INTERNATIONAL STANDARD

ISO
11673

First edition
2005-04-01

Unplasticized poly(vinyl chloride) (PVC-U) pressure pipes — Determination of the fracture toughness properties

Tubes en poly(chlorure de vinyle) non plastifié (PVC-U) sous pression — Détermination de la ténacité

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Contents

Page

Foreword

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ISO 11673 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 plastics materials and their accessories — Test methods and basic specifications*.

Introduction

Studies have been undertaken at the international level to determine a method of measuring the characteristics of unplasticized poly(vinyl chloride) (PVC-U) which influence the ability of the pipe to withstand brittle failure emanating from minor flaws in the pipe matrix.

These studies have demonstrated that a test which measures fracture toughness characteristics of the material fulfils these requirements.

The method involves immersing a prepared test piece in dichloromethane to identify the point around the circumference of the pipe where the gelation is at a minimum level. The fracture toughness of the pipe is likely to be at its minimum value at this section.

The fracture toughness of the pipe is then obtained by subjecting a pre-notched C-ring test piece to a flexural stress across a notch, which has been introduced at the point where the fracture toughness value is likely to be at its lowest value (as determined by the dichloromethane immersion test).

It is intended that individual product standards will specify the requirement for the fracture toughness test.

NOTE   Dichloromethane is now the accepted term for what was commonly referred to as methylene chloride.

Unplasticized poly(vinyl chloride) (PVC-U) pressure pipes — Determination of the fracture toughness properties

1   Scope

This International Standard specifies a method for determining the minimum fracture toughness, after a specified loading time, of unplasticized poly(vinyl chloride) (PVC-U) pressure pipes. It also makes provision for measuring alternative levels of fracture toughness.

2   Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6259-2, *Thermoplastics pipes — Determination of tensile properties — Part 2: Pipes made of unplasticized poly(vinyl chloride) (PVC-U), chlorinated poly(vinyl chloride) (PVC-C) and high-impact poly(vinyl chloride) (PVC-HI)*

ISO 9852, *Unplasticized poly(vinyl chloride) (PVC-U) pipes — Dichloromethane resistance at specified temperature (DCMT) — Test method*

3   Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
pipe wall thickness
e
value of the measurement of the wall thickness at any point around the circumference of the pipe, rounded up to the nearest 0,05 mm, expressed in millimetres

3.2
pipe wall thickness at the reference point
e_{ref}
value of the measurement of the wall thickness at the reference point on the pipe circumference where the notch is cut

3.3
C-ring
ring, as test piece, cut from the test specimen such that the cut surfaces are perpendicular to the longitudinal axis of the pipe

3.4
yield stress
σ
tensile stress at yield, expressed in megapascals[1)]

3.5
stress intensity factor
K
product of the yield stress, σ, perpendicular to the crack, a geometrical correction factor, y, and the square root of the notch depth, a

NOTE 1   See Annex A.

NOTE 2   The term *factor* is used because it is common usage, even though the value has dimensions.

3.6
critical stress intensity factor
K_C
value of the stress intensity factor at the critical condition of crack propagation where $K = K_C$

3.7
plain strain fracture toughness
K_{IC}
value of the stress intensity factor when the crack under load actually starts to enlarge under a plane-strain loading condition around the crack tip

NOTE   If the specimen dimensions satisfy the validity criteria for plane-strain fracture toughness, then $K_C = K_{IC}$. K_{IC} is a material property.

3.8
reference line
line drawn along the axis of the pipe sample to mark the point at which the notch is to be cut

NOTE   This will be either the point of greatest attack following the dichloromethane test or, if attack is even all the way round, the point of the thinnest wall section.

4   Symbols

a crack length (notch depth) in millimetres;

d_m moment arm;

e pipe wall thickness in millimetres;

e_{ref} measured wall thickness, in millimetres, at the reference point on the pipe circumference where the notch is cut;

F_e wall thickness factor;

F_r ring geometric factor;

m test mass;

m_c C-ring mass;

[1)]   1 MPa = 1 N/mm^2.

2

m_L support clamp mass;

σ_c fracture stress;

σ_{pc} plastic collapse stress under plane stress conditions;

σ_y uniaxial yield stress.

5   Principle

On the basis of the prior response of a test piece to immersion in dichloromethane, a C-ring section from a pipe is notched on its internal face at the region of maximum attack by dichloromethane. The notched sample is then subjected to a sustained flexural stress for a specified test period.

