INTERNATIONAL STANDARD

ISO 10147

First edition
1994-12-01

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

Reference number
ISO 10147:1994(E)

Foreword

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International Standard 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*.

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Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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 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.

3 Solvent

3.1 Xylene, analytically pure, to which 1 % antioxidant [2,2-methylene-bis(4-methyl-6-*t*-butylphenol)] has been added.

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. Expose only in a ventilated hood. Check the effectiveness of the hood before starting the tests. Do not inhale the vapour. Excessive inhalation of the vapour may cause dizziness, headache or both. In the event of excessive vapour inhalation, seek fresh clean air.

4 Apparatus (see figure 1)

4.1 Reflux condenser, of the general type.

4.2 Round-bottomed flask, of at least 500 ml capacity (a 2 000 ml flask is suitable for up to six determinations at one time).

4.3 Heating mantle, to fit the flask (see 4.2), and with sufficient heating capacity to boil xylene (boiling range: 138 °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 125 µm ± 25 µm. The wire cloth or mesh shall be free of grease, oil or other contaminants soluble in xylene. If necessary, it shall be washed with acetone and dried before use.

4.5 Lathe with automatic feed, for machining the test piece, or a **microtome** or other cutting tools.

4.6 Vacuum oven, or an **oven with forced ventilation**, 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 Test pieces

5.1 Preparation of test pieces

Each test piece shall comprise a slice or shavings having a thickness of between 0,1 mm and 0,2 mm, taken from a cross-section of the pipe or fitting to include the full wall thickness for at least one circumference. The mass of the test piece shall be between 0,5 g and 1,0 g.

NOTE 1 It is recommended that a lathe is used to machine test pieces from pipe. Alternatively, a microtome or other cutting tools may be used and may be necessary for obtaining test pieces from fittings.

5.2 Number of test pieces

At least two tests pieces shall be used.

6 Procedure

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

6.2 Place a test piece in the cage 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 solution to maintain total immersion.

6.4 Boil the solution vigorously to ensure good agitation for 8 h ± 5 min.

6.5 Carefully remove the cage and the residue of the test piece from the boiling solution and dry the exterior of the cage with a cloth.

6.6 Complete the drying of the cage and the residue together by placing them for 3 h in either

a) a vacuum oven (4.6) at 140 °C ± 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 (4.6) with an adequate extraction facility, at 140 °C ± 2 °C.

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

6.8 Repeat procedures 6.1 to 6.7 at least once, using a fresh test piece each time.

7 Calculation and expression of results

7.1 Calculate the degree of crosslinking for an individual test piece, G_i, as a percentage by mass of insoluble material, using the following equation:

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

where

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

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

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

Express the result to the nearest whole number.

7.2 Calculate the average degree of crosslinking, G, from the individual results. If the two individual results differ from one another by three points of degree of crosslinking, repeat the test with two new test pieces.

8 Test report

The test report shall include the following information:

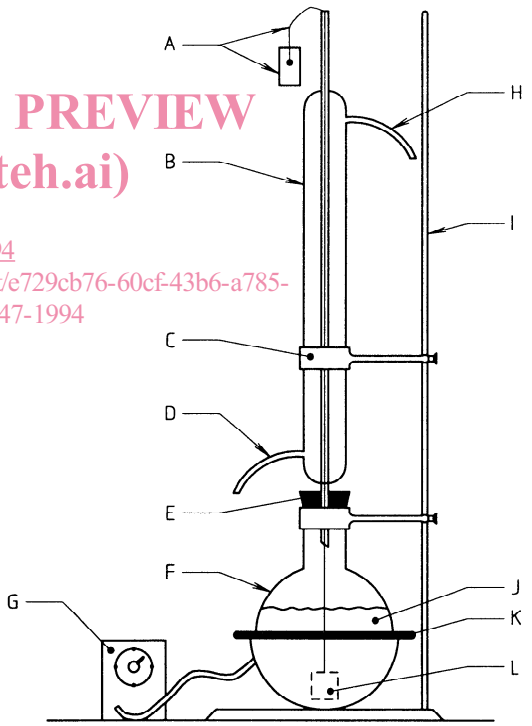
a) a reference to this International Standard;

b) identification of the test pieces;

c) the degree of crosslinking, G_i, for each test piece and the mean value G;

d) details of any variation in the specified procedure and of any abnormal behaviour observed during the test;

e) the date of the test.



Key

A Identification tag and fine wire attached to cage
B Reflux condenser
C Ring-stand clamp
D Water inlet
E Ground-glass joint or cork
F Wide-necked round-bottomed flask
G Variable transformer
H Water outlet
I Ring stand
J Xylene
K Heating mantle
L Wire cage enclosing a test piece

Figure 1 — Extraction apparatus

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ICS 23.040.20; 23.040.45

Descriptors: plastics products, polyethylene, cross-linked materials, plastic tubes, pipe fittings, tests, determination, cross-linking, extraction methods.

Price based on 2 pages