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Plastics piping systems — Multilayer pipes — Determination of the oxygen permeability of the barrier pipe

Systèmes de canalisations en plastiques — Tubes multicouches — Détermination de la perméabilité à l'oxygène de la couche barrière d'un tube

Reference number
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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 17455 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*.

Introduction

In response to the worldwide demand for specifications, requirements and test methods for multilayer pipes, WG 16 of ISO/TC 138/SC 5 was created at a meeting held in Kyoto, Japan, in 1998. The working group then started drafting three test standards (including ISO 17455) for multilayer pipes:

- ISO 17456, *Plastics piping systems — Multilayer pipes — Determination of long-term strength*;
- ISO 17454, *Plastics piping systems — Multilayer pipes — Test method for the adhesion of the different layers by using a pulling rig*.

Only multilayer pipes are dealt with in this International Standard and for these purposes cross-linked polyethylene (PE-X) as well as adhesives are to be considered as a thermoplastics material.

Plastics piping systems — Multilayer pipes — Determination of the oxygen permeability of the barrier pipe

1 Scope

This International Standard specifies two test methods for determining the oxygen permeability of barrier pipe: the dynamic (Method I) and the static (Method II). In principle, both methods give the same results. The method to be applied is not application-dependent, but can be specified in the referring standard.

2 Principle

The principle is measurement of the oxygen transfer through the wall of the test piece under specified conditions.

The oxygen increase is measured in a system of which the test piece forms part. Oxygen can only be transported through the wall of the test piece. Therefore, the increased amount of oxygen in the closed system is the result of the functioning of the barrier layer of the test piece.

NOTE A certain continuous oxygen permeation through the barrier layer is allowed.

3 Terms and definitions

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

3.1
multilayer pipe
pipe comprising layers of different materials

3.2
multilayer M pipe
multilayer pipe comprising layers of polymers and one or more metal layers

NOTE The wall thickness of the pipe consists of at least 60 % polymer layers.

3.3
multilayer P pipe
multilayer pipe comprised of two or more polymer layers

3.4
inner layer
layer in contact with the liquid or gas

3.5
outer layer
layer exposed to the outer environment

3.6
embedded layer
layer between the outer and inner layer

NOTE There can be more than one embedded layer.

3.7
barrier layer
layer intended to prevent or greatly diminish oxygen transport from outside the pipe into the inside water

NOTE For multilayer P pipes, the barrier layer is normally not stress-designed.

3.8
closed system
system which comprises stainless steel pipes, couplings and a tap, as well as the test piece, allowing only oxygen to permeate from the outside to the inside of the test piece

3.9
flux
oxygen permeability of the barrier layer of the pipe

4 Symbols and abbreviated terms

A_{barr}	outside surface of the barrier layer, in square metres (m^2)
$C_{\mathrm{ox},\,t}$	oxygen concentration after time , in grams per cubic metre (g/m^3)
F_{ox}	flux expressed in oxygen transfer per unit per area (of the barrier layer),in grams per square metre per hour ($g/m^2 \cdot h$)
T	test temperature (40°C or 80°C), expressed in degrees Centigrade
V_{app}	volume of the closed system, excluding the volume of the test piece, in cubic metres (m^3)
V_{pipe}	volume of the test piece (pipe sample), in cubic metres (m^3)
d_{b}	outside diameter of the barrier layer, millimetres (mm)
d_{e}	manufacturer's nominal outside diameter, expressed in millimetres (mm)
d_{i}	inside diameter of the test piece (pipe), in millimetres (mm)
l	length of the test piece, in metres (m)
P_{a}	standard atmospheric pressure (1 000 mbar at 20°C), expressed in bar1)
P_{fin}	atmospheric pressure at the end of the measurement, expressed in bar
P_{init}	atmospheric pressure at the start of the measurement, expressed in bar
t	test period, in hours (h)
β_{pr}	dimensionless correction factor to atmospheric pressure

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1) 1 bar = 0,1 MPa = 10^5 Pa; 1 MPa = 1 N/mm^2

$\int_0^6 O_{2,\mathrm{fin}}$ total amount of oxygen after six hours, in grams (g)

$\int_0^{6=0} O_{\mathrm{H_2O,init}}$ total amount of oxygen in the water at the start of the test, in grams (g)

5 Apparatus

The test assembly shall include the following main elements:

NOTE 1 The test temperature (40°C or 80°C) is specified in the relevant product- or system standard.

NOTE 2 1 ppb = 1 g/m^3.

5.1 Oven, capable of maintaining a constant temperature of (40 ± 0,5) °C and/or (80 ± 0,5) °C.

5.2 Test rig, a closed system consisting of stainless steel parts of pipes, couplings, valves (only for Method II) and taps, including the test piece.

