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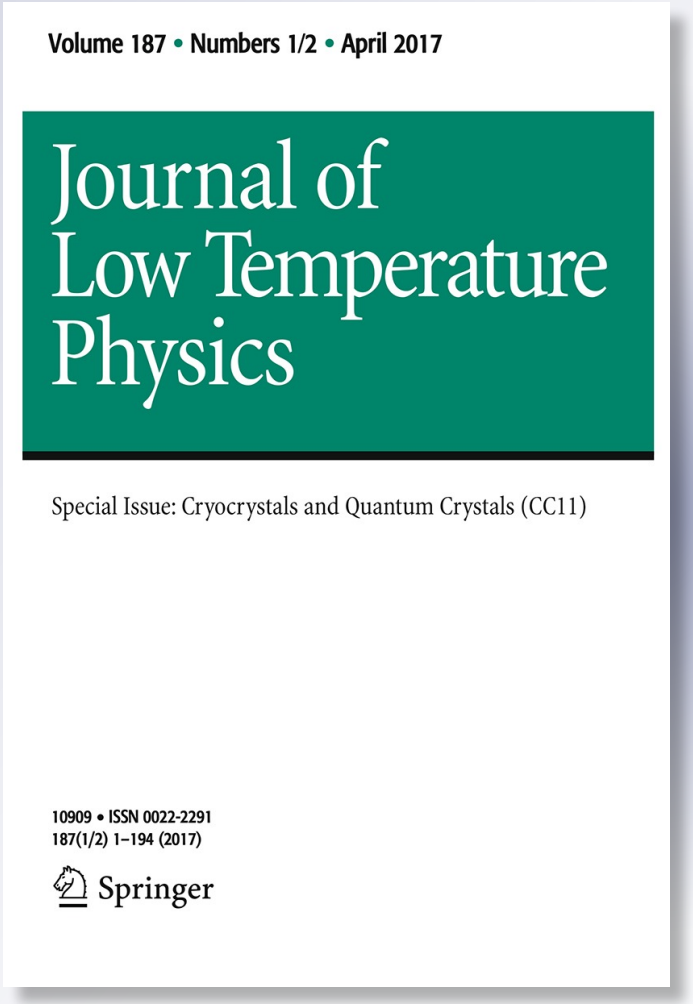

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Experimental Investigation of Thermal Conductivity of Meat During Freezing

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Abstract The cryogenic technologies of processing and storage of agricultural products are becoming increasingly indispensable in the food industry as an important factor of ensuring food safety. One of such technologies is the shock freezing of meat, which provides a higher degree of preservation of the quality of frozen products in comparison with traditional technologies. The thermal conductivity of meat is an important parameter influencing the energy consumption in the freezing process. This paper presents the results of an experimental investigation of the temperature dependence of the thermal conductivity of beef. The measurements were taken by using a specially designed measurement cell, which allows covering the temperature range from 80 to 300 K.

Keywords Thermal conductivity · Freezing · Meat

1 Introduction

Ever-increasing demands to improve the quality, biological availability and taste qualities of food, as a whole, and meat products, in particular, require developing new advanced technologies of their processing and subsequent storage. Development and implementation of the food shock freezing technology is one of the important examples of a technology that meets those objectives [1–3]. Despite intensive fundamental

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and applied research in this area over the past 40–50 years, this subject still remains relevant. This is due to the fact that in recent years, most studies focused on optimizing the process of cryotreatment to achieve a reduction in the prime cost of the final product [4,5]. The most important condition in this respect is the knowledge of the mechanical and thermal properties of frozen products, such as density, specific heat capacity, thermal diffusivity and thermal conductivity. Given the fact that the water content in meat ranges from 47 to 78% [6], it is evident that these properties are largely determined by the properties of solid water formed in the course of the cryotreatment. It is well known that [7,8], depending on the rate of formation of the solid phase of water, it may be formed as large hexagonal crystals (Ih), polycrystalline cubic crystals (Ic) and, upon ultra-fast cooling (quenching), an amorphous glassy state (ASW—amorphous solid water). It is clear that upon freezing of water into the solid phase the mechanical impact of its expansion on the muscle tissue of meat will largely determine the degree of its preservation upon freezing. In addition, heat-conducting properties of meat undergoing freezing significantly depend on the structure of the ice formed in the intercellular and intracellular spaces of the muscle tissue [9], i.e., on the cryotreatment temperature. The knowledge of this information will allow selecting the most economical mode of shock treatment in order to achieve the desired freezing temperature. This kind of information is particularly relevant for the selection of optimal cooling modes of sufficiently large quantities of meat. In this case, the efficiency of cryotreatment will depend largely on the speed of transition of the temperature front within the cooled sample at various stages of freezing.

