

Plastics — Differential scanning calorimetry (DSC) —

Part 6: Determination of oxidation induction time

ICS 83.080.10

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The UK participation in its preparation was entrusted to Technical Committee PRI/21, Plastics test methods, which has the responsibility to:

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- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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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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Reference number
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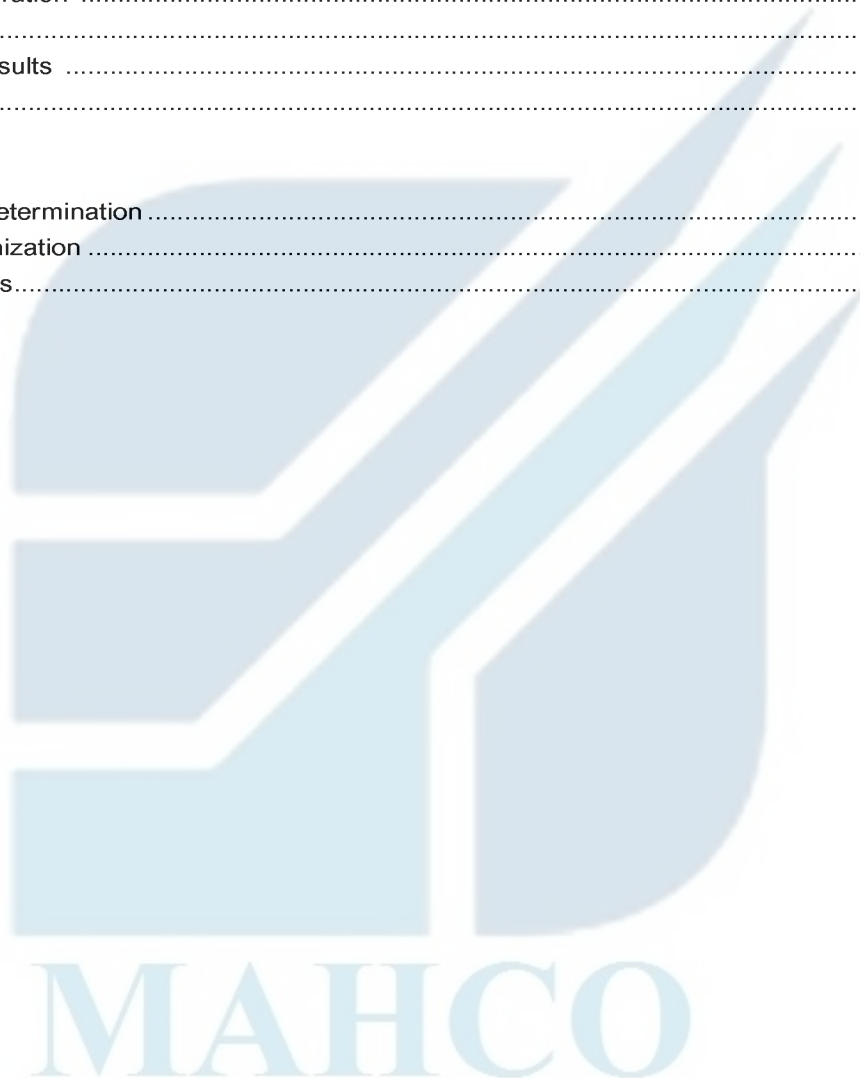
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Foreword

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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.

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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.

2 Normative references

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.

9.6 Scanning temperature measurement

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

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.

