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Improving the way of determination substances thermal physical characteristics by direct heating thermistor method

***Abstract**. This work devoted to investigating for improving methods possibility of determining material thermal characteristics measured by direct heating thermistor method and selecting the optimum settings heating sensor. Presented the results of experimental researches with control fluids with the help of the developed device, which gave an opportunity to evaluate the effectiveness of the proposed improvements. Recommended to reduce errors in the measurement by direct heating thermistor used method of determining thermal conductivity materials thermistor temperature under heating pulse of electric current with regard to thermal characteristics thermistor probe, thermistor power changes during it`s heating and temperature of the sample.*

Streszczenie.** Praca poświęcona jest badaniom nad poprawą metod obliczania właściwości cieplnych materiałów mierzonych metodą termistora bezpośredniego ogrzewania i doborem optymalnych ustawień czujnika ogrzewania. Przedstawiono wyniki eksperymentalnych badań płynów kontrolnych za pomocą opracowanego urządzenia, które dało możliwość oceny skuteczności proponowanych ulepszeń. Zalecane do zmniejszenia błędów w pomiarze za pomocą termistora bezpośredniego ogrzewania metody określania temperatury termistorowej materiałów termistorowych w impulsie grzewczym prądu elektrycznego w odniesieniu do charakterystyki cieplnej sondy termistorowej, zmian mocy termistora podczas jego ogrzewania i temperatury próbki. **(Poprawa sposobu określania fizycznych właściwości termicznych substancji metodą termistorowego ogrzewania bezpośredniego).

Keywords: direct heating thermistor method, thermal conductivity of materials, thermistor.
Słowa kluczowe: metoda termistora bezpośredniego ogrzewania, przewodność cieplna materiałów, termistor.

Introduction

When determining the coefficient of thermal conductivity by direct heating thermistor method there is an error of calculation, the value of which depends on the method by determining the coefficient of thermal conductivity. This problem can be solved by optimal and accurate determination of the calibration coefficients used in the calculations. In this work has been carried out the research of errors occurring in the application of known methods and proposed a new method for determining the thermal conductivity of substances to improve the accuracy of their measurement.

Formulation of the problem

The purpose of this work is to research the error of measurement for existing methods for the determination of thermophysical characteristics (TPC) measured by the direct heating thermistor method to increase the measurement accuracy by choosing the optimal method of mathematical calculations for the thermal conductivity of substances under study by measuring the temperature of the thermistor's heating during it`s heating under the action of electric current.

Main research material

The essence of the direct heating thermistor method is to use the phenomenon of it`s self-heating due to flow through it of electric current. The temperature of the self-heating thermistor will depend on the environment in which it is located, that is, from the TPC of the research substance, which the thermistor has a thermal contact with.

For an ideal model of a thermistor sensor in the ball form with radius r with ideal thermal conductivity, provided that the thermistor is surrounded by the research material, the coefficient of thermal conductivity is determined by the formula [1]:

$$\lambda = \frac{P_t}{4\pi r \Delta T} \tag{1}$$

where λ – thermal conductivity coefficient of the investigated material, W/(m·K); P_t – thermistor power, W; r – radius of the head of the thermistor, m; ΔT – temperature of the thermistor, °C.

Thus, to determine the coefficient of thermal conductivity by direct heating thermistor method it is necessary to measure the temperature of the thermistor heating, which has a thermal contact with the test substance. The accuracy of determining the thermistor's power and it`s heating temperature affects the accuracy of the calculation of the test substance thermal conductivity according to the measurement data. Therefore, was carried out an analysis of existing methods for determining the thermal conductivity used by different researchers and experimental studies about determine the measurement error by these methods.

Typically, all measurement errors, depending on the cause of their occurrence, can be divided into methodological (method errors), instrumental [2], subjective (operator errors) [3], as well as errors in the processing of measurement results.