6   Preliminary test for response to immersion in dichloromethane

6.1   Reagents

6.1.1   **Dichloromethane**, analytical grade

WARNING — The boiling temperature of dichloromethane is low (40 °C) and consequently has a high vapour pressure at ambient temperature. Furthermore, it can be toxic to skin and eyes. It is therefore necessary to take precautions when handling dichloromethane or test pieces, which have been immersed in it. The vapours are also toxic; the threshold limit value (TLV) comparable to the maximum admissible concentration (MAC) being 100 ml/m^3 (ppm). Ventilation of the room or the area in which the container is placed and where the test pieces are dried is, therefore, essential.

The consistency of colour and purity of the dichloromethane should be checked regularly, for example by measuring the refractive index. Whenever the refractive index differs by $\pm$ 0,002 from the original value, the dichloromethane should be changed. Any alternative checking procedure which gives the same result is acceptable.

NOTE   Dichloromethane, technical grade, contains small quantities (1 % maximum each) of chloromethane (CHCl), of trichlormethane (CHCl$_3$) and tetrachloromethane (CCl$_4$). It has been noted that even if the level of these impurities were to attain 5 % in total, the results would not be significantly different.

6.2   Apparatus

6.2.1   Cutting equipment, capable of cutting an external chamfer on the test piece such that the chamfer penetrates at least 90 % of the wall thickness of the test piece with a minimum length of 10 mm when it is cut at an angle inclined to the longitudinal pipe axis.

6.2.2   Covered tank, resistant to dichloromethane, e.g. of glass, stainless steel.

6.2.3   Thermostatic control equipment, capable of maintaining the temperature of the dichloromethane at either (15 $\pm$ 2) °C or (20 $\pm$ 2) °C.

6.3   Test specimen

6.3.1   The test specimen for both the dichloromethane test and fracture toughness test shall be a pipe of length not less than 200 mm.

6.3.2   A reference line shall be drawn along the complete length of the test specimen, and marked with arrowheads to indicate the extrusion direction in such a way that the pipe is not scored. An arrowhead in the same direction shall be marked on the C-ring test piece (see 6.3.3 and 6.3.4).

6.3.3 A ring of width (30 ± 3) mm shall be cut from the test specimen in such a manner that the cut surfaces are perpendicular to the longitudinal axis of the pipe. This C-ring is retained for the fracture toughness test in accordance with Clause 7.

6.3.4 The remainder of the test specimen is used for testing for the effect of dichloromethane. This shall be carried out in accordance with ISO 9852.

Where the tank is not big enough to take a large diameter pipe, it is permissible to cut the test piece longitudinally into sections, provided that the resultant sections can be related to the reference line.

6.4 Procedure

6.4.1 Place the chamfered end(s) of the test piece in dichloromethane such that the chamfer is completely submerged at (15 ± 2) °C for a period of (30 ± 1) min or submerged at (20 ± 2) °C for a period of (15 ± 1) min.

6.4.2 Remove the test piece to a well-ventilated area, e.g. a fume cupboard, until the dichloromethane has evaporated from its surface.

6.5 Assessment of results

6.5.1 For the purpose of the dichloromethane test, the attack is classified by the extent of the lightening of the chamfered surfaces. There are three types of attack, classified as follows.

— Type 1: no apparent lightening of the surface under consideration.

— Type 2: overall uniform lightening of the surface under consideration.

— Type 3: non-uniform lightening of the surface under consideration. The area where the attack is greatest or the lightening is most dense shall be identified.

NOTE   Lightening of the chamfer surfaces can be difficult to discern in pale coloured or white pipe specimens. In such cases, the observation of surface roughening can serve to indicate the extent of attack by dichloromethane.

6.5.2 After carrying out an assessment as described in 6.5.1, inspect the chamfer surface and record the type of attack in relation to the reference line.

## 7 Determination of fracture toughness

7.1 Apparatus

7.1.1 Broach or other **cutting equipment**, capable of cutting a notch of uniform depth, of included angle 45 ± 2°, such that the variation in wall thickness as measured from the outside surface to the notch tip does not exceed 0,1 mm. The tip of the notch shall have a radius not exceeding 0,025 mm. To ensure that this is so, the broach or cutting equipment shall be sharpened every 500 cuts.

