INTERNATIONAL STANDARD

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Optics and optical instruments — Lasers and laser-related equipment — Test method for absorptance of optical laser components

Optique et instruments d'optique — Lasers et équipements associés aux lasers — Méthode d'essai du facteur d'absorption des composants optiques pour lasers

Reference number
ISO 11551:2003(E)

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Contents

Page

Foreword

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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.

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 all such patent rights.

ISO 11551 was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 9, *Electro-optical systems*.

This second edition cancels and replaces the first edition (ISO 11551:1997), which has been technically revised.

Introduction

To characterize an optical component, it is important to know its absorptance. When radiation impinges upon a component, a part of that radiation is absorbed, increasing the temperature of the component. In this International Standard only the part of the absorbed power/energy that is converted into heat is measured. If enough energy is absorbed, the optical properties of the component may be changed, and the component may even be destroyed. Absorptance is the ratio of the radiant flux absorbed to the radiant flux of the incident radiation.

In the procedures described in this International Standard, the absorptance is determined calorimetrically as the ratio of power or energy absorbed by the component to the total power or energy, respectively, impinging upon the component. The assumption is made that the absorptance of the test sample is constant within the temperature fluctuations experienced by the component during the measurement and is independent of both the position of the irradiating beam on the sample surface and the power density of the impinging radiation.

For several bulk materials like CdTe, the absorptance depends on the position of the irradiating beam on the sample surface. Several infrared materials exhibit a strong dependence of absorptance on temperature, especially at high temperatures.

INTERNATIONAL STANDARD **ISO 11551:2003(E)**

Optics and optical instruments — Lasers and laser-related equipment — Test method for absorptance of optical laser components

1 Scope

This International Standard specifies procedures and techniques for obtaining comparable values for the absorptance of optical laser components.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-6:1992, *Quantities and units — Part 6: Light and related electromagnetic radiations*

ISO 11145:2001, *Optics and optical instruments — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 11145 and ISO 31-6 and the following apply.

3.1
absorptance

α

ratio of the radiant flux absorbed to the radiant flux of the incident radiation

NOTE The definition of absorptance used for this international Standard is limited to absorptance processes which convert the absorbed energy into heat. For certain types of optics and radiation, additional non-thermal processes may result in absorption losses which will not be detected by the test procedure described here (see Annex A).

4 Symbols and units of measure

Table 1 — Symbols and units of measure

Symbol	Term	Unit
c_i	Thermal capacity of test sample, holder, etc.	J/(kg·K)
$d_{\sigma x}$, $d_{\sigma y}$	Beam width on test sample	mm
m_i	Mass of test sample, holder, etc.	kg
P	cw power	W
P_{av}	Average laser power for continuous pulse mode operation	W
P_{pk}	Typical peak power for repetitive pulse mode operation	W
t_{B}	Duration of irradiation	s
Δt	Time interval	s
T_{amb}	Ambient temperature	K
ΔT	Temperature difference	K
α	Absorptance	1
β	Angle of incidence	Rad
γ	Thermal loss coefficient	1/s
λ	Wavelength	nm

5 Preparation of test sample and measuring arrangement

Storage, cleaning and the preparation of the test samples are carried out in accordance with the manufacturer’s instructions for normal use.

The environment of the testing place consists of dust-free filtered air with less than 50 % relative humidity. The residual dust is reduced in accordance with cleanroom class 7 as defined in ISO 14644-1:1999. In this connection, an environment free from draughts is very important in order to keep thermal disturbances and heat loss by convection as small as possible. Measurements in ambient atmosphere and vacuum may have different influences on the measured absorptance.

A laser shall be used as the radiation source. To keep errors as low as possible, the laser power chosen for measurements is as high as possible but without causing any deterioration to the component.

