT) has been used. For the best accuracy of the calculation of the signal parameters a common acquisition path has been used for sampling voltages u_1 i u_2 and a start of both signals acquisition has been assured exactly in the same phase of excitation signal. It was possible to fulfil this condition by generation of excitation signal using Direct Digital Synthesis (DDS), in which a chain of samples of sinusoidal waveform produced in 12-bit D/A converter is realized utilizing a common clock signal synchronised with the acquisition of signal samples u_1 and u_2. Let us note, that the described solution guarantees a precise fulfillment of the condition for N samples to take an exact place in the k periods of excitation, ensuring that in the DFT spectrum analysed, exactly one non-zero line adequate to excitement frequency exists, (assuming linearity of object under test).

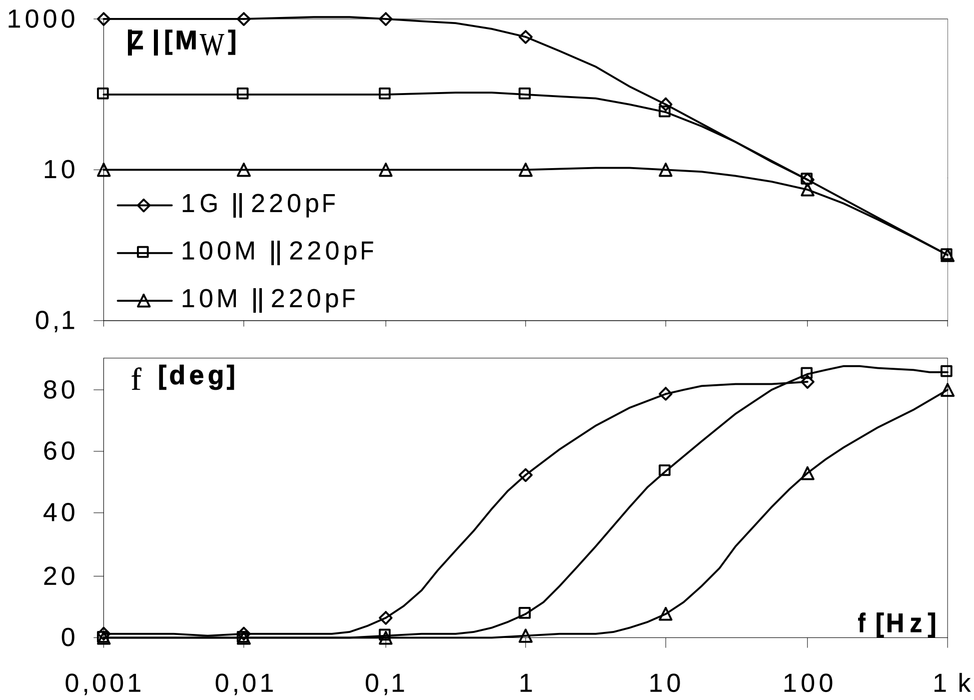

Figure 4. Impedance spectrum for two-terminal RC network.

Impedance measurement (modulus and phase) takes three stages:

- acquisition of N samples of voltage u_1 utilizing 12-bit A/D converter working with sampling frequency of f_s,
- collection of analogous N samples of voltage u_2 with the same measuring path, utilizing multiplexer in the Sample&Hold input,
- execution of DFT for both samples sets to calculate modulus ($|U_1|,|U_2|$) and phase (φ_1, φ_2). To do this the Labview built-in algorithm has been used. The modulus and the phase of the measured impedance have been calculated using equations:

$$|Z| = \frac{|U_2|}{|U_1|} \cdot R_R, \qquad \phi = \varphi_2 - \varphi_1. \tag{2}$$

The result of impedance measurement has been presented in Fig. 4 and 5.