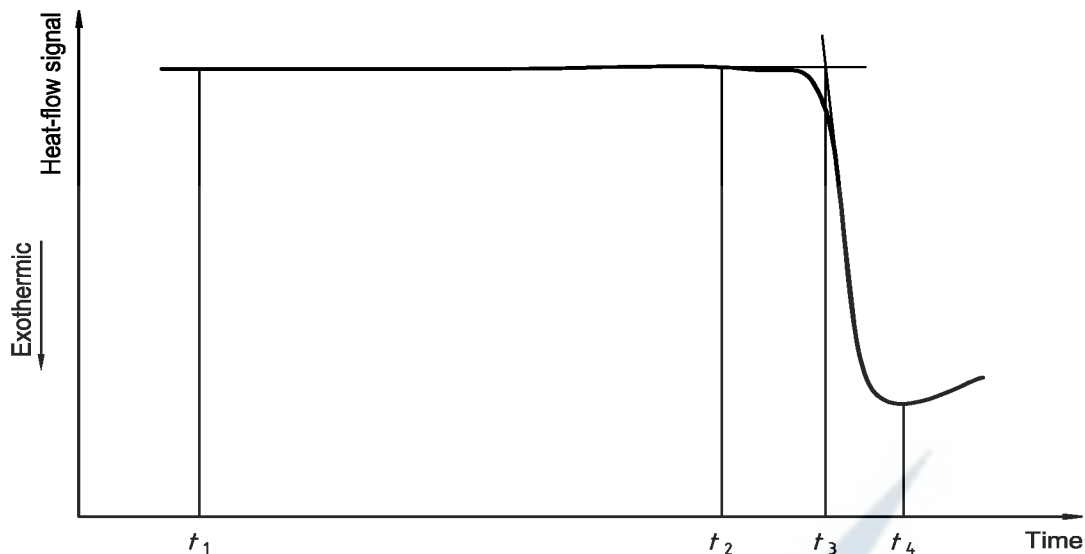
Turn on the recorder. If the instrument being used does not have an isothermal temperature-control-mode feature, follow the procedure outlined in annex A or, alternatively, ensure accurate temperature control by monitoring and adjusting continually, as required.

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

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

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

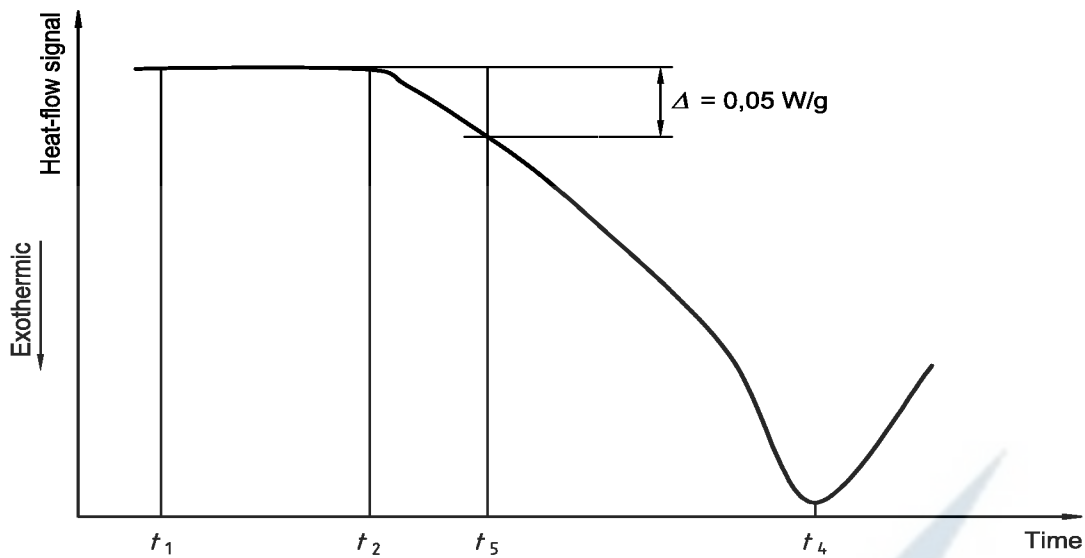
The data is plotted with the heat-flow rate on the y -axis, versus time on the x -axis. The x -axis shall be expanded as much as possible to facilitate analysis.

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.

