TECHNICAL SPECIFICATION

ISO/TS 19022

First edition
2016-02-01

Plastics — Method of controlled acceleration of laboratory weathering by increased irradiance

Plastiques — Méthode d'accélération contrôlée du vieillissement en laboratoire par irradiance accrue

Reference number
ISO/TS 19022:2016(E)

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

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

Introduction

A realization of the acceleration of laboratory weathering under controlled conditions is an essential requirement for delivering reliable and fast prediction of material durability. In this connection, the correlation to real use aging behaviour is being checked constantly.

The fundamental parameters of a weathering test are simulated solar radiation, heat and moisture. The induced change in the material properties, among other things, is determined by the irradiance level and relative spectral irradiance of simulated solar radiation incident on the material surface during the test, the surface temperature, and the level of moisture. An increase in some well-known weather parameters, continuously monitored outdoors, offers opportunities to speed up the weathering process outdoors and in the laboratory. Since 1967, acceleration of outdoor weathering with instruments for intensified weathering using concentrated solar radiation (according to ISO 877-3) became a common practice. By concentrating the natural solar radiation with Fresnel mirrors, irradiances of five to six times higher than the maximum natural level has been reached. Already in 1996, a screening procedure with very high irradiances for dyed textiles were developed which enabled the reduction of the test duration for lightfastness grades from five days to seven days to two and a half days.[5]

However, the applicability of an increased irradiance for deterministic acceleration of weathering without a specific knowledge of material properties requires that the degradation of material (at constant temperature and moisture conditions) has to be dependent on the applied radiant exposure only, irrespective of the irradiance level and resulting exposure duration used during the test. For some materials fulfilling this criterion, the acceleration of weathering has been demonstrated successfully.[6][7][8][9] This criterion is not always fulfilled since an increase in the irradiance might not always produce the expected increase in the weathering acceleration due to possible and a priory unknown to the operator nonlinear dependence of the photochemical processes on the irradiance level. Moreover, the overall material degradation might be strongly affected by the other weather parameters which can be modified due to the increased irradiance.

There are limitations in using increased irradiances. Therefore, the applicability and the limits of this weathering acceleration approach are determined by the properties of the specific material and have to be investigated systematically in each particular case. In this respect, it is of essential importance to validate an appropriate test procedure under controlled conditions in laboratory with an artificial radiation source which can provide high irradiances above the natural level with the relative spectral irradiance closely mimicking the natural solar radiation. Simultaneously, the temperature of the sample specimen surface and of the chamber air is kept constant in a wide range of irradiance level. In addition, the usual wetting and rain option have to be available.

Plastics — Method of controlled acceleration of laboratory weathering by increased irradiance

1 Scope

This Technical Specification specifies a test method which allows predicting the aging rate of material specimens, e.g. plastics, under interest independent of the aging mechanisms as a function of radiant exposure. The UV irradiance of a simulated solar radiation (with a laboratory radiation source) will be extended above the normal maximum level on earth surface while keeping all relevant temperature parameters fixed.

NOTE For translucent plastics, the surface temperatures are below the white standard temperature. In addition, the maximum temperature is not on the irradiated surface, it is somewhere inside the plastic material.

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.

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

ISO 4892-1, *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance*

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

ISO 10640, *Plastics — Methodology for assessing polymer photo ageing by FT-IR and UV-visible spectrometry*

ISO/TR 17801, *Plastics — Standard table for reference global solar spectral irradiance at sea level — Horizontal, relative air mass 1*

CIE Publication No. 85:1989, *Solar spectral irradiance*

3 Terms and definitions

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

3.1
radiant exposure
H
radiant energy per unit

Note 1 to entry: Radiant exposure is given by the following formula:

$$H = \int E \times \mathrm{d}t$$

where

E is the irradiance, in watts per square metre (W × m^{-2});

t is the exposure time, in seconds (s).

Note 2 to entry: H is therefore expressed in joules per square metre (J × m^{-2}).

3.2
solar radiation
global solar radiation
solar radiant flux, both direct and diffuse, received on a horizontal plane unit area from a solid angle of 2π steradians

[SOURCE: ISO/TR 17801, 3.1, modified — irradiance has been replaced by radiation]

Note 1 to entry: In this Technical Specification, “solar radiation” always means “global solar radiation”.

