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Plastics — Methods of exposure to determine the wavelength dependent degradation using spectrally dispersed radiation

Plastiques — Méthodes d'exposition pour déterminer la dégradation dépendante de la longueur d'onde en utilisant un rayonnement dispersé spectralement

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Plastics are used outdoors and indoors where they are exposed to solar radiation, to solar radiation filtered by window glass and to artificial radiation sources for long periods. Therefore, information on the wavelength dependent degradation of a polymer property (e.g. optical and mechanical) within the ultraviolet and visible solar spectrum is important. The results of this test determine the spectral sensitivity of a property change over the range of the ultraviolet and the visible solar spectrum.

Plastics — Methods of exposure to determine the wavelength dependent degradation using spectrally dispersed radiation

1 Scope

This document specifies methods of determining the spectral response of all kind of plastics materials to ultraviolet and visible radiation by an irradiation test with spectrally dispersed irradiation.

NOTE Typical specimens that are evaluated include: films, liquids, plaques, pellets, powders, sheets and discs.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 4582, *Plastics — Determination of changes in colour and variations in properties after exposure to glass-filtered solar radiation, natural weathering or laboratory radiation sources*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following 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
short-pass filter
filter that transmits wavelengths shorter than the cut-off wavelength while rejecting longer wavelengths, and characterized by a sharp transition from maximum to minimum transmittance

[SOURCE: ISO 9370:2017, 3.31]

3.2
long-pass filter
filter that transmits wavelengths longer than the cut-on wavelength while rejecting shorter wavelengths, and characterized by a sharp transition from minimum to maximum transmittance

[SOURCE: ISO 9370:2017, 3.21]

3.3
file specimens
portion of the material to be tested which is stored under conditions in which it is stable and which is used for comparison between the exposed and unexposed states

[SOURCE: ISO 4892-1:2016, 3.2]

3.4
action spectrum
description of the spectral efficiency of radiation to produce a particular polymer response (change in a specific property of a specific polymer) as a function of wavelength

Note 1 to entry: Data of an action spectrum are specific to the polymer but independent of the radiation source. This term is also known as spectral sensitivity.

3.5
reciprocal linear dispersion of the radiation
wavelength dispersion in the focal plane

Note 1 to entry: It is expressed in nm/mm.

3.6
spatial resolution of the exposure device
width of the image of the entrance slit in the specimen plane, to be calculated by the slit width multiplied by the reciprocal linear dispersion

3.7
spectral irradiance
E_λ
irradiance per wavelength interval

Note 1 to entry: It is typically reported in watts per square metre per nanometre ($W \cdot m^{-2} \cdot nm^{-1}$).

[SOURCE: ISO 9370:2017, 3.20]

3.8
spectral radiant exposure
time integral of *spectral irradiance* (3.7)

Note 1 to entry: It is expressed in joules per square metre per nanometre ($J \cdot m^{-2} \cdot nm^{-1}$).

4 Principle

A specimen is exposed to spectrally dispersed radiation. This exposure causes wavelength dependent degradation of a test specimen. The position of the degradation of a test specimen is a function of wavelength. Analysis of these parameters enables a quantitative evaluation of the degradation as a function of wavelength.

Additionally, the action spectrum can be quantified, by spatial measurement of both spectral irradiance and the resulting changes in the test specimen property.

Temperature control of the test specimen may be used.

NOTE General information on the test method is described in Annex A. Examples of devices for spectrally dispersed irradiation are shown in Annex B. Examples of test result are shown in Annex C.

5 Apparatus

5.1 Exposure device

The radiation source shall provide radiation covering the entire wavelength range under investigation, which is typically around 200 nm to 700 nm. If the light source generates ozone, the ozone shall be removed using an ozone treatment system.

NOTE 1 Typically, a Xenon arc lamp is used.

The radiation is directed to an entrance slit of an optical system. The width of the entrance slit determines the spectral resolution and the irradiance of spectrally dispersed radiation. A narrower entrance slit results in higher resolution and lower irradiance. Conversely, a wider entrance slit results in lower resolution and higher irradiance.

The radiation through the entrance slit is spectrally dispersed by means of a diffraction grating which should be widely illuminated. In order to optimize the deflection to get only one deflection order for a specific wavelength range, a blaze grating may be used.

