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


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

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© BSI 2011

ISBN 978 0 580 72509 8

ICS 23.040.20; 23.040.45

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2011.

Amendments issued since publication

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

Tubes et raccords en polyéthylène réticulé (PE-X) — Estimation du degré de réticulation par le mesurage du taux de gel
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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10147 was prepared by Technical Committee ISO/TC 138, Plastics pipes, fittings and valves for the transport of fluids, Subcommittee SC 5, General properties of pipes, fittings and valves of plastic materials and their accessories — Test methods and basic specifications.

This third edition cancels and replaces the second edition (ISO 10147:2004), which has been technically revised. It also incorporates the Amendment ISO 10147:2004/Amd.1:2008.
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 ≥ 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 ± 25) µm 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.
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 $(0.2 \pm 0.02) \text{mm}$, 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 $\geq 0.2 \text{g}$.

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 $(90 \pm 2) \, ^\circ\text{C}$ 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 $(140 \pm 2) \, ^\circ\text{C}$.

6.7 Allow to cool to ambient temperature and weigh the residue (mass $m_3$) or the cage, lid and residue (mass $m_2$) 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.
British Standards Institution (BSI)

BSI is the independent national body responsible for preparing British Standards and other standards-related publications, information and services. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions. It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover.

Tel: +44 (0)20 8996 9001 Fax: +44 (0)20 8996 7001

BSI offers Members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Tel: +44 (0)20 8996 7669 Fax: +44 (0)20 8996 7001
Email: plus@bsigroup.com

Buying standards

You may buy PDF and hard copy versions of standards directly using a credit card from the BSI Shop on the website www.bsigroup.com/shop. In addition all orders for BSI, international and foreign standards publications can be addressed to BSI Customer Services.

Tel: +44 (0)20 8996 9001 Fax: +44 (0)20 8996 7001
Email: orders@bsigroup.com

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Knowledge Centre.

Tel: +44 (0)20 8996 7004 Fax: +44 (0)20 8996 7005
Email: knowledgecentre@bsigroup.com

Various BSI electronic information services are also available which give details on all its products and services.

Tel: +44 (0)20 8996 7111 Fax: +44 (0)20 8996 7048
Email: info@bsigroup.com

BSI Subscribing Members are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration.

Tel: +44 (0)20 8996 7002 Fax: +44 (0)20 8996 7001
Email: membership@bsigroup.com

Information regarding online access to British Standards via British Standards Online can be found at www.bsigroup.com/BSOL

Further information about BSI is available on the BSI website at www.bsigroup.com/standards

Copyright

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. This does not preclude the free use, in the course of implementing the standard of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained. Details and advice can be obtained from the Copyright & Licensing Manager.

Tel: +44 (0)20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Tel +44 (0)20 8996 9001
Fax +44 (0)20 8996 7001
www.bsigroup.com/standards