There are many other factors which influence the choice of the optimal mode of the meat freezing. The contents of fat, water and other parameters which largely determine the main characteristics of freezing samples depend on animal husbandry in the region. Thus, to optimize the cryotreatment processes of meat, the production requires corresponding thermal studies for nearly every new batch of meat.

2 Task Description and Experiment

In this work, we present the results of the study of dynamic characteristics of the process of meat freezing at different initial temperatures of cryotreatment, as well as the results of the study of thermal conductivity of meat samples depending on the temperature and freezing mode. This study specifically focuses on the choice of the cryotreatment mode, providing the possibility of varying the cooling rate of the samples up to the shock freezing condition at the maximum sample cooling rate. It is assumed that at the low rates of freezing the water component of meat will form a crystalline structure (hexagonal ice), while the ultra-fast cooling will lead to the formation of the amorphous phase. Accordingly, dynamic characteristics of cooling and thermal properties of these samples, including thermal conductivity, will be substantially different due to the difference in the properties of crystalline and amorphous forms of solid water [7,8].

The objective of this study was to determine the influence of freezing temperature on the thermal conductivity of beef depending on the sample freezing rate. Below we describe the design of an apparatus and the method employed for measuring the

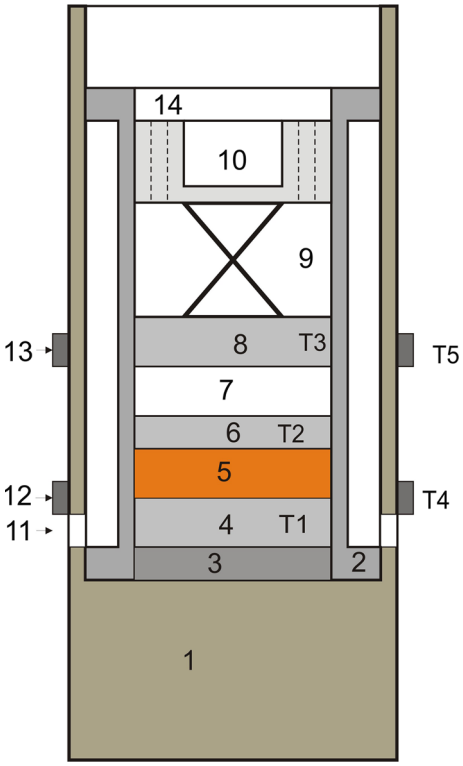

Fig. 1 Design of the measuring cell: *1*—outer casing; *2*—inner casing; *3*—heat exchanger; *4*—lower cryostat; *5*—thermal conductivity reference plate; *6*—measuring plate; *7*—test sample; *8*—top cryostat; *9*—spring; *10*—cover; *11*—cut slot; *12*, *13*—protection ring; *14*—measuring holes (Colour figure online)

thermal conductivity of meat samples in the temperature range from 100 to 300 K. A more detailed description thereof is given in Ref. [9]. We used the relative hot plate measurement method [8,9]. The desired temperature is maintained by means of an M-325 temperature controller (Lakeshore, USA). The measurement of temperature is taken using DT-670B-SD and K30AWG thermocouples (Lakeshore), calibrated with a PT-111-3S platinum resistance thermometer from the same manufacturer. The data acquisition system is based on a multi-channel analog-to-digital converter coupled to a data processing system. The measurement frequency is 10 Hz.