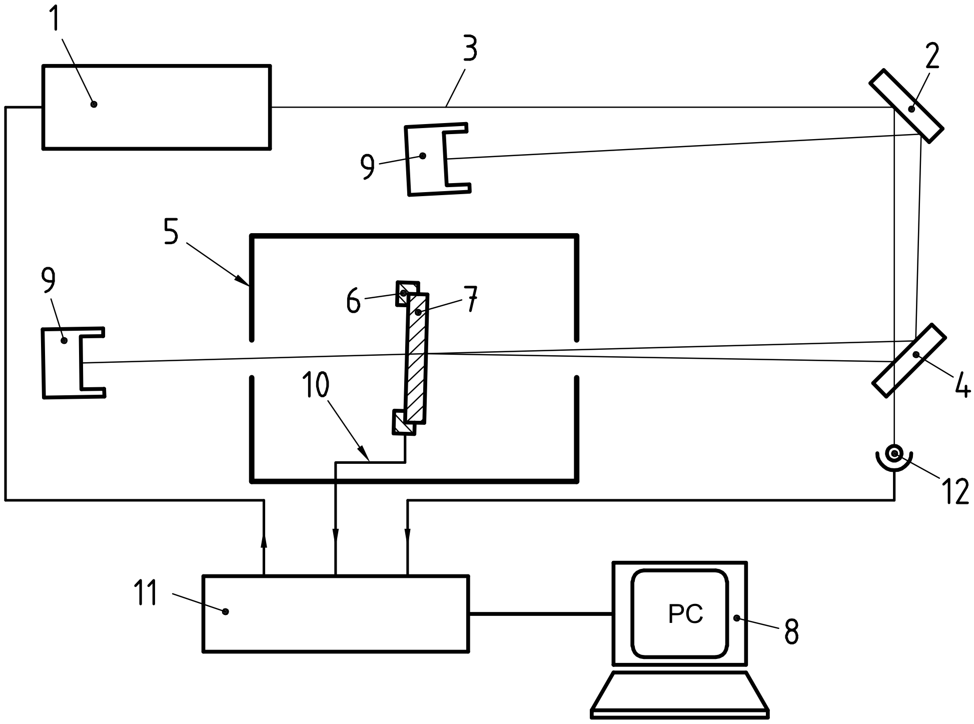
Wavelength, angle of incidence and state of polarization of the laser radiation used for the measurement shall correspond to the values specified by the manufacturer for the use of the test sample. If ranges are accepted for these three quantities, any combination of wavelength, angle of incidence and state of polarization may be chosen from those ranges.

The sample is mounted in a suitable holder. The thermal sensors are either connected directly to the sample surface, or attached to the sample holder. Good thermal contact between sensor and sample or between holder and sample shall be achieved. Precaution shall be taken to avoid a possible drop in thermal conductance between temperature sensor and test sample.

In order to increase the precision of the measurements, the sample should be mounted inside a chamber designed for thermal shielding, with apertures for the laser beam. Special attention shall be given to ensure that the temperature measurement itself does not cause a change of the sample temperature.

Suitable diaphragms should be placed in the beam path in front of and behind the test sample to ensure that only the test sample is irradiated by the measuring beam and that reflected or stray radiation will not strike the holder or the chamber walls. The number of transmissive optics employed for beam guiding should be minimized in order to reduce possible distortions by multi-reflections or scattered radiation. The transmitted and reflected partial beams shall be directed on to beam dumps with minimized back scatter.

Figure 1 shows a schematic measuring arrangement. The curved folding mirror M1 is recommended for imaging the laser output window on to the sample in order to avoid diffracted radiation influencing the measurement.



Key

1	laser	7	test sample
2	mirror M1	8	personal computer
3	optical axis	9	beam stop
4	mirror M2	10	thermal sensor
5	test chamber	11	control unit
6	sample holder	12	power detector

Figure 1 — Typical arrangement for measurement of the absorptance

6 Characteristic features of the laser radiation

The following physical quantities are needed for characterizing the laser radiation used for the test:

— wavelength λ;

— angle of incidence β;

— state and degree of polarization;

— beam widths on the test sample $d_{\sigma x}$, $d_{\sigma y}$;

- average power P_{av} for cw or continuously pulsed lasers;
- typical peak power P_{pk} and pulse energy Q in the case of continuously pulsed lasers;
- duration of irradiation t_{B}.

