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Plastics

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

Platiques

Détermination des changements de coloration et des variations de propriétés après exposition au rayonnement solaire sous verre, aux agents atmosphériques ou aux sources de rayonnement de laboratoire

Norme Marocaine homologuée

Par décision du Directeur de l'Institut Marocain de Normalisation N° B.O N° , publiée au

Cette norme annule et remplace la norme NM ISO 4582 homologuée en 2004.

Correspondance

La présente norme est une reprise intégrale de la norme ISO 4582 : 2017.

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Avant-Propos National

L'Institut Marocain de Normalisation (IMANOR) est l'Organisme National de Normalisation. Il a été créé par la Loi N° 12-06 relative à la normalisation, à la certification et à l'accréditation sous forme d'un Etablissement Public sous tutelle du Ministère chargé de l'Industrie et du Commerce.

Les normes marocaines sont élaborées et homologuées conformément aux dispositions de la Loi N° 12- 06 susmentionnée.

La présente norme marocaine NM ISO 4582 a été examinée et adoptée par la Commission de Normalisation des des Matières plastiques (1).

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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 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 the following URL: 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*.

This fourth edition cancels and replaces the third edition (ISO 4582:2007), which has been technically revised. The main changes compared to the previous edition are as follows:

— due to the withdrawal of all parts of ISO 7724, the colour measurement procedure has been revised.

Introduction

A number of different exposure techniques can be used to provide information on the effects of environmental stresses such as simulated solar radiation, heat and water on plastics [see ISO 877 (all parts) and ISO 4892 (all parts)]. Each exposure test has its own particular application and relevance. When determining changes in a particular property or attribute of a material subjected to different exposures, the same evaluation methods should be used after all exposures to ensure meaningful results.

Results for plastics subjected to exposure tests are strongly dependent on the type of exposure conditions used, the type of plastic being tested and the property being evaluated. A result obtained for one property may not be the same as that for a different property of the same material, even if the same exposure test is used. This document is not intended to establish a fixed procedure for conducting the exposure test, but is intended to provide a set of specific procedures used to express the results for change in a characteristic property of the material after it has been exposed. It is up to the user to determine which exposure conditions are most relevant to the specific material and the service conditions being used.

Test methods should be selected to determine changes in appearance and properties of the exposed material with its proposed application in mind. The exposure test used should be devised to discriminate among materials based on such changes. This document suggests typical properties that can be used to determine changes in plastics which have been subjected to exposure tests.

NOTE Because of large differences in the spectral distribution of the radiation sources used, there can be large differences in results for the same plastics exposed in the various devices described in ISO 4892 (all parts). Therefore, comparisons between plastics are intended to be made only based on results from exposures in the same type of device and under the same conditions. For optimum comparisons, plastics are expected to be exposed at the same time in the same device.

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

1 Scope

This document specifies methods to determine changes in colour and other appearance properties, and variations in mechanical or other properties, of plastics that have been exposed to glass-filtered solar radiation, to natural weathering or to simulated solar radiation from a laboratory source. The procedure used to analyse data depends on whether the test used to characterize the materials being exposed is destructive or non-destructive. The exposures are conducted under conditions described in specific exposure standards.

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 105-A01, *Textiles — Tests for colour fastness — Part A01: General principles of testing*

ISO 105-A02, *Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour*

ISO 105-A03, *Textiles — Tests for colour fastness — Part A03: Grey scale for assessing staining*

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

ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

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

ISO 11664-1, *Colorimetry — Part 1: CIE standard colorimetric observers*

ISO 11664-2, *Colorimetry — Part 2: CIE standard illuminants*

ISO 11664-3, *Colorimetry — Part 3: CIE tristimulus values*

ISO 11664-4, *Colorimetry — Part 4: CIE 1976 L*a*b* Colour space*

CIE Publication No. 15, *Colorimetry*

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:

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

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

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

3.2
file specimen
portion of the material to be tested which is stored under conditions in which it is stable, and is used for comparison between the exposed and the original state

3.3
test specimen
specific portion of the material upon which the testing is to be performed

3.4
replicate specimens
identical pieces of the test material being evaluated which are all exposed, conditioned and tested at the same time

4 Determination of changes in colour or other appearance attributes

4.1 General

When a polymeric material is exposed to 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 or artificial weathering exposure.

