BS EN 16472:2014

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Plastics — Method for artificial accelerated photoageing using medium pressure mercury vapour lamps

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National foreword

This British Standard is the UK implementation of EN 16472:2014.

The UK committee voted consistently against the enquiry and final drafts of this standard since the committee was of the opinion that the title of the standard should have been about determining the photo oxidation of materials by FT-IR and carbonyl formation, rather than the procedure of accelerated photo aging.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Plastics - Method for artificial accelerated photoageing using medium pressure mercury vapour lamps

Plastiques - Méthode de photovieillissement artificiel accéléré utilisant des lampes à vapeur de mercure à moyenne pression

Kunststoffe - Verfahren zur künstlich beschleunigten Alterung bei Verwendung von Quecksilberdampflampen

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Foreword

This document (EN 16472:2014) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2014 and conflicting national standards shall be withdrawn at the latest by October 2014.

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Introduction

When a polymeric material is exposed to natural UV radiation and other moderate environmental stresses, the change in most physical properties is attributable to chemical ageing, and the extent of the chemical changes can be related to the duration of the exposure under natural outdoor weathering conditions.

This method attempts to maximize the acceleration of photoageing using elevated UV irradiance and temperature that still keep the fundamental photoageing mechanism equivalent to that found in natural ageing. Temperature increase above the natural level should be limited so that the photothermal transformation exceeds any pure thermal conversion. A medium pressure mercury lamp, with radiations of wavelength lower than 290 nm properly filtered out, gives a relevant source with high UV emission intensity and low IR emission.

One of the main interests in use of artificial accelerated photoageing tests is to able to provide a relevant lifetime estimate of polymeric materials exposed in natural outdoor conditions.

The relevance of artificial ageing can be determined by comparing the chemical changes that occur in the accelerated test to those that occur in natural weathering (see ISO 10640). Kinetic analysis is recommended to determine the rate of degradation under different conditions of ageing in order to rank different formulations or to determine the range of acceleration possible for an artificial ageing test compared to a given natural outdoor weathering exposure (without distortion of the photodegradation mechanism of the polymer).

Chemical changes control the degradation of mechanical properties and contribute to changes in the visual appearance of polymer materials during photoageing. These chemical changes may be analysed primarily by IR spectroscopy, with additional analyses using UV/visible spectroscopy during the photoageing of polymers.

1 Scope

This European Standard specifies a method for carrying out artificial accelerated photoageing of test specimens by exposing them to medium pressure filtered mercury vapour lamp as light source, under controlled temperature conditions.

2 Normative references

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

EN ISO 4892-1:2000, *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance (ISO 4892-1:1999)*

ISO 4582, *Plastics — Determination of changes in colour and variations in properties after exposure to daylight under glass, natural weathering or laboratory light sources*

ISO 9370, *Plastics — Instrumental determination of radiant exposure in weathering tests — General guidance and basic test method*

ISO 10640, *Plastics — Methodology for assessing polymer photoageing by FTIR and UV/visible spectroscopy*

3 Terms and definitions

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

3.1
control
material which is of similar composition and construction to the test material and which is exposed at the same time for comparison with the test material

Note 1 to entry: An example of the use of a control material would be when a formulation different from one currently being used is being evaluated. In that case, the control would be the plastic made with the original formulation.

[SOURCE: EN ISO 4892-1:2000]

3.2
reference material
material of known performance

4 General

When correctly powered and maintained, the plasma of a medium pressure mercury arc discharge emits mainly UV and the visible radiation.

This lamp allows the acceleration of the photochemical process by high UV irradiance without high infrared emission.

Specimens of the samples to be tested are exposed to the laboratory light source under controlled temperature condition. The temperature activates the photochemical process.

Optionally, the samples can be exposed to immersion and/or dark periods. The design of the equipment shall achieve the appropriate specifications as well as the UV irradiance (radiant exposure) and temperature set points.

For comparing the performance of the test material to that of the control, it is recommended that at least one control be exposed during each test.