7.1.2 Loading device, capable of applying the required force to the test piece such that the maximum initial change in the moment arm (d_m) is not greater than d_m ± 2 % (see Figure 1).

7.1.3 Timing device, accurate to ± 1 s over a period of 15 min.

7.1.4 Rigid support clamps, an example of which is shown in Figure 2. The support clamps shall have an inside radius equal to the outside radius of the pipe and shall be sufficiently rigid to maintain this radius under the load imposed by the test mass (m).

NOTE   The clamps maintain the geometry of the C-ring, which ensures that a controlled bending moment is transmitted to the notched section, and this has been accounted for in the method of calculation.

7.1.5   Mounting device, capable of mounting the assembly such that under load the rotation of the specimen is prevented.

NOTE   Rotation of the specimen would shorten the moment arm (d_m) (see Figure 2). A counter balance is one method of restricting the rotation.

7.1.6   Balance, capable of weighing to an accuracy of $\pm$ 5 g.

a) **Location of test mass**	b) **Alignment of notch**
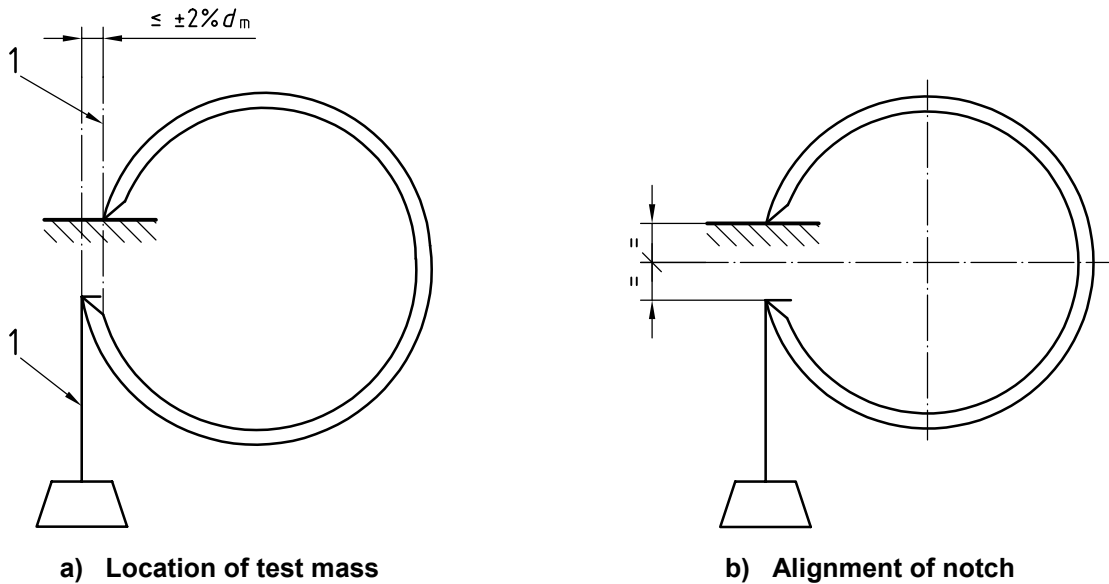
Key
1   centre line of support

Figure 1 — Tolerance on location of test mass and notch after test mass applied

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Dimensions in millimetres

Key

1 hanger
2 test mass (m)
3 support clamp
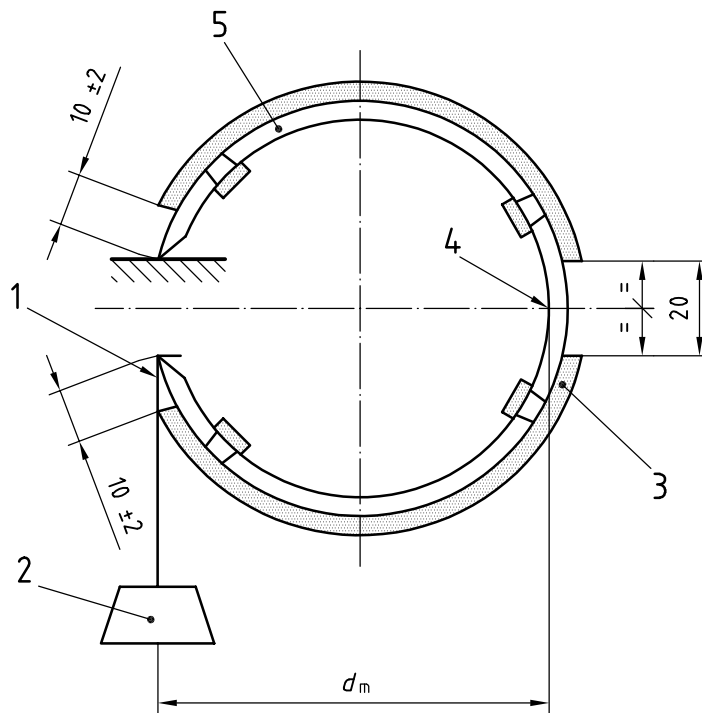
4 notch (25 % e_{ref}), see detail X in Figure 3
5 test piece