For optical geometry, various imaging optical elements whose spectral characteristics should be optimized for ultraviolet and visible radiation can be used. An optical short-pass or long-pass filter should be used to exclude unwanted radiation such as stray radiation.

The image of the entrance slit shall be focused at a specimen mounting port. A specimen can be irradiated with radiation wavelengths separated along the horizontal axis of the specimen plane.

The range of wavelengths irradiating the specimen can be adjusted by moving or switching the angular position of the grating.

NOTE 2 For a simple check of the wavelength positions, interference filters can be used.

The width of the entrance slit and the design of the optical system dictates the spatial resolution. When determining the evaluation criteria of a property being examined, the spatial resolution should be considered.

Another relevant characteristic is the reciprocal linear dispersion of the radiation as wavelength range per specimen width (in nm/mm).

The height of the image in the specimen plane depends on the optical system design.

NOTE 3 As the lamp arc locally varies in temperature, the emitted spectral irradiance is not homogeneous over the arc height. By using a cylindrical mirror instead of a spherical one, height range as well as height homogeneity is increased, at the same time decreasing spectral irradiance/intensity.

Temperature control of the specimen under test may be used. Typically, the test is performed at ambient temperature. Other specimen temperatures may be used upon agreement with the interested parties and shall be reported. Because of the low irradiance of the dispersed radiation the radiation heating can be neglected.

Liquids, pellets and powder specimens are tested using a cell. Unless specified, this cell shall be made of clear, optically, transparent material (typically UV grade quartz).

5.2 Spectroradiometer

For measuring the spectral irradiance at various positions in the specimen plane, a spectroradiometer should be used. A calibrated diffusing detector with an entrance aperture diameter not greater than the spatial resolution of the exposure device enables the best characterization.

5.3 Measuring device for property change

For a quantitative evaluation, the property to be investigated shall be measured before and after the respective exposure. The lateral resolution of the measuring device should be adapted to the spatial resolution of the exposure device.

6 Test specimens

6.1 Specimen preparation and conditioning

The methods used for the preparation of test specimens can have a significant impact on test results.

The methods used for specimen preparation and conditioning should preferably be closely related to the methods used to qualify the material in its application. Therefore, the methods used for preparing and conditioning the specimen shall be agreed upon by the interested parties. A complete description of the methods used for preparing and conditioning the specimen shall be included in the test report.

6.2 Specimen conditioning

6.2.1 Condition test specimens in accordance with ISO 291, unless otherwise specified.

NOTE In case of photosensitive materials, storage in a dark and refrigerated environment can be advantageous.

6.2.2 When characterizing the optical and mechanical properties of the specimen, condition the specimen in accordance with ISO 291 before measurements. If the mechanical properties of the specimen are known to be sensitive to moisture content, the duration of conditioning may be longer than specified in ISO 291.

6.2.3 It is essential that colour measurement, visual evaluation of the specimen's colour and assessment of other optical properties be performed immediately after exposure. If conditioning the specimens in accordance with ISO 291 is desired before testing, the conditioning and the duration shall be based on agreement. The specimen conditioning shall be reported.

7 Exposure parameter

7.1 Radiation

Contact the manufacturer of the apparatus for specific spectral irradiance data, the reciprocal linear dispersion of the radiation and the spatial resolution. These parameters should be reported in the test report.

If necessary, measure the spectral irradiance at various positions, x, in the specimen plane. This measurement shall not be done during specimen exposure, as it should be performed in the exact specimen exposure height. Spectral irradiance integrals, $E_\lambda(x)$, as well as peak wavelengths, $\lambda(x)$ are related to the measured horizontal positions x in the specimen plane and should be presented as a table in the test report. As the spectral irradiance is averaged over the width of the irradiance detector, the detector's diameter is essential for this characterization.

NOTE 1 Minimum error and the highest precision are achieved when the detector width is less than the spatial resolution of the system.

If necessary, determine the reciprocal linear dispersion of the radiation as wavelength range per specimen width (in nm/mm). This can be done by relating the peak wavelengths of spectral irradiance measurements to the respective horizontal positions in the specimen plane.

NOTE 2 The spatial resolution is measured as FWHM of a spectral irradiance peak, using a detector that is smaller than the slit image in the specimen plane. Or it is calculated as the entrance slit width multiplied by the reciprocal linear dispersion.

During the exposure of the specimen, the irradiance should be controlled or checked.

