A UNIFIED SERIES OF INSTRUMENTS FOR THERMOPHYSICAL MEASUREMENTS

S. E. Buravoi, V. V. Kurepin,
G. S. Petrov, E. S. Platunov,
V. I. Solov'ev, V. B. Yasyukov,
and V. M. Kozin

UDC 536.2.083

A set of industrial instruments is discussed for the investigation of the thermal conductivity, heat capacity, thermal diffusivity, and heats of phase transitions of a wide class of solid substances from thermal insulators to metals in the temperature region from −100 to + 400°C.

General Characterization of the Set

The set of methods and instruments discussed in the article provides for measurement of the thermophysical properties of solid materials having a thermal conductivity λ = 0.1-100 W/m·°K. The instruments form a unified series and as its foundation they have the basic construction of the IT-400 dynamic calorimeter (a device for measuring thermophysical properties up to 400°C) (Fig. 1).

The model was developed in connection with the preparation of the thermophysical apparatus for industrial production. The new instruments differ from the DK-400 instruments [1, 2], which were developed earlier and have found wide popularity, by an increased degree of unification of the construction and the measurement systems, by high reliability and simplicity of servicing, and by technological efficiency of preparation and assembly. The measurement methods and the heating and cooling units have been changed considerably. In particular, the water cooling of the housing and core of the calorimetric device was replaced by gaseous cooling, natural and forced.

Four models of instruments for the independent or combined investigation of thermophysical properties have now been created on the foundation of the basic construction. In each instrument the measurements are made on specimens of cylindrical shape 15 mm in diameter and from 1 to 30 mm high. A test is set up in the mode of monotonic (nearly linear) heating of the specimen at an average rate of about 0.1°K/sec at temperature drops on the specimen of 3-30°K. The temperature dependence of the parameter being studied is determined in one test. In all the instruments the test mode (the temperature drops on the specimen and the nonlinearity of the heating) is chosen in such a way that the corrections for the variation of the thermophysical properties and the heating rate under ordinary conditions (outside the zone of phase transitions) do not exceed 1-2% and they can be ignored in the calculating equations [1].

The IT-400 instrument consists of two units: measurement and supply units; the instrument complex also includes a galvanometer or digital voltmeter with a sensitivity of 1 μV. The supply unit is similar to that used earlier in the instrument of [2] and provides linear variation of the voltage on the heater and regulation of the temperature of the adiabatic shell. It contains an LATR-1 variator, two RD-09P2 motors, and a UPD1-03 amplifier.

Let us examine the construction of the measurement unit on the example of the IT-λ-400 instrument for measuring the thermal conductivity of thermal insulators. It contains elements of the measurement system and the calorimetric device (Fig. 2). The measurement system provides for the determination of the temperature level from −100 to +400°C at fixed points every 25°K, using a potentiometer and switch built into the instrument and at the corresponding positions of another switch, the measurement of the temperature drop on the test specimen and of the calorimeter signal directly with the galvanometer.

State Special Construction Bureau of Thermophysical Instrument Building, Leningrad. Leningrad Technological Institute of the Refrigeration Industry. Translated from Inzhenerno-Fizicheski Zhurnal, Vol. 38, No. 3, pp. 420-428, March, 1980. Original article submitted June 4, 1979.

 0022-0841/80/3803-0240$07.50

The main components of the calorimeter (Fig. 2) are the housing 1, a detachable thermal insulation shell 2, and a metallic core. The housing 1, made of Dural, consists of two parts and has outside ribbing to intensify heat transfer from its surface. The lower part of the housing is fastened to the horizontal board of the measurement unit; the upper part can be moved in the vertical direction along a guide. The thermal insulation shell is also made from two equal parts in the form of boxes of nickel foil filled with slag wool, asbestos, aluminum foil, and other insulation. The core of the calorimeter consists of metallic parts only and has a square shape outside. The heating unit 3 and the protective adiabatic shell 7 are made of Dural and are equipped with a system of grooves on the lateral surface in which flat, two-channel, porcelain tubes containing Nichrome wire 0.5 mm in diameter are located. The leads are doubly wound. The tubes on parts 3 and 7 are fastened with nickel shielding. The heating unit and the shell are equipped with a system of openings through which the coolants are supplied with the help of connecting pipes 9 and conical funnels. The system of openings developed provides sufficiently uniform cooling of the entire core. The adopted system for cooling the calorimeter housing and core proved to be very efficient. The absence of water cooling considerably simplified the construction, increased the reliability, and made the instrument self-contained. In this case the temperature of the calorimeter housing was no higher than 65°C during a test with a core temperature of 400°C. The time for cooling the core to −150°C with liquid nitrogen does not exceed 10-15 min at a total consumption of about 2 liters.

