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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**

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

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## Foreword

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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 second edition cancels and replaces the first edition (ISO 10147:1994), which has been technically revised.

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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 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.

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## 3 Solvent

**3.1 Xylene**, analytically pure, to which 1% of antioxidant has been added [either 2,2-methylene-bis(4-methyl-6-*t*-butylphenol) or 3-(3,5-di-*t*-butyl-4-hydroxyphenyl) propionate, or a combination of both].

**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. Excessive inhalation of the vapour may cause dizziness, headache or both. In the event of excessive vapour inhalation, seek fresh, clean air.

## 4 Apparatus

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

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

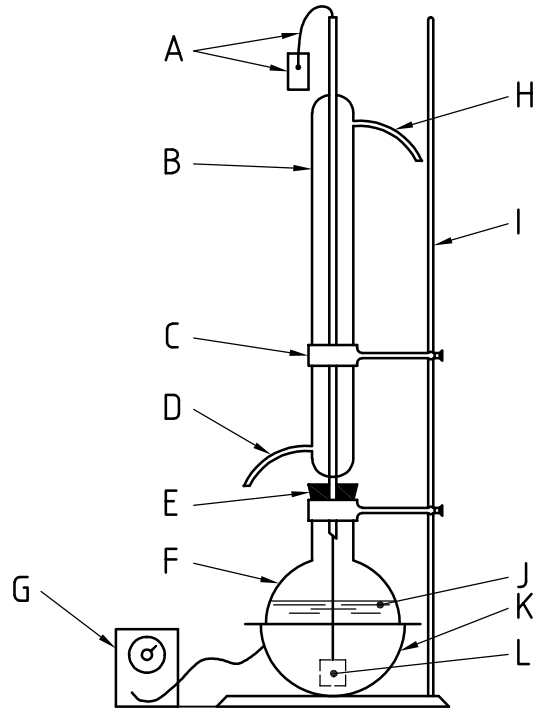
NOTE A 2 000 ml flask is suitable for up to six determinations at one time.

**4.3 Heating mantle**, to fit the flask (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 of  $(125 \pm 25) \mu\text{m}$ . 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**

- A identification tag and fine wire attached to cage
- B reflux condenser
- C ring clamp
- D water inlet
- E ground-glass joint or cork
- F wide-necked round-bottomed flask
- G variable transformer
- H water outlet
- I stand
- J xylene
- K heating mantle
- L wire cage enclosing test piece

**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**

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

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.

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

## 6 Procedure

- 6.1** Weigh a clean, dry cage, including its lid (see 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 solvent to maintain total immersion and to provide a ratio by mass of solvent to test piece of at least 200:1.
- 6.4** Boil the solvent vigorously to ensure good agitation for 8 h  $\pm$  30 min.
- 6.5** Carefully remove the cage and the residue of the test piece from the solvent after the time specified in 6.4. If the residue can be easily separated from the cage, place the residue only in pure boiling xylene for 5 min. If the residue cannot be separated from the cage and lid, place the complete assembly in pure boiling xylene for 5 min.
- NOTE Take care when removing the cage from the boiling solution (see warning in 3.1).
- 6.6** Complete the drying of the residue, or cage, lid and residue, by placing them for at least 3 h in:
- 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 a forced-ventilation oven (see 4.6), with an adequate extraction facility, kept at  $(140 \pm 2)$  °C.
- 6.7** After drying, allow to cool to ambient temperature and weigh the residue (mass  $m_4$ ), or 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 test piece as the percentage by mass of the insoluble material, using one of the following equations, as appropriate:

when the residue only 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.

## 8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for identification of the test piece;
- c) the degree of crosslinking,  $G$ , of the material in the test piece;
- 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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