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Optics and photonics — Optical materials and components — Test method for homogeneity of infrared optical materials

Optique et photonique — Matériaux et composants optiques — Méthodes d'essai pour déterminer l'homogénéité des matériaux optiques infrarouges

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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This document was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 3, *Optical materials and components*.

Optics and photonics — Optical materials and components — Test method for homogeneity of infrared optical materials

1 Scope

This document specifies the principle, apparatus, condition, sample, procedure and data processing of measuring homogeneity of infrared optical materials.

It is applicable to the determination of homogeneity of infrared optical materials, such as infrared optical glass, infrared crystals and infrared ceramics, which are opaque to visible wavelengths and whose transmission optical spectra are beyond 0,78 µm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 10110-7, *Optics and photonics — Preparation of drawings for optical elements and systems — Part 7: Surface imperfections*

ISO 10110-8, *Optics and photonics — Preparation of drawings for optical elements and systems — Part 8: Surface texture; roughness and waviness*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
peak-to-valley value of refractive index
PV value
Δn_{PV}
difference between the maximum and the minimum values of refractive index distribution of an infrared optical material in its cross-sectional area of the definition area

3.2
standard deviation value of refractive index
Δn_{STD}
value which is expressed with the square root of the sum of the squares of the differences of both the distribution values and the average value of refractive index of an infrared optical material divided by the sampling number of the distribution

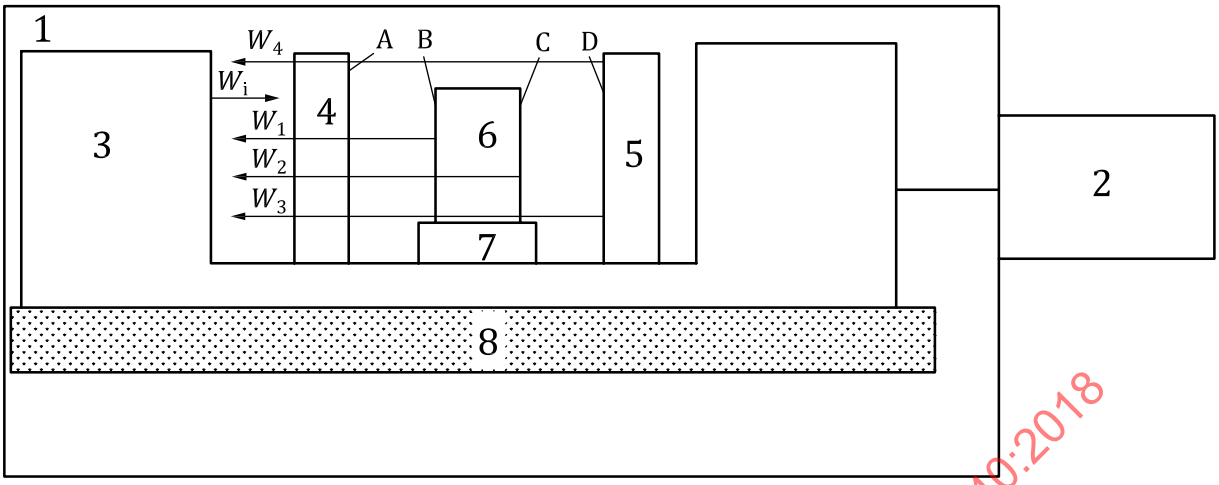
3.3
homogeneity
gradual variation of the refractive index distribution within an optical element in the prescribed direction (mostly perpendicular to optical path) and within the prescribed cross-section

3.4
homogeneity value
level of inconsistency of the refractive index distribution of an infrared optical material in the prescribed direction (mostly perpendicular to optical path) and within the prescribed cross-sectional area and per unit thickness of infrared optical materials, which is expressed in the PV value and the *standard deviation value of refractive index* (3.2)

4 Principle

For a wedge-shaped sample, measure with a digitalized infrared interferometer the wavefront interferograms of the front surface and the rear surface of the sample. Then measure the interferogram of the wavefront going forth and back through the sample in a double pass configuration. Repeat the double pass measurement without the sample in the measurement. Calculate the PV value and the standard deviation value of infrared refractive index (simply called four-step method).

For a plane parallel plate sample, separately measure the wavefront interferograms in a double pass configuration with and without the sample in the test cavity. Calculate the PV value and the standard deviation value of infrared refractive index (simply called two-step method). The schematic diagram of the measurement of homogeneity is shown in Figure 1.



