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# Thermal Conductivity Measurement

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There are three mechanisms whereby energy can be transported from one region of space to another under the influence of a temperature difference. One is by transmission in the form of electromagnetic waves (radiation); the second is the process of convection, in which a bulk or local motion of the material effects the transport; and the final process is that of thermal conduction, when energy is transported through a medium. In most practical situations, energy transport is accomplished by all three processes to some extent, but the relative importance of each contribution varies markedly. For example, within an evacuated region, radiation is the sole mechanism of transport; whereas, in an opaque solid, conduction is the only mechanism possible.

These processes of heat transfer are often very important in a wide variety of scientific and industrial applications. In the cooling of cast or crystalline materials (e.g., metals, semiconductors, or polymers) from a molten state to a solid state, the heat transfer within the material can have a profound effect on the final properties of the solid. Equally, the heat transfer in a foodstuff is a determinant of its cooking, freezing, or processing time, while the size of equipment needed to heat or cool the liquid or gas stream in a chemical plant depends sensitively on the heat transfer within and between one stream and another.

For these reasons, there has been great interest in the understanding and description of all three heat transfer processes. Among the three, that of thermal conduction is the simplest to describe in principle, since the empirical law of Fourier simply states that the heat transported by conduction per unit area in a particular direction is proportional to the gradient of the temperature in that direction. The coefficient of proportionality in this law is known as the thermal conductivity and denoted here by the symbol  $\lambda$ . Many important materials, whether made of pure chemical components or mixtures, are of uniform composition throughout and for them the thermal conductivity is a true physical property of the material, depending often only on the temperature, pressure, and composition of the sample. However, particularly in the solid state, the thermal conductivity can depend on the direction of the heat flow, for example, in the case of a molecular crystal.

It is also conventional to speak of the thermal conductivity of various types of composite materials such as bricks, glass-fiber insulation, carbon-fiber composites, or polymer blends. In this case, the thermal conductivity is taken to be the empirical constant of proportionality in the linear relationship between a measured heat transport per unit area and the temperature difference over a prescribed distance in the material. The thermal conductivity is not then, strictly, a property of the material, since it can often

depend on a large number of parameters, including the history of the material, its method of manufacture, and even the character of its surface. However, this distinction between homogeneous and inhomogeneous materials is often ignored and leads to more than a little confusion, especially where intercomparisons among measurements are concerned.

The fact that in most practical situations all three heat transfer mechanisms are present greatly complicates the process of measurement of the thermal conductivity. Thus, much early work in the field is substantially in error, and it has been really quite difficult to devise methods of measurement that unequivocally determine the thermal conductivity. For that reason, the instruments to be described in the following sections often seem to be rather far removed from the apparent simplicity implied by Fourier's Law.

### 33.1 Fundamental Equations

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The essential constitutive equation for thermal conduction relates the heat flux in a material to the temperature gradient by the equation:

$$Q = -\lambda \nabla T \quad (33.1)$$

It is not possible to measure local heat fluxes and gradients; thus, all experimental techniques must make use of an integrated form of the equation, subject to certain conditions at the boundaries of the sample. All experiments are designed so that the mathematical problem of the ideal model is reduced to an integral of the one-dimensional version of Equation 33.1, which yields, in general:

$$Q_a = G \lambda \Delta T \quad (33.2)$$

in which  $G$  is constant for a given apparatus and depends on the geometric arrangement of the boundaries of the test sample. Typical arrangements of the apparatus, which have been employed in conjunction with Equation 33.2, are two flat, parallel plates on either side of a sample, concentric cylinders with the sample in the annulus and concentric spheres.

Techniques that make use of Equation 33.2 are known as steady-state techniques and they have found wide application, some of which are discussed below. They are operated usually by measuring the temperature difference  $\Delta T$  that is generated by the application of a measured heat input  $Q_a$  at one of the boundaries. The absolute determination of the thermal conductivity,  $\lambda$ , of the sample contained between the boundaries then requires a knowledge of the geometry of the cell contained in the quantity  $G$ . In practice, it is impossible to arrange an exactly one-dimensional heat flow in any finite sample so that great efforts have to be devoted to approaching these circumstances and then there must always be corrections to Equation 33.2 to account for departures from the ideal situation.

