TECHNICAL REPORT

ISO/TR 22007-5

First edition
2011-04-01

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)

Reference number
ISO/TR 22007-5:2011(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword iv
Introduction v
1 Scope 1
2 Symbols and definitions 1
3 Specimen preparation and characterization 1
4 Measurement apparatus 2
5 Measurement procedure 3
6 Calculations 3
7 Results and conclusions 3
8 Results 3
9 Uncertainty and repeatability 4
10 Acknowledgment 4
Annex A (informative) Instructions sent to interlaboratory comparison participants: Procedure for thermal conductivity and diffusivity intercomparison in support of the development of ISO 22007 parts 1-4 9
Annex B (informative) Laboratory 1 results 12
Annex C (informative) Laboratory 2 results 18
Annex D (informative) Laboratory 3 results 22
Annex E (informative) Laboratory 4 results 29
Annex F (informative) Laboratory 5 results 31
Bibliography 34

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or such patent rights.

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.

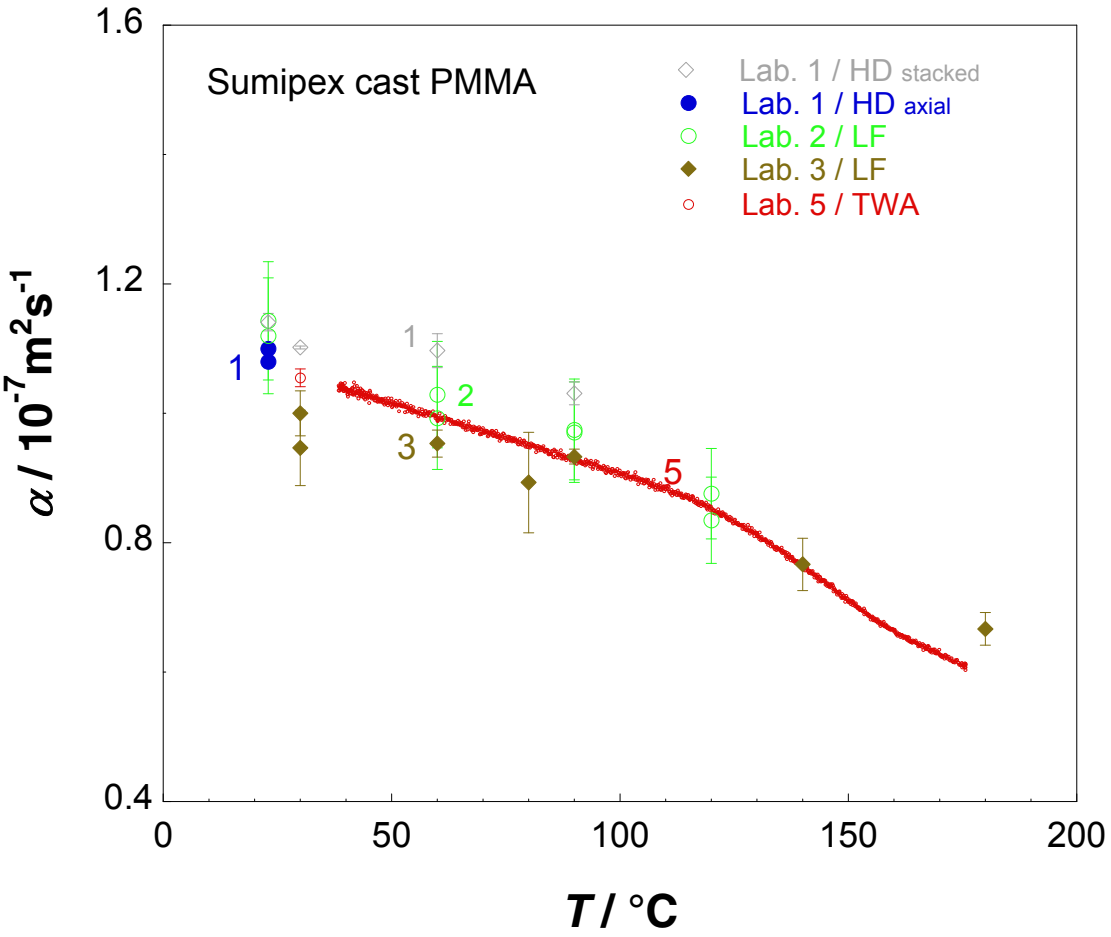

Figure 1 - Thermal diffusivity of Sumipex cast PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values:- Lab. 2 by the Laser flash method (LF) (thickness d = 2 mm), Lab. 3 by LF (d = 1,14 mm), and Lab. 5 by the Temperature 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).

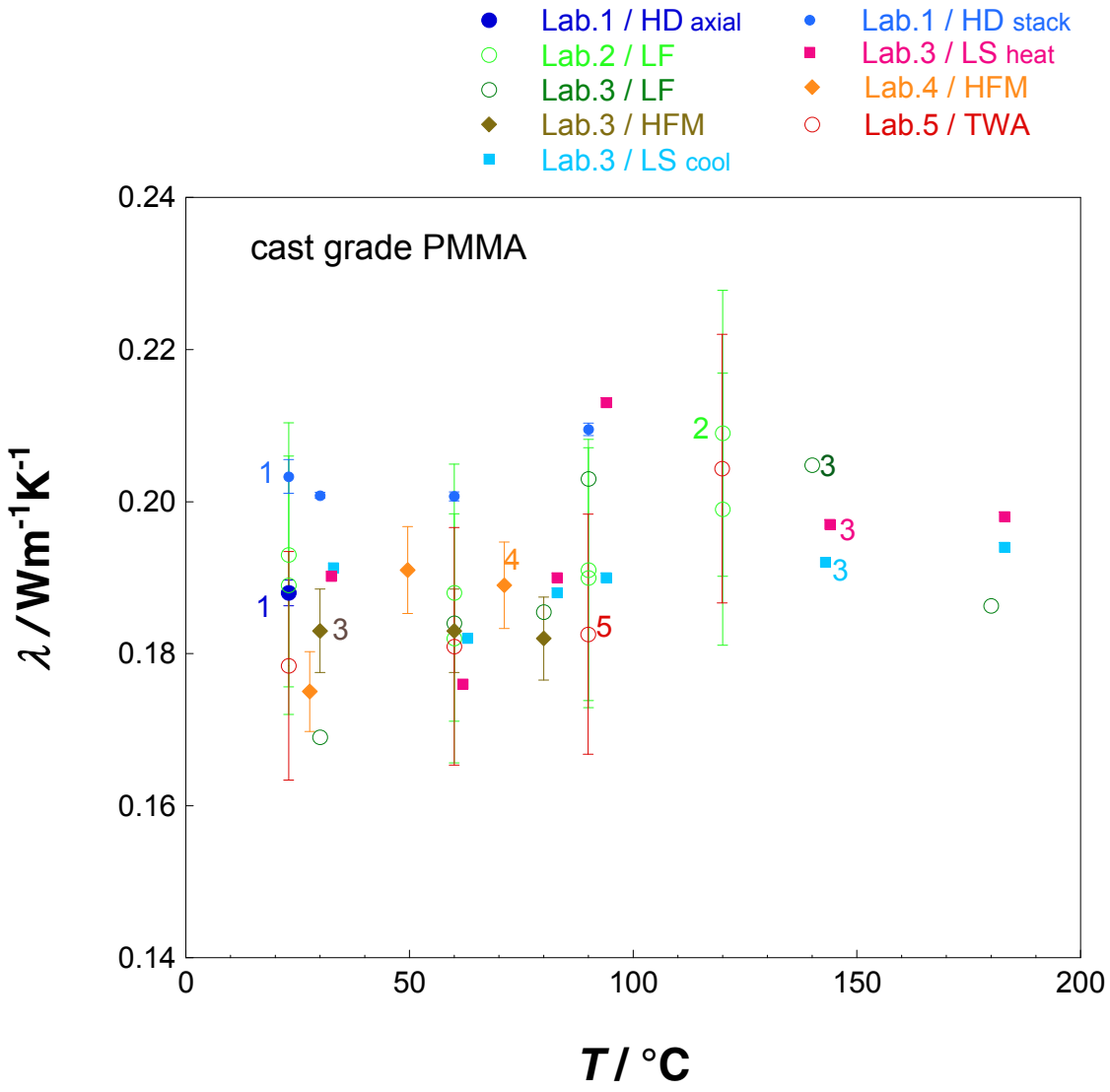

Figure 2 - Thermal conductivity of Sumipex cast PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values:- Lab. 1 by the Hot disk method (HD) (thickness d = 2 mm for axial measurement, d = 4 mm for isotropic measurement), 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).

