although the specific heat of AMg5 is higher. This is explained in that the actual cryoagent consumption at fixed Γ is determined by the volume specific heat of the cryostat material, which is higher in steel because of its higher density. In addition, in choosing the material, the higher strength characteristics of steel must be taken into account; this allows structures with lower Γ to be used.

NOTATION

C, specific heat; i, specific enthalpy; m, liquid mass; $\dot{m}$, mass rate of pumping-out; M, cryostat mass; P, pressure; $\dot{Q}$, heat flux; $\tilde{Q} = (\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_1$, $\tilde{\tilde{Q}} = (\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_2$, dimensionless parameters; r, radius; T, temperature; τ, time; V, volume; $\dot{V}$, volume rate of pumping-out; u, specific internal energy; $\Gamma = V_W/V_{L0}$, geometric parameter; $\theta = (m_0 - m)/m_0$, relative mass fraction of pumped-out liquid; $\psi = (V_0 - V)/V_0$, relative volume fraction of pumped-out liquid; δ, wall thickness of cryostat; ρ, density. Indices: V, vapor; L, liquid; 0, initial conditions; tr.p, triple point; s, saturation; w, wall; cr, critical point.

LITERATURE CITED

1. B. I. Verkin, V. F. Getmanets, and R. S. Mikhal'chenko, Thermal Physics of Low-Temperature Sublimational Cooling [in Russian], Kiev (1980).
2. C. Sindt, Cryogenics, 10, No. 5, 372-380 (1970).
3. R. S. Mikhal'chenko, V. T. Arkhipov, V. F. Getmanets, et al., in: Low-Temperature Engineering [in Russian], Kiev (1979), pp. 31-47.
4. A. Mironer, Cryogenics, 26, No. 1, 46-47 (1986).
5. A. B. Grachev, B. S. Voroshilov, and V. M. Brodyanskii, Inzh.-Fiz. Zh., 24, No. 6, 1007-1012 (1975).
6. V. O. Kruglov, K. V. Maslov, A. A. Motornaya, et al., Software for EC Computer. Library of Programs on Numerical Methods in PL/1 [in Russian], Kharkov (1984).
7. V. E. Seregin, Program in PL/1 for Calculating the Thermophysical Properties of Hydrogen, Oxygen, Nitrogen, Argon, and Methane. Preprint 29-85 [in Russian], Physicotechnical Institute of Low Temperatures, Academy of Sciences of the Ukrainian SSR, Kharkov (1985).
8. I. G. Kozhevnikov and L. A. Novitskii, Thermophysical Properties of Materials at Low Temperatures (Handbook) [in Russian], Moscow (1982).
9. A. M. Domashenko, V. P. Kachura, and N. V. Filin, Processes and Technology in Cryogenic Engineering [in Russian], Balashikha (1975), pp. 144-155.
10. N. V. Filin, A. M. Domashenko, V. P. Kachura, et al., in: Physical Constants and Properties of Materials. No. 9. Thermophysical Properties of Substances and Materials [in Russian], Moscow (1976), pp. 180-190.
11. B. I. Verkin, R. S. Mikhal'chenko, V. T. Arkhipov, et al., in: Low-Temperature Engineering [in Russian], Kiev (1979), pp. 3-21.
12. R. R. Caren and R. M. Coston, Adv. Cryogen. Eng., 13, 450-462 (1968).

RAISING THE ACCURACY OF MEASURING THE DIELECTRIC PERMITTIVITY OF FLUIDS

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UDC 621.317.33

Dielectric permittivity measurements are performed for certain nonpolar liquid dielectrics with a relative error of 0.05% by using a perfected phase method.

The dielectric permittivity is one of the most characteristic macroscopic dielectric parameters and its knowledge is necessary practically everywhere that such substances and materials are utilized. Exact knowledge of the dielectric permittivity in application to liquid substances permits reliable information to be obtained about the nature of thermal motion of molecules, about the molecular structure (mutual molecule orientation, dynamics

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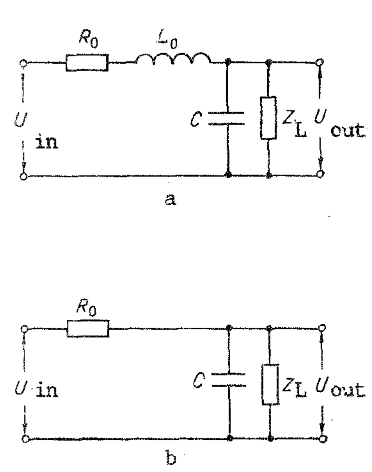