If the application of heat to one region of the test sample is made in some kind of time-dependent fashion, then the temporal response of the temperature in any region of the sample can be used to determine the thermal conductivity of the fluid. In these transient techniques, the fundamental differential equation that is important for the conduction process is:

$$\rho C_p \frac{\partial T}{\partial t} = \nabla \cdot (\lambda \nabla T) \quad (33.3)$$

which arises from an elementary energy balance in the absence of any other processes and in which  $\rho$  is the density of the material and  $C_p$  its isobaric heat capacity. In most, but not all, circumstances, it is acceptable to ignore the temperature dependence of the thermal conductivity in this equation and to write:

$$\frac{\partial T}{\partial t} = \frac{\lambda}{\rho C_p} \nabla^2 T = a \nabla^2 T \quad (33.4)$$

in which  $a$  is known as the thermal diffusivity.

Experimental techniques for the measurement of the thermal conductivity based on Equation 33.4 generally take the form of the application of heat at one surface of the sample in a known time-dependent manner, followed by detection of the temperature change in the material at the same or a different location. In most applications, every effort is made to ensure that the heat conduction is unidirectional so that the integration of Equation 33.4 is straightforward. This is never accomplished in practice, so some corrections to the integrated form of Equation 33.4 are necessary. The techniques differ among each other by virtue of the method of generating the heating, of measuring the transient temperature rise, and of the geometric configuration. Interestingly, in one geometric configuration only, is it possible to determine the thermal conductivity essentially independently of a knowledge of  $\rho$  and  $C_p$ , which has evident advantages. More usually, it is the thermal diffusivity,  $a$ , that is the quantity measured directly, so that the evaluation of the thermal conductivity requires further, independent measurements.

In the following sections, brief descriptions of the specific applications of these general principles are given. The examples chosen for study are intended to cover the full spectrum of materials and thermodynamic states and, in each case, attention is concentrated on a method that has proved most accurate and is widely used. The steady-state and transient techniques are considered separately.

## 33.2 Measurement Techniques

### Steady-State Methods

The steady-state methods employed for the measurement of the thermal conductivity of fluids and solids have most often employed the geometry of parallel plates so that it is that configuration described here in two variants. Coaxial cylinder equipment has largely been used within the preserve of the research laboratory, with the apparatus of Tufeu and Le Neindre [1] an excellent example of the genre.

#### Parallel-Plate Instrument

A schematic diagram of a guarded parallel-plate instrument is shown in Figure 33.1. The sample is contained in the gap between two plates (upper and lower) maintained a distance  $d$  apart by spacers. A small amount of heat,  $Q_a$ , is generated electrically in the upper plate and is transported through the sample to the lower plate. Around the upper plate, and very close to it, is placed a guard plate. This plate is, in many instruments, maintained automatically at the same temperature as the upper plate so as to reduce heat losses from the upper surface of the upper plate and to most nearly secure a one-dimensional heat flow at the edges of the sample.

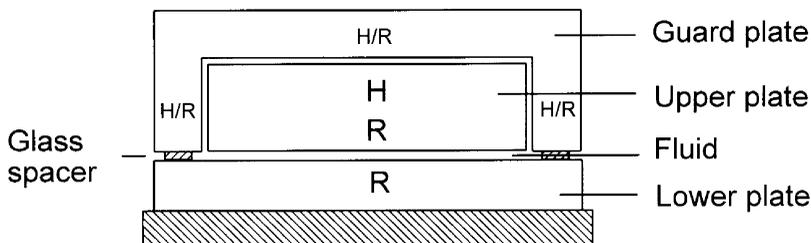


FIGURE 33.1 Schematic diagram of a guarded parallel-plate instrument. (H = heater, R = resistance thermometer).

The temperatures at the surfaces of the upper and lower plates are measured very precisely, as is the electric input of energy, so that the thermal conductivity can, in principle, be evaluated from the equation:

$$Q_a = \frac{A\lambda\Delta T}{d} \quad (33.5)$$

where  $\Delta T$  is the measured temperature difference, and  $A$  is the area of the upper plate.

