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Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 5: Results of interlaboratory testing of poly(methyl methacrylate) samples

Plastiques — Détermination de la conductivité thermique et de la diffusivité thermique —
Partie 5: Résultats d'essais interlaboratoires du poly(méthacrylate de méthyle)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an international Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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ISO/TR 22007-5 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 22007 consists of the following parts under the general title *Plastics — Determination of thermal conductivity and thermal diffusivity*:

— *Part 1: General principles*

— *Part 2: Transient plane heat source (hot disc) method*

— *Part 3: Temperature wave analysis method*

— *Part 4: Laser flash method*

— *Part 5: Results of interlaboratory testing of poly(methyl methacrylate) samples* [Technical Report]

Introduction

The purpose of this document is to record the results of the interlaboratory comparison of measurements of the thermal conductivity and thermal diffusivity of poly(methyl methacrylate) PMMA specimens, as a source of information in support of the development of the series of standards on thermal conductivity and diffusivity of plastics, ISO 22007 [1 - 4].

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 5: Results of interlaboratory testing of poly(methyl methacrylate) samples

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This Technical Report presents the results of interlaboratory testing for the determination of thermal conductivity and thermal diffusivity of two poly(methyl methacrylate) (PMMA) materials by means of the transient and the modulated methods presented in ISO 22007 parts 2 to 4 [1 - 4] and additional transient and steady state methods.

The instructions for the intercomparison are presented in Annex A with key items reproduced in the main part of this Technical Report.

The detailed results of individual laboratories are presented in Annexes B to F.

2 Symbols and definitions

Symbol	Meaning	Unit
α	Thermal diffusivity	m^2/s
d	Thickness of specimen	m
λ	Thermal conductivity	W/(m·K)

For definitions of the terms used, the reader is referred to ISO 472 [5] and ISO 22007-1 [1].

3 Specimen preparation and characterization

3.1 Specimens

Two types of PMMA material were used in the intercomparison:

- Sumipex 000 (cast grade), Lot. 6621114, supplied by Sumitomo Chemical Co. Ltd, Japan [6]. Referred to as "Sumipex cast PMMA" herein. Sheet thickness ≈ 2 mm.
- AAJHF (extruded grade), supplied via NPL, UK. Referred to as "extrusion grade PMMA" herein. Sheet thickness ≈ 3 mm.

The Sumipex cast PMMA was supplied in sheet form only whereas the extrusion grade PMMA was supplied in both sheet and pellet forms.

3.2 Specimen preparation

Depending on the test method, test specimens needed to be prepared from the sheet samples. For the temperature wave analysis the specimens were reduced in thickness. For laser flash testing they were reduced in thickness by one laboratory, but not by the second laboratory. For transient line source testing the specimens were prepared by cutting small pieces from the sheet for insertion into the barrel of the instrument. For Hot Disk testing, most of the data reported are for measurements on single sheets, although two sheets were stacked in some cases to form the test specimen (see Table 1).

4 Measurement apparatus

The experimental apparatus is described in ISO 22007 Parts 1 - 4 and in further detail in references 7 – 17.

Table 1 - The measured thermal properties and various specimen sizes for the methods used in this study.

Method / Lab No.	Measured parameter (thermal conductivity and/or thermal diffusivity)	Nominal specimen thickness mm	Specimen size mm (ϕ: diameter)	Additional pre-treatments
Hot Disk / 1	λ, α, (ρC_p)[1]	2, 3 (4, 6: stacked)	ϕ 5, ϕ 10	
Laser flash / 2	α	2	ϕ 10	silver paint (30 µm)
Laser flash / 3	α	1,14 – cast, 1,49 – extruded	ϕ 12,7	sputtered graphite
Transient line-source probe / 3	λ	moulded in-situ	50	moulded in-situ
Heat flow meter / 3	λ	2, 3	ϕ 50	
Heat flow meter / 4	λ	2, 3	ϕ 80	
Temperature wave analysis / 5	α	0,01	3 x 5	

[1] The factor ρC_p, the specific heat per unit volume J/(m^3.K), is determined from the ratio of the measured thermal conductivity λ and thermal diffusivity α values where ρ is the density (kg/m^3) and C_p is the specific heat capacity per unit mass (J/kg.K).