When conducting indirect measurements performed using simplified algorithms, errors arise that are related not so much to the measurement process, but above all to calculations errors. For example, such errors are encountered when integrating experimentally obtained dependences [4,5]. The reason for such errors is also [1,6,7,8]:

- accepted simplifications and tolerances;
- mathematical model error (inadequate model);
- calculation formula simplification;
- error in calculations;
- error of certain corrections and correction coefficients;
- insufficient measurement data filtering.

In determining the thermal conductivity, used the methods given in [8,9,10], that is, the calculation of the coefficient of thermal conductivity is carried out in the following ways:

• for the value of the thermistor heating temperature, which has a thermal contact with the test substance [6, 7],

• by mathematical processing of the thermistor heating thermogram during the momentum of its heating [9, 10],

• comparing the thermistor heating temperature, which has a thermal contact with the test substance and the thermistor temperature, which has a thermal contact with the reference substance [11]. Also, by comparing the thermistor power needed to heat it to a certain temperature of the thermistor, which has a thermal contact with the test substance and the thermistor power required to heat it to the same temperature that has a thermal contact with the reference substance [12].

In the latter case, the parameters of the heating mode of the thermistor are compared, which are proportional to the thermal conductivity of the reference and investigated materials, for example, proportional to the frequency of electric pulses applied to the thermistor [1].

The calculation of thermal conductivity coefficient is made on the value of the thermistor heating temperature, which has a thermal contact with the material [6, 7]:

$$\lambda_{s.m.} = \frac{BQ}{(T_{hot} - T_{cold}) - A*Q} \tag{2}$$

where: $\lambda_{s.m.}$ – thermal conductivity coefficient of the investigated material, kcal/sec·m·°C; T_{hot} – the temperature of the thermistor heated by electric current, °C; T_{cold} – thermistor temperature before electric current heating, °C; A, B – coefficients obtained as a result of gauge tests with reference materials with known thermophysical characteristics; Q – amount of heat released by the thermistor under the action of electric current, kcal/sec.

The thermistor temperature before it is heated by electric current and after heating is determined by the formula:

$$T = \frac{1}{C_1 + C_2 * \ln R + C_3 * (\ln R)^3} \tag{3}$$

where: T – thermistor temperature (T_{hot} and T_{cold}), °C; C_1, C_2, C_3 – calibration coefficients, which determine the dependence of the actual temperature of the thermistor on the value of it`s resistance; R –thermistor resistance, which is determined prior to the heating of the thermistor by electric current and after heating. In this case, voltage measurement is carried out on the thermistor and the current flowing through the thermistor, Ohm.

With the use of this method, given in [6, 7], the absolute value of the temperature of the investigated substance, the change in the thermistor power during its heating, and the significantly simplified true characteristic of the $T=f(R)$ thermistor were not taken into account.

Coefficient of thermal conductivity calculation, which is carried out by mathematical processing of the thermistor heating thermogram during the momentum of its heating [8, 9], while the thermistor has a thermal contact with the research material, carried out by the formula:

$$\lambda_{s.m.} = \frac{1}{a_1 \dfrac{\int_{t-t_h}^{t-t_h+2,69} \Delta T(t)dt}{\int_0^{t_h} P(t)dt} - a_2} \tag{4}$$

where: $\lambda_{s.m.}$ – coefficient of thermal conductivity of the investigated material, W/(m·K); a_1, a_2 – empirical coefficients; t – time, *sec*.; t_h – time during which the thermistor is heated by the pulse of an electric current, *sec*.; $\Delta T(t)$ – dependence of the heating thermistor temperature during the pulse of it`s heating from the time of heating (thermistor heating termogram), °C; $P(t)$ – dependence of the thermistor power during the momentum of it`s heating from the time of heating, W.

