APPENDIX 2

Using the same substitution of (I), (II), we may state that

$$\Delta t(\tau) = \int_0^{\tau} \frac{1/\overline{a_1}}{\lambda_1 \sqrt{\pi}(\varkappa+1)(\tau-\xi)} \left\{(1-\varkappa)\exp\left(-\frac{\delta^2}{a_1(\tau-\xi)}\right) - 2\exp\left(-\frac{\delta^2}{4a_1(\tau-\xi)}\right) + (1+\varkappa) + \sum_{n=1}^{\infty}\left[(1-\varkappa)\exp\times\right.\right. \times\left(-\frac{(n+1)^2\delta^2}{a_1(\tau-\xi)}\right) + (1+\varkappa)\exp\left(-\frac{n^2\delta^2}{a_1(\tau-\xi)}\right) - 2\exp\left[-\frac{(2n+1)^2\delta^2}{4a_1(\tau-\xi)}\right]\left.\left.\right]\left(\frac{\varkappa-1}{1+\varkappa}\right)^n\right\} q(\xi)\,d\xi\,. \quad \text{(XI)}$$

NOTATION

$q(\tau)$, non-steady-state heat flux through the heat meter; λ_1, a_1, δ, thermal conductivity, thermal diffusivity, and thickness of the heat meter, respectively; λ_2, a_2, thermal conductivity and thermal diffusivity, respectively, of the base of the heat meter; $\Delta t(\tau)$, temperature gradient over the thickness of the heat meter; η, index of thermal inertia; τ, time; s, parameter of Laplace transform; $t_1(x, \tau)$, temperature of the heat meter at point x; $t_2(x, \tau)$, temperature of the base; t_c, ambient temperature; $Y_q(s)$, transfer function from the heat flux $q(\tau)$ to the temperature gradient $\Delta t(\tau)$.

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ACCURACY OF TEMPERATURE MEASUREMENTS IN DETERMINING THE THERMAL CONDUCTIVITY OF SUBSTANCES BY STATIONARY METHODS IN THE RANGE OF MODERATE TEMPERATURES

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An empirical comparison is made of the accuracy of platinum-rhodium–platinum and Chromel–Alumel thermocouples in determining the thermal conductivity of substances.

The main errors present in determinations of thermal conductivity are connected with temperature measurements. The most important of these errors, in turn, is that arising in the measurement of the temperature difference ϑ, which is necessary to determine the temperature gradient in the specimen. This difference is three-dimensional [1] and, when measured with a differential thermocouple, is calculated as the ratio of the readings of the thermocouple Δy to its sensitivity β. These values are measured with a high degree of accuracy by modern electrical instruments. The low accuracy in the measurement of ϑ is connected [1] with the process of determining β. One of the main reasons for this is that, in a given thermophysical experiment, the thermocouple may be used under conditions which differ sharply from those under which it was calibrated on special units in the thermometric laboratory. In particular, this difference leads to a situation whereby the nonuniformity of the thermoelectrodes in these two cases is manifest in different temperature fields and is a source of unknown (with respect to both sign and magnitude) additional emf's in the thermocouple circuit.

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Two types of thermocouples are widely used in the interval 20-800°C in exact measurements of thermal conductivity: platinum-rhodium–platinum (10% Rh) and Chromel–Alumel [2-4]. For example, the use of the former is recommended in [2], the latter was used in [3], and both types were used in [4]. The goal of the present work is to empirically evaluate the error in the determination of the sensitivity of these thermocouples and thus to determine their metrological capabilities in precision thermophysical studies.

We made Chromel–Alumel thermocouples out of wire 0.3 mm in diameter. We used the same coils of Chromel and Alumel wire in preparing all of the thermocouples tested. No special studies were made or treatments given with regard to the thermoelectrodes. The latter were insulated with quartz. The insulation on the working ends of the thermocouples was replaced with a two-channel ceramic during the experiment as the situation required.

The platinum-rhodium–platinum thermocouples were made from wires 0.4 mm in diameter in accordance with GOST 21007-75 and GOST 10821-75. The purity of the platinum wire used was characterized by a ratio of resistance at 100°C to resistance at 0°C equal to 1.39258. Before making the thermocouples, we degreased the 1.5-m-long electrodes in hydrochloric acid and washed them with distilled water. The working ends, 0.5 m long, were mounted in a two-channel ceramic. We prepared seven thermocouples, annealing each of them at 1100°K for 4.5 h. Three of the thermocouples, which shall hereafter be referred to as the "reference" thermocouples, were subjected to thorough study in the temperature-measurements laboratory of the All-Union Scientific-Research Institute of Metrology (VNIIM) and calibrated at the solidification points of tin, zinc, antimony, and silver [5]. The maximum divergence of the thermoemf's of these three thermocouples was 0.1°K. The thermoemf values turned out to be close to the values recommended in international tables [6]. We therefore represented the results of the calibrations in the intervals between the reference points in the form of a graph of corrections with respect to these tables [6]. For the above "reference" thermocouples of the solidification points of tin, zinc, antimony, and silver, these corrections turned out to be −1.8, −1.9, +2.3, and 8.3 μV, respectively.

