BRITISH STANDARD

BS EN
728 : 1997
BS 2782 :
Part 11 :
Method 1103D :
1997

Plastics piping and ducting systems — Polyolefin pipes and fittings — Determination of oxidation induction time

The European Standard EN 728 : 1997 has the status of a British Standard

ICS 23.040.20

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee PRI/61, Plastics piping systems and components, upon which the following bodies were represented:

British Gas plc
British Plastics Federation
British Plumbing Fittings Manufacturers' Association
British Valve and Actuator Manufacturers' Association
Chartered Institution of Water and Environment Management
Department of the Environment (British Board of Agrèment)
Department of the Environment (Building Research Establishment)
Department of Transport
Electricity Association
Health and Safety Executive
Institute of Building Control
Institute of Materials
Institution of Civil Engineers
Institution of Gas Engineers
National Association of Plumbing, Heating and Mechanical Services Contractors
Pipeline Industries Guild
Plastics Land Drainage Manufacturers' Association
Society of British Gas Industries
Society of British Water Industries
Water Companies Association
Water Services Association of England and Wales

The following bodies were also represented in the drafting of this standard, through subcommittees and panels:

ERA Technology Ltd.
Engineering Equipment and Materials Users' Association
RAPRA Technology Ltd.

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on
15 July 1997

Amendments issued since publication

Amd. No.	Date	Text affected

The following BSI references relate to the work on this standard:
Committee reference PRI/61
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Contents

National foreword

This British Standard has been prepared by Technical Committee PRI/61 and is the English language version of EN 728 : 1997 *Plastics piping and ducting systems — Polyolefin pipes and fittings — Determination of oxidation induction time*, published by the European Committee for Standardization (CEN).

It is incorporated into BS 2782 *Methods of testing plastics : Part 11 : Thermoplastics pipes, fittings and valves*, as Method 1103D : 1997, for association with related test methods for plastics materials and plastics piping components.

This test method has been prepared for reference by other standards under preparation by CEN for specification of plastics piping and ducting systems and components. It has been implemented to enable experience of the method to be gained and for use for other fresh applications.

It is also for use for the revision or amendment of other national standards as practicable, but it should not be presumed to apply to any existing standard or specification which contains or makes reference to a different test method until that standard/specification has been amended or revised to make reference to this method and adjust any requirements as appropriate.

Cross-references

Publication referred to	Corresponding British Standard
ISO 293	BS 2782 : Part 9 : Method 901A : 1988 *Methods of testing plastics : Part 9 Sampling and test specimen preparation : Method 901A Compression moulding test specimens of thermoplastics materials*
ISO 1133	BS 2782 : Part 7 : Method 720A : 1997[1)] *Methods of testing plastics : Part 7 : Rheological properties : Method 720A Determination of melt flow rate of thermoplastics*

NOTE 1. For other applications, attention is drawn to BS 2782 : Part 1 : Methods 134A and 134B : 1992 *Determination of the oxidation induction time of thermoplastics.*

NOTE 2. Attention is drawn to the use in figure 1 of 'D' to designate the horizontal axis, for temperature and in figure 2 of 'F' to designate the time axis. This is inconsistent with ISO/TR 10837 : 1991, and with other test methods prepared by CEN/TC 155 and ISO/TC 138, which generally use '*T*' to designate a temperature axis and 'time' or '*t*' to designate the time axis. '*F*' is generally used to designate a force. Hence the use of 'D' and 'F' as presented in EN 728 : 1997 should not be copied into other contexts if it can be avoided.

Warning note. This British Standard, which is identical with EN 728 : 1997, does not necessarily detail all the precautions necessary to meet the requirements of the Health and Safety at Work etc. Act 1974. Attention should be paid to any appropriate safety precautions and the method should be operated only by trained personnel.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 6, an inside back cover and a back cover.

[1)] Under preparation.