NOTE 3 This is accomplished by continuous measurement at a specific wavelength in the specimen plane or by continuous measurement of the irradiance of the Xenon arc lamp.

7.2 Specimen temperature

Typically, the test is performed at standard atmosphere in accordance with ISO 291. Other specimen temperatures may be used upon agreement with the interested parties and shall be reported.

7.3 Test duration

Test duration is dependent on the spectral sensitivity of the specimens and the spectral irradiance in the exposure specimen plane.

NOTE In case of nonlinear progress of the property change, it is useful to vary the exposure duration. The radiant exposure for similar property change is determined at the various wavelengths.

8 Procedure

8.1 Specimen optical and mechanical properties measurements

8.1.1 Follow the procedures described in ISO 4582 for measuring properties of test specimens before and after exposure. Refer to the relevant International Standards for the specific procedures for measuring specimen properties, for example, micro hardness conforming to ISO 14577-1 or molecular mass conforming to the ISO 16014 series.

NOTE The spectral irradiance is not constant with wavelength.

8.1.2 If non-destructive tests are used to measure the properties of the materials being tested, the properties of the specimens shall be measured before testing. The same properties are then measured after each exposure period. Care should be taken to ensure that the measurement of the property is obtained in exactly the same position on the specimen.

8.1.3 If destructive tests are used to measure the properties of the materials being tested, sufficient quantities of file specimens will be needed. It is recommended that the value of the material property obtained by destructive testing after exposure be compared to the same property measured on a set of file specimens, measured at the same time as the exposed specimens. Alternatively, the value of the property after exposure may be compared to that value obtained prior to exposure.

8.2 Mounting test specimens

Attach the test specimens to the specimen mounting port so that the specimens are not subject to any applied stress.

If desired, a portion of each test specimen may be shielded from the radiation by an opaque cover throughout the exposure. This produces an unexposed area directly adjacent to the exposed area for comparison. This technique is useful for checking and evaluating the results of the exposure. The reported data shall always be based on a comparison to file specimens.

8.3 Exposure

Before mounting the specimens in the specimen mounting port, ensure that the apparatus is operating under the test conditions. Program the apparatus to operate continuously. Maintain the test conditions throughout the exposure, keeping any interruptions to service the apparatus and to inspect the specimens to a minimum.

If it is necessary to remove a test specimen from the test apparatus, take extreme care not to touch the exposed surface or alter the position of the specimen in the specimen mounting port. If the test specimen was removed from the specimen mounting port, return the test specimen to the specimen mounting port with its exposed surface oriented in the same direction and in the same position as it was before removal.

9 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 21475:2019;

b) description of the exposure device, including

- width of the entrance slit,
- reciprocal linear dispersion of the radiation as wavelength range per specimen width (x nm/mm),
- table with measured peak wavelengths, integrated spectral irradiance, and horizontal positions,
- spatial resolution,
- detector diameter of the spectroradiometer;
- spectral wavelength range;

c) wavelength of measurement and/or control, and irradiance;

d) description of the material tested;

e) dimensions and shape of the test specimens and the nature and area of the surface tested;

f) details of the preparation of the test specimens, including any cleaning treatment applied before testing;

g) the result of the spectral irradiance check and the wavelength calibration;

h) temperature of the specimen;

i) duration of the test;

j) the result of test, optical, mechanical and other properties;

k) any deviation from the test procedure specified;

l) the date of the test.

Annex A
(informative)

General information on the test method using spectrally dispersed radiation

A.1 General

The spectral sensitivity is only valid for the chosen test specimen, the defined property change and the given exposure conditions.

A.2 Previous history of the test specimen

Any pre-irradiation or pre-conditioning of the test specimen may degrade the performance of some specimens. Pre-irradiation or pre-conditioning may affect the crystalline structure that affects performance.

A.3 Thickness of the test specimen

The absorptivity of a translucent specimen changes with thickness. Hence, the degradation may vary with specimen thickness.

A.4 Increasing temperature

An elevated specimen temperature increases the rate of photo degradation of most polymers and influences the spectral sensitivity.

A.5 Other important parameters

Contamination such as moisture content or residual chemicals on a specimen will influence the results.

A.6 Further consideration

The method generally has plusses and minuses.

Because spectral irradiance can be easily measured in the plane of the exposed test specimen surface, this procedure can be used to determine the absolute actinic effects of the spectral regions of solar radiation within narrow spectral bands.

a) Spectral sensitivity shows the sensitivity of a primary reaction which in practice may be followed by secondary reactions caused by radiation with longer wavelengths and leads to the final ageing result obtained in practice.