Whether the sample is a fluid or a solid, the electric energy generated in the upper plate is not all conducted to the lower plate. Thus, it is necessary in all cases to account for spurious heat losses and for all except opaque materials for the radiative transfer between the two surfaces. When the sample is transparent to radiation, this correction is straightforward and can be reduced by means of surface coating the plates to reduce their emissivity; but when the material adsorbs radiation, the problem is much more complicated and has been the subject of some controversy in the past, which has since been resolved (p.147 of [2]). In many cases, the effort of performing absolute measurements cannot be justified so that the ratio  $A/d$  is determined by calibration with a material of known thermal conductivity.

### Measurement on Fluids

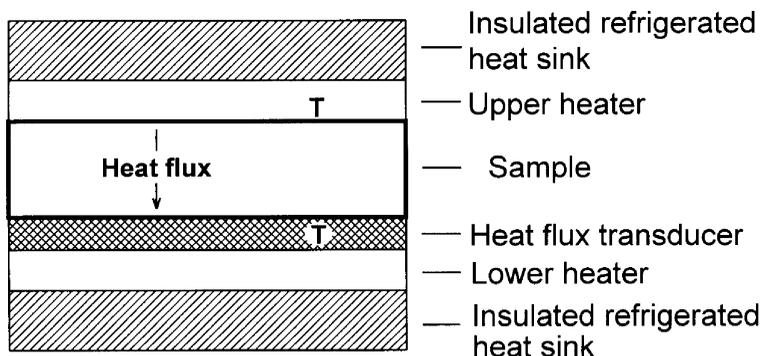
Measurements with parallel-plate instruments on fluids have been performed for a considerable period of time. The technique has particular advantages in some special regions of thermodynamic space but requires great attention to detail if accurate results are to be obtained. In the most accurate instruments for fluids, the gap between upper and lower plates is kept as small as possible (perhaps as small as 0.2 mm). This has the benefit of reducing the effect of heat flows that are not normal to the heat surfaces, but more importantly the small gap contributes to the reduction of the heat transferred by bulk convective motion of the fluid. Indeed, if very small temperature differences are employed by heating the top plate (to have a stable density gradient) *and* considerable care is taken to align the parallel plates normal to the earth's gravitational acceleration, then the effects of convective heat transfer can be rendered negligible (p.154 of [2]). It is a fact of history that the necessary care with this instrument has been taken by only a few workers so that despite the fact that the instrument has been used in the temperature range from 4 K to 800 K, and for pressures up to 250 MPa, only some of the measurements are reliable.

An example of what can be achieved with a parallel-plate instrument is provided by the work of Mostert [3] and Sakonidou [4] at the van der Waals laboratory in Amsterdam. They have used the technique near the critical point of a pure fluid or mixture where the extreme values of the compressibility make the fluid exceedingly prone to convection. In such circumstances, the small vertical extent of the fluid layer required for this technique avoids large density variations in the test layer and, combined with the fact that very small temperature differences (0.3 mK) can be employed, has enabled measurements of the thermal conductivity to be conducted to within 100 mK of the critical temperature at the critical density. Under these circumstances, the thermal conductivity of a pure fluid reveals an enhancement that is, in principle, infinite at the critical point itself.

The unique characteristics of the parallel-plate instrument mean that it is the method of choice for work near the critical region of a material. The arguments above pertaining to the care required for reliable measurements militate against the production of commercial instruments.

### Measurements on Solids

A parallel-plate instrument of exactly the same type as has been described for fluids has been employed for solids. However, the spacing of the two plates is normally significantly greater for solids, owing to the difficulty of preparing very thin samples of solids. One essential difficulty with solid samples in this configuration is the contact between the two heater surfaces and those of the sample. Unlike the case for fluids, the contact cannot be made uniform at the molecular level. There is therefore always the possibility of an unaccounted interfacial heat transfer resistance. It seems likely that these considerations contribute to the wide differences between values reported for the same sample by different authors.



**FIGURE 33.2** Schematic diagram of a heat flow-meter instrument. (T = thermocouple).

A more popular implementation of the parallel-plate configuration for solids is the so-called heat flow-meter instrument. When applied to materials such as building insulation, the dimensions of this type of instrument can be very large.

