
**Petroleum and natural gas industries —
External coatings for buried or
submerged pipelines used in pipeline
transportation systems —**

**Part 4:
Polyethylene coatings (2-layer PE)**

*Industries du pétrole et du gaz naturel — Revêtements externes des
conduites enterrées ou immergées utilisées dans les systèmes de
transport par conduites —*

Partie 4: Revêtements à base de polyéthylène (PE bicouche)



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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 21809-4 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 2, *Pipeline transportation systems*.

ISO 21809 consists of the following parts, under the general title *Petroleum and natural gas industries — External coatings for buried or submerged pipelines used in pipeline transportation systems*:

- *Part 1: Polyolefin coatings (3-layer PE and 3-layer PP)*
- *Part 2: Fusion-bonded epoxy coatings*
- *Part 3: Field joint coatings*
- *Part 4: Polyethylene coatings (2-layer PE)*
- *Part 5: External concrete coatings*

Introduction

It is necessary that users of this part of ISO 21809 be aware that further or differing requirements can be needed for individual applications. This part of ISO 21809 is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This can be particularly applicable where there is innovative or developing technology. Where an alternative is offered, it is the responsibility of the vendor to identify any variations from this part of ISO 21809 and provide details.

Petroleum and natural gas industries — External coatings for buried or submerged pipelines used in pipeline transportation systems —

Part 4: Polyethylene coatings (2-layer PE)

1 Scope

This part of ISO 21809 specifies the requirements for qualification, application, inspection, testing, handling and storage of materials for plant application of two-layer polyethylene coatings (2-layer PE) applied externally for the corrosion protection of bare steel pipe for use in pipeline transportation systems for the petroleum and natural gas industries as defined in ISO 13623.

NOTE Pipes coated in accordance with this part of ISO 21809 are considered suitable for additional protection by means of cathodic protection.

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 31-0:1992, *Quantities and Units — Part 0: General principles*

ISO 62, *Plastics — Determination of water absorption*

ISO 306, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)*

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 527-3, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets*

ISO 868, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1133, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

ISO 1872-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 1183 (all parts), *Plastics — Methods for determining the density of non-cellular plastics*

ISO 4625-1, *Binders for paints and varnishes — Determination of softening point — Part 1: Ring-and-ball method*

ISO 4892-2:2006, *Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

ISO 21809-4:2009(E)

ISO 8501-1:2007, *Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness — Part 1: Rust grades and preparation grades of uncoated steel substrates and of steel substrates after overall removal of previous coatings*

ISO 8502-3:1992, *Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness — Part 3: Assessment of dust on steel surfaces prepared for painting (pressure-sensitive tape method)*

ISO 8502-6, *Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness — Part 6: Extraction of soluble contaminants for analysis — The Bresle method*

ISO 8502-9, *Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness — Part 9: Field method for the conductometric determination of water-soluble salts*

ISO 8503-4:1988, *Preparation of steel substrates before application of paints and related products — Surface roughness characteristics of blast-cleaned steel substrates — Part 4: Method for the calibration of ISO surface profile comparators and for the determination of surface profile — Stylus instrument procedure*

ISO 8503-5:2003, *Preparation of steel substrates before application of paints and related products — Surface roughness characteristics of blast-cleaned steel substrates — Part 5: Replica tape method for the determination of the surface profile*

ISO 10474, *Steel and steel products — Inspection documents*

ISO 11124 (all parts), *Preparation of steel substrates before application of paints and related products — Specifications for metallic blast-cleaning abrasives*

ISO 11125 (all parts), *Preparation of steel substrates before application of paints and related products — Test methods for metallic blast-cleaning abrasives*

ISO 11357-6, *Plastics — Differential scanning calorimetry [DSC] — Part 6: Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)*

ISO 13623, *Petroleum and natural gas industries — Pipeline transportation systems*

ISO 15512, *Plastics — Determination of water content*

ASTM D1693, *Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics*

ASTM D3236, *Standard Test Method for Apparent Viscosity of Hot Melt Adhesives and Coating Materials*

SSPC-SP1, *Solvent Cleaning*

3 Terms and definitions

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

3.1 applicator

company that undertakes the coating application in accordance with the provisions of this part of ISO 21809

NOTE If the compounding of the top layer is done prior to or during the application process by the applicator, then he is considered as manufacturer (see 3.7).

3.2 batch

quantity of coating material as defined by the manufacturer

3.3**batch certificate**

certificate of analysis issued by the manufacturer

3.4**certificate of compliance**

one of the types of documents defined by ISO 10474 that is issued in accordance with the purchase requirements

3.5**cutback**

length of pipe left uncoated at each end for joining purposes

3.6**holiday**

coating discontinuity that exhibits electrical conductivity when exposed to a specific voltage

3.7**manufacturer**

company responsible for the manufacture of coating material(s)

3.8**manufacturer's specification**

document that specifies the characteristics, test requirements and application recommendations for the coating materials

3.9**maximum design temperature**

maximum temperature that the coating can be exposed to during operation

3.10**pipe diameter length**

any length along the pipe axis equal to the specified outside diameter of the pipe

3.11**pipeline**

those facilities through which fluids are conveyed, including pipe, pig traps, components and appurtenances, up to and including the isolating valves

[ISO 13623]

3.12**pipeline transportation system**

pipeline with compressor or pump stations, pressure control stations, flow control stations, metering, tankage, supervisory control and data acquisition system (SCADA), safety systems, corrosion protection systems, and any other equipment, facility or building used in the transportation of fluids

3.13**purchaser**

company responsible for providing the product order requirements

3.14**test report**

document that provides the quantitative test results for tests conducted in accordance with the requirements of this part of ISO 21809

3.15**test ring**

sample taken from production coated pipe

4 Abbreviated terms

BST	black standard temperature
ESCR	environmental stress cracking resistance
MFR	melt flow rate; designated as symbol q_{MFR} for use in mathematical expressions and equations
PE	polyethylene
ppd	per pipe diameter
SAW	submerged arc welding
UV	ultraviolet
2LPE	2-layer polyethylene coating

5 General requirements

5.1 Rounding

Unless otherwise stated in this part of ISO 21809, to determine conformance with the specified requirements, observed or calculated values shall be rounded to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with ISO 31-0:1992, Annex B, Rule A.

NOTE For the purpose of this provision, the rounding method of ASTM E29 is equivalent to ISO 31-0:1992, Annex B, Rule A.

5.2 Compliance with this part of ISO 21809

A quality system and an environmental management system should be applied to assist compliance with the requirements of this part of ISO 21809.

NOTE ISO/TS 29001 gives sector-specific guidance on quality management systems and ISO 14001 gives guidance on the selection and use of an environmental management system.

The applicator shall be responsible for complying with all of the applicable requirements of this part of ISO 21809. It shall be permissible for the purchaser to make any investigation necessary in order to be assured of compliance by the applicator and to reject any material and/or coating that does not comply.