5 Apparatus

5.1 Laboratory light source

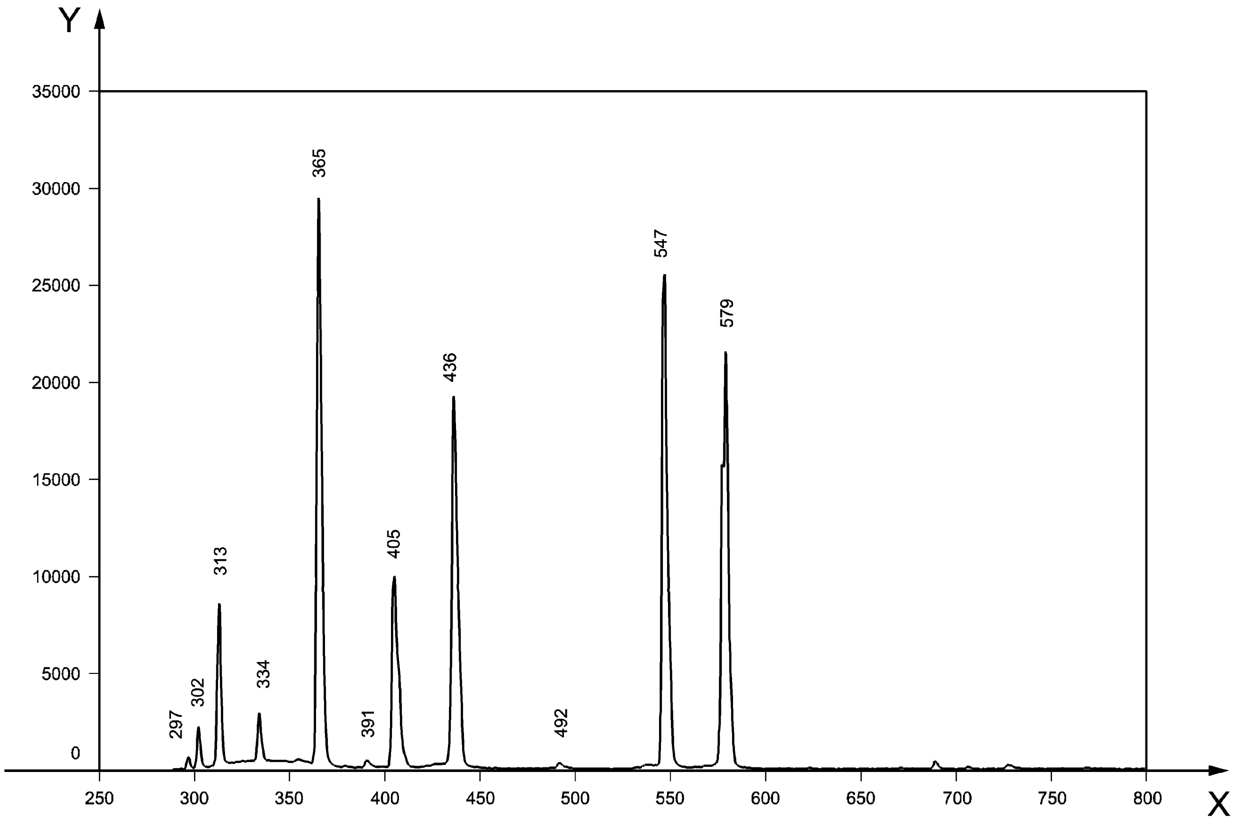
5.1.1 General

Medium pressure mercury vapour lamps consist of a quartz burner filled with a mixture of gas and mercury where the discharge takes place, the burner being located in a borosilicate bulb.

These lamps are available in different power categories.

The radiation they emit consists of lines of variable intensity within the range from 250 nm to 800 nm. Irradiance at wavelengths shorter than 290 nm is filtered out by the bulb. Therefore only lines at wavelength 297 nm, 302 nm, 313 nm, 334 nm, 365 nm, 391 nm, 405 nm, 436 nm, 492 nm, 547 nm and 579 nm remain.

A typical filtered spectrum of a medium pressure mercury vapour lamp is shown in Figure 1.



Key

X wavelength, nm

Y spectral irradiance, $mW \cdot m^{-2} \cdot nm^{-1}$

Figure 1 — Typical spectrum of a filtered medium pressure mercury vapour lamp

The filtered light emitted by a medium pressure mercury-arc does not simulate full spectrum sunlight but can be used to investigate photochemical phenomena. The relevancy to outdoor data shall be carefully considered. The only requirement is a relevant control of the chemical change in the solid state under polychromatic light.