In such instruments (see [Figure 33.2](#)), the upper heater plate is set at a higher temperature than the lower one. The hot and cold surface temperatures of the sample are measured with the two thermocouples permanently installed on the adjacent surface plates, while a precalibrated heat flow transducer on the lower plate measures the magnitude of the heat flux through the sample. The thermal conductivity is calculated directly from Equation 33.5. In some cases, contact resistances (i.e., insulation) can be characterized and controlled by employing a pressurized gas in the sample chamber. Commercial instruments of this type are included in the listing of [Table 33.1](#).

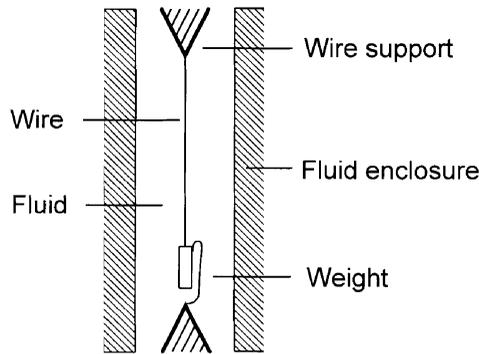
## Transient Methods

There are rather more transient techniques that have achieved popularity than steady-state instruments. This is because transient techniques generally require much less precise alignment and dimensional knowledge and stability. Furthermore, some of the techniques have distinct advantages that arise from the speed of the measurement. Here we have space to describe only one technique in detail which has, in a variety of ways, far greater applicability.

**TABLE 33.1** Companies That Make Thermal Conductivity Instruments

Type of instrument	Temperature range	Supplier	Approximate price (U.S.)
Transient hot disk			
Thermal Analyser TPS	290–1,000 K	K-analys AB	\$20,000
Guarded parallel plate			
Thermatest GHP-300	290–1,000 K	Holometrix Inc.	Variable
TCT 416	290–340 K	NETZSCH	\$30,000
Heat flow meter			
Rapid-k RK-70	290–500 K	Holometrix Inc.	Variable
Unitherm 2021	290–500 K	ANTER	\$18,000
Radial heat-flow			
Orton D.C.A.	290–1400 K	Orton	\$50,000
Laser flash			
Thermaflash 1100	290–1300 K	Holometrix Inc.	Variable

*Note:* Prices are only indicative.



**FIGURE 33.3** Schematic diagram of a transient hot-wire instrument for fluids. (Note that the hot wire is kept under constant tension by a weight.)

### Transient Hot-Wire Technique

In this technique, the thermal conductivity of a material is determined by observing the temporal evolution of the temperature of a very thin metallic wire (see Figure 33.3) after a step change in voltage has been applied to it. The voltage applied results in the creation of a line source of nearly constant heat flux in the fluid. As the wire is surrounded by the sample material, this produces a temperature field in the material that increases with time. The wire itself acts as the temperature sensor and, from its resistance change, its temperature change is evaluated and this is related to the thermal conductivity of the surrounding material.

According to the ideal model of this instrument, an infinitely long, line source of heat possessing zero heat capacity and infinite thermal conductivity is immersed in an infinite isotropic material, with physical properties independent of temperature and in thermodynamic equilibrium with the line source at time  $t = 0$  at a temperature  $T_0$ . The heat transferred from the line source to the sample when a stepwise heat flux,  $q$ , per unit length is applied, is assumed to be entirely conductive. Then the temperature rise of the material at a radial distance,  $r_0$ , which it transpires, is the same as the temperature rise at the surface of a wire of radius  $r_0$ ,  $\Delta T_i(r_0, t)$  is given by:

$$\Delta T_i(r_0, t) = T(r_0, t) - T_0 = \frac{q}{4\pi\lambda} \left[ \ln \left( \frac{4at}{r_0^2 C} \right) + \frac{r_0^2}{4at} + \dots \right] \quad (33.6)$$

In the above equation,  $C$  is a known constant [5]. The equation suggests that, provided the radius of the wire is chosen small enough so that the second term on the right-hand side of Equation 33.6 is negligible, the thermal conductivity of the fluid can be obtained from the slope of the line  $\Delta T_i$  vs.  $\ln t$ . Any practical implementation of this method of measurement inevitably deviates from this ideal model. The success of the technique, however, rests on the fact that by proper design, it is possible to construct an instrument that can operate very closely to the ideal model, while at the same time small departures can be treated by a first-order analysis [5].