4 Symbols and abbreviated terms

CHT	chamber air temperature (ambient air temperature)
BST	black-standard thermometer (insulated surface temperature sensor)
BPT	black-panel thermometer (uninsulated surface temperature sensor)
WST	white standard thermometer (insulated surface temperature sensor)
WPT	white panel thermometer (uninsulated surface temperature sensor)

5 Principle

A xenon or fluorescent UV radiation source (other radiation sources are possible), fitted with filters (if necessary), is used to simulate preferably the relative spectral irradiance of solar radiation according to CIE Publication No. 85:1989, Table 4 or solar radiation filtered by window glass. A standard test method (e.g. ISO 4892-2, ISO 4892-3) or a differing/non-standardized weathering test method shall be conducted as a basis for further investigations. The base level test shall use weathering parameters (irradiance, temperatures, relative humidity) which are not above a maximum natural level (e.g. irradiance in the wavelength range 300 nm to 400 nm smaller than 66 W/m^2). As a second step, the irradiance is increased step by step above the natural level. While the irradiance is increased above the maximum level (at least three levels) or decreased, all other test parameters (relative spectral irradiance, chamber air temperature, relative humidity) shall be kept constant (unchanged). If used, the influence of a spray/dry cycle shall be carefully considered. The test results (e.g. colour change, carbonyl formation) shall be plotted as a function of the radiant exposure.

6 Apparatus

6.1 General

The equipment comprises a climate chamber with a chamber air temperature and relative humidity measurement device. In the climate chamber, included is a radiation source. The radiation source may generate UV, visible radiation, and infrared radiation similar to solar radiation with appropriate filter systems. A cooling system for the laboratory simulated solar radiation source and a fixture for the specimens are included in the chamber as well.

6.2 Test chamber

The design of the test chamber may vary, but it shall be constructed from inert material. In case of radiation sources including VIS and IR, it shall be equipped with a blower which generates a defined airflow to be directed across the specimens. In addition to the controlled lamp wattage, the test chamber shall provide for control of chamber air temperature radiation shielded. For exposures that require control of humidity, the test chamber shall include humidity-control facilities that meet the requirements of ISO 4892-1.

NOTE If the lamp system (one or more lamps) is centrally positioned in the chamber, the effect of any eccentricity of the lamp(s) on the uniformity of exposure may be reduced by using a rotating frame carrying the specimens or by repositioning or rotating the lamps.

Should any ozone be generated from operation of the lamp(s), the lamp(s) shall be isolated from the test specimens and operating personnel. If the ozone is in an air stream, it shall be vented directly to the outside of the building.

6.3 Laboratory radiation source

6.3.1 General

The laboratory solar radiation sources (e.g. xenon arc lamp, fluorescent UV lamp) may emit radiation from below 270 nm in the ultraviolet through the visible spectrum and into the infrared. In order to simulate solar radiation with lamps which emit the full spectrum of solar radiation (UV, VIS, and IR radiation), filters shall be used to remove short wavelength UV radiation. In addition, filters to remove infrared radiation may be used to prevent unrealistic heating of the test specimens.

NOTE 1 Solar spectral irradiance for a number of different atmospheric conditions is described in CIE Publication No. 85. The benchmark solar radiation used in this Technical Specification is that defined in CIE No. 85:1989, Table 4. A recalculated Table 4 is available in ISO/TR 17801 (better wavelength resolution).

NOTE 2 If laboratory radiation sources are used which emit serious amounts in the IR and VIS range, surface temperatures are affected by surface heating. Then, actions to adjust specimen surface temperatures have to be taken.

6.3.2 Spectral irradiance

Filters are used to filter xenon-arc or other laboratory radiation sources in order to simulate solar radiation (CIE Publication No. 85:1989, Table 4). The minimum and maximum levels of the relative spectral irradiance are given in the used standard applications (e.g. ISO 4892-2, ISO 4892-3).

During the test, the relative spectral irradiance shall not be changed considerably. An evaluation method shall be used to compare the relative spectral irradiance at different irradiance levels.

NOTE 1 The deviation of the relative spectral irradiance at the lowest level from the highest level can be calculated according to ISO/TR 18486.

NOTE 2 The following wavelength ranges are recommended for the comparison of the relative spectral irradiance: 290 nm to 320 nm, 320 nm to 340 nm, 340 nm to 360 nm, and 360 nm to 400 nm.

NOTE 3 Xenon arc and fluorescent UV lamps can run at variable wattages (which the lamp is specified for) without a significant change of the relative spectral irradiance.

