TECHNICAL REPORT

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Second edition
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Plastics — Use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor weathering conditions

Plastiques — Utilisation d'éprouvettes de référence en polyéthylène pour l'évaluation des conditions de vieillissement climatique

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

For an explanation of 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 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 second edition cancels and replaces the first edition (ISO/TR 19032:2006), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the shipping address of PERS in 3.1 has been cancelled;

— a cautionary sentence has been added in Clause 4.

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

The method described in this document demonstrates the use of polyethylene reference specimens (PERS) for monitoring conditions in weathering tests used for plastics. PERS has double bonds in its molecular structure, which are easily oxidized to produce carbonyl groups. The change in carbonyl index of PERS is produced by the combined effects of ultraviolet (UV) and temperature. Therefore, the carbonyl groups proportionally increase, depending on the received UV and temperature. Based on this relationship, the effect of UV radiation and temperature on PERS can be expressed quantitatively. For laboratory-accelerated exposures, PERS is also sensitive to changes in the chamber air temperature. The effect of moisture was not determined in the study.

Plastics — Use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor weathering conditions

1 Scope

This document describes a method that demonstrates the use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor conditions in weathering tests used for plastics.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Background information

Degradation of plastics in an outdoor environment is mainly influenced by the ultraviolet radiation received, environmental temperature, moisture, etc. Especially in photo-oxidation induced from ultraviolet radiation, temperature plays a very important role. Measuring the ultraviolet radiation during the exposure period is useful for comparison of the result of the exposure test, but it is not enough to compare the exposure results. Therefore, it is very important to find some index that can be used to evaluate the complex effect of received ultraviolet radiation and environmental temperature. PERS is used to characterize the level of combined effect of ultraviolet radiation and temperature, and its characteristic proportionally increases depending on the UV radiation and temperature received.

NOTE The test results presented in this document were generated with PERS from Japan Weathering Test Center 1-3-7 Shibakoen Minatoku Tokyo Japan. This material is no longer available. No new material has been qualified to be used as a replacement in this document at the time of its publication.

5 Material

5.1 General

PERS is high-density polyethylene polymerized using molybdenum dioxide as a catalyst, containing the trans-form vinylene group. Other basic properties are as follows:

— absorbance ratio of trans-form vinylene group to methylene group: 1,0 to 1,3;

— melt flow rate (2,16 kg, 190 °C): 0,2 g to 0,4 g/10 min;

— density: 950 kg/m^3 to 965 kg/m^3;

— thickness: (0,2 ± 0,02) mm.

5.2 Preparation of PERS

After kneading for 5 min, the material between two rolls whose surfaces are heated at 150 °C to 170 °C are cut into small pieces of 0,4 g to 0,5 g.

After pre-heating for 90 s in a compression moulding machine with a surface that is heated at 160 °C to 180 °C, the material is compressed for 60 s, cooled in a compression moulding machine with a surface temperature of 30 °C to 40 °C for 60 s, and the press sheet of the thickness mentioned above is prepared.

6 Procedure

6.1 Method for measuring the carbonyl index of PERS

An infrared (IR) spectrophotometer should be used as the measuring apparatus.

The infrared absorption spectra is measured after irradiation, in the range of 2 200 cm^{-1} to 1 600 cm^{-1}. In this case, the same method for a quantitative analysis is used for the scanning speed.

The carbonyl index is determined in accordance with Formula (1), based upon infrared absorbance spectra of exposed PERS. Absorbance at near 2 020 cm^{-1} peak is employed as an internal standard to correct for sample film thickness, while absorbance at near 1 715 cm^{-1} peak is used to indicate carbonyl group content. A typical diagram of IR absorbance is shown in Figure 1.

$$A_r = \frac{A_{1\,715}}{A_{2\,020}} \tag{1}$$

where

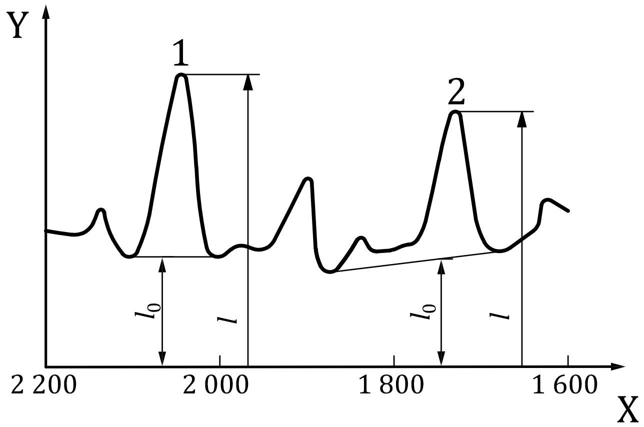
A_r is the absorbance ratio (carbonyl index);

$A_{1\,715}$ is the absorbance at near 1 715 cm^{-1} $(I - I_0)$;

$A_{2\,020}$ is the absorbance at near 2 020 cm^{-1} $(I - I_0)$;

I_0 is the absorbance measured by the base-line method at individual wave number;

I is the absorbance at the peak of individual wave number.



Key

Y absorbance

X wavenumber (cm^{-1})

1 peak near 2 020 cm^{-1}

2 peak near 1 715 cm^{-1}

Figure 1 — IR absorbance diagram showing base line and peak absorbance

6.2 Round robin test of laboratory light-source exposure devices with PERS

6.2.1 General

It is well known that the degradation of plastics materials or products used outdoors will occur by the combined effect of ultraviolet radiation and temperature, or other factors. It is also recognized that the reproducibility in the laboratory light-source exposure test will vary with the change in ultraviolet spectrum distributions and the chamber temperatures caused by deterioration of lamps and filters with operating time, even if ultraviolet radiation and black standard temperature (BST) or black panel temperature (BPT) are under constant conditions.