Additional optical filters may be used for specific applications. Annex A provides information on additional filtering of lamp UV radiations.

Ensure the lamp has been pre-aged for 100 h prior to use, since the transmittance spectrum of borosilicate bulb may change significantly during this initial period.

NOTE Commonly, the light output (intensity and wavelength) does not vary more than 20 % during the lifetime of the lamps (see 5.4 or 8.1).

5.1.2 Spectral irradiance of medium pressure mercury vapour lamps

The minimum and maximum levels of the relative spectral irradiance in the UV region are given in Table 1.

Table 1 — Relative spectral irradiance of medium pressure mercury vapour lamps

Spectral passband (λ = wavelength in nm)	Relative spectral irradiance [a b c]	
	Minimum %	Maximum %
290 ≤ λ ≤ 300	0,0	2,0
300 < λ ≤ 320	5,0	20,0
320 < λ ≤ 360	8,0	14,0
360 < λ ≤ 380	46,0	61,0
380 < λ ≤ 400	1,0	5,0
400 < λ ≤ 420	14,0	25,0

[a] This table gives the irradiance in the given passband, expressed as a percentage of the total irradiance between 290 nm and 420 nm. To determine whether a mercury vapour lamp meets the requirements of this table, the spectral irradiance shall be measured from 250 nm to 420 nm. The total irradiance in each wavelength passband is then summed and divided by the total irradiance from 290 nm to 420 nm.

[b] The minimum and maximum limits in this table are based on a round robin test performed by five laboratories on several lamps from three suppliers, by using different spectroradiometers. The spectroradiometers shall be calibrated and shall have a FWHM (full width at half maximum) resolution ≤ 2,5 nm.

[c] The minimum and maximum columns will not necessarily sum to 100 % because they represent the minima and maxima for the measurement data used. For any individual spectral irradiance, the percentages calculated for the passbands in this table will sum to 100 %. For any individual mercury vapour lamp, the calculated percentage in each passband shall fall within the minimum and maximum limits given. Exposure results can be expected to differ if obtained using mercury-arc apparatus in which the spectral irradiances differ by as much as that allowed by the tolerances. Contact the manufacturer of the mercury vapour apparatus for specific spectral irradiance data for the mercury vapour lamp and filters used.

5.1.3 Irradiance uniformity

The irradiance at any position in the area used for specimen exposure shall be at least 80 % of the maximum irradiance. Requirements for periodic repositioning of specimens when this requirement is not met are described in EN ISO 4892-1.

NOTE For some materials of high reflectivity, high sensitivity to irradiance and temperature, periodic repositioning of specimens is recommended to ensure uniformity of exposures, even when the irradiance uniformity in the exposure area is within the limits.

5.2 Test chamber

The test chamber comprises a box the walls of which are made of a chemically inert material that will not affect test results and in which the following facilities may be located:

a) light source(s), consisting of one or more medium pressure mercury vapour lamps;

b) temperature measuring system that allows to control the temperature of a chosen test specimen;

c) inlets and outlets to renew air in the test chamber;

d) frame supporting the specimen holders (see 5.3). It is recommended to use a rotating frame carrying the specimen holders to improve the uniformity of the exposure.

5.3 Specimen holders

Specimen holders and their supporting frames shall be made of a chemically inert material that will not affect test conditions and test specimens (for example non-oxidizing alloys of aluminium or stainless steel). The specimen holders shall have minimal influence on the temperature of the test specimens.

If a backing is used, it may affect the results, as may any space between the backing and the test specimen, particularly with transparent specimens, and shall be agreed on between the interested parties.

5.4 Radiometer

The irradiance of the lamp may be checked by means of a radiometer in the UV wavelength range, preferably between 290 nm and 420 nm or between 300 nm and 400 nm.

When a UV radiometer is used, the UV irradiance shall be measured by using a calibrated radiometer, so that it receives the same radiation as the specimen surface. If it is not positioned in the specimen plane, it shall have a sufficiently wide field of view and be calibrated for irradiance at the specimen distance.