The heating temperature of the thermistor during the pulse heating is determined by the formula:

$$\Delta T(t) = \frac{1}{(H_0 + H_1 * \ln R(t) + H_2 * (\ln R(t))^2 + H_3 * (\ln R(t))^3 + H_4 * (\ln R(t))^4) - 273,15} - T_0 \tag{5}$$

where: $\Delta T(t)$ – dependence of the heating thermistor temperature during the pulse of its heating from the time of heating (thermistor heating thermogram), °C; H_0, H_1, H_2, H_3, H_4 – gauge coefficients that determine the dependence of the actual temperature of the thermistor on its resistance value; $R(t)$ – the thermistor resistance, which is determined in this case during the heating of the thermistor by the pulse of the electric current by a fraction of the measured voltage on it and the current flowing through it during the pulse action, *Ohm*.

Then:

$$P(t) = \frac{U(t)^2}{R(t)} \tag{6}$$

where: $P(t)$ – dependence of the thermistor power during the momentum of its heating from the time of the heating, W; $U(t)$ –voltage dependence on the thermistor during the duration of the pulse of its heating from the time of the heating, V; $R(t)$ – resistance of the thermistor, which is determined, in this case, during the heating of the thermistor by the pulse of the electric current by a part of the measured voltage on it and the current flowing through it during the pulse action, Ohm.

When using this method of thermal conductivity coefficient determining [8, 9], the absolute value of the investigated substance temperature is also not taken into account. In addition, in this method, in the determination of the coefficients $H_0,..H_4$ and the empirical coefficients a_1, a_2 by calibration testing, as well as the determination of coefficients of the polynomial of functions $T=f(t)$, $P=f(t)$, according to the measurement data, there is an error. The total error of determining the coefficient of thermal conductivity, taking into account a significant number of these coefficients, is significant and exceeds 3%.

Calculation of the thermal conductivity coefficient, which is carried out by comparing the capacity of the thermistor required for it`s heating to a certain temperature of the thermistor in thermal contact with the test substance and the thermistor power required to heat it to the same temperature in thermal contact with the reference substance with known thermophysical properties [1] , is carried out according to the formula:

$$\lambda_{s.m.} = K * \lambda_{g.m.} \frac{F}{F_0} \tag{7}$$

where: $\lambda_{s.m.}$ – coefficient of thermal conductivity of the investigated material, W/(m·K); $\lambda_{g.m.}$ –coefficient of thermal conductivity of the material taken as gauge, W/(m·K); K – gauge coefficient determined by gauge testing; F – pulses frequency of electric current passing through the thermistor, in which the thermistor is heated to a normalized temperature T_0 in case when the thermistor has a thermal contact with the research material, Hz; F_0 – pulses frequency of electric current passing through a thermistor in which the thermistor is heated to a normalized temperature T_0 in the case when the thermistor has a thermal contact with the reference material, Hz.

The calculation of the thermal conductivity coefficient, which is carried out by comparing the temperature of the thermistor heating, which has a thermal contact with the research substance and thermistor heating temperature, which has a thermal contact with the reference substance with known thermophysical properties [10], is carried out by the formula:

$$\lambda_{s.m.} = K * \lambda_{g.m.} \frac{T_{\Delta 0}}{T_{\Delta X}} \tag{8}$$

where: $\lambda_{s.m.}$ – thermal conductivity coefficient of the investigated material W/(m·K); K – coefficient of proportionality; $T_{\Delta 0}$ – heating temperature of the gauge liquid with a known coefficient of thermal conductivity, ^{o}C; $T_{\Delta X}$ – heating temperature of the research liquid, ^{o}C; $\Lambda_{g.m.}$ – thermal conductivity of the gauge material, W/(m·K).

The heating thermistor temperature is defined as the difference between the initial and final values of the temperature of the thermistor by the thermistor heating thermogram during the duration of the pulse heating.

Using the comparison method, the absolute value of the temperature for studied and reference substances, the change in the thermistor's power during its heating, the influence of the thermistor shell on its heating temperature, and the nonlinearity of the characteristic $T=f(R)$ of the thermistor were not taken into account in [1, 11].

