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Polyethylene coatings for steel pipes and fittings

Requirements and testing

<u>DIN</u> 30 670

Umhüllung von Stahlrohren und -formstücken mit Polyethylen

Supersedes July 1980 edition.

In keeping with current practice in standards published by the International Organization for Standardization (ISO), a comma has been used throughout as the decimal marker.

This standard has been prepared jointly by *DIN Deutsches Institut für Normung e. V.* and *DVGW Deutscher Verein des Gasund Wasserfaches e. V.* (German Gas and Water Engineers' Association). The Standard has been adopted into the DVGW Codes of practice on gas and water.

Dimensions in mm

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Scope and field of application

This standard specifies requirements for and methods of testing extruded and fused polyethylene coatings applied at the works to DIN 2448 or DIN 2458 steel pipes that will be in contact with soil or water. It is intended to be used for quality assurance purposes. When supplied with normal type coatings (N), pipes in compliance with this standard are suitable for continuous service temperatures of up to 50 °C, and with special type coatings (S), for temperatures up to 70 °C. For the purposes of this standard, the term 'steel pipe' includes fittings manufactured from such pipes.

2 Concepts

2.1 Indentation hardness

The indentation hardness is a measure of the resistance of coatings to the penetration of a test cylinder under specified conditions.

2.2 Light ageing

For the purposes of this standard, light ageing is ageing of coatings by exposure to filtered xenon arc radiation in a

laboratory apparatus at a given irradiance, combined with periodic wetting.

2.3 Minimum coating thickness

The minimum coating thickness is the required thickness of coatings at any point.

2.4 Continuity

The continuity of coatings is defined as its freedom from defects such as cracks or pinholes.

2.5 Percentage elongation at failure

The percentage elongation at failure is the increase in the original gauge length of a test piece at the moment of failure, expressed as a percentage of the original gauge length.

2.6 Bond strength

For the purposes of this standard, the bond strength is the force required to peel a strip of coating from the pipe under specified conditions.

Continued on pages 2 to 7

2.7 Impact strength

The impact strength is defined as the impact energy coatings can withstand under specified conditions.

2.8 Coating resistivity

The coating resistivity is the electrical resistance of coatings per unit area.

2.9 Heat ageing

For the purposes of this standard, heat ageing is ageing of coatings by exposure to hot air at a given temperature for a specified period.

3 Designation

Coatings shall be designated as being of the normal (N) or special (S) type (cf. subclauses 4.2.3, 4.2.5 and 4.2.8) and as being applied to a normal (n) or reinforced (v) thickness (cf. subclause 4.2.1), as follows:

Coating DIN 30 670 - N-n Coating DIN 30 670 - N-v Coating DIN 30 670 - S-n

Coating DIN 30670 - S-v

4 Requirements

4.1 Requirements for substrate

Immediately prior to application of the coating, the surface of the steel pipe (substrate) shall be clean (i.e. free from dirt, oil, grease, welding beads, moisture) and blasted to comply with standard preparation grade Sa 2½ as defined in DIN 55 928 Part 4.

4.2 Requirements for coating

4.2.1 Minimum thickness

The minimum coating thickness shall be as specified in table 1.

Table 1.

Nominal size of pipe	Minimum thickness of normal (n) coating, in mm
Up to DN 100	1,8
Over DN 100 up to DN 250	2,0
Over DN 250 to below DN 500	2,2
From DN 500 to below DN 800	2,5
From DN 800	3,0

The minimum coating thickness shall be 0,7 mm greater ('reinforced' (v) type) where coatings will be subjected to particularly high mechanical stress.

Along any 1 m length of pipe, the coating thickness may be up to 10 % less than the minimum required, provided the thinner area does not cover more than 5 cm².

The ends of pipes less than DN 500 in size shall be free from adhesive over a length of 50 mm, those of a least DN 500 in size, over a length of 100 mm, unless otherwise specified by the purchaser.

The length of uncoated pipe ends shall not exceed 150 mm, unless otherwise specified by the purchaser.

4.2.2 Continuity

The coating shall be continuous (i.e. free from pinholes). This requirement shall be deemed satisfied if no breakdown in the coating occurs during spark testing (cf. subclause 5.3.2).

4.2.3 Bond strength

When the coating is tested as specified in subclause 5.3.3, not more than 24 hours after application, the mean force required to lift it off shall,

- a) at a test temperature of (20 ± 5) °C, be at least 35 N per cm of test piece width;
- at a test temperature of (50 ± 5) °C, be a least 15 N per cm of test piece width for type N coatings, and 25 N per cm for type S coatings.

