International Standard

ISO 4577

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 — Méthode à l'étuve

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ISO 4577-1983 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4577 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in May 1982.

It has been approved by the member bodies of the following countries:

Australia	Germany, F.R.	Romania
Austria	Hungary	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Sri Lanka
Canada	Italy	Sweden
China	Jamaica	Switzerland
Czechoslovakia	Japan	United Kingdom
Egypt, Arab Rep. of	Korea, Rep. of	USA
Finland	Netherlands	USSR
France	Poland	

The member body of the following country expressed disapproval of the document on technical grounds:

India

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

1 Scope and field of application

This International Standard 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.

2 References

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

ISO 1133, *Plastics — Determination of the melt flow rate of thermoplastics.*

ISO 1191, *Plastics — Polyethylenes and polypropylenes in dilute solution — Determination of viscosity number and of limiting viscosity number.*

ISO 1873, *Plastics — Polypropylene and propylene-copolymer thermoplastics —*

Part 1: Designation.

Part 2: Determination of properties.[1]

3 Principle

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

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

4 Apparatus

4.1 Oven, 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 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.

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1) At present at the stage of draft.

4.2 Thermometers, covering 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.

NOTE — 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.

4.3 Specimen holder.

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 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.

4.3.1 Biaxially rotated specimen holder (see figure 1).

The frequency of rotation about the horizontal and vertical axes shall be 1 to 3 min^{-1}.