Chemical changes control the degradation of mechanical properties and contribute to changes in the visual appearance of polymer materials during photoageing. These chemical changes are analysed primarily by IR spectroscopy, with additional analyses using UV/visible spectroscopy during the photoageing of polymers. The analysis at this earliest stage of degradation allows the identification of the critical oxidation products, allows the stoichiometry of reactions to be checked and, in some cases, indicates weak points in the polymer material (e.g. a weakness in the specific structure of the polymer, such as a double bond, an ether group or a urethane group, unstable colorant, lack of UV stabilizers, or migration of low-molecular-mass components of formulations to the surface and their accumulation there).

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. It should be pointed out that, in some cases, oxidation products can be partially eliminated by hydrolysis, or erosion caused by water under humid climates (e.g. southern Florida) or by wind under very dry climates (e.g. Arizona). 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). In addition, these analyses can be used as a tool for developing improvements in polymers and polymeric products.

Methodologies to measure chemical changes in plastics after exposure to glass-filtered solar radiation, natural weathering or laboratory radiation sources are expressed in ISO 10640.

4.2 Changes in colour

4.2.1 Principles

Changes in colour of plastics test specimens exposed in accordance with the specific exposure standard (see Introduction) are determined by one of the following methods:

a) an instrumental method;

b) visual assessment using a scale.

4.2.2 Apparatus

4.2.2.1 Instruments for measuring colour or changes in colour, conforming to the following requirements:

— CIE 1964 standard colorimetric observer (10° observer), as specified in ISO 11664-1;

— CIE standard illuminant D65 (recommended) or A (for metamerism index), as specified in ISO 11664-2;

— CIE tristimulus values or CIE 1976 L*a*b* Colour space, as specified in ISO 11664-3 and ISO 11664-4.

For light-transmitting specimens, instruments shall conform to the requirements of CIE Publication No. 15.

4.2.2.2 Grey scale for assessing change in colour, in accordance with ISO 105-A02 or ISO 105-A03. In this scale, grade 1 corresponds to the strongest contrast, and grade 5 to zero contrast (two samples with identical colour).

NOTE The dark grey scale of ISO 105-A02 is well suited to assessing the extent of fading of relatively strong colours or deep shades. The use of the near-white grey scale of ISO 105-A03 can be found preferable for assessing the discolouration, e.g. yellowing, of white or near-white specimens.

4.2.3 Test specimens

Specimens of test and control materials shall conform to the requirements of the appropriate International Standard dealing with the specific exposure method used. Whenever possible, a control material of known weathering properties shall be included in the exposure experiment. Unless otherwise specified, at least three replicate specimens of each material being exposed shall be used.

4.2.4 Procedure

4.2.4.1 General

The specific procedure used for assessment of colour changes and any surface cleaning shall be agreed upon by all interested parties and shall be included in the test report. Determine colour changes as specified in the appropriate International Standard.

Typically, colour changes are determined at a series of exposure stages in order to evaluate the rate of colour change caused by exposure. In some cases, colour change is determined after a predetermined or specified exposure increment. Measurement or visual assessment of colour should be made as soon as possible after specimens are removed from exposure in order to minimize the effect of dark reactions, although in some cases it is preferable to condition the specimens for e.g. 24 h after removal from exposure as appearance properties assessed just after removal from exposure may vary, depending whether the specimen was removed at the end of a wet exposure period or at the end of a dry exposure period.

NOTE Because of variability in exposure results, comparison of colour changes of different materials is best done when the materials are simultaneously exposed in a single exposure device or at the same exterior location.

4.2.4.2 Instrumental assessment

Measure colour on all specimens before exposure and after each exposure stage. If required, measure colour on file specimens of each material when measuring colour on exposed specimens.

For opaque specimens, both colour and gloss may change due to ageing. When measuring the colour and determining the colour change of opaque specimens with instruments use one of the following measuring conditions:

— to get colour values independent of surface changes, use di:8° or 8°:di, according to CIE Publication No. 15 (often used for smooth specimens);

— to get best correlation to visual perception, use de:8°, 8°:de, or 45°:0°, according to CIE Publication No. 15 (often used for rough or matt specimens).

NOTE 1 If the gloss changes, the diffuse part of the surface reflexion will change and consequently the colour values measured excluding the specular reflexion change too.