4.2.4 Impact strength

When tested in accordance with subclause 5.3.4, the coating shall withstand 30 impacts without any electrical breakdown occurring.

4.2.5 Indentation hardness

When tested in accordance with subclause 5.3.5, the indentation depth of type N coatings shall not exceed 0,2 mm at a temperature of (23 ± 2) °C, nor 0,3 mm at (50 ± 2) °C. In the case of type S coatings, it shall not exceed 0,3 mm at a temperature of (70 ± 2) °C.

4.2.6 Percentage elongation at failure

When tested in accordance with subclause 5.3.6, the percentage elongation at failure shall be at least 200 %.

4.2.7 Coating resistivity

When tested in accordance with subclause 5.3.7, the coating resistivity, determined after conditioning the test piece in the test medium for 100 days, shall be not less than $10^8\,\Omega$ m².

The ratio of resistivity after 100 days to that after 70 days shall be not less than 0,8, provided the resistivity is only one power of 10 above that specified for 100 days of conditioning.

4.2.8 Heat ageing

When tested in accordance with subclause 5.3.8, the melt flow rate shall not deviate by more than 35 % of the original value.

4.2.9 Light ageing

When tested in accordance with subclause 5.3.9, the melt flow rate of the test pieces shall not deviate by more than 35 % of the original value.

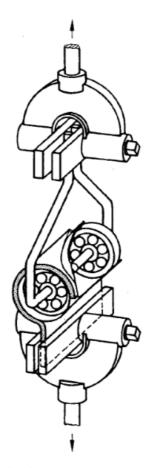


Figure 1. Type 1 test assembly for determining bond strength (taken from the September 1982 edition of DIN 30 674 Part 1)

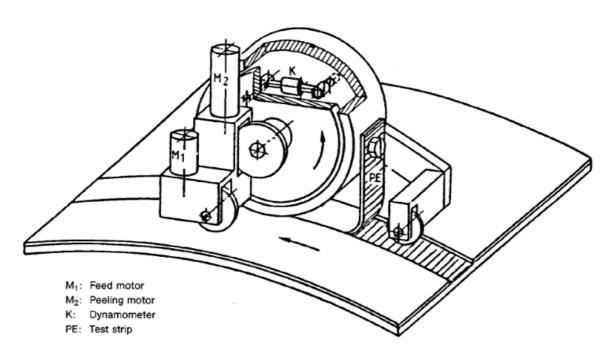


Figure 2. Type 2 test assembly for determining bond strength

5 Testing

5.1 General

Testing may be carried out by the manufacturer or by a recognized test house.

The manufacturer shall be responsible for ensuring compliance with the requirements specified in subclauses 4.1.1, 4.1.2 and 4.2.1 to 4.2.3.

For each coating material and application process, the manufacturer shall provide proof of compliance with the requirements specified in subclauses 4.2.6 to 4.2.9 at intervals of at least three years.

Unless otherwise specified below, samples shall be understood to be pipes or pipe sections.

5.2 Inspection documents

Issuing a DIN 50 049 inspection document shall be the subject of agreement.

5.3 Procedure

5.3.1 Coating thickness

The coating thickness shall be measured at points uniformly distributed along the length and about the circumference of the pipe, using a non-destructive (e.g. magnetic) method which permits measurements to be made to an accuracy of 10 %

5.3.2 Continuity

The coating shall be tested for continuity by means of spark testing, using commercially available high voltage equipment complying with DIN VDE 0433 Part 2, the voltage of which is discharged via a sphere spark gap. The test voltage shall be 25 kV. The electrode (e.g. metal brush) shall be in contact with the coating surface, since any air gap would falsify results.

When a fault is present, a spark will be seen, or the test equipment will emit a signal.

Note. Spark testing is intended to reveal any discontinuities in the coating, not to test the breakdown resistance of a continuous coating.

5.3.3 Bond strength

The bond strength of the coating shall be determined by means of testing using one of the test assemblies illustrated in figures 1 and 2 or equivalent equipment. The coating shall be peeled off at right angles to the pipe surface at a rate of 10 mm/min. Three samples each shall be tested at a temperature of (20 ± 5) °C and (50 ± 5) °C. If one of the samples fails to meet the requirements, the test shall be repeated on five further samples, none of which shall fail.