The calorimetric devices of each model differ only in the construction of the copper base 4 on which the thermocouples, calorimeter, and test specimen are placed. The base 4 and the heating unit 3 are connected with screws and special braces to the lower half of the housing, while the adiabatic shell is connected to the upper moveable part. The braces are used to bring out the heater and thermocouple electrodes. Chromel–Alumel thermocouples 0.2 mm in diameter, insulated in the hot zone of the core by ceramic tubes about 1 mm in diameter, are used for the temperature measurements. Thermocouples 8, sheathed with stainless steel tubes, are used to measure the temperature of the adiabatic shell. The tubes are fastened to the base 4 and simultaneously are the guides for the shell. All the thermocouples are led out through the fixed part of the core and are connected to the cold-junction block.

In all the instruments a test is performed in the following order. The upper part of the calorimeter housing is raised together with the adiabatic shell. After the test specimen is mounted, the core is cooled with liquid nitrogen to the lower temperature level and the heater is turned on. During the heating the temperature level is followed through one of the thermocouples and measurements of the necessary signals are made. When the limiting test temperature is reached, the core is turned off and cooled.

Let us examine the special features of the methods and the measurement circuits of the separate instruments in more detail.

IT-λ-400 Dynamic Calorimeter

It is designed to investigate the thermal conductivity of materials with $\lambda = 0.1\text{-}5$ W/m·°K. The measurement system differs from that known earlier in [1, 2] by the use of a metallic calorimeter for measuring the heat flux entering the specimen. Such a solution considerably simplifies the measurement process and increases the accuracy, since the heating rate does not change in this case and the demands on the mode of operation of the adiabatic shell are lowered, while the corrections introduced into the calculating equation partially compensate for each other.

The test specimen 5 (Fig. 2), in the form of a disk 15 mm in diameter and 0.5-5 mm high with ground contact surfaces, is placed between the copper rod 6 and the copper contact plate 10 of the calorimeter. The size of the rod is chosen [1] so that its heat capacity c_r is at least 5-10 times greater than the heat capacity c_s of the specimen:

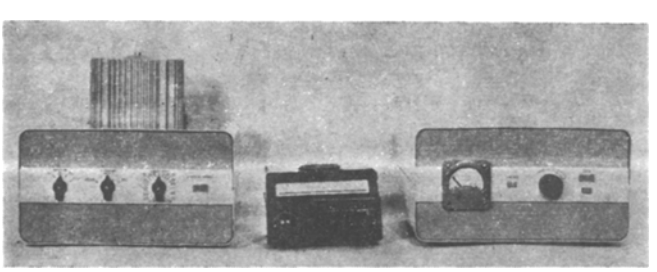

Fig. 1. General view of the IT-400 instruments.

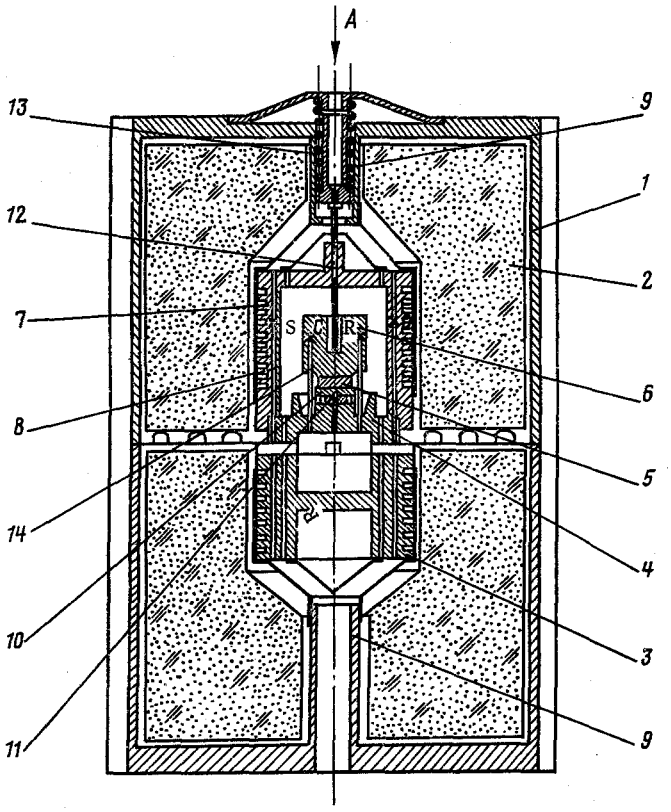

Fig. 2. Calorimetric device of the IT-λ-400 instrument.

$$(c_s/2c_r) \leqslant 0.1. \tag{1}$$

The rod is placed on two tubes 14 and is tightened by a spring 13 through a shaft 12. The tubes 14 are fastened to the base and are used simultaneously for the placement of the two thermocouples R.