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 728

January 1997

ICS 83.140.30

Descriptors: Plastic tubes, thermoplastic resins, polyolefins, pipe fittings, tests, determination, thermal stability, oxidation

English version

Plastics piping and ducting systems — Polyolefin pipes and fittings — Determination of oxidation induction time

Systèmes de canalisations et de gaines en plastiques — Tubes et raccords en polyoléfine — Determination du temps d'induction à l'oxydation

Kunststoff-Rohrleitungs- und Schutzrohrsysteme — Rohre und Formstücke aus Polyolefinen — Bestimmung der Oxidations-Induktionszeit

This European Standard was approved by CEN on 1996-10-27. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN 728 : 1997 E

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 155, Plastics piping systems and ducting systems, the secretariat of which is held by NNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 1997, and conflicting national standards shall be withdrawn at the latest by July 1997.

This standard is based on ISO/TR 10837 : 1991 *Determination of the thermal stability of polyethylene (PE) for use in gas pipes and fittings*, published by the International Organization for Standardization (ISO). It is a modification of ISO/TR 10837 : 1991 for reasons of applicability to other plastics materials and/or other test conditions and alignment with texts of other standards on test methods.

The modifications are:

- advice is provided on possible application of the method to additional thermoplastics;
- test parameters, except those common to all plastics, are omitted;
- no material-dependent requirements are given;
- editorial changes have been introduced.

The material-dependent parameters and/or performance requirements are incorporated in the System Standard(s) concerned.

This standard is one of a series of standards on test methods which support System Standards for plastics piping systems and ducting systems.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This standard specifies a method for measuring the oxidation induction time in oxygen at a specified temperature of polyolefin materials for or from pipes or fittings.

It may be used for assessing the thermal stability of either raw materials or finished products.

2 Normative references

This standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 293 *Plastics — Compression moulding test pieces of thermoplastic materials*

ISO 1133 *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

3 Principle

It is assumed that a polyolefin material for manufacture of pipe and/or fittings will incorporate an additive package which includes one or more antioxidants or other stabilizers.

The time for which the material, with its additive package consisting of antioxidant, stabilizers and other additives present in a test piece, inhibits oxidation is measured while the test piece is held isothermally at a specified temperature in a stream of oxygen.

The progress of the oxidation is monitored by measuring the difference in energy flow ($\Delta \dot{Q}$) or temperature (ΔT) between the test piece pan and reference pan of a thermal analyser and recording this difference against time.

The oxidation induction time (OIT) is then derived from this record as the period during which the difference in energy flow or temperature remains constant (see figure 2) between the test piece pan and reference pan.

This time can be indicative of the effective residual antioxidant level and reflects the time the test piece can be exposed in pure oxygen at the test temperature before the onset of thermal degradation. In normal atmospheric conditions this time will be longer.

Depending upon the material and the pipe or fitting processing, dimensions and service conditions, the methods of sample and test piece preparation may be crucial to the consistency of the results and their significance.

NOTE. It is assumed that the following test parameters are set by the standard making reference to this standard:

a) the test temperature, T, for the reference pan (see **5.1**);

b) the methods of sample and test piece preparation (see **6.2**) and, if applicable, the moulding temperature [see a) of **6.1**];

c) the number of test pieces (see **6.3**).

It is recommended to choose a temperature which normally results in induction times of at least 10 min.

4 Materials

4.1 Oxygen

An oxygen supply with a purity of at least 99,5 %.

4.2 Nitrogen

A nitrogen supply with a purity of 99,998 %.

4.3 Reference materials

Two or more temperature reference materials (calibration standards) of high purity metal having melting temperatures in the vicinity of the testing temperature, T [see a) of the note to clause **3**].

When T lies between 190 °C and 220 °C (typical for testing polyolefins), the calibration metals shall be as follows:

– indium (melting point 156,6 °C) with a purity grade of at least 99,99 %;

– tin (melting point 231,9 °C) with a purity grade of at least 99,99 %;

where the melting point is derived from the onset in the DSC diagram (shown as A in figure 1).

