

Thermal conductivity of bis-tetramethyltetraselenafulvalene perchlorate [(TMTSF)₂ClO₄]

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We have measured the thermal conductivity of bis-tetramethyltetraselenafulvalene perchlorate [(TMTSF)₂ClO₄] in the temperature range from 300 to 6 K and in magnetic fields to 8 T in the low-temperature regime. Due to the small thermal conductance of these samples as compared to residual gases or radiation we have employed a technique which measures both the heat flow into the sample at the hot end and heat flow out of the sample at the cold end, as well as the temperature drop across the sample. We find that the thermal conductivity is monotonically increasing with decreasing temperature from 100 to 12 K as for conventional solids but in contrast to the previously published results of Djurek and co-workers. Below the anion ordering temperature at 24 K the thermal conductivity increases at a faster rate due to reduced scattering. There is a slight reduction of the thermal conductivity at low temperatures in a magnetic field of 8 T.

INTRODUCTION

The family of organic conductors formed by charge transfer from the TMTSF molecule has exhibited intriguing and controversial phenomena. The TMTSF molecules form conducting stacks which lie in planes separated by a variety of anions: PF₆, ClO₄, ReO₄, FSO₃, etc.¹⁻⁴ These compounds, now known as Bechgaard salts, have the stoichiometry (TMTSF)₂X. At low temperatures there are a wide variety of phase transitions which depend on the particular anion and on the application of moderate pressure or high magnetic field. The compounds are metallic at room temperature but most undergo a metal-insulator transition to a low-temperature semiconducting state. In some cases this involves the spin-density-wave (SDW) instability (PF₆, SbF₆) (Ref. 5) while in other cases there is a charge-density-wave insulator which apparently is caused by an anion ordering with the periodicity of the Fermi wavelength.⁶ In both cases the application of moderate pressure (approximately 10 kbar) suppresses the SDW or the anion ordering and the material remains metallic down to approximately 1 K at which temperature there is a superconducting transition.⁷ Other salts exhibit an anion ordering transition which is at a different wave vector than k_F and hence does not cause a gap at the Fermi surface.⁸ In this case the transport is not greatly affected and the materials remain metallic.

(TMTSF)₂ClO₄, the compound used in the present study, is superconducting at ambient pressure.² On cooling from room temperature there is a monotonic increase in the conductivity. However, at 24 K there is an anion ordering transition at wave vector $(0, \frac{1}{2}, 0)$ which doubles the unit cell in the b direction (perpendicular to the highly

conducting a axis but in the conducting plane⁸). This transition leaves the material metallic and the reduced scattering from the anions lowers the resistivity on further cooling.⁹ The material becomes superconducting at 1.4 K. Below the anion-ordering transition there is also a large magnetoresistance observed when the magnetic field is perpendicular to the conducting (a - b) plane.¹⁰ Other magnetotransport properties also become large below 24 K.¹¹

The above description pertains to samples which have been slowly cooled through the anion-ordering transition. Rapid cooling through 24 K prevents the anion ordering and leads to a frozen anion "glass."¹² In the rapid cooled state (TMTSF)₂ClO₄ undergoes a spin-density-wave transition at ~ 6 K and it does not become superconducting.¹³ Unless specifically mentioned the remainder of the paper will deal with the slow cooled state.

The resistance of (TMTSF)₂ClO₄ is decreasing down to the superconducting transition. Coupled with the very large magnetoresistance observed below approximately 30 K this led to the suggestion that superconducting fluctuations were playing a substantial role in the transport properties to temperatures as high as 30 K.¹⁴ Measurements of the thermal conductivity and comparison with the electrical conductivity is a classic way of seeing whether a single particle or a collective mechanism are responsible for the electronic transport. If the ratio of thermal-to-electronic transport is equal to the Lorentz number times the temperature then the Weideman-Franz (WF) law holds. Simply stated this law works when there are three degrees of freedom (x, y, z) for each charge carrier in the system. In a superconductor the electronic current is carried by the superconducting condensate while the heat is

carried by the excitations. Thus the WF ratio drops abruptly to zero (because of the infinite electrical conductivity), while the thermal conductivity smoothly goes to zero as temperature is lowered below the transition temperature. Thus a measurement of the thermal conductivity should help resolve the controversy over the proposed superconducting fluctuations, if the electronic contribution can be separated from the phonon contribution.

