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Use of the Transient Plane Source Technique for Rapid Multiple Thermal Property Measurements[1]

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This paper describes work at NPL to evaluate the capability of the transient plane source (TPS) technique using various sensor sizes and different types of materials that include solids (Perspex, alumina, extruded polystyrene, agar gel, and ice) and liquids (water and silicone oil). The aim of the present work is to investigate use of the TPS technique on materials where probe size, contact, and internal specimen convection are potentially important issues. Following validation of the technique on the NPL solid reference materials, measurements were carried out on ice using TPS and the NPL guarded hot plate (GHP) to illustrate the probe-to-sample thermal contact resistance issue. Measurements on silicone oil were compared to GHP and the NPL transient hot wire (THW) technique where the probe size/short times are crucial. In addition, measurements on water and agar gel were made to illustrate the influence of natural convection. Although the TPS is a multi-property technique, the focus of this work was on thermal conductivity.

KEY WORDS: agar gel; alumina; ice; Perspex; silicone oil; thermal conductivity; transient technique; water.

1. INTRODUCTION

There is a growing need across a range of industries for rapid, compact, and preferably *in situ* devices for measurement of thermophysical properties. More typically, thermal properties are measured using laboratory-based apparatus designed for particular property ranges and materials.

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Among the transient techniques are the hot wire, hot-strip, and transient plane source (TPS) techniques. The main differences among these techniques are the shape of the resistive element and their ability to cover as large a range of transport properties as possible with satisfactory accuracy.

A resistive element is used as the heat source and often also as a temperature sensor. This element can consist of either a wire [1], strip [2], or thin layer of an electrically conducting material [3], and its temperature coefficient is such that the temperature of the element can be deduced precisely from its resistance.

The element should be thin and the resistance as large as possible in order to provide (i) applicability for small samples and (ii) high sensitivity temperature measurement. The thinnest element can be achieved as an evaporated thin film, but the resulting film temperature coefficient of resistance can change with time as the element undergoes temperature excursions and, in addition, the element is not very robust. By providing a practical compromise between high element resistance and element thickness, Gustafsson developed the TPS technique [4] in which the conducting pattern has a total electrical resistance that is higher and, therefore, more

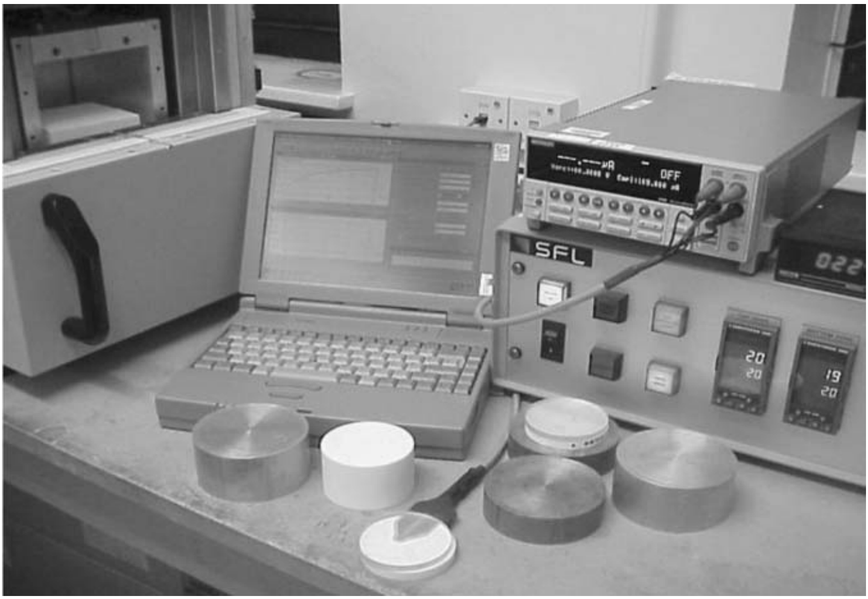

Fig. 1. TPS sensor and samples with computer, PSU, voltmeter, and chamber for the temperature range −60 to 400°C.