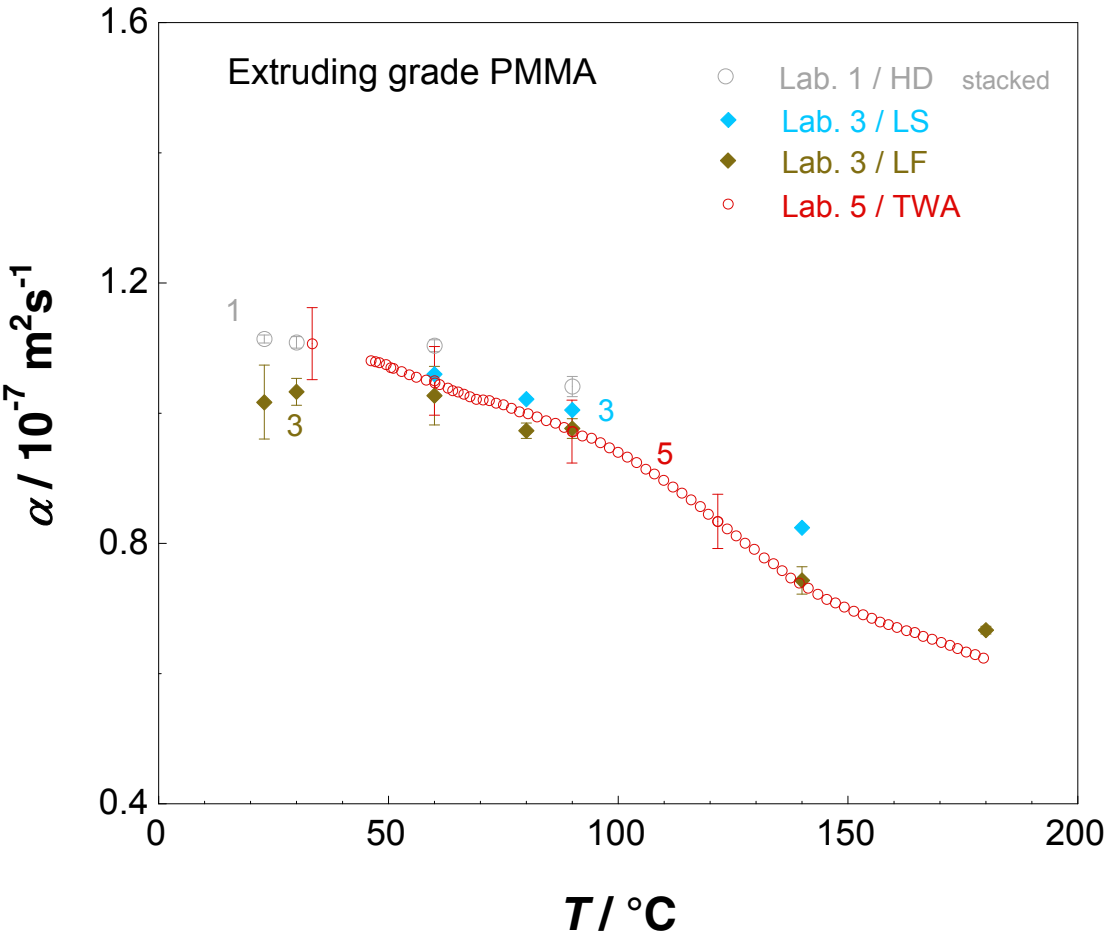

Figure 3 - Thermal diffusivity of the extrusion grade PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values: Lab. 3 by the Laser flash method (LF) (thickness d = 1,49 mm), and Lab. 5 by the Temperature wave analysis method (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).

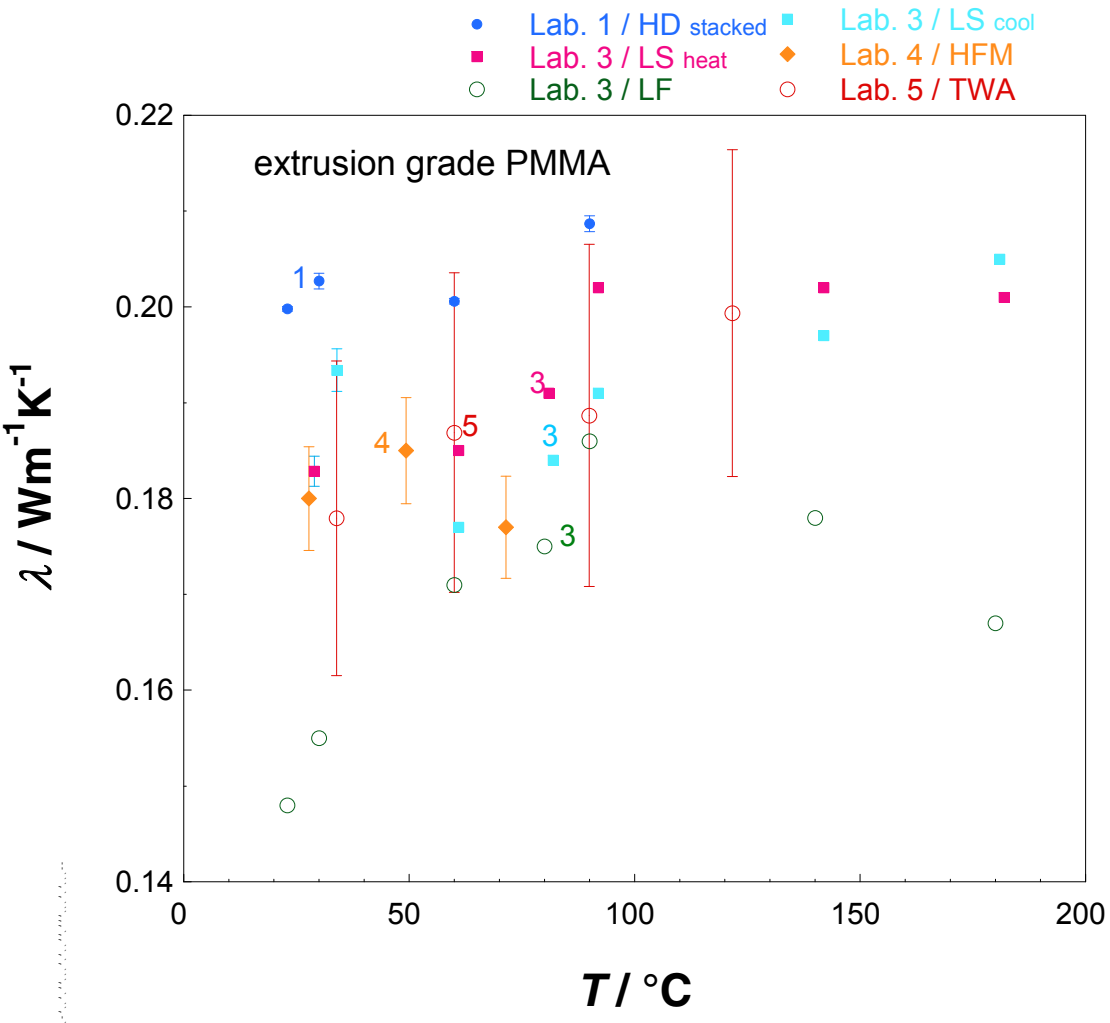

Figure 4 - Thermal conductivity of the extrusion grade PMMA in the through-thickness direction measured by the different laboratories at various temperatures *T*:
(i) directly measured values: Lab. 1 by the Hot disk method (HD) (thickness d = 6 mm with two species stacked), Lab. 3 by the Line source method ((LS) (d = 3 mm), and Lab. 4 by the Heat flow meter method (HFM) (d = 3 mm);
(ii) calculated values from thermal diffusivity: Lab. 3 by the Laser flash method (LF) (d = 1,49 mm), and Lab. 5 by the Temperature wave method (TWA) (d = 0,012 mm).

Annex A
(informative)
Instructions sent to interlaboratory comparison participants: Procedure for thermal conductivity and diffusivity intercomparison in support of the development of ISO 22007 parts 1-4

A.1 IMPORTANT INFORMATION

If you have any comments or questions concerning this intercomparison (the procedure, sample preparation etc) please send them to us before 25 February 2007 so that the issue(s) can be discussed BEFORE any participants prepare specimens or commence testing.

All results and associated documentation to be returned by 14 April 2007 if possible, please. If there are any problems with this schedule, please contact us as soon as possible.

A.2 INTRODUCTION

A.2.1 Thank you for agreeing to participate in this intercomparison on the measurement of thermal conductivity and diffusivity of polymers. This is a preliminary intercomparison amongst the project leaders that will shortly be expanded to a larger intercomparison including other organisations. The purpose of this preliminary intercomparison is to resolve any major issues that become apparent before involving the larger number of participants.