4.3.2 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.
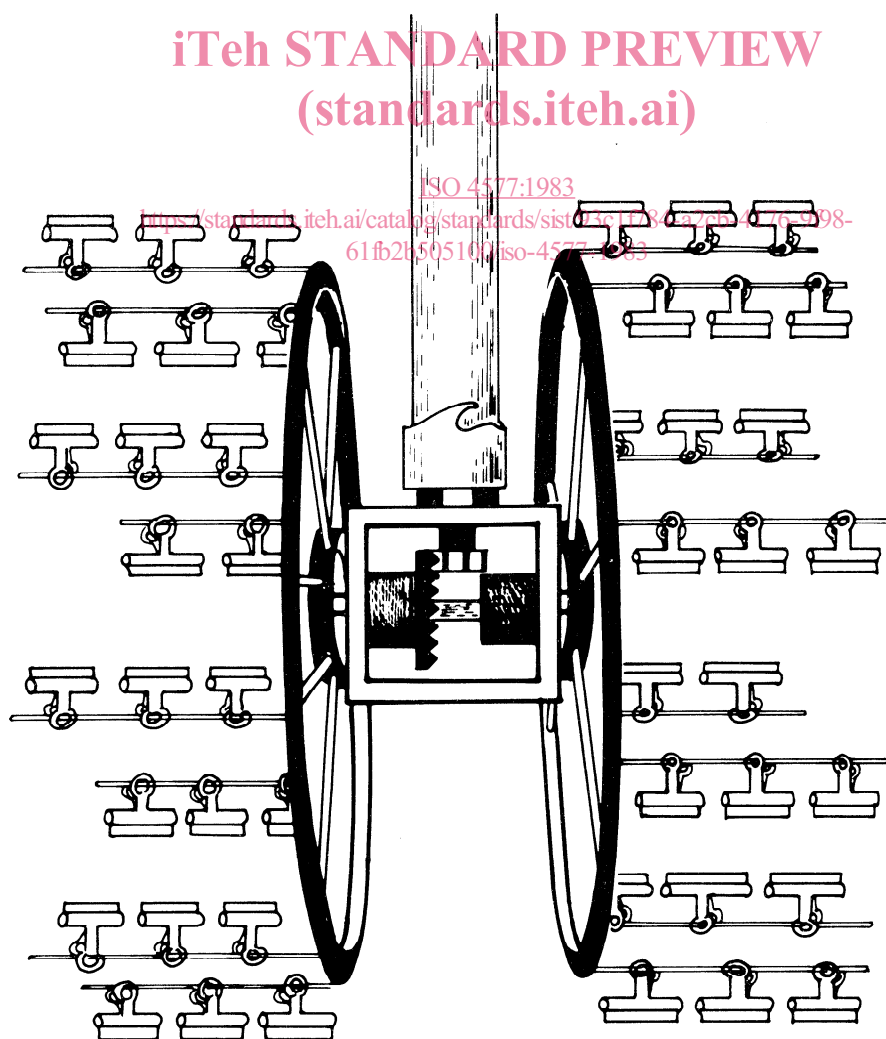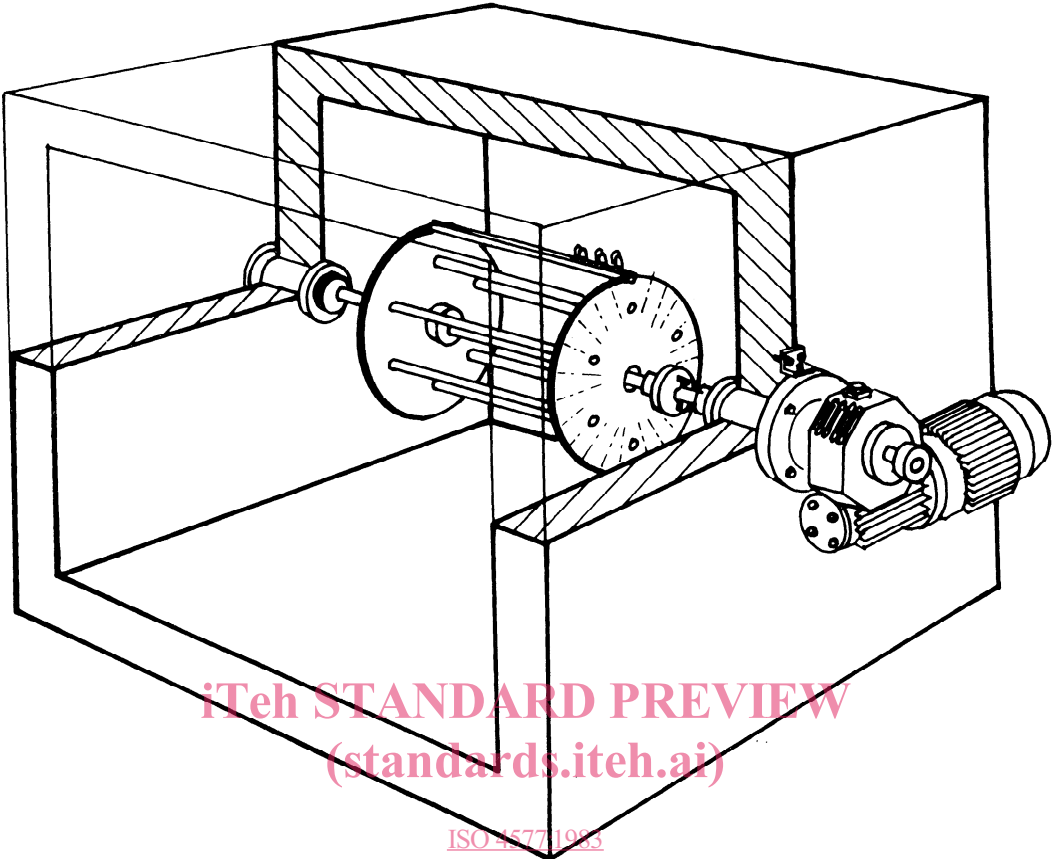

