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Designation: D 5930 – 01

## Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique<sup>1</sup>

This standard is issued under the fixed designation D 5930; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*

1.1 This test method covers the determination of the thermal conductivity of plastics over a temperature range from  $-40$  to  $400^{\circ}\text{C}$ . The thermal conductivity of materials in the range from  $0.08$  to  $2.0$   $\text{W/m}\cdot\text{K}$  can be measured covering thermoplastics, thermosets, and rubbers, filled and reinforced.

1.2 The values stated in SI units are to be regarded as standard.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish proper safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no current ISO document that duplicates this test method.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus<sup>2</sup>

C 518 Test Method for Steady-State Heat Flux and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus<sup>2</sup>

C 1113 Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique)<sup>3</sup>

D 618 Practice for Conditioning Plastics for Testing<sup>4</sup>

D 883 Terminology Relating to Plastics<sup>4</sup>

D 2717 Test Method for Thermal Conductivity of Liquids<sup>5</sup>

E 177 Practice for Use of the Terms Precision and Bias in

#### ASTM Test Methods<sup>6</sup>

E 1225 Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique<sup>6</sup>

### 3. Terminology

3.1 *Definitions*—Terminology used in this standard is in accordance with Terminology D 883.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *temperature transient, n*—the temperature rise associated with the perturbation of a system, initially at a uniform temperature. The system does not attain thermal equilibrium during the transient.

3.2.2 *thermal conductivity, n*—the time rate of steady heat flow/unit area through unit thickness of a homogeneous material in a direction perpendicular to the surface induced by a unit temperature difference.

3.2.2.1 *Discussion*—Where other modes of heat transfer are present in addition to conduction, such as convection and radiation, this property often is referred to as the apparent thermal conductivity,  $\lambda_{app}$ .

3.2.2.2 *Discussion*—Thermal conductivity must be associated with the conditions under which it is measured, such as temperature and pressure, as well as the compositional variation of the material. Thermal conductivity may vary with direction and orientation of the specimen since some materials are not isotropic with respect to thermal conductivity.

3.2.3 *thermal diffusivity*—a heat-transport property given by the thermal conductivity divided by the thermal mass, which is a product of the density and the heat capacity.

#### 3.3 Symbols:

3.3.1  $C$ —Probe constant.

3.3.2  $\lambda$ —Thermal conductivity,  $\text{W/m}\cdot\text{K}$ .

3.3.3  $Q$ —Heat output per unit length,  $\text{W/m}$ .

3.3.4  $T_2$ —The temperature (K) recorded at time  $t_2$ .

3.3.5  $T_1$ —The temperature (K) recorded at time  $t_1$ .

#### 3.4 Subscript:

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 04.06.

<sup>3</sup> Annual Book of ASTM Standards, Vol 15.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 05.02.

\*A Summary of Changes section appears at the end of this standard.

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- 3.4.1 *av*—average.
- 3.4.2 *app*—apparent.
- 3.4.3 *ref*—reference.

#### 4. Summary of Test Method

4.1 *Line-Source Technique*—This is a transient method for determining thermal conductivity (1, 2).<sup>7</sup> A line source of heat is located at the center of the specimen being tested. The apparatus is at a constant initial temperature. During the course of the measurement, a known amount of heat produced by the line-source results in a heat wave propagating radially into the specimen. The rate of heat propagation is related to the thermal diffusivity of the polymer. The temperature rise of the line-source varies linearly with the logarithm of time (3). This relationship can be used directly to calculate the thermal conductivity of the sample. The line-source of heat can be achieved in a number of ways. In this test method, it is in the form of a probe as described in 7.2.

#### 5. Significance and Use

5.1 The relative simplicity of the test method makes it applicable for a wide range of materials (4, 5). The technique is capable of fast measurements, making it possible to take data before the materials suffer thermal degradation. Alternatively, it is possible to study the effect of compositional changes such as chemical reaction or aging (6). Short measurement times permit generation of large amounts of data with little effort. The line-source probe and the accompanying test specimen are small in size, making it possible to subject the sample to a wide range of test conditions. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute.