Using a twin motor saw, an incision shall be made in the coating, at least 20 mm but not more than 50 mm wide, in the direction of the pipe circumference, down to the substrate, and another incision made at right angles to the cuts just made. The test piece so obtained shall be gripped, and a length of about 20 mm lifted from the pipe surface. Disregarding the first and last 20 mm of test strip, the force used to lift off the strip shall be continuously recorded using, for example, a dynamometer. The mean result obtained shall be expressed in N. Any test strip section (defined as being 25 mm in length) for which the mean force applied (in the peeling mode) is more than 25 % lower than the value specified in subclause 4.2.3 shall not be included in the evaluation.

For testing at (50 ± 5) °C, the sample shall be incised as described above and heated to about 60 °C. The temperature of the external surface of the test piece shall be continuously monitored using, for example, a fast-response contact thermometer. Measurements shall start being recorded

as soon as the temperature has dropped to 55 °C, and the test completed before it reaches 45 °C.

5.3.4 Impact strength

In the impact test, a sphere with a diameter of 25 mm shall be dropped onto the sample, which shall be supported so that it does not yield under the impact of the falling weight. The test temperature shall be $(23\pm2)\,^{\circ}\mathrm{C}$, and the drop height approximately 1 m. The impact energy, E, shall be equal to $(5\cdot\varphi)$ J per mm of coating thickness, with a tolerance of 5 %. The factor φ (cf. table 2) accounts for the curvature of the pipe, it being permissible to reduce the impact energy by this amount by reducing the mass of the falling weight or the drop height.

Table 2.

Nominal size (DN)	φ
From 200	1,0
65 to below 200	0,85
Less than 65	0,70

The falling weight shall be guided so as to minimize friction and so that the direction of fall is normal to the pipe surface. The pipe shall be subjected to impact 30 times, the distance between two points of impact being at least 30 mm. Following that, a test voltage of 25 kV shall be applied, and it shall be checked whether breakdown occurs.

5.3.5 Indentation hardness

Indentation hardness testing shall be carried out on three test pieces, taken from coating not more than 2 mm thick, which has been removed from the pipe and freed of adhesive. (It may be necessary to abrade the reverse side of the test piece to obtain the required thickness.) If one of the test pieces fails to meet the requirements, the test shall be repeated on five further test pieces, none of which shall fail.

The indentor shall be a 250 g metal rod to which an additional weight can be attached. A metal pin with a flat face, 1,8 mm in diameter (or 2,5 mm² in area), shall be fitted centrally at the lower end of the rod. The total mass of the assembly shall be 2,5 kg (corresponding to a pressure of 10 N/mm²).

For measurement of indentation depth, a penetrometer with dial gauge, which permits measurements to be made to an accuracy of 0,1 mm, is required.

Testing shall be carried out at temperatures of (23 ± 2) °C and (50 ± 2) °C, the higher temperature being (70 ± 2) °C in the case of type S coatings, using a temperature-controlled water bath, if necessary. After conditioning the test piece for one hour at the test temperature, the indentor (without the additional weight) shall be slowly and carefully lowered on the test piece, and the zero value set at the penetrometer within five seconds. Following this, the additional weight shall be attached to the indentor and, after 24 hours, the depth of penetration read from the penetrometer.

5.3.6 Percentage elongation at failure

The coating of three pipes shall be tested for elongation at failure, from which five test pieces each shall be taken. The test pieces shall have the form of specimen no. 3 (if necessary, half that size) or no. 4, as specified in DIN 53 455. One test piece per pipe shall be permitted fo fail.

In the case of extruded coatings, test pieces shall be taken either by neutralizing the adhesive or by heating the pipe to a maximum of 80 °C. In the case of fused coatings, test pieces shall be taken after the pipe has been heated to a maximum of 100 °C.

Testing shall be performed as described in DIN 53 455, at a temperature of (23 ± 2) °C and a rate of 50 mm/min (or 25 mm/min in the case of half-size test pieces).

5.3.7 Coating resistivity

Three test pieces having a minimum test area of 0,03 m² shall be tested, none of which shall fail.

A sodium chloride solution with a concentration of 0,1 mol/l shall be used as the test medium. The test equipment shall be made up of a counterelectrode with a surface area of not less than 10 cm², a d.c. source with an output voltage of not less than 50 V, an ammeter and a voltmeter. The test pieces shall be exposed to the test medium for 100 days at a temperature of (23 ± 2) °C, in one of the ways described below.