6 Information supplied by the purchaser

6.1 General information

The purchase order shall include the following information:

- a) the number of this part of ISO 21809 and year of publication (ISO 21809-4:2009);
- b) pipe quantity, outside diameter, minimum wall thickness, minimum, maximum and nominal length; grade of steel;
- c) bare pipe standard or specification designation, e.g. ISO 3183;
- d) coating system classification and thickness;

- e) cutback length and tolerances for both ends of pipe;
- f) pipeline maximum design temperature, in degrees Celsius;
- g) test temperature for flexibility;
- h) maximum cathodic disbondment radius at maximum design temperature.

6.2 Additional information

If applicable, purchase orders shall include the following information:

- a) additional surface treatments;
- b) plant inspection by the purchaser;
- c) increased test-ring length;
- d) test-ring location;
- e) test frequency for additional test rings;
- f) additional markings;
- g) handling procedures;
- h) storage procedures;
- i) waiver of test reports;
- j) other special requirements.

7 Coating classification

7.1 General

The coating shall consist of two layers:

- a) 1st layer, composed of one of the following adhesives:
 - asphaltic modified rubber,
 - non-asphaltic-modified rubber (e.g. butyl based),
 - hot melt,
 - polymeric;
- b) 2nd layer, consisting of the PE top layer.

7.2 Coating systems

The coating system shall be classified in accordance with Table 1.

Table 1 — Coating systems

Coating systems	A	B	C
1st layer	Asphaltic-modified rubber adhesive	non-asphaltic-modified rubber adhesive	hot melt or polymeric adhesive
2nd layer	PE	PE	PE

NOTE Temperature resistance of coating systems varies based on the polyethylene and adhesive systems specified.

7.3 Coating thickness classes

The total thickness of the coating shall be in accordance with Table 2.

Table 2 — Minimum total coating thickness

Nominal pipe diameter mm	Coating thickness ^{a,b} mm		
	A minimum adhesive: 0,15	B minimum adhesive: 0,2	C minimum adhesive: 0,25
< 115	0,75	0,95	1,25
115 to 275	0,85	1,00	1,40
276 to 510	1,00	1,20	1,60
511 to 760	1,25	1,40	1,80
> 760	1,35	1,75	2,00

^a The total thickness may be reduced by a maximum of 10 % on the weld seam for SAW welded pipes.

^b The choice of coating thickness is dependent on the condition of the soil or ground, laying method onshore and offshore, service constraints and pipe dimensions. These are minimum values and can be changed according to country or project specific requirements.

The applicator shall identify the materials and shall confirm that the certificates comply and relate to the specified materials.

8 Materials

8.1 Pipe

The pipe being coated shall conform to the pipe standard or specification that is specified in the purchase order.

CAUTION — Pipe conforming to such standards or specifications does not necessarily have a surface condition that is appropriate for the application of coating, e. g. temporary coating, salt contamination, slivers.

8.2 Coating materials

8.2.1 General

The applicator shall use materials that are

- a) certified by the manufacturer as in accordance with the requirements of 8.2.2 and 8.2.3;
- b) identified with the following:
 - manufacturer's name,
 - product description,
 - mass of material,
 - batch number,
 - location of manufacture,
 - temperature and humidity requirements for transportation and storage,
 - the year, month and day of manufacture, and
 - expiry date;
- c) handled, transported and stored prior to use in accordance with the manufacturer's recommendations.

8.2.2 Adhesive

Each batch of adhesive shall be tested by the manufacturer in accordance with the requirements of Table 3 or Table 4. Test results shall be reported in accordance with ISO 10474 and shall be made available to the applicator on request. A batch certificate shall be provided by the manufacturer to the applicator.

Table 3 — Minimum requirements for adhesives in systems A and B

Properties	Requirements		Test method
	A	B	
Viscosity	Within 30 % of manufacturer's specification, expressed in millipascal-seconds	N/A	ASTM D3236
Specific gravity at (25 ± 3) °C	0,9 to 1,2	0,95 to 1,5	ISO 1183
Melt flow rate	N/A	Within 20 % of manufacturer's specification, expressed in grams per 10 min	ISO 1133
Ring and ball softening point	At least 20 °C above maximum design temperature	N/A	ISO 4625-1

Table 4 — Minimum requirements for adhesives in system C

Properties	Requirements (hot melt)	Requirements (polymeric)	Test method
Viscosity	Within 20 % of manufacturer's specification, expressed in millipascal-seconds	N/A	ASTM D3236
Water absorption	< 0,1 %	N/A	ISO 62
Ring and ball softening point	At least 20 °C above maximum design temperature	N/A	ISO 4625-1
Melt flow rate	N/A	Within 20 % of manufacturer's specification, expressed in grams per 10 min	ISO 1133
Moisture content	N/A	< 0,1 %	ISO 15512
Vicat softening temperature A/50 (9.8N)	N/A	≥ 85 °C	ISO 306

8.2.3 Polyethylene

8.2.3.1 General

The applicator shall use polyethylene material that is in compliance with Table 5. Test results shall be reported in accordance with ISO 10474.

Table 5 — Minimum requirements for polyethylene top layer

Properties	Requirements	Test method
Density of base resin ^a	≥ 0,930 g/cm ³	ISO 1183
Melt flow rate at 190 °C: 2,16 kg	Within 20% of manufacturer's specification	ISO 1133
Elongation at break at 23 °C: ± 3 °C	≥ 600 %	ISO 527-2, ISO 527-3
Tensile yield strength at 23 °C: ± 3 °C ^b	≥ 10 MPa	ISO 527-2, ISO 527-3
Vicat softening temperature A/50 (9,8 N)	≥ 95 °C	ISO 306
Water absorption	≤ 0,05 %	ISO 62
Hardness Shore D	≥ 45	ISO 868
ESCR (50 °C, F50, Cond. B, 100 % Igepal CO 630)	≥ 300 h	ASTM D 1693
Oxidation induction time (intercept in the tangent method) ^c	≥ 30 min at 210 °C, or ≥ 10 min at 220 °C	ISO 11357-6
UV resistance and thermal ageing ^d	$\Delta q_{MFR} \geq 35 \%$	Annex A

^a The test specimen shall be a compression-moulded sheet annealed for 30 min at 100 °C

^b The test specimen shall be a 2 mm thick compression-moulded sheet prepared in accordance with ISO 527-2, strained at 50 mm/min. The specimen may also be prepared according to ISO 1872-2.

^c Manufacturer shall select one test condition only.

^d This test shall be performed only if UV additives have been included by manufacturer.

8.2.3.2 Compounding during processing

The polyethylene shall be compounded and stabilized by additives against UV and thermal ageing.

If the compounding is done by the applicator during the coating process, the applicator shall qualify the process, conduct all the relevant tests versus the requirements of this part of ISO 21809, and shall certify that the compounded polyethylene is in compliance with this part of ISO 21809.