In the device under consideration the calorimeter is mounted on the copper base and is built on the scheme of an auxiliary wall. The working layer of the calorimeter is a plate 11 of 12Kh18N19T steel. Several openings are made in the plate 11 to increase the thermal resistance and lower the heat capacity of the working layer. The base 4 and the plates 10 and 11 are soldered together with silver solder and are equipped with a thermopile of Chromel and Alumel for measuring the temperature drop in the working layer of the calorimeter. The "hot" junctions of the thermopile are located in the base 4 while the "cold" ones are in the plate 10. The thermocouple 11, used to measure the temperature drop in the specimen, is also located in the plate 10. Adiabatic conditions of heating of the rod are maintained by the shell heater with the help of thermocouples R (in the rod) and S (in the shell). An amplifier of type UPD1-03, to the output of which an RD-09P2 motor is connected, is used for the regulation.

The equation for calculating the coefficient of thermal conductivity without corrections for nonlinearity can be obtained from the equations of heat balance for the middle layer of the calorimeter and the test specimen:

$$Q_c(\tau) = k_c^*(t)\,\vartheta_c(\tau) = \left(\frac{1}{2}\,c_c + c_p + c_s + c_r\right) b - \Delta Q, \tag{2}$$

$$Q_s(\tau) = \frac{\lambda(t)}{\delta}\,S\vartheta_s(\tau) = \frac{\lambda}{\delta}\,S\,\frac{\vartheta_\Sigma}{1+\sigma_c} = \left(\frac{1}{2}\,c_s + c_r\right) b - \Delta Q. \tag{3}$$

Here $Q_C(\tau)$ and $Q_S(\tau)$ are the heat fluxes passing through the middle cross sections of the calorimeter and the specimen at a given time τ; $\vartheta_C(\tau)$, $\vartheta_S(\tau)$, and ϑ_Σ, temperature drops on the calorimeter, the test specimen, and measured between the rod and the contact plate of the calorimeter; δ and S, thickness and cross-sectional area of the specimen; c_C, c_p, c_S, and c_r, total heat capacities of the working layer of the calorime-

ter, the plate and specimen, and the rod; $b = d\vartheta/d\tau$, heating rate; ΔQ, heat flux to the rod through the pivots due to the nonidentical nature of the thermocouples R and S and the regulator error; $k_c^*(t)$, coefficient of heat transfer of the calorimeter.

In (2) and (3) it is assumed that the rates of heating of the rod, the specimen, the plate, and the working layer of the calorimeter differ insignificantly. An analysis shows that outside the zones of structural transitions in a specimen with $|k_b\vartheta| < 0.02$ this difference does not exceed tenths of a percent [1] ($k_b = (1/b)(db/dt)$ is the relative temperature coefficient of variation of the rod heating rate).

From (2) and (3), after simple transformations with allowance only for terms of the first order of smallness, we can obtain

$$\lambda(t) = \frac{\delta}{P_0(t)}, \quad P_0(t) = \frac{S}{k_c}\frac{\vartheta_\Sigma}{\vartheta_c}(1+\sigma) - 2P_{con}, \tag{4}$$

$$k_c = k_c(t) = k_c^*(t)\frac{c_r}{\frac{1}{2}c_c + c_p + c_r}, \tag{5}$$

$$\sigma = \frac{c_s}{2(c_s + c_r)}. \tag{6}$$

In Eq. (4) $2P_{con}$ and σ are corrections allowing for the thermal contact resistance and the nonidentical nature of the thermocouples R and P, as well as the heat capacity c_s of the specimen. From the structure of (4) it is seen that the corrections σ and $2P_{con}$ can compensate for each other. Calculations show that when $2P_{con} = (0.5\text{-}1)\cdot 10^{-4}$ m$^2\cdot$°K/W one can choose the specimen thickness so that the total correction does not exceed 1% and can be ignored. In this case the calculating equation can be simplified considerably:

$$\lambda(t) = k_c(t)\frac{\delta}{S}\frac{\vartheta_c(\tau)}{\vartheta_\Sigma(\tau)}. \tag{7}$$

The parameter $k_c(t)$ is a "constant" of the instrument, does not depend on the properties of the test specimen, and can be determined either from tests with KV quartz glass as a standard heat-conduction substance [3] or without a specimen, using the rod as the heat-capacity standard [1, 2]. The parameter $2P_{con}$ is determined in tests with a copper specimen.