The thermal conductivity of $(\text{TMTSF})_2\text{ClO}_4$ was measured previously by Djurek *et al.*^{15,16} They found that the thermal conductivity decreased sharply as temperature was lowered below approximately 30 K, in sharp contrast to the electronic conductivity which was rising. In addition they discovered that a magnetic field raised the thermal conductivity at low temperature so that in a moderate field it remained almost temperature independent below 30 K. This was regarded as very strong evidence for the existence of superconducting fluctuations.¹⁴ However, the interpretation in Refs. 15 and 16 was that the thermal conductivity was dominated by phonons rather than electrons. A straightforward indication of superconducting fluctuations was thus not evident. We decided to repeat the experiments to see what the mechanism for reduction of phonon thermal conductivity could be and particularly to investigate the striking magnetic field dependence.

In contrast to the earlier measurements we have found that the thermal conductivity increases with decreasing temperature, as is conventional in the temperature range above approximately 10 K for both electrons and phonons and that there is very little magnetic field dependence, as would be expected for phonons dominating the thermal conductivity. We associate the slight reduction in thermal conductivity that we see in a large magnetic field with the small electronic contribution. Unfortunately the determination of the Lorenz ratio is difficult in the present samples since the sample "cracking" appears to have a greater effect on the measured electrical conductivity than on the thermal conductivity. In the range 24–100 K our values range from 0.3 to over 2 times the classical value.

EXPERIMENTAL METHOD AND SETUP

Since our results are different from those previously reported it is necessary to explain how the experiment was done and what complications may arise. Thermal-conductivity measurements are much more difficult than electrical-conductivity measurements. An electric current usually flows directly through the wires and the sample. On the other hand, thermal currents are not only transferred by electrons, but by phonons, neutral-gas particles, and photons (black-body radiation). A determination of the thermal conductivity therefore requires careful determination of the heat-current flow field. It is not sufficient to assume that the entire heat current flows through the sample. This is especially true for the present organic conductor whose thermal conductivity is very low and whose cross section is small, increasing the surface-to-volume ratio.

In the experiments of Ref. 15, two assumptions were made: (1) that the thermal current went through the sample with no loss to surrounding gas or radiation and (2)

that all of the contact thermal resistances were negligible relative to that of the sample. In our initial experiments we eliminated the second of these problems by using the apparatus shown in Fig. 1(a). This is the thermal conductivity equivalent of a conventional four-probe electrical-conductivity measurement. In this configuration the samples were mounted on constantan leads which had the advantage that their properties are independent of magnetic field, but the disadvantage that the relaxation time was quite long. With the help of Djurek another apparatus was constructed with a faster relaxation time but utilizing a heated copper wire to control the temperature drop across the sample. This wire is of course strongly magnetic-field-dependent in its thermal properties.

The final configuration was a modification of Djurek's design to monitor the heat into the sample, the heat out of the sample, and the temperature drop along the sample as shown in Fig. 1(b). By using two thermocouples on the sample, instead of a single differential thermocouple, it was also possible to simultaneously measure the electrical conductivity.

The apparatus is constructed from a solid piece of oxygen-free high-conductivity (OFHC) copper as shown at the top of Fig. 1. The leads are thermally anchored outside the copper block and are fed to the underside of the flat plate where they are soldered to No. 18 insulated copper wires which are glued (GE7031) into holes drilled 0.001 in. larger than the wire diameter. A copper can is fitted over the apparatus and screws are used to tighten it onto an indium washer. Thus the sample and associated thermocouples and leads "see" a uniform temperature as they are surrounded by $\frac{1}{8}$ -in. thick copper. A copper wire 2 in. long is soldered to the copper plate and twisted into a coil. At the other end a 25- Ω heater made from manga-