4.4 Solvent

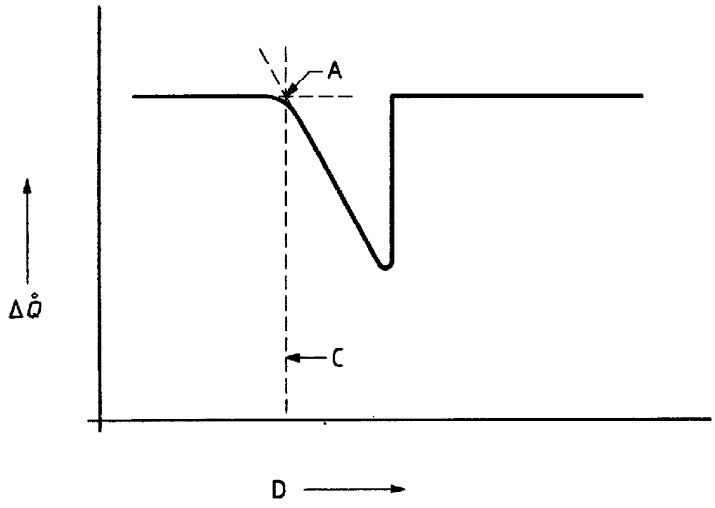
A solvent of appropriate composition (see **7.2**), analytical grade.

5 Apparatus

5.1 *Differential scanning calorimeter (DSC) or differential thermal analyser (DTA)*, capable of:

a) recording the difference in energy flow, $\Delta \dot{Q}$, or in temperature, ΔT, between the test piece pan and the reference pan against time (see clause **7**);

b) exposing a test piece in an open or ventilated aluminium pan to a flow of 50 ml/min ± 10 % of nitrogen and 50 ml/min ± 10 % of oxygen in turn so that each gas changeover is effected in not more than 1 min. The pan shall have a flat, smooth base capable of making good contact with the cell base and with a test piece respectively;



$\Delta \dot{Q}$ Difference in energy flow

A Onset temperature (melting point)

C Melting point

D Temperature

Figure 1. Typical calibration curve

c) increasing the temperature over the range of 140 °C to 250 °C at a rate of (1 ± 0,1) °C/min when the cell contains either a temperature calibration device or a calibration metal (see **4.3** and **7.1**);

d) increasing the test piece pan temperature T over the range from 50 °C to the test temperature at a rate of (20 ± 2) °C/min (see **7.2**);

e) stabilizing the temperature at (T ± 0,3) °C within 3 min of first reaching (T ± 0,3) °C;

f) maintaining the test temperature, T, within ± 0,3 °C for the duration of the test (see **7.2**).

NOTE. The design of the instrument oven should ensure that the test piece compartment is exposed to the required gas flow [see b)].

5.2 *Temperature measurement device*, capable of continuously monitoring the test piece pan temperature with a resolution of 0,1 °C.

NOTE 1. Test piece pan temperatures are used as the values for test results.

NOTE 2. This device can be integral with the DSC or DTA apparatus (see **5.1**), but this is not essential. A high impedance digital voltmeter with a resolution of 1 mV has been found suitable when connected to a thermocouple and the associated cold junction, or cold compensator, of the thermal analyser.

5.3 *Analytical balance*, capable of weighing a test piece (see clause **6**) to a limit of error of 0,1 mg.

5.4 *Gas flow control and measuring devices*, capable of providing the required flow rate (see **7.1** and **7.2**). Rotameters are suitable, if they are calibrated against a positive volume displacement device, e.g. a soap bubble flowmeter or equivalent.

5.5 *Timer*, comprising a stopwatch or equivalent.