5 Measurement procedure

The procedures used were as specified in the relevant parts of ISO 22007 [2 - 4] for the methods covered by that standard. The other methods are specified by ASTM D5930 [7] for the line source probe technique, by ASTM E1530 [8] for the guarded heat flow meter method, and as described by [9] for the second heat flux meter method. Experimental details and variations from these references are reported in the intercomparison instructions, Annex A, and in the individual laboratory test reports, Annexes B to F.

6 Calculations

All laboratories carried out the necessary analyses of their raw data to determine thermal conductivity, thermal diffusivity and heat capacity values.

7 Results and conclusions

The test reports of the individual laboratories are presented in Annexes B to F along with tabulated data as provided or abbreviated as appropriate.

8 Results

The results of the measurements are presented in Figures 1 - 4. In addition, in each of these figures, values of thermal diffusivity have been calculated from thermal conductivity, or vice-versa, to demonstrate the level of agreement between the two types of measurement.

The individual results were typically within a range of approximately ± 10 % of the mean value at any given temperature for both thermal conductivity and thermal diffusivity [18].

The reasons for the discrepancy in results are not entirely clear from the intercomparison and require further examination to reduce further the variation in results.

Three particular issues highlighted by the intercomparison that should be covered by good measurement practice are:

- Need to ensure that the specimens are of the appropriate thickness for the test method, satisfying any criteria on thickness that the method imposes. This may necessitate machining of the specimen to an appropriate thickness.
- Effect of anisotropy of the sample. When using the Hot Disk method, testing can yield either anisotropic properties or bulk properties depending on the specific method used. As properties of polymers can be anisotropic, normally due to processing induced effects, it may be necessary to take this into account in testing, depending on the application for the data.
- When calculating thermal diffusivity from thermal conductivity, and vice-versa, it is important to assess the uncertainties in the specific heat capacity values used as these can contribute significantly to the overall uncertainty in calculated values. In the testing carried out here the specific heat capacity values varied by up to approximately ± 10 % from the mean, and density values by ± 1 % from the mean. This would contribute an uncertainty of approximately 10 % to the calculation of thermal diffusivity (see Table 2).

9 Uncertainty and repeatability

Estimates of the uncertainties or repeatabilities of the experimentally measured and calculated values are presented in Table 2. The uncertainty of measurement (coverage factor k = 2) was calculated according to the *Guide to the expression of uncertainty in measurement* [19]. The expanded uncertainty was calculated when thermal diffusivity was calculated from thermal conductivity, or vice-versa, by the use of the equation $\lambda = \alpha C_p \rho$ according to the *Guide to the expression of uncertainty in measurement*. In Table 2 the uncertainties are shown with the k-numbers in parenthesis; values without k-numbers are the repeatabilities.

Table 2 - Estimates of the uncertainties or repeatability for the experimental and calculated values.

Sumipex cast PMMA				
	ρ	C_p	α	λ
Lab. 1	-	0,25 % - 2,89 % * (ISO 22007-2)	0,32 % - 3,16 % (ISO 22007-2)	0,12 % - 0,52 % (ISO 22007-2)
Lab. 2	1 % (ISO 1183-1)	4 % (ISO 11357-4)	8 % (k = 2) (ISO 22007-4)	9 % (k = 2) (calc, ISO 22007-4)
Lab. 3	per standard ASTM D792	- (ASTM E1269-05)	0,49 % - 2,9 % (ASTM E1461-01)	3 % (ASTM E1530)
Lab. 4	0,08 %	1,8 % - 4,8 %	-	3 %
Lab. 5	-	-	2,6 % (k = 2) (ISO22007-3)	8,4 % ** (k = 2) (calc, ISO22007-3)
Extrusion grade PMMA				
	ρ	C_p	α	λ
Lab. 1	-	0,12 % - 1,95 % * (ISO 22007-2)	0,16 % - 1,6 % (ISO 22007-2)	0,07 % - 0,35 % (ISO 22007-2)
Lab. 3	per standard ASTM D792	- (ASTM E1269-05)	0,87 % - 5,6 % (ASTM E1461-01)	per standard ASTM D5930
Lab. 4	0,22 %	3,2 % - 4,3 %	-	3 %
Lab. 5	-	-	5,0 % (k = 2) (ISO 22007-3)	9,5 % *** (k = 2) (calc, ISO 22007-3)

* apparent value as ρC_p

** calculated with Lab. 2 C_p and density data

*** calculated with Lab. 2 and Lab. 4 C_p and density data

10 Acknowledgment

We express our special thanks to Sumitomo Chemical Co. Ltd. for supplying us the cast PMMA.

