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**Plastics — Differential scanning calorimetry  
(DSC) —**

**Part 6:  
Determination of oxidation induction time**

*Plastiques — Analyse calorimétrique différentielle (DSC) —  
Partie 6: Détermination du temps d'induction à l'oxydation*  
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 11357 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11357-6 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 11357 consists of the following parts, under the general title *Plastics — Differential scanning calorimetry (DSC)*:

- Part 1: *General principles*
- Part 2: *Determination of glass transition temperature*
- Part 3: *Determination of temperature and enthalpy of melting and crystallization*
- Part 4: *Determination of specific heat capacity*
- Part 5: *Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion*
- Part 6: *Determination of oxidation induction time*
- Part 7: *Determination of crystallization kinetics*
- Part 8: *Determination of amount of absorbed water*

Annex A forms a normative part of this part of ISO 11357. Annexes B and C are for information only.

# Plastics — Differential scanning calorimetry (DSC) —

## Part 6:

### Determination of oxidation induction time

**WARNING** — The use of this part of ISO 11357 may involve hazardous materials, operations or equipment. This part of ISO 11357 does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this part of ISO 11357 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard information is given in subclause 5.3.

#### 1 Scope

This part of ISO 11357 specifies a method for the determination of the oxidation-induction time (OIT) of polymeric materials by differential scanning calorimetry (DSC). It is applicable to polyolefin resins that are in a fully stabilized/compounded form, either as raw materials or as finished products. It may be applicable to other plastics.

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#### 2 Normative references

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The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11357. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11357 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

ISO 11357-1:1997, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

#### 3 Terms and definitions

For the purposes of this part of ISO 11357, the terms and definitions given in ISO 11357-1 plus the following term and definition apply.

##### 3.1

##### oxidation induction time

##### OIT

a relative measure of a stabilized material's resistance to oxidative decomposition

**NOTE** It is determined by the calorimetric measurement of the time interval to the onset of exothermic oxidation of a material at a specified temperature in an oxygen atmosphere, under atmospheric pressure.

## 4 Principle

The time for which the antioxidant stabilizer system present in a test specimen inhibits oxidation is measured while the specimen is held isothermally at a specified temperature in a stream of oxygen. The OIT is an assessment of the level (or degree) of stabilization of the material tested. It is dependent on the surface area of specimen available for oxidation.

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 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 OIT is the time interval between the initiation of oxygen flow and the onset of the oxidative reaction. The onset of oxidation is signalled by an abrupt increase in the specimen's evolved heat or temperature and may be observed by a differential scanning calorimeter (DSC). The OIT is determined from the data recorded during the isothermal test.

**NOTE** The OIT 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, OIT results are often used to select optimum resin formulations. Volatile antioxidants or differences in activation energies of oxidation reactions may generate poor OIT results even though they may perform adequately at the intended use temperature of the finished product. It should also be noted that, since the test is carried out in pure oxygen, the OIT under normal atmospheric conditions will be longer.

## 5 Apparatus and materials

See ISO 11357-1:1997, clause 5.

**5.1 The DSC instrument** used shall be capable of maintaining stability within  $\pm 0,1$  °C at the test temperature over the duration of the test, typically 60 min. It shall be able to achieve a maximum temperature of at least 600 °C.

With some instruments, there may be modes of operation that promote precise isothermal temperature control. If available, they shall be used. If not, the test shall be conducted at two different isothermal temperatures, with each having been ascertained as accurately as possible. The data is then interpolated or extrapolated to the specified test temperature (see annex A). Alternatively, the specimen temperature can be monitored and adjusted continually, as required. Instruments incapable of automatic operation will additionally require a timer to monitor elapsed time.

**5.2 The pans** should preferably be open aluminium pans or closed ventilated pans. Other pans may be used by agreement between the interested parties.

**NOTE** The material composition of the pan can influence the OIT 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. Polyolefins used in the wire and cable industry typically require copper or aluminium pans, whereas polyolefins used in geomembrane and vapour-barrier film applications exclusively use aluminium pans.

**5.3 Oxygen**, 99,5 % ultra-high-purity grade (extra dry).

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

**5.4 Nitrogen**, 99,99 % ultra-high-purity grade (extra dry).

**5.5 Gas-selector switch and regulators**, needed to switch between nitrogen and oxygen. The distance between the gas-switching point and the instrument cell shall be kept small, with a dead time of less than one minute, to minimize the switching volume. Accordingly, for a flow rate of 50 ml/min, the dead volume shall be 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.

**5.6 Flowmeter**, of the rotameter or soap-film flowmeter type, for oxygen gas-flow calibration. Rotameters shall be calibrated against a positive-displacement device.

