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THERMAL CONDUCTIVITY OF SOLID CH_2Cl_2

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The isobaric thermal conductivity of solid CH_2Cl_2 was investigated by the linear-flow method under saturated vapor pressures in the temperature range from 80 K to the corresponding melting temperature and then recalculated for a constant density of the samples. The temperature dependence of isochoric thermal conductivity of solid CH_2Cl_2 is explained within a model in which the heat is transferred by phonons and above the phonon mobility edge by "diffusive" modes migrating randomly from site to site. The pressure dependence of the thermal conductivity of solid CH_2Cl_2 has been obtained.

Key words: heat transfer, thermal pressure, phonons, "diffusive" modes.

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I. INTRODUCTION

The results of the research of the high-temperature (at the temperatures close to or above the Debye temperature $T \geq \Theta_D$) thermal conductivity of molecular crystals [1–3] point to some features which go beyond the framework of traditional ideas about heat transfer processes in dielectric crystals. According to the theoretical predictions [4–6], at $T \geq \Theta_D$ the lattice thermal conductivity should be inversely proportional to the temperature $\Lambda \propto 1/T$. It is usually admitted that in dielectric crystals the heat is transferred by phonons, which are quanta of energy of every vibrational mode. In the quantum picture, such transferring processes could be described through the destruction of some quanta of elastic energy, and the creation of new ones. The most likely processes is three-quantum scattering in which two quanta are destroyed and one created or vice versa.

To obey the $1/T$ law, the volume of crystals should remain invariable, because the modes would otherwise change and so would the temperature dependence of the thermal conductivity. However, the experimental results for isochoric thermal conductivity Λ_v of molecular crystals show considerable deviations from the $1/T$ dependence [1–3,7,8.] Essentially all of the basic concepts of heat transfer were created mainly on the basis of studies of the simplest crystalline structure: atomic crystals. Therefore, features typical of molecular crystals were not taken into account in them. One of these features that can affect the temperature dependence of the thermal conductivity is the translation-rotation coupling. It should be noted that the additional factor which can determine the temperature dependence of the thermal conductivity at $T \geq \Theta_D$ also causes thermal conductivity to approach its lower limit.

As temperature increases, the phonon–phonon scattering processes enhance and the phonon mean-free path decreases, but it cannot become smaller than half the phonon wavelength $\lambda/2$ [9]. If all vibrational modes scatter for a distance of $\lambda/2$, the thermal conductivity reaches its lower limit $\Lambda_{\min}$:

$$\Lambda_{\min} = \left(\frac{\pi}{6}\right)^{1/3} k_{\mathrm{B}} n^{2/3} \sum_i \upsilon_i \times \left\{ \left(\frac{T}{\Theta_i}\right)^2 \int_0^{\Theta_i/T} \frac{x^3 e^x}{\left(e^x - 1\right)^2} dx \right\} \quad (1)$$

The summation is over three (one longitudinal and two transverse) sound modes with the sound velocities υ_i, Θ_i is the Debye cut-off frequency for each polarization in Kelvins: $(\Theta_i = \upsilon_i \left(\hbar/k_{\mathrm{B}}\right) \left(6\pi^2 n\right)^{1/3})$, $n = 1/a^3$ is the number of atoms per unit volume, a is the lattice parameter. The calculated values of $\Lambda_{\min}$ (1) were as a rule considerably smaller than experimental ones [2,8,10,11]. The most obvious reason for this difference is that the site to site transfer of the rotational energy was not taken into account. In molecular crystals the heat is transferred by mixed translation-rotation modes, whose heat capacity is saturated in proportion to the total molecular degrees of freedom [12]. Taking into account this feature of molecular crystals, lower limit of the thermal conductivity can be represented as [10]

$$\Lambda^*_{\min} = \frac{1}{2} \left(\frac{\pi}{6}\right)^{1/3} \left(1 + \frac{z}{3}\right) k_{\mathrm{B}} n 2/3 \left(\upsilon_\ell + 2\upsilon_t\right), \quad (2)$$

where υ_ℓ and υ_t are the longitudinal and transversal sound velocitie, respectively, z is the number of rotational degrees of freedom.

The purpose of this paper was to study basic features of high-temperature heat transfer in solid CH_2Cl_2.