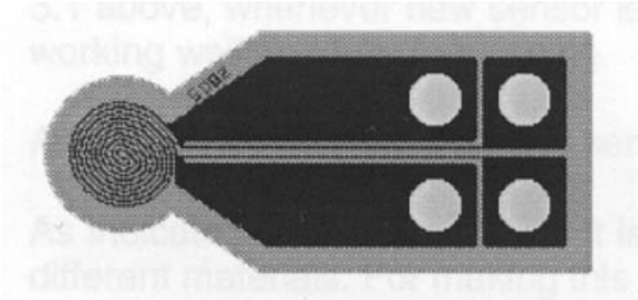

Fig. 2. The TPS sensor: a double spiral of 10 **mm** thick nickel sandwiched between two layers of 25 **mm** thick Kapton insulation.

sensitive than either the hot strip or hot wire, both of which are alternatives to the TPS.

The TPS is a commercial compact instrument (Fig. 1) that employs a transient plane heater/sensor combination to measure the thermal properties of materials. It has been designed to measure thermal conductivity, thermal diffusivity, and specific heat per unit volume and can be applied to a wide range of materials, having thermal conductivities from 0.02 to 200 $W \cdot m^{-1} \cdot K^{-1}$ [5].

The commonly used TPS sensor is a 10 μm thick nickel double-spiral (Fig. 2) and can be used from 10 to 1000 K. The sensor has a 25 μm thick layer of Kapton insulation to keep it electrically isolated; this also helps to maintain its planarity and provide mechanical strength. The probe can be used over the temperature range 10 to 500 K with Kapton insulation while, for temperatures up to 1000 K, a 0.1 mm thick layer of mica insulation is used.

2. THEORY

The TPS sensor is sandwiched between two distinct specimen halves for solids, or fully immersed in a single specimen for powders, pastes, and liquids. A constant current is applied, sufficient to increase the sensor temperature by 1 to 2 K. As the sensor temperature changes, so does its resistance, and by recording the resistance of the probe for a pre-set time, one can establish the sensor temperature variation versus time.

The time-dependent resistance $R(t)$ of the TPS sensor during the transient recording is expressed as follows:

$$R(t) = R_0[1 + \alpha\, \overline{\Delta T(\tau)}]$$

where $R_0 = R(t=0)$, $\tau = (t/\theta)^{1/2}$, and $\theta = a^2/K$. The constant, a, is the radius of the sensor disc, K is the thermal diffusivity, $\overline{\Delta T(\tau)}$ is the mean value of the temperature increase of the TPS sensor, and α is the temperature coefficient of the resistance [4]. It is important to be aware of the influence of the specimen boundary in using the TPS technique, since the Gustafsson model assumes the sample to be infinitely large with, therefore, no boundaries.

The probing depth, ΔP, is the thickness of the specimen assumed to affect the actual measurement. This helps the experimenter determine the minimum size and the transient recording time required to approximate the infinite specimen required by the theory. This is defined by the following expression:

$$\Delta P = \beta\,(K t_{\max})^{1/2},$$

where β is a constant of the order of unity [4] and $t_{\max}$ is the total time of the transient recording. Probing depth and specimen size are intimately connected. The shortest distance from any point on the TPS sensor to the nearest point on any of the outer surfaces of the sample must always exceed the probing depth value to satisfy the "infinite sample" condition.

3. MEASUREMENTS AND RESULTS

The following materials were chosen for measurement:

Solids. NPL thermal conductivity reference specimens Perspex (polymethylmethacrylate) and alumina (99.5% pure, Deranox, Morgan Matroc), extruded cellular polystyrene (density $= 32\ \mathrm{kg \cdot m^{-3}}$).

Agar gel obtained from agar-agar granular powder (code A/1080/53, Fisher Scientific) and ice.

Liquids. Water and silicone oil.

3.1. Validation of the TPS Values for Thermal Conductivity

The extruded polystyrene, Perspex, and alumina were used to evaluate the TPS technique over the approximate thermal conductivity range 0.03 to 29 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$. Perspex and alumina were measured over the temperature range 20 to 70 °C.

3.1.1. Perspex

TPS measurements were performed with the sensor inserted between two 76 mm diameter and 10 mm thick pieces (Table I, Fig. 3). The thermal

Table I. Perspex, Alumina, and Extruded Polystyrene Results using TPS ($r = 6.394$ mm), GHP and AHF Techniques

Material	Temperature (°C)	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$) and std. dev. (%) TPS[a]		
				GHP
Perspex	20	0.194	1	0.191
	50	0.194	3	0.194
	70	0.200	3	0.196
				AHF
Alumina	20	28.6	3	
	40	25.3	5	25.4
	50	25.2	5	24.6
	60	24.6	3	23.8
	70	24.0	4	23.1
				GHP
Extruded polystyrene	24	0.03	1	0.03

[a] Averages of at least five measurements.