#### 6. Interferences

6.1 The line-source method produces results of highest precision with materials where intimate contact with the probe can be established, thereby eliminating effects of thermal contact resistance. These materials include viscous fluids and soft solids.

6.1.1 *Thermal-Contact Resistance*—In the solid state, a contact resistance can develop due to the interface between the specimen and the measuring device. Conventional methods attempt to account for this by introducing a conductive paste between the specimen and the sensor. This reduces, but may not eliminate, the effect of contact resistance. In the line-source method, contact resistance manifests itself as a nonlinearity in the initial portion of the transient (see Fig. 1). The technique has a method to account for this phenomenon. By extending the time of the measurement, it is possible to progress beyond the region of thermal-contact resistance, achieving a state where the contact resistance does not contribute to the measured transient (7). This state typically is achieved after about 10 to 20 s in the measurement. The larger the contact resistance, the greater is this time. It is, therefore, important to make a sufficiently long measurement to exclude the portion of

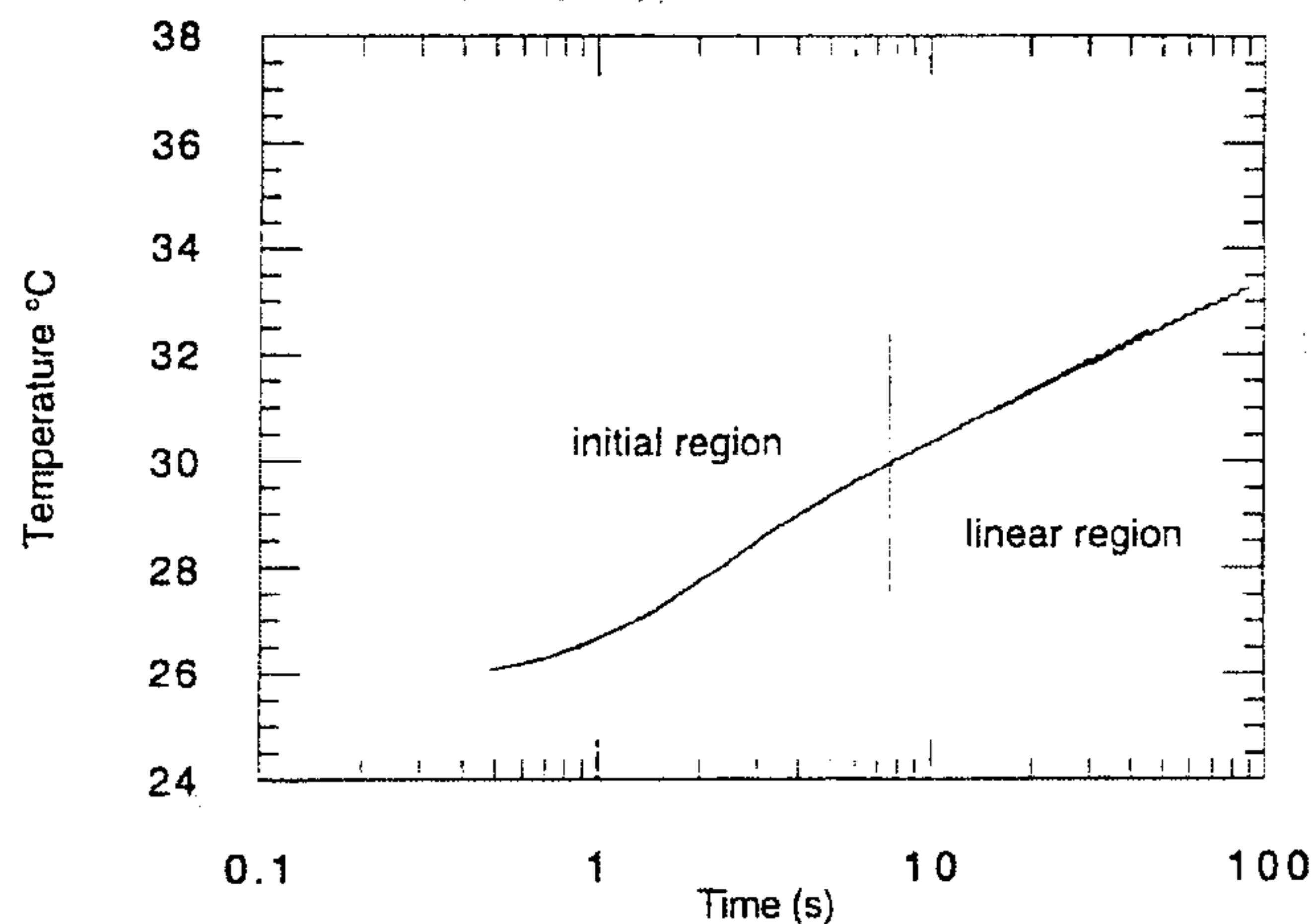


FIG. 1 Line-Source Transient

the transient that shows the effect of the contact resistance. The duration of measurement, however, cannot be too long, or else the heat wave can strike a sample boundary, thereby violating the theoretical conditions of the measurement.

6.1.2 *Shrinkage Upon Solidification*—Plastics tend to shrink significantly upon solidification. This shrinkage is especially so for the semi-crystalline materials, which experience a significant change in specific volume upon crystallization. This crystallization can result in large gaps being developed between the specimen and the sensing device. To account for shrinkage, a simple compression scheme described in 9.5 can permit the line-source probe to move downward to take up the slack. Steps also must be taken to minimize specimen volume so as to reduce the extent of shrinkage.

6.2 Measurements on inviscid fluids are subject to the development of convection currents which can affect the measurement. Because of the transient nature of the measurement, these effects are not as pronounced. They cannot be eliminated, however.

6.3 Although the technique is not limited by temperature, at measurements above 500°C, a significant amount of heat transfer occurs due to radiation so that only a  $\lambda_{app}$  can be measured.

#### 7. Apparatus

7.1 The apparatus consists of a line-source probe imbedded in a specimen contained in a constant-temperature environment. During the measurement, the line-source probe produces a precise amount of heat. The resulting temperature transient is recorded, preferably, on a computer data-acquisition system, as specified in 7.4. This transient is analysed to obtain the thermal conductivity.

