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## Heat Flux Meters for Comprehensive Measurement of Thermophysical Properties of Readily-Spoiled Foodstuffs

V.N. PAKHOMOV, A.G. MAZURENKO, L.V. DEKUSHA AND  
V.G. FEDOROV

A method of heat flux measurement for comprehensive determination of thermophysical properties of readily spoiled (labile) foodstuffs under alternating steady-state and transient heat transfer conditions is described. It is shown that it is advantageous to use the modular instruments that permit one to develop a series of heat flux meters for measuring thermophysical properties from typical modules. Methods for calibrating the instrument, for programming and performing experiments are described. The measuring errors are analyzed and the optimal operating modes of the instruments are defined.

\* \* \*

Many foodstuffs of vegetable and animal origin, and also products derived from them are altered by temperature and time. Correspondingly the thermophysical properties of food products are affected by the heat flux, temperature and rate of its variation, and the time over which heating takes place [1-3]. For purposes of process and thermal design, one needs data on the effective thermophysical properties of such foodstuffs which correct for the effect of manufacturing changes, or fit the real conditions of the food-processing industry.

The thermophysical properties of labile foodstuffs are normally measured by the comprehensive heat flux technique [4-6], whereby one alternates steady and transient conditions of heat transfer through a planar layer of the test material of thickness  $h$  and continuously measures the temperatures  $t_1$  and  $t_2$ , and heat flux densities  $q_1$  and  $q_2$  on both surfaces of this layer. The surfaces of

the layer are assumed to be isothermal, and the temperature sensors (thermocouples) and heat flux meters [7] are placed in the centers of surfaces, where the temperature field is virtually one-dimensional.

Under steady conditions the thermal conductivity is obtained from the expression

$$\lambda = qh(t_1 - t_2)^{-1},$$

and the result is referred to the temperature  $\bar{t}$ , averaged across the layer's thickness.

Under transient conditions the specimen is heated or cooled; here the mean layer temperature changes over the elapsing from  $\tau_{st}$  (start of transient conditions) to  $\tau_e$  (end of transient conditions), ranging from  $\bar{t}_{st}$  to  $\bar{t}_e$ . The quantity of heat  $Q$ ,  $J/m^2$ , accumulated (or released) by the layer is recorded by the heat flux meters, giving data for calculation of the mean effective specific heat during transient conditions:

$$cp = \frac{\int_{\tau_{st}}^{\tau_e} (q_1 - q_2) d\tau}{h(\bar{t}_1 - \bar{t}_{st})}.$$

The integral in this expression is evaluated graphically from the area  $S$  on the recorder chart contained between curves  $q_1(\tau)$  and  $q_2(\tau)$ . The result is referred to the mean temperature of the layer during the transient period.

The instruments for determining the thermophysical properties were designed by the modular unit technique. This gives a set of standard modular units from which it is possible to assemble thermophysical property sensors of the same overall design, but intended for different uses and exhibiting different performance. The heat flux meter-based instrument for measuring thermophysical properties can be subdivided into three functional parts: thermal conditions, unit consisting of a heat source and sink; container for the test specimen that is placed between the heat source and sink; and measuring cell, consisting of two heat flux meters and two thermocouples. The following devices are used as heat sources and sinks: chamber with a highly conducting bottom, through which a heat transfer fluid (water, or ethylene glycol) is pumped from an ultraprecise constant-temperature bath or from a refrigerator (when freon is used); electric heater in a metal housing; IR emitter made of NIK-220-1000 tr lamps; TOS-2 thermoelectric refrigerator [8]; high-conductivity plate with spine fins, which is submerged in the ultraprecise constant-temperature bath (containing water, or ethylene glycol) or cryostat (with liquid nitrogen).