Improved method for measuring the thermal conductivity of substances

Given the shortcomings that lead to measurement error increase, proposed a method for determining the thermal conductivity, taking into account the temperature of the test sample, the change in the thermistor power during its heating, and the thermophysical characteristics of the thermistor probe and the thermistor shell [12,13]. In order to determine the thermal conductivity of the proposed method, an analog-to-digital (ADC) converter for measuring the distortion signal of the measuring bridge is carried out, one of the arms of which is the thermistor. The thermistor has a thermal contact with the test substance. The construction of thermistor probes for liquid, solid and loose materials is shown in Fig. 1-3. Numerical values at the output of the ADC are proportional to the temperature of the thermistors. With the help of an electric pulse supplied to the measuring bridges, the thermistors warm up. During the instantaneous warming up at short intervals, the values at the output of the ADC are recorded in the memory of the microcontroller by developed device [12, 13], then transmitted to external PC, which generates a data file. These measurements are heating thermistors thermograms, according to which the temperature of their self-heating is determined.

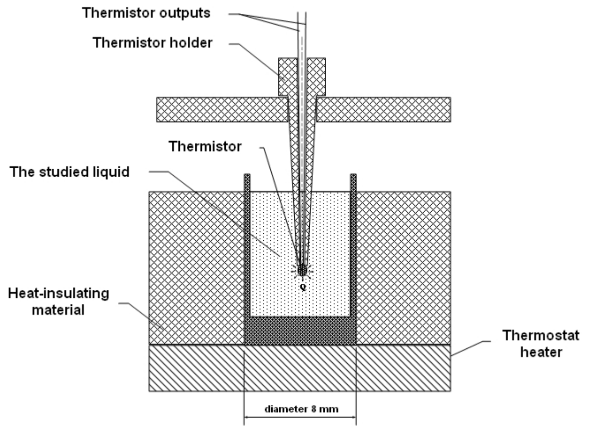

Fig. 1. Location of the thermistor probe and the test in studies of TPC fluids

Under the action of an electric current pulse, the thermal energy produced by the thermistor is dissipated by probe design and propagated in the test substance. The thermistor during the pulse gradually warms up.

The thermal conductivity coefficient by proposed method in the case where several thermistor probes in the device, are determined by the formula:

$$\lambda_{s.m.i} = \frac{P_T}{4\pi r\left(\left((N_{3mi} - N_{1mi}) - (N_{(+40)} - K_{ni}N_{1mi}) * K_{pi}\right) * K_{ki}\frac{T_{(+40)} - T_0}{N_{(+40)}} - \Delta T_0\right) * \frac{1}{K_{p.f.}}} 1 \quad (9)$$

where λ_{smi} is the coefficient of thermal conductivity of the studied material, which is defined by the data of measuring by the i-th thermistor probe; N_{3mi} is the numeric value in the final point of thermistor heating in the thermogram, obtained as a result of measuring by the i-th thermistor probe; N_{1mi} is the numeric value in the starting point of thermistor heating in the thermogram, obtained as a result of measuring by the i-th thermistor probe; K_{ni} is the coefficient that corrects the error of measuring by the i-th thermistor probe of the studied sample temperature; K_{pi} is the coefficient that corrects the dependency of the value of the difference N_{3mi} – N_{1mi}, measured by the i-th thermistor probe, on the temperature of the studied sample; K_{ki} is the coefficient that adjusts the sensitivity of the i-th thermistor probe (compensates for the error of value of the difference N_{3mi} – $N_{1m\ i}$, measured by the *i*-th thermistor probe, to the average value); $T_{(+40)}$ is the thermistor temperature that equals +40^{o}C; T_0 is the temperature of thermistor, at which N_{ADC}=0; $N_{(+40)}$ is the numeric value at the ADC output at the probe's temperature +40^{o}C, which is calculated under condition of linear dependency $N=f(T)$ [14,15,16].