The radiometer shall comply with the requirements outlined in EN ISO 4892-1 and ISO 9370.

Measurements shall be performed at each lamp replacement and according to the recommendations of the manufacturer of the apparatus.

5.5 Temperature sensor

The temperature sensor shall consist of:

a) a reference material subjected to radiation from the lamp and the flow of cooling air. The reference material may be a polymer material e.g. plate or film, carefully selected so as to be thermally representative of the test specimens exposed;

b) a measuring device whose function is to ensure that the temperature of the reference material is accurately measured. This device consists of a sensitive component (thermoelectric couple or platinum resistance sensor) located in the reference material or in contact with it so as to ensure effective heat transfer.

It is recommended to use an electronic device capable to regulate the temperature within ± 0,3 °C.

The temperature of the exposed specimen can vary in a range larger than those of the setting value. It may be relevant to get the temperature of the specimen.

Black-panel or black-standard thermometers according to EN ISO 4892-1 may be also used.

Annex B provides information on the temperature control during photoageing.

5.6 Temperature controller

An optimised design of air intakes and outlets is recommended so that the cooling air flow maintains the temperature (see 5.5) within specifications, whatever the set temperature value may be. The regulation system shall allow renewing the air of the test chamber.

Once the apparatus has been put into operation, the set temperature value shall be achieved in less than 15 min.

The test shall be stopped if the actual temperature is higher than 5 °C above the set temperature value.

5.7 Optional facilities

The specimens may be exposed to moisture in the form of water spray, condensation or by immersion cycles, with or without light.

NOTE Information on the influence of water and additive migration can be found in ISO 10640.

6 Test specimens

Refer to EN ISO 4892-1:2000, Clause 6.

7 Exposure conditions

7.1 Radiation

The check of the irradiance on the exposed area of the test specimens may be carried out by means of a radiometer as described in 5.4 in order to ensure the reproducibility of testing.

Independently of its level, the irradiance at any position shall satisfy the requirement of 5.1.3.

The total irradiance between 290 nm and 420 nm, measured at the surface of the test specimens, shall be ≥ 60 W/m^2.

The total irradiance shall be agreed upon by the interested parties.

7.2 Temperature

The set point of the temperature sensor shall be agreed upon by the interested parties, taking into account the nature of the materials going into the test chamber, the applications envisaged and the reference material used.

The temperature set point is preferably chosen between 45 °C and 80 °C.

7.3 Optional facilities

If an immersion cycle is used, the kind of aqueous solution, the frequency, temperature and duration of the immersion shall be reported.

If dark periods are introduced in the cycle, their frequency, temperature and duration shall be reported.

Cycles shall be agreed upon by the interested parties.

8 Procedure

8.1 Verification of the apparatus

Use a reference material with well known changes of properties during an exposure, e.g. the polyethylene reference specimen as defined in ISO/TR 19032 or appropriate metrological instruments in order to check the reproducibility of the test.

8.2 Mounting the test specimens

Arrange the specimens on the specimen holders in the equipment with the side to be examined facing the light source.

Attach the test specimens to the specimen holders in such a manner that the specimens are not subjected to any unintended mechanical stress.

Identify each test specimen by suitable indelible marking, avoiding the areas to be used for subsequent testing. As a check, a plan of the test-specimens positions may be made.

In order to ensure effective repeatability, it is recommended to load the specimen holder turntable completely, using samples that are similar in size and shape.

8.3 Exposure

Before placing the test specimens in the test chamber, be sure the apparatus is operating under the desired conditions described in Clause 7.

Maintain these conditions throughout the exposure.

Depending on the type of temperature sensor, make sure the test specimen used to check the temperature of the surface material is properly in contact with the temperature sensor.

Start the test for the specified period.

If it is necessary to remove a test specimen for a periodic inspection, take care not to touch the exposed surface or alter it in any way. After inspection, the test specimen shall be replaced into the test chamber with its test surface in the same orientation as before. If intermediate sampling is carried out during the test, replace the test specimens by other specimens so that the turntable is fully loaded.

8.4 Measurement of radiant exposure

If used, mount and calibrate the radiometer and its integration system so that the radiometer indicates the irradiance at the exposed surface of the test specimen

This radiant exposure shall be expressed in terms of incident radiant energy per unit area of the exposure plane, in joules per square metre, for the passband selected.