7.2 *Line-Source Probe*—The line-source probe contains a heater that runs the length of the probe (3). The length-to-diameter ratio of the probe must be greater than 20. The resistance of the line-source heater must be known to within  $\pm 0.1\%$ . The probe also contains a temperature sensor to measure the temperature transient. A typical sensor for the line-source probe is a high-sensitivity *J*-type thermocouple used because of its large Seebeck coefficient. The housing sheath of the probe must be robust enough to ensure that the

<sup>7</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

probe does not bend or deform under the adverse conditions it is subject to during measurements.

**7.3 Heater Power Source**—The power input to the line-source heater comes from a DC voltage source. The precision of the voltage source must be within  $\pm 0.25\%$  over the entire duration of the test.

**7.4 Recording Device**—The temperature transient from the line-source probe is recorded for the duration of the test. A temperature measurement device with a resolution of  $0.1^\circ\text{C}$  is required. Data are acquired for 30 to 120 s depending on the type of material. Typical temperature rises are between 2 and  $10^\circ\text{C}$  over the duration of the measurement. The frequency of data acquisition must be at least once every second.

**7.5 Specimen Environment**—A constant-temperature environment must be maintained through the duration of the test so as to provide a temperature stability in the specimen of within  $\pm 0.1^\circ\text{C}$ . Failure to attain this criterion can compromise the linearity of the transient, thereby affecting the test result. The environment should be free from excessive vibration.

**7.5.1 Ambient**—For measurements close to ambient, a stirred water bath may be used to maintain the test temperature. Alternatively, the specimen, adequately shielded to protect it from convection, may be placed in air.

**7.5.2 Cryogenic Temperatures**—The specimen, adequately shielded to protect it from convection, may be placed in a controlled cryogenic bath or chamber.

**7.5.3 Elevated Temperatures**—At temperatures above ambient, a special heated cell is required. This consists of a vertical cylindrical heated chamber, fitted with a removable plug at the bottom. The specimen is loaded from the top and can be discharged through the bottom, once the test is complete (see Fig. 2).

## 8. Conditioning

**8.1** Many thermoplastic materials need to be dried because moisture can affect the properties. Moisture causes molten

polymer samples to foam, which will affect the measured thermal conductivity. If conditioning is necessary, see the applicable material specification or Practice D 618.