The study was conducted in two stages. In the first stage, the thermocouples were calibrated as instruments for absolute measurements in the furnace of a unit designed to determine the thermal conductivity of metals and alloys [7]. To this end, we installed a copper cylinder 1 (Fig. 1) in the measurement block of the furnace at the location of the specimen. A hole was drilled along the axis of this cylinder and the ends of the thermocouples 2 with their junctions were placed in this hole. The thermocouples were calibrated up to 500°C from the readings of a small platinum resistance thermometer. Here, the ends 2 of the thermocouples being calibrated were fastened around the resistance thermometer 3 in such a way that the junctions were at a level corresponding to half the height of the thermometer. Prior studies of the thermometer in the temperature-measurements laboratory of VNIIM showed that its error was no greater than 0.01°K. In experiments up to 800°C, calibration in the furnace was performed with the three above-noted "reference" platinum-rhodium–platinum thermocouples. Here, the ends of the thermocouples being calibrated were bound in one group in such a way that the junctions were located at the midlevel of the hole. In both cases, the free space in the hole was filled with copper filings 4 to equalize the temperature field. The results of the calibrations were analyzed by the least squares method on a computer. For individual series, distinguished by the fact that several different thermocouples were studied after one, two, or three heatings to 800°C, the dependence of the thermoemf on temperature is approximated by an equation of the type

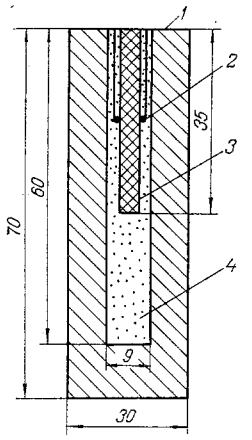
$$\Delta y = A_0 + A_1 T + A_2 T^2 + A_3 T^{-1} + A_4 T^{-2} + A_5 \ln T,$$

while the temperature dependence of sensitivity was obtained by differentiating this equation. A generalized calibration curve was constructed for each type of thermocouple from an analysis of the data obtained for all thermocouples of this given type.

Table 1 shows values of the sensitivity of the platinum-rhodium–platinum thermocouples and data characterizing the sensitivity error. It is apparent from Table 1 that, throughout almost the entire temperature range, there is a good agreement among the values of β^{I} for all of the investigated thermocouples, both within the individual series ($S_0^{I} < 0.1\%$) and between these series ($\delta^{I} \leqslant 0.2\%$). The values of δ^{I} are greater than 0.2% at the beginning of the interval, a fact due to the limited volume of data analyzed by the least squares method. We should point out the satisfactory agreement of the sensitivity values $\langle\beta^{I}\rangle$ obtained from the generalized function with the "stationary" values of sensitivity β_{st}^{I} calculated from the international tables with allowance for the above-noted corrections (the latter did not introduce any significant changes into the value of β_{st}^{I}).

Table 2 shows values of sensitivity for the Chromel–Alumel thermocouples and data characterizing the sensitivity error, with the values of β_{st}^{II} having been computed for Chromel–Alumel thermocouples from the tables of GOST 3044-77. It is apparent from Table 2 that the temperature dependence of β^{II} and $\langle\beta^{II}\rangle$ is of the same character as β_{st}^{II}, with a maximum close to 500°C. Except for this region, the values of $\langle\beta^{II}\rangle$ and β_{st}^{II} nearly coincide. The greatest divergence of δ^{II} between series is 1.2%, and it reaches 1.5% only at the ends of the temperature interval.

A comparison of δ^{I} and δ^{II} (Tables 1 and 2) shows that the furnace-calibration error of the absolute platinum-rhodium–platinum thermocouples is somewhat less than that of the Chromel–Alumel thermocouples.



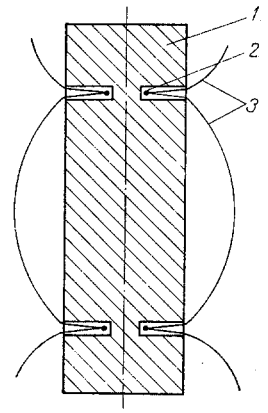


Fig. 1 Fig. 2

Fig. 1. Location of resistance thermometer and thermocouples in cylindrical copper block for thermocouple calibration.

Fig. 2. Location of differential thermocouples in cylindrical copper block to determine errors in temperature-difference measurement.