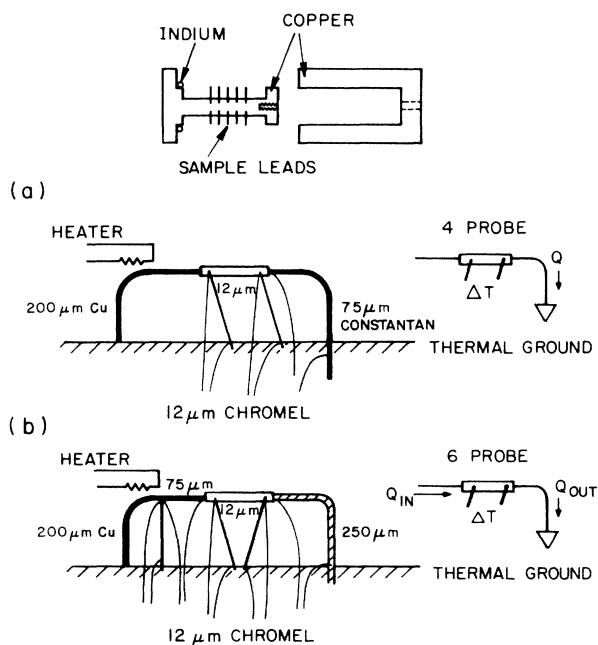


FIG. 1. (a) Schematic of a "four-probe" thermal-conductivity measurement apparatus. (b) Schematic of a "six-probe" apparatus. Inset: Schematic of a sample holder.

nin is glued to the wire and a 75-micron constantan wire is attached. The thermal conductance of the copper wire and the heat applied are used to vary the temperature across the rest of the sample-thermocouple arrangement. Two 12-micron chromel wires are attached to the 75-micron constantan to form a differential thermocouple which measures the heat going into the sample.

The sample is usually attached to the 75-micron constantan wire with silicon vacuum grease for ease of mounting and removal. However, we have also used silver paint, especially in cases where the electrical conductivity was simultaneously measured. The other end of the sample is connected with vacuum grease to a 250-micron constantan wire which is attached to thermal ground to complete the thermal circuit. Two 12-micron chromel leads are attached to the 250-micron constantan to form a differential thermocouple which measures the heat current leaving the sample. In most cases a 12-micron differential chromel-constantan thermocouple is placed on the sample to measure the temperature drop. In the case where electrical conductivity is also measured, two thermocouples are used to measure the temperature at two points along the sample. (The two-thermocouple measurement has more noise than the differential measurement.) Finally a chromel-constantan thermocouple is attached to the hot side of the 75-micron constantan wire to monitor the entire temperature drop. Since the 75- and 250-micron constantan wires are firmly attached, while the sample and its thermocouple are attached by vacuum grease, it is easy to remove and replace fragile samples.

With this apparatus we are able to measure the total temperature drop as well as the temperature drop across each of the elements (two constantan wires and the sample). We can then infer the temperature drop across the contacts. Also since the thermal conductivity of constantan is known, we measure the heat current into and out of the sample as well as the temperature drop across the sample. If there is no heat loss to radiation or residual gases, the heat through the constantan wires would be equal and the calculation of the thermal conductivity is trivial. Otherwise all of this information is necessary and the measurements and calculations are tedious.

The measurements are done by applying an offset square-wave voltage (on-off-on-off) to the heater and recording the changes in all of the thermocouples. At several temperatures the relaxation time is measured to be sure that the measurements are made after steady state has been reached. Most of the measurements were taken with a cycle frequency of 0.003 Hz (approximately 5 min period). Initially the signals were taken with two dual pen chart recorders and a single pen recorder to monitor the temperature. Later the data were digitally acquired and each measurement averaged over two on-off cycles. Any linear drifts in the temperature were corrected for in the analysis.

Typical cool down from 300 K to approximately 100 K took 24 h, while cool down from 100 to 6 K took approximately 12 h due to the absence of exchange gas from the vacuum can in which the sample holder was situated. A magnetic field was obtained with an 8 T superconducting magnet. Usually the cooling rate was sufficiently slow to

allow data acquisition during cool down. However, in many cases we also took data during slow warmups and with temperature stabilized with a capacitance temperature controller. The latter was always required for magnetic field runs.

The simplest case, when there is no heat flow out of the sample from radiation or the thermal conductivity of residual gases, the thermal conductivity can be obtained directly from the heat current flowing through the sample, the temperature drop across the heat-flow path and the geometry. For a rectangular prism the relation is

$$\frac{\kappa wt \Delta T}{l} = Q, \quad (1)$$

where κ is the thermal conductivity, w , t , and l are, respectively, the width, thickness, and length of the sample, and Q is the heat current in watts. This equation can easily be used when we experimentally find that the heat flow into the sample (as measured by the 75-micron constantan thermocouple) is equal to the heat flow out of the sample (as measured by the 250-micron thermocouple). Most of our data were taken under these conditions.