A schematic diagram of the measuring cell is shown in Fig. 1. The cell is mounted on outer casing 1 (steel type X18H10T) with the outer and inner diameters of 60 and 58 mm, respectively. The massive lower body part of casing 1 with the height of 60 mm functions as a heat exchanger between liquid nitrogen and the measuring cell. The main working parts of the cell are located in inner housing 2 made of vinyl plastic. The inner diameter of housing 2 is 45 mm, and all parts inserted into housing 2 have an appropriate diameter. The material of the inner housing is selected so that its thermal conductivity would not exceed the thermal conductivities of studied and reference plate. A copper heat exchanger 3 with the thickness of 8 mm is embedded into the lower part of housing 2. Heat exchanger 3 is required to ensure thermal contact between heat exchanger 1 and the measuring cell.

On the top surface of heat exchanger 3, a 5-mm-thick copper plate 4 is placed. A flat 25-watt nichrome heater and a T_1-type DT-670B-SD temperature sensor are built into copper plate 4 to measure and maintain the desired temperature. As such, plate 4 acts as a cryostat for the measuring cell. A reference plate 5 with thickness of 3.06 mm is made of PLEXIGLAS XT 20070 organic glass. This is used for a determination of the heat heat flow passing through the measuring cell [9]. On the top surface of reference plate 5, a copper measuring plate 6 is placed with the thickness of 3 mm and an integrated T_2-type K30AWG temperature sensor. The measuring plate enables averaged surface temperature values to be obtained for the samples, as well as to accurately determine the thicknesses of the reference plate and test samples regardless of the size of the temperature sensors. In this case, the temperature gradient forming across the thickness of plate 6 is negligible compared to the temperature gradients across the reference plate and the samples. Test samples with the thickness of 5–7 mm are placed between measuring plate 6 and top cryostat 8, which represents a copper disk with the thickness of 5 mm and an embedded 25-W flat nichrome heater and a T_3-type DT-670B-SD temperature sensor. With the help of the T_1- and T_3-type temperature sensors connected to the temperature controller, a given temperature difference between cryostats 4 and 8 was set and maintained. In order to fix geometric dimensions of the sample during the measurement, components 4–8 are moderately pressed using a spring 9 and secured using a cover 10. Inner casing 2 with measuring elements 3–10 is placed into outer casing 1 with the inner diameter of 58 mm and the wall thickness of 1 mm. To reduce the heat flow along the wall of casing 1, slots 11 are cut at its lower part at the bottom plane of cryostat 4, leaving 6 bridges with the width of 5 mm. The 6-mm space between housings 1 and 2 is used for tucking the connecting wires and filled with a heat insulating material. To compensate for a radial heat flow in the reference plate and the test sample, two protective rings 12 and 13 equipped with T_4- and T_5-type K30AWG temperature sensors and a 12 W nichrome heater is installed on the outer surface of housing 1 at the same level as the cryostats 4 and 8. The purpose of protection rings 12 and 13 is to maintain temperature values set by cryostats 4 and 8, respectively. A test sample is made in the form of a disk about 45 mm in diameter and 5–7 mm in thickness. During the sample preparation, account is taken of the direction of meat fibers—longitudinal or lateral. Separate measurements are conducted for each case. In this study, the fibers of meat were oriented perpendicular to the plate of the cryostat.

The measuring cell is placed vertically into a Dewar vessel (not shown), which is afterward filled with liquid nitrogen to a level of 30–40 mm above the bottom plane of heat exchanger 1. Upon cooling of the measuring cell, the values of temperature at the control points T_1–T_5 are registered. After approximately 30–40 min, the measuring cell goes into stationary mode at a given temperature, and measurements of the thermal conductivity are taken.

The described setup for the measurement of the thermal conductivity of organic samples employs the relative flat layer method, which is based on a comparison of the temperature gradients in the reference plate and test samples at constant heat flows, i.e., in accordance with the Fourier law:

$$Q = -\lambda\,(\mathrm{d}T/\mathrm{d}x) = -\lambda\,(\Delta T/\Delta x)\,. \tag{1}$$

In the stationary mode, when the heat flows through the reference plate and the test sample are equal, $Q_s = Q_x$, we obtain:

$$\lambda_s(\Delta T_s/\Delta x_s) = \lambda_x\,(\Delta T_x/\Delta x_x)\,. \tag{2}$$

Moving on to the notation introduced earlier, the thermal conductivity coefficient of the test sample λ_x can be expressed as:

$$\lambda_x = \lambda_r d_x/d_s(T_2 - T_1)/(T_3 - T_2), \tag{3}$$

where T_1 is the temperature of the lower cryostat; T_2 is the temperature of the measuring plate; T_3 is the temperature of the top cryostat; d_s is the thickness of the reference plate; d_x is the thickness of the test sample; λ_r is the thermal conductivity coefficient of the reference plate at the average temperature of the reference plate T_r; and λ_x is the sought coefficient of thermal conductivity at the average temperature of the sample T_x.