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Key

- t_1 point of changeover to oxygen flow
- t_2 onset of oxidation
- t_5 intercept in the offset method
- t_4 time to oxidation peak

Figure 2 — Offset method for calculation of OIT

11 Test report

The test report shall include the information required in ISO 11357-1:1997, clause 10, plus the following:

- a) the details and method of specimen preparation;
- b) the test temperature;
- c) the measurement technique used (i.e. tangent method, offset method or other procedure agreed upon);
- d) the OIT, in minutes (to three significant digits);
- e) details of any deviations in the conditions or the materials used from those specified in this part of ISO 11357.

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Annex A (normative)

Alternative OIT determination

A.1 If the DSC instrument being used does not have an isothermal temperature-control-mode feature, the following procedure can be used for OIT determination. Commence programmed heating (under a nitrogen flow of $50 \text{ ml/min} \pm 5 \text{ ml/min}$) from ambient temperature and record the thermal curve. Continue heating until $200 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ is reached. Discontinue programmed heating and equilibrate the specimen for 5 min to a constant temperature. Measure and record this temperature to $\pm 0,1 \text{ }^\circ\text{C}$. Once the equilibrium time has expired, change the gas to oxygen at a flow rate of $50 \text{ ml/min} \pm 5 \text{ ml/min}$. This changeover point to oxygen flow is considered 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). Upon completion of the test, switch the gas selector back to nitrogen and cool the instrument to ambient temperature. Repeat the entire test on a new specimen, thereby generating a second exotherm. The isothermal portion of these two tests must be different from each other by at least $1 \text{ }^\circ\text{C}$, but within $2 \text{ }^\circ\text{C}$ of the set temperature (for example, $200 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$).

A.2 Extend the recorded baseline beyond the oxidative reactive exotherm. Extrapolate the slope of this exotherm to intercept the extended baseline (see Figure 2). Measure the OIT to within $\pm 0,1 \text{ min}$ from zero time to the intercept point. Make a plot of OIT versus temperature. Interpolate between the two actual temperature values until an OIT is obtained that corresponds exactly to the set temperature (for example, $200 \text{ }^\circ\text{C}$). A sample calculation is given in Figure A.1.