   For a quantitative evaluation, the action spectrum $s_\lambda(x)$ can be calculated at all measured positions by the Formula (A.1):

$$s_\lambda(x) = \Delta P_\lambda(x) / H_\lambda(x) \quad \text{(A.1)}$$

where

$\Delta P_\lambda(x)$ is a change in an optical or mechanical property at position x on the specimen at wavelength λ;

$H_\lambda(x)$ is the radiant exposure expressed in J/m^2 at wavelength λ.

b) Only small specimens can be used in exposure to narrow spectral band radiation, this limits the type of property changes which can be measured.

c) Polychromators have increased stray radiation, which therefore, should be reduced or eliminated.

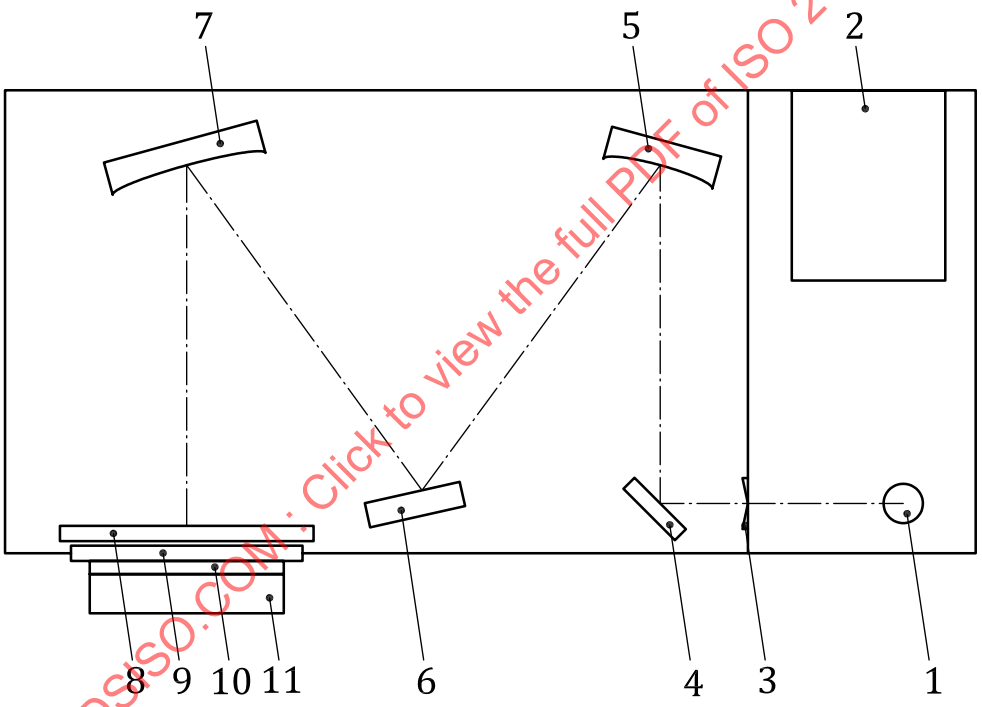
Annex B
(informative)

Examples of devices for spectrally dispersed irradiation

An example used in the test method described in this document is schematically shown in Figure B.1. Typically, two wavelength ranges, 220 nm to 520 nm and 400 nm to 700 nm, are used.

Typically, 160 mm opening port (irradiated portion) corresponds to wavelength of 300 nm in width, resulting in a reciprocal linear dispersion of 15 nm/8 mm.

During the test, the irradiance is measured at the 300 nm position for the investigated range of 220 nm to 520 nm or at 480 nm position for the range of 400 nm to 700 nm.