The maximum total systematic uncertainty in the measurements of the thermal conductivity coefficient is $\delta < 3$ %. Analysis of the random uncertainty based on 11 different measurements on the same sample revealed that for the confidence level of 0.95, random uncertainty does not exceed 4%. Thus, the total uncertainty in the measurements of the thermal conductivity coefficient is $\delta < 7$–8%.

The described installation is also used to study the dynamics of the samples' freezing, i.e., the rate of temperature changes over the sample thickness at various values of the cryostat temperature.

For these studies, the measurement method and apparatus were modified. The test sample was made in the shape of a disk of 45 mm in diameter and 15–18 mm in thickness. In parallel to the axial plane of the sample, two thermocouples were inserted into a depth of 20–22 mm. The distance from the lower plane of the sample to the first thermocouple and between the thermocouples was 5 mm. Elements 5–10 were removed from the measuring cell (Fig. 1), and lower cryostat 4 was cooled to a predetermined temperature. Upon reaching the cooling temperature, the sample was placed directly onto the surface of cryostat 4, after which on top of the sample a foam plastic insulating plate was placed slightly pressed by the spring 9 and lid 10. The measurements of the sample temperature were taken at a distance of 5 and 10 mm from cryostat 4 using the automated data acquisition system and the temperature controller. In this case, the cryostat temperature was kept constant in the range from 190 to 260 K.

3 Results and Discussion

Below we demonstrate the results of the measurements of temperature changes in the meat sample across its thickness during the freezing process. The aim of these experiments was to study the influence of the cryotreatment temperature on the dynamics of the sample freezing. In Fig. 2, the results of the measurements are presented. The following notations are used here: T_0 is the temperature of cryostat 4; T_1 is the temperature at the distance of 5 mm from the cryostat; and T_2 is the temperature at the

distance of 10 mm from the cryostat surface. The measurements were taken at three different temperatures T_0 of 190, 230 and 250 K.

The basic criterion for identifying the state of the sample following its freezing in our experiments is the presence or absence of a plateau in the thermogram which indicates the process of crystallization. As such, the absence of the plateau would indicate the transition of the water-containing component of meat into the amorphous (glassy) state. As we have noted, the transition into the crystalline or amorphous states upon freezing is determined by the freezing rate [7–9]. In our case, the cryotreatment temperature is the main factor influencing the freezing rate. For example, Fig. 2 shows that this temperature determines the character of temperature change in the sample.

Figure 2a shows the temperature evolution in the sample at the cryostat temperature of $T_0 = 190$ K. The thermograms T_1 and T_2 demonstrate the same character of the temperature change with time. The absence of the plateau indicates that upon cooling the test sample passes into the glassy state. Noteworthy is the presence of kinks in the thermograms in the range of 260–270 K. They indicate an acceleration of the cooling rate of the samples. The cooling rate values calculated for linear sections of the thermograms indicate a nearly threefold increase below the kink temperatures. We attribute this observation to the increasing thermal conductivity of meat upon transition to the solid state below the thermoscopic temperature, which leads to an increase in the heat transfer between the cryostat and the sample.

In Fig. 2b, the data on temperature changes in the sample at the cryotreatment temperature of $T_0 = 230$ K are shown. As seen, the character of the temperature changes at the examined points within the sample differs fundamentally. The thermogram T_1 exhibits a smooth and a gradual decrease in the cooling rate from 0.02 to 0.01 deg/s. At the same time, the thermogram T_2 demonstrates a flat plateau characteristic of the crystallization process in the time interval from 1200 to 1700 s. In this case, the cryoscopic temperature is $T = 272.3$ K, which agrees with the experimental data of other authors [10]. Therefore, we conclude that the observed difference in the shape of the thermograms is due to an insufficiently low cryotreatment temperature which does not ensure the required cooling rate. Thus, at the distance of 10 mm from the surface of the cryogenic impact the localized crystallization occurs, whereas the layers located closer to the cryostat freeze into the amorphous glass state (thermogram T_1). At the same time, it must be noted that the cooling rate of the sample (thermogram T_2) above and below the temperature $T = 272.3$ K is practically identical, equal to $V_2 = 0.02$ deg/s.