Thus, to determine the coefficient of thermal conductivity of a test specimen in a test with continuous heating of the core it is necessary to measure the temperature drop ϑ_Σ and the thermocouple readings ϑ_c at different temperature levels t.

Testing of the described system on specimens of plastic and of KV, K8, and TFI glasses [3] showed that the measurement error does not exceed 5-8% in the entire temperature range from −100 to +400°C. The calculated value of the error for materials with $\lambda = 5$ W/m·°K is 10% at a probability of 0.95.

IT-s-400 and IT-a-400 Instruments for Measuring Heat Capacity, Heats of Phase Transitions, and the Coefficient of Thermal Diffusivity

The metallic core of the IT-s-400 instrument for measuring heat capacity differs from that discussed in the preceding section only in that the contact plate 1 of the calorimeter is built in the form of a cylindrical ampule (Fig. 3a). The test specimen 2, 15 mm in diameter and 10 mm high, is placed inside the ampule in a sliding fit with contact lubricant, and the ampule is tightly covered by a lid 3. Two thermocouples R and U are mounted in the ampule-plate and one L in the base for temperature measurements. The thermocouple R is used together with the thermocouple S in the system for regulating the temperature of the adiabatic shell.

The equation for calculating the heat capacity can be obtained from the heat-balance equation

$$Q_c(\tau) = k_c(t)\,\vartheta_c = (c_s m_s + c_{con})b \tag{8}$$

for the middle layer of the calorimeter plate 4 and has the form

$$c_s(t) = \frac{1}{m_s}\left[\frac{k_c(t)\,\vartheta_c}{b} - c_{con}\right]. \tag{9}$$

Here m_s is the mass of the specimen; $k_c(t)$, coefficient of thermal conductivity of the calorimeter; $\vartheta_c = \vartheta_c(\tau)$ and $b = b(\tau)$, temperature difference on the calorimeter and the heating rate; c_{con}, total heat capacity of the ampule, the lid, and half the working layer of the calorimeter.

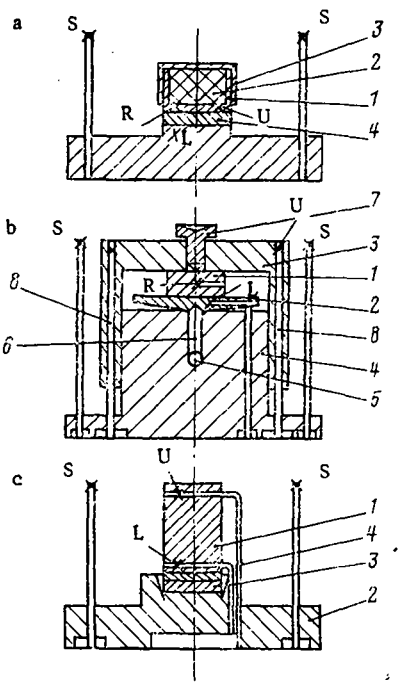

Fig. 3. Measurement cells of IT-s-400 (a), IT-a-400 (b), and IT-as-400 (c) instruments.

Another system is preferable from the point of view of operation. At small ϑ one can change to the measurement of the delay time τ_c of the temperature in the working layer, since $\vartheta_c/b = \tau_c$ [1]. Then (9) can be represented in a more convenient form,

$$c_s(t) = \frac{k_c(t)}{m_s}(\tau_c - \tau_c^0), \tag{10}$$

while in this case the determination of the heat capacity comes down to the measurement in a test of the delay time τ_c of the ampule temperature (U) with respect to the base (L) at different temperature levels.

The parameters $k_c(t)$ and $\tau_c^0(t)$ are "constants" of the instrument, and the first is determined from tests with a specimen of known heat capacity (copper, corundum) while the second is determined with an empty ampule. The calculating equations for them can be obtained directly from (10).