NOTE 2 If the gloss changes without a visually perceptible change in colour, for example after weathering, the colour values measured including the specular reflexion will generally not be influenced.

NOTE 3 ISO 18314-1 applies to paint films but gives useful hints on the measuring procedure.

For light-transmitting specimens, follow the procedures described in CIE Publication No. 15.

NOTE 4 ASTM E1347 also describes colour measurement of light-transmitting materials.

4.2.4.3 Visual assessment

Follow the procedure described in ISO 105-A01 when determining colour change by visual assessment. Use a grey scale meeting the requirements of ISO 105-A02 or ISO 105-A03. Compare the contrast rating of the exposed specimen and file specimens using the grey scale. The rating of colour change is the grade on the grey scale which shows the same contrast as between the exposed test specimen and an unexposed file specimen of the same material.

NOTE Current information about suppliers of grey scales can be obtained from the secretariat of ISO/TC 38/SC 1.

If the contrast observed lies between two ratings on the grey scale, it can be characterized by an intermediate rating. For example, a 3-4 rating signifies that, at the given exposure stage, the contrast between the exposed test specimen and the unexposed file specimen is greater than that of rating 4 on the grey scale, but less than that of rating 3.

Report the nature of the colour change in terms of the rating on the grey scale. In addition, the type of colour change shall also be determined and reported. Use the following terms to describe changes in hue, saturation, lightness or combinations of these changes:

a) for hue changes:	more blue or less blue
	more green or less green
	more red or less red
	more yellow or less yellow
b) for saturation changes:	less intense
	more intense
c) for changes in lightness:	lighter
	darker

A typical report of colour change by visual assessment would be as follows: "more yellow, less intense, lighter, ISO 105-A02/ISO 105-A03 grey scale 2-3".

4.3 Changes in other appearance properties

In addition to colour change, other appearance properties of plastics may change as a result of exposure. Determine changes in these appearance properties in accordance with the relevant International Standards. If the method used to assess the property change is not described in an International Standard, include a description of the method used when reporting results. Examples of tests used to determine change in typical appearance properties are shown in Table 1.

Table 1 — Methods used to measure change in typical appearance properties

Property assessed	ISO standard	Quantitative data
Gloss retention	ISO 2813[a]	yes
Light transmission	ISO 13468-1	yes
Haze	ISO 14782	yes
Chalking	ISO 4628-6[a]	scale[b]
Mass		yes
Dimensions		yes
Cracking or crazing		scale[b]
Delamination		scale[b]
Warping		scale[b]
Growth of microorganisms		scale[b]
Migration of components to surface		scale[b]

[a] Methods for paints applicable to plastics.

[b] See 6.2.2 for recommended descriptive scale.

5 Determination of changes in mechanical or other properties

5.1 Principles

Surface properties of a plastic can be much more sensitive to changes caused by weathering than bulk properties. Measurement of surface properties, or material properties greatly affected by surface properties, may be more informative in evaluating rigid plastics. The mechanical or other properties measured using destructive tests are determined on several sets of specimens:

a) on specimens selected as representative of the material prior to exposure (initial property determination);

b) on test specimens exposed for a chosen period in accordance with an appropriate International Standard for the specific exposure used;

c) (if required) on file specimens stored in the dark for the same period for which the corresponding test specimens have been exposed.

It is very important that all tests be conducted using exactly the same test procedure and the same specimen-conditioning environment.

Examples of mechanical-property tests which may be used to assess the effect of exposure are shown in Table 2. Such tests yield quantitative data but are destructive so that, if it is required to follow changes through the course of the exposure, an adequate number of replicate test pieces are needed for each exposure increment.

If a property is measured with a non-destructive test, it is recommended that the property be measured on each test specimen prior to exposure and after each exposure increment. Typical properties measured using non-destructive tests include mass, dimensions, surface gloss, transmittance and haze.

Table 2 — Typical mechanical-property tests used to assess the effect of exposure on plastics

Property assessed	ISO standard
Tensile properties, particularly extension at break	ISO 527
Flexural properties	ISO 178
Impact strength	
Charpy impact strength	ISO 179 (all parts)
Izod impact strength	ISO 180
Non-instrumented puncture test	ISO 6603-1
Instrumented puncture test	ISO 6603-2
Tensile impact test	ISO 8256
Vicat softening temperature	ISO 306
Temperature of deflection under load	ISO 75-1 and ISO 75-2
Dynamic mechanical thermal analysis	ISO 6721-1, ISO 6721-3 and ISO 6721-5
Chemical changes (for example using infrared spectroscopy)	

5.2 Apparatus

The apparatus shall conform to the appropriate International Standard for the determination of the property being measured.