8.3 Packaging

The coating materials shall be contained in packaging that is clearly labelled to identify the items specified in 8.2.1 b).

For bulk shipments, where the container is not specifically marked for that product (e.g. railcars), the manufacturer shall provide documentation to ensure traceability in accordance with the requirements of 8.2.1.

9 Coating qualification

The coating system shall be qualified by the applicator for each coating line.

Qualification shall be achieved by successful evaluation of pipe coated using the specified coating line. The requirements for testing of pipe samples are given in Table 6. The qualification shall be repeated in case of modifications to the coating line, application procedures or coating materials, including the following:

- material composition;
- change in manufacturer;
- location of manufacturing.

Table 6 — Minimum requirements for coating qualification testing

Property	Requirements for coating system			Test method
	A	B	C	
Cathodic disbondment, 28 days at 23 °C ± 3 °C	12 mm radius max.	20 mm radius max.	20 mm radius max.	Annex B
Cathodic disbondment, 28 days at max. design temperature	Purchaser's specification	Purchaser's specification	Purchaser's specification	Annex B
Impact resistance at 23 °C ± 3 °C	> 3,0 J/mm	> 3,0 J/mm	> 3,0 J/mm	Annex C
Indentation at 23 °C ± 3 °C	< 0,4 mm	< 0,4 mm	< 0,4 mm	Annex D
Flexibility at 0 °C and/or -30 °C	No cracking of PE at 2,5° ppd minimum	No cracking of PE at 2,5° ppd minimum	No cracking of PE at 2,5° ppd minimum	Annex E
Peel adhesion at 23 °C ± 3 °C	> 3,0 N/25 mm	> 10,0 N/25 mm	> 50 N/25 mm	Annexes F or G

10 Application

10.1 General

A coating being applied during production shall have been previously qualified in accordance with the requirements of Clause 9.

10.2 Surface preparation

10.2.1 Initial preparation

All dirt, deleterious matter and contaminants, such as oil and grease, shall be removed from the pipe prior to coating. If necessary, pipe shall be cleaned in accordance with the requirements of SSPC-SP1.

All steel defects and irregularities (e. g. laminations, slivers, scratches) shall be removed by an approved grinding method. Grinding of steel defects shall not reduce the wall thickness below the specified minimum wall thickness of the pipe.

All pipe shall be dry prior to entering the abrasive blast cleaning unit(s). Pipe temperature shall be at least 3 °C above the dew point immediately prior to abrasive blast cleaning.

10.2.2 Abrasive blast cleaning

The abrasives used in the coating plant shall be in accordance with the requirements of ISO 11124.

NOTE If the pipe being coated is of a high-strength grade, e.g. L555, L690 or L839 (USC equivalents: X80, X100 or X120, respectively) harder abrasives can be needed to provide the required cleanliness and surface profile.

The abrasives (including recycled materials) shall be maintained clean, dry and free from contaminants in accordance with ISO 11125 so as not to contaminate the substrate.

For system A and B, the cleanliness achieved prior to application shall be in accordance with the requirements of ISO 8501-1:2007, Grade Sa 2.

For system C, the cleanliness achieved prior to application shall be in accordance with the requirements of ISO 8501-1:2007, Grade Sa 2½.

For systems A and B, the height of the surface profile attained shall be within 40 µm and 100 µm as measured in accordance with the requirements of ISO 8503-4:1988, stylus method, or ISO 8503-5:2003, replica tape method.

For system C, the height of the surface profile attained shall be within 50 µm and 100 µm as measured in accordance with the requirements of ISO 8503-4:1988, stylus method or ISO 8503-5:2003, replica tape method.

If grinding is required after blast cleaning, the maximum allowable area of grinding shall be 10 cm² per metre of pipe length.

10.2.3 Surface cleanliness

The surface cleanliness shall be measured in accordance with the requirements of ISO 8502-3:1992, pressure-sensitive tape method. The maximum allowable level shall be Class 2.

10.2.4 Surface pre-treatment

If the applicator chooses a surface pre-treatment (e.g. deionized water or phosphoric acid pre-treatment), the pre-treatment process shall be agreed with the purchaser.

If surface pre-treatment is used for qualification, it shall be used for production.

If surface pre-treatment is not used, testing for the presence of soluble salts on pipe shall be undertaken in accordance with the requirements of ISO 8502-6 or ISO 8502-9. The maximum allowable level shall be 20 mg/m² after blasting.

If high levels (above 20 mg/m²) of soluble salts are measured, a surface pre-treatment cleaning process shall be agreed upon by the applicator and the purchaser.

10.3 Pipe temperature

Application temperature of the external pipe surface shall be as selected by the applicator within the manufacturer's recommendation.

10.4 Application of adhesive

The adhesive selected by the applicator shall be applied in accordance with the manufacturer's recommendation. The minimum adhesive thickness shall be as given in Table 2.

10.5 Application of polyethylene

The polyethylene may be applied immediately following the application of the adhesive to the pipe. The minimum polyethylene thickness shall be as given in Table 2.

10.6 Cutback

The cutback length and tolerances on both ends of the pipe shall be as specified in the purchase order.

NOTE The polyethylene can undergo growth or shrinkage following cutback due to temperature change.

11 Inspection and testing

11.1 Inspection

If specified in the purchase order, inspection shall be undertaken by the purchaser. All inspections shall be made at the place of application prior to shipment and shall be conducted without undue interference with the operation of the plant. The purchaser may require that the applicator set aside pipe as requested for inspection or testing, or both.

11.2 Testing

11.2.1 Incoming raw materials testing

The applicator shall conduct the sample preparation, testing and evaluation of the coating using suitable equipment at the application facility.

The minimum testing frequency shall be one sample on every batch of coating materials received.

The properties of the adhesives shall be in accordance with the requirements of Table 7 or Table 4.

Table 7 — Incoming raw materials requirements for adhesive in systems A and B

Properties	Requirements		Test method
	A	B	
Viscosity	Within 30 % of manufacturer's specification, expressed in millipascal-seconds	N/A	ASTM D3236
Melt flow rate	N/A	Within 20 % of manufacturer's specification, expressed in grams per 10 min	ISO 1133
Ring and ball softening point	At least 20° C above maximum design temperature	N/A	ISO 4625-1

The properties of the polyethylene shall be in accordance with the requirements of Table 8.

Table 8 — Incoming raw materials requirements for PE

Properties	Requirements	Test method
Melt flow rate	Within 20 % of manufacturer's specification	ISO 1133

11.2.2 In-process inspection / measurement by applicator

11.2.2.1 Surface finish

The surface finish shall be monitored and recorded a minimum of every 2 h during production to determine whether the cleanliness is in accordance with the requirements of 10.2.3. Pipes that fail to conform to the requirements shall be rejected and may be reblasted by agreement between purchaser and applicator.