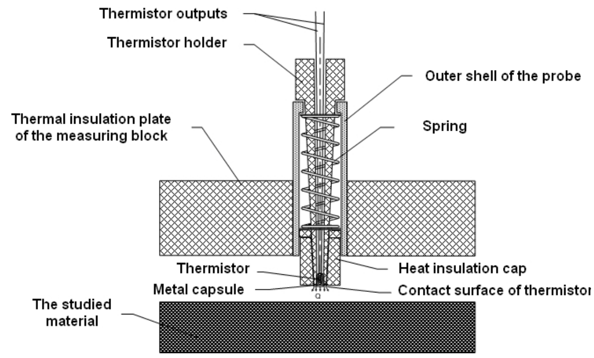

Fig. 2. Location of the thermistor probe and the test substance in studies of solid phase TPC

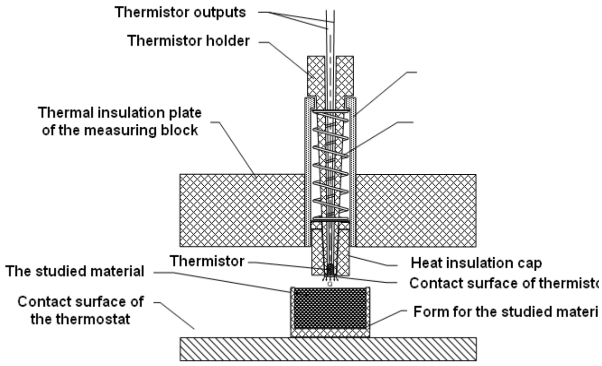

Fig. 3. Location of the thermistor probe and the test substance in TPC studies of conditionally solid and bulk materials [13]

The coefficients K_{ni}, K_{pi}, K_{ki} are determined individually for each thermistor in the course of calibration tests, by using reference fluids with known TPC as the studied ones. These coefficients are the characteristics of a thermistor probe. The coefficients ΔT_o, $K_{p.f.}$ are defined by the data of calibration tests, by using reference fluids with known TPC as the studied ones, but they are the mean values for all probes [13, 17,18].

The use of the proposed method increases the accuracy of measuring the thermal conductivity of substances by direct heating thermistor method.

Research results

A multichannel instrument for measuring the thermal conductivity of substances was used to conduct studies to determine measuring error for thermal conductivity coefficient by direct heating thermistor method [12, 13]. The device has the ability to measure the heating thermogram of all thermistor probes that are immersed in the test substance. The test substance has a good thermal contact with the thermistor and therefore, with such an error of measurement, will be minimal. The data obtained as a result of the test allows all of these methods to be used to determine the thermal conductivity. As research substances used: distilled water; 0,9% NaCl solution in distilled water; glycerin; 96% solution of ethyl alcohol in distilled water [19,20].

Prior to research, the device was calibrated using reference fluids known TPC.

When calculating the coefficient of thermal conductivity by the value of the temperature of the thermistor heating by the formula (2) [6], the value of the heating temperature is determined by the heating thermogram as the difference between the final and the initial temperatures of the thermistor heating. Corrective factors A, B – obtained as a result of gauge testing with reference materials with known thermophysical properties (distilled water and 96% ethyl alcohol solution in distilled water). The calibration coefficients (C_1, C_2, C_3) of the polynomial by function $T=f(R)$ determine the dependence of the actual thermistor temperature on the value of it`s resistance. They are determined during the calibration process, depending on the measured temperature of the thermistor from it`s resistance, which, in turn, is determined by the value of the voltage over the term and the current flowing through it. Knowing the characteristics of measuring bridge (the thermistors resistance and the pulse amplitude) and the gain of the amplifier of bridge imbalance signal, one can determine the voltage on the term and the current flowing through it. The thermistor's heating thermogram during the pulse heating is presented in Fig. 4. The values of the measurement error obtained during the calculations by this method are given in Table 1. In the same table the values of the error obtained during measurements in the manner indicated in literature are given [6, 20].