The transient hot-wire technique was first developed in the 1930s to measure the effective thermal conductivity of powders. However, its application to other materials was somewhat slower until in the late 1960s the new technology associated with electronics made it possible to measure small, transient resistance changes with high accuracy in a period of less than 1 s. This development, pioneered by Haarman [6], made it possible to complete the transient heating process so quickly that, despite the inevitability of convective motion in the fluid from time zero, the inertia of the fluid ensures that the fluid velocity is sufficiently small that there is no significant contribution to heat transfer. This fact prompted a rapid development of the measurement technique for fluids — first in gases and then in liquids — that was then followed by further developments in solids. The differences in the technique

between solids and fluids are rather small; thus, some aspects of the instrumentation for liquids are briefly discussed and the differences for solids are just outlined.

In the case of fluids, the instrumentation generally involves a wire some  $7\ \mu\text{m}$  to  $25\ \mu\text{m}$  in diameter (in order to reduce the correction owing to its heat capacity) and some 150 mm long. The wire is mounted vertically in a cylindrical cell containing the test sample. Often, a second wire differing only in length is employed to compensate automatically for effects at the ends of the wires via the electrical measurement system, but this can also be accomplished with potential taps [5]. Whenever possible, platinum is used for the wire material because its resistance/temperature characteristics are well known and it can be readily obtained in the form of wires with a diameter as small as  $5\ \mu\text{m}$ . When the material under test is electrically conducting, it is necessary to insulate electrically the wire from the fluid. A variety of techniques have been employed for this purpose that enjoy different degrees of success depending on the range of conditions to be studied. Near ambient temperature over a range of pressures, it has been found adequate to use a tantalum wire as the sensor that is electrolytically anodized to cover the wire with an insulating layer of tantalum pentoxide 100 nm thick [7]. Under more aggressive conditions, it has been necessary to employ ion-plating of the wire with a ceramic to secure the isolation [8]. In either case, the theory has been modified by the addition of a small correction.

In the case of solids, the need for the wire to be straight and vertical is removed by virtue of the rigidity of the material. Thus, Bäckström and colleagues [9] were able to employ a wire embedded as an arc within the compressed solid matrix of the material under study, particularly at very high pressures (up to 4 GPa).

The transient hot-wire technique has a unique advantage among transient methods that the thermal conductivity of the test material can be evaluated directly from the slope of the line relating the temperature rise of the wire to the logarithm of time. The heat capacity and density of the test material are required only to evaluate small corrections. Furthermore, the exact dimensions of the heating element and the cell are also unimportant so that the method avoids the intricate alignment problems of the parallel-plate technique while securing absolute measurements of the property. Despite these advantages and its wide application to measurements in gases, solids, and liquids, there has been no commercial development of an instrument of this kind, presumably because of the delicacy of the long, thin wire in the case of devices for fluids, and the difficulty of sample preparation for solids.

### Hot-Disk Instrument

A transient technique for which there is a commercial version suitable for solid materials is the transient hot-disk instrument shown in Figure 33.4.

The sensor in this case comprises a thin metal strip, often of nickel, wound in the form of a double-spiral in a plane. It is printed on, and embedded within, a thin sandwich formed by two layers of a material that is a poor electric conductor but a good thermal conductor. This disk heater is then, in turn, placed either between two halves of a disk-shaped sample of solid material or affixed to the outside of the sample.