11.2.2.2 Surface profile

At least once every 4 h of production, the external surface profile on two pipes shall be measured and recorded using a profilometer, replicating film or purchaser-approved equivalent. The profile shall be in accordance with the requirements of 10.2.2. Pipes that fail to conform to the requirements shall be rejected and may be reblasted by agreement between purchaser and applicator.

11.2.2.3 Visual inspection

After cleaning, each pipe shall be visually inspected for surface defects and surface imperfections that can cause holidays in the coating. Such surface imperfections shall be removed by grinding, provided that the remaining wall thickness is within specified limits. Pipe containing surface defects shall be rejected or repaired at the purchaser's option.

11.2.2.4 Application temperature

The surface temperature of the pipe during the coating application shall be monitored and controlled within the limits determined by the applicator.

Once the coating temperature is established, the temperature shall be monitored and recorded once every hour.

Any change in the coating parameters, e.g. line speed, can necessitate a modification in the frequency of temperature measurement.

11.2.2.5 Coating thickness

The total thickness and the thickness of the individual components shall be measured at four random locations along the pipe at different circumferential positions, using a coating thickness gauge that has been calibrated at least once every working shift (to a maximum of 12 h), and shall be recorded.

Such tests shall be conducted on each of the following, as applicable to the coating run lengths:

- first pipe coated in the run;
- one pipe from the subsequent lot of four pipes sequentially coated in the run;
- one pipe from the subsequent lot of 10 pipes sequentially coated in the run;
- one pipe from the subsequent lot of 35 pipes sequentially coated in the run; and
- one pipe from the subsequent lot of 50 pipes sequentially coated in the run.

If the coated pipe does not meet the thickness requirements of 7.3, it shall be rectified in accordance with the requirements of 12.1.

11.2.3 Holiday inspection

11.2.3.1 General

The entire coated surface of each length of pipe shall be inspected with a holiday detector having a search electrode made of conducting material.

For inspection, the direct-current potential of the detector shall be set to exceed 10 V per micrometre of nominal coating thickness, up to a maximum of 25 kV. The detector shall be calibrated at least once every working shift (to a maximum of 12 h).

11.2.3.2 Acceptance criteria

No holidays shall be permitted in finished coating.

Pipe having less than three holidays shall be repaired in accordance with the requirements of 12.2. Any pipe having three or more holidays, or a holiday having an axial length greater than 300 mm shall be stripped and recoated in accordance with the requirements of 12.3.

11.2.4 Production test rings

11.2.4.1 Facilities

The applicator shall have suitable facilities available at the application plant for the preparation, testing and evaluation of test ring samples for tests described in Table 9.

The applicator shall have the option of conducting the tests directly on the coated pipe or on cut test rings.

11.2.4.2 Test rings

Test rings shall be obtained from the end and shall be of sufficient size to provide the mandatory tests in accordance with Table 9.

11.2.4.3 Testing requirements

The minimum test frequency of Table 9 shall be one test ring per pipe diameter every working shift (up to a maximum of 12 h).

For pipe that is stripped and recoated, at least one test ring of the stripped and recoated pipe shall be taken for each order item. If specified in the purchase order, additional test rings shall be taken.

Table 9 — Production coating testing requirements

Properties	Requirements			Test method
	A	B	C	
Cathodic disbondment: 24 h, (65 ± 3) °C	12 mm radius max.	20 mm radius max.	20 mm radius max.	Annex B
Peel adhesion at (23 ± 3) °C	> 3,0 N/25 mm	> 10,0 N/25 mm	> 50,0 N/25 mm	Annexes F or G
Impact resistance at (23 ± 3) °C	> 3,0 J/mm	> 3,0 J/mm	> 3,0 J/mm	Annex C

11.2.4.4 Retests

11.2.4.4.1 If a test fails to conform to the specified requirements, either

- a) the test that failed shall be repeated using two additional test samples taken from the originally tested end of the affected pipe, or
- b) all pipe coated after the previous acceptable test and prior to the next acceptable test shall be stripped and recoated in accordance with the requirements of 12.3.

If both retests conform to the specified requirements, the coated pipe shall be accepted.

11.2.4.4.2 If one or both of the retests fail to conform to the specified requirements, either

- a) all pipe coated after the previous acceptable test and prior to the next acceptable test shall be stripped and recoated in accordance with the requirements of 12.3, or
- b) subject to the approval of the purchaser, further retesting may be performed to determine which pipes coated after the previous acceptable test are acceptable. Pipes that are not acceptable shall be stripped and recoated in accordance with the requirements of 12.3.

12 Repair of coated pipe

12.1 Rectification of below-thickness coating

If required by 11.2.2.5, coated pipe that does not meet the thickness requirements but meets the requirements of Table 9 may be overcoated by applying adhesive and polyethylene in accordance with the requirements of 10.4 and 10.5.

At least one production test ring shall be taken from the over-coated pipe during each 12 h working shift. The test rings shall be prepared and tested in accordance with the requirements of 11.2.4. The peel adhesion between the polyethylene layers shall be tested using the applicable method and acceptance criterion in accordance with Table 9.

12.2 Repair

If required by 11.2.3.2 or 14.1, coated pipe shall be repaired in accordance with 12.2 and 12.3.

The repair shall be performed by the application of a compatible adhesive-lined heat-shrink sleeve, patch material or melt stick.

If a sleeve or patch is used, the repair shall overlap at least 50 mm of the undamaged coating. If a melt stick is used, the repair shall overlap at least 10 mm of the undamaged coating.

The repair material shall be applied in accordance with the manufacturer's recommendations.

All repairs shall be holiday-tested in accordance with the requirements of 11.2.3.

12.3 Stripping and recoating

If required, all rejected coated pipe shall be stripped to bare metal and new coating applied in accordance with the requirements of Clause 10.

The temperature during the stripping operation shall not exceed 275 °C.

The applicator shall record the identity of each stripped and recoated pipe.

13 Markings

13.1 General

Coated pipe shall be marked in accordance with the requirements of 13.2 and with any additional markings specified in the purchase order. Additional markings as desired by the applicator shall be by agreement.

13.2 Required markings

The following markings shall be placed on the coating:

- applicator's name or mark;
- number of this part of ISO 21809 and year of publication: ISO 21809-4:2009;
- markings required by the applicable pipe specification or standard;
- date of coating application; and
- pipe number.

Marking shall be carried out using a method, such as stencil painting or printing, to ensure that the identification is legible and indelible.

14 Handling and storage in the coating area

14.1 Handling

Coated pipe shall be handled in a manner that avoids damage to the pipe, pipe ends and coating. If specified in the purchase order, the applicator shall submit details of the handling procedures; such procedures shall include loading requirements where the applicator is responsible for loading.

Pipe that is damaged during processing shall be repaired in accordance with the requirements of the applicable pipe specification or standard.

Coating that is damaged after the holiday inspection shall be repaired in accordance with the requirements of 12.2 or by stripping and recoating in accordance with 12.3.