8.5 Determination of changes in properties after exposure

The chemical variations in the polymeric material may be followed by using FTIR spectroscopy, as described in ISO 10640.

It is also possible to determine the property changes according to ISO 4582. However it is recommended that the macroscopic properties have been correlated with the chemical changes if possible to avoid incorrect conclusions.

Other analytic methods may also be used for the determination of changes in properties after exposure.

It shall be reported if specific storage conditions were applied for the aged specimens before analysis.

9 Test report

The test report shall contain the following information:

a) reference to this European Standard;

b) full description of the specimen including:

   1) their origin;

   2) a complete description of the method used for preparation of the test specimens.

c) description of the exposure device, including:

   1) type of apparatus;

   2) reference of lamps (age and lifetime);

   3) description of filters, if used;

   4) if required, the irradiance at the test specimen surface (including the bandpass in which the radiation was measured);

   5) type of temperature sensor used;

   6) if used, a complete description of the optional facility(ies) if it is not part of the apparatus itself.

d) reference material used for the test;

e) control, if used;

f) description of the method used to mount the test specimens in the specimen holders, as well as a description of any device used to hold the test specimen;

g) complete description of the exposure cycle, including:

   1) test duration;

   2) set point for test temperature;

   3) if used, a description of the immersion cycle (solution, temperature,frequency and duration);

   4) if used, the frequency, temperature and duration of dark cycles;

h) test results:

   1) in accordance with ISO 10640, If FTIR spectroscopy is used to follow the chemical changes;

   2) in accordance with ISO 4582, if used.

i) date(s) of the exposure test.

Annex A
(informative)

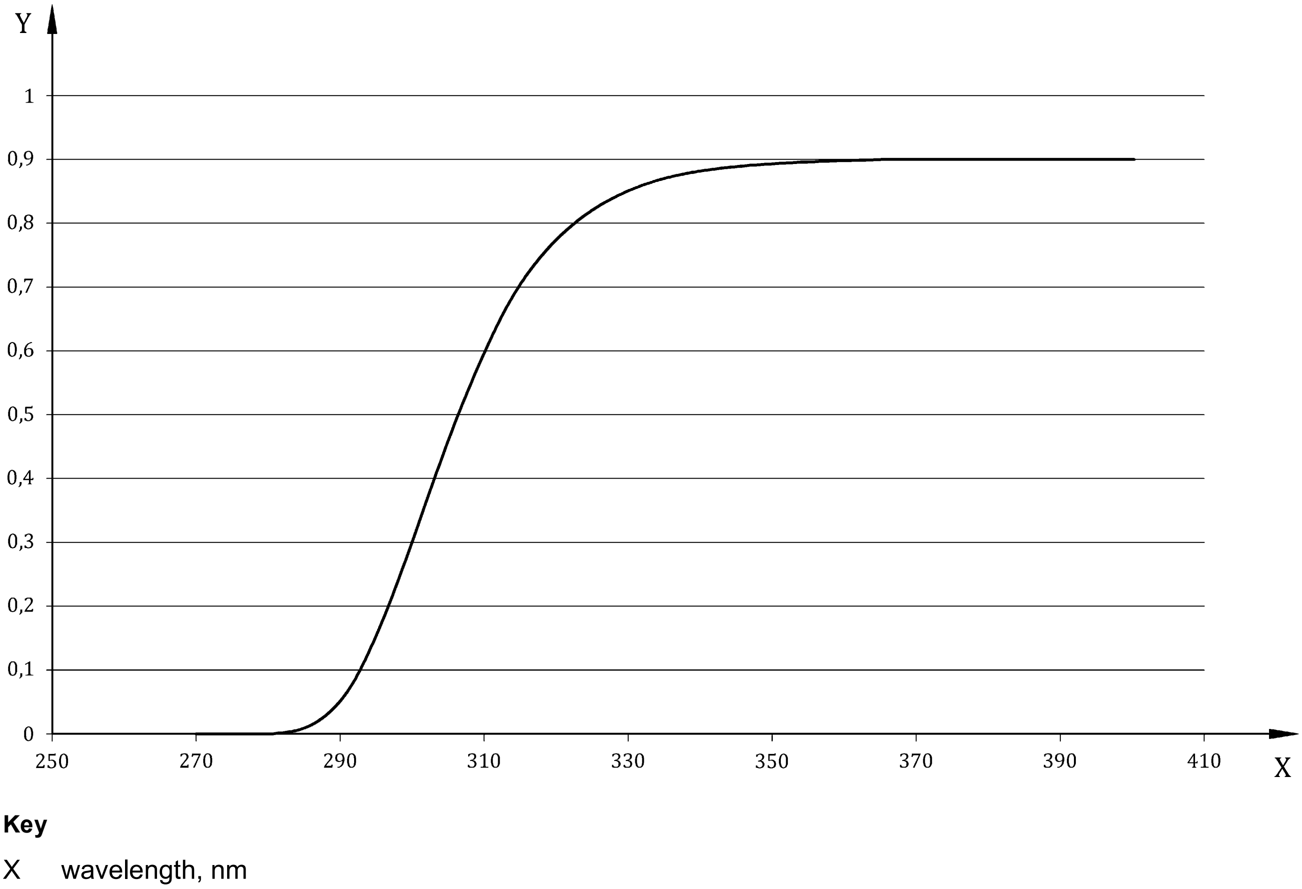
Additional filtering of lamp UV radiations