- a) The pipe shall be placed horizontally in a plastic container with lateral openings of suitable size. The space between container walls and test piece shall be sealed with a non-conductive material. The test medium shall be poured in, covering the pipe to a depth of at least 100 mm.
- b) Plastic pipe sections shall be bonded to the surface of the sample (pipe or pipe section) using a non-conductive adhesive. The former shall then be filled with the test medium.
- c) One end of a pipe shall be sealed with non-conductive material so that the steel surface is not in contact with the test medium. The pipe shall then be placed vertically in a vessel containing the test medium. The pipe may be removed from the medium and wetted with an electrolyte solution to measure resistivity.

The coating shall be tested for continuity (cf. subclause 5.3.2) before being tested for resistivity.

For the purposes of measurement, the positive pole of the d.c. source shall be connected to the steel pipe, and the negative pole to the counterelectrode, which shall then be immersed in the test medium. The coating resistivity, $r_{\rm u}$, in Ω m², shall be calculated from the following equation:

$$r_{\rm u} = \frac{U \cdot S}{I}$$

where

U is the voltage between counterelectrode and steel pipe, in V:

S is the test area, in m2;

I is the intensity of current flowing through the coating, in A

The current flowing through the d.c. source is equal to that flowing through the coating if there are only negligible leakage currents along the surface between the test area and the steel at the pipe ends. Leakage current earthing complying with DIN VDE 0303 Part 3 shall be provided where the intensity of measuring current, *I*, is high. The results of measurement are only correct if the leakage current intensity is substantially lower than *I*.

5.3.8 Heat ageing

Heat ageing shall be carried out using an oven with forced circulation. The test pieces, which have been taken from the coating and freed of adhesive, shall be not more than 2 mm thick. (It may be necessary to abrade the reverse side of the test piece to obtain the required thickness.) Type N test pieces shall be subjected to ageing at a temperature of 100 °C for 100 days (2400 h), and type S test pieces, for 200 days (4800 h).

The test pieces shall be removed from the oven at intervals of 400 h, and the melt flow rate determined in accordance with DIN 53 735, using condition D for type N, and condition T for type S test pieces.

5.3.9 Light ageing

To determine the resistance of coatings to light ageing, the changes in the melt flow rate of test pieces shall be determined after a specified period of exposure to filtered xenon arc radiation and periodic wetting, in accordance with DIN 53 387, test condition 1-A-X.

Sampling shall be carried out in accordance with DIN 53 735, using condition D for type N, and condition T for type S test pieces. Test pieces which have been removed from the pipe and freed of adhesive shall be not more than 2 mm thick. (It may be necessary to abrade the reverse side of the test piece to obtain the required thickness.)

Before exposure and after a radiant exposure of about 1,2 GJ/m², the melt flow rate shall be determined in accordance with DIN 53 735.

Evaluation shall be made after a total radiant exposure of 7 GJ/m².

6 Marking

Steel pipes as covered in this standard shall be clearly and durably marked with at least the following particulars:

- a) manufacturer's mark or registered trade mark;
- b) DIN number;
- symbol denoting type of coating (N or S) and thickness (n or v) (cf. clause 3).

Example:

(Mark) - PE coating DIN 30 670 - N-n

Standards referred to

DIN 2448 Seamless steel pipes and tubes; dimensions and mass per unit length

DIN 2458 Welded steel pipes and tubes; dimensions and mass

DIN 30 674 Part 1 Polyethylene coatings for ductile iron pipes

DIN 50 049 Inspection documents for the delivery of metallic materials

DIN 53 387 Artificial weathering and ageing of plastics and elastomers by exposure to filtered xenon arc radiation

DIN 53 455 Tensile testing of plastics

DIN 53 735 Determination of melt flow rate of thermoplastics

DIN 55 928 Part 4 Corrosion protection of steel structures by the application of organic or metallic coatings; preparation

and testing of surfaces

DIN VDE 0303 Part 3 Testing of materials used in electrical engineering; measurement of electrical resistance of non-metallic

materials

DIN VDE 0433 Part 2 High-voltage generation and measurement; voltage measurements using sphere gaps (one sphere

earthed)

ASTM G 8-90 Test method for cathodic disbonding of pipeline coatings

Previous editions

DIN 30 670: 02.74, 07.80.