In measuring the heats of phase transitions one must record curves of $\vartheta_c(t)$ and $t_U(\tau)$ in time. In this case a thermopile of five to eight junctions must be mounted in the calorimeter to amplify the signal. As in other similar cases, the calculation is made from the corresponding area of the curve of $\vartheta_c(\tau)$ and the known coefficient of thermal conductivity $k_c(t)$.

We note that Eqs. (9) and (10) do not allow for nonlinearity of the heating or a temperature dependence of the thermophysical properties of the test specimen. When necessary, the corresponding corrections can be found in [1].

As shown by investigations of quartz glass, aluminum oxide (leucosapphire and α-corundum), and 12Kh18N9T steel, the error in measuring heat capacity does not exceed 3-5% in the entire working temperature range.

The internal construction of the metallic core of the IT-a-400 instrument for measuring the coefficient of thermal diffusivity of thermal insulators and semiconductors with λ = 0.1-5 W/m·°K is shown in Fig. 3b. A method of monotonic symmetrical heating of the plate analogous to that of [2] is used in the instrument. The only differences involve the structural working out of the calorimeter and are directed at simplifying the adjustment and increasing the accuracy and reliability of the measurements.

The test specimen 1 (Fig. 3b), in the form of a disk 15-20 mm in diameter and 6-12 mm high with an opening at the center, is placed between the lower moveable plate 2 and the upper contact plate 3. The copper base 4 is fastened to the heater unit and contains an eccentric 5 and a cylindrical pin 6. With the help of the latter the lower contact plate can be moved within limits of 0.5 mm in the vertical direction, regulating the

thermal contact between the plate 2 and the base during the adjustment. Symmetry of the heating of the test specimen is thereby assured. After the adjustment the position of the eccentric is fixed with setscrews.

The upper contact plate 3 is made in the form of a hollow cylinder, the inner surface of which is in contact with the base 4 in a sliding fit. A locking contact 7 with a screw, by which the specimen is pried off of the plate after a test, is located on the end of the plate 3.

There are four thermocouples in the device for temperature measurements. One of them (L) is mounted in the lower plate, another (R) in the specimen, and two thermocouples (U) are located inside the tubes 8, which are fastened to the base 4 and are simultaneously used as guides for the upper plate 3. Adiabatic conditions between the shell and the plate 3 are maintained by the shell heater with the help of one thermocouple U and the thermocouple S in the shell, which improves the symmetry in heating the specimen and increases the measurement accuracy. In the process of heating the temperature is followed from the middle thermocouple in the specimen, and the delay times τ_{UR} and τ_{LR} of the temperature of the middle point R behind the temperatures of the upper U and lower L contact plates are measured at different levels.

The coefficient of thermal diffusivity is calculated from an equation analogous to that of [2]:

$$a(t) = \frac{\delta^2}{(\tau_{UR} + \tau_{LR} - \tau_0)} (1 - \sigma_\alpha + \sigma_\beta), \tag{11}$$

$$\sigma_\alpha = 2\left(\frac{\delta}{R}\right)^2 \frac{\mathrm{Bi}}{2 + \mathrm{Bi}}, \quad \sigma_\beta = 2\beta t, \quad \mathrm{Bi} = \frac{\alpha R}{\lambda}. \tag{12}$$

Here 2δ and $2R$ are the thickness and diameter of the specimen; $\alpha(t)$, coefficient of heat exchange at its lateral surface; $\tau_0(t)$, correction for contact thermal resistance and for the nonidentical nature of the thermocouples (it is determined as a "constant" in a test with a copper specimen); β and λ, tentative values of the coefficients of linear expansion and thermal conductivity of the specimen; σ_α and σ_β, corrections for lateral heat exchange and linear expansion of the specimen.

Testing of the system on standard specimens [3] showed that, as in the instrument of [2], the error does not exceed (3-8)% in the entire temperature range.

IT-as-400 Instrument for Comprehensive Investigation of the Thermophysical Properties of Metals

The basic configuration of instruments for determining the thermal diffusivity and heat capacity of materials with λ = 5-100 W/m·°K is analogous to that discussed in [4] and is shown in Fig. 3c in application to the basic model of the unified IT-400 instrument. The test specimen 1, 15 mm in diameter with two radial openings 1 mm in diameter near the end surfaces, is placed on the base 2 with the calorimeter 3, structurally similar to those of the IT-λ-400 instrument. Two movable thermocouples L and U, sheathed by stainless steel tubes 4, are inserted in the specimen. The height of the specimen is chosen as a function of its thermal conductivity: 20-25 mm for λ = 5-15 W/m·°K, 25-30 mm for λ = 15-50 W/m·°K, 30-35 mm for λ = 50-100 W/m °K. In a test the calorimeter signal ϑ_c, the specimen heating rate $b(\tau)$, and the delay time τ_{UL} at the point U relative to the point L are measured at different temperature levels in the process of heating. Adiabatic conditions for the open surface of the specimen are maintained by the shell heater with the help of thermocouples U and S.

