## INTERNATIONAL STANDARD

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### Plastics — Polypropylene and propylene-copolymers — Determination of thermal oxidative stability in air — Oven method

Plastiques — Polypropylène et copolymères de propylène — Détermination de la stabilité à l'oxydation à chaud dans l'air —

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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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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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 <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>. (standards.iteh.ai)

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This second edition cancels and replaces the first edition (ISO 4577:1983), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

- the normative references have been updated;
- the mandatory <u>Clause 3</u> "Terms and definitions" has been added the subsequent clauses have been renumbered.

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 <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

# Plastics — Polypropylene and propylene-copolymers — Determination of thermal oxidative stability in air — Oven method

#### 1 Scope

This document specifies a method for the determination of the resistance of moulded test specimens of polypropylene and propylene-copolymers to accelerated ageing by heat in the presence of air using a forced draught oven.

The method represents an attempt to estimate the service life of parts fabricated from propylene plastics.

The stability determined by this method is not directly related to the suitability of the material for use when different environmental conditions prevail.

NOTE The specified thermal levels are considered sufficiently severe to cause failure of commercial grades of heat-stable propylene plastics within a reasonable period of time. If desired, lower temperatures can be applied to estimate the performance of propylene plastics with lower heat stabilities.

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#### 2 Normative references

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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 291, Plastics — Standard atmospheres for conditioning and testing

ISO 19069-2, Plastics — Polypropylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties

#### 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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>

#### 4 Principle

Accelerated ageing of test specimens is performed by heat in the presence of air using a forced draught oven. Visual examination and determination of the time to failure is done.

Under the severe conditions of this test, the specimens undergo degradation at a rate dependent upon the thermal endurance of the propylene plastic under examination.

For the purpose of this document, the time to failure of the material is taken as the number of days after which the specimen shows localized crazing, crumbling and/or discoloration.

If a more reliable estimate of the life-temperature relationship of propylene plastics is required, the test may be conducted at several temperatures and the data interpreted through use of the Arrhenius

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relation, by plotting the logarithms of times to failure against the reciprocals of the temperatures in kelvins. Temperatures in the range from 100 °C to 150 °C, with intervals of 10 °C, are suggested for this purpose.

#### 5 Apparatus

#### 5.1 Oven

The oven shall be of mechanical convection type, capable of controlled circulation of air, with adjustable air intake and exhaust, equipped with a specimen holder and a temperature control system capable of adjustment to meet the following conditions.

- a) Exhaust rate: at least one oven-chamber volume in 10 min.
- b) Air velocity: from 0, 75 m/s to 1 m/s at any oven position occupied by the test specimens.
- c) Temperature control: range up to 200 °C and with control throughout the working range to the nearest 1 °C. The temperature control shall include a device to prevent temperature overrides. It is recommended that a device be used for recording the temperature inside the oven.

#### 5.2 Thermometers

Thermometers shall cover the temperature range up to 200 °C, graduated in 1 °C intervals or better. Each thermometer shall be tested for bulb stability and shall be calibrated at 150 °C.

In order to control the homogeneity of the temperature in the oven, it is recommended that at least two thermometers be used, one in the upper part of the working volume of the oven, and one in the lower part. The test temperature is the mean value of the indications of the two thermometers.

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## **5.3 Specimen holder** https://standards.iteh.ai/catalog/standards/sist/8d785105-bd68-4547-a8b7-71cffe974a4c/iso-4577-2019

The specimen holder may be biaxially rotated, uniaxially rotated or stationary, provided that the test specimens are in a stream of air having a relative velocity between 0,75 m/s and 1 m/s.

Illustrations of suitable apparatus for biaxially and uniaxially rotated specimen holders are shown in Figures 1 and 2, respectively.

Biaxial rotation increases the probability that all specimens will be exposed similarly. In case of dispute, the use of biaxial rotation shall be the reference method.

#### 5.3.1 Biaxially rotated specimen holder (see Figure 1)

The frequency of rotation about the horizontal and vertical axes shalt be 1 min<sup>-1</sup> to 3 min<sup>-1</sup>.

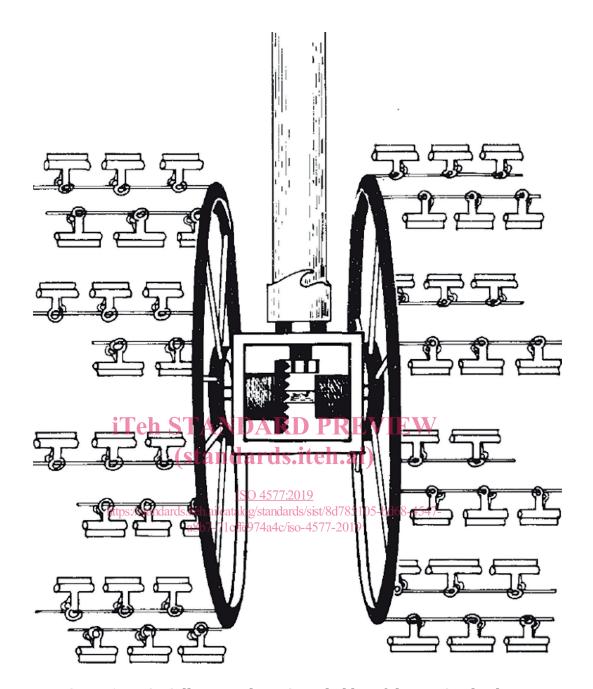


Figure 1 — Biaxially rotated specimen holder of the Ferris wheel type

#### **5.3.2** Uniaxially rotated specimen holder (see Figure 2)

The drum peripheral velocity shall be such that the air stream impinges on the flat section of the test specimens at not more than 1 m/s.