The data analysis is considerably more tedious when there is heat loss through the sample. However, using the experimental setup described above, it is possible to unambiguously obtain both the thermal conductivity of the sample and the heat flow out of the sample due to other thermal paths. The heat flow out of a slab $w dx$ of the sample is proportional to the temperature difference $T(x) - T_a$ where T_a is the ambient temperature (of the copper sample holder) and the exposed area $2(t + W) dx$. Thus the heat flowing out of this slab along the x direction is reduced from that entering the slab by

$$\frac{dQ}{dx} = R [T(x) - T_a], \quad (2)$$

where $R_1 = 4\sigma T^3 2(t + W)$ for radiation losses (σ is the Stefan-Boltzmann constant). For residual gases R is proportional to the thermal conductivity of the gas and other geometrical factors. The temperature along the sample is then obtained by integrating the heat flow divided by the thermal conductivity

$$T(x) = T_0 + \int_0^x \frac{Q(x) dx}{\kappa'}, \quad (3)$$

where $K' = Kwt$. These equations neglect the temperature profile in the sample cross section. The solution of these equations, with the appropriate boundary conditions determined by T_0 , T_a , $Q(0)$ (from the 250-micron constantan), $Q(1)$ (from the 75-micron constantan) is

$$\Delta T = \frac{1(0)(e^{a l_1} - e^{a l_2}) - A(e^{a l_2} + e^{-a l_2} - e^{a l_1} - e^{-a l_1})}{\kappa' \alpha},$$

$$A = \frac{Q(1)e^{-a l} - Q(0)}{e^{-2a l} - 1}, \quad (4)$$

$$\alpha \equiv \sqrt{R/\kappa'} = \frac{R(T_0 - T_a)}{[Q(0) - 2A]}.$$

Here l_1 and l_2 are the position of the differential thermocouple which gives ΔT along the sample. We thus have

three coupled transcendental equations for the three unknowns A , R , and κ' and the values can be computed from the experiments. The heat-flow profile along the sample is

$$Q(X) = Ae^{-\alpha x} + [Q(0) - A]e^{\alpha x} \quad (5)$$

and is therefore completely determined. The equations are however very time consuming to numerically compute and it is much more convenient to take data under conditions where the heat loss is minimum [$Q(1) = Q(0)$] in which case the equations simplify to the form given in Eq. (1). Note however, that it is imperative to have the information that $Q(1) = Q(0)$ in order to convincingly make this simplification.

At room temperature the dominant heat loss from the desired path is by black-body radiation. However, there is also considerable loss from residual gas unless great care is taken. Pumping on the vacuum can with a diffusion pump system reading a vacuum of 1×10^{-7} Torr, we observed a significantly decreasing heat loss while pumping for a period of two days. At temperatures below 100 K the measurements were independent of time after pumping for approximately 2 h with the same system. As an example of the heat loss from residual gases, the ratio of heat flowing through the 75-micron constantan to that flowing through the 250-micron constantan was approximately 200 at atmospheric pressure, approximately 20 after pumping for an hour, and approximately 3 after two days. At 100 K this ratio was approximately $1 \pm 10\%$ after 2 h. (The main difference is probably the additional cryopumping of the walls at low temperature as opposed to their outgassing at high temperature.) From our room-temperature measurements we would strongly suggest that any measurement not made with information about both the heat entering and leaving the sample is suspect. This should be especially true for cases where the sample is surrounded by a fluid as in a pressure bomb.

EXPERIMENTAL RESULTS

In Fig. 2 we have shown the thermal conductivity of $(\text{TMTSF})_2\text{ClO}_4$ from 100 to 6 K. Throughout the entire range we have calculated R using Eq. (1). Below 100 K R_{10} (ratio of heat in to heat out) was approximately $1 \pm 10\%$ so that the easier calculation [Eq. (1)] is sufficient. As we see from this figure the thermal conductivity is essentially temperature independent from 100 to 50 K with a value of 20 mW/cm K. The correctly calculated

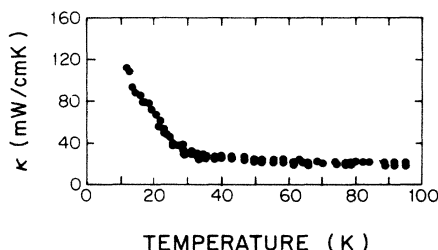


FIG 2. Thermal conductivity of a crystal of $(\text{TMTSF})_2\text{ClO}_4$ calculated from analog traces of thermocouple voltages.