Fig. 1

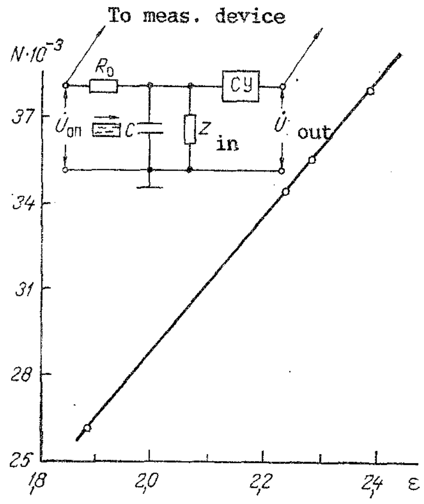

Fig. 2

Fig. 1. Measuring transducer electrical circuits, in principle, in the form of phase-shifting loops of the RLC (a) and RC (b) types.

Fig. 2. Connection diagram of the primary transducer and dependence of the digital code of the measuring device on the dielectric permittivity of different (see Table) nonpolar liquid dielectrics.

of rotational motion, etc.) [1]. To a definite degree the dielectric permittivity reflects the nature of intermolecular interaction, knowledge of which is necessary for the analysis of thermodynamic and transport properties of liquids, etc. On the other hand, the dielectric permittivity is used as a parameter for checking and identifying liquid media, its magnitude allows a judgment on the kind of fluid, the degree of its purity, the concentration of impurities, admixtures, and other properties. The development of scientific investigations of the properties of substances, the working out of new technologies, the creation of new substances and materials with complex assigned properties require the development of methods and facilities assuring a substantial rise in the accuracy of measuring different physical characteristics, including the dielectric permittivity.

Judgments on the dielectric permittivity are ordinarily made from the change in the electric capacitance of the measuring capacitor that is filled with the dielectric under investigation [2] or is set in contact with it [3]. Different methods of measurement and measuring schemes are realized here. Bridge circuits in which the information parameter is the current or voltage occurring when the electrical bridge is unbalanced are used extensively. From the viewpoint of information theory of measuring apparatus, methods based on measuring the signal amplitude possess the lowest resolution and measurement accuracy. Resonance frequency primary measuring transducers utilized also do not assure the accuracy required because of their low quality. A phase method is used in which the phase difference of harmonic electrical oscillations is measured at the input and output of a phase shifting loop containing a measuring capacitor before and after it is filled with the dielectric [4]. The disadvantage of such a method is the presence of a systematic error for the absolute phase measurement, which also reduces measurement accuracy.

A perfected modification of the phase method that eliminates systematic measurement errors is used in this paper. This is achieved because the measuring system developed records only the increment in the phase difference of signals from the output and input of the primary transducer that results during filling of a measuring cell of capacitance type with the liquid dielectric being investigated. The measuring system contains a module that multiplies the phase-difference increment 10^n time, n = 1, 2, 3, 4. For a reference signal frequency of 10^6 Hz, which is supplied to the measuring transducer input and is the reference for the subsequent conversion and recording modules, the intrinsic relative frequency instability introduced by the system is assured at the 10^{-13} level. This permits recording the increment of the phase difference of the input and output signals with a $2\pi \cdot 10^{-6}$ rad error, which raises the measurement accuracy of the dielectric permittivity by approximately an order as compared with traditional methods.

Utilization of a phase-shifting loop as measuring transducer imposes substantial requirements on the selection of the type of these latter, the method of its connection in the

TABLE 1. Results of Experimental Investigations of the Dielectric Permittivity of Certain Nonpolar Liquid Dielectrics

Fluid	ε [6]	N_{av}	σ(N)	$\frac{\sigma N}{N}\cdot 100\%$
Hexane	1,889	26204	14,3	0,05
Carbon tetrachloride	2,236	34449	12,4	0,04
Carbon Benzene	2,283	35676	10,4	0,028
Toluene	2,383	37907	21	0,05

measurement loop, and the nominal value of its elements. Loops of the RC, CR, RLC, etc. types are the simplest phase-shifting loops most extensively utilized for measurements. Connection of the measuring transducer in the measuring loop should be realized in such a manner that firstly the load resistance, for which the input impedance of the measuring device is used, would weakly affect the steepness of the transfer function argument of the measuring transducer, and secondly, would eliminate the influence of parasitic capacitances practically completely. The transducer elements should have such nominal values as to assure the measurement process on a section with maximal steepness of the phase characteristic.

