INTERNATIONAL STANDARD

ISO 11357-6

Third edition
2018-03

Plastics — Differential scanning calorimetry (DSC) —

Part 6:
Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 6: Détermination du temps d'induction à l'oxydation (OIT isotherme) et de la température d'induction à l'oxydation (OIT dynamique)

Reference number
ISO 11357-6:2018(E)

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Published in Switzerland

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 11357-6:2008), which has been technically revised. The main changes compared to the previous edition are as follows:

— the normative references in Clause 2 have been updated;

— techniques for purge gas flow control have been extended.

A list of all parts in the ISO 11357 series can be found on the ISO website.

Introduction

The measurement of oxidation induction time or temperature described in this document provides a tool to assess the conformity of the material tested to a given formulation of plastics compounds, but it is not intended to provide the concentration of antioxidant. Different antioxidants can have different oxidation induction times or temperatures. Due to interaction of the antioxidant with other substances in the formulation, different oxidation induction times or temperatures can result even with products having the same type and concentration of antioxidant.

Plastics — Differential scanning calorimetry (DSC) —

Part 6:
Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)

1 Scope

This document specifies methods for the determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT) of polymeric materials by means of differential scanning calorimetry (DSC). It is applicable to polyolefin resins that are in a fully stabilized or compounded form, either as raw materials or finished products. It can be applicable to other plastics.

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 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

ISO 472, *Plastics — Vocabulary*

ISO 8986-2, *Plastics — Polybutene-1 (PB-1) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 11357-1, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

ISO 17855-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 11357-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

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

3.1
oxidation induction time
isothermal OIT
relative measure of a stabilized material's resistance to oxidative decomposition, determined by the calorimetric measurement of the time interval to the onset of exothermic oxidation of the material at a specified temperature in an oxygen or air atmosphere, under atmospheric pressure

Note 1 to entry: It is expressed in minutes (min).

3.2
oxidation induction temperature
dynamic OIT
relative measure of a stabilized material's resistance to oxidative decomposition, determined by the calorimetric measurement of the temperature of the onset of exothermic oxidation of the material when subjected to a specified heating rate in an oxygen or air atmosphere, under atmospheric pressure

Note 1 to entry: It is expressed in degrees Celsius (°C).

Note 2 to entry: The oxidation induction temperature is also called oxidation onset temperature (OOT).

4 Principle

4.1 General

The time for which, or the temperature up to which, an antioxidant stabilizer system present in a test specimen inhibits oxidation is measured while the specimen is held isothermally at a specified temperature or heated at a constant rate in an oxygen or air atmosphere. The oxidation induction time or temperature is an assessment of the level (or degree) of stabilization of the material tested. Higher test temperatures will result in shorter oxidation induction times; faster heating rates will result in higher oxidation induction temperatures. The oxidation induction time and temperature are also dependent on the surface area of the specimen available for oxidation. It should be noted that tests carried out in pure oxygen will result in a lower oxidation induction time or temperature than tests performed under normal atmospheric-air conditions.

NOTE The oxidation induction time or temperature can be indicative of the effective antioxidant level present in the test specimen. Caution should be exercised in data interpretation, however, since oxidation reaction kinetics are a function of temperature and the inherent properties of the additives contained in the sample. For example, oxidation induction time or temperature results are often used to select optimum resin formulations. Volatile antioxidants or differences in activation energies of oxidation reactions can generate poor oxidation induction time or temperature results, even though the antioxidants can perform adequately at the intended temperature of use of the finished product.

4.2 Oxidation induction time (isothermal OIT)

The specimen and a reference material are heated at a constant rate in an inert gaseous environment (a flow of nitrogen). When the specified temperature has been reached, the atmosphere is changed to oxygen or air maintained at the same flow rate. The specimen is then held at constant temperature until the oxidative reaction is displayed on the thermal curve. The isothermal OIT is the time interval between the initiation of oxygen or air flow and the onset of the oxidative reaction. The onset of oxidation is indicated by an abrupt increase in the specimen's evolved heat and may be observed by a differential scanning calorimeter (DSC). The isothermal OIT is determined in accordance with 9.6.1.

4.3 Oxidation induction temperature (dynamic OIT)

The specimen and a reference material are heated at a constant rate in an oxygen or air atmosphere until the oxidative reaction is displayed on the thermal curve. The dynamic OIT is the temperature of the onset of the oxidative reaction. The onset of oxidation is indicated by an abrupt increase in the specimen's evolved heat and may be observed by a differential scanning calorimeter (DSC). The dynamic OIT is determined in accordance with 9.6.2.

5 Apparatus and materials

5.1 General

See also ISO 11357-1.

Subclauses 5.5 to 5.8 shall be followed as applicable (Subclauses 5.7 and 5.8 are required only for oxidation induction time measurements).

5.2 DSC instrument

The DSC instrument shall be able to achieve a maximum temperature of at least 500 °C. For oxidation induction time measurements, it shall be capable of maintaining an isothermal stability of ±0,3 K at the test temperature over the duration of the test, typically 60 min.