7 Test procedure

7.1 General

The following auxiliary tests shall be performed on a regular basis and whenever the measuring arrangement has been altered.

7.2 Calibration

7.2.1 Calibration of the power signal

Calibrate the power signal by placing a calibrated laser power detector at the location of the test components and, in order to obtain correct calibration, compare the measured laser power to the signal of the power monitor used during absorptance tests.

7.2.2 Calibration of the temperature signal

Calibrate the temperature signal by fixing a test sample, to which a calibrated thermal sensor is attached, to the sample holder. Compare the temperature signals of this calibrated detector and the sensors used during absorptance tests while varying the ambient temperature slowly over a range of a few kelvins at the typical test temperature.

7.2.3 Calibration of the thermal response

For certain types of sample materials and geometries, the temperature rise induced by the absorbed heat may differ from the theoretical response expected for ideal materials having infinite thermal conductivity. In these cases, a correction factor f_{c} shall be determined, which compensates for the influence of such phenomena on the absorptance test result. f_{c} is unity if the influence of limited thermal conductivity can be neglected.

For calibration, a reference sample of known absorptance, which is similar to the samples under investigation with respect to substrate geometry, heat capacity and thermal conductivity, is tested for absorptance as described below. The irradiation time and evaluation method used for calibration shall be the same as for other typical absorptance tests for which the calibration shall be valid.

Depending on the evaluation method used for the absorptance test, the correction coefficient can be calculated by substituting the value of the known calibration sample absorptance for α in equations 2 (see 8.3) or 5 (see 8.4), and solving for f_{c}.

NOTE 1 A known absorptance can be achieved by applying a thin, high-absorbing coating to the sample surface area that is exposed to irradiation. High absorptance values can be determined with sufficient accuracy, e.g. by measuring the fraction of transmitted, reflected and scattered radiation. For absorptance testing of samples with high absorptance values, the laser power should be suitably attenuated in order to avoid damage to the samples and to ensure that the resulting temperature rise is in the same order of magnitude as the temperature which is achieved for typical measurements.

NOTE 2 An alternative to irradiating a calibration sample of known absorptance with the laser beam, the thermal energy may be deposited electrically in the test sample by attaching an electric resistor to the tested surface. The absorbed power is given by RI^2, where R is the electrical resistance and I is the electric current during “irradiation”. Care should be taken to ensure good thermal contact between resistor and sample. Furthermore, especially in the case of samples with low thermal conductivity, the area of the resistor should match the area irradiated by the laser beam under normal test conditions.

7.2.4 Measurement of the background signal

For maximum accuracy and suppression of possible signal distortions, the imaging and alignment of the laser beam shall be optimized. A measurement with an empty holder or with an absorptance-free component can be used to verify that the measuring arrangement is not influenced by reflected or stray radiation. The amplitude of the temperature fluctuations during the test interval shall be at least one order of magnitude below the temperature rise occurring during an absorptance test.

7.3 Determining the absorptance

The absorptance of optical components is determined calorimetrically by means of a measuring arrangement as shown in Figure 1. Before measurement commences, thermal equilibrium shall be established, so that non-linear temperature drift and temperature noise are at least one order of magnitude below the maximum temperature rise induced by irradiation. The maximum temperature rise during the test should not exceed a few kelvins.

If the absorptance is dependent either on the power or energy density of the impinging radiation, or the temperature of the test sample, this shall be noted in the test report. The test shall be performed under the conditions of the foreseen use of the components.