Key

W_i wavefront of the incident light, in μm

W_1 wavefront error of interferogram resulting from interference between the wavefront reflected by the flat surface A and the wavefront reflected by the front surface B of the sample, in μm

W_2 wavefront error of interferogram resulting from interference between the wavefront reflected by the flat surface A and the wavefront reflected by the rear surface C of the sample, in μm

W_3 wavefront error of interferogram resulting from interference between the wavefront reflected by the flat surface A and the wavefront transmitted through the sample and reflected back from standard mirror D and then transmitted through the sample again, in μm

W_4 wavefront error of interferogram resulting from interference between the wavefront reflected by the flat surface A and the wavefront reflected back from standard mirror D in absence of the sample (so-called empty cavity measurement), in μm

1 thermostatic chamber

2 interferogram analysis device

3 infrared interferometer (including an image sensor)

4 infrared reference flat

5 standard mirror

6 sample

7 precise adjustment stage

8 vibration isolation platform

Figure 1 — Schematic diagram of the measurement of infrared material homogeneity

5 Apparatus

5.1 Apparatus arrangement

The measurement apparatus, which consists of an infrared interferometer, an infrared reference flat, precision adjustment stage, a standard mirror and an interferogram analysis device to collect data, to process data and to display data are arranged according to Figure 1. The apparatus shall be placed onto a vibration isolation platform. Examples of infrared interferometers are given in Annex A. The infrared interferometers may include various kinds of phase-shifting methods which can guarantee measurement accuracy, such as piezoelectrical ceramic phase-shifting or Fourier transform phase-shifting.

5.2 Optical module of interferometer

The radiation spectrum of interferometer should be within the spectral range of transmission of optical materials. Generally, this should be a DL (Diode Laser) light source with an output wavelength of 1,55 μm, a He-Ne laser light source with an output wavelength of 3,39 μm, a CO_2 laser light source with an output wavelength of 10,6 μm or QCLs (Quantum Cascade Lasers). The aperture of the output beam should be more than that of the sample. Alternatively use additional optics to change the diameter of the output optical beam or employ a sufficient aperture stitching algorithm for sub-aperture measurements.

5.3 Reference flat

The surface formation deviation should not be more than 0,05 λ (λ = 0,633 μm) and its aperture should generally be more than that of the sample.

5.4 Standard mirror

The surface formation deviation should not be more than 0,05 λ (λ = 0,633 μm) and its aperture should be more than that of the sample.

5.5 Image sensor

The working wavelength band of an infrared image sensor should be within the transmission spectrum of the sample measurement. The corresponding object spatial resolution of the image sensor should be high enough to guarantee the measurement accuracy.

5.6 Computer data collecting, processing and displaying system

The computer data collecting, processing and displaying system should have a software that is able to collect the interferograms of the measurement wavefronts and calculate the PV value and the standard deviation value of refractive index.

5.7 Thickness measurement equipment

The thickness measurement equipment shall have an uncertainty not more than 0,01 mm.

6 Test conditions

6.1 Temperature

The environmental temperature of the measurement should be 22 °C ± 5 °C, with the temperature tolerance being not more than ±0,2 °C. See Annex B.

6.2 Relative humidity

The environmental relative humidity of the measurement should not be more than 70 %.

6.3 Vibration isolation

The vibration isolation device to be used shall be capable of eliminating the effect of the vibration from the outside to the interferometer and the sample. It should be provided for performing high accuracy measurements. It is recommended as the technical parameters of the vibration isolation device that the vibration isolation frequency of the device should be less than 1,2 Hz –2,0 Hz in the horizontal direction, 1,2 Hz –2,0 Hz in the vertical direction and its amplitude should be less than 1,2 μm.

6.4 Airflow

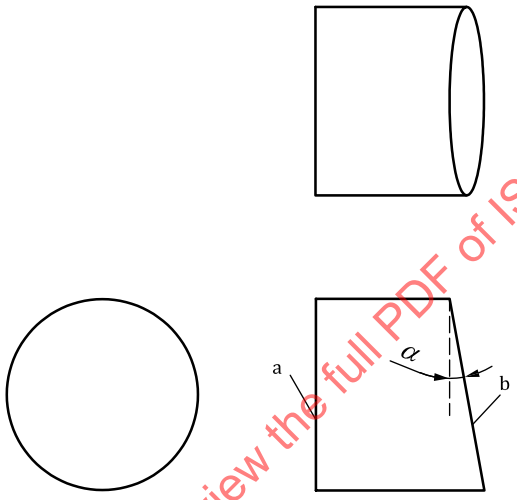
The airflow velocity of the test environment should not affect the measurement precision.