The quantities $a(t)$ and $c(t)$ being determined are calculated from the equations [4]

$$a(t) = -\frac{h_L^2 - h_U^2}{2(\tau_{LU} - \tau_0)} (1 + \sigma_\alpha + \sigma_\beta), \tag{13}$$

$$c_s(t) = \frac{1}{m_s}\left[\frac{k_c(t)\,\vartheta_c}{b} - c^*\right](1 - 2\sigma_\alpha), \tag{14}$$

$$\sigma_\beta = 2\beta t, \quad \sigma_\alpha = \frac{1}{3}\,\frac{\alpha d}{\lambda}\,\frac{H^2}{d^2}. \tag{15}$$

Here h_L and h_U are the distances from the upper end of the specimen to the axes of the openings; m_s, d, and H, mass, diameter, and height of the specimen; $k_c(t)$, heat-transfer coefficient of the calorimeter, W/mV; τ_0, c^*, σ_α, and σ_β, corrections allowing for the nonidentical nature of the thermocouples (τ_0), lateral heat exchange of the specimen (c^*, σ_α), the heat capacity c_{con} of the working layer and that of the contact plate of the calorimeter (c^*), and the linear expansion σ_β of the specimen; λ and β, tentative values of the coefficients

of thermal conductivity and linear expansion of the specimen; $\alpha(t)$, heat-exchange coefficient at the lateral surface of the specimen. It must be noted that the role of the correction is different in (13) and (14). For example, the corrections σ_α become appreciable only for temperatures above 200°C, and for materials with $\lambda \geq 30$ W/m ·°K they can be ignored altogether. The correction c* depends on the nonidentical nature of the thermocouples U and S; it may be equal to zero, but it must be determined upon each change of thermocouples. The parameters $k_c(t)$, c^*, and τ_0 are determined from calibration tests with copper specimens (solid or hollow) and are calculated from the equations

$$\tau_0 = \tau_{LU}^r - \frac{h_{LR}^2 - h_{UR}^2}{2a_r}, \tag{16}$$

$$k_r(t) = \frac{c_s m_{s,r} b_r - c_s m_{s,p} b_p}{\vartheta_c^r - \vartheta^p}, \tag{17}$$

$$c^*(t) = \frac{k_c \vartheta_c}{b_r} - c_s m_{s,r}. \tag{18}$$

Here the indices r and p refer to values determined in tests with solid and hollow copper specimens, respectively.

The measurement errors for the IT-as-400 instrument do not exceed 1-3% for heat capacity and 3-8% for the coefficient of thermal diffusivity, depending on the thermal conductivity of the test material.

In conclusion, we note that the systems discussed are far from exhausting the possibilities of the IT-400 unified model. It appears that other instruments can also be created on its basis with some additions, particularly for the comprehenisive investigation of semiconductors, differential thermal analysis, the determination of linear expansions, the investigation of the thermophysical properties of liquids and gases at standard pressures, etc.

All-Union State Standard 23630. 1-79-23630. 3-79 "Plastics. Methods of determining thermophysical characteristics in the temperature range from minus 100 to plus 400°C" was developed on the basis of instruments of the IT-400 series.

The instruments will be produced commercially by the Aktyubinsk Instrument Repair Factory.

LITERATURE CITED

1. E. S. Platunov, Thermophysical Measurements on a Monotonic Regime [in Russian], Énergiya, Leningrad (1974), pp. 30-134.
2. E. S. Platunov et al., "An instrument for comprehensive thermophysical measurements (DK-asλ-400)," Izv. Vyssh. Uchebn. Zaved., Priborostr., 15, No. 1, 130-133 (1972).
3. O. A. Sergeev, Metrological Foundations of Thermophysical Measurements [in Russian], Standarty, Moscow (1972).
4. V. V. Kurepin, S. E. Buravoi, and E. S. Platunov, "An instrument for measuring the true heat capacity and thermal diffusivity of metals," Teplofiz. Vys. Temp., 9, No. 3, 611-616 (1971).