The test is performed in three successive intervals:

- the pre-irradiation interval $[t_0, t_1]$ (at least 30 s);
- the heating interval $[t_1, t_2]$ ($t_B = t_2 - t_1$ = 5 s to 300 s) during which the laser beam is switched on and impinges on the test sample surface;
- the cooling interval of at least 200 s.

For test samples with high thermal losses, the irradiation should end significantly before the temperature rise saturates due to a balance of absorbed power and thermal loss.

During the test, the sample temperature signal $T(t)$ and the laser power signal $P(t)$ are recorded. The resulting calorimetric data sets $[t_k, T(t_k)]$ and $[t_k, P(t_k)]$ with k, enumeration index, are stored for the evaluation of the absorptance.

8 Evaluation

8.1 General

The mass, m_i, of components heated during irradiation (test sample, holder, etc.) is determined by weighing. Thermal capacities are taken from tables.

For the calculation of absorptance, two alternative methods can be used: the exponential method or the pulse method. In general, the pulse method is applicable for irradiation times up to 120 s, while the exponential method can be applied for irradiation times from 60 s. Which method is preferable depends also on the individual properties of the tested specimen and the circumstances of the test. In many cases it can be helpful to apply both methods and gain additional information on the uncertainty in the result by comparing the two results.

8.2 Elimination of drift

Preceding the calculation of absorptance, the calorimetric data shall be analysed with respect to the possible occurrence of temperature drift phenomena. In the presence of temperature drift, a linear fit shall be performed to the temperature data sampled before irradiation. The approximated drift influence described by the resulting linear fit function is eliminated by subtracting the extrapolated fit values from the raw temperature data recorded during the test.

8.3 Exponential method

Determine the parameters A_h, B_h, γ_h by fitting an exponential function to the temperature data [t_k, $T(t_k)$] recorded during the irradiation interval (heating period of the component):

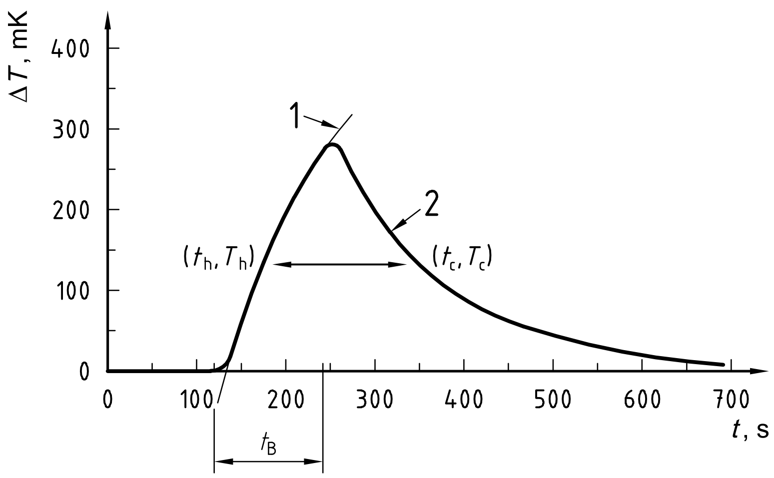
$$\sum\left[T(t_k)-A_\mathrm{h}-B_\mathrm{h}\exp\left(-\gamma_\mathrm{h}t_k\right)\right]^2 = \min \tag{1}$$

NOTE γ_h corresponds to the thermal loss coefficient of the heated system, which describes the cooling process in absence of heat sources as $\dot{T}=-\gamma(T-T_\mathrm{amb})$. If γ is known from earlier measurements or other sources, the fitting procedure can be simplified by fixing γ_h at this value.

Calculate the absorptance from these fit parameters as

$$\alpha=\frac{f_\mathrm{c}\gamma_\mathrm{h}A_\mathrm{h}\sum\limits_i m_i c_{\mathrm{p}i}}{P} \tag{2}$$

In case of pulsed laser operation, substitute P by P_av. See Figure 2.