A.1 Additional filtering of UVB radiations for chromophoric polymers exposed to outdoor conditions

Polymers with UVB absorbing chromophoric units [aromatic polymers [e.g. polycarbonate, poly(ethylene terephthalate), polystyrene, unsaturated polyester resin) or polyamide] undergo a direct photochemistry and the photolysis processes are therefore enhance, depending on the lamp type for a given supplier and on the relative irradiance in the UVB range.

As shown in Table 1 (from 290 nm to 320 nm), the relative spectral irradiance uncertainty in the UVB range is quite large and a value close to the minimum one is recommended. In order to minimize the UVB radiation it is recommended to use an optical filter, e.g. reference N-WG305[1)] of 2 mm thickness. See Figure A.1.

In any case the filter shall not be placed against the exposed surface of the specimen.



Key

X wavelength, nm

Y transmittance

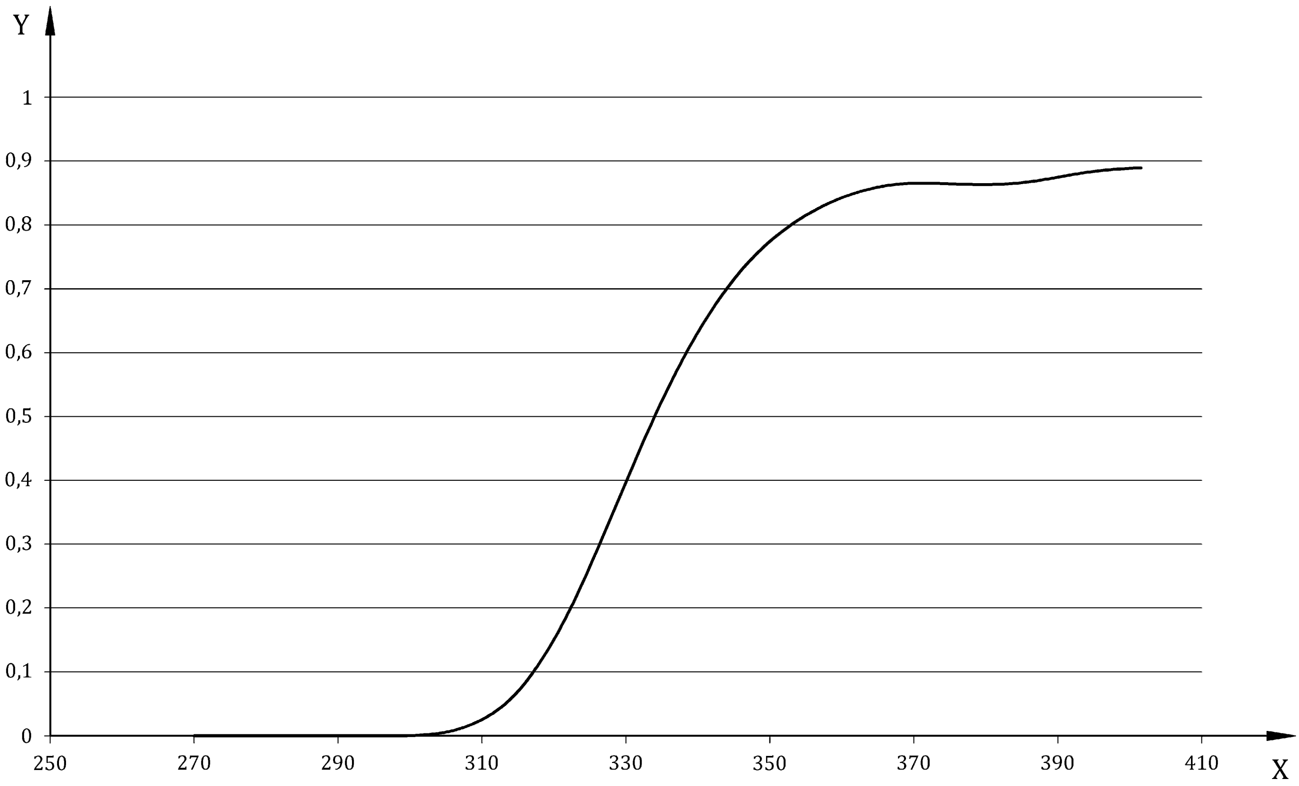
Figure A.1 — Example of transmittance versus wavelength of an optical filter