II. THE OBJECT

The solid CH_2Cl_2 under the pressure of its own saturated vapor has only one crystallographic modification. It has orthorhombic spatial symmetry P_{bcn} with four molecules per unit cell [13–15]. It is known from the Raman spectrums and IR absorption (20 K and 77 K)

data [16,17] that the translational modes take the frequency band up to 80 cm^{-1} and partially overlap the librational modes in the 70-150 cm^{-1} (100–210K). The dipole moment of the CH_2Cl_2 molecule is $1.6D$. The lower intramolecular mode corresponds to the frequency of 285 cm^{-1} (410 K). Nuclear quadrupole resonance (NQR) on the ^{35}Cl nuclei has been observed in CH_2Cl_2 up to melting temperature $T_m = 176.3\,K$ [18]. These data also indicate that there are no molecular reorientations at frequencies above 10^4 sec^{-1}.

To date, the thermal conductivity of solid CH_2Cl_2 has been measured at premelting temperatures on several isochores [19].

III. ISOBARIC THERMAL CONDUCTIVITY AND EXPERIMENTAL TECHNIQUE

The isobaric thermal conductivity Λ_p of solid CH_2Cl_2 was investigated by the linear-flow method [8] under saturated vapor pressure in the temperature range from 80 K to the corresponding melting temperature. The measurement ampoule, in which the sample was grown, was a tube of Kh18N9T stainless steel with the length of 70 mm and an inner diameter of 7.2 mm. The temperature sensors were germanium resistance thermometers and a Copper–Constantan thermocouple, which were mounted on copper rings attached to the cell. The measurements were made by a modified heat potentiometer [20], which made it possible to minimize the error in determination of the thermal conductivity. Uncontrolled heat fluxes due to thermal radiation were reduced considerably with a radiation shield, on which a system of thermocouples and a precision heat controller reproduced the temperature field of the measuring cell. The polycrystalline samples were grown from the gas phase at the pressure of around 1.6 bar, with the temperature of the bottom of the measuring ampoule maintained close to the temperature of liquid nitrogen. The growth procedure took around 6 hours. The samples had a granular structure. Most of the grains were of 1.5 to 3 mm in the linear size. The measurements were conducted on two samples of 99.8% purity. The accuracy of the thermal conductivity measurement is 5%.

The results of the measurements are presented in Fig. 1. The different symbols correspond to the two different samples. The solid line shows smoothed values for isobaric thermal conductivity. The dashed line in the lower part of Fig. 1 is the lower limit of the thermal conductivity Λ_{min} (1) calculated for the isobaric case according to Cahill and Pohl, in the framework of the Einstein model of diffusional heat transfer directly from atom to atom [9]. From Fig. 1 it can also be noted that at premelting temperatures the experimental thermal conductivity approaches its lower limit, being no more than twice that values Λ_{min} (1). The isobaric thermal conductivity of solid CH_2Cl_2 decreases with an increase in the temperature as $\Lambda_p \propto T^{-1.21}$. Such a dependence of isobaric thermal conductivity is typical for simple molecular crystals [1,8]. It can be related to two major reasons. Firstly, the isobaric thermal conductivity also depends upon a thermal expansion of samples [12]. The second reason related to nearing thermal conductivity to its lower limit. The temperature dependence of isobaric thermal conductivity can be described in the framework of Leibfried& Schlömann formula [4] taking into account thermal expansion of samples.

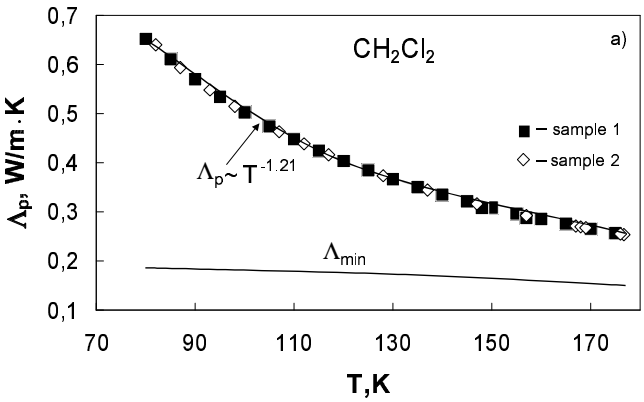

Fig. 1. Isobaric thermal conductivity Λ_p of solid CH_2Cl_2. The solid lines are the fitting curves for isobaric thermal conductivity. The lower limit of thermal conductivity Λ_{min} (1) is calculated for the isobaric case, in the framework of the Einstein model of diffusional heat transfer directly from atom to atom [9].