6 Test pieces

6.1 Preparation of test piece from raw materials

Cut one or more test pieces (see **6.3**), each having a mass of (15 ± 2) mg, from a melt flow extrudate obtained in accordance with ISO 1133, or prepare one or more test pieces as follows:

a) prepare a compression-moulded plaque in accordance with ISO 293. Limit heating to 2 min at the moulding temperature specified by the referring standard;

b) cut a cylindrical sample with a diameter not less than half the inside diameter of the test piece pan;

NOTE. It is recommended to use test pieces with a diameter of approximately 6 mm.

c) cut a test piece from the cylinder to give a test piece mass of (15 ± 2) mg.

6.2 Preparation of test pieces from a pipe or fitting

Cut samples from the pipe or fitting in accordance with the referring standard, so as to obtain one or more test pieces (see **6.3**) each having a mass of (15 ± 2) mg.

NOTE. For testing of thick-walled polyethylene pipe or fittings the following method has been found suitable.

Recommended procedure for test piece preparation for pipes and fittings.

Obtain a cross section of the wall of the pipe and/or fitting by use of a core drill directed radially through the wall, so that the diameter of the core preferably is just less than the inner diameter of the test pan for the test instrument [see note to item b) of **6.1**] and care is taken not to overheat the sample during the cutting operation. Cut from the core test pieces of the specified mass in the form of discs as follows.

Select at least the inner-wall surface zone, outer-wall surface zone and mid-wall zone as the sample points from the core which are to be tested individually, unless surface effects are of prime interest. In such cases cut the discs only from the inner and outer surfaces and test them with surface side uppermost.

6.3 Number of test pieces

The number of test pieces shall be as specified in the referring standard.

7 Procedure

7.1 Calibration

7.1.1 *General*

Carry out the procedures given in **7.1.2** and **7.1.3** each at the frequency necessary to ensure that results obtained in accordance with **7.2** are obtained under the specified conditions.

7.1.2 *Temperature calibration*

7.1.2.1 Ensure that the oven is properly clean, e.g. by heating up in a nitrogen atmosphere at a temperature of approximately 500 °C to 550 °C for at least 10 min followed after cooling by a cleaning with a cloth, if necessary.

Establish an oxygen flow of 50 ml/min ± 10 % through the apparatus at a temperature of at least 10 °C below the expected melting point of one of the calibration metals, e.g. indium or tin (see **4.3**).

Heat the calibration metal in a sealed aluminium pan at a rate of 1 °C/min until the melting endotherm is recorded, using an empty sealed aluminium pan as reference. Obtain a plot of ΔT or $\Delta \overset{o}{Q}$ against temperature (e.g. see figure 1). If the apparatus does not automatically do so, mark the indicated temperature on a chart at intervals in the region of the endotherm so that the melting point can be determined with a precision of ± 0,1 °C.

Take the melting point of the metal to be the temperature given by the intercept of the extended baseline and the extended tangent to the first slope of the endotherm, i.e. the onset temperature shown as A in figure 1.

7.1.2.2 Repeat the procedure given in **7.1.2.1** using a piece of the other calibration metal.

7.1.2.3 Adjust the apparatus so that the indicated melting point of indium lies within (156,6 ± 0,3) °C and that of tin lies within (231,9 ± 0,3) °C.

NOTE. The described dynamic calibration procedure according to **7.1.2.3** is expected to correspond to the isothermal calibration within the experimental error.

7.1.3 *Time calibration*

For a chart output, use a timer (**5.5**) to check that the pen moves along the abscissa at a known rate.

