
**Plastics — Determination of apparent
activation energies of property changes
in standard weathering test methods**

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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).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Temperature is an important influencing factor on correlation and acceleration in lightfastness and weathering testing. This is acknowledged in international general weathering standards like ISO 4892-1 or ISO 16474-1.

The Arrhenius concept describes the influence of temperature on the reaction rate of chemical reaction. This model is also used (with limitations) to describe the reaction rate of photochemical/weathering reactions. Several assumptions and limitations should be taken into account; the Arrhenius concept might not be sufficient to describe complex degradation behaviours. However, this basic approach might help to better understand the influence of temperature on weathering degradation of polymeric materials.

An important material specific property to describe the temperature influence on degradation reactions is the activation energy E_a , which describes the required energy barrier for a chemical reaction to occur. E_a is only valid for a specific material and a specific degradation pathway. In weathering, this degradation pathway is usually attributed to a specific property change.

The Arrhenius principle is applied to determine the temperature dependency of the thermal degradation of polymers. The thermal activation energy can be calculated based on ISO 11358-2. There is no such standard to determine the activation barrier for weathering or photo-chemical degradation of polymers.

Limited information is available on the activation energies of photochemical degradation/weathering reactions. To determine activation energies, weathering experiments at least at two different temperatures are required. Due to this high effort, available activation energies are often calculated based on unsuitable weathering experiments (for example based on outdoor weathering and accelerated weathering).

There are some basic test requirements which help to increase the significance of the Arrhenius concept and the determination of the activation energy in accelerated weathering. These requirements are described in this document.

NOTE 1 Different property changes, for example yellowing and surface cracking of one material can have different activation energies.

NOTE 2 In this approach, the activation energy is considered as temperature independent for the selected temperature range.

The determination of activation energies of photo-chemical degradation processes allows including the temperature into the evaluation and correlation of different weathering experiments as quantitative factor. Knowing the activation energy improves the reliability and predictive value of artificial weathering.

NOTE 3 The use of the Arrhenius concept in artificial weathering simplifies complex chemical degradation processes and is therefore, in this context, a limited model to estimate the temperature dependency in weathering. However, by following some basic experimental preconditions, the relevance of the estimates can be increased.

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1 Scope

This document describes a test method to determine the activation energy (E_a) of photochemical degradation reactions in accelerated weathering tests according to the Arrhenius model in the most comprehensive manner.

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 4582, *Plastics — Determination of changes in colour and variations in properties after exposure to glass-filtered solar radiation, natural weathering or laboratory radiation sources*

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

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

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

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

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3 Terms, definitions and abbreviated terms

3.1 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:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

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

3.1.1

activation energy

E_a

energy, above that of the ground state, which is to be added to an atomic or a molecular system to allow a particular process to take place

Note 1 to entry: It is expressed in $\text{J}\cdot\text{mol}^{-1}$.

Note 2 to entry: The activation energy, E_a , is derived from the Arrhenius concept (see [Annex A](#)).

3.1.2

effective irradiance

E_{eff}

spectral irradiance triggering the relevant photo-degradation processes for the reactions under consideration

$$E_{\text{eff}} = \int E_{\lambda} \cdot s_x(\lambda) d\lambda$$

where

E_{λ} is the spectral irradiance in $W \cdot m^{-2} \cdot nm^{-1}$;

$s_x(\lambda)$ is the action spectrum normalized to its maximum.

Note 1 to entry: If different test conditions are compared using radiation sources with the same spectral irradiance distribution, often the ratio of the total irradiances is used instead of the ratio of the effective irradiance.

3.2 Abbreviated terms

CHT chamber air temperature (ambient air temperature)

BST black standard temperature (measured with insulated surface temperature sensor)

BPT black panel temperature (measured with un-insulated surface temperature sensor)

WST white standard temperature (measured with insulated surface temperature sensor)

WPT white panel temperature (measured with un-insulated surface temperature sensor)

4 Principle

The Arrhenius formula deals with effects of varying levels of temperature on property change rates caused by exposure to solar radiation. This concept is used in the thermal degradation of plastics, for example in ISO 11358-2 and ISO 2578. To determine the activation energy, E_a , two or more weathering experiments shall be performed at different temperatures, but under otherwise equal conditions, until the same specified property change is reached. [ISO 23706:2020](https://standards.iteh.ai/catalog/standards/sist/73ea2852-b7ce-4f98-9a63-de0b1d1bde5c/iso-23706-2020)

The only difference between the exposures shall be the specimen surface temperature. All other parameters (spectral irradiance, relative humidity, cycles, etc.) shall be the same in all exposures. If the specimen surface temperature cannot be measured directly and/or controlled, a reference surface temperature (BST/BPT or WST/WPT, as similar to the relevant specimen surface temperature as possible) shall be the only varying parameter instead.

Different radiation sources can be used.

If radiation sources are used which emit IR radiation (see [Table 1](#), Type A-1, e.g. Xenon arc according to ISO 4892-2), the specimen will show an inhomogeneous temperature distribution over the thickness, due to radiation heating. In such case, only thin samples should be investigated, or the considered property change should be related to the specimen surface, not to the bulk. If radiation heating occurs, vary the surface temperature by values of about 5 K to 10 K, as the systematic errors during surface temperature measurement can cancel each other out.

If radiation sources are used which do not emit IR radiation, but the specimen are cooled from the backside (see [Table 1](#), Type A-2, e.g. UVA according to ISO 4892-3), the specimen will show an inhomogeneous temperature distribution over the thickness. In that case the same limitations as above apply.