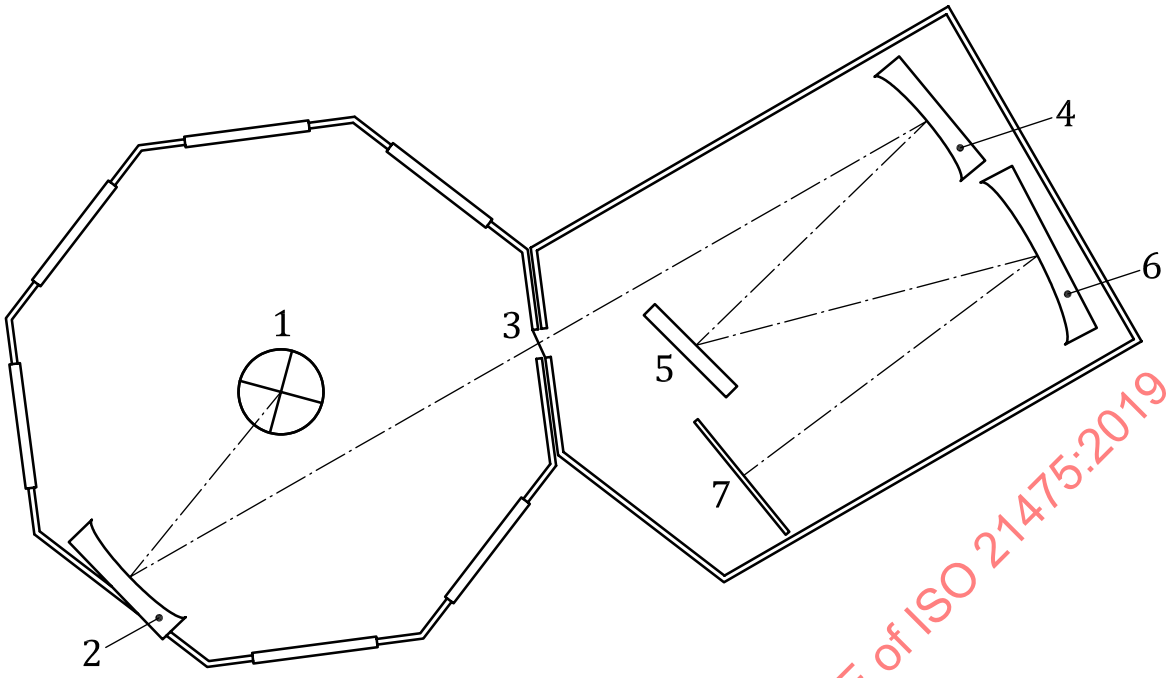
Key

1 radiation source
2 ozone treatment system
3 entrance slit
4 flat mirror
5 concave mirror
6 flat mirror grating
7 concave mirror
8 optical short-pass, long-pass filter
9 specimen mounting port
10 test specimen
11 specimen temperature controller

Figure B.1 — Typical schematic diagram — Example 1

NOTE Stray light level is the ratio of a measured signal in the blocking range of an absorption filter to the maximum desired signal. Stray light results from a wavelength range which is not part of the measured wavelength range and depends on the radiation source used and the geometry of the spectrometer. Stray light influences the dynamic range of the spectrometer.

Another example used in this test method is schematically shown in Figure B.2. Typically, a wavelength ranges from 250 nm to 500 nm is used, with a reciprocal linear dispersion of 4 nm/mm.



Key

1	xenon arc lamp	5	grid
2	spherical mirror	6	cylindrical mirror
3	slit	7	specimen plane
4	concave mirror		

Figure B.2 — Typical schematic diagram — Example 2

Annex C
(informative)