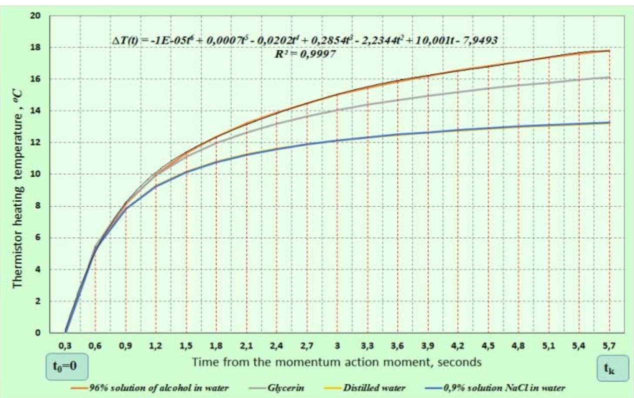

Fig. 4. Thermistor heating thermograms in $T=f(t)$ of various substances (96% solution of ethyl alcohol, glycerol, distilled water, 0.9% NaCl solution in water).

On the basis of the thermistors thermogram measurement for the process of thermistor heating, the functions $T=f(t)$ and $P=f(t)$ are determined, respectively, in Fig. 4 and Fig. 5. According to formula (4), the calculation of thermal conductivity coefficient value by the method of mathematical processing for heating thermistor thermogram [8, 9]. When integrating the experimental dependences $T=f(t)$ and $P=f(t)$, the time interval at which the integration is carried out is equal to the duration of the thermistor heating up pulse. The values of the measurement error, which were obtained during the calculations of this method, are given in Table 1. In the same table the values of the error obtained from measurements in the manner indicated in literature [8, 23] are given.

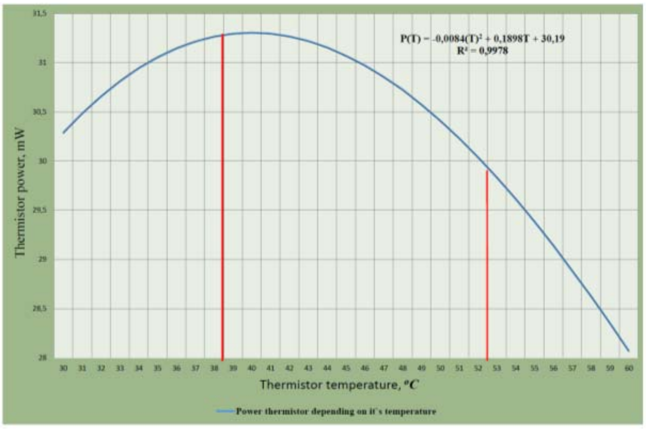

Fig. 5. The dependence of the thermistor's power on it`s temperature $P=f(t)$ during the heating (between red vertical lines, the line on the left – the initial heating temperature, the right line – the final heating temperature).

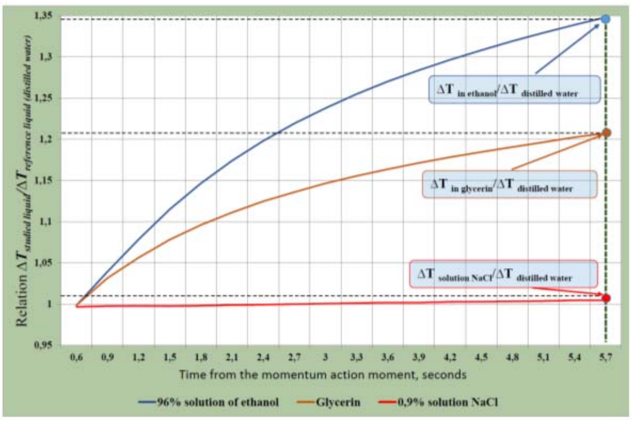

Fig. 6. Measured carried out by the tests, the value of the thermistor heating temperature ratio in the test substances (96% solution of ethyl alcohol, glycerol and 0.9% NaCl solution in distilled water) during the heating pulse.