7 Sample

7.1 Outline

7.1.1 Wedge shaped sample

The drawing of wedged sample can be seen in Figure 2.



Key

a surface B (transmission plane of the sample).

b surface C (transmission slope of the sample).

α wedge angle of the sample

Figure 2 — Drawing of the wedge shaped sample

7.1.2 Parallel plane sample

The parallelism of both surfaces of the parallel plane sample shall not be more than 30″.

7.2 Thickness

The ratio of diameter and thickness of sample should not be generally more than 5:1. For example, the diameter is 50 mm and the thickness should not be less than 10 mm.

The thickness value t_i shall be measured at the minimum 3 points in different regions evenly distributed on the edge of the sample with thickness measurement equipment. The mean value t_0 of the thickness shall then be calculated. A thicker sample is better for the measurement precision. A thicker sample should amplify the homogeneity value of a sample.

7.3 Wedge angle

The wedge angle α shown in Figure 2 shall be as small as possible, but at the same time it shall be large enough to ensure that the light rays reflected from the rear surface of the wedge shaped sample do not

enter the observation field of view of the interferometer. The minimum wedge angle α_{min} is calculated by Formula (1):

$$\alpha_{min} = \frac{1}{2}\sin^{-1}\left(\frac{\sin\theta}{n_0}\right) \quad (1)$$

where

α_{min} is the minimum wedge angle of the sample;

θ is half the fieldangle of view of interferometer;

n_0 is the nominal refractive index of wedge shaped sample.

NOTE In the case of the four-step interferometry, the wedge angle of the sample in general can be 10′ or so. The nominal refractive index n_0 of wedge shaped sample is available by product manual.

7.4 Polished surfaces

7.4.1 Wedge shaped sample

To prevent the measurement from being affected by the surface defects and deformation of the sample, both surface B (transmission plane) and surface C (transmission slope) of the sample shall be polished to the flatness specified in Twice (with surface error mutual offsetting) in Table C.2 of Annex C and their surface roughness R_q specified in ISO 10110-8 less than 0,005 µm. The surface imperfection shall be better than 5/5 × 0,40 L5 × 0,006 (ISO 10110-7).

7.4.2 Parallel plane sample

To prevent the measurement from being affected by the surface defects and deformation of the sample, both surface B (transmission plane) and surface C (transmission slope) of the sample shall be polished to the flatness specified for Once in Table C.2 of Annex C and their surface roughness R_q specified in ISO 10110-8 less than 0,005 µm. The surface imperfection shall be better than 5/5 × 0,40 L5 × 0,006 (ISO 10110-7).

8 Procedure

8.1 Four-step method

8.1.1 Clean the surface of the sample.

8.1.2 Soak the sample thermally within the measurement environment for a period that allows it to achieve a thermal stability of ±0,2 °C. See Annex B.

8.1.3 Turn on the power of the digitalized interferometer and preheat until interferometer is thermally stabilized.

8.1.4 Adjust the standard mirror so that the reflected wavefront of surface A of the reference flat coincide with the reflected wavefront of the standard mirror to form an equal optical path interference. Then collect the wavefront error of interferogram W_4.

8.1.5 Place the sample on the precision adjustment stage and adjust the standard mirror so that the ray transmitted through the sample returns along its original incident light path after being reflected by the standard mirror. Then collect the wavefront error of interferogram W_3.

8.1.6 Cover the surface D of the standard mirror with a plate opaque to infrared radiation. Adjust the sample so that surface B is parallel with surface A of the reference flat. Collect the wavefront error of interferogram W_1.

8.1.7 Keep on covering surface D of the standard mirror and adjust the sample so that the sample's rear surface C is perpendicular to the refracted light of surface B. Collect the wavefront error of interferogram W_2.

8.1.8 Choose not less than three points in different places at the edges of the sample and measure with a thickness measurement equipment, record the data of thickness t_i and calculate the mean value t_0 of sample thickness.