IV. ISOCHORIC THERMAL CONDUCTIVITY

To compare correctly experimental results of thermal conductivity with theory it is necessary to use data at constant density to exclude the effect of thermal expansion. In accordance with this assertion, the experimental results were recalculated to a constant volume of samples $V_{mol} = 47.1$ cm^3/mole [19], which are occupied by the samples at the growth temperature (80 K). The calculations were performed according to the formula [12]:

$$\Lambda_v = \Lambda_p \left(\frac{V(T)}{V_0} \right)^g, \tag{3}$$

where Λ_v is the isochoric thermal conductivity, $V(T)$ is the current molar volume of the free sample [21], V_0 is the molar volume for which the recalculation is carried out, and $g = -(\partial \ln \Lambda / \partial \ln V)_T$ is the Bridgman coefficient (Tab. 1) [21]. Figure 2 shows the isochoric thermal conductivity recalculated to the corresponding molar volume (black squares). Recalculation results are in good agreement with isochoric data cited in [19] for the same conditions (P,T,V). The isochoric thermal conductivity of solid CH_2Cl_2 decreases with increasing temperature slower than suggested by the $\Lambda \propto 1/T$ ($\Lambda_v^{CH_2Cl_2} \propto T^{-0.85}$). The last one, qualitatively conforms

to the case of strong phonon scattering, when mean free path of vibrational modes are substantially limited and approaches to phonon wavelength. In this respect, to elucidate the reasons for such deviations isochoric thermal conductivity from the dependence $1/T$, we used Debye model of the heat transfer [4] and assumption of Roufosse and Klemens [21] about lower limit of the phonon scattering length. According to the Debye formalism, the lattice thermal conductivity can be expressed as

$$\Lambda = \frac{k_{\mathrm{B}}}{2\pi^2 \upsilon^2} \int_0^{\omega_{\mathrm{D}}} l(\omega)\omega^2 \, d\omega, \tag{4}$$

where ω_{D} is the Debye frequency ($\omega_{\mathrm{D}} = (6\pi^2)^{1/3}\upsilon/a$), $l(\omega)$ is the phonon mean free path, and ω is the angular frequency. At $T \geq \Theta_{\mathrm{D}}$ the phonon mean free path is mainly determined by the U-processes and for perfect crystal can be written as

$$l_u(\omega) = \upsilon / AT\omega^2, \tag{5}$$

$$A = \frac{18\pi^3}{\sqrt{2}} \frac{k_B \gamma^2}{m a^2 \omega_{\mathrm{D}}^3}, \tag{6}$$

where the Grüneisen parameter $\gamma = -\left(\partial \ln \Theta_D / \partial \ln V\right)_T$, m is the average atomic (molecular) weight.

Expression (6) is not applicable if $l(\omega)$ becomes of the order or smaller than half the phonon wavelength $\lambda/2 = \pi\upsilon/\omega$. A similar situation was considered previously for the case of U-processes alone [21]. Let us assume that in the general case

$$l(\omega) = \begin{cases} \upsilon / A\omega^2 T, & 0 \leq \omega \leq \omega_0, \\ \alpha\pi\upsilon/\omega, & \omega_0 < \omega \leq \omega_D, \end{cases} \tag{7}$$

where α is the numerical coefficient of the order of unity. The frequency ω_0 can be found from the condition

$$\frac{\upsilon}{AT\omega_0^2} = \frac{\alpha\pi\upsilon}{\omega_0}, \tag{8}$$

It equals

$$\omega_0 = 1/\alpha\pi AT, \tag{9}$$

Condition (8) is a well-known Ioffe–Regel criterion which implies localization. We can therefore assume that the excitations whose frequencies are above the phonon mobility edge ω_0 are "localized" or "diffusive". Since completely localized modes do not contribute to thermal conductivity, we supposed that the localization is weak and the excitations can hop from site to site diffusively, as was suggested by Cahill and Pohl [9].

