



BSI Standards Publication

**Pipes and fittings made of  
crosslinked polyethylene  
(PE-X) — Estimation of the  
degree of crosslinking by  
determination of the gel  
content**

### National foreword

This British Standard is the UK implementation of EN ISO 10147:2012. It is identical to ISO 10147:2011. It supersedes BS EN 579:1994 and BS ISO 10147:2011, which are withdrawn.

The UK participation in its preparation was entrusted by Technical Committee PRI/88, Plastics piping systems, to Subcommittee PRI/88/4, Test Methods - Plastic Piping.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Pipes and fittings made of crosslinked polyethylene (PE-X) -  
Estimation of the degree of crosslinking by determination of the  
gel content (ISO 10147:2011)

Tubes et raccords en polyéthylène réticulé (PE-X) -  
Estimation du degré de réticulation par le mesurage du  
taux de gel (ISO 10147:2011)

Rohre und Fittings aus vernetztem Polyethylen (PE-X) -  
Berechnung des Grades der Vernetzung durch  
Bestimmung des Gel-Gehaltes (ISO 10147:2011)

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## Foreword

The text of ISO 10147:2011 has been prepared by Technical Committee ISO/TC 138 "Plastics pipes, fittings and valves for the transport of fluids" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 10147:2012 by Technical Committee CEN/TC 155 "Plastics piping systems and ducting systems" the secretariat of which is held by NEN.

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# Pipes and fittings made of crosslinked polyethylene (PE-X) — Estimation of the degree of crosslinking by determination of the gel content

## 1 Scope

This International Standard specifies a method for the assessment of the degree of crosslinking in crosslinked polyethylene (PE-X) pipes and fittings by determination of the gel content by solvent extraction.

## 2 Principle

The mass of a test piece taken from a pipe or a fitting is measured before and after immersion of the test piece in a solvent for a specified period of time. The degree of crosslinking is expressed as the percentage by mass of the insoluble material.

For the minimum applicable values for the degree of crosslinking refer to the product or system standards.

## 3 Solvent

**3.1 Xylene**, an isomeric mixture with a purity  $\geq 98$  % volume fraction and a boiling range of 137 °C to 144 °C, to which a 1 % volume fraction of antioxidant has been added.

The antioxidant may be either 2,2-methylene-*bis*(4-methyl-6-*tert*-butylphenol) or antioxidants based on 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl) propionate or a combination of these.

**WARNING — Xylene is a harmful and inflammable solvent that can be absorbed through the skin and, as such, should be handled carefully. Attention is drawn to any relevant regulations and associated exposure limits. Use only in a ventilated hood. Check the effectiveness of the hood before starting the test. Do not inhale the vapour. The appropriate safety equipment should be worn. Excessive inhalation of the vapour may cause dizziness, headache or both. In the event of excessive vapour inhalation, seek fresh, clean air.**

## 4 Apparatus

The following apparatus is required in order to carry out the test.

**4.1 Reflux condenser**, of the general type shown in Figure 1.

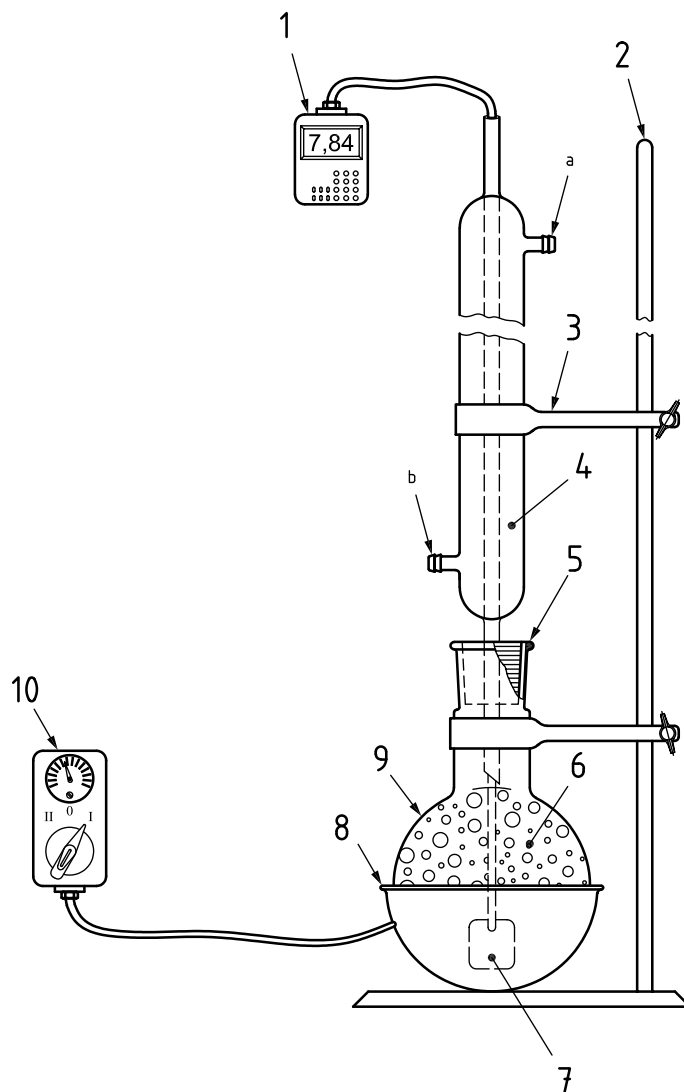
**4.2 Round-bottomed flask**, of at least 500 ml capacity.