5.3 Water circulation pump, capable of a variable delivery with a capacity range of from 0,15 dm^3/min to 0,5 dm^3/min.

5.4 Oxygen sensor, Capable of functioning at (40 ± 0,5) °C and/or (80 ± 0,5) °C, with a range of from 0,1 ppb to 20 ppm[2].

5.5 Water pressure meter, with a range of (1 ± 0,1) to (4 ± 0,1) bar.

5.6 Atmospheric pressure meter, with a range of from (965 ± 1) mbar to (1035 ± 1) mbar.

5.7 Water flow meter, with a range of from (0,15 ± 0,05) dm^3/min to (0,5 ± 0,05) dm^3/min.

5.8 Water temperature meter, capable of functioning at (40 ± 0,05) °C and/or (80 ± 0,05) °C.

5.9 Air temperature meter, capable of functioning at (40 ± 0,05) °C and/or (80 ± 0,05) °C.

5.10 Airtight vessel, for preparation of water with an oxygen concentration of < 10 ppb (nominally oxygen-free)

NOTE 3 Normally, sink plates or nitrogen are used to remove the oxygen from the water by purging.

5.11 Test medium, deionized water with PH 7 (demi/water).

5.12 Registration device, capable of registering (graphical writer or computer) oxygen concentration as a function of time.

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[2] “Parts per billion (ppb)” and “parts per million (ppm)” are used exceptionally in this International Standard in order to correspond to other, closely related and already published standards. The accepted SI form for the expression of a volume fraction is in units of microlitres per litre (µl/l), or, alternatively, as 10^{-6}, or as a percentage by volume (% by volume); for mass fractions it is expressed in micrograms per gram (µg/g). See ISO 31-0:1992, 2.3.3, and ISO 31-8-15:1992.

6 Test piece

6.1 Number of test pieces

Unless otherwise specified, the number of pipe test pieces shall be one.

6.2 Preparation

The test piece shall have a free length of (20 ± 0,5) m.

Prepare the test piece in accordance with the manufacturer's instructions, taking into account the minimum free length.

The free length between the couplings as well as the inside diameter of the test piece shall be measured and recorded.

7 Preconditioning

The test piece shall be preconditioned in accordance with the referring product or system standard.

When the relevant product or system standard gives no conditioning time, the conditioning time in relation to the wall thickness according ISO 1167 [1] is recommended.

8 Calibration of the test assembly

8.1 Connect the closed system, without the test piece, to the oxygen sensor and pump.

8.2 Remove all air (bubbles) from the system, which has been filled with water.

8.3 Using the vessel, circulate the water at a maximum of 0,5 dm^3/min to produce water with an oxygen concentration of < 10 ppb.

NOTE 1 dm^3/min = 0,0016 m^3/s.

8.4 Stop the production of this water after the oxygen sensor shows a steady state concerning the oxygen concentration in the water.

8.5 After at least 15 min stop the calibration procedure.

9 Preparation for testing

9.1 Installation procedure

9.1.1 Connect the test piece in the closed system using the relevant stainless steel couplings.

9.1.2 Fill the closed system with water and remove all air (bubbles).

9.1.3 Establish the required pressure and temperature.

9.2 Removing the oxygen

9.2.1 Using the vessel, circulate the water through the closed system to produce water with an oxygen concentration of < 10 ppb.

9.2.2 Stop the production and circulation of this water after the oxygen sensor shows a level of $\leqslant$ 220 ppb (220 µg/l) for a test temperature of 40 °C, or 100 ppb (100 µg/l) for a test temperature of 80 °C.

NOTE As long as the final oxygen concentration is much smaller than the solubility values per Annex A, the driving force of the transfer of oxygen can be considered as being constant.

10 Measuring procedure

10.1 General

For measuring the oxygen increase, two different methods are allowed: Method I and Method II.

NOTE With Method I the water is circulated; whereas, in Method II it is stagnant.

10.2 Dynamic test method (Method I) (see Figure 1)

10.2.1 Circulate the water continuously with a volume flow of maximum 0,5 dm^3/min through the system without refreshment. Maintain the temperature of the water as well of the surrounding air at the specified values. Maintain the inside water at the specified pressure.

10.2.2 Using the oxygen sensor, continuously measure the total amount of the oxygen in the water.

10.2.3 After one hour, start the measurement of the oxygen concentration in the inside water.

NOTE During 5 h, the rate of change in oxygen concentration in the inside water is constant (Stage 2 in Figure 1).

10.2.4 Repeat the procedure according to 9.2 before each measurement.

10.2.5 Only when three measurements in succession have values within 5 % of each other during Stage 2, calculate the value of oxygen concentration in the inside water, F_{ox}.