For high-precision measurements, an isothermal stability of ±0,1 K is recommended.

5.3 Crucibles

Specimens shall be placed in open or closed ventilated crucibles that allow unperturbed contact with the surrounding atmosphere. Preferably, crucibles shall be made of aluminium. Crucibles made of different materials may be used by agreement between the interested parties.

NOTE The composition of the crucible material can influence the oxidation induction time or temperature test result significantly (that is, including any associated catalytic effects). The type of containment system used depends on the intended application of the material being tested. Polyolefin resins used in the wire and cable industry typically require copper or aluminium crucibles whereas, for polyolefin resins used in geomembrane and vapour-barrier film applications, only aluminium crucibles are used.

5.4 Flowmeter

For gas flow calibration, a flow rate measuring device such as a rotameter or soap-film flowmeter or a gravimetric method shall be used together with a flow-adjusting valve. Mass flow controlling devices shall be calibrated against a positive-displacement device.

NOTE Suitable factory-provided gas flow rate calibration is also acceptable.

5.5 Oxygen

The oxygen used shall be of 99,5 % purity grade or better.

WARNING — The use of pressurized gas requires safe and proper handling. Furthermore, oxygen is a strong oxidizer that accelerates combustion vigorously. Keep oil and grease away from equipment using or containing oxygen.

5.6 Air

The pressurized air used shall be dry and free of oil and grease.

5.7 Nitrogen

The nitrogen used shall be of 99,99 % purity grade or better.

5.8 Gas-selector switch and regulators

The DSC apparatus used for oxidation induction time measurements needs to be switched between nitrogen and oxygen or air. The distance between the gas-switching point and the instrument cell shall be kept as short as possible, with a dead time of less than 1 min, to minimize the switching volume. Accordingly, for a flow rate of 50 ml/min, the dead volume will be equal to or less than 50 ml.

NOTE Increased precision can be obtained if the dead time is known. One possible means of determining dead time is to carry out a test using a non-stabilized material which will oxidize immediately in the presence of oxygen. The induction time from this test will provide a correction for subsequent OIT determinations.

6 Test specimens

6.1 General

See ISO 11357-1.

Specimens shall have a constant thickness of (650 ± 100) µm and parallel surfaces, shall be flat and shall not show any burrs or scars.

Specimen discs shall be small enough to lay flat in the crucible and shall not be stacked to increase mass.

NOTE Depending on the material and its process history, dimensions and service conditions, the methods of sample and specimen preparation can be crucial to the consistency of the results and their significance. In addition, the surface to volume ratio of the test specimen, poor specimen uniformity, residual stresses or lack of contact between specimen and crucible can affect test precision adversely.

If measurements of the OIT profile across the specimen thickness are required, specimens of significantly lower thickness than 650 µm may need to be used. This shall be noted in the test report.

6.2 Specimens from compression-moulded plates

Following ISO 293 or any other relevant polyolefin product standard such as ISO 17855-2 for PE, ISO 19069-2 for PP or ISO 8986-2 for PB-1, the test sample shall be compression-moulded into sheet of thickness complying with 6.1 to yield consistent specimen morphology and thickness. Alternatively, a specimen of suitable thickness can be cut from a thicker compression-moulded plate. If no heating time is specified in the relevant product standard, heating at the moulding temperature shall be limited to 5 min. Preferably, a bore-hole cutter shall be used to punch out from the plate a disc of diameter just less than the inner diameter of the sample crucible.

NOTE Specimen mass will vary depending on disc diameter. For a typical diameter of 5,5 mm, specimen discs cut from sheet will have a mass of approximately 12 mg to 17 mg, depending on the density of the material.

6.3 Specimens from injection-moulded plates or melt flow extrudates

Specimens may also be obtained from injection-moulded samples of thickness complying with 6.1, e.g. prepared in accordance with ISO 294-3 or any other relevant polyolefin product standard such as ISO 17855-2 for PE, ISO 19069-2 for PP or ISO 8986-2 for PB-1. Preferably, a bore-hole cutter shall be used to punch out a disc of diameter just less than the inner diameter of the sample crucible.

Specimens can also be cut from melt flow indexer extrudates. In this case, the specimen shall be cut perpendicular to the extrudate length. A visual inspection of the specimen shall be performed to ensure that it is free of voids. Preferably, a microtome shall be used to cut specimens to a constant thickness of (650 ± 100) µm.

6.4 Specimens from finished parts

Examples of such parts are pipes and fittings. Disc-shaped pieces shall be cut from the finished part in accordance with the referring standard so as to obtain specimens of thickness (650 ± 100) µm.

The following procedure is recommended to prepare specimens from thick-walled finished parts: obtain a cross-section of the wall by using a core drill directed radially through the wall, where the diameter of the core is just less than the inner diameter of the sample crucible. Take care not to overheat