8.2 Two-step method

8.2.1 Clean the surface of the sample.

8.2.2 Soak the sample thermally within the measurement environment for a period that allows it to achieve a thermal stability of ±0,2 °C. See Annex B.

8.2.3 Turn on the power of the digitalized interferometer and preheat until interferometer is thermally stabilized.

8.2.4 Adjust the standard mirror so that the reflected wavefront of surface A of the reference flat coincide with the reflected wavefront of the standard mirror to form an equal optical path interference. Then collect the wavefront error of interferogram W_4.

8.2.5 Place the sample on the precision adjustment stage and adjust the precision adjustment stage so that the reference flat is parallel with the transmission surface of the sample. Then collect the wavefront error of interferogram W_3.

8.2.6 Choose not less than three points in different places at the edges of the sample and measure with a thickness measurement equipment, record the data of thickness t_i and calculate the mean value t_0 of sample thickness.

9 Data processing

9.1 Calculation of refractive index error distribution with the four-step method

Calculate $\Delta n(x, y)$ of the refractive index distribution of the sample according to Formula (2) with the computer software.

$$\Delta n(x, y) = \frac{1}{2\times 10^3 t_0}[n_0(W_3 - W_4) - (n_0 - 1)(W_2 - W_1)] \quad (2)$$

where

$\Delta n(x, y)$ is the deviation of refractive index distribution;

t_0 is the arithmetic mean of the thickness of the sample, in mm;

n_0 is the nominal value of the refractive index of the sample at the working wavelength.

9.2 Calculation of refractive index error distribution with the two-step method

Calculate $\Delta n(x, y)$ of the refractive index distribution of the sample with double pass interferometers such as Fizeau and Twyman-Green interferometers according to Formula (3) with the computer software.

$$\Delta n(x, y) = \frac{n_0}{2\times 10^3 t_0}(W_3 - W_4) \tag{3}$$

Calculate $\Delta n(x, y)$ of the refractive index distribution of the sample with single pass interferometers such as the Mach Zehnder interferometer according to Formula (4) with the computer software.

$$\Delta n(x, y) = \frac{n_0}{1\times 10^3 t_0}(W_3 - W_4) \tag{4}$$

9.3 Calculation of the PV value and the standard deviation value of refractive index

Calculate the PV value and the standard deviation value of the refractive index distribution of the sample respectively according to Formula (5) and Formula (6).

$$\Delta n_{PV} = Max[\Delta n(x, y)] - Min[\Delta n(x, y)] \tag{5}$$

$$\Delta n_{STD} = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\left[\Delta n(x, y) - \overline{\Delta n(x, y)}\right]^2} \tag{6}$$

where

Δn_{PV} is the maximum deviation of the refractive index distribution of the sample;

Δn_{STD} is the standard deviation of the refractive index distribution of the sample;

N is the number of sampling;

i is the serial number of measurement;

$\overline{\Delta n(x, y)}$ is deviation mean value of the refractive index distribution.

10 Calculation of measurement value and measurement uncertainty

10.1 Calculation of measurement value

Measure a sample m times according to the procedure of Clause 8 and respectively calculate the means of the PV values and the standard deviation values of refractive index of a sample according to Formula (7) and Formula (8).

$$\overline{\Delta n_{PV}} = \left(\sum_{i=1}^{m}\Delta n_{PVi}\right)\times 1/m \tag{7}$$

$$\overline{\Delta n_{STD}} = \left(\sum_{i=1}^{m}\Delta n_{STDi}\right)\times 1/m \tag{8}$$

where

m is the number of measurement times;

$\overline{\Delta n_{PV}}$ is the mean of measuring Δn_{PV} for m times;

Δn_{PVi} is the value of Δn_{PV} for number i of the measurement;

$\overline{\Delta n_{STD}}$ is the mean of measuring Δn_{STD} for m times;

Δn_{STDi} is the value of Δn_{STD} for number i of the measurement.

10.2 Calculation of measurement uncertainty

Calculate the uncertainty of $\overline{\Delta n_{PV}}$ and $\overline{\Delta n_{STD}}$ according to Formula (9) and Formula (10), respectively.

$$u_{PV} = \sqrt{\frac{\sum_{i=1}^{m}\left(\Delta n_{PVi} - \overline{\Delta n_{PV}}\right)^2}{m(m-1)}} \tag{9}$$

$$u_{STD} = \sqrt{\frac{\sum_{i=1}^{m}\left(\Delta n_{STDi} - \overline{\Delta n_{STD}}\right)^2}{m(m-1)}} \tag{10}$$

where

u_{PV} is the uncertainty of the mean of Δn_{PV};

u_{STD} is the uncertainty of the mean of Δn_{STD}.