10.3 Static test method (Method II) (see Figure 2)

10.3.1 After the step according to 9.2.2 (the water circulation has been stopped), close the valves on both sides of the test piece to isolate (make airtight) the test piece from the closed system. To prevent a possible vacuum, first close the outlet valve and then the inlet valve.

10.3.2 Maintain the temperature of the water as well of the surrounding air at the specified values. Maintain the pressure of the inside water at the specified pressure.

NOTE The water is stagnant inside the test piece without refreshment.

10.3.3 Leave for a period of 6 h to let oxygen diffuse into the stagnant water.

10.3.4 After 6 h, open the valves on both sides of the test piece and force the oxygen-free water (coming from the vessel) out the pipe.

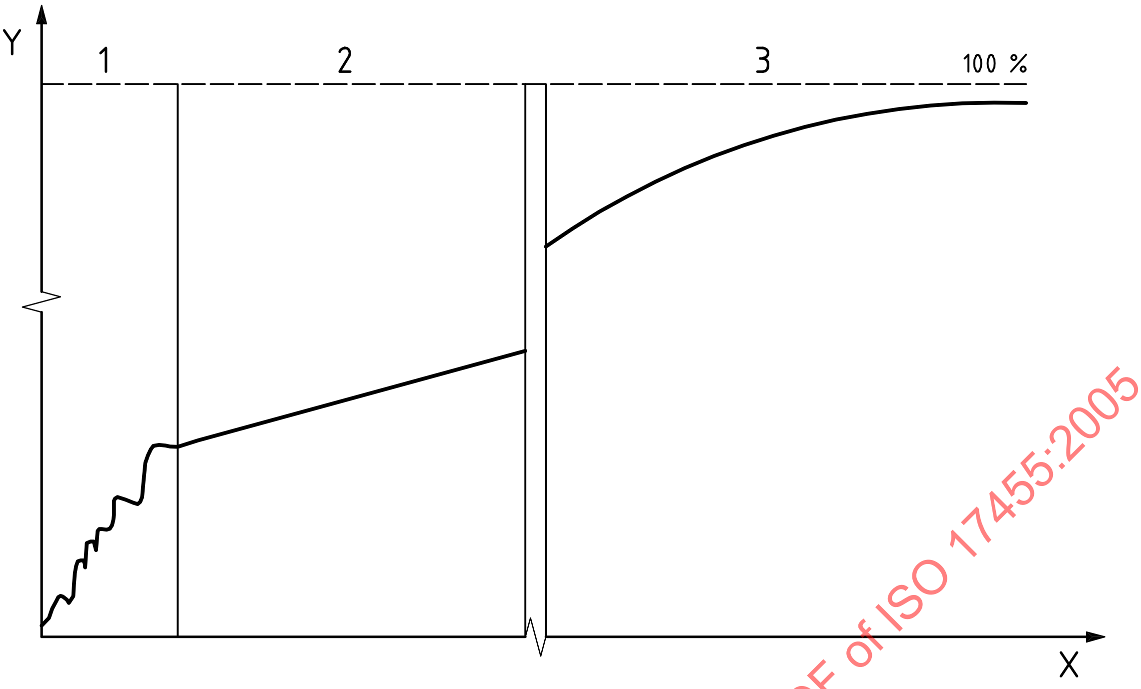
10.3.5 Using the oxygen sensor, measure the total amount of oxygen that issues.

NOTE 1 The water coming from the vessel forces out the stagnant water.

NOTE 2 When the observed concentration of oxygen is displayed as a function of time in Figure 2, the integration of the curve shows the total amount of oxygen flowing past the sensor.

10.3.6 Repeat the procedure according to 9.2 before each measurement.

10.3.7 Only when three measurements in succession have values within 5 % of each other, use the value of the total amount of oxygen in the inside volume of water for calculating F_{ox}.



Key

X time *t*, h

Y oxygen concentration, g/m^3

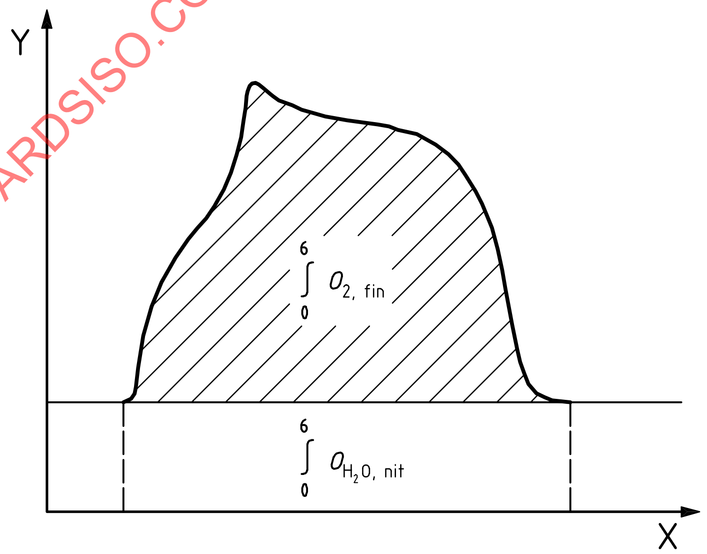
1 stage 1

2 stage 2

3 stage 3

NOTE When the oxygen increase in time is graphically shown, as here, there are three different stages. In Stage 2, the rate of oxygen increase is constant. In Stage 3 the maximum solubility is reached.