Let us consider a measuring transducer in the form of a RLC phase-shifting loop as represented in Fig. 1a. We will consider C to be the sensing element of the transducer and the output to be capacitive. The phase difference between the signal from the transducer output and the reference signal for such a loop has the form

$$\varphi = -\operatorname{arctg}\frac{\omega_0(L_0+R_0Z_LC)}{R_0+Z_L-\omega_0^2L_0Z_LC}, \tag{1}$$

where Z_L is the load resistance of the measuring transducer. Then the change in phase difference caused by insertion of an object is written as

$$\operatorname{tg}\Delta\varphi = -\frac{\omega_0^2L_0Z_L\Delta C}{\omega_0L_0+\omega_0R_0Z_LC_0+\omega_0R_0Z_L\Delta C}, \tag{2}$$

where C_0 is the intrinsic capacitance of the measuring cell, and ΔC is the increment in the capacitance caused by insertion of the object. The sign takes account of the negative value of $\Delta\phi$ for such a type of measuring transducer.

We obtain an expression for the relative measurement of the capacitance from (2), which under the condition of maximal sensitivity of the measuring transducer [5]

$$\omega_0^2L_0C_0 = \frac{Z_L+R_0}{Z_L},$$

has the form

$$\frac{\Delta C}{C_0} = -\frac{\dfrac{Q\omega_0L_0}{R_0+Z_L}+1}{1+\dfrac{Q}{\operatorname{tg}\Delta\varphi}}, \tag{3}$$

where $Q = \omega_0L_0/R_0$.

Using the relation between the relative change in the capacitance and the dielectric permittivity for two-terminal cells in conformity with GOST 22372-77; we obtain on the basis of (3)

$$\varepsilon = 1-\frac{C_0}{C_0-C_c}\,\frac{\dfrac{Q}{\omega_0Z_LC_0}+1}{1+\dfrac{Q}{\operatorname{tg}\Delta\varphi}}, \tag{4}$$

where C_c is the cell constant defined by the formula

$$C_c = \frac{C_0\varepsilon_K-C_K}{\varepsilon_K-1}, \tag{5}$$

and C_K is the capacitance of the measuring cell filled with the calibration fluid with a previously known ε_K.

It is seen from (4) that the resolution in determining the dielectric permittivity by a phase method under the condition that values of the capacitances C_0 and C_c are known with very high accuracy

$$\delta\varepsilon = \frac{C_0}{C_0 - C_c}\left(\frac{1}{\omega_0 Z_L C_0} + \frac{1}{Q}\right)\delta(\Delta\varphi). \quad (6)$$

As is seen from (6), for Q = 1 and $Z_L = \infty$ the resolution on the dielectric permittivity agrees with the resolution in phase. For Q > 1 and a finite value of Z_L as holds in the majority of cases in practice, the resolution of the measurements for identical Z_L grows additionally as Q grows. When selecting the value of Q it is usually necessary to be guided by the fact that the measurement process should occur on the linear section of the phase characteristic of the measuring transducer in the whole range of variation of the sensing element capacitance in addition to the requirements imposed on the resolution.

For a measuring transducer in the form of the RC loop displayed in Fig. 1b, the appropriate expressions for the phase characteristic and the dielectric permittivity under the condition of maximal sensitivity of the measuring transducer

$$\omega_0 R_0 C_0 = 1 + \frac{R_0}{Z_L} \quad (7)$$

are written in the form

$$\varphi = -\operatorname{arctg}\frac{\omega_0 R_0 Z_L C}{R_0 + Z_L}, \quad (8)$$

$$\varepsilon = 1 - \frac{C_0}{C_0 - C_c}\,\frac{2\operatorname{tg}\Delta\varphi}{1 + \operatorname{tg}\Delta\varphi}, \quad (9)$$

while the resolution is in this case

$$\delta\varepsilon = \frac{2C_0}{C_0 - C_c}\,\delta(\Delta\varphi). \quad (10)$$

Comparing (6) and (10), we see that the resolution for measurement using a measuring transducer in the form of an RC loop in the case of a high-resistance load is approximately 2Q times smaller than when using the RLC loop. However, for many specific measurement problems in practice, it is completely sufficient to assure an error determined by (10). For those investigations when still higher resolution is necessary or it is necessary to measure small changes in the dielectric permittivity, it is more expedient to use a measuring transducer in the form of an RLC loop.