5.3 Test specimens

For measurement of the property of interest, test specimens shall conform to the appropriate International Standard dealing with the property measurement method. Unless otherwise specified, use at least three replicate specimens of each material being evaluated when non-destructive tests are performed. Use at least five replicates of each material being evaluated when destructive tests are performed.

NOTE For properties measured with destructive tests, exposed specimens can be in the form of a sheet from which the specimens for the particular test are cut. However, there can be differences in results between tests conducted where individual test specimens are directly exposed, and tests where individual test specimens are cut from a larger piece that has been subjected to the exposure.

Test specimens shall be conditioned after machining (see ISO 2818). In addition, it may also be necessary to precondition the sheets prior to machining to facilitate specimen preparation.

5.4 Procedure

5.4.1 Determination of initial properties

Unless otherwise specified in the relevant International Standards, condition the test specimens prior to the determination of initial properties in the standard atmosphere 23/50, class 1 and the appropriate conditioning period specified in ISO 291, or as agreed between the interested parties.

Determine the property or properties to be evaluated in accordance with the relevant International Standards, or as agreed between the interested parties (see 5.1).

5.4.2 Storage of file specimens

Store file specimens in the dark, unless otherwise specified in the standard atmospheres 23/50, class 1 according to ISO 291.

If increased moisture sensitivity is expected, storing at low relative humidity could be more useful, to avoid ageing processes during storage. Such conditions shall be agreed upon all parties.

The storage conditions used for reference specimens shall be agreed upon by all interested parties and shall be stated in the test report.

5.4.3 Determination of properties after exposure

Condition the exposed test specimens and the file specimens (see 5.1) in the standard atmosphere 23/50, class 1 or under conditions agreed upon by all interested parties. Any difference in the conditioning procedures used for initial property determination and for exposed specimens shall be included in the test report.

Using the same measurement method, determine the same property (or properties) on both exposed and, if required, file specimens as was determined on the initial test specimens (see 5.4.1).

NOTE 1 If hygroscopic properties change during ageing (e.g. due to surface accumulation of migrated additives), water content of the specimens after conditioning can be changed. For following measurements, this water absorption effect can interfere with the ageing effect to be investigated (e.g. tensile strength of polyamide material).

NOTE 2 With some tests, the results depend upon which side of the test specimen is exposed. In bending tests, for example, different results are obtained according to whether the exposed surface or the unexposed surface of the test specimen is placed under tension.

6 Expression of results

6.1 Changes in colour

6.1.1 Instrumental measurements

Using the instrumentally measured colour coordinates, determine the colour difference of each replicate specimen in accordance with ISO 11664-4, in L*a*b* colour space. Calculate the mean colour difference and the standard deviation of the mean.

NOTE Yellowing as a typical ageing effect can be directly expressed as Δb* value. Changes in ΔL lightness can often be related to formation of microcracks.

6.1.2 Visual assessment

Determine the change in colour as described in 4.2.4.3.

6.2 Changes in other appearance properties

6.2.1 Instrumental measurements

Instrumental methods used to characterize appearance properties such as gloss or transparency are typically non-destructive. When non-destructive tests are used, the property is measured on all test specimens before exposure and after each exposure increment.

If change in appearance property is measured by a non-destructive instrumental method, determine the mean and standard deviation for the property change in accordance with the procedures given in ISO 2602. For properties measured using non-destructive tests, the formulae for determining the mean and the standard deviation for the property change are given in A.1. If required, calculate the 95 % confidence interval for the property change as well.

In some cases, it may be useful to determine the percent retention of an appearance property after exposure. When percent property retention is determined using a non-destructive test, calculate the mean and standard deviation using the formulae given in A.2 after each exposure period where properties are measured.

6.2.2 Visual assessment of change in appearance attributes

Changes in appearance and surface properties which have been estimated qualitatively shall be expressed on a scale agreed between the interested parties. The following is recommended:

— none;

— barely perceptible;

— slight;

— moderate;

— substantial.