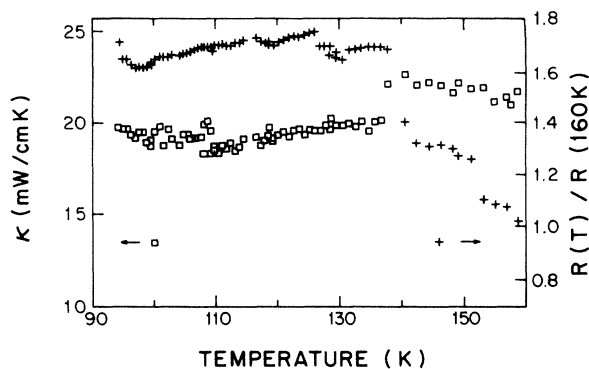


FIG 3. Thermal conductivity and resistance measured on the same sample in the region of several "cracks."

value for κ at 300 K is also 20 mW/cm K. Below 50 K the thermal conductivity increases as temperature is further reduced.

In Fig. 3 we show the thermal conductivity and electrical resistivity measured simultaneously on the same sample. Below 200 K the electrical conductivity shows a series of drops associated with sample "cracks" as reported previously. Note that although these "cracks" affect the electrical transport, the thermal transport is affected less. Thus the cracks must be somewhat transparent to the phonons.

In Fig. 4 we show our data from 50 to 6 K. We see that the thermal conductivity is approximately monotonically increasing down to approximately 12 K and then rapidly decreases. Note that below the anion-ordering temperature of 24 K the thermal conductivity rises faster as temperature is decreased.

In Fig. 5 we show a blowup of the region near the anion-ordering temperature of 24 K plotting both resistance and thermal conductivity.

We have measured the thermal conductivity of a total of six different crystals of $(\text{TMTSF})_2\text{ClO}_4$ in three different apparatuses with temperature slowly drifting down from 40 to 6 K, slowly drifting up in temperature, or temperature stabilized with a capacitance temperature controller. The results are always qualitative as shown in

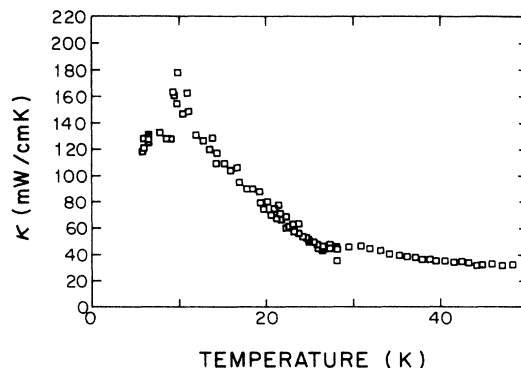


FIG 4. Thermal conductivity, digitally acquired in the region of anion ordering.

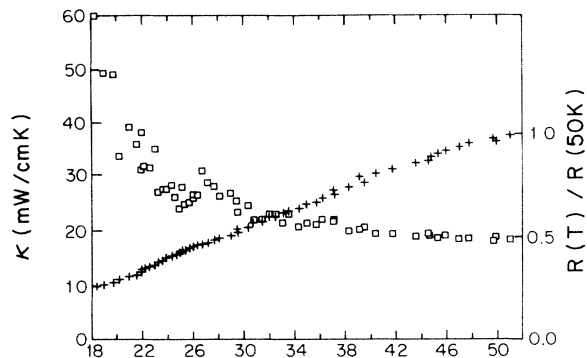


FIG 5. Digitally acquired thermal conductivity and resistance in the region of the anion-ordering transition.

Figs. 2, 4, and 5, but the absolute value of the thermal conductivity varies by about $\pm 30\%$ due to the inaccuracy of the sample geometry measurements and the presence or absence of sample cracks. When normalized at the same temperature the data from different samples agree to approximately $\pm 10\%$.

In two of the samples the thermal conductivity from 40 to 10 K was measured in the presence of an 8 T magnetic field perpendicular to the a - b plane. Aside from an increase in noise to signal, there was no observable change in the thermal conductivity within an error of approximately 25% (from the additional noise and the estimated 10% combined uncertainty in the magnetic field dependence of our thermocouples, thermometers, etc.). Another sample was temperature stabilized at 8 and 15 K and the thermal conductivity measured in zero field and 8 T. Taking data for many sample gradient cycles we were able to distinguish a $20\% \pm 10\%$ decrease in the thermal conductivity at both temperatures.