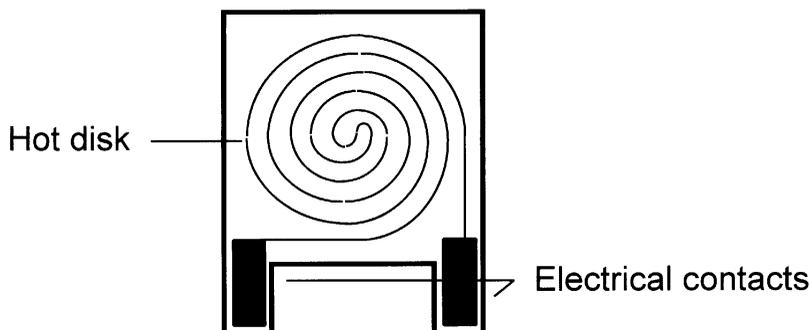
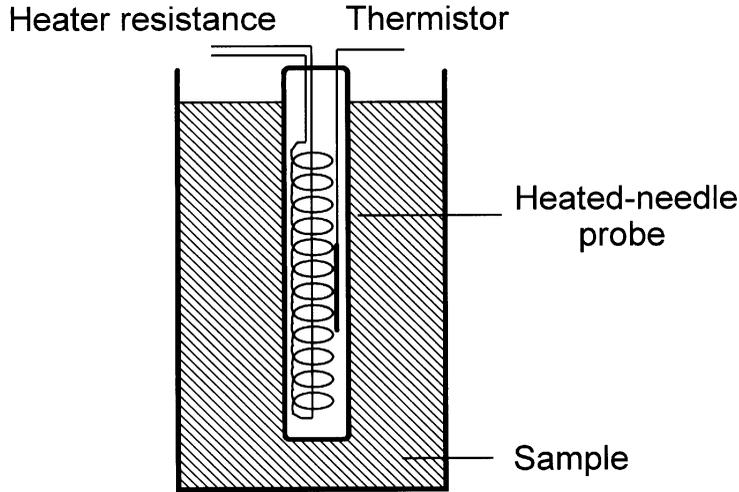


FIGURE 33.4 Schematic diagram of a transient hot-disk instrument.



**FIGURE 33.5** Schematic diagram of a transient heated-needle probe.

However it is configured, the essential measurement performed is the same as that for the hot-wire technique, and the temperature history of the sensor when subject to known electrical dissipation is inferred from its resistance change. In the most recent version of the instrument, developed by Gustafsson [10], and also available commercially, the interpretation of the data is accomplished via a numerical solution of the differential equation rather than by some analytical approximation to it. The technique is used frequently for studies of polymer composites, glasses, superconductors, and insulating materials.

### **Heated-Needle Probe**

A further commercial device exists for the measurement of the thermal conductivity of granular materials such as powders and soils, natural materials such as rock and concrete and, indeed, of food. The probe is shown schematically in [Figure 33.5](#) where it is seen that it consists of a thin, hollow, metallic needle (diameter 3 mm) containing an electric heater and a separate thermistor as a probe of the temperature history of the needle following initiation of a heat pulse [11]. The temperature history of the probe is generally interpreted with the aid of the equation appropriate to a transient hot-wire instrument but in a relative manner whereby its response is calibrated against known standards. This rather simplistic approach to the analysis of a somewhat complex cell inevitably restricts the accuracy that can be achieved, but does provide a measurement capability where no other technique is viable. It is often employed for measurements in inhomogeneous samples such as rocks or soils where it is simply the effective thermal conductivity that is required.

### **Laser-flash Instrument**

A final transient technique is that which has become known as the laser-flash technique developed originally for measurements in solids but occasionally used on liquids, particularly at high temperatures.

[Figure 33.6](#) contains a schematic diagram of the instrument as it is available today in a commercial form. The sample is illuminated on one face with a laser pulse of very short duration and high intensity. The absorption of the laser energy on the front face of the sample causes the generation of heat at that front surface, which is subsequently transmitted throughout the sample to the back face of the sample where the temperature rise is detected with an infrared remote sensor. The interpretation of measurements is based on a one-dimensional solution of Equation 33.4 subject to an initial condition of an instantaneous heat pulse at one location.

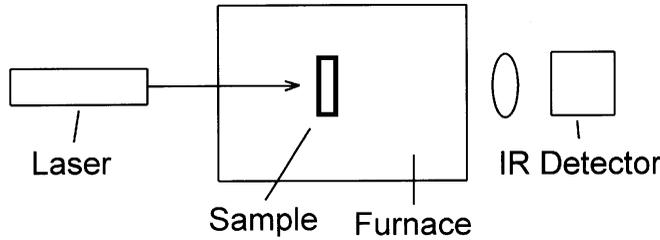


FIGURE 33.6 Schematic diagram of a laser-flash instrument.