Key

1 fit

2 relative temperature

Figure 2 — Example for exponential method evaluation

8.4 Pulse method

The temperature decay curve after irradiation shall be extrapolated graphically to the time $(t_1 + t_\mathrm{B})/2$ (middle of irradiation interval), measuring the extrapolation temperature T_ext. It is recommended to carry out this extrapolation by calculating the best fit parameters A_c, B_c, γ_c, performed on the temperature data with negative slope recorded after irradiation:

$$\sum\left[T(t_k)-A_\mathrm{c}-B_\mathrm{c}\exp\left(-\gamma_\mathrm{c}t_k\right)\right]^2 = \min \tag{3}$$

and to calculate the extrapolation temperature T_ext as

$$T_\mathrm{ext}=A_\mathrm{c}+B_\mathrm{c}\exp\left(-\gamma_\mathrm{c}\frac{t_1+t_2}{2}\right) \tag{4}$$

The absorptance is calculated as

$$\alpha = \frac{f_\mathrm{c}\, T_\mathrm{ext} \sum_i m_i c_{\mathrm{p}i}}{P t_\mathrm{B}}\; 0{,}5\gamma_\mathrm{c} t_\mathrm{B} \,/\, \sinh(\gamma_\mathrm{c} t_\mathrm{B}/2) \qquad (5)$$

In case of pulsed laser operation, substitute P with P_av. See Figure 3.

$$0{,}5\gamma_c t_B/\sinh(\gamma_c t_B/2)$$

NOTE The term $0{,}5\gamma_\mathrm{c} t_\mathrm{B}/\sinh(\gamma_\mathrm{c} t_\mathrm{B}/2)$ can be approximately unity, if $\gamma_\mathrm{c} t_\mathrm{B} < 0{,}5$ is valid.

If $0{,}5 \leqslant \gamma_\mathrm{c} t_\mathrm{B} \leqslant 2$ the term $0{,}5\gamma_\mathrm{c} t_\mathrm{B}/\sinh(\gamma_\mathrm{c} t_\mathrm{B}/2)$ can be approximated by $\left(1-\gamma_\mathrm{c}^2 t_\mathrm{B}^2/24\right)$.

For $\gamma_\mathrm{c} t_\mathrm{B} > 2$ Equation (5) is applicable.

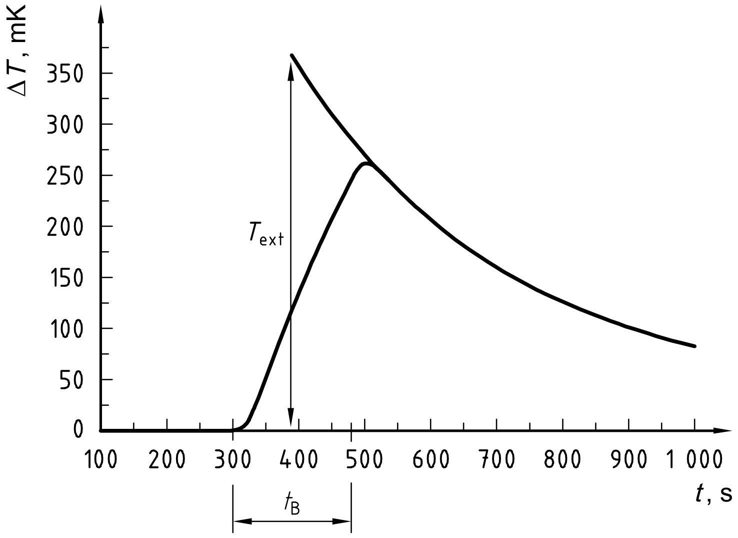


Figure 3 — Example for pulse method evaluation

9 Test report

The test report shall contain the following information.

a) General information

1) test has been performed in accordance with ISO 11551:2003;

2) date of test;

3) name and address of test organization;

4) name of individual performing the test.

b) Information concerning the test sample (if available)

1) type of test sample;