## 6 Test specimens

See ISO 11357-1:1997, clause 6, also taking into account the following recommendations/requirements:

- a) The following sample preparation procedures are recommended: following ISO 293, the test sample is compression-moulded into sheet of thickness  $250\ \mu\text{m} \pm 15\ \mu\text{m}$  prior to analysis to yield consistent sample morphology and mass. Limit heating to 2 min at the moulding temperature. Use a bore-hole cutter to punch out a disc from the sheet. Specimen discs small enough to lay flat in the pan, cut from the sheet, will have a mass of approximately 5 mg to 20 mg, depending on sample density. Do not stack specimens to increase mass.
- b) Specimens may also be prepared from injection-moulded samples or from melt flow indexer extrudates. In the latter case, cut the specimen perpendicular to its length. Perform a visual inspection of the specimen to ensure that it is free of voids.

**NOTE** Depending on the material and its process history, dimensions, and service conditions, the methods of sample and test specimen preparation may be crucial to the consistency of the results and their significance. In addition, the surface to volume ratio of the test specimen, poor sample uniformity, residual stresses or lack of contact between specimen and pan can affect test precision adversely. If the sample requires homogenization prior to analysis, the melt-compounding procedure given in annex B is recommended.

- c) Preparation of test specimens from a pipe or fitting, for example: cut disc-shaped pieces from the pipe or fitting in accordance with the referring standard so as to obtain specimens of mass 5 mg to 20 mg. The following procedure shall be used to prepare specimens from thick-walled pipes or fittings: obtain a cross-section of the wall of the pipe or fitting by use of a core drill directed radially through the wall, so that the diameter of the core is just less than the inner diameter of the sample pan. Take care not to overheat the specimen during the cutting operation. Cut discs of specified mass from the core. If the surface effects are of prime interest, cut discs from the inner and outer surfaces and test them with the original surface facing upward. If the characteristics of the base material are desired, cut a middle-section disc by removing the outer and inner surfaces.

## 7 Test conditions and specimen conditioning

See ISO 11357-1:1997, clause 7.

## 8 Temperature calibration

This procedure uses a two-point calibration step. For example, indium and tin are used as the calibrants for polyolefins since their respective melting points encompass the specified analysis temperature range (180 °C to 220 °C). If other plastics are analysed, the calibrants may need to be changed. Calibrate the instrument in accordance with ISO 11357-1:1997, clause 8. Calibration shall be performed under nitrogen using closed pans.

Use the following melting profiles:

Indium: ambient to 145 °C at 10 °C/min, 145 °C to 165 °C at 1 °C/min

Tin: ambient to 220 °C at 10 °C/min, 220 °C to 240 °C at 1 °C/min

## 9 Procedure

### 9.1 Setting up the apparatus

See ISO 11357-1:1997, subclause 9.1.

## 9.2 Loading the test specimen into the pan

See ISO 11357-1:1997, subclause 9.2.

If the specimen is cut from the inner or outer surface of a pipe or fitting, place it in the pan with the surface of interest facing upward. Weigh the test specimen to the nearest  $\pm 0,5$  mg, as heat flow is not measured in this case. Place the specimen disc into the appropriate pan type. If a cover is necessary, it shall be pierced to permit flow of oxygen to the specimen. Do not seal the pans unless they are ventilated.

Do not handle the test material or pan with bare hands. Either use tweezers or wear gloves.

## 9.3 Insertion of pans

See ISO 11357-1:1997, subclause 9.3.

## 9.4 Nitrogen flow

Use the same purge gas flow rate that was used to calibrate the instrument. Any change in flow rate or gas requires re-calibration. A typical nitrogen gas flow rate is  $50 \text{ ml/min} \pm 5 \text{ ml/min}$ .

## 9.5 Sensitivity adjustment

Adjust the sensitivity so that the difference in vertical height on the stepped change curve becomes at least 10 % or more of the full scale of the recording device. Modern instruments do not need this adjustment.

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## 9.6 Scanning temperature measurement

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Allow 5 min of nitrogen pre-purge at ambient temperature prior to beginning the heating cycle.

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Commence programmed heating of the specimen under nitrogen flow from ambient temperature to the test temperature, commonly  $200 \text{ }^\circ\text{C}$  for polyethylenes, at a rate of  $20 \text{ }^\circ\text{C/min}$ . Other test temperatures may be used as specified by a reference standard or by agreement between the interested parties. Particularly, specimens yielding OITs of less than 10 min should be retested at a lower temperature. Specimens yielding OITs of greater than 60 min should be retested at a higher temperature.

When the set temperature has been reached, discontinue programmed heating and equilibrate the sample for 5 min at the set temperature.