14.2 Storage

The applicator shall utilize methods for yard storage to avoid damage to the coating. If specified in the purchase order, the applicator shall submit details of the facilities and the methods for storage.

15 Test reports and certificates of compliance

Unless specified in the purchase order that test reports are waived, the applicator shall furnish test reports to the purchaser in accordance with ISO 10474.

The applicator shall furnish certificates of compliance stating that the coating has been manufactured, applied, inspected and tested in accordance with the requirements of this part of ISO 21809 and any other requirements specified in the purchase order, and that the results of the coating tests and other required tests have been found to conform to such requirements.

Annex A (normative)

UV ageing test and thermal ageing test

A.1 UV-ageing

A.1.1 General

The test consists of subjecting PE material test samples to the continuous irradiation of a xenon lamp under given temperature and humidity conditions.

The change in the PE material shall be assessed by the variation in its elongation at break or in its melt flow rate.

A.1.2 Equipment

The equipment shall consist of an irradiation chamber equipped with a xenon lamp; see ISO 4892-2.

A.1.3 Sampling

The test shall be carried out on a sample of PE in accordance with ISO 1872-2 for PE.

A.1.4 Procedure

The size of the sample shall be large enough to make 10 test specimens. Expose the test specimen under the following conditions:

- artificial weathering; see ISO 4892-2:2006, method A, Table 1);
- black standard temperature equal to $65\text{ °C} \pm 3\text{ °C}$;
- relative humidity equal to $(65 \pm 5)\%$;
- spray cycle: $18\text{ min} \pm 0,5\text{ min spray}/102\text{ min} \pm 0,5\text{ min dry}$;
- total radiant energy equal to 7 GJ/m^2 ;
- continuous exposure.

Calculate the arithmetic mean, \bar{q}_{MFR1} , of the results obtained. Calculate the melt flow rate, q_{MFR0} , in accordance with ISO 1133 on a test sample of identical shape that has not been placed in the test chamber.

A.1.5 Results

A.1.5.1 Melt flow rate

Calculate the variation of the melt flow rate after exposure, Δq_{MFR} , expressed as a percentage, using Equation (A.1):

$$\Delta q_{\text{MFR}} = \frac{\bar{q}_{\text{MFR}1} - q_{\text{MFR}0}}{q_{\text{MFR}0}} \times 100 \quad (\text{A.1})$$

where

$q_{\text{MFR}0}$ is the initial melt flow rate measured before exposure;

$\bar{q}_{\text{MFR}1}$ is the mean of the melt flow rates measured after exposure.

A.1.5.2 Elongation at break

The elongation at break after exposure, tested in accordance with ISO 527-3, shall be a minimum of 150 %.

A.2 Thermal ageing

A.2.1 General

The test consists of subjecting PE material test samples to the effect of dry heat from a thermostatically controlled oven.

The change in the PE material shall be assessed by the variation in its melt flow rate.

A.2.2 Equipment

The equipment shall consist of a thermostatically controlled oven with air circulation maintaining a test temperature within ± 2 °C.

A.2.3 Sampling

The test shall be carried out on a sample of PE material in accordance with ISO 1872-2 for PE.

A.2.4 Procedure

Carry out the test at a temperature of 100 °C ± 3 °C.

Take three melt flow rate measurements on the test sample in accordance with ISO 1133.

Calculate the arithmetic mean, $\bar{q}_{\text{MFR}1}$, of the results obtained. Calculate the melt flow rate, $q_{\text{MFR}0}$, in accordance with ISO 1133 on a test sample of identical shape that has not been placed in the oven.

A.2.5 Test results

Express the variation of the melt flow rate, Δq_{MFR} , after 2 400 h or 4 800 h, as a percentage using Equation (A.1).

Annex B (normative)

Cathodic disbondment test

B.1 General

The test consists of assessing the resistance to disbondment of damage to the coatings when exposed to cathodic polarization.

The test shall be performed on test specimens taken from the coated components previously subjected to holiday-detection, in which an artificial defect of a defined size has been drilled. The test may be performed on the coated component without cutting test specimens.

B.2 Equipment

B.2.1 Electrical source, consisting of a stabilized d.c. power unit that is the source for the voltage and the current.

The current source shall be capable of supplying 20 mA to each test area simultaneously. A cathodic polarization potential of – 1 500 mV to a saturated calomel reference electrode shall be maintained; see also B.2.3.

B.2.2 Electrolytic cell, having a typical configuration as shown in Figure B.1 for large-diameter components and in Figure B.2 for small-diameter components.

The electrolytic cell shall be comprised of the following:

- rigid plastic pipe with a minimum internal diameter of 50 mm; the height shall be such that the total volume of the electrolyte is equal to or greater than 150 ml with a minimum height of the electrolyte of 70 mm;
- rigid plastic cover in which holes are drilled to allow the passage of the electrodes and any other measuring instruments deemed necessary, and to allow the escape of hydrogen.

B.2.3 Electrodes, consisting of the following.

B.2.3.1 Reference electrode, capable of producing a suitable potential (B.2.1).

The electrode is placed in an electrode holder situated in a glass pipe with a porous end diaphragm. The end of this assembly shall be placed approximately 10 mm from the surface of the coating and approximately 20 mm from the coating defect; see Figure B.1.

The reference electrode used shall be suitable for the test temperature required.

B.2.3.2 Auxiliary electrode (anode), consisting of an inert material, e.g. platinum wire with a diameter of 0,8 mm to 1,0 mm.

It shall be immersed in the electrolyte (within approximately 10 mm over the coating defect).

B.2.3.3 Working electrode (cathode), represented by the artificial defect, which shall be 6 mm in diameter, with a maximum depth of 0,5 mm in the steel substrate; see B.3 and Figure B.3.

The ratio of the surface area of the anode to that of the cathode (surface of artificial defect) shall be greater than 1.

B.2.4 Electrolyte, consisting of a 3 % mass fraction solution of NaCl in distilled or deionized water.

The solution shall be made from analytical grade sodium chloride.

The pH of the electrolyte at $23\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ during the test shall be in the range of 6 to 9.

The height of the electrolyte in the cell shall be $75\text{ mm} \pm 5\text{ mm}$.

B.2.5 Heating equipment, suitable to establish and to maintain the test temperature of the sample.

If not heated in an oven, the temperature shall be checked on the steel surface by an appropriate means, e.g. a temperature sensor.

B.3 Sampling

The test samples shall be cold cut from a coated component and shall have a minimum size of $80\text{ mm} \times 80\text{ mm}$, unless the test is performed on the body of the coated component.

Tests samples shall not be taken from the weld area.

For each sample, the thickness of the area of the coating subject to the test shall be measured and recorded.

The integrity of the coating on all test samples shall be checked by holiday detection.

A 6 mm diameter hole (see Figure B.3) shall be drilled through the coating in the centre of the test sample using a standard drill bit. The depth of the drilled hole in the steel substrate shall not exceed 0,5 mm. At the initiation of the test, the total surface area subject to the test shall be free from residual coating.