10.3 Frequency of measuring sample

In order to achieve a better measurement result, the measurements should be repeated at least 3 times. Measurements more than 5 times can improve measurement results and reduce their uncertainties a little, but would greatly increase measurement cost.

11 Expanded uncertainty of the measurement

The absolute expanded uncertainty of this method is 2×10^{-6} to 2×10^{-5}.

12 Test Report

The test report shall include at least the following (see Annex D):

a) laboratory name and contact information;

b) test method and instrument;

c) test wavelength;

d) client;

e) sample name and specification;

f) environmental temperature and humidity;

g) homogeneity value of the sample;

h) tester, reviewer and test date;

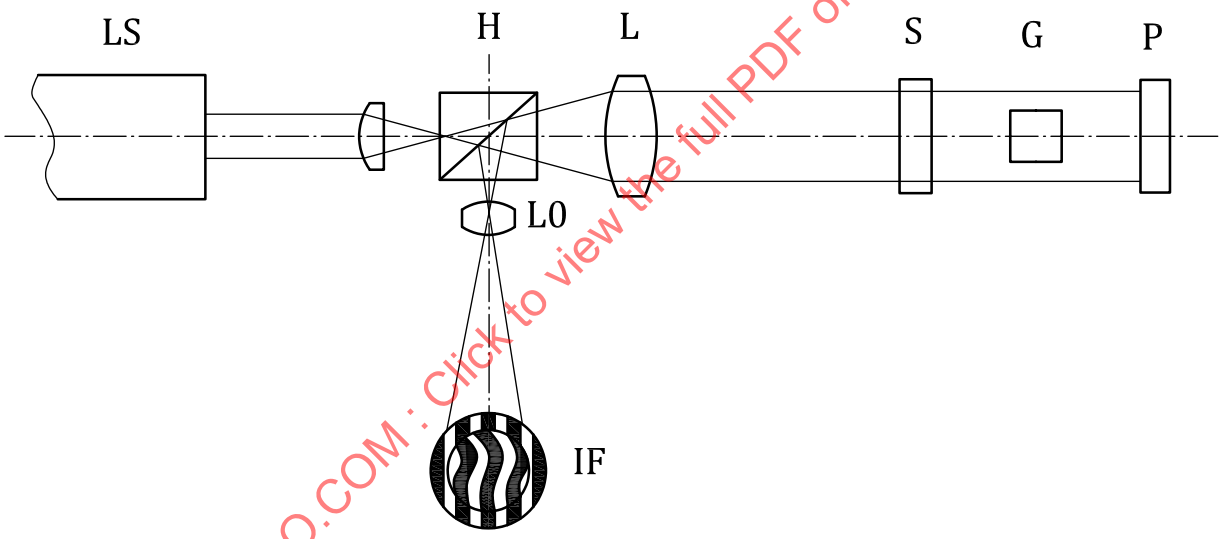
i) remarks.

Annex A
(informative)

Infrared interferometer

The infrared interferometer is a device that generates interference fringes by splitting the parallel rays with uniform wavefronts into two with a semi-transparent plane mirror (beam splitter), and after making each ray pass through difference paths, shifts the wavefronts slightly and then superimposes them again.

