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

Plastiques — Détermination des changements de coloration et des variations de propriétés après exposition à la lumière du jour sous verre, aux agents atmosphériques ou aux sources lumineuses de laboratoire



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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4582 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 4582:1980), which has been technically revised.

Annex A forms an integral part of this International Standard. Annex B is for information only.

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Introduction

A number of different exposure techniques can be used to provide information on the effects of environmental stresses such as light, heat and water on plastics (see ISO 877 and ISO 4892). 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 standard 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 test 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 consideration for its proposed application. The exposure test used should be devised to discriminate among materials based on such changes. This standard suggests typical properties that can be used to determine change in plastics which have been subjected to exposure tests.

NOTE — Because of large differences in the spectral distribution of the light sources used, there can be large differences in results for the same plastics exposed in the various devices described in ISO 4892. Therefore, comparisons between plastics should only be made based on results from exposures in the same type of device and under the same conditions. For optimum comparisons, plastics should be exposed at the same time in the same device.

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

1 Scope

This International Standard describes procedures used to determine changes in colour and other appearance properties, and variations in mechanical or other properties, of plastics that have been exposed to daylight behind glass, natural weathering or light 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 105-A01:1994, *Textiles — Tests for colour fastness — Part A01: General principles of testing.*

ISO 105-A02:1993, *Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour.*

ISO 105-A03:1993, *Textiles — Tests for colour fastness — Part A03: Grey scale for assessing staining.*

ISO 178:1993, *Plastics — Determination of flexural properties.*

ISO 179 (both parts), *Plastics — Determination of Charpy impact properties.*

ISO 180:—¹⁾, *Plastics — Determination of Izod impact strength.*

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 306:1994, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST).*

ISO 527 (all parts), *Plastics — Determination of tensile properties.*

1) To be published. (Revision of ISO 180:1993)

ISO 877:1994, *Plastics — Methods of exposure to direct weathering, to indirect weathering using glass-filtered daylight, and to intensified weathering by daylight using Fresnel mirrors.*

ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval.*

ISO 2813:1994, *Paints and varnishes — Determination of specular gloss of non-metallic paint films at 20°, 60° and 85°.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 4628-6:1990, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 6: Rating of degree of chalking by tape method.*

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

ISO 6603-1:—²⁾, *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 1: Non-instrumented impact test.*

ISO 6603-2:—³⁾, *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 2: Instrumented puncture test.*

ISO 6721-1:1994, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles.*

ISO 6721-3:1994, *Plastics — Determination of dynamic mechanical properties — Part 3: Flexural vibration — Resonance-curve method.*

ISO 6721-5:1996, *Plastics — Determination of dynamic mechanical properties — Part 5: Flexural vibration — Non-resonance method.*

ISO 7724 (all parts), *Paints and varnishes — Colorimetry.*

ISO 8256:1990, *Plastics — Determination of tensile-impact strength.*

ISO 13468-1:1996, *Plastics — Determination of the total luminous transmittance of transparent materials — Part 1: Single-beam instrument.*

ISO 14782:—⁴⁾, *Plastics — Determination of haze of transparent materials.*

NOTE — The above list is not exhaustive and other standard methods for the determination of properties may also be suitable (see 5.1).

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 control: A 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: A 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.

2) To be published. (Revision of ISO 6603-1:1985)

3) To be published. (Revision of ISO 6603-2:1989)

4) To be published.

3.3 masked area: A portion of the exposed specimen which is protected from light exposure by masking. The masked area is not protected from heat and moisture.

3.4 test specimen: A specific portion of the material upon which the testing is to be performed.

3.5 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 Changes in colour

4.1.1 Principles

Changes in colour of plastics test specimens exposed in accordance with the specific exposure standard (see clause 2) are determined by one of the following methods:

- a) instrumental methods;
- b) visual assessment using a scale.

4.1.2 Apparatus

4.1.2.1 Instruments for measuring colour or changes in colour, conforming to the requirements specified in ISO 7724.

4.1.2.2 Grey scale for assessing change in colour in accordance with ISO 105-A02 or ISO 105-A03 (see also annex B to this International Standard). 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 may be found preferable for assessing the discolouration, e.g. yellowing, of white or near-white specimens.

4.1.3 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 (see clause 2). 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.

4.1.4 Procedure

4.1.4.1 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.

NOTES

1 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.

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

4.1.4.2 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. Follow the procedures described in ISO 7724 when measuring colour and determining colour change with instruments.

4.1.4.3 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/A03 grey scale 2-3".

4.2 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 relevant International Standards. If the method used to assess 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.

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.