The test area shall be degreased using a suitable solvent and then rinsed with potable water and subsequently dried.

B.4 Procedure

Seal the plastic pipe forming the electrolytic cell using a suitable sealant, e.g. a chemically inert adhesive. The artificial defect shall be in the centre of the cell.

Fill the cell with the NaCl electrolyte (B.2.4).

Apply a negative cathodic potential between the reference and working electrodes (see Figure B.1 or Figure B.2), with an accuracy of $\pm 10\text{ mV}$. If a saturated calomel electrode is used, the potential shall be $-3\ 500\text{ mV}$ for the 24 h , $(65 \pm 3)\text{ }^{\circ}\text{C}$ test and $-1\ 500\text{ mV}$ for all other test durations.

Continue the test for the test period required. Readjust the level of the electrolyte with distilled or deionized water, if necessary.

B.5 Investigation procedure

After the test, remove the cell with the electrolyte. Rinse the test sample with water, and cool and dry it. Carry out the examination within 1 h of removing the samples from the test equipment.

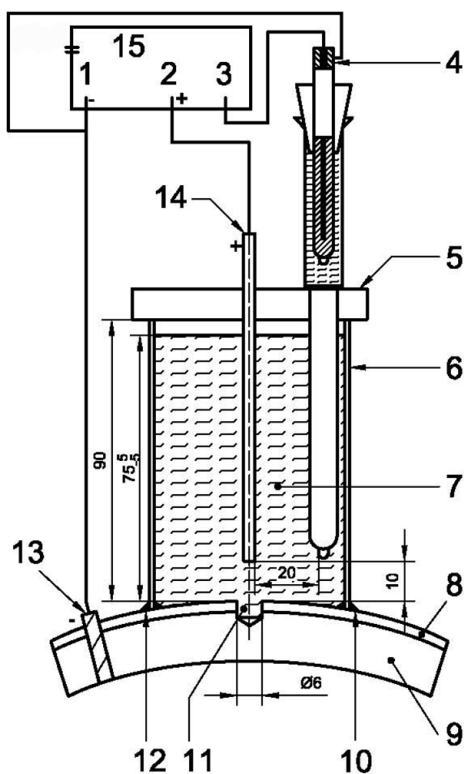
After drying, examine the area of the coating subjected to the test in accordance with the following method.

- Inspect and assess each coating immediately after the test period. Detach the plastic pipe from the test site. Using a lint-free paper towel, wipe along the surface of the coating and cathode area to remove moisture.
- Make about 12 radial incisions using a sharp knife through the coating to the substrate extending outwards from the holiday for a distance of at least 40 mm. Make these incisions at an angle of approximately 30° from each other.
- Insert the knife point into the centre portion of the holiday down to the metal substrate. Using a gentle levering action, slowly peel away a radial section of coating, continuing until firm adhesion is encountered. As loss of adhesion is not always obvious, carefully examine the substrate for signs of residual coatings, which indicates that disbonding has not occurred.
- Repeat with each radial segment.

NOTE Different methodologies for removing the polyethylene can be agreed.

B.6 Test results

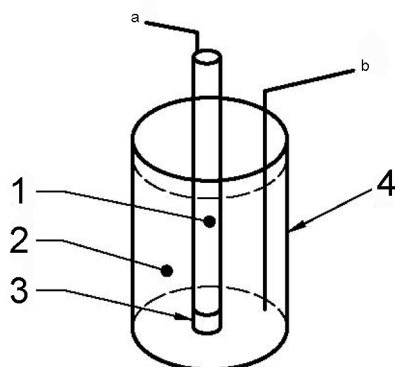
The result of the length of the cathodic disbondment is defined as the arithmetic mean value of the 12 single values. The mean value shall be reported.



Key

- | | |
|---|--|
| 1 working electrode | 9 steel test piece |
| 2 electrode (anode) | 10 sealing material |
| 3 electrode (reference) | 11 artificial defect |
| 4 reference electrode | 12 sealing material |
| 5 plastic cover | 13 electrode (cathode) |
| 6 plastic pipe (minimum internal \varnothing 50 mm) | 14 platinum electrode \varnothing 0,8 mm to 1,0 mm (anode) |
| 7 electrolyte \geq 150 ml | 15 potentiostat |
| 8 coating | |

Figure B.1 — Electrolytic cell for large-diameter pipe

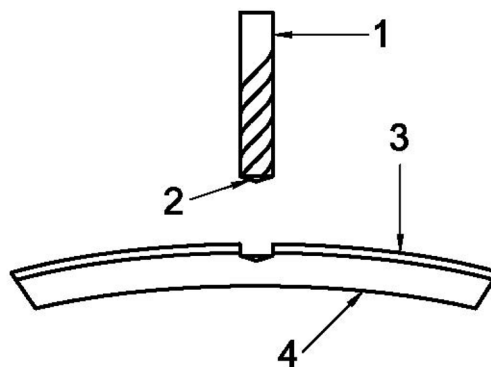


Key

- 1 holiday
- 2 electrolyte
- 3 end cap
- 4 beaker

- a to negative lead (-)
- b to positive lead (+)

Figure B.2 — Electrolytic cell for small-diameter pipe



Key

- 1 fluted and mill face mill \varnothing 6 mm
- 2 conic end
- 3 coating
- 4 steel

Figure B.3 — Drilling of an artificial defect

Annex C (normative)

Impact test

C.1 General

The test consists of verifying the strength of the coating by the impact of a punch of defined shape falling directly onto the coating from a fixed height and at a fixed temperature. The test shall be carried out on pipes or cut samples. This test shall not be carried out on pipes with a diameter of less than 50 mm.

C.2 Drop-weight equipment

The equipment shall consist of a drop weight testing machine comprised of the following:

- straight guide made of steel, aluminium or plastic, rigid and non-deformable, of inside diameter between 40 mm to 60 mm, of length at least 1,30 m and incorporating a smooth and even inside surface. The guide shall be provided with
 - 1) support and levelling devices (for example, two spirit levels for the horizontal plane and a plumb line for the vertical plane); and
 - 2) a graduated rod that makes it possible to determine the drop height to an accuracy of 5 mm; other guides may be used by agreement;
- hard steel punch, with a hemispherical head free from notches, porosity or other surface irregularities and with a diameter of 25 mm. A small metal rod of diameter 6 mm shall be fixed perpendicular to the flat face of the head and in its centre. This rod shall be long enough to hold the additional weights required for the tests. The punch shall be equipped with a suitable system for raising the weights to the required height. The mass of this assembly shall be $1 \text{ kg} \pm 0,005 \text{ kg}$;
- sufficient number of additional weights, in the form of metal discs (preferably made of stainless steel) with an outside diameter fitting the internal diameter of the straight guide and incorporating a central hole of a suitable diameter. The mass of each disc shall be measured to an accuracy of $\pm 5 \text{ g}$.