Some of the versions of the thermal conditions unit, made of a complement of aforementioned devices, are shown in Fig. 1. In each arrangement the source is on the top, the sink is on the bottom, and the specimen container is between them. The unit shown in Fig. 1a is comprised of two identical chambers, maintained at constant but different temperatures, so that there is transfer of heat through the specimen. The working temperature range of this unit is  $-10$  to  $90^{\circ}\text{C}$ . This is also the temperature range of the unit shown in Fig. 1b, comprised of an electric heater and a chamber maintained at constant temperature. Both these units are equipped with ultraprecise constant-temperature baths with cooling systems and are used primarily for determining the temperature dependence of the thermophysical properties when heating the specimen. The unit in Fig. 1c consists of an IR emitter and a constant-temperature chamber and is employed at room temperatures. It allows one to generate low-thermal inertia transient conditions at heat fluxes from  $10$  to  $10^4 \text{ W/m}^2$ . The units in Fig. 1d and 1e are similar to the unit in Fig. 1b, but have different heat sinks, which permit one to generate lower temperatures and to perform experiments with both heating and cooling of the specimen. The temperature range of unit 1d is  $-20$  to  $70^{\circ}\text{C}$ , whereas that of the unit in Fig. 1e is  $-150$  to  $100^{\circ}\text{C}$ . The unit in Fig. 1d employs a thermoelectric refrigerator with water-cooled hot junctions. Here the temperature of the working surface of the heat sink is adjusted either by measuring the current supplied to the thermopile of the refrigerator, or by measuring the temperature of the water cooling it. The unit in Fig. 1e uses a sink with a large open, heat-releasing surface. The temperature of the working surface is regulated either by measuring the temperature of the coolant, or by measuring the depth of submersion of the spine fins into the coolant. The latter method is used in the case of liquid-nitrogen cooling [6]. The unit in Fig. 1f consists of two identical chambers, maintained at constant temperatures, which have on their working surfaces flat electric heaters that release heat at a uniform density. The chambers are connected in parallel in the loop of one of the ultraprecise constant temperature bath and their temperatures are identical. The heaters ensure transfer of heat through the specimen and precise regulation of the specimen temperature. The range of temperatures of unit 1f is  $-20$  to  $+90^{\circ}\text{C}$ . With this setup, the experiments with heating and cooling of specimens can be performed at different rates of temperature change over a heat flux range of  $10$  to  $10^3 \text{ W/m}^2$ .

The specimen-holding container is used to impart to the specimen, in particular a liquid, the shape of a plate and to protect it from the chemical and thermal effect of the surrounding medium. The container has a reserve space that receives the excess volume of the test liquid generated by thermal expansion. Our instruments employ two types of specimen containers—horizontal and vertical (Fig. 2), and these determine whether the entire instrument assembly is then vertical or horizontal.

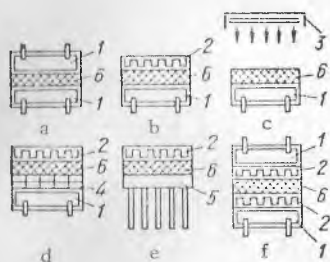


Fig. 1

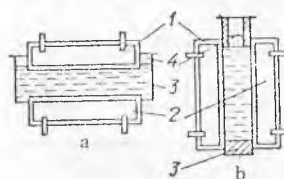


Fig. 2

Fig. 1. Different models of the thermal-conditions unit of the thermophysical-properties meter.

1) Chamber maintained at constant temperature; 2) electric heater; 3) IR heater; 4) thermoelectric refrigerator; 5) plate maintained at constant temperature; 6) specimen container.

Fig. 2. Horizontal (a) and vertical (b) specimen containers.

1) and 2) heat source and sink; 3) retaining ring; 4) reserve space.

The horizontal container sits on the heat sink. The test material (in excess amounts) is loaded from the top, and then the heat source is installed in the proper position. The source is also fastened by means of supports of equal height, fastened on the working surface of the sink. Another method is to fasten it on a translational mechanism. Such a container is used for low-viscosity liquids and of paste-type heterogeneous mixtures, in which there can occur separation of component by gravity, since when the heat flux is from the top, it prevents natural convection and suppresses the separation.

The vertical container consists of a ring, placed between the heat source and sink, which fixes their mutual arrangement. The ring has ports for loading and unloading the test material. Such a container is used for liquids, whose physical properties are such that there is no natural convection ( $Cr \cdot PR < 1000$ ) [9], and for loose materials, since it provides rather reliably uniform contact between the specimen and the working surfaces of the instrument.

The measuring cell contains two heat flux meters and two thermocouples. Instruments used for different purposes use different measuring cells, which differ in the arrangement of the heat flux meters and the thermocouples. Three models of the cell are shown in Fig. 3. In the first (Fig. 3a) the heat flux meters and thermocouples are cemented with an epoxy compound to the working surfaces of the heat source and sink. The main shortcoming of this cell is the uncontrolled increase in thermal contact resistances

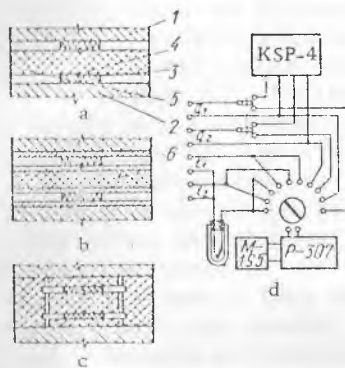


Fig. 3

Fig. 3. Measuring cells of the instrument for measuring thermophysical properties (a, b, c) and their electrical schematic (d).