Experimental data corresponding to the cryotreatment temperature of $T_0 = 250$ K are shown in Fig. 2c. Taking into account the nature of time dependence of thermograms T_1 and T_2, it can be concluded that in the given cooling mode the crystallization of the sample extends, at least up to a distance of 5 mm from the plane of cryostat 4 throughout its thickness. The crystallization plateau in this case is not properly distinguished and is extended in time due to the openness of the system and non-negligible heat exchange between the layers remaining in different phase states. Furthermore, at low cooling temperature gradients the difference in the concentration of mineral salts in the intercellular and intracellular spaces may result in different crystallization temperatures.

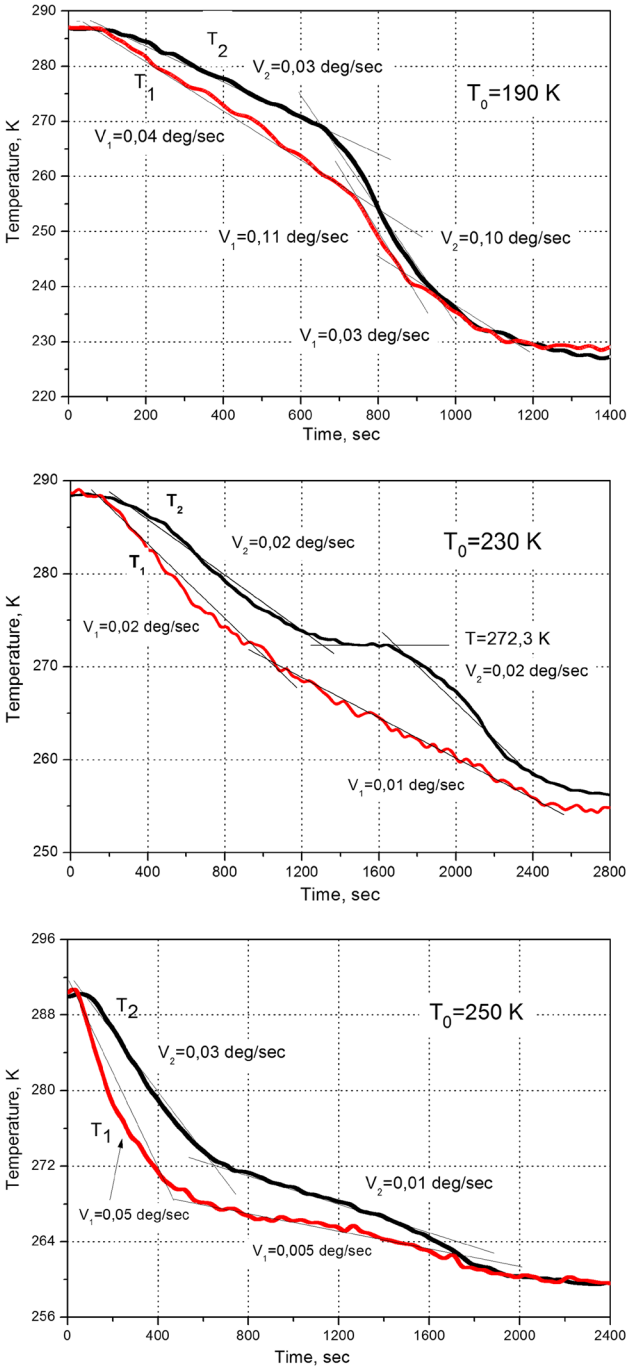

Fig. 2 Changes in temperature of meat samples at distances of 5 mm from cryostat 4 (T_1) and 10 mm from cryostat 4 (T_2) corresponding to various values of cryotreatment temperature T_0: **a** 190 K, **b** 230 K, **c** 250 K (Colour figure online)