If $\omega_0 > \omega_{\mathrm{D}}$ the mean free path of all modes exceeds $\lambda/2$ and the thermal conductivity is determined exceptionally by the processes of phonon scattering. Substituting (5) in (4) we have the known $1/T$ law:

$$\Lambda_{\mathrm{ph}} = \frac{k_{\mathrm{B}}\omega_{\mathrm{D}}}{2\pi^2 \upsilon A T}, \tag{10}$$

At $\omega_0 \leq \omega_{\mathrm{D}}$ the integral of thermal conductivity (4) is subdivided into two parts describing the contributions to the heat transfer from the low-frequency phonons and high-frequency "diffusive" modes:

$$\Lambda = \Lambda_{\mathrm{ph}} + \Lambda_{\mathrm{dif}}, \tag{11}$$

In the high-temperature limit ($T \geq \Theta_{\mathrm{D}}$) these contributions are:

$$\Lambda_{\mathrm{ph}} = \frac{k_{\mathrm{B}}\omega_0}{2\pi^2 \upsilon A T}, \tag{12}$$

$$\Lambda_{\mathrm{dif}} = \frac{\alpha k_{\mathrm{B}}}{4\pi\upsilon}\left(\omega_{\mathrm{D}}^2 - \omega_0^2\right), \tag{13}$$

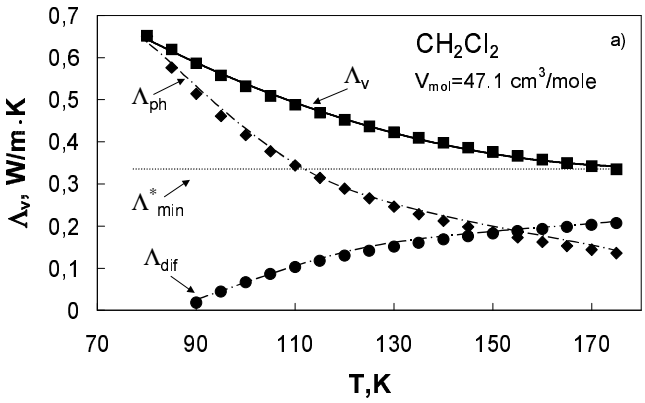

Fig. 2. Isochoric thermal conductivity Λ_v of solid CH_2Cl_2 (squares). The solid line is the fitting curve for isochoric thermal conductivity. Λ_{ph} and Λ_{dif} are relative contributions of phonons and "diffusive" modes, calculated according to Eqs. (12), (13). The lower limit of thermal conductivity Λ^*_{min} (2) is calculated taking into account the possibility of the site to site rotational energy transfer.

a, 10^{-10} m	υ, m/s	γ	α	A, 10^{-16} s/K	g
4.2	1770	2.9	1.7	1.33	4.6

Table 1. Debye model parameters of thermal conductivity used in fitting and other quantities which were used for the calculation.

To our knowledge, no information at all is available on the velocity of sound in solid CH_2Cl_2. In this respect, the phonon velocity (Table 1) was calculated by the method described in [22,23]. The Debye temperature was determined by using the boundary frequency of translational modes from the Raman and IR absorption data at 20 K. The necessary initial data were taken from [16,17].

The computer fitting of the thermal conductivity using Eqs. (11–13) was performed by the least square method, varying the coefficients A and α. The parameters of the Debye model for thermal conductivity used in the fitting (a, v), and the fitted values A and α are listed in Table 1 along with the Bridgman coefficient obtained in the experiment [19].

The fitting results for isochoric thermal conductivity are shown in Fig. 2 (solid line). The same figure shows the contributions (dot-and-dash lines) to the heat transfer from the low-frequency phonons $\Lambda_{\rm ph}$ and the high-frequency "diffusive" modes $\Lambda_{\rm dif}$ (calculated by Eqs. (12), (13)). The dotted line shows the lower limit of thermal conductivity $\Lambda^*_{\rm min}$ (2) calculated of taking into account the possibility of site to site rotational energy transfer. It is seen (Fig. 2) that in solid CH_2Cl_2 the "diffusive" behavior of the oscillatory modes starts above 90K. As temperature rises the amount of heat transferred by the "diffusive" modes increases. Above 150 K most of the heat is transported by "diffusive" modes. It can be seen from Fig. 2 that the results obtained by using the above model fit the experimental results well and maximum differences do not exceed 5%. The minimal values of thermal conductivity $\Lambda_{\rm v}$ (Fig. 2) is 1.04 times higher than $\Lambda^*_{\rm min}$ calculated by Eq. (2), and 1.5 times higher than $\Lambda_{\rm min}$ calculated by Eq. (1). The discussion of the lower limit of thermal conductivity of molecular crystals brings up the inevitable question: should the site-to-site transport of the rotational energy of the molecules be taken into account? The above correlation between the $\Lambda_{\rm min}$ and $\Lambda^*_{\rm min}$ suggests the positive answer.