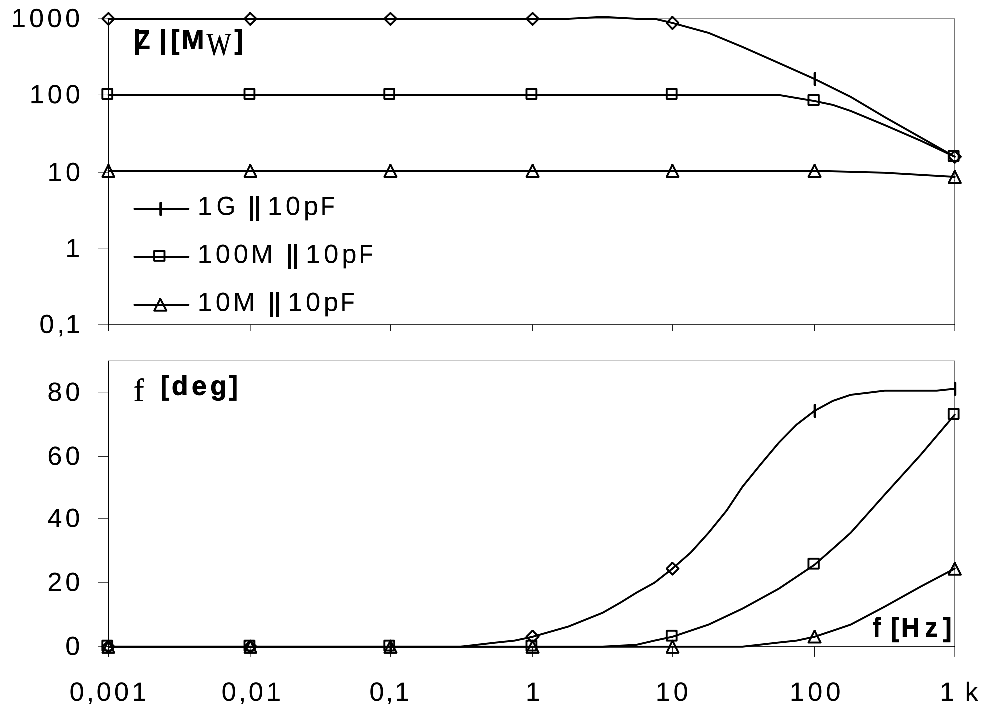

Figure 5. Impedance spectrum for two-terminal RC network.

To estimate the measurement accuracy in the realised system, an identification of the elements parameter of the two terminal parallel circuit (R_x, C_x) measured has been performed on the basis of their calculated impedance parameter values (|Z| i ϕ) using equations:

$$C_X = \frac{1}{|Z| \cdot \omega} \cdot \sin \varphi, \qquad R_X = \frac{|Z|}{\cos \varphi}. \tag{3}$$

The relative error of the computed elements of a two-terminal network under investigation has been determined, taking advantage of the nominal values R and C on the basis of their values measured with 0,1% accuracy. A typical relative error has been presented in Fig. 6 illustrating two measurement cases carried out at 1 Hz excitement frequency: 1 GΩ connected in parallel with 220 pF and 1 GΩ with 10pF. The first of them represents a situation when both the impedance components are comparable, the second one takes place when the capacitance has 20-times less influence on resultant |Z| of the two-terminal network than the resistor. In computation the constant parasitic capacity 1,5 pF of connection range resistor R_R in the feedback of the operational amplifier in the input circuitry has been taken into consideration.

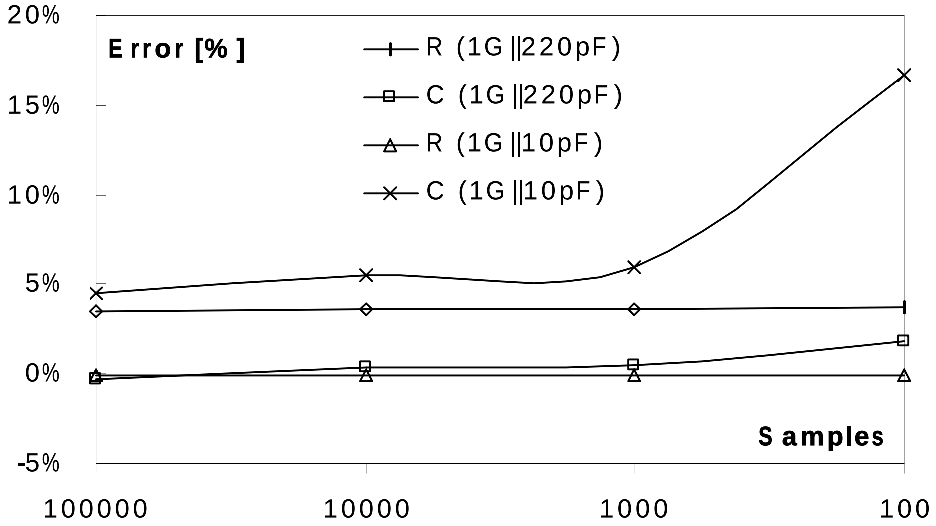

Figure 6. Relative error of identification of two-terminal network elements.

The error has been calculated for a different number of samples for one period of generated sinusoidal waveform, assuming a constant number (10000) of collected samples of voltages u_1 and u_2. The graph analysis allows to formulate the following conclusions:

- an element weakly shunted (1 GΩ||10pF) is measured with an error of less than 0,1%,
- elements shunted almost identically to each other (1 GΩ||220 pF) are calculated with an error in range of 0,5÷3% depending on low shunt difference,
- an element strongly shunted (1 GΩ||10 pF) is measured with the highest error rising with shunting.

All errors indicate a rising tendency depending on the decrease of samples per period, which is particularly evident in the case of elements highly shunted. A similar effect on the error has the number of the collected samples in DFT procedure.

Generation of the measuring signal, the acquisition of voltages u_1 and u_2 samples and the calculations based on equations (2) and (3) have been controlled via Labview based panel shown in Fig. 7.