The temperature rise at the back face of a sample of thickness  $l$  and radius  $r$ , is therefore given by [12]:

$$\Delta T(l, t) = \frac{Q}{\rho C_p l \pi r^2} \left[ 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(-\frac{n^2 \pi^2 a t}{l^2}\right) \right] \quad (33.7)$$

where  $Q$  is the energy absorbed at the front surface at time zero. The thermal diffusivity of the sample,  $a$ , is then often deduced from the measurement of the time taken for the back face of the sample to reach one half of its maximum value. The technique has the very distinct advantage that it does not require physical contact between the test sample and the heater or detector. For this reason, it is a particularly appropriate technique for use at high temperatures or in aggressive environments.

However, there are a number of precautions that must be taken to ensure accurate results. First, the theory should be modified to account for non-unidirectional heat flow. Secondly, care must be taken to ensure that no radiation incident on the front face penetrates to the back face for transparent samples. Due care must also be taken to match the laser power to the system being studied so that there is neither fusion nor ablation at the front face that can distort the results. Finally, when the fluid state is studied, due care should be taken to eliminate convective heat transport. Seldom are all of these precautions adopted in routine work, so that some results obtained with the technique are of dubious validity. Nevertheless, the method has seen widespread application to a wide range of materials, including composites, polymers, glasses, metals, refracting materials, insulating solids, and coatings.

Finally, the radial heat-flow method should also be mentioned. This is a transient technique in which the sample is heated and cooled continuously [13]. From the recording of the temperature gradient via thermocouples, the thermal diffusivity is obtained and thus the thermal conductivity is calculated. The advantage of the radial heat-flow method is that the measurements are fast and only small temperature gradients are necessary in the sample. A commercially available instrument operating according to this technique is listed in Table 33.1.

### 33.3 Instrumentation

Table 33.1 lists various instruments for the measurement of the thermal conductivity of solids, while the addresses of the suppliers are shown in Table 33.2. As already mentioned, to our knowledge, there is no company that produces instruments specifically for the measurement of the thermal conductivity of fluids.

### 33.4 Appraisal

Naturally, the technique to be employed for the measurement of thermal conductivity depends on the type of sample to be studied and the range of conditions to be employed. For fluid samples under most conditions, a variant of the transient hot-wire method must be the preferred technique. Under favorable

**TABLE 33.2** Addresses of Companies That Make Thermal Conductivity Instruments

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ANTER Corporation 1700 Universal Road Pittsburgh, PA 15235-3998 Tel: (412) 795-6410 Fax: (412) 795-8225	NETZSCH Gerätebau GmbH Wittelsbacherstraße 42 D-95100 Selb/Bayern, Germany Tel: (9287) 88136 Fax: (9287) 88144
HOLOMETRIX Inc. 25 Wiggins Avenue Bedford, MA 0170-2323 Tel: (617) 275-3300 Fax: (617) 275-3705	ORTON The Edward Orton JR. Ceramic Foundation P.O. Box 460 Westerville, OH 43081 Tel: (614) 895-2663 Fax: (614) 895-5610
K-ANALYS AB Seminariegatan 33 H S-752 28 Uppsala, Sweden Tel: (46) 18 50 01 66 Fax: (46) 18 54 36 38	

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conditions, an accuracy of  $\pm 0.3\%$  can be achieved and a level of  $\pm 1\%$  is possible under all but the most aggressive circumstances. Near the critical region of fluids, a parallel-plate instrument is essentially the only viable method. For molten materials at high temperature, while a variant of the hot-wire system has advantages, the laser-flash technique is very attractive but an accuracy of no better than 10% is then to be expected.

For solids, the hot-disk or laser-flash techniques have many features that recommend them when the sample is amenable to appropriate preparation. In those cases, an accuracy of a few percent should be possible but is rarely attained. For samples such as rocks, the heated needle-probe is undoubtedly the only viable technique.

It should be emphasized again here that when a sample is inhomogeneous, the quantity determined is not the thermal conductivity of any element of it, but rather an effective value suitable for engineering purposes. It is not an intrinsic thermophysical property of the material.

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