A.2.2 In summary, the objectives of this intercomparison are to assess the repeatability, reproducibility and comparability of the transient and the modulated techniques covered by ISO 22007 Parts 1 - 4 and of other techniques that may also be incorporated into this series in the future. The intention is that the findings of the intercomparison will be incorporated into ISO 22007 (or at least Part 1) as part of the precision statements, and will contribute to the development of all parts of this Standard.

A.2.3 This intercomparison is based on testing of PMMA materials to obtain thermal conductivity and thermal diffusivity data at a range of temperatures. Information that may be of use on the materials are given below.

Sumipex cast PMMA: T_g transition range starts around 100 °C, completed by approx 130 °C (at 10 °C/min); degrades above 220 °C; drying time should be 80 °C for 5 hours. A percentage of water absorption of PMMA is 0,3 %/24Hr (depending on the relative humidity), and the saturated water absorption is 2 %.

NPL thermoplastic PMMA: extrusion grade PMMA; MFI 1,6 g/10 mins (230 °C/ 3,8 kg); drying conditions 75 °C for 4 hours; Tg transition range by DSC approx 90 °C to 130 °C; typical die process temperatures 220 °C to 240 °C; decompose above 280 °C BUT may degrade at lower temperatures - recommend keep to below 240 °C.

A.2.4 This document prescribes the procedure to be followed for those measurements.

A.2.5 Tests shall be performed in accordance with the appropriate parts of ISO 22007. The latest versions of the relevant documents shall be used. Please contact myself if you need a copy.

A.2.6 Where testing using a method not currently covered by ISO 22007, state which Standard/procedure was used. If an in-house method was used please provide documentation describing the procedure and equipment.

A.3 THERMAL CONDUCTIVITY / DIFFUSIVITY TEST PLAN

A.3.1 Condition the material using the standard atmosphere (ISO 291) of (23 +/- 1)°C and relative humidity (50 % +/- 5 %) for at least 4 hours before testing.

A.3.2 The recommended test temperatures:

Cast Sumipex PMMA:

Recommended measurement temperatures are 23 °C, 30 °C (with repeats at this temperature), 60 °C and 90 °C and additional optional temperatures of 140 °C and 180 °C.

Thermoplastic NPL PMMA: (i.e. extrusion grade PMMA):

Recommended measurement temperatures are 30 °C (with repeats at this temperature), 140 °C and 180 °C, with additional optional temperatures of 23 °C, 60 °C and 80 °C.

PLEASE NOTE IT IS HIGHLY DESIRABLE THAT DATA ARE OBTAINED AT ATLEAST 30 °C FOR BOTH MATERIALS BY ALL TECHNIQUES

Participants are encouraged to test at other temperatures and, if possible, above the Tg for the thermoplastic PMMA

A.3.3 At the test temperature of 30 °C repeat the test at least a further 4 times using identical test conditions. These repeat tests should involve the complete procedure to be a true repeat test including, for example, re-conditioning the sample, measuring the specimen dimensions and re-loading the sample into the instrument. Re-testing without going through the whole procedure is not correct practice to determine the true repeatability of the method.

A.3.4 Where possible, measure the sample density and/or mass before and after testing to assess moisture uptake during the test.

A.4 ADDITONAL TESTING / DATA REQUIREMENT

A.4.1 Conversion from thermal conductivity values to thermal diffusivity values, and similarly from thermal diffusivity to thermal conductivity, requires density and specific heat capacity values. All participants should (if possible) each measure these values and convert their own results (i.e. from thermal conductivity to diffusivity or vice-versa) using those values. On analysis of all of the participants' results, further analysis will be carried out using average values for both density and specific heat capacity. The measurement of these parameters is a part of the overall measurement of thermal conductivity and/or thermal diffusivity and so is considered a valid part of this intercomparison. It will enable correct comparison of the thermal conductivity with thermal diffusivity results.

A.4.2 Measure the density of the material at the same temperature(s).

A.4.3 Measure the specific heat capacity of the material at the same temperature(s).

Note: if resourcing is an issue for any particular laboratory then the emphasis should be on the thermal conductivity/diffusivity testing.

PLEASE KEEP ALL SPECIMENS FOR FUTURE REFERENCE / USE UNTIL OTHERWISE INSTRUCTED

A.5 REPORTING RESULTS

A.5.1 Provide all information, raw data ~(e.g. plot files) and results as requested in the excel spreadsheet. The spreadsheet may be used for this purpose. Alternatively, other means of saving the information can be used (e.g. where information is provided in text or image files direct from the instrument's software). Where the information requested is "not applicable" please enter N/A.

A.5.2 Consult ISO 22007-1 and the appropriate Part of ISO 22007 for additional reporting requirements.

A.5.3 Where thermal diffusivity has been measured give values of density and specific heat capacity used for conversion to thermal conductivity, and vice-versa. Report what methods were used for measurements of density and specific heat capacity, and provide information on the standards / procedures / techniques / instruments used. Report both thermal conductivity and thermal diffusivity data, but provide clear indication as to which is/are measured and which is derived.

A.5.4 Enter comments where the current wording of the standard causes difficulties in carrying out testing, or is deficient in instruction, etc.

NOTE: Please ensure that you have documented all the information necessary to enable another person to duplicate the measurement. Much of this is likely to be in the outputs from the instrument.

A.6 REPORT ADDITIONAL INFORMATION

In addition to the results please provide additional information on the following, where available:

A.6.1 Calibration procedures

· Details of calibration procedures

A.6.2 Reference materials

· Details of reference materials used and reference values

A.6.3 Calibration data

· Results of calibrations

A.6.4 Uncertainty analysis

· Provide uncertainty analyses where available.

· Give details of tolerances on measurement parameters, e.g. on dimensions, temperature measurements.

· Provide any further repeatability data on you r measurement system that you may have.

A.7 RETURNING RESULTS

A.7.1 All results and documentation to be returned in electronic format that is readable in either Excel or Word (e.g. please do not provide files that can only be read by the instrument's software).

Annex B
(informative)
Laboratory 1 results

B.1 Hot Disk report on the ISO group Round Robin test on two types of PMMA

The two samples were received, heat treated and measured as agreed. In addition to the agreed measurements, measurements were made of the anisotropic properties as these are very different for the two materials.

The Hot Disk method [2, 10, 11, 12] measures thermal conductivity and thermal diffusivity independently in each measurement in the basic set up (using two equal samples, one on each side of the sensor). Having these two values the specific heat per unit volume ρC_p can be calculated by dividing the thermal conductivity by the thermal diffusivity. It must be understood that this value of ρC_p is only correct for isotropic materials. When the sample is anisotropic, this ratio is simply

$$\left(\lambda_a \cdot \lambda_c\right)^{1/2} / \alpha_a$$

where the subscripts indicate the axis directions of the properties (e.g. λ_a is the thermal conductivity along the *a*-axis). It is assumed here that the properties along the *a*- and *b*-axes (mapping out the plane of the sensor) are the same but different from those along the *c*-axis.

By introducing an independently measured ρC_p value for an anisotropic sample, it is possible to calculate the thermal conductivity and thermal diffusivity for the two directions, normal and parallel to the sensor surface. A method for independently measuring the heat capacity has been developed by Gustavsson et al [10] but it is not a part of the standard ISO 22007-2. This method measures ρC_p at room temperature only, which means that other methods, like a drop-calorimeter or a precise DSC must be used at other temperatures. (An advantage with DSC is that it can be measured as a function of temperature).

For these measurements, where anisotropy is so clearly distinguishing the two samples, it was judged that this should be investigated. The samples' anisotropic properties as-received and after drying, have been measured at room temperature.

B.2 Hot Disk measurements on Sumipex cast PMMA – preliminary exploratory results

Two sheets of Sumipex cast PMMA were supplied from Japan. From these, two circular samples of diameter 50 mm were cut and used for the measurements. The thickness was measured at 2 mm.

First, the material was measured as-received at room temperature (RT), with the standard method and also with the anisotropic method. A value for ρC_p was measured on a smaller sample, cut with a diameter of 12 mm.

After the initial measurements, the recommended drying process was carried out: 80 °C in a furnace for 5 hours. After this treatment the samples were stored in desiccators.

The two 50 mm diameter samples were then mounted in a special metal sample holder together with the sensor (radius 2,001 mm), and put into an oil bath thermostat with temperature regulation.

The following measurements were performed:

Standard at 23 °C, 5 s measuring time, as-received sample
Anisotropic at 23 °C, with ρC_p measured on the as-received 12 mm sample
Anisotropic at 23 °C, with ρC_p measured on the dried 12 mm sample
Standard at 23 °C, 5 s
Standard at 30 °C, 5 s
Standard at 60 °C, 5 s
Standard at 60 °C, 10 s
Standard at 90 °C, 10 s
Standard at 90 °C, 5 s
Standard at 60 °C, 10 s
Standard at 60 °C, 5 s
Standard at 30 °C, 5 s
Standard at 23 °C, 5 s

All measurements were done with a sensor 7577, radius 2,001 mm. The power was 0,075 W in all cases. At lower temperatures the time was 5 s, but as temperature increased, the thermal diffusivity was lower and allowed for a longer measuring time of 10 s. This is why both 5 s and 10 s were tried at 60 °C and 90 °C. In both cases the probing depth was always below 2 mm.