When visual assessments of appearance attributes are made, it is recommended that a comparative reference guide be used, such as photographic standards which illustrate the subjective scale.

NOTE This scale is arbitrary and is best used when one individual assesses several test specimens at the same time. Because of differences between individuals conducting visual assessments, great care is necessary in interpreting results from different observations.

6.2.3 Changes in mechanical and other properties

Determine the mechanical or other properties of each test specimen in accordance with the relevant International Standards. Determination of mechanical properties often involves destructive tests on individual specimens. When destructive tests are used, compare the results obtained for the exposed specimens with those obtained for the same property measured on file specimens. This comparison can be made in three ways:

a) Measurements of the property of interest made on all replicates from the exposed specimens are compared with measurements of the property made on a set of specimens tested prior to exposure or with measurements made on file specimens made at the same time as the test specimens.

   Comparison of data is made using analysis of variance. Unless otherwise specified, the difference in the means of the property measured on exposed and initial or file specimens shall be different at the 95 % confidence level before any change can be considered statistically significant. Use the procedure for analysis of variance given in ISO 2602.

b) The mean and standard deviation for retention of the measured property are determined using data from measurements made on each replicate of the exposed specimens compared to the mean value of the property measured in the initial measurement or on file specimens. When a property is measured with a destructive test, use the equations given in A.3 to determine the mean and standard deviation for the percent property retention.

c) The mean and standard deviation for change in the property are determined using data from measurements made on each replicate of the exposed specimens compared to the mean value of the property measured in the initial property determination or on file specimens. When a property is measured with a destructive test, use the equations given in A.4 to determine the mean and standard deviation for the change in the measured property.

For some properties, for example change in surface gloss, mass or dimensions, results can also be expressed in the form of change in property measured with non-destructive tests. When non-destructive tests are used, use the equations given in A.1 to determine the mean and standard deviation for the property change or the equations in A.2 to determine the mean and standard deviation for the percent property retention.

It may also be useful to plot change in property against time or radiant exposure, but in many cases there can be a considerable spread of results within each set of test specimens and, unless this is taken into account, misleading conclusions can be drawn from a plot of arithmetic means. For some applications, calculation of the time or radiant exposure needed to produce a defined level of the measured property is required. When this type of data is to be reported, the mean and tolerance interval for the exposure time or radiant exposure shall be determined using the procedure described in A.5.

7 Precision

The precision of the results reported in accordance with this standard depends on the precision of the specific test methods used to measure the properties evaluated and the variability of the exposure test. Therefore, no specific precision statement is possible. The procedures specified in this standard attempt to give the user an indication of the precision of the results obtained by requiring reporting both the mean and standard deviation for all test results.

8 Test report

The test report shall include the following:

a) a reference to this document;

b) all details necessary for identification of the product tested;

c) a complete description of the exposure test used, including the following:

   1) for exposure to laboratory radiation sources:

      — the type of exposure device used,

      — the radiation source and filters used,

      — a complete description of the exposure cycle (temperature, period of radiation exposure, dark exposure and moisture exposure, etc.),

      — the length of the exposure stage,

      — a reference to the relevant International Standard;

   2) for exposure to natural weathering or glass- filtered solar radiation:

      — the exposure location,

      — the dates of exposure,

      — the total exposure duration,

      — if required, the radiant exposure of exposure and the passband in which it was measured,

      — a description of the conditions in which the specimens were mounted on the exposure rack,

      — a reference to the relevant International Standard;

d) a complete description of the methods used to measure reported properties, with reference to the relevant International Standard where appropriate;

e) the conditioning procedures used (where appropriate, by reference to the relevant International Standard);

f) if applicable, the procedure used for cleaning the test specimens (see the Note);

g) the colour change, from instrumental or visual assessment, at each exposure stage;

h) the mean and standard deviation for results determined in accordance with 5.4 and/or Clause 6.

NOTE Cleaning the surface of exposed specimens can affect appearance. More information about the possible effects of cleaning specimens prior to testing can be found in Annex B.

Annex A
(normative)

Statistical formulae based on ISO 2602 for determination of mean and standard deviation and procedure for determination of time to 50 % loss of property

A.1 Calculation of mean and standard deviation for property changes measured using non-destructive tests

Calculate the change c_i for each replicate specimen using Formula (A.1):

$$c_i = x_{0,i} - x_{t,i} \quad \text{(A.1)}$$

where

$x_{0,i}$ is the initial property value;

$x_{t,i}$ is the value of the property at exposure time t.