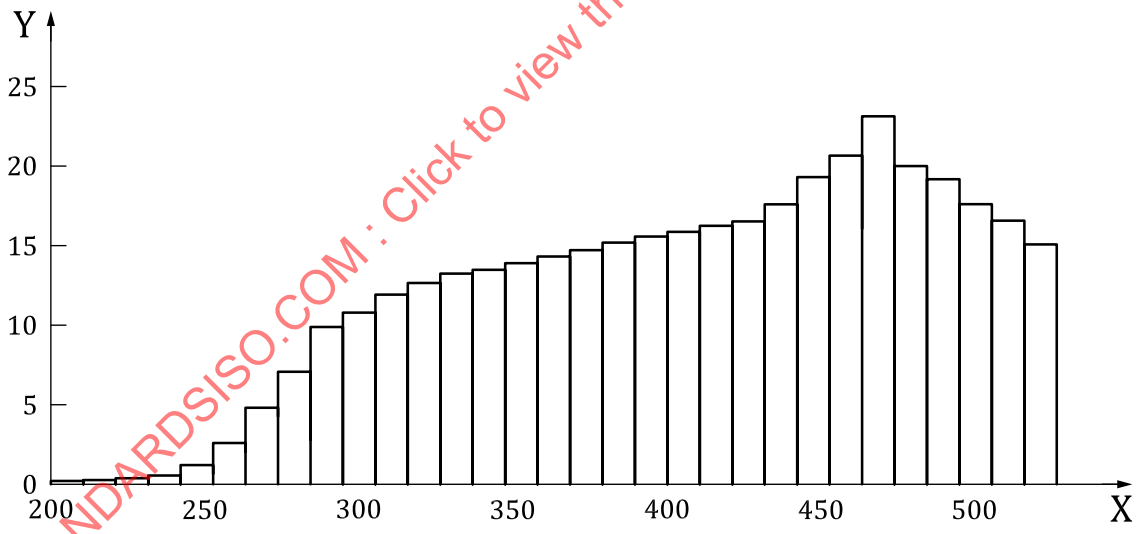
Examples of test results

C.1 General

This annex describes examples of test conditions, test specimens as well as test results under the test conditions for better understanding of the test method specified in this document. Test results for standard grade and weather resistant grade of typical polycarbonate are described in C.4. The test result for typical polypropylene is described in C.5.

C.2 Spectrally dispersed radiation test

An apparatus used for this test is shown in Figure B.1. These tests are carried out using a spectrally dispersed radiation test instrument SPX[1)]. A specimen is irradiated by spectrally dispersed radiation from 220 nm to 520 nm. Spectral irradiance at 300 nm is controlled 10 $W \cdot m^{-2} \cdot nm^{-1}$. A width of the entrance slit is 4 mm, a reciprocal linear dispersion of 15 nm/8 mm, spatial resolution (FWHM) is 8 nm, and a detector diameter is 3 mm. Each point of spectral irradiance is described in Figure C.1. Test period is 100 h.



Key

X wavelength (nm)

Y spectral irradiance ($W \cdot m^{-2} \cdot nm^{-1}$)

Figure C.1 — Spectral irradiance at sample

1) SPX is an example of a suitable product supplied by Suga Test Instruments Co., Ltd., Japan. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

C.3 Optical and mechanical property measurement

C.3.1 Colour measurement

Colour measurement is done according to CIE 15. Spectral reflectance from 380 nm to 780 nm is determined using de:8° geometry, and a measured diameter is 5 mm.

C.3.2 Micro-fourier transform infrared spectroscopy

Measurement of micro-fourier transform infrared spectroscopy in attenuated total reflectance (ATR) mode is carried out. The wavelength range is from 4 000 cm^{-1} to 600 cm^{-1}, resolution is 4 cm^{-1}, depth of measurement is 1 µm, and measured diameter is 0,1 mm.

C.3.3 Micro hardness

Measurement of micro hardness is done according to ISO 14577-1, and measurement condition is HM 0,004 9 3,45/5,00.

C.3.4 Molecular mass

Measurement of molecular mass is done according to ISO 16014 series. The number-average molecular mass (M_n) and the mass-average molecular mass (M_W) are measured.