Table B.1 - Sumipex cast PMMA, as-received sample

	TC W/(m.K)	**StDev.** %	***Diff*** mm^2/s	**StDev.** %	***Apparent*** ρC_p $MJ/(m^3K)$	**StDev.** %
Front Side*	0,2063	0,31	0,135	0,62	1,52	0,47
Back Side*	0,2065	0,12	0,134	0,32	1,54	0,25

NOTE: StDev. is used as the abbreviation for standard deviation.

Table B.2 - Sumipex cast PMMA, anisotropic properties of as-received sample (ρC_p = 1,733 $MJ/(m^3K)$ StDev. 0,36 %)

TC **Axial** W/(m.K)	**StDev.** %	***Diff*** **Axial** mm^2/s	**StDev.** %	***TC*** **Radial** W/(m.K)	**StDev.** %	***Diff*** **Radial** mm^2/s	**StDev.** %
0,1863	0,35	0,108	0,32	0,2302	0,52	0,133	0,51

Table B.3 - Sumipex cast PMMA, anisotropic properties of dried sample (ρC_p = 1,730 $MJ/(m^3K)$ StDev. 0,27 %)

TC **Axial** W/(m.K)	**StDev.** %	***Diff*** **Axial** mm^2/s	**StDev.** %	***TC*** **Radial** W/(m.K)	**StDev.** %	***Diff*** **Radial** mm^2/s	**StDev.** %
0,1897	0,87	0,110	0,86	0,2246	1,03	0,130	1,03

Table B.4 - Sumipex cast PMMA

Temperature °C	Time s	*TC* W/(m.K)	StDev. %	*Diff* mm^2/s	StDev. %	*Apparent* ρC_p $MJ/(m^3K)$	StDev. %
23	5	0,2051	0,15	0,132	0,99	1,55	0,82
30	5	0,2064	0,16	0,127	0,49	1,62	0,35
60	5	0,2113	0,52	0,123	2,60	1,72	2,08
60	10	0,2117	0,22	0,120	1,18	1,77	1,00
90	5	0,2156	0,36	0,117	1,47	1,85	1,14
90	10	0,2159	0,28	0,115	3,16	1,88	2,89
60	10	0,2121	0,15	0,120	0,71	1,77	0,53
60	5	0,2121	0,14	0,124	0,77	1,72	0,66
30	5	0,2038	0,08	0,126	0,54	1,63	0,46
23	5	0,2020	0,13	0,126	1,10	1,60	0,95
140	10	0,2109	0,17	0,091	1,01	2,32	0,98

*The first measurements on as-received samples, front side and back side facing the sensor, showed that the samples do not have a difference due to up/down, through the thickness.

These results indicate that the material is strongly anisotropic. The measured thermal conductivity and thermal diffusivity are some 20 % lower in the through (thickness) direction than in the plane. Even after annealing the anisotropic property remains, meaning that at 80 °C no re-organisation of the material has taken place. It is too far from the melting point. Measurements of standard or bulk samples (all the other measurements) show an effective average of thermal conductivity and thermal diffusivity over the sampled volume.

Since the material is obviously anisotropic, the expressions used in the table column headers should be understood as follows:

$TC = \left(\lambda_a \cdot \lambda_c\right)^{1/2}$

$Diff = \alpha_a$

and

$Apparent\ \rho C_p = \left(\lambda_a \cdot \lambda_c\right)^{1/2} / \alpha_a$

where *TC* is the apparent thermal conductivity and α_a is the apparent thermal diffusivity, accounting for specimen anisotropy.

There is a very clear trend that *TC* and *Apparent* ρC_p increase and *Diff* decreases with temperature, and that the changes are reversible.

Taking into account the very low standard deviations (based on 5 measurements and given for each value in the table) even the small differences in thermal conductivity are significant.

The changing of material properties due to heating cycles can be observed in Table B.5.

Table B.5 - Sumipex cast PMMA

	TC W/(m.K)	*Diff* mm^2/s	*Apparent* ρC_p $MJ/(m^3K)$
As-received Front Side	0,2063	0,135	1,52
As-received Back Side	0,2065	0,134	1,54
After heating to 80°C, 5H measured at RT	0,2051	0,132	1,55
Followed by a full cycle to 90 °C measured at RT	0,2020	0,126	1,60

B.3 Hot Disk measurements on the extrusion grade PMMA – preliminary exploratory results

Two sheets of PMMA were supplied by NPL. From these, two circular samples with diameter 50 mm were cut and used for the measurements. The thickness was measured at 3 mm.

First, the material was measured as-received at room temperature, with the standard method and also with the anisotropic method. A value for ρC_p was measured on a smaller sample, cut with a diameter of 12 mm.

After the initial measurements, the recommended drying process was carried out: 75 °C in a furnace for 4 hours. After this treatment the samples were stored in desiccators.

The two 50 mm diameter samples were then mounted in a special metal sample holder together with the sensor (radius 3,189 mm) and put into an oil bath thermostat with temperature regulation. The following measurements were performed:

Standard at 23 °C, 20 s measuring time, as-received sample
Anisotropic at 23 °C, 20 s, with ρC_p measured on the as-received 12 mm sample
Anisotropic at 23 °C, 20 s, after drying (with ρC_p measured on a dried 12 mm sample
Standard at 23 °C, 20 s
Standard at 30 °C, 20 s
Standard at 60 °C, 20 s
Standard at 80 °C, 20 s
Standard at 80 °C, 20 s
Standard at 60 °C, 20 s
Standard at 30 °C, 20 s
Standard at 23 °C, 20 s
Standard at 140 °C, 20 s

All measurements were done with a sensor 5465, radius 3,189 mm. The power was 0,075 W in all cases. At all temperatures the measuring time was 20 s. Using this sensor and measuring time, the probing depth was always below 3 mm (compared to the Sumipex cast PMMA sample, which was only 2 mm thick; this required a smaller sensor and shorter time).

Table B.6 - Extrusion grade PMMA, as-received sample

TC W/(m.K)	**StDev.** %	***Diff*** mm^2/s	**StDev.** %	***Apparent*** ρC_p $MJ/(m^3K)$	**StDev.** %
0,2022	0,35	0,121	20,1	1,66	1,95

Table B.7 - Extrusion grade PMMA, anisotropic properties of as-received sample (ρC_p = 1,615 MJ/(m^3K), StDev 0,52 %)

TC **Axial** W/(m.K)	**StDev.** %	***Diff*** **Axial** mm^2/s	**StDev.** %	***TC*** **Radial** W/(m.K)	**StDev.** %	***Diff*** **Radial** mm^2/s	**StDev.** %
0,2010	0,29	0,125	0,39	0,2004	0,26	0,125	0,25

Table B.8 - Extrusion grade PMMA, anisotropic properties of dried sample (ρC_p = 1,604 MJ/(m^3K), StDev. 0,25 %)

TC **Axial** W/(m.K)	**StDev.** %	***Diff*** **Axial** mm^2/s	**StDev.** %	***TC*** **Radial** W/(m.K)	**StDev.** %	***Diff*** **Radial** mm^2/s	**StDev.** %
0,2048	0,99	0,127	0,98	0,2094	1,33	0,130	1,35

Table B.9 - Extrusion grade PMMA

Temperature °C	Time s	*TC* W/(m.K)	StDev. %	*Diff* mm^2/s	StDev. %	*Apparent* ρC_p $MJ/(m^3K)$	StDev. %
23	20	0,2057	0,07	0,132	0,16	1,56	0,12
30	20	0,2074	0,27	0,131	1,60	1,59	1,35
60	20	0,2090	0,18	0,121	0,51	1,73	0,35
80	20	0,2127	0,09	0,116	0,30	1,84	0,30
down							
80	20	0,2126	0,05	0,116	0,18	1,84	0,14
60	20	0,2078	0,27	0,123	1,20	1,69	0,91
30	20	0,2067	0,08	0,131	0,36	1,58	0,35
23	20	0,2069	0,13	0,134	0,59	1,54	0,48
140	20	0,2135	0,16	0,088	0,72	2,43	0,58

These results indicate that this material is much less anisotropic than the Sumipex cast PMMA. The measured thermal conductivity and thermal diffusivity are about 2 % lower in the through (thickness) direction than in the plane for the sample.

Measurements of standard or bulk samples (all the other measurements) show an effective average of thermal conductivity and thermal diffusivity over the sampled volume, which therefore is reported as *apparent* ρC_p.