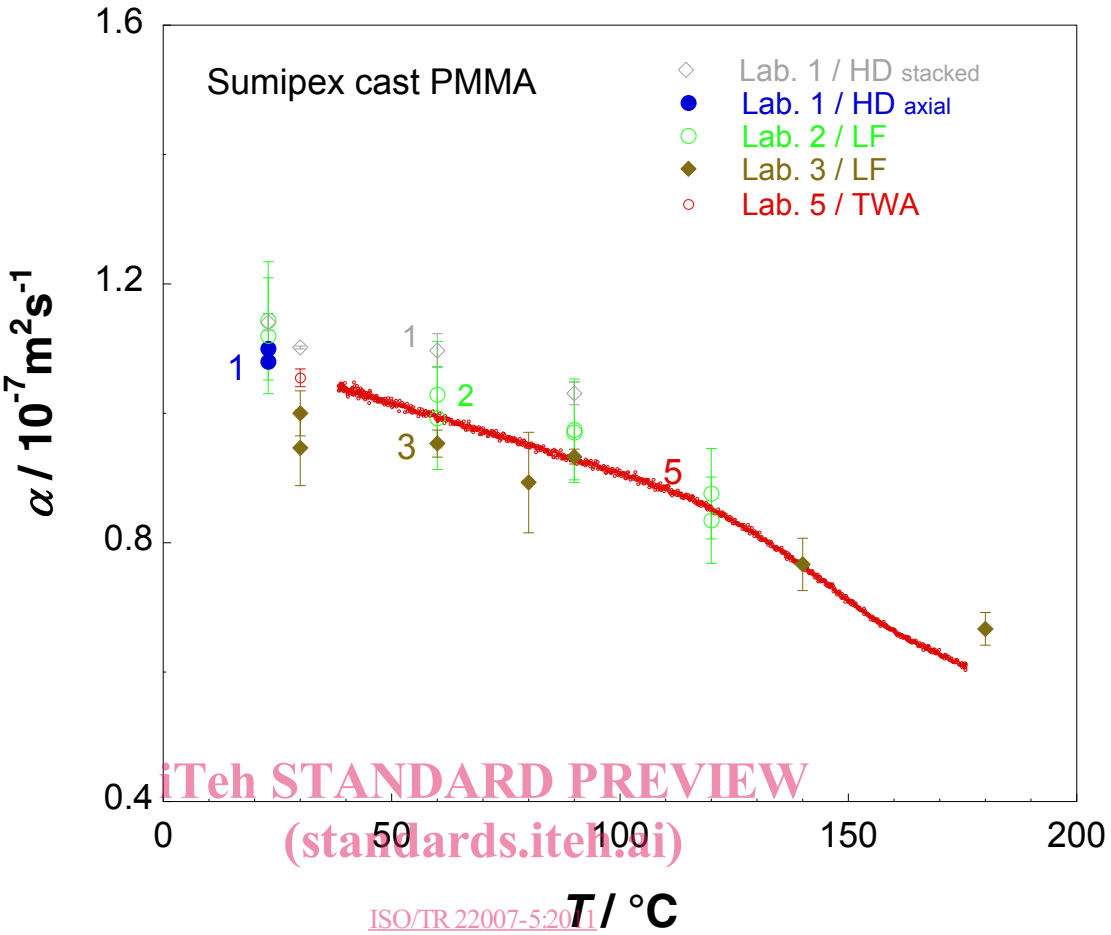

Figure 1 - Thermal diffusivity of Sumipex cast PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values:- Lab. 2 by the Laser flash method (LF) (thickness d = 2 mm), Lab. 3 by LF (d = 1,14 mm), and Lab. 5 by the Temperature wave analysis method (TWA) (d = 0,011 mm);
(ii) calculated values from thermal conductivity:- Lab. 1 by the Hot disk method (HD) (d = 2 mm for axial measurement, d = 4 mm for isotropic measurement).