V. PRESSURE DEPENDENCE OF A THERMAL CONDUCTIVITY

Molecular crystals are characterized by a large coefficient of thermal expansion. Therefore, in the course of isochoric investigations [3,4,6], a crystal sample upon heating experiences an increasing pressure produced by a measuring cell that is only slightly susceptible to thermal expansion. This process is similar to uniform compression with the negative sign, because the thermal pressure P is associated with thermal expansion of the studied sample. Upon uniform compression, the same pressure directed normally to the surface acts on each unit area of a crystal. This completely corresponds to the conditions arising in isochoric experiment. The temperature dependence of the thermal pressure (Fig. 3) in CH_2Cl_2 solid samples with the given constant molar volumes were calculated according to the assumption that the lattice heat capacity in the given temperature range ($T \geq \Theta_{\rm D}$) obeys the Dulong–Petit law, and from relationship [21]

$$\left(\frac{\partial P}{\partial T}\right)_V = \frac{\gamma C_V}{V_{\rm mol}}, \tag{14}$$

The necessary initial data were taken from [19]. As is seen from Fig. 3, the thermal pressure in the isochoric sample ($V_{\rm mol}$=47.1 cm^3/mole) changes by more than two orders of magnitude in the temperature range under investigation. The molar volume of the samples depend on their growth temperature. The results of calculations are in good agreement with the data obtained from direct measurement of the thermal pressure in solid CH_2Cl_2 [19] at premelting temperatures.

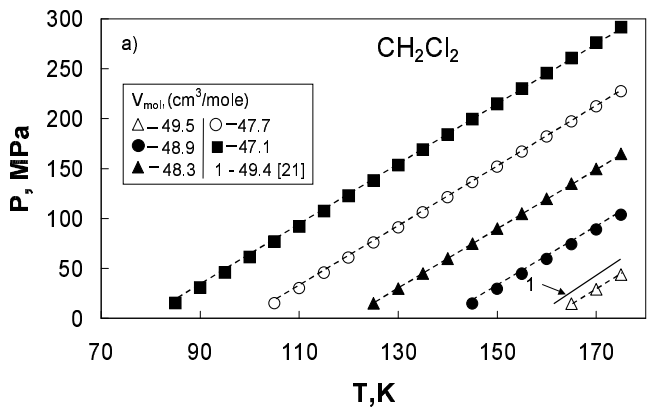

Fig. 3. Temperature dependence of thermal pressure for samples of solid CH_2Cl_2 with various molar volumes. Line 1 corresponds to experimental data [21].

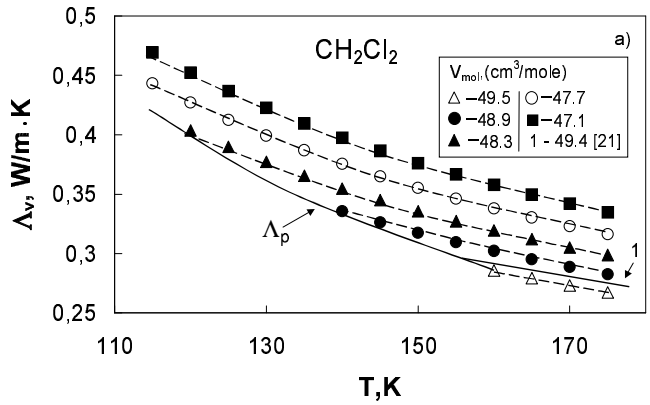

Fig. 4. Temperature dependence of isochoric thermal conductivity of solid CH_2Cl_2 with various molar volumes. Line 1 corresponds to the data of direct measurements of the isochoric thermal conductivity [21] at premelting temperatures.

The temperature dependences of the isochoric thermal conductivity Λ_v (Fig. 4) for the samples of solid CH_2Cl_2 with various molar volumes (these dependences are needed afterwards to obtain the pressure dependence of thermal conductivity $\Lambda_T(P)$) were calculated according to formula (3). Using the temperature dependences of the thermal pressure $P(T)$ (Fig. 3) and the isochoric ther-

mal conductivity (Fig. 4), were constructed the dependences $\Lambda_T(P)$ of the thermal conductivity on the pressure that arises in the studied samples as their volume changes. The results are displayed in Fig. 5. Using the methods of computer simulation, the thermal conductivity of CH_2Cl_2 was revealed to vary, as the pressure grows, according to the dependence