Figure 1 — Biaxially rotated specimen holder of the Ferris wheel type
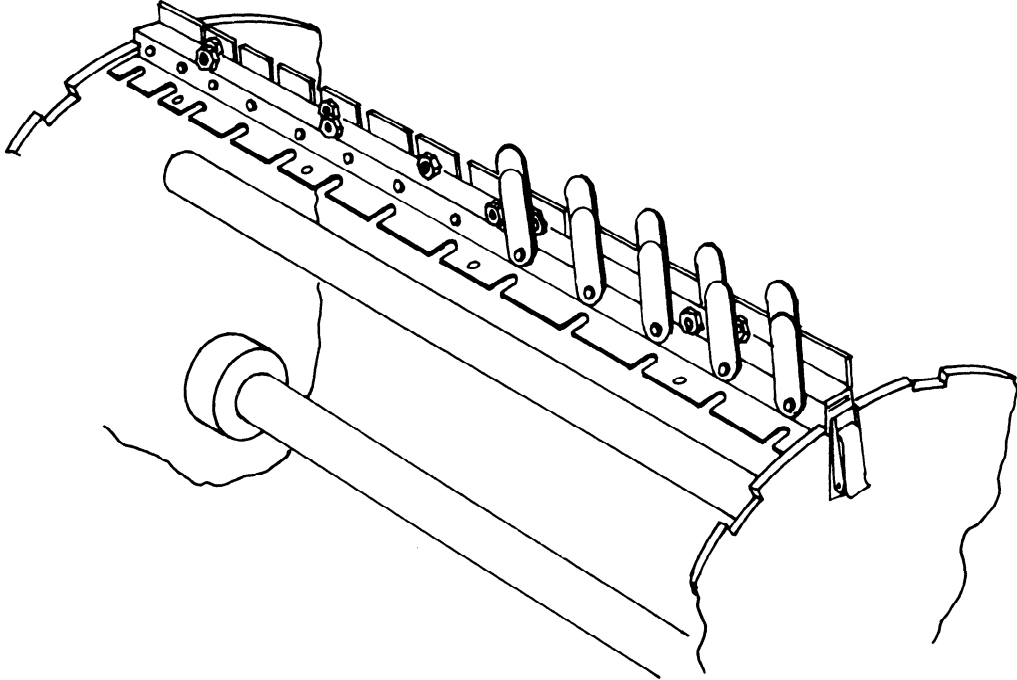
2

Figure 2 — Uniaxially rotated specimen holder — Drum rotator

4.4 Air velocity meter, non-directional, resistance wire type.

4.5 Die, to produce specimens of dimensions 50 mm × 10 mm from compression moulded sheet.

4.6 Metal clips, lined with fluorocarbon film or other materials that have no adverse effect on the oxidative stability of propylene plastics.

5 Test specimens

The test specimens shall be cut from compression moulded sheet prepared from granules or other homogeneous moulding material as described in ISO 1873/2.

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 ± 0,05 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.

6 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.

7 Test conditions

The recommended test temperature is 150 °C. During 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.

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 ± 1 °C and, if necesary, 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.

8 Procedure

WARNING — Handle specimens with caution to avoid contamination.

8.1 Attach the five specimens to the holder (4.3) by means of suitable metal clips (4.6), 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 (4.1) 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.

8.2 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 may be followed by determination of the viscosity number (see ISO 1191) and/or the melt flow rate (see ISO 1133) of the specimens at specified elapsed times of ageing.

9 Expression of results

Report the time to failure, i.e. the time, in days, after which visible indication of localized discoloration and crumbling appears. The area within 5 mm of the clips shall not be included in this evaluation.

10 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) the complete identification of the sample;

c) the average time to failure, in days at 150 °C[1], of the five specimens;

d) the range of times to failure, in days at 150 °C[1], of the five specimens;

e) the preparation or history of the specimen;

f) the thickness of the specimen;

g) the type of rotator;

h) the test temperature, if other than 150 °C;

j) details of conditioning (temperature and humidity), if appropriate (see clause 6).

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1) If the time to failure is less than 7 days at 150 °C, report as "less than 7 days at 150 °C" and in addition report the time (range of times) to failure at 140 °C (or at other temperatures used).

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