1) and 2) heat source and sink; 3) heat flux meter; 4) thermocouple; 5) test material; 6) temperature-equalizing plate.

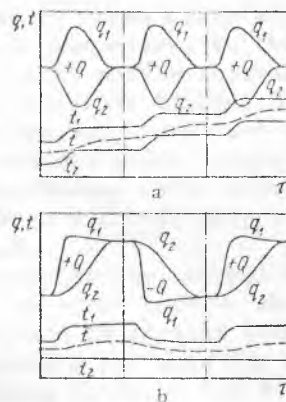


Fig. 4

Fig. 4. Temperatures and heat flux densities vs time in studies designed to yield the temperature (a) and time (b) curves.

in local cracking of the cemented joint, which results in distortion of the temperature field, and therefore also the measurements. In the second cell (Fig. 3b) the heat flux meters and thermocouples are installed between thin disks with isothermal surfaces. The disks are screwed to the heat source and sink. The thermal resistances of the disks in the center and over the periphery are the same. This reduces the distortion of the temperature field, although even in this cell it is possible to have a local rise in thermal contact resistance between the disks and the heat source and sink. In the third model (Fig. 3c) the heat flux probes and thermocouples are placed directly in the layer of the test material, one on top of the other. The inner part of the layer located between the heat flux meters is that on which the thermophysical properties are measured. The only distortions are those due to differences in thermal conductivity of the specimen and heat flux meters, but they are small, and can be corrected for.

The switching arrangement for connecting the heat flux probes and thermocouples to the recording instrumentation is of the two-wire type (Fig. 3d). Automatic measurement of transducer signals by the automatic potentiometer KSP-4 is duplicated by manual measurement on an P-307 high-resistance potentiometer with an M-195/2 galvanometer.

The heat flux meters are calibrated by three different methods. The first method involves the supply, by conduction, to the meter of a known heat flux from a calorimeter with active thermal insulation [10]. The second method involves radiative heating of the meter by an IR heater [7]. In the third method, performed after the heat flux meters are installed in the thermophysical properties instrument, one uses the results of experiments with reference materials. The thermocouples are calibrated with the help of the ultraprecision constant-temperature bath.

When measuring thermophysical properties the thermal resistances and specific heats of the heat flux probe layers, which protect the sensing elements from chemical and mechanical damage, are lumped together with the thermal resistance and specific heat of the specimen. Because of this the working formulas for determining  $\lambda$  and  $c\rho$  of the specimen are corrected by introducing the thermal resistance  $R_p$  and specific heat  $P_b$ . These corrections are determined in special experiments employing, as a minimum, two methods. In the first use is made of reference materials with known thermophysical properties ( $\lambda_r, c_r, \rho_r$ ) and, from the results of each experiment, the properties are determined from the formulas

$$R_p = \frac{\Delta t}{q} - \frac{h}{\lambda_r}, \quad P_p = \frac{Q}{\delta t} - c_r \rho_r h,$$

where  $\Delta t = t_1 - t_2$ , whereas  $\delta t = \bar{t}_e - \bar{t}_{st}$ . In the second method no consideration is given to the thermophysical properties of the reference material, and the corrections are determined from results of two experiments, performed with different thicknesses of test material

$$R_p = \frac{h''}{h'' - h'} \left[ \left( \frac{\Delta t}{q} \right)' - \frac{h'}{h''} \left( \frac{\Delta t}{q} \right)'' \right];$$

$$P_p = \frac{h''}{h'' - h'} \left[ \left( \frac{Q}{\delta t} \right)' - \frac{h'}{h''} \left( \frac{Q}{\delta t} \right)'' \right].$$

(the quantities designated by one and two primes correspond, respectively, to the first and second experiment).