NOTE 4 For natural and artificial sun radiation, the irradiance of 60 W/m^2 in the wavelength range of 300 nm to 400 nm or 0,51 $W/(m^2 \times nm^{-1})$ at 340 nm indicates the one sun level. This does not mean that higher irradiances can appear (66 W/m^2 at 300 nm to 400 nm or 0,8 W/m^2 at 340 nm acc. new calculation of CIE 85, Table 4).

6.3.3 Irradiance uniformity

The irradiance at any position in the area used for specimen exposure shall be at least 90 % of the maximum irradiance.

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.

6.4 Radiometer

A radiometer shall be used to measure and control irradiance during the test procedure. The radiometer shall comply with the requirements outlined in ISO 4892-1 and ISO 9370.

6.5 Test chamber temperature and relative humidity

The chamber temperature sensor shall located, radiation-shielded, and combined with a sensor which measure the relative humidity in the exhaust air duct.

For exact calibration of the field chamber temperature and relative field sensor, it is necessary to move a calibrated working reference standard and the instrument sensor to about the same position so that a balanced temperature and humidity can be set for the measuring sensors and the chamber air. Calibration takes place as soon as the whole system is in thermal balance.

6.6 Black-standard/black-panel thermometer

The black-standard or black-panel thermometer used shall comply with the requirements for these devices given in ISO 4892-1.

The preferred maximum surface temperature device is the black-standard thermometer.

6.7 Wetting and humidity-control equipment

6.7.1 General

Specimens may be exposed to moisture in the form of water spray or condensation, or by immersion. If condensation, immersion or other methods are used to expose the specimens to moisture, details of the procedures and exposure conditions used shall be included in the exposure report.

NOTE Wetting cycles can have significant influence on the test results. Results can differ for keeping constant either the wetting duration (recommended for bulk related properties and/or thick samples) or the number of wetting cycles (recommended for surface related properties and/or thin samples).

6.7.2 Relative-humidity control equipment

For exposures where relative-humidity control is required, the location of the sensors used to measure the humidity shall be as specified in ISO 4892-1.

6.7.3 Spray system

The test chamber may be equipped with a means of directing an intermittent water spray onto the fronts or backs of the test specimens under specified conditions. The spray shall be uniformly distributed over the specimens. The spray system shall be made from corrosion-resistant materials that do not contaminate the water employed.

The water sprayed onto the specimen surfaces shall comply with the requirements in ISO 4892-1.

6.8 Specimen holders

Specimen holders may be in the form of an open frame, leaving the backs of the specimens exposed, or they may provide the specimens with a solid backing. They shall be made from inert materials that will not affect the results of the exposure, for example, non-oxidizing alloys of aluminium or stainless steel. Brass, steel or copper shall not be used in the vicinity of the test specimens. The backing used might affect the results, as might any space between the backing and the test specimen, particularly with transparent specimens and shall be agreed upon between the interested parties.

6.9 Apparatus to assess changes in properties

If an International Standard relating to the determination of the properties chosen for monitoring the changes in properties exists (see, in particular, ISO 4582 or ISO 10640), the apparatus specified by the International Standard concerned shall be used.

NOTE Different material properties of the same material may act differently. The property choice is of basic relevance for evaluating the weathering acceleration.

7 Test specimens

Test specimens shall be according to ISO 4892-1.

8 Test method

8.1 Select a standard test method or create an own test method.

8.2 The test parameters are the following:

a) relative spectral irradiance (preferred simulated solar radiation acc. ISO/TR 17801);

b) irradiance;

c) chamber air temperature (CHT);

d) surface temperature of the specimen (BST or BPT);

e) relative humidity;

f) rain cycle.

NOTE For some tests, rain cycles are not specified or not necessary.

8.3 Select the lowest irradiance level from the following:

a) (60 ± 2) W × m^{-2} (300 nm to 400 nm);

b) $(0{,}51 \pm 0{,}02)$ W × m^{-2} × nm^{-1} (340 nm).

NOTE The lowest irradiance level is normally defined by the selected standard test method but irradiance levels below the standard irradiance level are also allowed.

8.4 Run the test at the lowest irradiance level.

8.5 Increase the irradiance above the natural level ≥60 W × m^{-2} (300 nm to 400 nm) and 0,51 W × m^{-2} (340 nm). The deviation from the set point (±x W × m^{-2}) grows about proportional with the irradiance.

8.6 Run the test at increased irradiance level while keeping all other test parameters constant [relative spectral irradiance, chamber air temperature (≤±3 °C), surface temperature or black-standard or black-panel temperature (≤± 3°C), relative humidity (≤±10 %) and rain cycle].