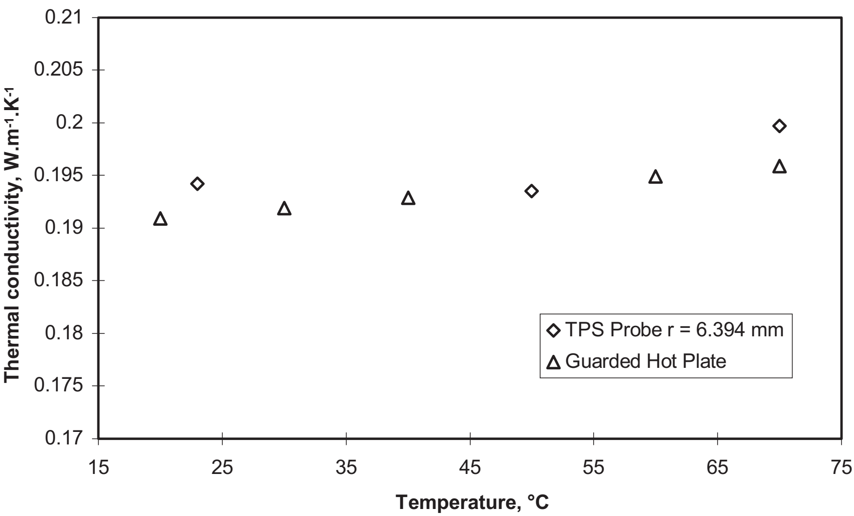

Fig. 3. Perspex thermal conductivity results, 20 to 70°C, using TPS and GHP.

conductivity results agreed to within 2% with the results obtained on the same samples using the NPL 76 mm guarded hot plate (GHP) as shown in Table I.

In the GHP two specimens are mounted on either side of a heater plate and sandwiched under pressure between a pair of cooled plates whose temperatures are steady to within $\pm 0.01°C$. Heat flow at the edges of the specimen is reduced by surrounding the specimens with a guard heater and blanket insulation. The temperature of the annular guard around the central heater plate is matched to the central part to within $\pm 0.01°C$, thus further reducing lateral heat flow to or from the specimens. Depending on the sample measured, periods of up to ten hours are allowed to establish thermal equilibrium once the required temperature drop has been established across the specimen. The thermal conductivity, λ, is derived from the equation: $Q = 2\lambda A(dT/dx)$ where Q is the power supplied to the central heater of effective area A, and dT/dx is the mean temperature gradient through the two specimens.

3.1.2. Alumina

TPS thermal conductivity measurements were obtained on two halves of the alumina samples (Table I and Fig. 4) and they agree to within 4%

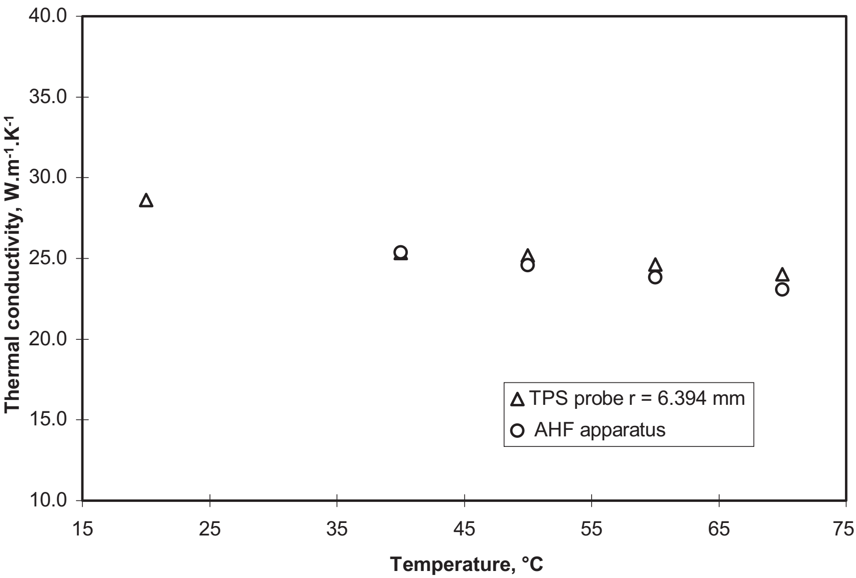

Fig. 4. Thermal conductivity results on alumina, 20 to 70°C, using TPS and NPL AHF apparatus.