Amendments

The following amendments have been made to the July 1980 edition.

a) A coating type suitable for service temperatures up to 70 °C (type S) has been included for the first time.

- b) The method of coating application is no longer of relevance.
- c) Different requirements have been made for coatings of greater thickness.
- d) The method for determining bond strength has been amended.
- e) The test for resistance to light ageing has been brought into line with DIN 53 387, and now includes wetting.

Explanatory notes

This standard has been jointly prepared by Technical Committee AA 5.2 of the Normenausschuß Gastechnik (Gas Technology Standards Committee), the Normenausschuß Wasserwesen (Water Practice Standards Committee), the Normenausschuß Rohre, Rohrverbindungen und Rohrleitungen (Pipes, Pipe Joint Assemblies and Pipelines Standards Committee) and the Normenausschuß Kunststoffe (Plastics Standards Committee).

The aim of this standard is to ensure that polyethylene pipe coatings provide adequate protection against the mechanical, thermal and chemical stresses occuring in service, transit and storage, and during installation. Pipes are coated by the extrusion of plastics sleeving or sheeting, or by the tusion of polyethylene powder. A coating thickness of 1 mm is adequate for corrosion protection. A greater thickness increases the resistance to mechanical stresses.

The specifications given here account for both higher demands in practice (in terms of mechanical stength and thermal stability) and recent developments in polyethylene materials research. Type S coatings should be used where service temperatures are high and where high mechanical strength is required.

The classification of polyethylene coatings into normal and reinforced coatings (the latter being 0,7 mm thicker) has proved expedient in practice and applies to both types of coating.

In the previous edition, the peel test (cf. subclause 5.3.3) was supplemented by a test involving the suspension of a weight. The latter is no longer specified, since test results are not reproducible. This edition specifies that the peel test be carried out at ambient and elevated temperature. Testing at low temperatures can be dispensed with, since bond strength usually increases as temperature decreases. Adhesives which become brittle at low temperatures are no longer used in practice because they do not meet the requirements for adequate bond strength at 50 °C.

Both fused and extruded coatings are required to have the same value of percentage elongation at failure, which justifies their being covered together here.

The heat ageing test at 100 °C is intended to give an indication of the minimum resistance of the coating to thermal stresses in storage and in service. Testing is accelerated and represents severe conditions. Since type S coatings are expected to conform to higher demands in service, the heat ageing test shall be carried out for twice as long as for the normal type.

Testing for resistance to light ageing is also accelerated, and shall be carried out in accordance with DIN 53 387. This is intended to prevent differences in test equipment from influencing test results. Wetting the test pieces is intended to prevent the formation of deposits on the test piece surface, which might falsify test results.

Experience gathering in transporting, laying and servicing pipes with polyethylene coating has shown that a high bond strength is necessary to avoid mechanical damage to the pipes. Based on experience and extensive research, it has also been shown that bond strength may decline over time, regardless of the ambient conditions, and that coating may disbond entirely if damaged. However, this involves neither an increased risk of corrosion (e.g. underrusting) nor an increase in the protective current density requirement in cathodic corrosion protection.

Bond strength is not a significant factor with regard to the protection the coating provides against corrosion, as long as the thickness and quality of the coating meet the requirements specified here. However, it is desirable that the coating adhere well over large areas to avoid mechanical damage during pipelaying. Therefore, except for the determination of coating resistivity, no other electrochemical tests are included here.

Determination of resistance to blistering is irrelevant because such does not occur in the coating materials

covered here. Similarly, determination of cathodic disbonding is not included, since this test does not provide relevant information with regard to the quality of polyethylene coatings and their long-term corrosion protection. Should determination of cathodic disbonding be required, American Standard ASTM G 8-90 specifies that the method described in DIN 30 671 be used. The values obtained, however, are only reproducible for the test conditions specified therein.

The responsible technical committee agreed that it would be useful to determine the tear resistance of polyethylene coatings. To this end, a number of studies have been conducted under various test conditions, but it has not been possible to obtain practical, reproducible results. Work in this regard is continuing.

Polyolefines other than polyethylene (e.g. polypropylene) may also be used to coat steel pipes and fittings. However, since some of the characteristics of these materials differ considerably from polyethylene, their properties cannot be determined in accordance with this standard. It is intended to prepare separate standards for these materials.

International Patent Classification

B 29 C 63/18 E 03 B 7/09 F 16 L 57/00 F 16 L 58/04

G 01 B 21/00 G 01 M 19/00