SLOVENSKI STANDARD
SIST EN 728:1999

01-julij-1999

Cevni sistemi iz polimernih materialov - Cevi in fitingi iz poliolefinov - Določanje indukcijskega časa oksidacije

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

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

Systemes de canalisations et de gaines en plastiques - Tubes et raccords en polyoléfine - Détermination du temps d'induction a l'oxydation

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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 - Détermination 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.

The European Standards exist 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

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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of an identical text or by endorsement, at the latest by July 1997, and conflicting national standards shall be withdrawn at the latest by July 1997.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 (ΔQ) 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 of 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, ΔQ, 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;

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).