Determine the mean property change using Formula (A.2):

$$\bar{c} = \frac{\sum_{i=1}^{n} c_i}{n} \quad \text{(A.2)}$$

where n is the number of replicates.

Calculate the standard deviation $s_{\bar{c}}$ of the mean property change using Formula (A.3):

$$s_{\bar{c}} = \sqrt{\frac{\sum_{i=1}^{n} \left(c_i - \bar{c}\right)^2}{n-1}} \quad \text{(A.3)}$$

A.2 Calculation of mean and standard deviation for percent property retention measured using non-destructive tests

Calculate the percent property retention R_i for each replicate specimen using Formula (A.4):

$$R_i = \frac{x_{t,i}}{x_{0,i}} \times 100 \quad \text{(A.4)}$$

where

$x_{0,i}$ is the initial property value;

$x_{t,i}$ is the value of the property at exposure time t.

Calculate the mean percent property retention $\bar{R}$ using Formula (A.5):

$$\bar{R} = \frac{\sum_{i=1}^{n} R_i}{n} \tag{A.5}$$

where n is the number of replicates.

Calculate the standard deviation $s_{\bar{R}}$ for the percent property retention using Formula (A.6):

$$s_{\bar{R}} = \sqrt{\frac{\sum_{i=1}^{n} \left(R_i - \bar{R}\right)^2}{n-1}} \tag{A.6}$$

A.3 Calculation of mean and standard deviation for percent property retention for properties measured using destructive tests

Calculate the mean of the measured property from the initial determination or from measurements on the file specimens, using Formula (A.7) or Formula (A.8):

When using initial property measurements

$$\bar{x}_0 = \frac{\sum_{i=1}^{n} x_{0,i}}{n} \tag{A.7}$$

where

$x_{0,i}$ is the initial property value;

n is the number of replicates.

When using measurements made on file specimens

$$\bar{x}_f = \frac{\sum_{i=1}^{n} x_{f,i}}{n} \tag{A.8}$$

where

$x_{f,i}$ is the value of the property for each file specimen;

n is the number of replicate file specimens.

Determine the percent retention $R_{e,i}$ for each of the exposed specimens using either Formula (A.9)

$$R_{e,i} = \frac{x_{e,i}}{\bar{x}_0} \times 100 \quad (A.9)$$

or Formula (A.10)

$$R_{e,i} = \frac{x_{e,i}}{\bar{x}_f} \times 100 \quad (A.10)$$

depending whether retention is expressed as a percentage of the initial value obtained for the specimens exposed or as a percentage of the value measured on file specimens, where $x_{e,i}$ is the value of the property for each exposed specimen.

Determine the mean percentage property retention $\bar{R}_e$ using Formula (A.11):

$$\bar{R}_e = \frac{\sum_{i=1}^{n} \bar{R}_{e,i}}{n} \quad (A.11)$$

where n is the number of replicate exposed specimens.

NOTE The mean percent property retention can be obtained from measurements made using non-destructive tests (see A.2) or destructive tests (this clause).

Use Formula (A.12) to determine the standard deviation $s_{\bar{R}_e}$ for percent property retention:

$$s_{\bar{R}_e} = \sqrt{\frac{\sum_{i=1}^{n} \left(R_{e,i} - \bar{R}_e\right)^2}{n-1}} \quad (A.12)$$

A.4 Calculation of mean and standard deviation for the change in property measured by destructive tests, when the change is relative to the mean of the initial values of the property or to the mean of the values obtained on file specimens

Determine the mean initial value of the property or the mean value from file specimens as described in A.3.

Use Formula (A.13) or Formula (A.14) to determine the change in property $c_{e,i}$ of each of the exposed specimens:

$$c_{e,i} = x_{e,i} - \bar{x}_0 \quad (A.13)$$

$$c_{e,i} = x_{e,i} - \bar{x}_f \quad (A.14)$$

where

$x_{e,i}$ is the value of the property for each exposed specimen;

$\bar{x}_0$ is the mean of the initial values of the property;

$\bar{x}_f$ is the mean of the values obtained on file specimens.