$$\Lambda_T = B(T) + C(T)P + D(T)P^2, \tag{15}$$

where B, C, and D are numerical coefficients, the values of which are listed in Table 2. With the increase in pressure at constant temperature, the thermal conductivity also increases (Fig. 5). The coefficient B is nothing else but the value of isobaric thermal conductivity. In equation (15) the coefficients C and D determine the influence of pressure on the thermal conductivity. By matching the units of the C and D coefficients with those of thermal conductivity it is not difficult to notice that the coefficients C and D can be determined as partial derivatives $(\partial\Lambda/\partial P)_T$ and $\left(\partial^2\Lambda/\partial P^2\right)_T$, respectively. Then dependence (15) can be written as

$$\Lambda_T = \Lambda_P + \left(\frac{\partial\Lambda}{\partial P}\right)_T P + \left(\frac{\partial^2\Lambda}{\partial P^2}\right)_T P^2 \tag{16}$$

The verification of this dependence, on the example of the C_6H_6 and CCl_4 solids, gives good accordance with the direct research results of the pressure dependences of thermal conductivity [24,25] (in the pressure range from 0.1 GPa to 1.8 the GPa distinctions between calculation (16) and experimental values do not exceed 7%).

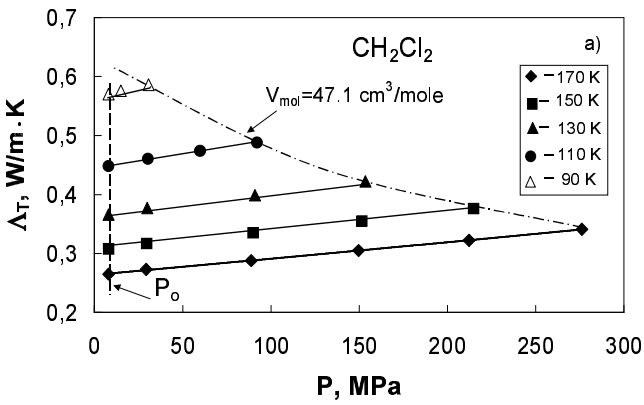

Fig. 5. Pressure dependence of thermal conductivity of solid CH_2Cl_2. P_0 corresponds to the values of thermal conductivity under saturated vapor pressure.

T, K	B, W/(m·K)	C, W/(m·K·Pa)	D, W/(m·K·Pa^2)	P, MPa
110	0.443	0.0006	$-8{\cdot}10^{-7}$	0.0÷92.1
130	0.363	0.0004	$-3{\cdot}10^{-7}$	0.0÷153.5
150	0.306	0.0003	$1{\cdot}10^{-8}$	0.0÷214.9
170	0.263	0.0003	$3{\cdot}10^{-8}$	0.0÷276.3

Table 2. Isotermal pressure dependence of thermal conductivity of CH_2Cl_2 fitted to equations of the form of $\Lambda_T = B(T) + C(T)P + D(T)P^2$.

VI. CONCLUSIONS

The current study presents some results of the investigation of high-temperature thermal conductivity in the orientationally ordered phases of molecular crystals, with solid CH_2Cl_2 as an example. The isobaric thermal conductivity of solid CH_2Cl_2 has been investigated in the temperature interval from 80 K to the melting temperature and then recalculated for a constant density of samples. The obtained experimental data was used in the calculation mechanism, which was suggested to determine the pressure dependence of thermal conductivity.

It is shown that the temperature dependence of the isochoric thermal conductivity of CH_2Cl_2 can be described within a model in which the heat is transferred by phonons and above the phonon mobility edge by "diffusive" modes migrating randomly from site to site. The phonon mobility edge ω_o is determined from the condition that the phonon mean free path restricted by the Umklapp processes cannot become smaller than half the phonon wavelength. It should be noted that the present model can be applied to the simulation of the isochoric thermal conductivity only in the orientationally ordered phases of molecular crystals, since thermal conductivity in the orientationally disordered phases of molecular crystals depends essentially on the character of the orientational molecular motion as well.

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ТЕПЛОПРОВІДНІСТЬ ТВЕРДОГО CH_2Cl_2

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Ізобарну теплопровідність твердого CH_2Cl_2 досліджено плоским стаціонарним методом під тиском власних насичених парів у діяпазоні температур від 80 K до температури плавлення. Результати експерименту перераховано до постійної густини зразків. Температурну залежність ізохорної теплопровідности твердого CH_2Cl_2 пояснено в межах моделі, у якій тепло переноситься фононами, а вище від межі рухливости фононів — "дифузними" модами, що міґрують випадково з вузла на вузол. Отримано залежність теплопровідности твердого CH_2Cl_2 від тиску.