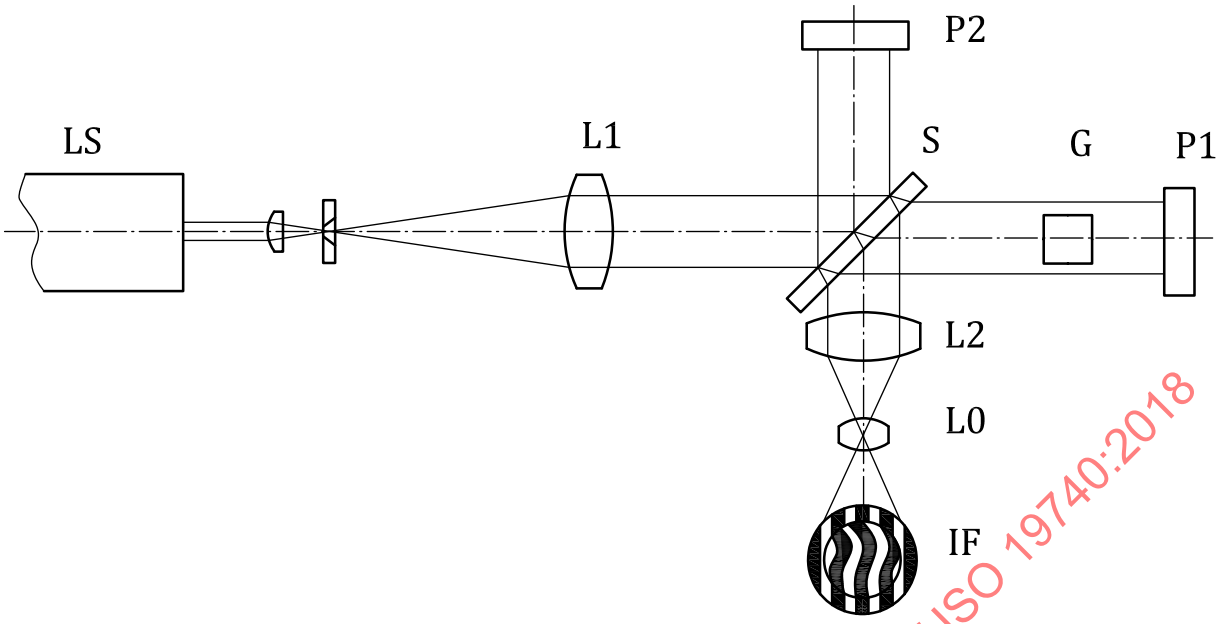
As examples of devices suitable for homogeneity measurement of infrared material sample, three types of interferometers are shown below. Figures A.1 and A.2 show the interferometers (including an image sensor and an interferogram analysis device to collect data, to process data and to display data in each one) of the type in which infrared radiation transmits through the sample twice, and Figure A.3 shows the interferometer (including an image sensor and an interferogram analysis device to collect data, to process data and to display data) of the type in which infrared radiation transmits through the sample once.



Key

G infrared optics sample
H beam splitter
L collimating lens
L0 imaging lens
LS infrared radiation source
P plane mirror
S infrared reference flat
IF interference fringes

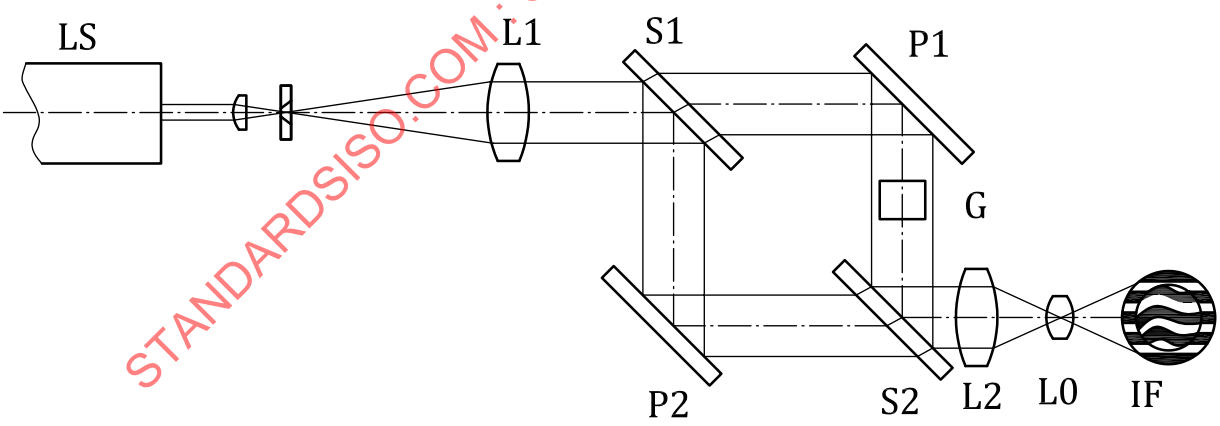
Figure A.1 — Fizeau interferometer



Key

G	infrared optics sample
L0	imaging lens
L1, L2	collimating lens
LS	infrared radiation source
P1, P2	plane mirror
S	beam splitter
IF	interference fringes

Figure A.2 — Twyman-Green interferometer



Key

G	infrared optics sample
L1, L2	collimating lens
L0	imaging lens
LS	infrared radiation source
P1, P2	plane mirror
S1, S2	beam splitter
IF	interference fringes

Figure A.3 — Mach-Zehnder interferometer

When an infrared optics sample is put into the path of one of the infrared radiation of these interferometers, infrared radiation travels through the sample, and then the wavefront irregularities (phase difference) are generated according to the difference of refractive indices within the infrared optics sample. When this radiation is superimposed on the other radiation of which the wavefront is uniform, the interferogram that shows the relative phase difference appears. The phase difference of infrared radiation is generated due to not only the inhomogeneity of the refractive index but also the poor flatness of a sample's end face.