7.4 Measurement of oxidation induction time

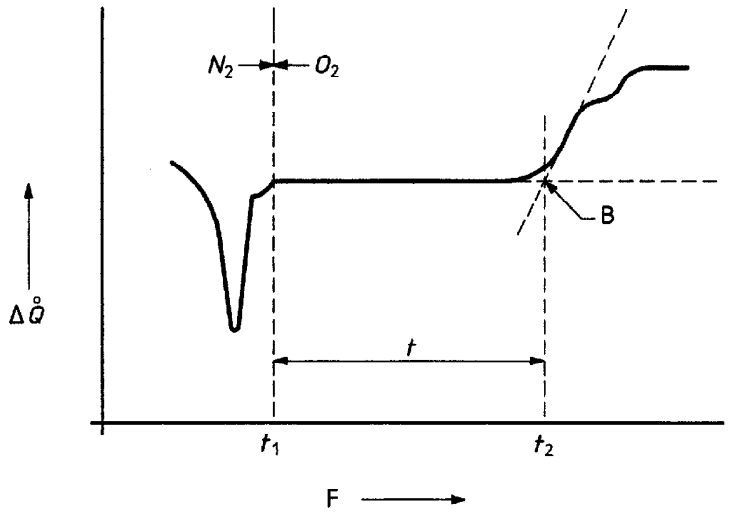
Before their use, ensure that the test piece pan(s) and reference pan are clean, e.g. by cleaning with an appropriate solvent (see **4.4**) such as hexane.

Establish a nitrogen flow of 50 ml/min ± 10 % through the DSC or DTA. Check that when a switchover to oxygen is made, the gas flow will continue at that rate. Revert to a nitrogen flow of 50 ml/min ± 10 %.

Put a test piece into the test piece pan, ensuring as good initial contact with the pan as possible. If the test piece includes the inner or outer surface of the source pipe or fitting (see clause **3**), place it in the pan with that surface uppermost.

Introduce an open or ventilated aluminium pan containing a test piece and an empty aluminium reference pan into the instrument. Purge the instrument oven with nitrogen for 3 min to expel any oxygen. Set the instrument both to raise the temperature from an ambient start temperature of 50 °C at a rate of 20 °C/min and to run isothermally at T °C until the temperature has stabilized. Start to record the thermogram as a plot of the energy flow difference ($\Delta \overset{o}{Q}$), or the temperature difference (ΔT), against time and record the time as t_0, when the temperature first reaches T °C.

Exactly 3 min after t_0, switch over to oxygen and ensure that this point t_1 is recorded on the thermogram. Continue to run the thermogram until the oxidation exotherm has occurred and has passed its maximum value (see figure 2).



$\Delta \dot{Q}$ Difference in energy flow

B Point of onset of degradation

t Oxidation induction time

F Time

Figure 2. Typical thermogram for polyethylene exhibiting an endothermic reaction

8 Interpretation of results

8.1 OIT calculation

The oxidation induction time, t, of each test piece shall be the time taken from the switchover to oxygen, t_1, to the time t_2 corresponding to the intercept of the extended baseline and the extended tangent drawn to the exotherm at the point of maximum slope, shown as B in figure 2.

NOTE. In cases when the interpretation using this procedure may be doubtful, the procedure for obtaining the oxidation induction time may need to be agreed upon by the parties involved [see i) of clause 9].

8.2 Behaviour of test piece

After the test, inspect the test piece and record details of any observed changes.

NOTE. Due to e.g. orientation, the test piece may change in size or deform in a manner which influences the area/thickness ratio. This may give erroneous results or high variations in the oxidation induction times.

9 Test report

The test report shall include the following information:

a) the reference to this standard and to the referring standard;

b) full identification of the product from which samples are taken;

c) the position(s) in the pipe or fitting wall cross section from which the test pieces were derived;

d) the mass of the test piece, in milligrams;

e) the oxidation induction time, t, of each test piece, and, if applicable, the mean, maximum and minimum values and a copy of the thermogram(s);

f) the test temperature, T, in degrees Celsius;

g) manufacture, model and type (DSC or DTA) of instrument used;

h) details of any changes in the test piece appearance (see **8.2**);

i) any factors which may have affected the results, such as any incidents or any operating details not specified in this standard;

j) the date of test.

List of references

See national foreword.

BS EN 728 : 1997
BS 2782 : Part 11 : Method 1103D : 1997

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