with values obtained with the NPL axial heat flow (AHF) [6] apparatus. In the AHF steady-state apparatus, linear heat flow is induced in a bar-shaped specimen firmly clamped between a guarded heater unit and a water-cooled reference specimen using a screw and spring arrangement. Heat energy is supplied at a known rate at one end of the specimen by the heater unit and constrained to flow axially along the specimen with minimum loss or gain. This is achieved by surrounding the specimen and reference specimens with a system of insulated heat shields whose temperatures are maintained steady to within $\pm 0.01°C$. Heat flux from the specimen is monitored using the reference specimens, which serve as a heat flow meter and heat sink for heat conducted through the specimen.

3.1.3. Extruded Polystyrene

The TPS thermal conductivity value at 24°C obtained for the 76 mm diameter and 10 mm thick extruded polystyrene samples was $0.03 \pm 0.0003\ W \cdot m^{-1} \cdot K^{-1}$ (Table I), which agrees to within 1% of the NPL value obtained through a recent intercomparison of guarded hot plate apparatus [7].

3.2. Probe-to-Sample Contact: Measurements on Ice

Since contact resistance is a very important parameter in the use of contact thermal measurements devices, the influence of an imperfect contact resistance was studied through measurements on ice using TPS and the NPL GHP.

It has been pointed out that there are no significant differences between published values of the thermal conductivity of ice—whether laboratory-grown single crystals, glacial single crystals, or commercial polycrystalline ice [8]. The following equation has been proposed for the thermal conductivity of ice, λ, at temperatures from 100 to 273 K:

$$\lambda = 1.16(1.91 - 8.66 \times 10^{-3} T + 2.97 \times 10^{-5} T^2)$$

where λ is in $W \cdot m^{-1} \cdot K^{-1}$ and T in °C. This yields a thermal conductivity of $2.374\ W \cdot m^{-1} \cdot K^{-1}$ at $-15°C$. The ASHRAE Handbook of Fundamentals [9] gives the thermal conductivity of ice at two temperatures, namely $2.24\ W \cdot m^{-1} \cdot K^{-1}$ at 0°C and $2.44\ W \cdot m^{-1} \cdot K^{-1}$ at -20 °C.

Assuming a linear variation of thermal conductivity with temperature, a value of $2.39\ W \cdot m^{-1} \cdot K^{-1}$ at $-15°C$ is obtained. The two approaches thus agree to within less than 1%. To illustrate the importance of specimen-to-sensor contact, measurements were performed using different assembly techniques:

Table II. TPS ($r = 6.394$ mm) Results on Ice

Specimen	Temperature (°C)	Power (W)	Thermal conductivity[a] ($W \cdot m^{-1} \cdot K^{-1}$) and std. dev. (%)	
ice (sensor frozen in pure water)	−15	0.12	2.33	4
ice (using Vaseline as a heat sink compound)	−15	0.12	1.789	5

[a] Averages of at least five measurements.

• Two pieces of ice, 76 mm diameter and 10 mm thick, with Vaseline as a heat sink compound while applying a moderate pressure to maintain a secure contact. The TPS-measured thermal conductivity was $1.79 \pm 0.09\ W \cdot m^{-1} \cdot K^{-1}$ at −15 °C (Table II) which is significantly lower than the literature value.

• For optimal contact the TPS sensor was frozen into distilled water and the measured thermal conductivity was $2.33 \pm 0.09\ W \cdot m^{-1} \cdot K^{-1}$ (Table II), a result which agrees to within 3 % of the literature value.

To compare with the TPS technique, the thermal conductivity was measured in the GHP and a value of $2.00\ W \cdot m^{-1} \cdot K^{-1}$ at −12.8 °C was obtained. This is once again lower than the expected value. Hence, despite our best attempts, it was apparently not possible to achieve an optimal contact with the specimen in the GHP apparatus.

3.3. Liquids

The importance of natural convection when measuring liquids was investigated by varying the sensor size and by comparing measurements on a liquid and a solid having approximately the same thermal properties. To measure a liquid the sensor was fully immersed and kept planar throughout the measurement. To maintain sensor planarity, the sensor was suspended under tension from three fine wires (Fig. 5). It is important to minimize the effects of natural convection since it is not accommodated in the mathematical model and to achieve this, one has to choose short times for transient heat flow from the probe, thus aiming to avoid the onset of significant convection. Also, since the characteristic measurement time θ depends on the radius, a, of the sensor ($\theta = a^2/K$), the sensor radius should be as small as possible.