Due to the fact that the material is almost isotropic, the value for ρC_p given in a standard measurement is very close to the measured value.

There is a very clear trend that thermal conductivity and heat capacity increases and thermal diffusivity decreases with temperature, and that the changes are reversible.

Taking into account the very low standard deviations (based on 5 measurements and given for each value in the table) even the small differences in thermal conductivity are significant. The difference between thermal conductivity in axial and radial direction in the anisotropic analysis of the sample is not significant, considering the standard deviation in the ρC_p measurement.

The changing of material properties due to heating cycles can be followed, Table B.10:

Table B.10 – Extrusion grade PMMA

	TC W/(m.K)	StDev. %	*Diff* mm^2/s	StDev. %	*Apparent* ρC_p $MJ/(m^3K)$	StDev. %
As-received	0,2022	0,35	0,121	20,1	1,66	1,95
After drying 75°C, 4 h	0,2057	0,07	0,132	0,16	1,56	0,12
After heating cycle to 80 °C	0,2069	0,13	0,134	0,59	1,54	0,48

B.4 Stacked specimens results

The results of stacking sheets, to respect specimen thickness criteria, are presented in Tables B.11 and B.12. Two stacked sheets were necessary to get a thickness sufficient to use a time that was long enough to get a *Total to Characteristic time* ratio of 0,45, which is within the analysis model range (0,3 - 1,0).

Table B.11 – Sumipex cast PMMA

Temperature °C	***TC*** W/(m.K)	**StDev.** %	***Diff*** m^2/s	**StDev.** %	***Apparent ρC_p*** $MJ/(m^3K)$	**StDev.** %
23	0,2033	1,1	1,141E-07	1,2	1,78	1,9
30	0,2008	0,2	1,102E-07	0,2	1,82	0,3
60	0,2007	0,3	1,097E-07	2,4	1,83	2,7
90	0,2095	0,4	1,031E-07	1,7	2,03	2,1

Table B.12 – Extrusion grade PMMA

Temperature °C	***TC*** W/(m.K)	**StDev.** %	***Diff*** m^2/s	**StDev.** %	***Apparent ρC_p*** $MJ/(m^3K)$	**StDev.** %
23	0,1998	0,12	1,114E-07	0,55	1,75	0,7
30	0,2027	0,4	1,109E-07	0,8	1,83	0,8
60	0,2006	0,14	1,104E-07	0,8	1,9	1,0
90	0,2087	0,4	1,041E-07	1,5	2,01	1,7

Annex C
(informative)
Laboratory 2 results

C.1 Laser flash specimen preparation

Two sheets of PMMA having a thickness of 2 mm (reference: Sumipex 000 lot 6621114) were received from one source and two sheets of PMMA having a thickness of 3 mm (reference: AAJHF002-3A) from a second source. Specimens for density, thermal expansion, specific heat and thermal diffusivity measurements were machined from one sheet of each type of PMMA. All specimens were dried at a temperature of 80 °C for 5 hours, and were stored in a desiccator.

As PMMA is not opaque to the laser wavelength (1,054 µm) used to measure thermal diffusivity, a thin layer (1 µm to 3 µm) of metallic coating was deposited on both faces of the thermal diffusivity specimens. Due to the low thermal diffusivity of PMMA and the relatively large thickness of the specimens, the energy deposited by the laser beam on the front face of the specimen had to be increased, in order to obtain a thermogram having a good signal/noise ratio. After some thermal diffusivity measurements, it appeared that the metallic coating did not resist to the repetition of laser impacts. It was then decided to change the coating and to apply a thin layer (≈ 30 µm) of silver paint. It was, however, not possible to acquire “good” thermograms with the 3 mm thick specimens. In consequence, the results presented hereafter were obtained on 2 mm thick sheets (Sumipex 000 lot 6621114).

C.2 Density measurements

C.2.1 Density measurements at 23 °C

The density is determined at 23°C according to the immersion method (ISO 1183-1 [20]) and is calculated by the following formula:

$$\rho_{23°C} = \frac{m_1 \cdot \rho_\ell}{(m_1 - m_2)}$$

where m_1 is the apparent mass of the specimen in air, m_2 is the apparent mass of the specimen in the immersion liquid (distilled water at 23 °C ± 0,1 °C) and ρ_ℓ is the density of the immersion liquid at 23 °C.

Measurements where performed on six specimens of Sumipex cast PMMA.

Table C.1 - Sumipex cast PMMA: density at 23 °C

Specimen	Density kg/m^3
1	1186
2	1183
3	1183
4	1185
5	1185
6	1184
Mean value	1184

C.2.2 Density measurements at *T* >23 °C

For an isotropic material, the density is determined by:

$$\rho_T = \frac{\rho_{23°C}}{\left(1+\alpha_L\right]_{T_0}^{T}\cdot(T-T_0)\right)^3}$$

where $\alpha_L\]_{T_0}^{T}$ is the mean linear thermal expansion coefficient between T_0 (23 °C) and T.

Table C.2 - Sumipex cast PMMA: density

Temperature °C	Density kg/m^3
23	1184
60	1174
90	1163
120	1152 (1)
(1) using the mean thermal expansion coefficient between 23 °C and 100 °C (see C.3)	

The uncertainty on density measurements is estimated to be ± 1 %.

C.3 Linear thermal expansion coefficient measurements

The linear thermal expansion coefficient was determined with a TMA according to ISO 11359-2 [21] standard. The tests were performed from -10 °C to 120 °C in a high purity helium atmosphere with a heating rate of 5 °C/min. Before the test, the TMA was calibrated under identical test conditions (temperature range, heating rate, atmosphere…) with reference specimens of known thermal expansion.

The mean linear thermal expansion coefficient $\alpha_L\]_{T_0}^{T}$ between *T* and T_0 is given by the following formula:

$$\alpha_L\]_{T_0}^{T} = \frac{1}{(T-T_0)}\cdot\frac{\Delta L\]_{T_0}^{T}}{L_{T_0}}$$

where $\Delta L\]_{T_0}^{T}$ is the expansion measured between T_0 to *T* and L_{T_0} is the length of the specimen at room temperature T_0 (usually T_0 = 23 °C).

Table C.3 - Sumipex cast PMMA: thermal expansion coefficient

Temperature °C	Thermal expansion coefficient 10^{-6} K^{-1}
23	-
60	80,0
90	88,2
100	93,5

Due to the presence of a glass transition towards 105 °C, the mean linear thermal expansion coefficient was not determined beyond 100 °C.

C.4 Specific heat measurements

The specific heat of Sumipex cast PMMA was determined under nitrogen atmosphere from 20 °C to 130 °C using a Differential Scanning Calorimeter DSC111 (Sétaram). These measurements were performed applying the stepwise-scanning method according to subclause 4.3 of the standard ISO 11357-4:2005 [22].

The total temperature range was divided into intervals of 5 K, which were successively scanned at a heating rate of 5 K/min. The DSC was calibrated in temperature using melting points of standard reference materials (e.g. Indium, Tin).

Table C.4 - Sumipex cast PMMA: specific heat capacity

Temperature °C	**Specific heat capacity** J/(kg.K)
23	1428
60	1557
90	1685
120	2071

The uncertainty on specific heat measurements was estimated to be ± 4 %.

C.5 Thermal diffusivity measurements

The thermal diffusivity was measured by “laser flash method” according to ISO 22007-4 [4]. Each thermal diffusivity value corresponds to the average of three consecutive measurements.

Table C.5 - Sumipex cast PMMA: thermal diffusivity

	Temperature °C	**Thermal diffusivity - measured value** 10^{-6} m^2/s	**Thickness** mm	**Thermal diffusivity - corrected value**(2) 10^{-6} m^2/s
Cycle 1	23	0,1143	2,273	0,1143
	60	0,1023	2,280	0,1029
	90	0,0963	2,286	0,0975
	120	0,0860	2,294 (3)	0,0876
Cycle 2	23	0,1120	2,273	0,1120
	60	0,0987	2,280	0,0993
	90	0,0960	2,286	0,0971
	120	0,0820	2,294 (3)	0,0835
After cycle 2	23	0,1140	2,273	0,1140

(2) with thermal expansion correction
(3) using the mean thermal expansion coefficient between 23 °C and 100 °C

After the measurements, the silver paint layer seemed degraded. A progressive modification of the deposit during the two thermal cycles could explain the huge repeatability (more than 4 % in some cases) obtained for three successive measurements. Taking into account this large repeatability, the uncertainty in these thermal diffusivity measurements was estimated to be ± 8 %.