2) manufacturer of test sample;

3) part identification, date of production;

   4) specifications by the manufacturer concerning storage, cleaning, etc.;
   5) specifications by the manufacturer for normal use.

c) Information concerning the test facility
   1) beam source;
      - type of beam source,
      - manufacturer,
      - manufacturer’s model designation;
   2) description of other relevant test equipment.

d) Test conditions
   1) wavelength;
   2) operating mode cw/pulsed;
   3) source parameter settings;
      - output power or energy (irradiation time),
      - pulse energy,
      - pulse duration,
      - pulse repetition rate;
   4) spatial and/or temporal beam profile, if necessary;
   5) beam width on test sample;
   6) angle of incidence;
   7) polarization;
   8) environmental conditions;
   9) cleaning;
   10) method of mounting of optical component.

e) Information concerning testing and evaluation
   1) test method used;
   2) surface quality/imperfections/contamination.

f) Comments concerning effects of changing absorptance, if relevant.

g) Error budget.

h) Test result(s).

Annex A
(informative)

Effects changing absorptance

A.1 General

The absorptance measured according to the methods given in this International Standard is, in certain cases, dependent on the parameters of the measurement (power, power density, irradiation time, etc.). Furthermore, absorptance may be influenced by ageing of the specimen and nonlinear behaviour. These effects must be taken into consideration, especially for measurements with UV radiation. The following mechanisms may contribute to the measured absorptance:

A.2 Colour centre formation

Especially in the UV spectral range, colour centres can be formed by irradiation. In this case the absorptance increases with time and a saturation may occur after irradiation with a certain radiation dose. Some colour centres can be annealed by heating the component resulting in a reduction of absorptance.

In the case of coated optics, high power irradiation may cause structural changes in optical coatings (e.g. post-oxidation). This can lead either to an increase or to a decrease of absorptance.

Thus, a single measurement reflects the absorptance only at the time the measurement is performed. For a detailed analysis of the data, the history of the sample (parameters of former irradiation cycles etc.) must be known.

A.3 Non linear absorption

In many samples, absorptance increases reversibly with increasing power density due to multi-photon absorption. The change depends on the wavelength of the radiation and may be large especially for UV radiation. For ultra-short pulses it may also influence absorptance in the VIS and NIR spectral range. For the power densities of practical use, only two-photon absorption is of importance, leading to a linear increase of absorptance with increasing power density.

If nonlinear absorption is assumed to significantly influence the absorptance of a sample, absorptance should be measured for a variety of power densities. The obtained data are fitted linearly as a function of the power density. The linear absorptance is given by the intercept with ordinate, the nonlinear absorption is given by the slope of the linear fit.

A.4 Further parameters influencing the measured absorptance value

Additional effects must be considered, which may change the measured absorptance value of the specimen:

- high power irradiation may cause cleaning effects on the surface of optical components, reducing the total absorptance;
- high power irradiation may cause degradation or ageing of optical components and optical coatings; in this case the absorptance may increase with time and with power density;
- cleaning procedures that are not appropriate for the specimen may cause an increase of absorptance.

Annex B
(informative)

Influence of signal distortions

B.1 Influence of the resolution of temperature measurement

It is important to be sure that the temperature rise is significantly higher than the resolution of the temperature measurement.

B.2 Influence of scattered radiation

Scattered radiation emerging from the component itself, or the coating, may hit the detector. Note that this kind of scattered radiation must be distinguished from scattered radiation coming from the beam-forming components. In particular, this is important for the UV range.