DISCUSSION

The previous experiments on the thermal conductivity of (TMTSF)₂ClO₄ presented very different results from those which we have found. If in our experiment we neglect all of the data except the total temperature drop and the temperature drop across the 75-micron constantan wire, then we have the same experiment as was used in Ref. 15. We find however that this calculation yields virtually the same results as we have reported in Figs. 2–5 from the total analysis. Thus the technique reported in the previous method should have worked. The only advantage of using our much more complicated setup is that we can see directly when the conditions are correct for doing the simpler experiment. The only way we have been able to obtain data similar to that previously reported is to introduce a small amount (several microns) of residual gases into the vacuum can and to use the simple analysis.

Note that the data in Figs. 2, 4, and 5 are from three different samples measured at three different runs and are all consistent with one another but vastly different from results in Ref. 15.

The drop in the thermal conductivity previously report-

ed, as temperature was lowered below 30 K, was interpreted as being evidence for the existence of superconducting fluctuations to high temperature. There were two problems with this interpretation. The first involved the work on other fluctuating superconducting systems which shows that the fluctuations enhance the thermal conductivity above the 3D T_c . Eventually at lower temperatures it decreases.^{17,18} The second is that Ref. 15 interprets the thermal conductivity as phonon dominated but with a phonon mean free path that is “controlled by scattering of the electrons.” If this were the case, there should be a large phonon-drag term observed in the thermopower (which is not seen), and superconducting fluctuations should reduce the phonon scattering. Thus, in either case, superconducting fluctuations should lead to an initial rise in the thermal conductivity rather than the reported fall below 30 K.

In this sense the data which we report here are more consistent with an interpretation in terms of high-temperature superconductivity fluctuation⁵ than were previous data. However, there is a much more straightforward interpretation of our results. Virtually all crystalline materials which have a thermal conductivity dominated by phonons have a thermal conductivity reminiscent in form to what we observe. As temperature is decreased below Θ_D the umklapp scattering is reduced and the mean free path increases exponentially. At low temperature the mean free path becomes impurity dominated and the thermal conductivity falls with the T^3 law of the specific heat of the phonon gas. The characteristic temperature for this turnover is $\Theta_D/10$. The electronic contribution has a similar behavior with a slightly different crossover.

The change in slope of the temperature dependence at 24 K correlates nicely with the anion-ordering transition. Above this transition there is another mechanism for scattering (off the rotating anion) for both electrons and phonons. As the anions freeze this scattering is reduced and the mean free paths increase, increasing the thermal conductivity. The electrical conductivity has likewise been observed to increase its temperature-dependent slope below this transition.

Finally, we observe that the application of a magnetic field slightly reduces the thermal conductivity, again unlike the previous results which show a strong increase. Note that the present data do not lend themselves to the magnon interpretation in Ref. 16. In some initial experiments we performed where the only thermal links to ground were via constantan and there was nothing in the measurement apparatus that was magnetic-field-dependent, only very small changes in any of the measured temperature drops occurred when 80 kG was applied perpendicular to the sample. In the newer apparatus, modeled after the one used in Refs. 15 and 16, all of the temperature drops changed dramatically upon application of a magnetic field, due to the thermal heat link through the copper wire (which has a large magnetoresistance). However, the thermal conductivity remained only slightly changed. Using a less sophisticated technique with more unknowns could lead to erroneous results when the basic temperature drop changes dramatically. We attribute the small change in the thermal conductivity in a

magnetic field to the electronic contribution. The conductivity of this material decreases rapidly with field in this temperature regime.¹⁰

Although we have no direct way of separating the electronic and lattice contributions there are two indications which suggest that the thermal conductivity is lattice dominated. The first is the small change in thermal conductivity upon application of a magnetic field which more than doubles the electrical resistance. The second is the rapid increase in the thermal conductivity below the anion ordering at 24 K. This increase is considerably more striking than the increase in the electrical conductivity below the same temperature.

CONCLUSION

We have measured the thermal conductivity of $(\text{TMTSF})_2\text{ClO}_4$ from room temperature to 6 K using a technique which can determine the heat flow due to radiation or residual gases. We find, contrary to previous re-

sults, that the thermal conductivity is monotonically increasing in the temperature regime from 100 to 12 K. The thermal conductivity throughout the range appears phonon dominated. Application of a magnetic field slightly reduces the thermal conductivity at low temperature. We find that these results are completely consistent with observations on other crystalline materials and that there is no need to involve exotic mechanisms.

We expect that this technique may be useful for studying other organic conductors with low thermal conductivity. Using ac heating at different frequencies may also be used to measure the thermal diffusivity and specific heat.

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