When calculating the thermal conductivity of thermistor heating temperature, which has a thermal contact with the test substance and the thermistor heating temperature, which has a thermal contact with the reference substance with known TPC [11,24,25,26], initially the thermogram heating is determined by the ratio $T_{\Delta 0}/T_{\Delta x}$ (7), that is, the heating temperature of thermistor, which has a thermal contact with the reference substance to the heating temperature of the thermistor, which has a thermal contact with the test substance. The investigated heat conductivity of the investigated material is calculated as the product of the thermal conductivity of the reference substance on resulting ratio and the coefficient K, which is determined by calibration testing. In this case, distilled water is taken for the reference liquid (thermal conductivity at +40°C – 0,629 W/(m·K). Measured by the tests carried out, the value of the ratio of the temperature of the thermistor heating in the studied fluids (96% solution of ethyl alcohol, glycerol and 0.9% solution of NaCl in distilled water) during the pulse heating, are shown in Fig. 6. Measurement error values obtained during calculations by this method are given in Table 1. In the literature earlier, measurement data of the error value obtained during calculations in this way are not given in the literature [11, 27].

Calculation of the thermal conductivity coefficient, which is carried out by comparing the capacity of the thermistor, necessary for it`s heating to a certain thermistor temperature in thermal contact with the investigated substance and the capacity of the thermistor needed to heat it to the same temperature in thermal contact with the reference substance in the research [1], was carried out by the (8). However, in this case, for the calculations, the ratio by the time of additional thermistor heating to the temperature of 13°C in the research fluid until the time of additional heating of the thermistor in the reference liquid to the same temperature determined by heating thermogram was taken. In this case, also distilled water is taken as reference liquid (thermal conductivity at +40 ^{o}C – 0,629 W/(m·K). In fig. 7 shows the technique of determining the time of additional thermistor heating to a temperature of 13°C in different investigated fluids.

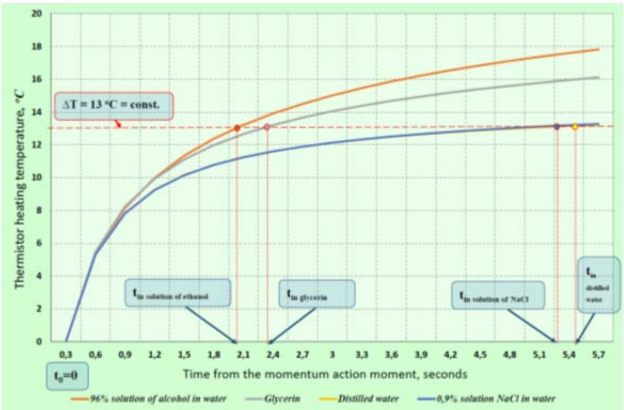

Fig. 7. Method of determining the time of additional thermistor heating to a temperature of 13^{o}C in different investigated fluids.

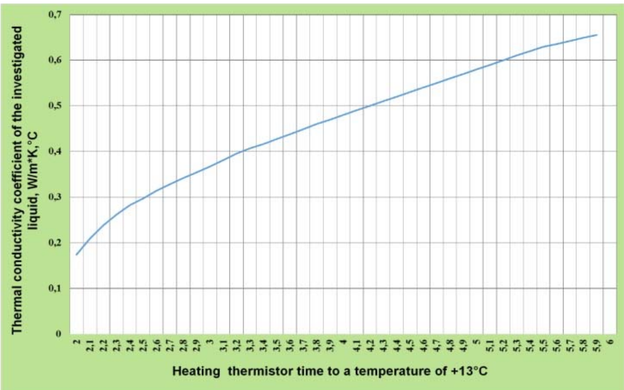

Fig. 8. Measured in the testing process, the real dependence of the value of the thermal conductivity coefficient for studied fluids from the time of thermistor heating to a temperature of 13^{o}C.