If radiation sources are used which do not emit IR radiation and the specimen are placed inside a climatic chamber with homogeneous temperature distribution (see [Table 1](#), Type B e.g. UVA-340 according to ISO 4892-3), the specimen will show a constant temperature over the thickness. Thus, also for thicker samples, bulk properties can be investigated. In such case, temperature can be varied in a larger range. Doing so, it should be taken into account that the activation energy, E_a , can vary over the temperature, due to differently relevant processes.

5 Apparatus

5.1 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, 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.

For the test equipment (including chamber design, laboratory radiation source, radiometer, temperature and humidity control equipment) the criteria as described in ISO 4892-1 and ISO 4892-2 (for Xenon-arc equipment) and ISO 4892-3 (for fluorescent UV devices) apply.

5.2 Surface temperature measurement

Equipment utilizing sample surface temperature sensors according to EN 16795 should be used. However, if no direct specimen surface temperature measurement is possible, the black standard or black panel sensors should be used instead as reference for dark test specimen and white standard or white panel sensors should be used as reference for light coloured test specimen.

NOTE Surface and bulk temperature of the specimens will typically adjust between CHT or WST and BST, depending on possible radiation heating, mounting position, or thermal properties of the specimens.

6 Test specimens

The test specimens shall be according to ISO 4892-1.

7 Test method

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7.1 Test conditions

Select a standard test method or create a test method as appropriate for the required material (e.g. ISO 4892-2, Cycle 1 for outdoor weathering of plastics).

The test parameters are:

- a) relative spectral irradiance (preferred simulated solar radiation according to 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, if specified/necessary.

NOTE 1 Rain or wetting cycles influence the specimen temperature. If rain cycles are used, the effective specimen temperature is used for evaluation (see [Annex C](#)).

NOTE 2 Rain cycles can increase the uncertainty of the method.

Test at conditions described in the selected test method.

7.2 Test duration

The test duration is either defined by the test standard used or as agreed between all interested parties (test duration, or until a specific property change is achieved).

The evaluation criteria are either defined by the test standard used or as agreed between all interested parties. The evaluation criteria should be determined after the exposure

For determination of Arrhenius factors, the sample surface temperature is the critical temperature. The sample surface temperature shall be measured or estimated according to 5.2.

7.3 Increased/decreased temperatures

Test at a reference temperature of, for example, -10 K, -5 K, +5 K, +10 K, or other temperature ranges compared to standard conditions (e.g. according to ISO 4892-2, Cycle 1). For dark materials, BST or BPT can be a suitable reference temperature. For light coloured materials WST or WPT as reference is recommended.

All other parameters [irradiance (*E*), radiant exposure (*H*), relative humidity (*RH*), rain cycle] shall be the same as described under standard conditions.

The test at increased or decreased temperatures shall be performed until the same property change is achieved as described under standard conditions.

For determination of the activation energy, E_a , the sample surface temperature is the critical temperature. The surface temperature shall be measured or estimated according to 5.2.

For the determination of the activation energy, E_a , the test shall be performed at standard conditions and at least at one increased or decreased temperature or at least two different temperatures within a range where no temperature influence on the degradation pathway is expected (no T_g transition).

Table 1 — Classification of instrument types with and without radiation heating and test specimen with inhomogeneous (Type A) and homogeneous (Type B) specimen temperature

Instrument Type		Suggested ΔT K	Temperature control by	Constant parameters
A-1	Radiation heating (e.g. Xenon arc)	e.g. $\pm 5, \pm 10$	Adjusting convection (blower speed) and/or control of IR radiation	E, E_λ, RH , rain cycle
A-2	No radiation heating (e.g. UVA), backside cooling	e.g. $\pm 5, \pm 10$	Adjusting CHT	E, E_λ, RH , rain or condensation cycle
B	No radiation heating (e.g. UVA), homogeneous specimen temperature	e.g. ± 20	Adjusting CHT	E, E_λ, RH , rain cycle

7.4 Calculation of activation energy, E_a

7.4.1 Exposures at two different temperatures

Calculate the activation energy, E_a , on the basis of two weathering tests according to the logarithmic Arrhenius formula shown in Formula (A.3).

7.4.2 Exposures at two or more different temperatures

Determine the activation energy, E_a , on the basis of two or more weathering tests graphically. When the natural logarithm of k is plotted versus the inverse of the temperature ($1/T$), the slope is a straight line with a value equal to $-E_a/R$ [according to [Formula \(A.4\)](#) and [Figure A.1](#)].

NOTE 1 The determination of E_a becomes more reliable, the more data points from different temperatures are used.

NOTE 2 The correlation coefficient can be calculated by Pearson correlation according to ISO 2578:1993, Annex B for data sets with more than three data points.

8 Exposure conditions

Follow the exposure conditions described in the selected test method or standard. Control the test parameters as defined in the selected test method or standard or according to ISO 4892-1, ISO 4892-2 or ISO 4892-3.

9 Procedure

9.1 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.

9.2 Mounting the test specimens

Attach the specimens to the specimen holders in the equipment in such a way 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 control, 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 control specimens stored in the dark.

9.3 Exposure

Before placing the specimens in the test chamber, be sure that the apparatus is operating under the desired conditions. Programme 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.

9.4 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 ($J \cdot m^{-2}$), in the wavelength band from 300 nm to 400 nm, or in joules per square metre per nanometre ($J \cdot m^{-2} \cdot nm^{-1}$) at the wavelength selected (e.g. 340 nm).