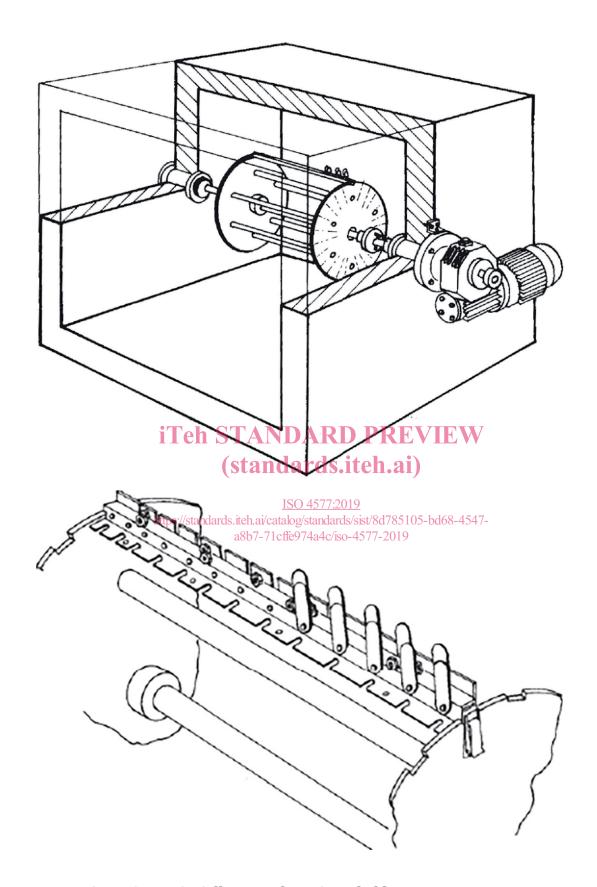


Figure 2 — Uniaxially rotated specimen holder — Drum rotator

**5.4 Air velocity meter**, non-directional, resistance wire type.

- **5.5 Die**, to produce specimens of dimensions 50 mm × 10 mm from compression moulded sheet.
- **5.6 Metal clips**, lined with fluorocarbon film or other materials that have no adverse effect on the oxidative stability of propylene plastics.

#### **6** Test specimens

The test specimens shall be cut from compression moulded sheet prepared from granules or other homogeneous moulding material as described in ISO 19069-2.

Specimens prepared by injection moulding or specimens cut from propylene plastics products may be used by agreement between the interested parties.

The specimens shall be 10 mm wide, 50 mm long and  $1.0 \text{ mm} \pm 0.05 \text{ mm}$  thick. Edges shall be smoothed, if necessary, to remove imperfections introduced by cutting.

By agreement between the interested parties, specimens of other thicknesses may be used.

At least five specimens shall be used for each test.

#### 7 Conditioning

In general, conditioning of specimens is not required. In case of dispute, the specimens shall be conditioned in one of the standard atmospheres specified in ISO 291 for not less than  $40\,h$ .

### 8 Test conditions (standards.iteh.ai)

The recommended test temperature is 15<u>0.°C4 During</u> the course of the test it shall not vary more than ±1 °C, measured at a single location of the working space, or more than ±1,5 °C throughout the oven.

a8b7-71cffe974a4c/iso-4577-2019 In the event that the average time to failure at 150 °C is less than 7 days, the test report shall indicate "less than 7 days at 150 °C" and the test shall be repeated at an oven temperature of 140 °C  $\pm$  1 °C and, if necessary, at lower temperatures at intervals of 10 °C until time to failure of 7 days is achieved.

NOTE If it is preferred to perform the test at 140 °C, even for materials lasting more than 7 days at 150 °C, this is permitted provided that this is stated in the test report.

#### 9 Procedure

**WARNING** — Handle specimens with caution to avoid contamination.

**9.1** Attach the five specimens to the holder (5.3) by means of suitable metal clips (5.6), which, together with their linings, shall be cleaned beforehand with trichloroethylene or any other suitable solvent to remove all traces of oil.

Place the specimens in the holder in the oven (5.1) such that the specimens are at least 30 mm apart and at least 50 mm from the walls of the oven, and start the rotation of the holder.

**9.2** Inspect the specimens visually at least once per day for failure; during inspection, rotation of the holder shall be stopped.

NOTE Visual inspection of the deterioration of the specimen surface, as a reliable evaluation of oxidative ageing endurance, is justified by the fact that oxidation of propylene thermoplastics usually takes place on the surface of the material before propagating inward. For special research purposes or in case of dispute, the oxidation can be followed by determination of the viscosity number (see ISO 1628-3) and/or the melt flow rate (see ISO 1133-1) of the specimens at specified elapsed times of ageing.