Since PERS can quantitatively evaluate, as carbonyl index, the combined effect of UV radiation and temperature, the carbonyl index obtained can reflect changes in a given environment.

In order to verify the repeatability and reproducibility of the specimens and exposure test, the round robin test (RRT) using PERS by laboratory light-source exposure devices in ISO/TC 61/SC 6 was conducted.

6.2.2 Xenon-arc-lamp exposure

The test conditions were according to ISO 4892-2[1]. The conditions are shown in Table 1. It was not requested to control the chamber temperature, but participants have been requested to report this temperature.

Each participant has been provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 1 — Exposure conditions for xenon-arc lamp

Filter	daylight filter
Irradiance	0,5 W/(m^2·nm) at 340 nm or 60 W/m^2 (300 nm to 400 nm)
BST or BPT	(65 ± 3) °C for BST or (63 ± 3) °C for BPT
Chamber temperature	Arbitrary
Water spray	102 min of light only followed by 18 min of light plus water spray
Humidity	(50 ± 5) %
Period	24 h, 48 h, 72 h and 96 h

6.2.3 Open-flame carbon-arc-lamp exposure

The test conditions were according to ISO 4892-4[2]. The conditions are shown in Table 2. The chamber temperature was not specified, but participants were requested to report this temperature.

Each participant has been provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 2 — Exposure conditions for open-flame carbon-arc lamp

Filter	Type 1 (Type 1 known as Corex 7058 filter)
BST or BPT	(65 ± 3) °C for BST or (63 ± 3) °C for BPT
Chamber temperature	Arbitrary
Water spray	102 min light only followed by 18 min of light plus water spray
Humidity	(50 ± 5) %
Period	24 h, 48 h, 72 h and 96 h

6.2.4 Fluorescence lamp exposure

The test conditions were according to ISO 4892-3[3]. The conditions are shown in Table 3. Irradiance was not specified at any intensity.

Each participant was provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 3 — Exposure conditions for fluorescent lamp

Lamp type	UVA340
Irradiance	Arbitrary
Mode	Mode 1: 4 h of dry UV exposure followed by 4 h of condensation
BPT	(63 ± 3) °C at UV exposure and (50 ± 3) °C at condensation
Period	8 h, 24 h, 32 h and 48 h

After each sample had been exposed for each exposure period, the carbonyl index was determined, based upon the method described in 6.1.

6.3 Outdoor exposure test of PERS

The result of the outdoor exposure test varies, even if it is conducted in the same place, because of differences due to the seasonal climate changes. Although it is useful to measure the amount of ultraviolet radiation for comparison of exposure tests, it is not enough in the comparison only to consider the amount of ultraviolet radiation, because plastics are influenced not only by ultraviolet radiation but by temperature or by moisture. Since PERS is influenced by the combined effect of ultraviolet radiation and temperature, PERS have been exposed in various places where the climate was different.

Six locations in different climates and different countries were selected: Sapporo, Choshi, Miyakojima (Japan), Serpong, Bandung (Indonesia) and Phoenix (USA). Locations and exposure angles are shown in Table 4.

Table 4 — Locations and exposure angles

Exposure site	Exposure angle	Latitude
Sapporo (Japan)	45° South	43° 03′ N
Choshi (Japan)	30° South	35° 43′ N
Miyakojima (Japan)	20° South	24° 44′ N
Serpong (Indonesia)	5°[a] South and north	6° 15′ S
Phoenix (USA)	34° South	33° 54′ N

[a] From November to February, the samples face south; and from March to October, they face north.

The conditions of the outdoor exposure test are based on the relevant ISO 877 part[4]. Three pieces of PERS were exposed for 1 month. By replacing exposed PERS with new ones, the exposure test was repeated in the following months successively. The exposure test was repeated successively for more than 24 months.

The practical procedure of the outdoor exposure test is shown below.

1) The conditions of the outdoor exposure test are based on the relevant ISO 877 part.

2) Prepare a minimum of three PERS and expose them for 1 month. It is desirable to expose them at the beginning of a calendar month, in order to evaluate the condition of the month.

3) By replacing exposed PERSs with new ones, the exposure test is repeated in the following months successively.

4) The exposure test is repeated successively for at least 12 months.

5) The accumulated value of the carbonyl index for each month will be adopted for the index of combined effect of UV radiation and temperature at the site of exposure.

6.4 Consistency of laboratory light-source exposure devices

It is recognized that changes in the characteristics of lamps and filters with time, and changes in the chamber temperature of laboratory light-source exposure devices, influence the reproducibility and repeatability of test results, even if the test is operated under constant conditions of ultraviolet radiation and BST or BPT.

Since the carbonyl index of a polyethylene reference specimen is proportional to the environment where ultraviolet radiation and temperature are compounded, consistency of the exposure environment could be monitored with this reference material.

In order to verify the correct operation of a particular xenon-arc-lamp device, the control limit was determined by PERS.

The test conditions were according to ISO 4892-2. For different operating times of lamp and filters, exposure was repeated three times.

The practical procedure was carried out as follows.

1) The conditions of the laboratory light-source exposure test are based on the relevant ISO 4892 part.

2) PERS should be irradiated for about 100 h. The number of PERSs should not be less than three.