Determine the mean change $\bar{c}_{e,i}$ in the property using Formula (A.15):

$$\bar{c}_{e,i} = \frac{\sum_{i=1}^{n} c_{e,i}}{n} \tag{A.15}$$

where n is the number of exposed specimens.

Determine the standard deviation of the mean change in the property using Formula (A.16):

$$s_{\bar{c}_{e,i}} = \frac{\sum_{i=1}^{n} \left(c_i - \bar{c}_{e,i}\right)^2}{n-1} \tag{A.16}$$

A.5 Calculation of exposure time or radiant exposure to e.g. 50 % loss in property

Use the following procedure to determine the mean exposure time or the mean radiant energy necessary to produce the defined property change, plus the corresponding tolerance interval:

a) Prepare at least five separate sets of specimens for exposure. Each specimen set shall contain at least three replicate specimens of the material being exposed.

b) Expose all the specimen sets at the same time in a single exposure device. Remove specimen sets sequentially so that the last set removed has been exposed sufficiently to produce more than a 50 % loss in the property of interest. Remove specimens at the end of an appropriate dry period in the exposure and store specimens in the dark at 20 °C to 25 °C and 50 % relative humidity until the property has been measured.

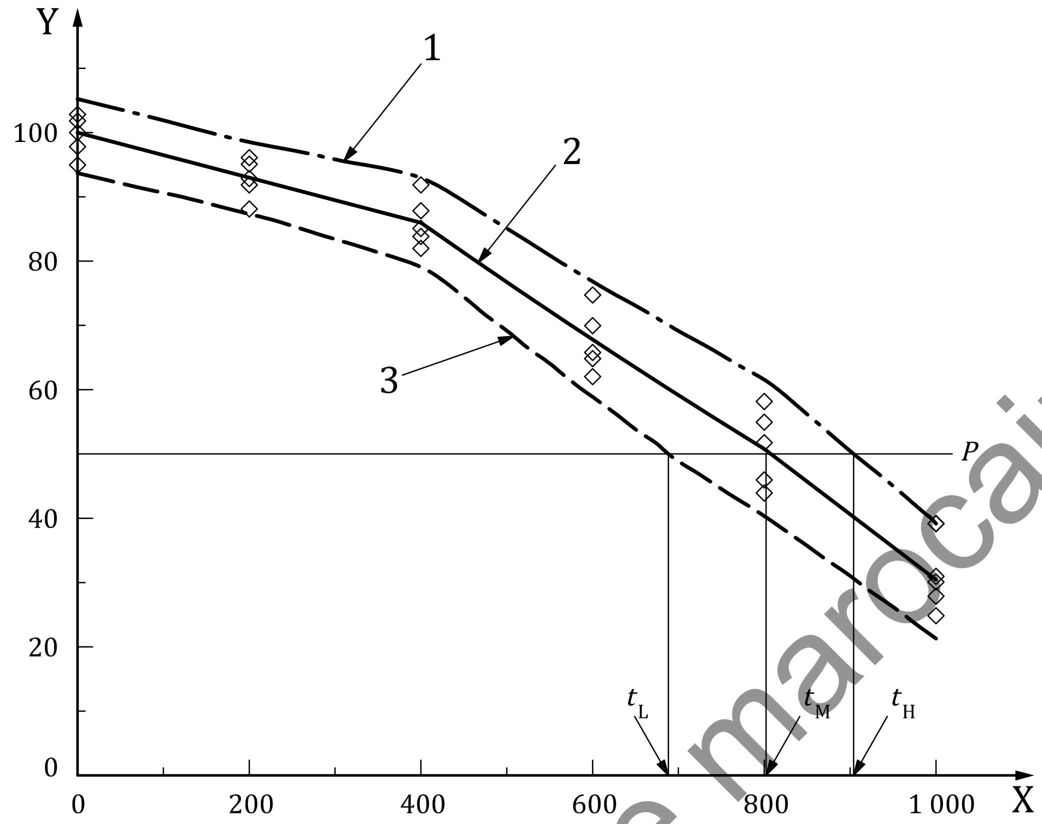
c) After each specimen set has been removed from the exposure device, determine the mean and standard deviation for the property of interest.

d) When all exposures have been completed, plot all data points, the mean, mean −2 × standard deviation and mean +2 × standard deviation for the property of interest as a function of exposure time in hours or radiant exposure.

e) Use graphical interpolation of the plots of mean −2 × standard deviation and mean +2 × standard deviation to determine the tolerance interval for the time or radiant exposure needed for the property of interest to fall to the defined value. Figure A.1 shows a typical plot and illustrates how the tolerance interval for the exposure time or radiant exposure necessary to produce a 50 % loss in the property is determined.