B.3 Influence of low thermal conductivity in the specimen

The standard evaluation methods given for the determination of the absorptance are based on a fundamental physical model, assuming a small temperature increase and a homogeneous temperature T_s of sample and holder at all times. For such a system, the temperature dynamics are described by the differential equation

$$\frac{\mathrm{d}T}{\mathrm{d}t} = \frac{\alpha P_\mathrm{av}}{C_\mathrm{eff}} - \gamma T \qquad \text{(B.1)}$$

which renders the following exponential functions as solutions for the temperature signal $T(t)$:

$$T(t) = T(t_1) + \frac{C_\mathrm{eff}}{\gamma \alpha P}\left\{1 - \exp\left[-\gamma\left(t - t_1\right)\right]\right\} \text{ during irradiation and} \qquad \text{(B.2)}$$

$$T(t) = T(t_2) + \frac{C_\mathrm{eff}}{\gamma \alpha P}\left\{1 - \exp\left[-\gamma\left(t - t_2\right)\right]\right\} \text{ after irradiation.} \qquad \text{(B.3)}$$

If laser components are constituted of materials with low thermal conductivities, the temperature dynamics during an absorptance measurement cannot be accurately described by this simple model of homogeneous temperature distribution. Instead, the finite sample thermal conductivity must be taken into account by solving a three-dimensional heat equation of the form

$$\nabla(\kappa / c_\mathrm{p} \nabla T) + Q(x, y, z) / c_\mathrm{p} = \eta \dot{T} \qquad \text{(B.4)}$$

where

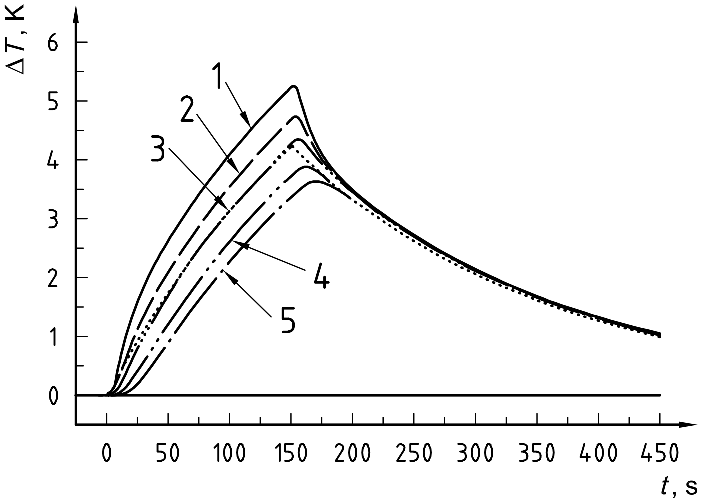
η is the mass density, in grams per cubic centimetre;

κ is the thermal conductivity, in watts per kelvin centimetre;

Q is the heat source, in watts per cubic centimetre.

The boundary conditions at the sample surfaces are determined by the thermal convection and radiation losses and by the thermal coupling to the holder and thermal sensors. Below is an example shall be given to illustrate the effects of limited thermal conductivity on laser calorimetry.

For a thin cylindrical test sample (absorptance: 1, thickness 3 mm, diameter 25 mm), the temperature signals have been simulated for a range of thermal diffusivities and for different radial locations of the thermal sensors. The beam diameter was assumed to be much smaller than the diameter of the sample. Figure B.1 shows the calculated temperature behaviour for one thermal diffusivity and for different radial locations of the temperature sensor. For comparison, the temperature curve for infinite thermal conductivity is included in the plot (0D model).