Figure 1 — Oxygen increase in a closed system — Method I



Key

X time *t*, h

Y oxygen concentration, g/m^3

Figure 2 — Oxygen concentration in a closed system — Method II

11 Calculation of flux

11.1 Dynamic method (Method I)

Calculate the outside surface of the barrier layer using Equation (1):

$$A_{\text{barr}} = \frac{\pi \times l \times d_{\text{b}}}{1\,000} \qquad (1)$$

Calculate the inside volume of the pipe using Equation (2):

$$V_{\text{pipe}} = \frac{\pi \times l \times d_{\text{i}}^2}{4 \times 10^6} \qquad (2)$$

Calculate the total rate of transfer of oxygen through the pipe wall, expressed in grams per hour, using Equation (3):

$$C_{\text{ox},t} = \frac{(V_{\text{pipe}} + V_{\text{app}})}{t} \qquad (3)$$

Calculate the influence of the atmospheric pressure using Equation (4):

$$\beta_{\text{pr}} = \frac{p_{\text{init}} + p_{\text{fin}}}{2p_{\text{a}}} \qquad (4)$$

Calculate the flux, F_{ox}, expressed in grams per square metre per hour, using Equation (5):

$$F_{\text{ox}} = C_{\text{ox},t} \frac{(V_{\text{pipe}} + V_{\text{app}})}{A_{\text{barr}} t} \beta_{\text{pr}} \qquad (5)$$

Calculate the flux per day, in milligrams per square metre, using Equation (6):

$$F_{\text{ox,day}} = 24\,000 \times F_{\text{ox}} \qquad (6)$$

11.2 Static method (Method II)

NOTE Integration of the curve of Figure 2 yields the total amount of oxygen passing the sensor for the total inside volume of water in the pipe. Dividing this amount by the barrier surface area of the pipe and by the testing time and taking into account the variation of pressure yields the flux.

Calculate the total oxygen content of the pipe after six hours, expressed in grams, using Equation (7):

$$\int_0^6 O_{2,\text{abs}} = \int_0^6 O_{2,\text{fin}} - \int_0^{6=0} O_{\text{H}_2\text{O,init}} \qquad (7)$$

Calculate the outside surface of the barrier layer, expressed in square metres, using Equation (8):

$$A_{\text{barr}} = \frac{\pi \times l \times d_{\text{b}}}{1\,000} \qquad (8)$$

Calculate the inside volume of the pipe using Equation (9):

$$V_{\text{pipe}} = \frac{\pi \times l \times d_{\text{i}}^2}{4 \times 10^6} \tag{9}$$

Calculate the influence of the atmospheric pressure using [Equation (5):]

$$\beta_{\text{pr}} = \frac{p_{\text{init}} + p_{\text{fin}}}{2 p_{\text{a}}}$$

Calculate the flux, expressed in grams per square metre per hour, using Equation (10):

$$F_{\text{ox}} = \int_0^6 O_{2,\text{abs}} \frac{1}{A_{\text{barr}} t} \beta_{\text{pr}} \tag{10}$$

Calculate the flux per day, in milligrams per square metre, using Equation (11):

$$F_{\text{ox,day}} = 24\,000 \times F_{\text{ox}} \tag{11}$$

Then, determine the flux per day in relation to the water volume of the (tested) pipe, using Equation (12):

$$F_{\text{ox,vol}} = \frac{F_{\text{ox}} \times A_{\text{barr}}}{V_{\text{pipe}}} \times 24 \tag{12}$$

12 Test report

The test report shall include the following information:

a) reference to this International Standard, as well as to the referring standard and the manufacturer's information;

b) complete identification of the multilayer pipe component of which the test piece was taken, including manufacturer, material type, code number, size, source and significant history, if any;

c) dimension of the test piece (pipe sample) used for the testing;

d) test conditions;

e) calculated values;

f) any observations of damage;

g) method applied (Method I or II);

h) all relevant parameters (P_{init}, P_{fin}, P_t, V_{pipe}, testing time, etc);

i) test results (F_{ox}, $F_{\text{ox, day}}$, $F_{\text{ox, vol}}$);

j) any factors that could have affected the results, such as incidents or any operational details not specified in this standard;

k) date of test.