C.3 Procedure

The test shall be carried out at a temperature of $23 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$. If provisions have been made to perform this test outside this temperature range, the method described shall be adapted by agreement between the applicator and the purchaser.

The coated pipe shall be placed on a rigid and stable horizontal support and, if necessary, the pipe interior shall be supported to reduce its elastic response.

Before carrying out an impact test, the holiday-detection test shall be performed to identify the defective points and to avoid making the impact at these locations.

For each point of impact, the drop-weight testing machine shall be installed perpendicular to the coating surface so that the loaded punch can fall freely without friction or resistance.

C.3.1 Basic method

Carry out ten impacts allowing the weight corresponding to the energy specified in Tables 6 and 9 to fall from a height of 1 m. Select the points of impact to avoid any protruding welds. Furthermore, the distance from the points of impact to the end of the pipe shall be at least 1,5 times the outside diameter of the pipe and at least 50 mm apart between the axes of the impacts. Then carry out the holiday-detection test at each location.

The hard steel punch shall be checked every 30 impacts. If damaged, it shall be replaced.

C.4 Test results

If no holidays are recorded on any of the ten impacts using the basic methods, the impact results are accepted.

Annex D (normative)

Indentation test

D.1 General

The test consists of measuring the indentation of a punch into the coating under fixed conditions of temperature and load.

D.2 Indentation test equipment

The equipment shall consist of

- an inside-ventilated oven or a circulating bath thermostatically controlled to ± 3 °C;
- a penetrometer consisting of
 - a cylindrical punch with a diameter of 1,8 mm (cross-sectional area of 2,5 mm²) on the top of which is mounted a weight. The assembly, punch plus weight, shall produce a total load of 2,5 kg, and
 - a dial gauge or any other measurement system accurate to $\pm 0,01$ mm.

D.3 Procedure

Perform the test three times on one PE coating sample.

Place the test sample, held within the penetrometer assembly, into the thermostatically controlled chamber and set to the test temperature. Keep the test sample in the chamber for 1 h. Record the readings on the dial gauge.

Lower the punch assembly, with a total mass of 2,5 kg, gently onto the specimen. The test duration shall be 24 h. Record the readings of the dial gauge at the conclusion of the exposure period.

D.4 Test results

The indentation shall be the difference between the dial gauge reading before and after the 24 h test duration.

The average of the three samples shall meet the requirements of Table 6.

Annex E (normative)

Flexibility test

E.1 General

This procedure shall be used to assess the flexibility of coatings applied to a bare steel substrate.

E.2 Equipment

E.2.1 Hydraulic press.

E.2.2 Bending mandrels with fixed radii.

E.2.3 Freezer.

E.2.4 Strain gauges, if applicable.

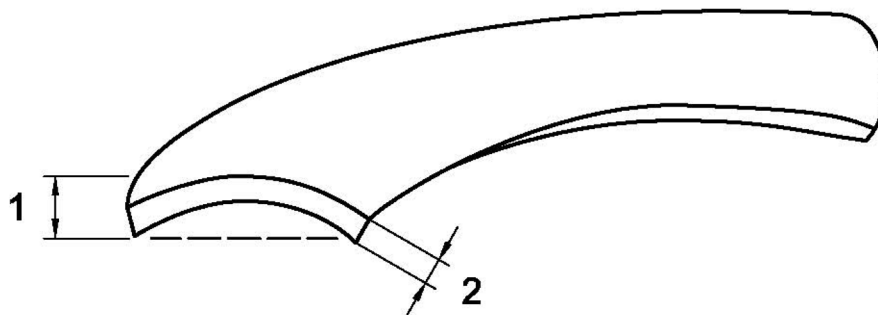
E.3 Specimen

Coated test samples shall be cold cut from a pipe ring with a minimum length of 200 mm parallel to the axis of the pipe. The width shall be approximately 25 mm.

E.4 Procedure

Determine the effective sample thickness, d , which includes the actual sample thickness and any curvature, by placing the specimen on a flat surface and measuring the effective thickness shown in Figure E.1.

Smooth the coating on the edge of the test sample to remove any potential stress risers. Place the test specimen in the freezer, cool it to the temperature of 0 °C and/or -30 °C, and hold it within that temperature range for a minimum of 1 h.



Key

- 1 effective thickness
- 2 actual thickness

Figure E.1 — Effective thickness

ISO 21809-4:2009(E)

Determine the mandrel radius, R , expressed in millimetres, that corresponds to an angle of deflection of $2,5^\circ$ per pipe diameter length by using Equation (E.1):

$$R = 22,42d \quad (\text{E.1})$$

where d is the effective sample thickness, expressed in millimetres.

Bend the test sample over a mandrel whose radius is not larger than that determined in accordance with the Equation (E.1). Bend the specimen such that the operation lasts no longer than 10 s and is completed within 30 s of the test samples having been removed from the freezer.

NOTE If the specimen exhibits peaking, the percent strain can be calculated by the use of strain gauges attached to the test sample.

E.5 Test results

Warm the bent test sample to $20\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$, and hold it in this temperature range for a minimum of 2 h. Within the next hour, visually inspect it for the presence of cracks.

Annex F (normative)

Peel strength test

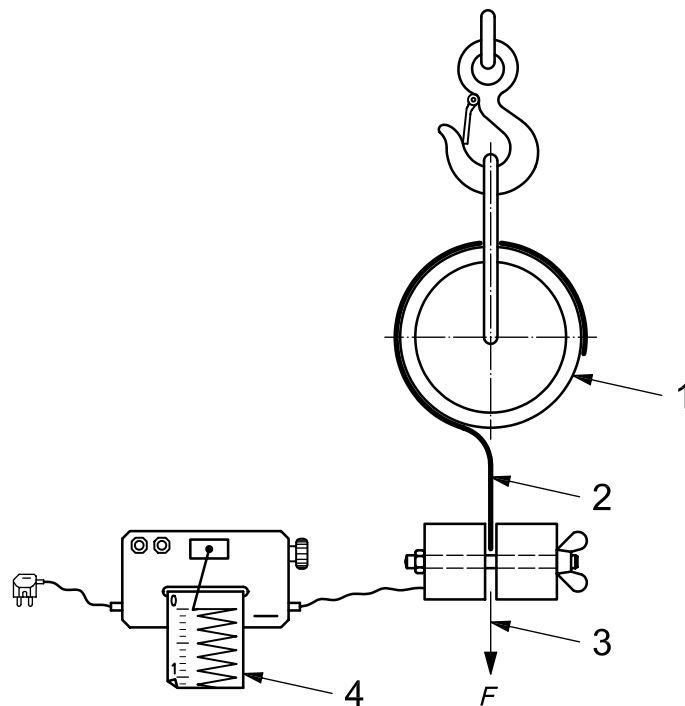
F.1 General

The method consists of measuring the force required for peeling the coating from the metal substrate of the pipe at a constant rate of pull; see Figures F.1 and F.2.