Report the mean and the upper and lower limits of the tolerance interval for the exposure time or radiant exposure to produce the defined change in the property.

NOTE This procedure is used for the data for a single experiment in a single laboratory. When the results from additional exposures in different laboratories are taken into consideration, the range of possible exposure times will be larger.



Key

X	time or radiant exposure (arbitrary units)
Y	level of measured property (arbitrary units)
◇	individual result for property measurement
1	upper 95 % confidence interval for property measurement results
2	mean for property measurement results
3	lower 95 % confidence interval for property measurement results
P	50 % loss in property of interest
t_L	lower limit of tolerance interval for time or radiant exposure to 50 % loss in property of interest
t_M	average time or radiant exposure to 50 % loss in property of interest
t_H	upper limit of tolerance interval for time or radiant exposure to 50 % loss in property of interest

Figure A.1 — Determination of tolerance interval for time or radiant exposure needed to produce desired property level in accelerated durability test

Annex B
(informative)

Possible effects of surface cleaning on assessment of exposure

Surfaces should preferably be cleaned using soft tissue paper soaked in distilled or deionized water with no additives and using the least abrasive/polishing action needed to be effective. However, cleaning the exposed surfaces of test specimens is likely to affect the appearance. This is particularly the case with naturally weathered specimens since the exposed surfaces are likely to become contaminated by dirt, microorganisms, etc. Cleaning is then needed to assess the underlying colour change of the test surfaces of opaque materials or the light transmission of transparent materials.

Cleaning may have a polishing effect on the surface that produces measurably increased gloss. It will also tend to remove microbiological growth together with any loosely adhering breakdown products of the material itself, the result of what is commonly known as “chalking” (see ISO 4628-6). As a result, it can be difficult to compare results between natural weathering sites or between laboratory accelerated exposures. The latter should not require cleaning to remove dirt, although chalking can occur. Accumulated dirt or chalking residues may have a protective effect on the exposed surface.

In order to examine more clearly the effects of cleaning, it is suggested that duplicate specimens be exposed, both in natural and laboratory tests. One specimen should be left uncleaned for the whole exposure, measuring, say, colour and gloss and noting any micro organic growth at the prescribed intervals. The other specimen should be measured in the uncleaned condition, then carefully cleaned and measured at the same intervals.

Bibliography

[1] ISO 75 (all parts), *Plastics — Determination of temperature of deflection under load*

[2] ISO 178, *Plastics — Determination of flexural properties*

[3] ISO 179 (all parts), *Plastics — Determination of Charpy impact properties*

[4] ISO 180, *Plastics — Determination of Izod impact strength*

[5] ISO 306, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)*

[6] ISO 527 (all parts), *Plastics — Determination of tensile properties*

[7] ISO 877 (all parts), *Plastics — Methods of exposure to solar radiation*

[8] ISO 2813, *Paints and varnishes — Determination of gloss value at 20°, 60° and 85°*

[9] ISO 2818, *Plastics — Preparation of test specimens by machining*

[10] ISO 4628-6, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 6: Assessment of degree of chalking by tape method*

[11] ISO 4892 (all parts), *Plastics — Methods of exposure to laboratory light sources*

[12] ISO 6603-1, *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 1: Non-instrumented impact testing*

[13] ISO 6603-2, *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 2: Instrumented impact testing*

[14] ISO 6721-1, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

[15] ISO 6721-3, *Plastics — Determination of dynamic mechanical properties — Part 3: Flexural vibration — Resonance-curve method*

[16] ISO 6721-5, *Plastics — Determination of dynamic mechanical properties — Part 5: Flexural vibration — Non-resonance method*

[17] ISO 8256, *Plastics — Determination of tensile-impact strength*

[18] ISO 13468-1, *Plastics — Determination of the total luminous transmittance of transparent materials — Part 1: Single-beam instrument*

[19] ISO 14782, *Plastics — Determination of haze for transparent materials*

[20] ISO 18314-1, *Analytical colorimetry — Part 1: Practical colour measurement*

[21] ASTM E 1347, *Standard Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry*