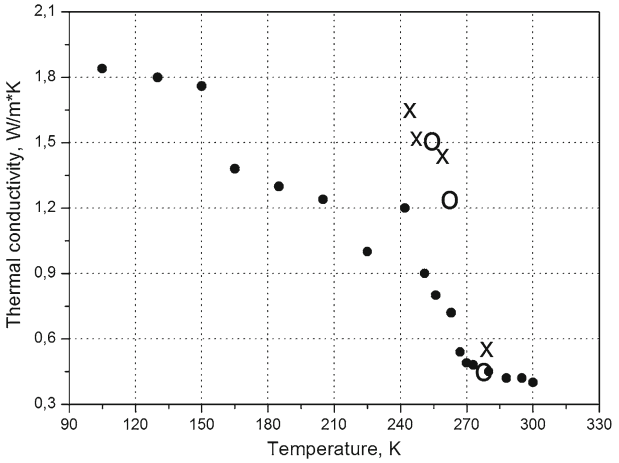

Fig. 3 Changes in thermal conductivity of a biological tissue sample (beef) during freezing: *filled circle*—this work, *open circle*—data presented in Ref. [11], *crosses*—data presented in Ref. [12]

The results of the measurements of thermal conductivity of beef during freezing are shown in Fig. 3. The data in the temperature range from 300 to 90 K were obtained as follows.

We begin with setting the initial value of the lower cryostat 4 temperature range from $T = 273$ to 300 K. Then, the temperature of the top cryostat 8 was stabilized 8–10 K above the value of the selected temperature of cryostat 4. After 20–30 min, a quasi-stationary mode was set, and then, the values of temperatures T_1, T_2 and T_3 were measured and the calculation of the thermal conductivity was done. Next, the measuring cell was cooled somewhat to lower temperature (8–10 degrees lower than the previous value). The temperature difference of 8–10 K between cryostats 4 and 8 was maintained with the help of the heaters. On reaching the quasi-stationary state, the values of the temperatures T_1, T_2 and T_3 were measured again, and the measurements of the coefficient of thermal conductivity were taken. This measuring cycle was repeated with step-like changes in the measuring cell temperature until the lowest temperature of 90 K was reached.

It is clear that the transition through the cryoscopic temperature leads to an abrupt increase in the thermal conductivity coefficient, which is associated with the transition of the sample into the solid state. Considering the character of the temperature change in the samples (Fig. 2), it can be assumed that in the temperature range from 240 to 270 K freezing leads to the formation of crystallites of the water-containing part of meat. Three data points corresponding to the thermal conductivity values at temperatures of 150, 130 and 100 K were obtained in the course of the rapid freezing of the samples to the stated values. As shown in Fig. 3, these values are higher than the values of thermal conductivity obtained during the step-like thawing.

The results of the measurements agree well with the data of other authors [11,12]. A small difference in the thermal conductivity values may be due to the difference in

the properties of the studied samples (the composition of muscle tissue and the water content of the samples).

4 Conclusions

Based on the obtained results, the cryoscopic temperature value of beef samples of $T = 272.3\,\mathrm{K}$ was determined (Fig. 2b).

The studies presented here suggest an existence of three freezing modes of beef samples depending on the character of temperature changes in the process of freezing. At cryotreatment temperatures below $T = 180\,\mathrm{K}$, the sample freezes with the transition of the water-containing component of meat into the quasi-amorphous state. This is evidenced by the monotonous character of the change in the sample temperature both above the cryoscopic temperature and substantially below it. At cryotreatment temperatures in the vicinity of $T = 230\,\mathrm{K}$, the freezing is characterized by a change in the mechanism of formation of the solid phase (Fig. 2b). Meat layers located closest to the cryotreatment surface freeze into the quasi-amorphous state; however, meat layers more distant from the impact surface undergo crystallization due to the lower cooling rate (thermogram T_2). Thus, under these conditions, the sample consists of layers frozen into different structural states, both crystalline and amorphous. At cryotreatment temperatures above $T = 240\,\mathrm{K}$, the samples are formed in the fully crystalline state.

The measurements of the temperature dependence of thermal conductivity of the meat samples indicate a step-like increase in thermal conductivity upon cooling through the cryoscopic temperature. Further decrease in temperature leads to a monotonic increase in the thermal conductivity coefficient. The samples obtained in the course of the shock freezing demonstrate even higher thermal conductivity values. The results are in fairly good agreement with the data of other authors.

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