To verify the correctness of the calculated formula, it was checked for research substances with known TPC. For this we substitute the known values of the thermal conductivity coefficient of known investigated substances in (8), and the results of testing the thermistor temperature up to 13^{o}C in these substances are obtained as a result of testing. The results of this research are presented in Fig. 8. It is clear from the graph that the real dependence of the thermal conductivity coefficient on time of it`s heating to 13^{o}C is nonlinear, although according to (8) it should be linear. This is due to influence on the process of heat exchange of the probe and the investigated material of the thermophysical characteristics of thermistor shell and the additional removal of heat by the elements of the probe design, which are not taken into account in the calculations and lead to significant error of research.

Measurement error values obtained during calculations by this method are given in Table 1. In the literature, measurement data of the error value obtained during calculations in this way were not previously given in the literature [1].

To determine the thermal conductivity by proposed method, taking into account the temperature of the test sample, changes in thermistor power during it`s heating, and the thermophysical characteristics of the thermistor probe and thermistor shell (by the formula (9)), the same thermogram data as in the previous methods were used. The method of carrying out measurements and detailed description for this method is given in [12]. Also in [12] the results of measurements are given and an estimation of measurement error in this way is presented. The obtained data value of measurement error in the course of researches is given in Table 1 in comparison with the previous data of the researches set forth in [12].

Table 1. The error of measuring the thermal conductivity, which occurs when applying different methods of determining the thermal conductivity, obtained as a result of research and measurement error, is given in the literature.

Method of the research	**Defined error, %**	**Error of the method according to the literature, %**	**Note**
For the heating thermistor temperature	3,5% -7% in the range 0,1-0,7 W/m·K	3% - 4% in the range 0,3-1,5 kcal/cm·sec·oC	[6][1]
By thermistor heating thermogram during the pulse heating	3.2% - 6,0% in the range 0,1-0,7 W/m·K	2,9% - 6,1% in the range0,3-0,5 W/m·K	[8][2]
In relation to the temperature of the heating in the investigated substance in comparison with the reference substance	3,0% - 5,0% in the range 0,1-0,7 W/m·K	---	[10][3]
By the ratio of the heating thermistor power to the same temperature in the investigated and reference substances	4% - 8,0% in the range 0,1-0,7 W/m·K	---	[1][3]
For the heating thermistor temperature determined by the thermogram with correction for the value of the test substance temperature and the change of power in the heating process	< 2,0% in the range 0,1-0,7 W/m·K	< 2,0% in the range 0,1-0,7 W/m·K	[11][4]

Notes:

1 – the method was used to measure soil moisture content of 3%, 17% and 26% [6]. Water and silicone oil were used as reference fluids. The authors note that the measured error > 10% was considered to be false and was withdrawn at subsequent calculations.

2 – the method was used for measuring glycerol solutions in water [8] with thermal conductivity of 0,605 W/m·K in proportions 90/10, 68/32, and 47/53.

3 – the authors don`t present in the literature the results of researches for determine the measurement error.

4 – the results of the conducted researches coincide with the results of previous studies.

Conclusions

Existing methods [1, 6, 7] for determining the thermal conductivity of the investigated substances do not take into account the value of the temperature of the research sample, the change in the thermistor power during it`s heating, the thermophysical characteristics of the thermistor probe and thermistor shell, therefore, have a significant measurement error greater than 3% [8, 9, 10]. This error is unevenly distributed in the range of values, so measurement of TPC in a wide range of these methods is impossible. In order to reduce the error of measurement, it is necessary to introduce additional correction factors in the calculation formulas (4, 7, 8), which must be determined during the calibration tests.

The method proposed by the authors [12, 13] for determining the coefficient of thermal conductivity of substances by (9) allows measurements with an accuracy of <2.0% and extending the range of values of the thermal conductivity with the specified accuracy for liquids from 0,1 to 1,0 W/m·K.

When designing new instruments for TPC substances measuring, it is necessary to take into account the shortcomings found during the research, which increase the error of determination of TPC.

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