A laser in an infrared wavelength band is the most efficient coherent light source in a single wavelength. By using this, it is possible to observe infrared radiation interference fringes of which the contrast is good, irrespective of the optical path difference.

Annex B
(informative)

Temperature stability for homogeneity measurements

Thermal gradients within the sample and within the optical components of the interferometer lead to wavefront distortion because of their refractive indices' dependence on temperature. In the wavefront registered by the interferometer it is not possible to separate the influences of thermal gradients from the wavefront distortion caused by the refractive index variation within the sample, where the quantity to be measured. Thermal gradients should be kept low by operating the interferometer in a temperature stabilized chamber and by giving the sample time enough to reach thermal equilibrium in the interferometer environment.

For high accuracy homogeneity measurements temperature stability in the interferometer chamber and for the sample should be better than the values given in Table B.1.

Table B.1 — Maximum admissible temperature variation in infrared optics samples to be measured for different homogeneity conditions with double pass interferometer

Dimension in degrees Celsius[a]

Homogeneity to be measured	For infrared material types with thermo-optical coefficient G[b]				
	5×10^{-5}/°C	10×10^{-5}/°C	15×10^{-5}/°C	20×10^{-5}/°C	40×10^{-5}/°C
10×10^{-5}	1,00	0,50	0,33	0,25	0,13
4×10^{-5}	0,40	0,20	0,13	0,10	0,05
2×10^{-5}	0,20	0,10	0,07	0,05	0,03
1×10^{-5}	0,10	0,05	0,04	0,03	0,02

[a] Admissible temperature variations are given in degrees Celsius.

[b] The thermo-optical coefficient G is an infrared material type specific value.

The limit values for temperature stability in Table B.1 come from the following consideration.

The wavefront distortion due to refractive index homogeneity in the sample measured with a double pass interferometer is

$$\Delta W_H = 2 \cdot \Delta n \cdot t_0$$

where

Δn is refractive index variation in the sample (peak-to-valley);

t_0 is sample thickness.

The wavefront distortion due to thermal gradients in the sample is

$$\Delta W_{Th} = 2 \cdot t_0 \cdot G \cdot \Delta T$$

where ΔT is temperature variation within the sample (peak-to-valley).

The thermo-optical coefficient G is defined by

$$G=\left[(n-1)\alpha+\frac{dn}{dT}\right]$$

With requiring that

$$\Delta W_{Th}<\frac{1}{2}\Delta W_H$$

follows that

$$\Delta T<\frac{\Delta n}{2\cdot G}$$

By far most infrared materials have thermo-optical coefficients G between 2 and 10 × 10^{-5}/°C. High refractive index germanium types lie higher up to almost 40 × 10^{-5}/°C. Extremely low index Al_2O_3 types lie below 1 × 10^{-5}/°C. The infrared material chalcogenide used very frequently for high homogeneity applications has a thermo-optical coefficient of 7 × 10^{-5}/°C.

For the calculation of G the following quantities have been used:

n_m is refractive index at the middle wavelength band infrared radiation 4 µm;

α is coefficient of thermal expansion valid for the temperature interval −30 °C to +70 °C;

dn/dT is refractive index change with temperature relative to air at the spectral middle wavelength band infrared radiation for the temperature interval +20 °C to +40 °C.

EXAMPLE Two examples for calculating the thermo-optical coefficient G is shown below.

Infrared material types are ZnS(CVD) and chalcogenide.

α	6,6 × 10^{-6}/°C; 20,5 × 10^{-6}/°C
dn/dT	4,3 × 10^{-5}/°C; 3,2 × 10^{-5}/°C
n_m	2,252; 2,7945
G	5,09 × 10^{-5}/°C; 6,88 × 10^{-5}/°C