C.6 Thermal conductivity determination

The thermal conductivity λ was determined by calculation using:

$$\lambda = a \cdot \rho \cdot c_p$$

Table C.6 - Sumipex cast PMMA: calculated thermal conductivity

	Temperature °C	Thermal diffusivity 10^{-6} m^2/s	Density kg/m^3	Specific heat capacity J/(kg.K)	Thermal conductivity W/(m.K)
Cycle 1	23	0,1143	1184	1428	0,193
	60	0,1029	1174	1557	0,188
	90	0,0975	1163	1685	0,191
	120	0,0876	1152	2071	0,209
Cycle 2	23	0,1120	1184	1428	0,189
	60	0,0993	1174	1557	0,182
	90	0,0971	1163	1685	0,190
	120	0,0835	1152	2071	0,199
After cycle 2	23	0,1140	1184	1428	0,193

The uncertainty in these calculated thermal conductivity values was estimated to be ± 9 %.

The behaviour of the coating (used in the case of semi-transparent material) during the thermal diffusivity tests has a big influence on the quality of the measurement results. It clearly appeared that the coatings we usually use for thermal diffusivity measurements of semi-transparent material were not suitable in this case.

The temperature calibrations were performed either with reference materials (for DSC and TMA, respectively according to ISO 11357-1 [23] and 11359-1 [24]), or by using a calibrated thermocouple (for the temperature calibration of the diffusivimeter).

The uncertainties of measurement (coverage factor k = 2) were calculated in the particular case of the Sumipex cast PMMA (semi-transparent material) according to the *Guide to the expression of uncertainty in measurement* [19]. They were estimated to be ± 4 % for specific heat, ± 8 % for thermal diffusivity and ± 9 % for thermal conductivity.

Annex D
(informative)
Laboratory 3 results

D.1 Sumipex cast PMMA

D.1.1 Solid density

Method: ASTM D 792 – 00 Density and Specific Gravity (Relative Density) of Plastics by Displacement, Method A
Instrument: Analytical balance

Specimen type: sheet
conditioning 40 hrs. 21 °C, 51 %RH
other preparation cut from sheet
Parameters: water temperature 22,3 °C
Uncertainty: per standard

Table D.1 - Sumipex cast PMMA: density

Replicate	Density kg/m^3
1	1192,1
2	1188,5
Mean	1190,3

D.1.2 Specific heat

Method: Based on ASTM E 1269 – 05 Determining Specific Heat Capacity by Differential Scanning Calorimetry
Instrument: Perkin Elmer DSC7
Specimen type: sheet
drying none
other preparation cut from sheet

Parameters:
purge gas N_2, purge gas purity 99,99 %, purge gas rate 25 ml/min
cooling rate 20 °C/min, initial temperature 180 °C, final temperature 20 °C
equilibration times 4 min
sample weight 3,97 mg
sample pans Al, volatile
Calibration standards:
temperature In, Zn
heat flow In
specific heat sapphire
Transition analysis:
extrapolated onset 110 °C, inflection point 100 °C, extrapolated end 92 °C

Table D.2 - Sumipex cast PMMA: specific heat capacity by DSC

Temperature °C	Specific heat capacity, C_p J/(kg.K)
23	1419
30	1460
60	1622
80	1744
90	1827
120	2183
140	2244
180	2348

D.1.3 Thermal diffusivity (laser flash)

Method: ASTM E1461-01
Standard Test Method for Thermal Diffusivity of Solids by the Flash Method
Instrument: Holometrix FLASH
Specimen type: disc
conditioning 40 hrs 23 °C 50 %RH
other preparation sputtering / 3x graphite
thickness 1,14 mm

Parameters: thermal pulse source laser
beam uniformity ensured using filters
response detector infrared
variation due to % rise < 3 %
repeat measurements 3 per temperature
Corrections: thermal expansion N/A
heat losses N/A
finite pulse time effects N/A
Uncertainty: per standard

Table D.3 - Sumipex cast PMMA: thermal diffusivity by laser flash

Temperature °C	**Thermal diffusivity** m^2/s	**StDev.** m^2/s	**Specific heat capacity** J/(kg.K)	**Thermal conductivity** W/(m.K)
30	1,00E-07	3,46E-09	1460	0,17
30	9,47E-08	5,86E-10	1460	0,16
60	9,53E-07	2,08E-09	1621	0,18
80	8,93E-08	7,77E-09	1744	0,19
90	9,33E-08	1,15E-09	1827	0,20
140	7,67E-08	4,04E-09	2244	0,20
180	6,67E-08	2,52E-09	2347	0,19

Table D.4 - Sumipex cast PMMA: thermal diffusivity by laser flash, retested

Temperature °C	**Thermal diffusivity** m^2/s	**StDev.** m^2/s	**Specific heat capacity** J/(kg.K)	**Thermal conductivity** W/(m.K)
23	1,18E-07	1,53E-09	1460	0,20
30	1,18E-07	5,77E-10	1460	0,21
60	1,14E-07	1,73E-09	1621	0,22
80	1,09E-07	2,08E-09	1744	0,23
90	1,06E-07	1,53E-09	1827	0,23
140	8,50E-08	1,00E-09	2244	0,23
180	7,93E-08	2,31E-09	2347	0,22

D.1.4 Thermal conductivity (heat flow meter)

Method: ASTM E1530 Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by Guarded Heat Flow Meter Technique
Instrument: Netzsch TCA-300

Specimen type: sheet
conditioning none
other preparation cut to 2"diameter size
thickness 2,03 mm
Accuracy: ±3 %
NOTE: Material softened beyond 90 °C

Table D.5 - Sumipex cast PMMA: thermal conductivity by heat flow meter

Temperature °C	**Thickness** mm	**Thermal resistance** m^2K/W	**Thermal conductivity** W/(m.K)
30	2,03	1,11E-02	0,184
30	2,03	1,11E-02	0,183
30	2,03	1,11E-02	0,183
30	2,03	1,11E-02	0,183
60	2,03	1,11E-02	0,183
80	2,03	1,12E-02	0,182

D.1.5 Thermal conductivity (line source)

D.1.5.1 Line source - cooling scan

Method: ASTM D 5930 - 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument: K-System II Thermal Conductivity System
Specimen type: sheet
drying 5 hrs 80 °C w/vac
other preparation cut to size

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 220 °C
initial temperature 180 °C
final temperature 30 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard

Table D.6 – Sumipex cast PMMA: thermal conductivity by line source (cooling scan)

Temperature °C	Thermal conductivity W/(m.K)
183	0,194
143	0,192
94	0,190
83	0,188
63	0,182
35	0,192
34	0,193
34	0,195
33	0,187
32	0,190
32	0,191

D.1.5.2 Line source - heating scan

Method: ASTM D 5930 - 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument: K-System II Thermal Conductivity System
Specimen type: sheet
drying 5 hrs 80 °C w/vac
other preparation cut to size

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 30 °C
initial temperature 30 °C
final temperature 180 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard

Table D.7 - Sumipex cast PMMA: thermal conductivity by line source (heating scan)

Temperature °C	Thermal conductivity W/(m.K)
183	0,198
144	0,197
94	0,213
83	0,190
62	0,176
34	0,191
33	0,192
33	0,182
32	0,192
32	0,193
31	0,191

D.2 PMMA pellets

D.2.1 Thermal conductivity (line source)

D.2.1.1 Line source – cooling scan

Method: ASTM D 5930 - 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument: K-System II Thermal Conductivity System
Specimen type: pellets
drying 4 hrs 75 °C w/vac
other preparation none

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 200 °C
initial temperature 180 °C
final temperature 30 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard

Table D.8 - Extrusion grade PMMA (pellets): thermal conductivity by line source (cooling scan)

Temperature °C	**Thermal conductivity** W/(m.K)
183	0,201
143	0,197
93	0,191
83	0,190
62	0,171
35	0,190
34	0,196
33	0,188
33	0,189
32	0,189
32	0,187

D.2.1.2 Line source – heating scan

Method: ASTM D 5930 – 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument: K-System II Thermal Conductivity System
Specimen type: pellets
drying 4 hrs @ 75 °C w/vac
other preparation none

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 30 °C
initial temperature 30 °C
final temperature 180 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard
NOTE: The same sample was used from the cooling scan experiment

Table D.9 - Extrusion grade PMMA (pellets): thermal conductivity by line source (heating scan)

Temperature °C	Thermal conductivity W/(m.K)
183	0,200
143	0,200
93	0,203
83	0,190
62	0,183
31	0,180
31	0,181
31	0,182
31	0,181
31	0,182
31	0,181

D.3 Extrusion grade PMMA

D.3.1 Solid density

Method: ASTM D 792 - 00
Density and Specific Gravity (Relative Density) of Plastics by Displacement, Method A
Instrument: Analytical balance
Specimen type: sheet
conditioning 40 hrs. 21 °C, 51 %RH
other preparation cut from sheet