F.2 Equipment

F.2.1 Tensile testing machine, capable of recording the peel force, which operates at a rate of pull of 10 mm/min, as shown in Figures F.1 or F.2.

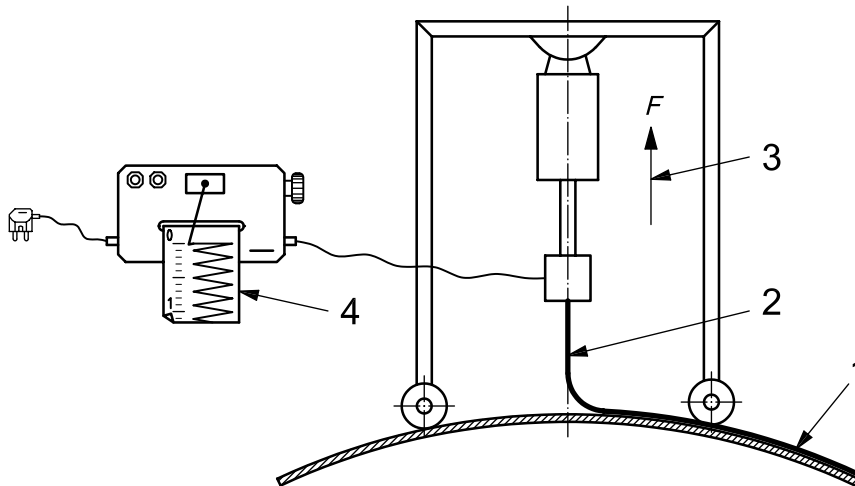
F.2.2 Cutting tool, e. g. knife.



Key

- 1 pipe sample
- 2 peeled coating
- 3 peeling force, F
- 4 measuring device

Figure F.1 — Testing equipment for small-diameter samples



- Key**
- 1 coated pipe sample
 - 2 peeled coating
 - 3 peeling force, F
 - 4 measuring device

Figure F.2 — Testing equipment for large-diameter samples

F.3 Procedure

Perform the test at temperatures in accordance with Tables 6 and 9.

Measure the temperature, by means of an adapted probe, on the external surface of the pipe at the root of the peeled strip.

Cut a sample of minimum length of 160 mm in the circumferential direction from the coated pipe. From this sample, peel a strip of coating 20 mm to 50 mm wide perpendicular to the axis of the pipe.

Arrange the pipe on its support and secure it to one of the gripping jaws of the testing machine. The system shall be such as to allow the reciprocal movement between pipe and testing machine at the prescribed pulling speed.

Record the peel force graphically over the agreed length using a constant peeling speed set to 10 mm/min.

F.4 Test results

Calculate the peel strength from the recorded data divided in five sections of 20 mm each. Calculate the average peel strength for each of these sections.

The average peel strength shall be the arithmetic mean taken over a length of 100 mm. If this value is not automatically determined, the arithmetic mean may be estimated from the 20 mm bands across the 100 mm length.

The data shall meet the following two requirements.

- The lowest arithmetic mean from each of the 20 mm sections shall meet the requirements of Tables 6 and 9.
- No single point in each of the 20 mm sections shall be lower than 30 % of the specified value of Tables 6 and 9.

Annex G (normative)

Peel strength test — Hanging mass

G.1 General

The method consists of measuring the rate of peeling the coating from the metal substrate of the pipe at a constant load; see Figure G.1.

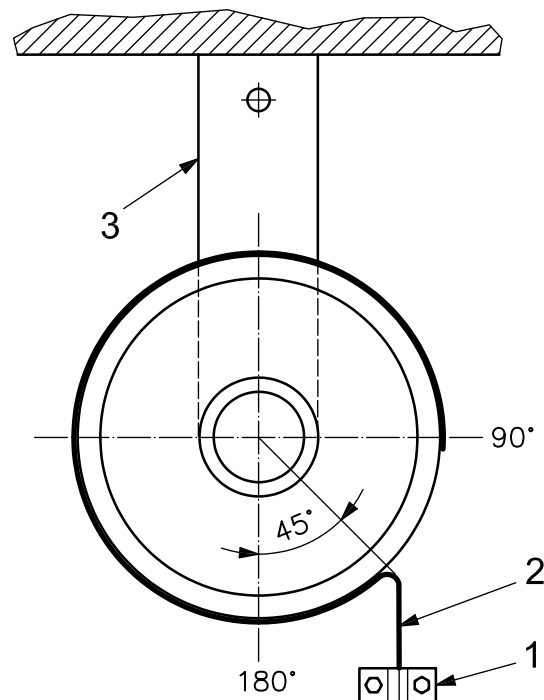
G.2 Equipment

G.2.1 Masses, weighing 0,306 kg for system A, 1,02 kg for system B or 5,1 kg for system C.

G.2.2 Cutting tool, e.g. knife.

G.2.3 Timing device.

G.2.4 Attaching device.



Key

- 1 mass
- 2 peel strip
- 3 attaching device

Figure G.1 — Hanging mass test (end view)

G.3 Test specimens

Each test specimen shall consist of a complete pipe or test ring cut from a pipe. The test ring shall be approximately 150 mm long.

G.4 Test parameters

The test temperature shall be (20 ± 3) °C.

The peel strip shall be (25 ± 1) mm wide;

The peel orientation shall be circumferential

G.5 Procedure

- 1) Condition the sample at (20 ± 3) °C for at least 1 h before starting the test.
- 2) Make two cuts (25 ± 1) mm apart through the coating to the steel substrate with the cutting tool from the 90° position to the 180° position. Cut the coating at a right angle at the 90° position and peel it off down to the 135° position as shown in Figure G.1.
- 3) Attach the appropriate mass to the free end of the strip as shown in Figure G.1.
- 4) Measure the peeling time, t , expressed in minutes, from the 135° position to the 180° position. Alternatively, terminate peeling when the test time calculated in accordance with Equation (G.1) has been exceeded:

$$t = \frac{(0,125 \times 3,14 \times D)}{10} \quad (\text{G.1})$$

where D is the specified outside diameter, expressed in millimetres.

- 5) Measure the distance peeled, expressed in millimetres, from the time the test was initiated until it was terminated.
- 6) Calculate the peeling speed, V_p , expressed in millimetres per minute, using Equation (G.2):

$$V_p = \frac{l}{t} \quad (\text{G.2})$$

where

l is the distance peeled, expressed in millimetres;

t is the peeling time, expressed in minutes.

If the peeling speed exceeds 10 mm/min, the test shall be considered to have failed.

G.6 Test results

If a report is required, the following information shall be reported:

- a) date of testing;
- b) specified pipe outside diameter;
- c) mass, expressed in kilograms;
- d) distance peeled, expressed in millimetres;
- e) peeling time, expressed in minutes;
- f) peeling speed, expressed in millimetres per minute.

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