Key

1 r = 6 mm
2 r = 7 mm
3 0D model
4 r = 9 mm
5 r = 12 mm

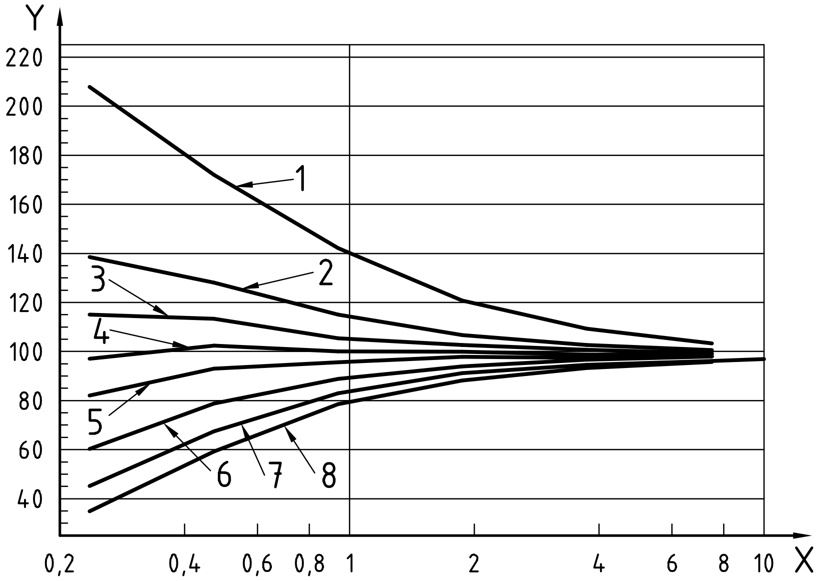
Figure B.1 — Dependence of temperature signal and thermal sensor location

It can be seen, that with increasing distance of the temperature sensor from the laser beam (the sample centre), the temperature rise is delayed. Also the maximum temperature is smaller.

After irradiation, all curves converge.

Obviously, there is an optimum location of the thermal sensor with respect to the best similarity between theoretical curves regarding thermal conductivity and the infinite thermal conductivity model (for this example, approximately 7 mm.)

Further theoretical calculations for other ratios of thermal conductivity to specific heat capacity were performed. The absorptance values determined from the simulated data are plotted in Figure B.2. The absorptance results are given relative to the theoretical absorptance value for infinite thermal conductivity.



Key

X κ, W(m·K)

Y deviation of measurement, %

1 detector position 5 mm
2 detector position 6 mm
3 detector position 6,5 mm
4 detector position 7 mm
5 detector position 7,5 mm
6 detector position 8,5 mm
7 detector position 10 mm
8 detector position 12,5 mm

Figure B.2 — Relationship between theoretical absorptance results, thermal diffusivity and thermal sensor position on cylindrical sample

From Figure B.2 it is evident that the influence of the temperature sensor position increases with decreasing thermal diffusivity. While for many typical optical materials in FIR applications (e.g. copper, aluminium, germanium, ZnSe) the influence can be neglected, it has to be considered for glass-like materials. By selecting a suitable location for the temperature detectors (e.g. ca. 7 mm for ∅ 25 mm optics), the influence of the thermal conductivity on an absorptance measurement can be widely reduced. With the performance of calibrations as described in Clause 7, possible errors induced by the effects of low thermal conductivity can be further suppressed.

Annex C
(informative)

Algorithm for parameterizing the temperature data

If fitting parameters A, B, $C = 1/\gamma$ have to be found for describing a range of data points $t_k, T(t_k)$ (with k counting variable and $t_{k+1} > t_k$) as an exponential function of the type

$$T(t_k) \approx A + B\exp\left(-t_k / C\right),\ k_1 \leqslant k \leqslant k_2, \tag{C.1}$$

the following approach is recommended, which reduces this problem of non-linear fitting to two simple linear regressions:

1) Calculate the derivative δT of the data set

$$\delta T_k = \frac{T(t_{k+1}) - T(t_{k-1})}{t_{k+1} - t_{k-1}},\ k_1 < k < k_2 \tag{C.2}$$

2) Calculate C (reciprocal loss coefficient) by minimizing the sum (linear regression by variation of A, C):

$$\sum\nolimits_{k_1<k<k_2} \left[T(t_k) - A + C\delta T_k\right]^2 \tag{C.3}$$

3) Calculate A, B by minimizing the sum (linear regression by variation of A, B):

$$\sum\nolimits_{k_1<k<k_2} \left[T\left(t_k\right) - A + B\exp\left(t_k / C\right)\right]^2 \tag{C.4}$$

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