TABLE 1. Temperature Dependence of Sensitivity of Platinum-Rhodium – Platinum Thermocouples

t, °C	β^{I}_{st}, μV/°K	$\langle\beta^{I}\rangle$, μV/°K	δ^{I}, %	β^{I}, μV/°K, series 1	series 2	series 3	series 4	S^{I}_{0}, %, series 5	series 6
100	7,3	7,32	0,54	7,31	7,29	7,30	7,35	0,04	0,06
200	8,4	8,45	0,12	8,44	8,45	8,45	8,46	0,06	0,07
300	9,1	9,13	0,22	9,12	9,12	9,12	9,14	0,09	0,07
400	9,6	9,57	0,10	9,56	9,56	9,57	9,57	0,09	0,07
500	9,9	9,90	0,10	9,89	9,89	9,90	9,90	0,90	0,07
600	10,2	10,21	0,10	—	—	—	10,21	—	0,06
700	10,6	10,54	0,10	—	—	—	10,54	—	0,07
800	10,9	10,89	0,10	—	—	—	10,89	—	0,08

TABLE 2. Temperature Dependence of Sensitivity of Chromel – Alumel Thermocouples

t, °C	β^{II}_{st}, μV/°K	$\langle\beta^{II}\rangle$, μV/°K	δ^{II}, %	β^{II}, μV/°K, series 1	series 2	series 3	series 4	series 5	S^{II}_{0}, %, series 2	series 3	series 5
100	41,5	41,80	1,5	41,34	42,37	42,37	42,41	42,44	0,10	0,30	0,10
150	40,0	40,07	0,01	40,06	40,03	40,07	40,07	40,16	0,03	0,16	0,04
200	40,0	39,98	0,02	39,87	39,96	39,96	39,97	40,06	0,06	0,14	0,06
300	41,5	41,05	0,8	40,71	41,04	41,29	41,29	41,27	0,10	0,06	0,15
400	42,5	42,01	0,8	41,68	41,89	42,09	42,10	42,15	0,08	0,04	0,11
500	42,5	42,42	0,5	42,11	42,23	42,27	42,33	42,38	0,08	0,13	0,01
600	42,5	42,31	0,6	—	42,20	42,04	42,04	42,11	0,06	0,02	0,09
700	42,0	41,79	0,7	—	41,96	41,61	41,53	41,54	0,41	0,06	0,17
750	41,5	41,41	1,2	—	41,80	41,38	41,25	41,18	0,54	0,44	0,21
800	41,0	40,96	1,4	—	41,62	41,16	40,97	40,80	0,69	0,74	0,24

The second stage of the study consisted of comparing the accuracy of differential (rather than absolute) thermocouples. To this end, the junctions 2 of differential thermocouples were mounted in copper blocks 1 having the configuration of the specimen (Fig. 2). The electrodes 3 were mounted in two-channel tubes, the diameters of which were 1.5 mm and 2 mm for the Chromel – Alumel and platinum-rhodium – platinum thermocouples, respectively. The distance between the channels was 40 mm. The junctions were installed at a depth of 8 mm in one case and about 15 mm in another. We studied four differential thermocouples of each type. The measurements showed that the maximum divergence of the readings was the same for all of the differential thermocouples and was equal to 1.5% of the measured temperature

difference of 5-10°C, although the data in Tables 1 and 2 lead us to expect that this error would be less for the platinum-rhodium–platinum thermocouples. Such a large error is possibly a reflection of inexact positioning of the thermocouple junctions relative to the specimen height and the insulation of the electrodes, and it is always present in these experiments despite precautions that are taken. Thus, the complete study shows that platinum-rhodium–platinum thermocouples offer no distinct advantages over Chromel–Alumel thermocouples in determining the thermal conductivity of solids in an actual thermophysical experiment.

NOTATION

T, t, temperature; ϑ, temperature difference; Δy, thermocouple readings; A_i, parameters of approximating equation; β, sensitivity of thermocouple; $\langle\beta\rangle$, sensitivity found from the generalized function; β_{st}, "standard" values of sensitivity; S_0, standard deviation of sensitivity for a given series; δ, maximum deviation of β from $\langle\beta\rangle$ in different series; I, II, indices indicating that the values pertain to platinum-rhodium–platinum and Chromel–Alumel thermocouples, respectively.

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RELATIONSHIP BETWEEN THERMAL CONDUCTIVITY, SPEED OF SOUND, AND ISOBARIC HEAT CAPACITY OF LIQUIDS

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Using two liquids – water and toluene – as an example, the author determines the dependence of the coefficient of thermal conductivity on the speed of sound and isobaric molar heat capacity for high state parameters.

The relationship between the coefficient of thermal conductivity, speed of sound, and isobaric molar heat capacity for pure liquids at atmospheric pressure was established by the formulas of Sakiadis and Kouts [1] and Filippov [2]:

$$\lambda = Lu(c_p\rho)\,, \tag{1}$$

$$\lambda = \psi u(c_p\rho)^{2/3}k^{1/3}\,. \tag{2}$$

Equations (1) and (2) were derived with the assumption that heat transfer occurs by means of hyperacoustic oscillations of the medium. Values of λ were computed from Eq. (1) for 69 different liquids, where the standard deviation was 13% [1], and from Eq. (2) for 19 normal liquids, with a standard deviation of 7% [2].

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