1) from SCHOTT AG, Hattenbergstrasse 10, 55122 Mainz (Germany).

A.2 Additional filtering of UV radiations for polymers exposed to indoor conditions

To achieve a spectral distribution meeting the minimum and maximum limits for window glass, a spectral transmittance for window glass of between 0,1 and 0,2 at 320 nm and a minimum of 0,8 at 380 nm is preferred. See Figure A.2.

In any case the filter shall not be placed against the exposed surface of the specimen.



Key

X wavelength, nm

Y transmittance

Figure A.2 — Example of transmittance versus wavelength of a glass window filter

Annex B
(informative)

Temperature control during photoageing

The temperature of the exposed specimen is of main importance during photoageing because a lot of processes are thermally activated (e.g. free radical photodegradation processes of the polymer, diffusion of additives, diffusion of oxygen). The acceleration of the photoageing follows an Arrhenius type law with the temperature that takes in account an aggregate activation energy which includes the superposition of several processes during photoageing. If the temperature is susceptible to vary during a defined cycle for exposure it is recommended to check the variation of the temperature of the specimen surface.

It is worth to note that no pure thermal degradation of the polymer material shall occur during the whole duration of the ageing test at the chosen temperature set point. In case of doubt, a check can be carried out by placing a specimen in an oven at the same temperature and for the same duration.

A filtered mercury medium pressure source is poor in long wavelength visible emission (lines at 547 nm and 579 nm) and does not emit any significant infrared wavelength. The heating of the specimen by visible and IR light absorption, depending on the colour and the nature of the polymer material, is very limited.

An accurate control of the effective temperature of the test specimen is essential but it is difficult to perform due to the heterogeneity of the temperature in the chamber (cooling airflow turbulence, water aspersion or immersion cycle or other cycle, etc.). Furthermore, different systems for temperature regulation and control are used by the devices on the market. In any case, a control of the temperature set point should be carried out during a photoageing test. The technique and the results of the control of the temperature, during an ageing test shall be described in the test report, so that the repeatability and the reproducibility of the ageing test can be ensured.

One way to overcome this difficulty, the use a polyethylene reference specimen PERS as defined in ISO/TR 19032 is recommended. The rate of photooxidation of the PERS offers a convenient and reliable technique to check at the same time the UV radiation and the temperature received by the specimen during a test.

The PERS film should be exposed for a fixed time (e.g. 48 h or 72 h). The oxidation level should be measured by means of IR spectrometry in the transmission mode. The absorbance ratio, i.e. absorbance at 1 713 cm^{-1}/absorbance at 2 020 cm^{-1}, gives a checking value for the severity of the ageing test.

Bibliography

ISO/TR 19032, *Plastics — Use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor weathering conditions*

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