Parameters: water temperature 22,4 °C
Uncertainty: per standard

Table D.10 - Extrusion grade PMMA: density

Replicate	Density kg/m^3
1	1185,7
2	1187,0
Mean	1186,4

D.3.2 Specific heat

Method: Based on ASTM E 1269 - 05
Determining Specific Heat Capacity by Differential Scanning Calorimetry (DSC)
Instrument: Perkin Elmer DSC7
Specimen type: sheet
drying none
other preparation cut from sheet

Parameters:
purge gas N_2, purge gas purity 99,99 %, purge gas rate 25 ml/min
cooling rate 20 °C/min, initial temperature 180 °C, final temperature 20 °C
equilibration times 4 min
sample weight 5,84 mg
sample pans Al, volatile
Calibration standards: temperature In, Zn
heat flow In
specific heat sapphire
Transition analysis:
extrapolated onset 110 °C, inflection point 101 °C, extrapolated end 91 °C

Table D.11 - Extrusion grade PMMA: specific heat capacity by DSC

Temperature °C	Specific heat capacity, C_p J/(kg.K)
23	1227
30	1262
60	1407
80	1520
90	1602
120	1955
140	2016

D.3.3 Thermal diffusivity (laser flash)

Method: ASTM E1461-01
Standard Test Method for Thermal Diffusivity of Solids by the Flash Method
Instrument: Holometrix FLASH
Specimen type: disc
conditioning 40 hrs 23 °C, 50 %RH
other preparation sputtering / 3x graphite
thickness 1,489 mm

Parameters: thermal pulse source laser
beam uniformity ensured using filters
response detector infrared
variation due to % rise < 3 %
repeat measurements 3 per temperature
Corrections: thermal expansion N/A
heat losses N/A
finite pulse time effects N/A
Uncertainty: per standard

Table D.12 - Extrusion grade PMMA: thermal diffusivity by laser flash

Temperature °C	Thermal diffusivity m^2/s	StDev. m^2/s	Specific heat capacity J/(kg.K)	Thermal conductivity W/(m.K)
23	1,017E-07	5,686E-09	1227	0,148
30	1,033E-07	2,082E-09	1263	0,155
60	1,027E-07	4,509E-09	1407	0,171
80	9,733E-08	1,155E-09	1520	0,175
90	9,767E-08	1,528E-09	1602	0,186
140	7,433E-08	2,082E-09	2016	0,178
180	6,667E-08	5,774E-10	2111	0,167

D.3.4 Thermal conductivity

D.3.4.1 Line source – cooling scan

Method: ASTM D 5930 - 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument: K-System II Thermal Conductivity System
Specimen type: sheet
drying 4 hrs, 75 °C w/vac
other preparation cut to size

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 200 °C
initial temperature 180 °C
final temperature 30 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard

Table D.13 - Extrusion grade PMMA: thermal conductivity by line source (cooling scan)

Temperature °C	Thermal conductivity W/(m.K)
181	0,205
142	0,197
92	0,191
82	0,184
61	0,177
36	0,193
35	0,194
35	0,192
34	0,193
34	0,198
33	0,191
33	0,193

D.3.4.2 Line source - heating scan

Method: ASTM D 5930 - 01
Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique
Instrument K-System II Thermal Conductivity System
Specimen type: sheet
drying 4 hrs, 75 °C w/vac
other preparation cut to size

Parameters:
calibration material 60,000 cstk PDMS
probe constant 0,733
probe length 50 mm
loading temperature 30 °C
initial temperature 30 °C
final temperature 180 °C
probe voltage 2,5 V
acquisition time 45 s
Uncertainty: per standard

Table D.14 - Extrusion grade PMMA: thermal conductivity by line source (heating scan)

Temperature °C	Thermal conductivity W/(m.K)
182	0,201
142	0,202
92	0,202
81	0,191
61	0,185
29	0,181
29	0,184
29	0,184
29	0,182
29	0,183
29	0,185
29	0,181

Annex E
(informative)
Laboratory 4 results

E.1 Sumipex cast PMMA

Table E.1 - Sumipex cast PMMA: thermal conductivity by heat flux

Nominal temperature °C	90	60	30
Average temperature °C	71,2	49,6	27,7
Thermal conductivity W/(m.K)	0,189	0,191	0,175

Estimate of repeatability (based on testing of similar material) 3 % at 95 % confidence level.

Table E.2 - Sumipex cast PMMA: specific heat capacity by DSC

Temperature °C	**Specific heat capacity** J/(kg.K)			**Mean specific heat capacity** J/(kg.K)	**StDev** J/(kg.K)
	Test reference				
	5 - 035	**6 - 036**	**7 - 037**		
23	1359	1390	1268	1339	64
30	1393	1425	1304	1374	62
40	1440	1471	1351	1421	62
50	1492	1523	1405	1473	61
60	1547	1575	1460	1527	60
70	1596	1623	1500	1573	65
80	1646	1665	1550	1620	62
90	1708	1721	1615	1681	58
100	1772	1781	1695	1750	47
110	1912	1921	1844	1892	42
120	2241	2270	2183	2231	44
130	2189	2187	2114	2164	43
140	2217	2211	2138	2188	44
150	2259	2246	2177	2227	44
160	2308	2284	2224	2272	43
170	2490	2445	2404	2446	43
180	2574	2548	2470	2531	54

Table E.3 - Sumipex cast PMMA: density by Archimedes principle

Temperature °C	**Operator#1 test no.**	**Density** kg/m^3		**Operator#2 test no.**	**Density** kg/m^3
23	1	1184,9		1	1186,6
23	2	1187,4		2	1187,1
23	3	1186,1		3	1186,2
23	4	1186,5		4	1186,5
23	5	1185,3		5	1186,2
Mean		1186,0		Mean	1186,5
StDev.		1,0		StDev.	0,4

E.2 Extrusion grade PMMA

Table E.4 - Extrusion grade PMMA: thermal conductivity by heat flux meter

Test temperature	°C	71,5	49,3	27,8
Thermal conductivity	W/(m.K)	0,177	0,185	0,180

Estimate of repeatability (based on testing of similar material) 3 % at 95 % confidence level.

Table E.5 - Extrusion grade PMMA: specific heat capacity by DSC

Temper-ature °C	**Specific heat capacity** J/(kg.K)									**Mean C_p** J/(kg.K)	**StDev. C_p** J/(kg.K)
	Test reference										
	# 2-022	**# 2-024**	**# 2-025**	**# 3-027**	**# 2-028**	**# 1-033**	**# 4-038**	**# 6-040**	**# 4-042**		
30	1334	1328	1319	1338	1295	1363	1417	1426	1431	1361	51
40	1389	1383	1376	1394	1347	1421	1465	1474	1478	1414	48
50	1440	1428	1418	1445	1394	1477	1514	1527	1526	1463	49
60	1498	1472	1462	1505	1437	1538	1568	1585	1576	1516	54
70	1551	1513	1504	1562	1477	1599	1621	1640	1617	1565	58
80	1591	1559	1551	1601	1521	1644	1682	1704	1665	1613	64
90	1646	1612	1607	1659	1574	1704	1741	1758	1721	1669	65
100	1720	1678	1676	1736	1641	1786	1815	1830	1796	1742	68
110	1929	1804	1807	1888	1768	1944	1972	1976	1961	1894	81
120	2051	2009	2016	2063	1971	2126	2139	2148	2156	2075	69
130	2092	2049	2061	2101	2011	2175	2181	2201	2182	2117	70
140	2129	2082	2100	2138	2045	2218	2221	2243	2217	2155	72
150	2168	2121	2141	2175	2080	2261	2263	2285	2255	2194	74
160	2200	2156	2177	2206	2115	2297	2299	2320	2289	2229	74
170	2230	2192	2212	2235	2147	2334	2332	2352	2325	2262	75
180	2256	2228	2249	2263	2179	2367	2362	2382	2359	2294	74

Table E.6 - Extrusion grade PMMA: density by Archimedes principle

Temperature °C	**Operator#1 test no.**	**Density** kg/m^3		**Operator#2 test no.**	**Density** kg/m^3
23	1	1183,1		1	1186,3
23	2	1185,6		2	1185,4
23	3	1187,2		3	1184,9
23	4	1182,3		4	1184,2
23	5	1188,4		5	1185,7
	Mean	1185,3		**Mean**	1185,3
	StDev.	2,6		**StDev.**	0,8

Annex F
(informative)
Laboratory 5 results

F.1 Sumipex cast PMMA

F.1.1 Thermal diffusivity (temperature wave analysis)

Method ISO 22007-3
Instrument ai-Phase mobile 1
Specimen The specimen was sliced from the cast sheet by using a micro-tomb into a thickness of 11 μm
with an area size of 3 mm x 5 mm.
Conditioning 48 hrs 30 °C
Heater size 1 mm x 4 mm
Sensor size 0,5 mm x 2 mm
Frequency 100 Hz - 1 kHz
Temperature scan rate 0,4 °C/min
Frequency at a temperature scan 260 Hz
Repeat measurements 400 per one temperature
Uncertainty 2,6 %