By using the method described and a measuring transducer of RC type, the dielectric permittivity was measured of chemically pure nonpolar liquid dielectrics with low losses, for which the organic fluids hexane, carbon tetrachloride, benzene, and toluene were selected. A capacitance type measuring cell fabricated from molybdenum glass was used as sensing element. The working part of the cell is a coaxial capacitor, made by depositing a gold layer on the wall of glass cylinders. The electrode leads are connected to a coaxial joint on the cell plate. The intrinsic capacitance of the cell is C_0 = 26.5 pF. The cell is connected in series with the active resistor R_0 through a coaxial junction thereby forming a measuring transducer in the form of a phase-shifting RC loop. The transducer output is connected to the input of the measuring device with a low input resistance (on the order of 150 Ω) through a matching unit MU for which a dividing emitter follower was used. The input impedance Z_{in} of the matching unit which is the transducer load was approximately 13 kΩ. To a considerable degree this permitted elimination of the influence of the low input resistance of the measuring device on the steepness of the argument of the transfer function of the phase-shifting loop, and therefore, on the transducer sensitivity, and practically completely getting rid of the influence of parasitic capacitances in the form of the connecting cables and input capacitance of the measuring device [5] on the measurement result. The connection circuit for the measuring transducer is shown in Fig. 2. To assure maximal transducer sensitivity, the value of the resistance R_0 was selected such that the initial phase difference between the output and input signals would be $\pi/4$. the volume of fluid filling was 10^{-2} liters. Pumping the fluid out and drying the cell are performed after each measurement by using a vacuum pump, to which the measuring cell was connected through a fluid trap. The reference signal frequency was 1 MHz. Measurements for all the fluids were performed at t =20°C.

Twenty fillings of the measuring cell for each fluid were performed with each of the dielectrics being investigated. The results are represented in Fig. 2. The values of ε for these fluids were taken from [6]. As should have been expected, the dependence between the increment of the phase difference and the dielectric permittivity is linear in nature. The mean values of the measured phase-difference increments as well as the measurement error characteristics are presented in Table 1. The phase-difference increments were recorded in the form of a digital code N connected with the $\Delta\phi$ by the relationship

$$N = \frac{10^6}{2\pi}\Delta\varphi. \quad (11)$$

The sensitivity of the measuring system to a change in the phase difference, or in other words, the value of the counting unit in the smallest place of the code being recorded was $2\pi\cdot 10^{-6}$ rad which, as is seen from (10), corresponds to a resolution of approximately $1.3\cdot 10^{-5}$ in ε. The relative error of measurement ε does not exceed 0.05%.

As the measurement object, the nonpolar dielectrics named above were not selected randomly. These fluids are utilized extensively as standards for the calibration of measuring cells since they satisfy the following requirements: easily purified, chemically stable, low electrical conductivity, and small dielectric losses. Depending on the purposes of the measurements, the dielectric permittivity of such fluids must be known to the accuracy of the third, and sometimes even the fourth term after the decimal since the accuracy of the dielectric permittivity measurement of a specific object of investigation with analogous properties will be determined to a considerable degree by the accuracy of giving the calibration curve. The phase method described with the high resolution of the measurements can be used successfully for such purposes, in particular, for an express analysis of the composition of liquid media.

NOTATION

R, C, L, electrical resistance, capacitance, and inductance of the measuring transducer elements; $\omega_0 = 2\pi f_0$, f_0, frequency of the electrical oscillations of the reference signal; ε, dielectric permittivity; ϕ, phase difference of signals at the input and output of the measuring transducer.

LITERATURE CITED

1. M. I. Shakhparonov, Methods of Investigating the Thermal Motion of Molecules and the Configuration of Fluids [in Russian], Moscow (1963).
2. F. Ame, Dielectric Measurements [Russian translation], Moscow (1967).
3. I. G. Matis, Electric Capacitance Transducers for Nondestructive Control [in Russian], Riga (1982).
4. A. A. Kulikovskii (ed.), Handbook on Radio Electronics [in Russian], Vol. 2, Moscow (1968), pp. 453-486.
5. V. I. Krylovich and V. V. Mikhal'kov, Vestsi Akad. Nauk BSSR, Ser. Fiz.-Énerg. Nauk, No. 1, 90-95 (1987).
6. Ya. Yu. Akhadov, Dielectric Properties of Pure Fluids [in Russian], Moscow (1972).