The working equations for determining the thermal conductivity of a specimen from the results obtained under steady-state conditions and of the bulk specific heat of the specimen from results obtained under transient conditions are

$$\lambda = h \left[ \frac{2e_{\Delta t} k_t}{(k_{q_1} e_{q_1} + k_{q_2} e_{q_2})} - R_p \right]^{-1},$$

$$c\rho = \frac{1}{h} \left[ \frac{Sm_q m_r}{(e_{i_k} - e_{i_{st}}) k_t} - P_p \right],$$



where  $e$  is the thermal emf, generated by the heat flux meter or thermocouple designated by the pertinent subscript;  $k$  is the instrument factor for the heat flux meter or thermocouple  $m_q$  and  $m_\tau$  are the scales of the chart of the recording potentiometer.

The experimental conditions are repeated in cycles. The simplest cycle consists of an initial steady, followed by transition and final steady modes. Such a cycle makes it possible to determine the entire set of thermophysical properties on a single specimen in a single experiment. In addition to  $\lambda$  and  $c\rho$ , one determines the enthalpy gain  $\Delta i$  over the interval from  $\bar{t}_{st}$  to  $\bar{t}_e$ , the thermal diffusivity  $a$  and the thermal activity  $b$

$$\Delta i = \frac{1}{\rho h} [S k m_q m_\tau - P_d k_i (e_{i_k} - e_{i_{st}})],$$

$$a = -\frac{\lambda_{st} + \lambda_e}{2c\rho}, \quad b = \sqrt{0.5(\lambda_{st} + \lambda_e) c\rho}.$$

In the course of the experiment the experimental modes alternate without a break, and the final steady-state mode of one cycle is the start of the next cycle. The experimental modes are preset in accordance with the experimental program, which is a sequence of modes of heat supply to the specimen by the heat source (and removal by the sink) and is worked out *a priori*. This sequence depends on the purpose of the experiments and methods for controlling the thermal conditions unit. Thus, for example, in investigating the temperature dependence of thermophysical properties each successive transition mode is set up to occur at a temperature differing by 1 to 3°K from that of the preceding transition mode by simultaneously raising the temperature of the heat source and sink by the same number of degrees (this is done by means of the thermal conditions unit shown in Fig. 1a). The temperatures and heat flux densities in such an experiment are plotted in Fig. 4a. Here it is possible to preset the overall rate of change in temperature and the mean heat flux to the specimen.

In investigating the behavior of thermophysical properties as a function of time, transient conditions about the specified temperature are generated by alternating heating and cooling of the specimen. This is done by raising and reducing the heat from the source while maintaining the sink at the same temperature (here one uses the thermal conditions unit shown in Fig. 1b). The range of temperature fluctuations in this experiment is 0.5 to 2.0°K. A graph of temperatures and heat flux densities is shown in Fig. 4b.

In analyzing the error in determining thermophysical properties with these instruments we determined the probable sources of systematic errors by the technique described by Sergeyev [11]. The following systematic errors were taken into account: nonuniformity of temperature field in the specimen, temperature dependence of the specimen's thermal conductivity, effect of radiative and convective heat transfer, heat losses along wires, effect of idle characteristics of the instrument. Among sources of



instrumental errors are error in calibrating the heat flux meters and thermocouples, dynamic calibration error in measuring transient heat fluxes, error of secondary measuring instruments, and error in measuring the specimen thickness. Some of the systematic errors can be eliminated (or significantly reduced) by proper design and programming of the measurements, the remaining are incorporated into the instrument characteristics and determined in the course of calibration.

The random errors were estimated statistically. Under steady-state conditions over the thermal conductivity range of 0.05 to 0.70 W/m-°K and temperature interval from 5 to 90°C, the relative rms error in determining the thermal conductivity is between 2.5 and 3 percent, whereas over the specimen thermal conductivity range of 0.7 to 1.3 W/m-°K and temperature interval -70 and 5°C it is 3 to 5 percent. The rms error in determining specific heat under transient conditions is minimized by analysis and selection of optimal transition modes [12] and experimental design, by the Box-Wilson method, with respect to the factor of type of the transition regime (rate of accumulation of heat by the specimen under transient conditions) and the factor of thickness of the specimen [13]. At optimum values of these factors the relative rms error in determining the specific heat at temperatures between 5 and 90°C is 3 to 5 percent, and for the temperature range from -70 to 5°C it is 5 to 7 percent.

Using the instruments described above, we have investigated the thermophysical properties of various readily spoiled liquid, viscous and bulk foodstuffs, including milk and food, dairy cream, cheese, concentrated and skimmed milk, grain mash, fruit and berry desserts, semiprocessed biscuit materials, emulsions, mixed feed, granular rice, table-salt solutions, glucose and saccharose solutions, and solutions of benzylpenicillin.

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Institute of Food Technology,  
Kiev



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