**Figure 2 — Typical arrangement of test specimen and equipment**

7.2 Preparation of test piece

7.2.1 Take for the test piece the 30 ± 3 mm pipe ring (C-ring) prepared in accordance with 6.3.3.

7.2.2 Measure and record the external diameter of the test piece rounded to the nearest 0,05 mm.

7.2.3 For test pieces having type 1 and type 2 attack of the chamfered surface, as assessed in the dichloromethane test (see 6.5.1), measure and record the wall thickness rounded to the nearest 0,05 mm and the width of the test piece rounded to the nearest 0,1 mm at the reference line.

For test pieces having type 3 attack of the chamfered surface, measure and record the wall thickness rounded to the nearest 0,05 mm and the width of the test piece rounded to the nearest 0,1 mm at the area corresponding to that of greatest attack, using the reference line as datum.

7.2.4 Cut a notch in the bore of the test piece at the point at which the wall thickness was measured, so that the notch traverses the complete width of the test piece to a depth of 25 % $\pm$ 0,1 mm of the wall thickness at that point (see Figure 3).

7.2.5 Cut out a (20 ± 2) mm section from the ring diametrically opposite the notch (see Figure 3).

7.2.6 Ensure that the clamps are fixed symmetrically within (10 ± 2) mm of the notch tip and the cut ends of the ring as shown in Figure 2.

Dimensions in millimetres
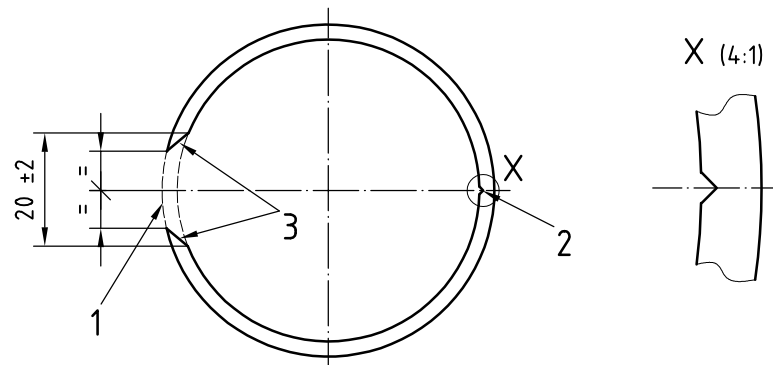
Key

1 section cut from pipe ring sample

2 notch (25 % e_{ref})

3 free ends

Figure 3 — C-ring test specimen

7.3 Conditioning

Condition the test piece, including the clamps at (23 ± 2) °C for an appropriate period conforming to the time given in Table 1.

Table 1 — Conditioning time

Pipe wall thickness e mm	Minimum conditioning time min
$e < 8,7$	30
$8,7 \leqslant e < 13,8$	60
$e \geqslant 13,8$	120

7.4 Procedure

7.4.1 Weigh the C-ring (m_c) and lower support clamp (m_L) to the nearest 5 g.

7.4.2 Calculate the test mass according to 7.4.4.

7.4.3 Whilst maintaining the temperature at (23 ± 2) °C, support the test piece on the cut-out section opposite the notch and apply the test mass to the test piece. Ensure the specimen is aligned equally about the centre line of the notch. At least, ensure that the spacing of the upper and lower arms about the centreline of the notch is equal in respect to one another to within 3 mm. A typical test arrangement is shown in Figure 2. Maintain the force for a period of at least 15 min or until the piece fails across the notch, whichever is the shorter.

