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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

Plastiques — 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

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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.