## 9. Preparation of Test Specimen

**9.1** The test specimen may be prepared from samples, which can be in the form of plastic pellets, liquids, foams, or soft solids. The specimen-preparation method depends on the type of material being tested. If the material is believed to be anisotropic, at least three specimens must be tested. Specimens must be longer than the line-source probe and large enough in radius to have at least 4 mm of material surrounding the probe, so that the expanding heat wave will not strike a boundary during the measurement.

**9.2 Viscous Liquids**—These include pastes and semisolids. Pour or extrude the specimen into a test tube or similar cylindrical container. The container must be filled with sufficient quantity of fluid such that the probe is immersed completely.

**9.3 Soft Solids**—Insert the line-source probe directly into the specimen, taking care to see that it does not bend during insertion. The specimen can be of any size or shape as long as it is larger than the minimum specified in 9.1. In the case where the specimen cannot be penetrated without being destroyed, it is permissible to drill a pilot hole that is smaller than the probe diameter to aid in insertion.

**9.4 Thermoplastics in the Melt**—Preheat the sample cell to the lowest processing temperature of the thermoplastic. Loading specimens at a low temperature is desirable to ensure an air-free specimen. Pour a charge of the specimen, typically in pellet or powder form, into the cell and compress into a homogeneous specimen. Several charges, tamped well, may be needed to fill the sample cell. When the specimen is well molten, insert the probe so as to be near the axial center of the specimen. Sealing systems may be employed to contain the specimen. For thermally unstable materials, follow material manufacturers' recommendations on temperature exposure limits.

**9.5 Solid Thermoplastics**—Load the sample in the same manner as in 9.4. The following precautionary steps are needed to account for shrinkage of the specimen as it solidifies. The probe shall be fitted with a dynamic sealing system permitting it to move with the shrinking specimen. Static loads can then be placed on the probe to help maintain contact as the plastic shrinks. These loads optimally will apply a pressure of 1 to 7 MPa on the specimen.

**9.6 Thermosets and Rubber**—Preheat the sample cell to a loading temperature, above the glass transition, where the specimen is fluid enough to be molded but will not undergo significant reaction (6). If the sample cell is to be reused, wipe the walls of the cell with a thin layer of a release agent such as silicone oil to prevent the cured specimen from bonding to the cell. Charge or pour the uncured specimen in the same manner as in 9.4. For best results, do not coat the probe with release agents since this might affect the test results.

## 10. Calibration

**10.1** The actual probe and sample cell differ in many ways from the theoretical situation, which assumes an infinitely long