7.4.4 Calculation of test masses

Calculate the applicable test mass as follows.

a) Select either

    1) the 15 min yield strength obtained from regression data, or

    2) the 15 min yield strength measured in accordance with Annex C.

b) Select a default value of 4,5 MPa·m$^{1/2}$ for the fracture toughness or calculate the appropriate fracture toughness (K_{IC}) commensurate with the yield stress of the material, using Equation (1):

$$\left(\frac{K_{IC}}{\sigma_y}\right)^2 = 0,0081 \tag{1a}$$

    i.e. $K_{IC} = 0,09 \times \sigma_y$ (1b)

c) Calculate the value of the pipe wall thickness factor (F_e) using Equation (2):

$$F_e = \frac{e^2}{29,43\pi} \times \sigma_{pc} \times \cos^{-1}\left[\exp\left(\frac{-\pi^2 K_{IC}^2}{0,008 y^2 \sigma_{pc}^2 a}\right)\right] \tag{2}$$

where σ_{pc} is the plastic collapse stress under plane stress conditions calculated using Equation (3):

$$\sigma_{pc} = 1,891 \times \sigma_y \times \left(1 - \frac{a}{e_{ref}}\right)^2 \tag{3}$$

where

a is the crack length (notch depth) in millimetres;

e_{ref} is the pipe wall thickness, in millimetres, at the point where the notch is cut;

y is 1,914 for a/e_{ref} = 0,25.

NOTE σ_{pc} = 1,064 × σ_y for a/e_{ref} = 0,25

d) Calculate the value of the ring geometry factor (F_r) using Equation (4):

$$F_r = \frac{w}{d_m} \quad \text{(see Annex A)} \tag{4}$$

where

w is the width of the C-ring;

d_m = ($D - e_{ref}$) and D is the external diameter of the pipe, in millimetres.

e) Calculate F_c using Equation (5):

$$F_c = \frac{m_c}{4} + \frac{m_L}{2} \tag{5}$$

f)   Calculate the test mass (m) using Equation (6):

$$m = \left(F_r \times F_e \right) - F_c \tag{6}$$

NOTE 1   A detailed explanation of the calculation of the factors F_e and F_r is given in Annex A.

NOTE 2   Calculation of the test mass can be done by taking into account the values of the pipe diameter, C-ring width, notch depth and wall thickness. A simple computer spreadsheet can be useful for this.

8   Expression of results

At the conclusion of the test: record a pass if the test piece is intact at the end of the 15 min; record a fail if the test time is not achieved.

9   Test report

The test report shall include the following information:

a)   reference to this International Standard, i.e. ISO 11673;

b)   identification of the pipe:

—   nominal size,

—   nominal wall thickness (e) or pipe series (S);

c)   date of test;

d)   value of fracture toughness required;

e)   results of test as summarized in Clause 8.

Annex A
(normative)

Mathematical calculations

A.1 For notch-sensitive materials, including unplasticized poly(vinyl chloride) (PVC-U), failure may be predicted in the presence of a sharp notch using a fracture mechanics model. The relationship between the applied stress (σ) and the stress intensity factor (K) is defined by Equation A.1.

$$K = \sigma \times y \times \sqrt{a} \tag{A.1}$$

where

y is the geometrical correction factor;

a is the notch depth.

The applied stress at failure is defined as the critical applied stress (σ_c) and the resultant stress intensity factor is then called the critical stress intensity factor (K_c).

$$K_c = \sigma_c \times y \times \sqrt{a} \tag{A.2}$$

Where the dimensions of the test piece are large by comparison with those of the notch, so that plane strain conditions prevail, $K_c = K_{IC}$.

K_{IC} is the plane strain fracture toughness and is an intrinsic property of the material.

However, when PVC-U pipes are tested as C-rings, the dimensions of the test piece do not satisfy the criteria for valid plane strain conditions. In particular, the relatively low wall thicknesses of pipes generally do not satisfy the depth validity criterion. With low depth values and high toughness levels, it is possible for the stress on the section of material ahead of the notch to approach the collapse stress of the material. In such cases, it is necessary to modify the simple linear elastic equation [Equation (2)] used to calculate the stress needed to derive a valid toughness value. Compensation for this deficiency in the sample dimensions is provided by using a solution to the Bilby, Cottrell, Swindon model to predict the critical stress as shown in Equation A.3.