**4.3 Heating mantle**, to fit the flask (4.2), and with sufficient heating capacity to boil xylene (boiling range: 137 °C to 144 °C).

**4.4 Cage**, with lid, large enough to contain a test piece (see Clause 5).

The cage shall be made of wire cloth or mesh, of aluminium or stainless steel, with a pore size of  $(125 \pm 25)$   $\mu\text{m}$  and sufficient to retain the sample. The wire cloth or mesh shall be free of grease, oil or other contaminants soluble in xylene. If not, it shall be washed with acetone and dried before use.

**4.5 Lathe with automatic feed**, for machining out test pieces, or **microtome** or **other suitable cutting tool**.



**Key**

- 1 identification tag and fine wire attached to cage
  - 2 stand
  - 3 ring clamp
  - 4 reflux condenser
  - 5 ground-glass joint or cork
  - 6 xylene
  - 7 wire cage enclosing test piece
  - 8 heating mantle
  - 9 wide-necked round-bottomed flask
  - 10 variable transformer
- a Water out.  
b Water in.

**Figure 1 — Extraction apparatus**

**4.6 Vacuum oven or forced-ventilation oven**, capable of maintaining the specified conditions (see 6.6).

**4.7 Balance**, capable of weighing the cage, with or without a test piece, to an accuracy of 1 mg.

## 5 Preparation of test pieces

The test pieces are prepared in accordance with the following instructions.

Any protective layer on the pipe or test sample shall be removed prior to preparation of test pieces.

Unless otherwise specified in the referring standard, at least two test pieces shall be prepared.

Each test piece shall comprise a slice or shaving having a thickness of  $(0,2 \pm 0,02)$  mm, taken from a cross-section of the pipe or fitting to include the full wall thickness round at least one circumference unless specified otherwise by the referring standard. The mass of the test piece shall be  $\geq 0,2$  g.

The degree of crosslinking can vary through the wall thickness of a pipe or fitting and therefore, for surface or midwall measurements, test pieces shall be machined accordingly.

It is recommended that a lathe be used to machine test pieces from pipe. Alternatively, a microtome or other suitable cutting tool can be used to obtain the test pieces from fittings.

## 6 Procedure

**6.1** Weigh a clean, dry cage including the lid (see 4.4), to an accuracy of 1 mg (mass  $m_1$ ).

**6.2** Place a test piece in the cage, close the lid and weigh the cage and test piece together to an accuracy of 1 mg (mass  $m_2$ ).

**6.3** Place the cage and test piece in the flask (4.2) and ensure that there is sufficient xylene solvent to maintain total immersion and to provide a ratio by mass of solvent to test piece of at least 200:1.

The solvent can be re-used after distillation with the addition of a further 1 % volume fraction of antioxidant (3.1). In case of dispute, use a new or freshly distilled solution.

**6.4** Boil the solvent vigorously to ensure good agitation for  $8 \text{ h} \pm 30 \text{ min}$ .

**6.5** Carefully remove the cage and the residue of the test piece from the solution after the time specified in 6.4.

**CAUTION — Take care when removing the cage from the boiling solution (see 3.1).**

**6.6** Complete the drying of the residue, or cage, lid and residue, by placing them for at least 3 h in:

- a) either a vacuum oven (see 4.6), kept at  $(90 \pm 2)$  °C under a vacuum (negative pressure) of at least 0,85 bar (85 kPa), i.e. approximately 0,15 bar absolute pressure or less; or
- b) a forced-ventilation oven (see 4.6), with an adequate extraction facility, kept at  $(140 \pm 2)$  °C.

**6.7** Allow to cool to ambient temperature and weigh the residue (mass  $m_4$ ) or the cage, lid and residue (mass  $m_3$ ) to an accuracy of 1 mg.

## 7 Calculation and expression of results

Calculate the degree of crosslinking,  $G$ , of the material in the individual test pieces as the percentage by mass of the insoluble material, using one of the following equations as appropriate:



when only the residue was weighed:

$$G = \frac{m_4}{m_2 - m_1} \times 100$$

when the cage, lid and residue were weighed:

$$G = \frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

$m_1$  is the mass of the cage and lid, in milligrams;

$m_2$  is the mass of the original test piece, the cage and the lid, in milligrams;

$m_3$  is the mass of the residue, cage and lid, in milligrams;

$m_4$  is the mass of the residue, in milligrams.

Express the result to the nearest whole number.

The mass of any filler present in the material shall be taken into account.

The average degree of crosslinking,  $G_a$ , can be calculated from the individual results of a number of samples.

## 8 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e ISO 10147, and, if applicable, to the standards making reference to this one;
- b) all details necessary for the identification of the test piece;
- c) the degree of crosslinking,  $G$ , for the individual test pieces and the average,  $G_a$ , stating the number of samples tested;
- d) details of any variation in the specified procedure and of any abnormal behaviour observed during the test;
- e) the date of the test.





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