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

Property assessed	ISO standard	Quantitative data
Gloss retention	ISO 2813 ¹⁾	yes
Light transmission	ISO 13468-1	yes
Haze	ISO 14782	yes
Chalking	ISO 4628-6 ¹⁾	scale ²⁾
Mass		yes
Dimensions		yes
Cracking or crazing		scale ²⁾
Delamination		scale ²⁾
Warping		scale ²⁾
Growth of microorganisms		scale ²⁾
Migration of components to surface		scale ²⁾

1) Methods for paints applicable to plastics.
2) See 6.2.2 for recommended descriptive scale.

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
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
Dynamic mechanical thermal analysis	ISO 6721, parts 1, 3 and 5
Chemical changes (for example infrared spectroscopy)	

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.

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 used. Use at least five replicates of each material being evaluated when destructive tests are used.

NOTE — For properties measured with destructive tests, exposed specimens may be in the form of a sheet from which the specimens for the particular test have to be cut. However, there may 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.

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, condition the test specimens prior to the determination of initial properties in one of the atmospheres and using the tolerances and the appropriate period specified in ISO 291, or as agreed between the interested parties. For hygroscopic plastics, comparative testing of moisture-sensitive properties (i.e. mechanical, electrical) should be done after the samples have been brought to the same moisture content, preferably the one that corresponds to the equilibrium at 50 % RH and 23 °C.

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 under normal laboratory conditions, using one of the standard atmospheres specified in ISO 291. Store moisture-sensitive materials in an atmosphere that will not produce changes due to water absorption. This can be accomplished by storing at low relative humidity or in a moisture-proof container. 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) under the same conditions as those used for the determination of the initial properties (see 5.4.1) 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 — 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 7724-3. Calculate the mean colour difference and the standard deviation of the mean.

6.1.2 Visual assessment

Determine the change in colour as described in 4.1.4.3.

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 clause 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 clause A.2 at 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 to use a comparative reference guide 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.

6.3 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 clause 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 clause 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 clause A.1 to determine the mean and standard deviation for the property change or the equations in clause 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 energy, 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 clause 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) reference to this International Standard;
- 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 light sources
 - the type of exposure device used,
 - the light source and filters used,
 - a complete description of the exposure cycle (temperature, period of light 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 daylight behind glass
 - the exposure location,

- the dates of exposure,
 - the total exposure time,
 - if required, the radiant energy of exposure and the bandpass 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 appropriate 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.

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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 as follows:

$$c_i = x_{0,i} - x_{t,i}$$

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 \bar{c} using the equation

$$\bar{c} = \frac{\sum_{i=1}^n c_i}{n}$$

where n is the number of replicates.

Calculate the standard deviation $s_{\bar{c}}$ of the mean property change using the equation

$$s_{\bar{c}} = \sqrt{\frac{\sum_{i=1}^n (c_i - \bar{c})^2}{n-1}}$$

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 as follows:

$$R_i = \frac{x_{t,i}}{x_{0,i}} \times 100$$

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} as follows:

$$\bar{R} = \frac{\sum_{i=1}^n R_i}{n}$$

where n is the number of replicates.

Calculate the standard deviation $s_{\bar{R}}$ for the percent property retention as follows:

$$s_{\bar{R}} = \sqrt{\frac{\sum_{i=1}^n (R_i - \bar{R})^2}{n-1}}$$

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 the following equations:

When using initial property measurements

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

where

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

n is the number of replicates.

When using measurements made on file specimens

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

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 the equation

$$R_{e,i} = \frac{x_{e,i}}{\bar{x}_0} \times 100$$

or the equation

$$R_{e,i} = \frac{x_{e,i}}{\bar{x}_f} \times 100$$

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 the equation

$$\bar{R}_e = \frac{\sum_{i=1}^n R_{e,i}}{n}$$

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 clause A.2) or destructive tests (this clause).

Use the following equation to determine the standard deviation $s_{\bar{R}_e}$ for percent property retention:

$$s_{\bar{R}_e} = \sqrt{\frac{\sum_{i=1}^n (R_{e,i} - \bar{R}_e)^2}{n-1}}$$

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 clause A.3.

Use one of the following equations to determine the change in property $c_{e,i}$ of each of the exposed specimens:

$$c_{e,i} = x_{e,i} - \bar{x}_0$$

$$c_{e,i} = x_{e,i} - \bar{x}_f$$

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 in the property $\bar{c}_{e,i}$ using the equation

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

where n is the number of exposed specimens.

Determine the standard deviation of the mean change in the property using the equation

$$s_{\bar{c}_{e,i}} = \sqrt{\frac{\sum_{i=1}^n (c_{e,i} - \bar{c}_{e,i})^2}{n-1}}$$