$$\sigma_c = \frac{2}{\pi} \sigma_{pc} \cos^{-1} \left\{ \exp\left[-\left(\frac{\pi^2 K_{IC}^2}{8 y^2 \sigma_{pc}^2 a} \right) \right] \right\} \tag{A.3}$$

where

σ_{pc} is plastic collapse stress under plane stress conditions;

K_{IC} is plane strain fracture toughness;

a is crack length (notch depth), in millimetres;

y is 1,914 for $a/e = 0,25$.

$$\sigma_{pc} = 1,891\ \sigma_y \left(1 - \frac{a}{e}\right)^2 \tag{A.4}$$

and

$$y = \sqrt{\pi} \times \left[1,12 - 1,39\left(\frac{a}{e}\right) + 7,32\left(\frac{a}{e}\right)^2 - 13,1\left(\frac{a}{e}\right)^3 + 14,0\left(\frac{a}{e}\right)^4 \right] \tag{A.5}$$

NOTE   When a/e = 0,25, then y is 1,914.

The stress due to bending in a rectangular beam is defined according to Equation (A.6).

$$\sigma = \frac{6M}{e_b^2 w} \tag{A.6}$$

where

   M is the bending moment;

   e_b is the thickness of the beam;

   w is the width of the C-ring.

It can be shown [1] that:

$$F_e = \frac{e^2}{3g_n\pi} \sigma_{pc} \cos^{-1}\left\{ \exp\left[-\left(\frac{\pi^2 K_{IC}^2}{8y^2 \sigma_{pc}^2 a} \right) \right] \right\} \tag{A.7}$$

where g_n is the gravitational constant,

then

$$m_t = F_e \times F_\gamma \tag{A.8}$$

However, the mass of the C-ring itself and the lower clamps supporting the ring contribute to the moment arm. Therefore, the mass m to be applied is determined by subtracting this contribution from m_t.

$$m = m_t - \left(\frac{m_c}{4} + \frac{m_L}{2} \right) = m_t - F_c \tag{A9}$$

$$m = \left(F_e \times F_r \right) - F_c \tag{A.10}$$

Annex B
(informative)

Further guidance

B.1   This International Standard specifies a method for establishing the minimum fracture toughness level for unplasticized poly(vinyl chloride) (PVC-U) pressure pipe in specific ranges of wall thickness as detailed in Table 2. This concept is based on the assumption that the constant logarithmic reductive slope holds over a considerably long time, up to 100 years survival. Measurement of the fracture toughness values at longer times than 15 min can provide a reductive slope and, as a result, can give a regression equation which allows a more reliable toughness value to be quoted for PVC-U pressure pipes. This aspect of testing is therefore to be explored further and the test specification amended as appropriate.

B.2   In general, the technique of introducing a notch in the specimen has a significant effect on the fracture toughness of brittle materials, including PVC-U. However, once critical sharpness of the notch tip is attained, the fracture toughness becomes independent of the tip radius. If a notch having a tip radius less than the critical limit (0,050 mm) can be made, a notching apparatus other than a broach can also be used.

B.3   A reduction in sharpness of a broaching tool leaves residual stress around the tip of the notch even if the radius of the notch tip is less than 30 µm. However, daily checking of the tool is difficult because there is no effective method to measure the sharpness of the blade. It is necessary to set the maximum cutting cycle of the broaching tool that keeps the notch tip free from residual stress and of the required sharpness. As a precaution, it is recommended that the blade be resharpened after every 500 cuts.

Annex C
(normative)

Determination of yield strength

C.1 Scope

When the value of yield strength of the pipe material is not taken from regression data, the value of yield strength to be used in the calculations shall be determined as follows.

C.2 Method

The 15 min yield stress value shall be determined in accordance with ISO 6259-2, except that duplicate tensile specimens shall be tested at crosshead speeds of 5, 10, 25, 50 and 100 mm/min. For each specimen, the time to yield and the yield stress will be recorded. A graph of log yield stress versus log time shall be plotted and the yield stress at a time of 15 min obtained by applying a power law curve fit to the data.

C.3 Calculation

Calculate the mean yield strength of the three specimens that yielded within (15 ± 2) min.

Bibliography

[1] WHITTLE, A., BURFORD, R.P. and HOFFMAN, M. Improvements to C-ring fracture toughness test for poly(vinyl chloride) pipes. *Plastics, Rubber and Composites*, Vol. 129, No. 8. p. 417, 2000

ISO 11673:2005(E)

ICS 23.040.20

Price based on 14 pages