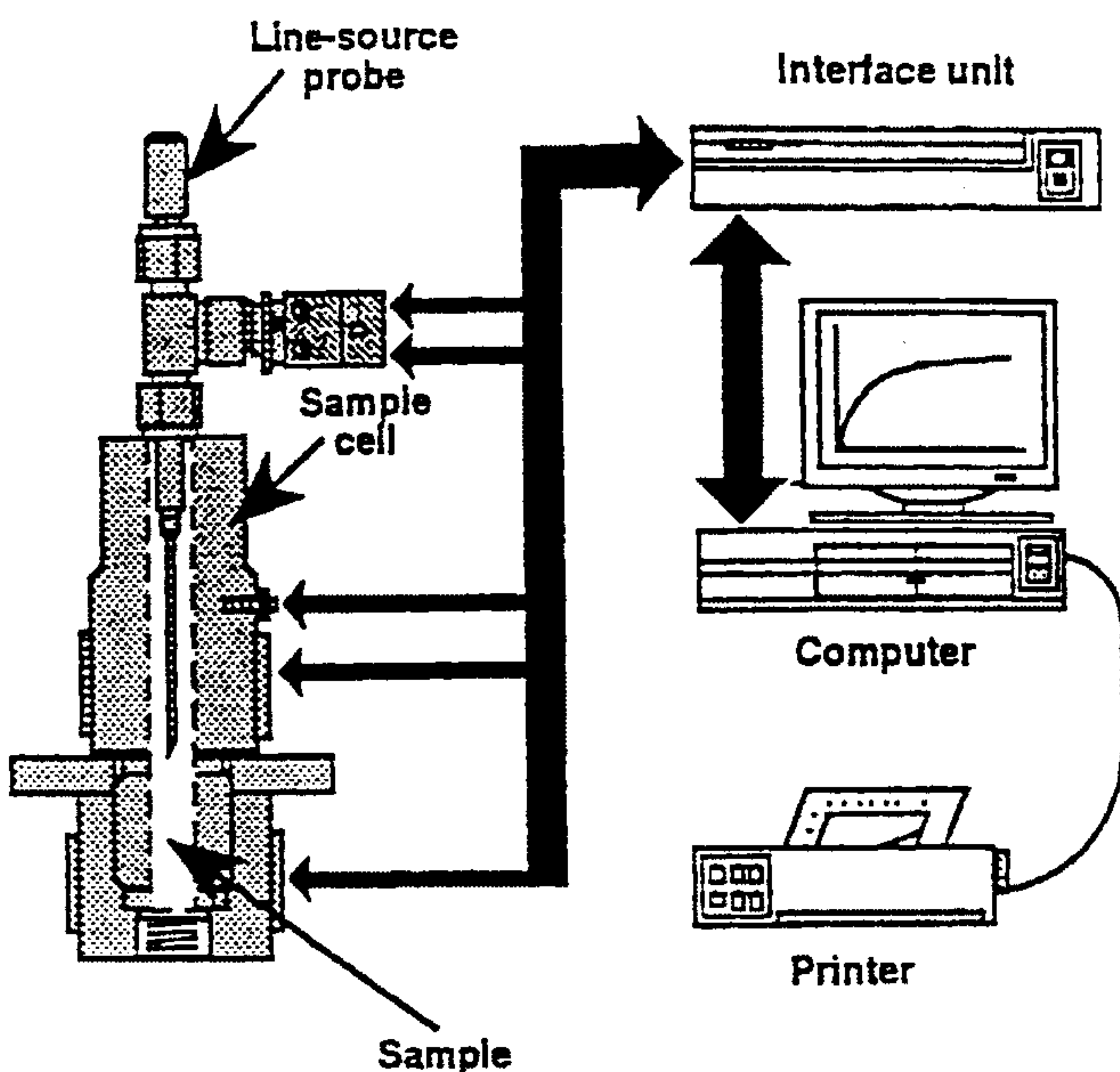


FIG. 2 Adaptation for Measurements at Elevated Temperatures

probe in an infinite specimen. Some of the non-idealities can be minimized by judicious design of the probe and sample cell. Practical limitations in probe construction, however, are such that a calibration is needed to account for such effects as the thermal mass of the probe and the fact that the precise length of the line-source cannot be determined. A probe constant is obtained by calibrating the probe against a material of known thermal conductivity. The constant depends on the probe characteristics and has no significant temperature sensitivity. The ideal value of the probe constant is 1.0. Typical values range from 0.8 to 0.9.

**10.2 Reference Material**—An ideal reference material is a well-characterized, viscous liquid. Such a system will not be subject to thermal contact resistance effects. Because of the high viscosity, convection effects are not present. A polydimethylsiloxane fluid<sup>8</sup> is in common use as a reference material. This material has a thermal conductivity of 0.16 W/m.K at room temperature (8, 9).

**10.3 Probe Calibration**—In the equations in 12.2, the probe constant is set to 1.0. The line-source probe is immersed in the reference material of known thermal conductivity  $\lambda_{ref}$  which is at the reference temperature. At least five measurements must be performed as outlined in Section 11. The resulting values of thermal conductivity are calculated based on 12.2, for each case and the results averaged yielding  $\lambda_{av}$ .

$$C = \lambda_{ref} / \lambda_{av} \quad (1)$$

Calibration should be verified at least once a month.

## 11. Procedure

**11.1** Before beginning experiments, examine the line-source probe. The probe must be straight and its surface free of residues, oil, or water. Nonresidue solvents and copper gauze are recommended for cleaning. The sample cell must be clean and free of residues.

**11.2** Prepare the specimen and insert the probe as applicable for the appropriate sample type, as described in Section 9. Ensure that the probe is secure and the system is not subject to any form of vibration or temperature fluctuation.