Turn on the recorder. If the instrument being used does not have an isothermal temperature-control-mode feature, follow the procedure outlined in annex A or, alternatively, ensure accurate temperature control by monitoring and adjusting continually, as required.

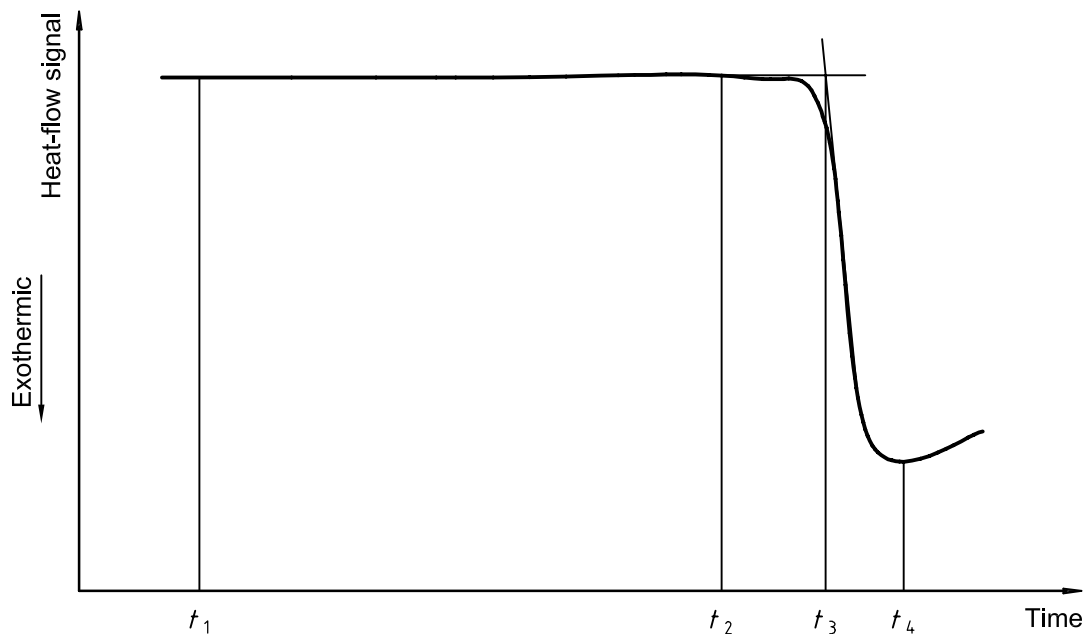
Once the equilibrium time has expired, change the gas to oxygen at a flow rate of  $50 \text{ ml/min} \pm 5 \text{ ml/min}$  (calibrated for oxygen). Mark this changeover point to oxygen flow as the zero time of the experiment.

Continue isothermal operation until at least 2 min have elapsed after the steepest point of the exotherm has been displayed (see Figure 1). Alternatively, the test may be terminated if the time requirements stated in the product specification have been met.

Upon completion of the test, switch the gas selector back to nitrogen and cool the instrument to ambient temperature. If additional testing is being conducted, cooling the instrument cell below  $60 \text{ }^\circ\text{C}$  to  $70 \text{ }^\circ\text{C}$  should be sufficient to avoid any further thermal oxidation of the sample.

The test frequency shall be established by the user. As a minimum requirement, specimens shall be tested in duplicate with the lower value reported.



**Key**

- $t_1$  point of changeover to oxygen flow  
 $t_2$  onset of oxidation  
 $t_3$  intercept in the tangent method  
 $t_4$  time to oxidation peak

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**Figure 1 — Typical oxidative thermal stability curve — Tangent method of analysis**

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Clean the DSC cell of contamination by heating to 600 °C for 5 min in air (or oxygen), at frequencies dictated by good laboratory practice.

## 10 Expression of results

The data is plotted with the heat-flow rate on the  $y$ -axis, versus time on the  $x$ -axis. The  $x$ -axis shall be expanded as much as possible to facilitate analysis.

Extend the recorded baseline beyond the oxidative-reaction exotherm. Extrapolate the steepest tangent to this exotherm to intercept the extended baseline (see Figure 1). Measure the OIT to within  $\pm 0,1$  min from zero time to the intercept point.

The tangent method described above is the preferred means of determining the intercept point, but the selection of the appropriate tangent to the exotherm curve may be difficult if the exothermic peak has a leading edge. Exothermic peaks with leading edges may occur if the oxidation reaction is slow. If the selection of the appropriate baseline is not obvious using the tangent method, an offset method may be used. Draw a second baseline parallel to the first baseline at a distance of 0,05 W/g (Figure 2) from the first baseline (see note). The intersection of this second line with the exotherm signal is defined as the onset of oxidation.

NOTE Other procedures or values for the distance from the baseline may be used by agreement between the interested parties.