Several increased irradiance levels shall be used (at least four).

NOTE Photo reactions may depend on the Arrhenius relation (a ΔT of 1 °C can cause a property change of about 8 %).

8.7 Plot the test results (property changes, e.g. colour change or carbonyl formation, see also ISO 4582) as a function of the radiant exposure.

NOTE 1 When the property change is plotted as a function of radiant exposure, the measured data will vary even if the test data are measured at the same radiant exposure and the law of reciprocity maybe fulfilled. On the other hand, it is not always practical to stop a test at the same radiant exposure.

NOTE 2 Other evaluations of the test are possible, e.g. if the test data are available at the same radiant exposure two irradiance levels can be compared directly.

8.8 Calculate the deviations from a fit of the data (Pearson correlation coefficient can be appropriate).

NOTE For a good correlation, the Pearson correlation coefficient is >0,9.

9 Exposure conditions

9.1 Radiation

Unless otherwise specified, control the irradiance at the levels indicated in the selected standard. Other irradiance levels may be used when agreed upon by the interested parties. The irradiance and the pass band in which it was measured shall be included in the exposure report.

9.2 Temperature

9.2.1 Black-standard and black-panel temperature

If a black-standard or a black-panel temperature is specified in the selected standard, these surface temperature sensors shall be used to define the temperature level during the test procedure.

Other surface temperatures may be used when agreed upon by the interested parties, but shall be stated in the exposure report.

If water spray is used, the temperature requirements apply to the end of the dry period. If the thermometer does not reach a steady-state during the dry period after the short water-spray part of the cycle, check whether the specified temperature is reached during a longer dry period and consider using this longer dry period.

NOTE During the water-spray part of the cycle, the black-standard or black-panel temperature will be close to that of the water used.

9.2.2 Chamber air temperature

Exposures can be run either with the chamber air temperature (CHT) controlled at a specified level or allowing the CHT to find its own level.

Exposures with controlled CHT are preferred.

If the CHT is not controlled, the specific surface temperature of the specimens shall be measured.

NOTE For xenon arc lamps (or similar full spectrum solar simulators: UV, VIS, IR radiation), the possible specimen surface temperature is limited by the temperature of the air surrounding the sample (CHT) as the lower limit and the BST temperature as the upper limit temperature (massive plastic samples can have higher temperatures than the temperature indicated by a BST). It is then assumed that the actual specimen temperature lies somewhere between the two cited limits.

9.2.3 Specimen surface temperature

The specimen surface temperature is the decisive measure. It is recommended to measure the surface temperature. In addition, a WST or WPT sensor can help to evaluate the specimen temperature.

Nevertheless, the test should be controlled by BST or BPT and CHT.

NOTE If the test method described under Clause 8 is used (increasing irradiance up to threefold solar irradiance with unchanged BST or BPT and CHT), the temperature change of the specific specimen surface is smaller than the tolerance described in 8.6.

9.3 Humidity of chamber air

9.3.1 Relative humidity of chamber air

Exposures can be conducted either with the relative humidity controlled at a specified level or allowing the relative humidity to find its own level.

Exposures with controlled relative humidity are preferred.

NOTE For some materials, the relative humidity might not be important.

9.3.2 Spray cycle

The spray cycle used shall be as agreed between the interested parties, but is normally defined by the used test method.

NOTE Spray cycles can have significant effect on the exposure result and has to be carefully considered. By increasing irradiance, either the wetting duration or the number of spray cycles during a test is reduced.

10 Procedure

10.1 General

It is recommended that at least three test specimens of each material evaluated be exposed in each run to allow statistical evaluation of the results.

10.2 Mounting the test specimens

Attach the specimens to the specimen holders in the equipment in such a manner that the specimens are not subject to any applied stress. Identify each test specimen by suitable indelible marking, avoiding areas to be used for subsequent testing. As a check, a plan of the test-specimen positions may be made.

If desired, in the case of specimens used to determine change in colour and appearance, a portion of each test specimen may be shielded by an opaque cover throughout the exposure. This gives an unexposed area adjacent to the exposed area for comparison. This is useful for checking the progress of the exposure, but the data reported shall always be based on a comparison with file specimens stored in the dark.

10.3 Exposure

Before placing the specimens in the test chamber, be sure that the apparatus is operating under the desired conditions. Program the apparatus with the selected conditions to operate continuously for the required number of cycles at the selected exposure conditions. Maintain these conditions throughout the exposure, keeping any interruptions to service the apparatus and to inspect the specimens to a minimum.