**11.3** In general, low power outputs are desirable to prevent excessive perturbation of the system. The amount of power input to the line-source heater is dependent on the characteristics of the material. A high-thermal-diffusivity material dissipates heat at a faster rate so that a larger amount of heat is needed. Conversely, low-thermal-diffusivity materials, such as foams and insulations require a small amount of heat input.

**11.4** Allow the system to attain temperature stability, as defined in the criteria in 7.5. Supply the heat input to the line-source. Simultaneously, record the temperature and time for the duration of the measurement.

**11.5** Multiple measurements may be made if desired at these same conditions, provided that the system is permitted to stabilize prior to each measurement. Where a change of physical state occurs over the range of conditions tested, acquire at least two measurements in each state.

**11.6** Change the sample environment conditions for the next measurement. Continue the process until the desired range of test conditions have been covered.

**11.7** Clean up the system by extracting the probe under the same conditions as those under which it was loaded. The only exception to this rule is in the case of thermosets where the specimen has solidified around the probe. Rubbers can be cut away from the probe using a sharp razor. Rigid thermosets are removed by thermal decomposition using a Bunsen burner. Remove all adhering materials. Do not scratch, mar, or bend the probe during cleaning.

**NOTE 2—Precaution:** This procedure must be performed with adequate fume removal. The probe temperature must be monitored so that it does not exceed permissible limits.

**11.8** Inspect the specimen for voids. If specimen has been subjected to conditions where it might have undergone degradation, note and report such evidence.

## 12. Calculation

**12.1** Plot the temperature rise against log time. Obtain the slope of the linear portion of the curve. Neglect initial nonlinearities, which occur because of the heat wave propagating through the walls of the probe and also due to contact resistance, where the phenomenon exists (7).

**12.2** Perform the calculations using the following equation:

$$\lambda = \frac{C Q^1}{4\pi \text{Slope}} \quad (2)$$

where:

$$\text{Slope} = \frac{T_2 - T_1}{\ln(t_2 / t_1)} K \quad (3)$$

**12.3 Test Conditions**—The temperature at which the thermal conductivity is to be reported is the equilibrium temperature of the specimen prior to the start of the measurement, reported to the nearest whole number. If the time of the measurement is to be recorded, report the time at the start of the measurement. Report any other conditions that are unique to the measurement.

## 13. Report

**13.1** Report the following information:

**13.1.1** Description of the material and test specimen.

**13.1.2** Description of the apparatus.

**13.1.3** Reference material used for calibration including its thermal conductivity value.

**13.1.4** Details of the probe, including probe constant, length, and power output.

**13.1.5** Thermal conductivity at the relevant test conditions, such as temperature or time.

**13.2** Report if the specimen has undergone any physical or chemical change during testing.

## 14. Precision and Bias

**14.1** Table 1 is based on a repeatability study involving a single laboratory. The materials used were unfilled polypropylene and polycarbonate resins. Measurements were performed

<sup>8</sup> Polydimethylsiloxane, trimethylsiloxyterminated, 60 000 CS, from Huls America, Bristol, PA 19007.

**TABLE 1 Repeatability Data for Thermal Conductivity (W/m·K) of Polypropylene and Polycarbonate**

Material	Test Temperature, °C	Average	$S_r^A$	$r^B$
Polypropylene	200	0.164	0.0019	0.0053
Polycarbonate	280	0.263	0.0058	0.0162

<sup>A</sup> $S_r$  = within-laboratory standard deviation of the average.

<sup>B</sup> $r$  = within-laboratory repeatability limit =  $2.8 \times S_r$ .

by a single technician on a single day. Each test result is an individual determination. Five measurements were obtained for each material.

14.2 Attempts to develop a full precision and bias statement for this test method have not been successful. Because this test

method does not contain round-robin based precision data, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.30 (Section D20.30.07), ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.

14.3 There are no recognized standards on which to base an estimate of bias for this test method.

## 15. Keywords

15.1 line-source probe; line-source technique; plastics; rubber; thermal conductivity; thermoplastics; thermosets

## REFERENCES

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## SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

*D 5930-01:*

(1) Added repeatability statement to Section 14, Precision and

Bias.

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