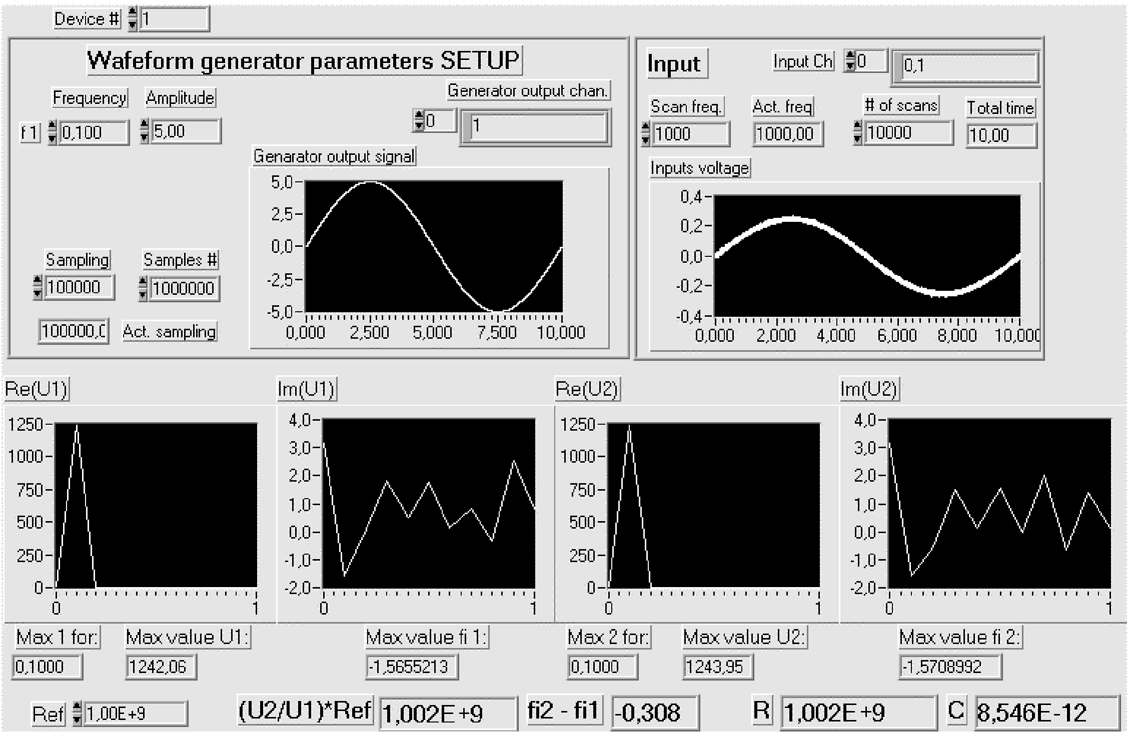

Figure 7. The control panel for measuring impedance system.

4 SUMMARY

There is a need for special instrumentation to proceed with the diagnosis because of very high impedance of objects under test and a wide frequency range. For very low frequencies (<10 Hz) one can hardly use the well known phase sensitive detectors because of the required long average times. The presented laboratory measuring system utilizes the DDS technique to generate the sinusoidal measuring signal and it uses the DFT procedure to calculate the impedance parameters of two-terminal network under tests. The system measurement allow to determine an optimal number of samples of a generated signal period at an order of 100000 (for strongly shunted elements) and a number of collected samples in the DFT procedure at an order of 10000. The measurement error 0,1% appears for an element shunted by a value of an order greater and at a level of 1% when identical shunting is used for measuring frequencies in the range of 1 mHz÷100 Hz. In view of obtained results, the assumed system conception has proved to be right and this allows, in the next step, to realize a microsystem designed for field-worthy monitoring of anti-corrosion coatings.

REFERENCES

[1] J. R. Dygas, M. W. Breiter, Measurements of large impedances in a wide temperature and frequency range, *Electrochimica Acta*, Vol. 41, Nos. 7/8, pp. 993-1001, 1996.
[2] G. Chiodelli, P. Lupotto, Experimental Approach to the Impedance Spectroscopy Technique, *J. Electrochem. Soc*, Vol. 138, No. 9, pp. 2703-2711, September 1991.
[3] A. Miszczyk, K. Darowicki, Z. Klenowicz, Use of Impedance Spectroscopy to Characterise Lining Performance in Simulated Flue Gas Desulfurization Systems, *Corrosion*, NACE, vol. 53, 1997, p. 572.
[4] 1294 High Impedance Interface – Solartron UK.
[5] A. Miszczyk, originally in polish language: Charakterystyka impedancyjna pow³ok organicznych przeznaczonych do ochrony instalacji odsiarczania spalin metoda mokra (in English: The impedance characteristic of the organic coatins designed for protections of fuel gas desulfurization systems using wet method), PhD Dissertation directed by K. Darowicki, Technical University of Gdansk, Chemical Department, Gdansk 1998.
[6] J. Hoja, Z. Czaja, Accuracy processing analysis of the measuring impedance system based on four vector method, *Proceedings of the 9th International Symposium on Electrical Instruments in Industry*, IMEKO TC-4, Glasgow, 8-9 September 1997, pp. 229-232.
[7] J. Hoja, Error analysis for the four-vector method of measurement of RLC elements, *5-th International Symposium on Electrical Measuring Instruments for Low and Medium Frequencies*, IMEKO TC-4, Vienna (Austria), April 8-10, 1992, Part I, pp. 93-100.

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