Expose the test specimens, the radiometer and the surface temperature sensor for the specified period.

If it is necessary to remove a test specimen for periodic inspection, take care not to touch the exposed surface or alter it in any way. After inspection, return the specimen to its holder or to its place in the test chamber with its exposed surface oriented in the same direction as before.

10.4 Measurement of radiant exposure

Mount and calibrate the radiometer so that the irradiance at the exposed surface of the test specimen is reported.

Express the exposure stages in terms of radiant exposures, in joules per square metre ($J \times m^{-2}$), in the wavelength band from 300 nm to 400 nm, or in joules per square metre per nanometre ($J \times m^{-2} \times nm^{-1}$) at the wavelength selected (e.g. 340 nm).

10.5 Determination of changes in properties after exposure

These shall be determined as specified in ISO 4582 or in ISO 10640 as far as possible. Other properties may be used if agreed upon by all interested parties.

Different material properties of the same material may act differently. The property choice is of basic relevance for evaluating the weathering acceleration.

11 Test report

Test report shall be according to ISO 4892-1.

Annex A
(informative)

Principle of reciprocity

The generalization of the influence of the irradiance level on the acceleration of material degradation is provided by the so-called principle of reciprocity.[10] In the framework of this principle, all material property changes are classified into the following two classes:

a) materials whose induced degradation during the weathering test is independent on the irradiance level at fixed applied radiant exposure when all other weathering parameters are kept constant;

b) all other materials.

Material property changes belonging to the first class are said to fulfill the principle of reciprocity. Here, the degradation of material is governed by the level of the radiant exposure only and in this case, the acceleration of weathering is limited by the maximal irradiance level which can be reached while keeping all other relevant parameters fixed. Therefore, the exposure duration can be simply shortened by the enhancement factor of the irradiance level. The situation is different for the second class of materials. In this case, the rate of induced degradation of some material property under interest ΔP can be obtained through the Formula (A.1):[10]

$$\Delta P = E^p \tag{A.1}$$

where

ΔP is the change of some material property;

E is the irradiance level;

p is the so called Schwarzschild coefficient.

NOTE p may vary between 0,5 and 1,0.

Knowledge of p provides a precise estimation of the aging behaviour at elevated level of irradiance. Therefore, the procedure to validate this parameter is an essential condition for realization of deterministic weathering acceleration.

Bibliography

[1] ISO 877-3, *Plastics — Methods of exposure to solar radiation — Part 3: Intensified weathering using concentrated solar radiation*

[2] ISO 4892-2, *Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

[3] ISO 4892-3, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

[4] ISO/TR 18486, *Plastics —Calculation of the goodness of fit of the spectral distribution of a solar simulator to a reference solar spectral distribution*

[5] Rieker J., & Wobser K. *Untersuchung über die Anwendbarkeit der Kurzzeitbelichtung für die betriebliche Kontrolle der Lichtechtheitseigenschaften farbiger Textilien*", Melliand Textilberichte 9, 1996

[6] Chin J., Nguyen T., Byrd E., Martin J. *The Law of Reciprocity Applied to High Radiant Flux Weathering Studies*", 2nd European Weathering Symposium, 2005, Natural and Artificial Ageing of polymers, CEEES Publication No. 6, 2005, ISBN 3-9808382-9-3, p. 105 - 114

[7] Hardcastle H.K. *A Characterization of the Relationship Between Light Intensity and Degradation Rate for Weathering Durability*", 2nd European Weathering Symposium, 2005, Natural and Artificial Ageing of polymers, CEEES Publication No. 6, 2005, ISBN 3-9808382-9-3, p. 115 - 130

[8] Stuck J.-W. *Erfahrungen und neue Möglichkeiten bei der Schnellbelichtung", DEK Fachtagung „Echtheitsprüfungen in der Textilindustrie*", Gelnhausen, Oktober 1996 9/9

[9] Boxhammer J. Kürzere Prüfzeiten bei thermischer und strahlungsbedingter Alterung von polymeren Werkstoffen durch verschärfte Prüfbedingungen", Tagungsband der 29. Jahrestagung der GUS, 2000, pp. 8.1–8.15.

[10] Trubiroha P., Geburtig A., Wachtendorf V. *The Principle of Reciprocity and its Limits*", 3rd European Weathering Symposium, 2007, Natural and Artificial Ageing of polymers, CEEES Publication No. 8, 2007, ISBN 978-3-9810472-3-3, p. 243 – 258

ICS 83.080.01

Price based on 10 pages