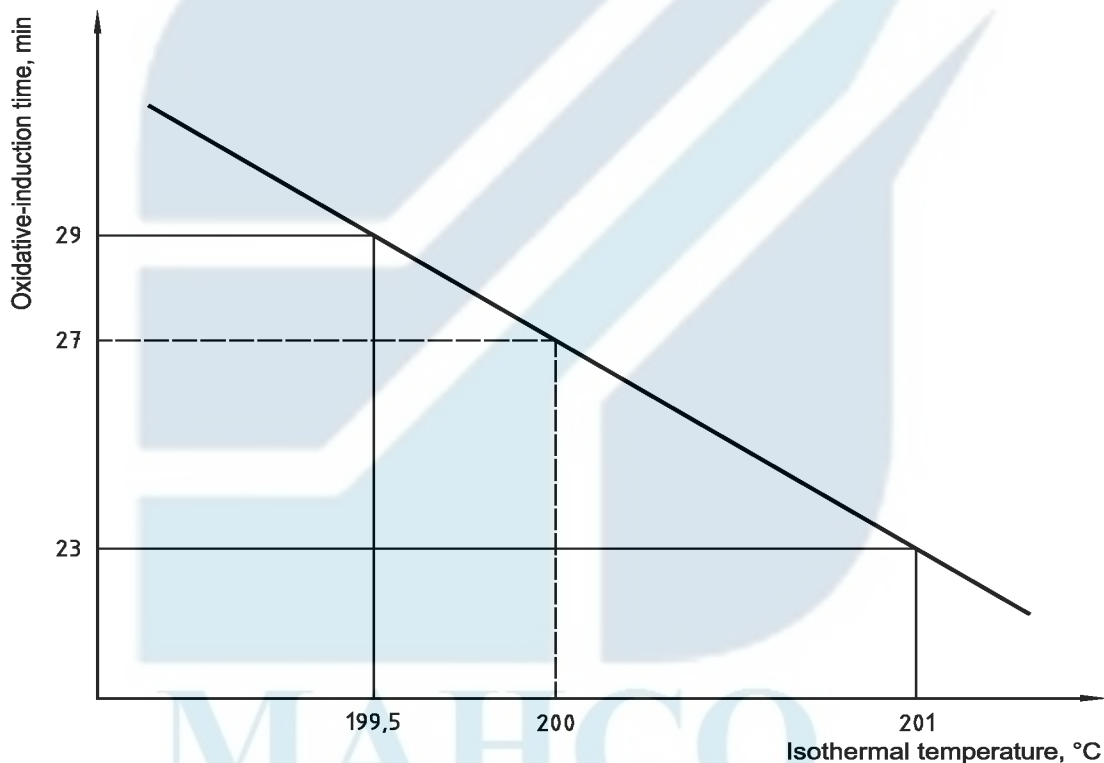


Figure A.1 — Example of the linear-interpolation technique used to estimate the oxidative-induction time (OIT) at $200 \text{ }^\circ\text{C}$

Annex B
(informative)

Sample homogenization

B.1 If desired, the test sample may be homogenized prior to OIT analysis to minimize local variations of stabilizer concentration. A torque rheometer equipped with a mixing head is suitable for such purposes.

B.2 Each sample should be blended at 60 r/min for 10 min, using a chamber temperature of 150 °C to 160 °C for polyethylene resins and 180 °C to 190 °C for polypropylene resins. The mixing chamber should be purged with nitrogen to suppress polymer and additive degradation.

B.3 After a mixing time of 10 min, quench-cool the samples in liquid nitrogen or ice water. After sufficient cooling, compression-mould the required amount of material into sheet form.



Annex C (informative)

Precision and bias

C.1 The data in Table C.1 are based on a round robin conducted in 1991, following ASTM E 691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*, involving four polyethylene materials. The test materials were obtained from three laboratories. The OIT of each sample was determined in duplicate on two separate days (that is, a total of four OIT determinations). Of the fourteen laboratories that participated in the round-robin exercise, eleven laboratories used heat-flux-type instruments and three laboratories used power-compensation-type instruments.

NOTE 1 The precision and bias data for other polyolefins may differ from the polyethylene material data shown in Table C.1.

NOTE 2 The sample preparation steps for each test material (that is, sheet compression moulding) were performed by one laboratory. Each participating laboratory received a set of specimen discs die-cut from each of the moulded sheets and samples of indium and tin required for instrument calibration. Each participating laboratory furnished its own specimen holders.

Table C.1 — Precision data for the oxidative induction time of polyethylenes

All times in minutes

Material	PE-LD	PE-LD	PE-LLD	PE-HD
Average	24,0	83,4	120	163
s_r	2,8	9,2	7,8	8,3
s_R	4,3	17,4	14,6	21,7
r	8,0	25,7	21,9	23,2
R	12,2	48,8	40,8	60,9

C.2 The following explanations of r and R are intended to present only a meaningful way of considering the approximate precision of this test method. The data in Table C.1 should not be applied rigorously to acceptance or rejection of material, as those data are specific to the round robin, and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in ASTM E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The following principles would then be valid for such data.

Concept of r and R : If s_r and s_R have been calculated from a large enough body of data, and for test results that were averages from testing four specimens, the following applies:

Repeatability limit, r (comparing two test results for the same material, obtained by the same operator using the same equipment on the same day): the two test results should be judged not equivalent if they differ by more than the r value for that material.

Reproducibility limit, R (comparing two test results for the same material, obtained by different operators using different equipment in different laboratories): the two test results should be judged not equivalent if they differ by more than the R value for that material.

Any judgment in accordance with the above would have an approximately 95 % (0,95) probability of being correct.

C.3 Bias: There are no recognized standards by which to estimate the bias of this test method.

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