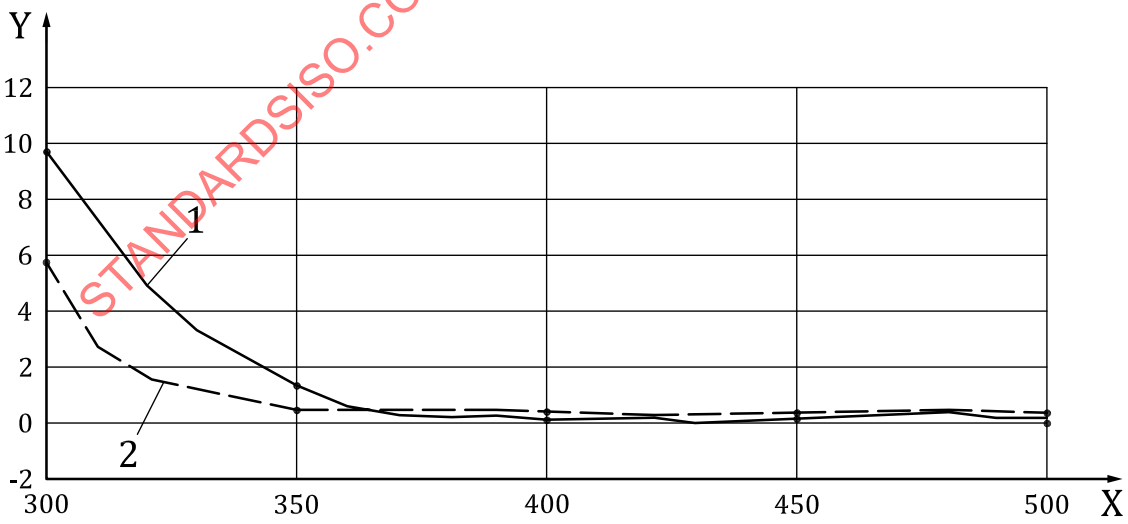
C.4 Test results of polycarbonate

C.4.1 Specimen

Two types of polycarbonate are used in this test. One is "standard grade", PC1, another is "weather resistant grade", PC2. The dimension of each specimen is 174 mm × 38 mm and 4 mm thickness.

C.4.2 Result of colour measurement

The test result of change of yellowness index (Δ YI) done according to ISO 17223 is shown in Figure C.2. The test result is not normalized.



Key

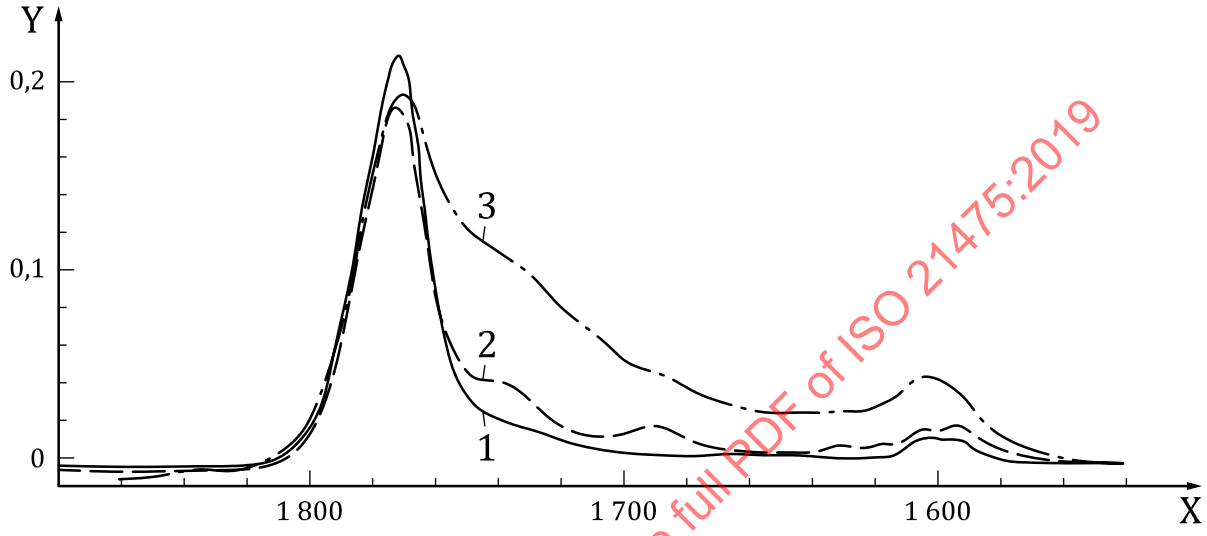
X	wavelength (nm)	1	PC1
Y	change of yellowness index (Δ YI)	2	PC2

Figure C.2 — Result of change of yellowness of PC1 and PC2

Figure C.2 shows that PC1 yellowness is increased around 300 nm significantly, although PC2 is rather stable. Δ YI of PC2 is less than 1 for wavelengths greater than 330 nm.