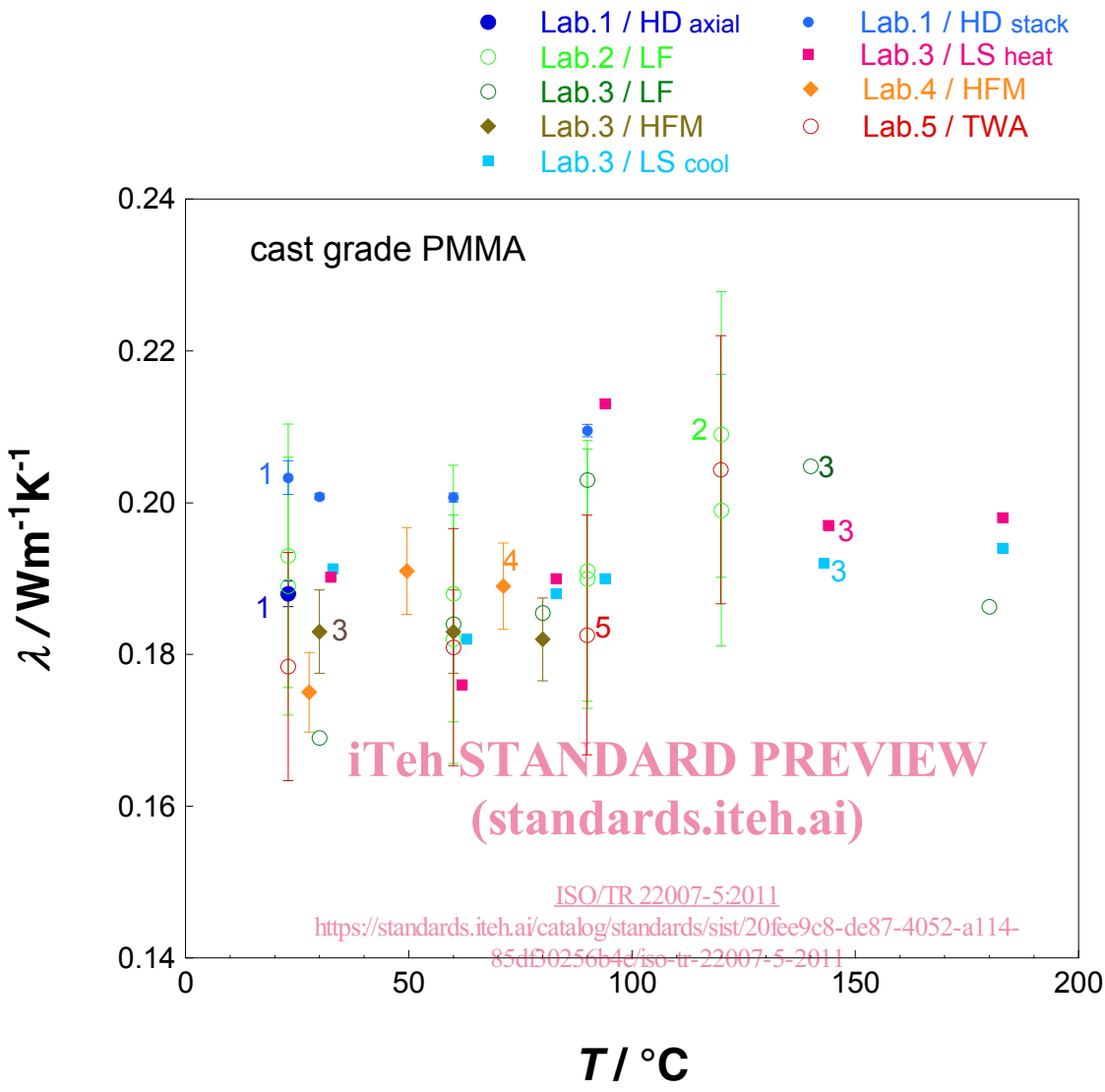

Figure 2 - Thermal conductivity of Sumipex cast PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values:- Lab. 1 by the Hot disk method (HD) (thickness d = 2 mm for axial measurement, d = 4 mm for isotropic measurement), Lab. 3 by the Heat flow meter method (HFM) (d = 2 mm) and the Line source method (LS) (d = 2 mm), and Lab. 4 by HFM (d = 2 mm);
(ii) calculated values from thermal diffusivity:- Lab. 2 by the Laser flash method (LF) (d = 2 mm), Lab. 3 by LF (d = 1,14 mm), and Lab. 5 by the Temperature wave analysis method (TWA) (d = 0,011 mm).