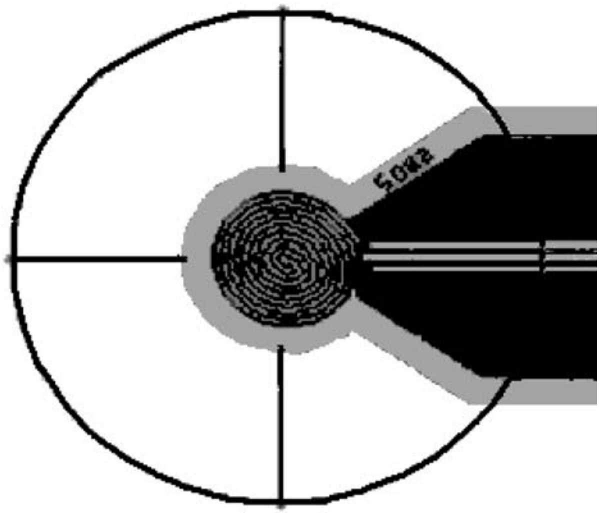

Fig. 5. Three fine wires attached to the insulation to maintain the sensor plane.

3.3.1. Silicone Oil

TPS measurements of the thermal conductivity of silicone oil at 25°C, using sensors with radii $r1 = 6.394$ mm and $r2 = 3.3$ mm, depended significantly on the size of the sensor, as shown in Fig. 6 and Table III. The

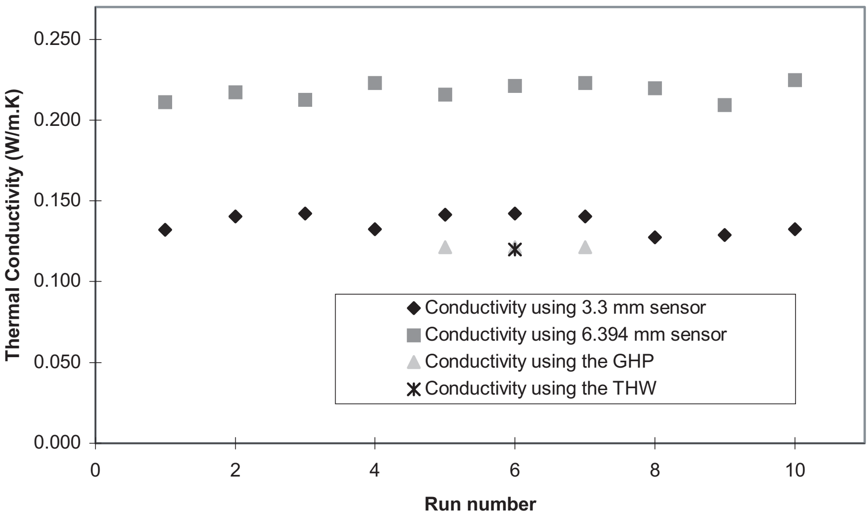

Fig. 6. Comparison of silicone oil results at 25°C using TPS with 2 different sensor sizes, the GHP, and the THW.

Table III. Experimental Results on Silicone Oil Measured at 25°C Using TPS with Two Different Sensor Sizes; THW; and GHP

Technique	Sensor radius (mm)	Power (W)	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$) and std. dev. (%)	
TPS[a]	6.394	0.08	0.22	2
TPS[a]	3.3	0.04	0.14	4
GHP			0.12	
THW			0.12	

[a] Averages of at least five measurements.

results were compared with the NPL transient hot wire (THW) apparatus [10] and the NPL one-sided guarded hot plate in which a cell containing a 4 mm thick sample of liquid is placed below a guarded heater plate so that the liquid is heated from the top to inhibit convection. Extruded polystyrene insulation is placed on top of the heater and the whole assembly sandwiched under pressure between a pair of cold plates, controlled at a constant temperature.

The thermal conductivity measured by the TPS using a sensor radius of 6.394 mm was 0.22 $W \cdot m^{-1} \cdot K^{-1}$ (Table III). This is significantly higher than the value obtained by the THW of 0.12 $W \cdot m^{-1} \cdot K^{-1}$ (Table III). The latter compares very well with the steady-state GHP results, as shown in Fig. 6, whereas the thermal conductivity measured by the TPS using a sensor radius of 3.3 mm at 25 °C is within 16% of the results obtained by the THW and the GHP. Even with the smaller sensor, the TPS results differ significantly from those of the THW and GHP, almost certainly due to convection.