C.4.3 Result of micro-fourier transform infrared spectroscopy

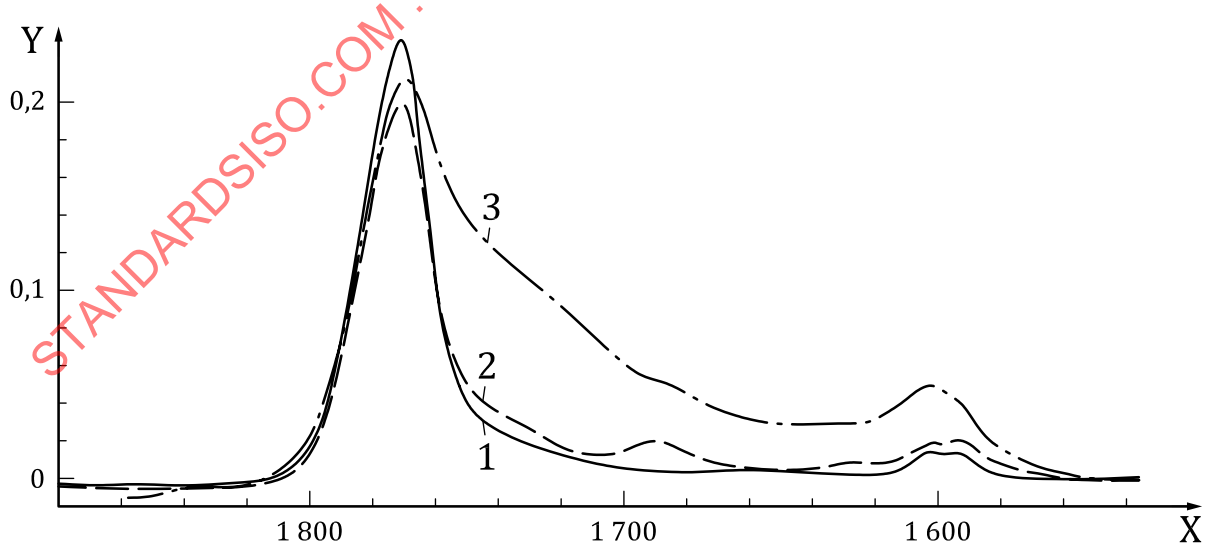
Each specimen was assessed before exposure, and after irradiation with 310 nm and 480 nm radiation. Test result of micro-fourier transform infrared spectroscopy (PC1 and PC2) is described in Figures C.3 and C.4.



Key

X wavenumber (cm^{-1})
Y absorptance
1 before exposure
2 point irradiated by 310 nm radiation
3 point irradiated by 480 nm radiation

Figure C.3 — Result of micro-fourier transform infrared spectroscopy of PC1



Key

X wavenumber (cm^{-1})
Y absorptance
1 before exposure
2 point irradiated by 310 nm radiation
3 point irradiated by 480 nm radiation

Figure C.4 — Result of micro-fourier transform infrared spectroscopy of PC2

Each result of PC1 and PC2 is almost same and is shown below.

a) A peak intensity of carbonic acid ester bond near 1 780 cm^{-1} on irradiated point by 310 nm and 480 nm radiation is less than before exposure.

b) A stretching vibration in the C = 0 bond of the carboxylic acids near 1 690 cm^{-1} on 310 nm is more active than before exposure.

c) A peak intensity by ketone and ester near 1 740 cm^{-1} to 1 710 cm^{-1} on 310 nm is higher than before exposure.

d) Reactions of b) and c) are shown on 480 nm but weaker than reactions of 310 nm.

C.4.4 Result of micro hardness

Micro hardness was measured for each specimen before exposure, and after irradiation with 310 nm and 480 nm radiation and the result is shown in Table C.1.

Table C.1 also shows the result of ratio of hardness compared with before exposure. It increases only at a point irradiated by 310 nm radiation of PC1. It seems the 310 nm radiation causes a cross-linking reaction at that point.

Table C.1 — Result of micro hardness of PC1 and PC2

Specimen		Micro hardness HM 0,004 9 3,45/5,00 (N/mm^2)	Ratio
PC1	Before exposure	150,0	100 %
	310 nm	173,6	116 %
	480 nm	155,9	104 %
PC2	Before exposure	161,8	100 %
	310 nm	161,8	100 %
	480 nm	163,8	101 %

C.4.5 Result of molecular mass

Molecular mass was measured for each specimen before exposure, and after irradiation with 310 nm and 480 nm radiation and the result is shown in Table C.2.

Table C.2 also shows the ratio of M_W for the exposure condition to M_W before exposure. It decreases at each point of both PC1 and PC irradiated by 310 nm radiation and PC1 is more decrease than PC2. It seems the 310 nm radiation causes a decomposition reaction in that point.

Table C.2 — Result of molecular mass of PC1 and PC2

Specimen		M_n	M_W	Ratio of M_W
PC1	Before exposure	13 900	50 300	100 %
	310 nm	12 400	46 400	92 %
	480 nm	13 600	49 500	98 %
PC2	Before exposure	8 420	50 200	100 %
	310 nm	8 200	48 000	96 %
	480 nm	8 540	49 800	99 %