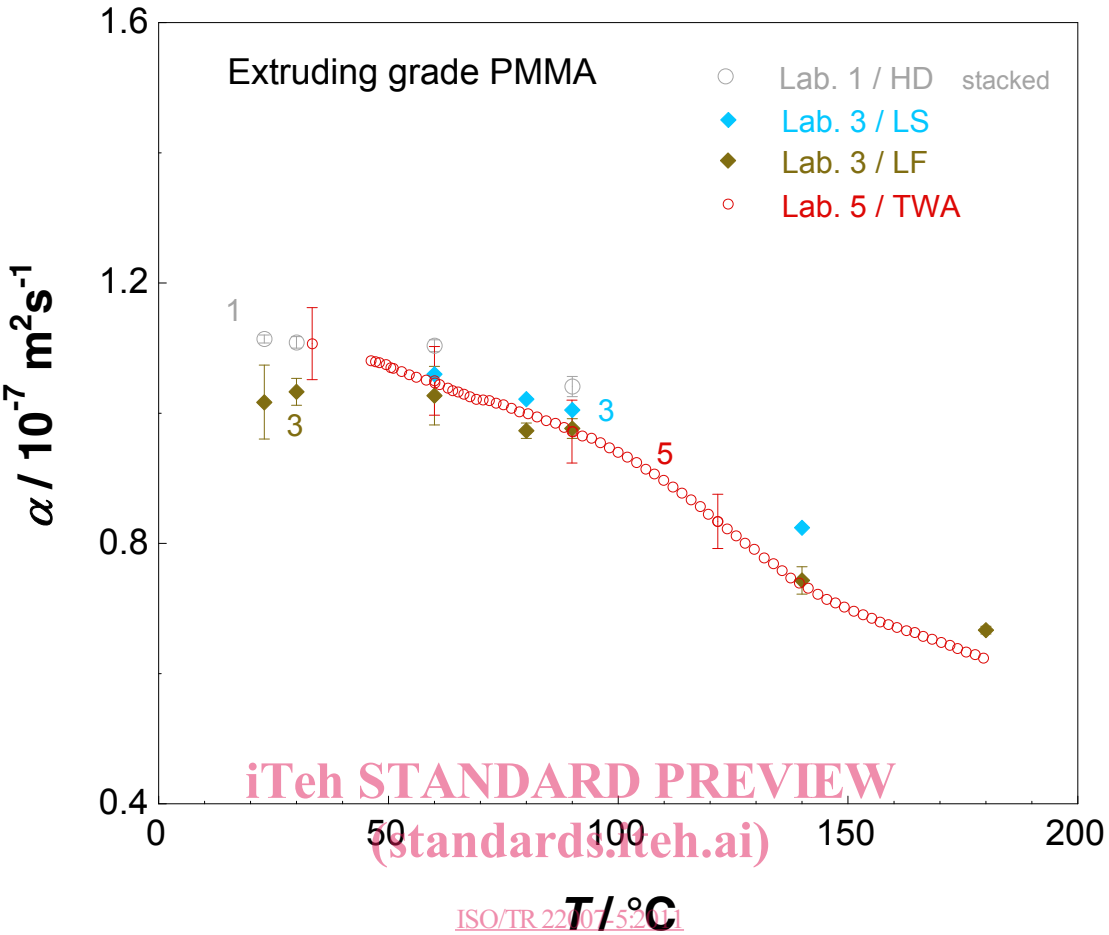

Figure 3 - Thermal diffusivity of the extrusion grade PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values: Lab. 3 by the Laser flash method (LF) (thickness d = 1,49 mm), and Lab. 5 by the Temperature wave analysis method (d = 0,012 mm);
(ii) calculated values from thermal conductivity:- Lab. 1 by the Hot disk (HD) method (d = 6 mm with two pieces stacked), and Lab. 3 by the Line source (LS) method (d = 3 mm).