Table F.1 - Sumipex cast PMMA: thermal diffusivity by temperature wave analysis

Temperature °C	Thermal diffusivity m^2/s
40,0	1,04E-07
45,0	1,03E-07
50,0	1,02E-07
54,9	1,01E-07
60,0	9,95E-08
65,1	9,84E-08
69,9	9,73E-08
75,0	9,62E-08
80,0	9,51E-08
85,0	9,40E-08
90,0	9,29E-08
95,1	9,18E-08
100,0	9,07E-08
105,0	8,95E-08
110,0	8,82E-08
115,0	8,68E-08
119,9	8,52E-08
125,0	8,32E-08
130,0	8,11E-08
135,0	7,88E-08
140,0	7,64E-08
145,0	7,38E-08
150,0	7,12E-08
155,0	6,87E-08
160,0	6,65E-08
165,0	6,46E-08
170,0	6,28E-08
175,0	6,11E-08

F.1.2 Thermal conductivity determination

The thermal conductivity λ was determined by calculation using $\lambda = a \cdot \rho \cdot c_p$

Table F.2 - Sumipex cast PMMA: thermal conductivity by calculation

Temperature °C	α m^2/s	**StDev.** m^2/s	Cp^* J/(kg.K)	**StDev.** J/(kg.K)	ρ^{**} kg/m^3	**StDev.** kg/m^3	λ W/(m.K)	**StDev.** W/(m.K)
23	1,06E-07	1,38E-09	1430	57,1	1180	1,21	0,179	7,51E-03
60	9,95E-08	1,29E-09	1560	62,3	1170	11,7	0,182	7,82E-03
90	9,29E-08	1,21E-09	1690	67,4	1160	11,6	0,182	7,89E-03
120	8,52E-08	1,11E-09	2070	82,8	1150	11,5	0,203	8,83E-03

$^*C_p$: cited values from the results of Lab. 2
$^{**}\rho$: cited values from the results of Lab. 2

The uncertainty in thermal diffusivity measurement (coverage factor k = 2) was calculated for Sumipex cast PMMA according to the *Guide to the expression of uncertainty in measurement* [19]. The expanded uncertainty was calculated for thermal conductivity using the experimental values for thermal diffusivity and the equation $\lambda = \alpha\ C_p\ \rho$. The uncertainties were estimated to be $\pm$ 2,6 % for thermal diffusivity and $\pm$ 8,4 % for thermal conductivity.

F.2 Extrusion grade PMMA

F.2.1 Thermal diffusivity (temperature wave analysis)

Method ISO22007-3
Instrument ai-Phase mobile 1
Specimen The specimen was sliced from the cast sheet using a micro-tomb into a thickness of 12 μm with an area size of 3 mm x 5 mm
Conditioning 48 hrs 30 °C
Heater size 1 mm x 4 mm
Sensor size 0,5 mm x 2 mm
Frequency 100 Hz - 1 kHz
Temperature scan rate 1,0 °C/min
Frequency at a temperature scan 280 Hz
Repeat measurements 400 per one temperature
Uncertainty 5,0 %

Table F.3 – Extrusion grade PMMA: thermal diffusivity by temperature wave analysis

Temperature °C	Thermal diffusivity m^2/s
50,1	1,07E-07
55,3	1,06E-07
60,1	1,05E-07
65,2	1,03E-07
70,6	1,02E-07
75,0	1,01E-07
80,4	1,00E-07
84,3	9,89E-08
90,2	9,72E-08
96,2	9,55E-08
99,9	9,40E-08
106,0	9,15E-08
109,9	8,97E-08
115,9	8,68E-08
121,7	8,34E-08
125,6	8,12E-08
129,6	7,91E-08
135,7	7,59E-08
141,3	7,32E-08
145,4	7,14E-08
149,2	7,03E-08
155,2	6,85E-08
160,7	6,71E-08
164,6	6,63E-08
170,3	6,48E-08
175,7	6,34E-08
181,2	6,18E-08

F.2.2 Thermal conductivity determination

The thermal conductivity λ was determined by calculation as $\lambda = a \cdot \rho \cdot c_p$.

Table F.4 – Extrusion grade PMMA: thermal conductivity by calculation

Temperature °C	α m^2/s	StDev. m^2/s	C_p* J/(kg.K)	StDev. J/(kg.K)	ρ** kg/m^3	StDev. kg/m^3	λ W/(m.K)	StDev. W/(m.K)
34	1,11E-07	2,77E-09	1360	51	1180	11,8	0,178	8,21E-03
60	1,05E-07	2,63E-09	1520	54	1170	11,7	0,187	8,34E-03
90	9,72E-08	2,43E-09	1670	65	1160	11,6	0,189	8,93E-03
122	8,34E-08	2,09E-09	2080	69	1150	11,5	0,199	8,53E-03

*C_p : cited values from the results of Lab. 4.
**ρ : cited values from the results of Lab. 2 for Sumipex cast PMMA.

The uncertainty of measurement (coverage factor k = 2) was calculated for AAJHF extruded PMMA according to the *Guide to the expression of uncertainty in measurement* [19]. The expanded uncertainty was calculated for thermal conductivity using the experimental values for thermal diffusivity and the equation $\lambda = \alpha C_p \rho$. They were estimated to be ± 5,0 % for thermal diffusivity and ± 9,5 % for thermal conductivity.

Bibliography

[1] ISO 22007-1:2009 *Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 1: General principles*

[2] ISO 22007-2:2008 *Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 2: Transient plane heat source (hot disc) method*

[3] ISO 22007-3:2008 *Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 3: Temperature wave analysis method*

[4] ISO 22007-4:2008 *Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 4: Laser flash method*

[5] ISO 472:1999 *Plastics -- Vocabulary*

[6] *Sumipex Sheet Technical Data Book*, July 2003, Sumitomo Chemical Co., Ltd

[7] ASTM D 5930, *Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique*

[8] ASTM E 1530, *Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by the Guarded Heat Flow Meter Technique*

[9] Dawson, A., Rides, M., Allen, C. R. G., and Urquhart, J. M., Polymer–mould interface heat transfer coefficient measurements for polymer processing, *Polymer Testing,* **27** pp. 555-565 (2008)

[10] Gustavsson, M., Saxena, N. S., Karawacki, E., and Gustafsson, S.E., Specific Heat Measurement with the Hot Disk Thermal Constants Analyser, *Thermal Conductivity*, **23**, pp. 56-65 (1996)

[11] Gustafsson, S. E., Transient plane source techniques for thermal conductivity and thermal diffusivity measurements of solid materials, *Rev. Sci. Instrum.*, **62** (3), pp. 797-804 (1991)

[12] Lundström, D, Karlsson, B., and Gustavsson, M., Zeitschrift fur Metall kunde, **92**, 11 (2001)

[13] Hay, B., Flitz, J. R., Hameury, J., Rongione, L., Uncertainty of Thermal Diffusivity Measurements by Laser Flash Method, *Int. J. Thermophys.*, **26** (6), pp. 1883-1898 (2005)

[14] Lobo, H., and Cohen, C., *Polym. Eng. Sci.*, **30**, p. 65 (1990)

[15] Lobo, H., Thermal Conductivity and Diffusivity, *Handbook of Plastic Analysis*, Ch. 5, H. Lobo and J. Bonilla (eds.), Marcel Dekker (2003)

[16] Morikawa, J., Tan, J., and Hashimoto, T., *Polymer,* **36** (23), pp. 4439-4443 (1995)

[17] Hashimoto, T., *Data Book of Thermal Diffusivity of Polymers*, Youtes, Tokyo 1994

[18] Rides, M., Morikawa, J., Halldahl, L., Hay, B., Lobo, H., Dawson, A., and Allen, C., Intercomparison of thermal conductivity and thermal diffusivity methods for plastics, *Polymer Testing*, **28**, Issue 5, August 2009, pp. 480-489

[19] ISO/IEC Guide 98:1995 *Guide to the expression of uncertainty in measurement (GUM)*

[20] ISO 1183-1 *Plastics -- Methods for determining the density of non-cellular plastics -- Part 1: Immersion method, liquid pyknometer method and titration method*

[21] ISO 11359-2 *Plastics -- Thermomechanical analysis (TMA) -- Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

[22] ISO 11357-4:2005 *Plastics -- Differential scanning calorimetry (DSC) -- Part 4: Determination of specific heat capacity*

[23] ISO 11357-1 *Plastics -- Differential scanning calorimetry (DSC) -- Part 1: General principles*

[24] ISO 11359-1 *Plastics -- Thermomechanical analysis (TMA) -- Part 1: General principles*

ICS 83.080.01

Price based on 35 pages