3.3.2. *Water and Agar Gel*

The influence of natural convection was studied using water and a weak dilution of agar gel that has a thermal conductivity almost equal to that of water. The gel was prepared by diluting 0.4% by mass of agar-agar granular powder in pure water and the solution was heated for 12 to 15 minutes. The solution was then poured into a container where the hot disk sensor was kept planar by the arrangement shown in Fig. 5 and left to cool for two hours. With this approach the thermal contact resistance between probe and gel is reduced.

As shown in Fig. 7 and Table IV, the value for the agar gel measured by the TPS ($r = 6.394$ mm and $r = 3.3$ mm) agreed to within 2% with the

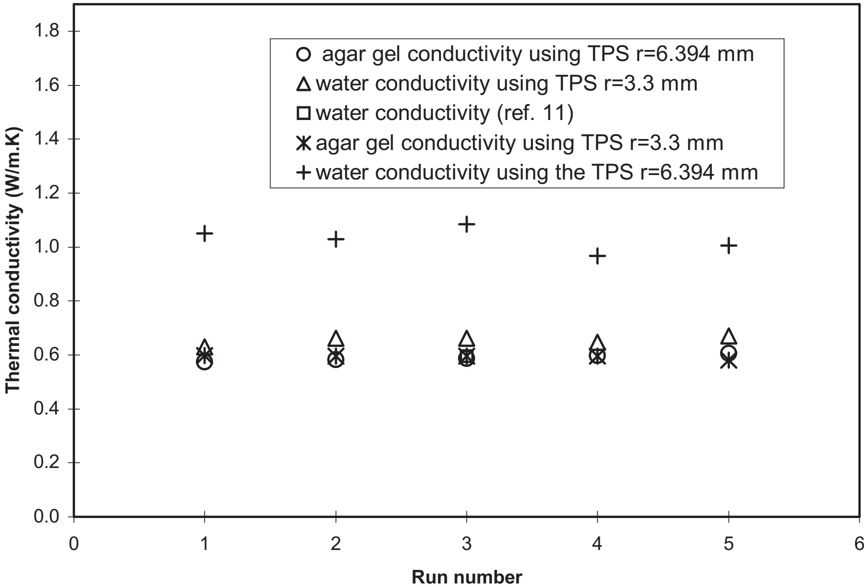

Fig. 7. Results of thermal conductivity on agar gel and pure water at ~25°C using the TPS and compared to literature.

recommended value of ~0.60 $W \cdot m^{-1} \cdot K^{-1}$ for water [11]. However, the value for water measured by the TPS ($r = 6.394$ mm) was significantly higher. Furthermore, the measured value using TPS ($r = 3.3$ mm at 25 °C) was 0.64 $W \cdot m^{-1} \cdot K^{-1}$, which is 13% higher. This was similar to the results obtained on silicone oil using the same sensor radius $r = 3.3$ mm (16%).

Table IV. Thermal Properties of Agar Gels and Pure Water using TPS

Specimen	Temperature (°C)	Sensor radius (mm)	Power (W)	Thermal conductivity[a] ($W \cdot m^{-1} \cdot K^{-1}$)	and std. dev. (%)
Agar gel	23	6.394	0.3	0.589	2
Agar gel	25	3.3	0.05	0.593	1
water	25	6.394	0.1	1.03	4
water	25	3.3	0.05	0.643	3

[a] Averages of at least five measurements.

4. CONCLUSION

The TPS technique has been validated for solid materials in the thermal conductivity range 0.03 to 28 $W \cdot m^{-1} \cdot K^{-1}$ using extruded polystyrene and NPL reference materials Perspex and alumina. The values were reproducible to within 5% and compared well with the NPL's guarded hot-plate and axial heat-flow techniques to within 4%.

Measurements carried out on ice indicated that the TPS technique can be used advantageously to address thermal contact imperfections in difficult materials.

Measurements on liquids showed the influence of the sensor size, but by choosing a small sensor and therefore short times, the onset of significant natural convection can be reduced. However, a discrepancy of $\sim 15\%$ was obtained for both silicone oil and water when compared to other techniques, so for liquids it is clear that further development of the probe or measurement technique is required to obtain better accuracy. Measurements on water and agar gel confirmed the significance of convection in fluids and, although it was reduced by choosing a small sensor and therefore short measurement time, it was not eliminated.

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