
**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)**

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

*Partie 1: Revêtements à base de polyoléfinés (PE tricouche et
PP tricouche)*



Reference number
ISO 21809-1:2011(E)

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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 21809-1 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*

A Part 6 dealing with multilayer fusion-bonded epoxy coatings (FBE), a Part 7 dealing with liquid coatings, a Part 8 dealing with thermal insulation coatings, and a Part 9 dealing with epoxy polyamide powder coatings (2-layer) are under preparation.

Introduction

It is necessary that users of this part of ISO 21809 be aware that further or differing requirements can be required 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 1: Polyolefin coatings (3-layer PE and 3-layer PP)

1 Scope

This part of ISO 21809 specifies requirements of plant-applied external three-layer polyethylene- and polypropylene-based coatings for corrosion protection of welded and seamless steel pipes for pipeline transportation systems in the petroleum and natural gas industries in accordance with ISO 13623.

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

2 Conformance

2.1 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 80000-1.

NOTE For the purpose of this provision, the rounding method of ASTM E29 is equivalent to ISO 80000-1.

2.2 Compliance with standard

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 the applicable requirements of this part of ISO 21809. The purchaser shall be allowed to make any investigations necessary to ensure compliance by the applicator and to reject any material and/or coating that does not comply.

3 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 179-1, *Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test*

ISO 179-2, *Plastics — Determination of Charpy impact properties — Part 2: Instrumented impact test*

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

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

ISO 1873-2, *Plastics — Polypropylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 2811 (all parts), *Paint and varnishes — Determination of density*

ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile matter content*

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

ISO 8130-2, *Coating powders — Part 2: Determination of density by gas comparison pycnometer (referee method)*

ISO 8130-3, *Coating powders — Part 3: Determination of density by liquid displacement pycnometer*

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, *Preparation of steel substrates before application of paints and related products — Test 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 — Test for the assessment of surface cleanliness — Part 6: Extraction of soluble contaminant 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, *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, *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 10474:1991, *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 11126 (all parts), *Preparation of steel substrates before application of paints and related products — Specifications for non-metallic blast-cleaning abrasives*

ISO 11127-6, *Preparation of steel substrates before application of paints and related products — Test methods for non-metallic blast cleaning abrasives — Part 6: Determination of water-soluble contaminants by conductivity measurement*

ISO 11357 (all parts), *Plastics — Differential scanning calorimetry (DSC)*

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

ISO 15512, *Plastics — Determination of water content*

ISO 80000-1, *Quantities and units — Part 1: General*

AS 3894-6, *Site testing of protective coatings — Determination of residual contaminants*

ASTM D792¹⁾, *Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement*

ASTM D1505, *Standard Test Method for Density of Plastics by the Density-Gradient Technique*

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

ASTM D4138, *Standard Practice for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive, Cross-Sectioning Means*

ASTM D4940, *Standard Test Method for Conductimetric Analysis of Water Soluble Ionic Contamination of Blasting Abrasives*

EN 10204:2004²⁾, *Metallic materials — Types of inspection documents*

SSPC-AB 1, *Mineral and Slag Abrasives*

SSPC-AB 2, *Cleanliness of Recycled Ferrous Metallic Abrasives*

SSPC-AB 3, *Ferrous Metallic Abrasive*

SSPC-SP 1³⁾, *Solvent Cleaning*

4 Terms and definitions

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

4.1

adhesion

bond between coating and substrate after environmental testing

1) American Society for Testing and Materials, 100 Harbour Drive, West Conshohocken, PA 19428-2959, USA.

2) CEN, European Committee for Standardization, Central Secretariat, Rue de Stassart 36, B-1050, Brussels, Belgium.

3) Society for Protective Coatings, 40 24th Street, 6th Floor, Pittsburg, PA 15222-4656, USA.

4.2

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 the applicator is regarded as the manufacturer (see 4.13).

4.3

application procedure specification

APS

document describing procedures, methods, equipment and tools used for coating application

4.4

batch

quantity of material produced in a continuous manufacturing operation using raw materials of the same source and grade

4.5

batch certificate

certificate of analysis issued by the manufacturer

4.6

certificate of compliance

document issued in accordance with ISO 10474 or EN 10204, stating compliance with the purchase order for coated pipes, but without mention of any test results, issued in accordance with the purchasing requirements

4.7

cutback

length of pipe left uncoated at each end for joining purposes

4.8

design temperature range

temperature range including maximum and minimum temperatures likely to be reached during transport, handling, installation and operation

NOTE The design temperature range of the coating may be narrower than that specified for the steel pipe material and/or the pipeline system.

4.9

end user

company (companies) that own(s) and/or operate(s)

4.10

holiday

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

4.11

inspection certificate 3.1.B

inspection certificate 3.1

document in accordance with ISO 10474 or EN 10204 giving the results of the testing of coated pipes, supplied and signed by a representative of the applicator authorized to issue such documents

4.12

inspection and testing plan

ITP

document providing an overview of the sequence of inspections and tests, including appropriate resources and procedures

4.13**manufacturer**

company responsible for the manufacture of coating material(s)

4.14**manufacturer's specification**

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

4.15**operating temperature**

maximum and/or minimum temperature that can be endured by a pipeline (component) and/or pipeline system during operation, and that shall be within the design temperature range

4.16**peel strength**

force required for peeling the coating from the substrate

4.17**pipe diameter length**

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

4.18**pipeline**

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

NOTE Adapted from ISO 13623:2009, definition 3.14.

4.19**pipeline 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

NOTE Adapted from ISO 13623:2009, definition 3.16.

4.20**procedure qualification trial****PQT**

application of a coating and subsequent inspection/testing of its properties, to confirm that the APS is adequate to produce a coating with the specified properties, carried out prior to the start of production

4.21**purchaser**

company responsible for providing the purchase order requirements

4.22**test report**

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

4.23**total coating thickness**

sum of all three layers

5 Symbols and abbreviated terms

5.1 Symbols

C	percentage conversion of FBE coating
d	effective sample thickness, expressed in millimetres
D	outside diameter of the pipe, expressed in millimetres
ΔH	exothermic heat of reaction, expressed in Joules per gram
M	mass, expressed in kilograms or grams
P_m	mass of pipe per metre length, expressed in kilograms per metre
dQ/dt	differential heat flow, expressed in watts per square metre
r	mandrel radius, expressed in millimetres
R_{MF}	melt flow rate, expressed in grams per 10 minutes
T_g	glass transition temperature, expressed in degrees Celsius
ΔT_g	difference in the glass transition temperature between two successive thermal analysis scans, expressed in degrees Celsius
w_m	mass fraction of moisture, expressed as a percentage
ρ_p	density of the epoxy powder, expressed in grams per litre

5.2 Abbreviations

APS	application procedure specification
DSC	differential scanning calorimetry
ESCR	environmental stress cracking resistance
FBE	fusion-bonded epoxy
HDPE	high-density polyethylene
IR	infrared
ITP	inspection and testing plan
LDPE	low-density polyethylene
MDPE	medium-density polyethylene
MFR	melt flow rate
NPS	nominal pipe size
PDL	pipe diameter length
PE	polyethylene
PP	polypropylene
PPT	preproduction trial
PQT	procedure qualification trial

SAW	submerged arc welding
UV	ultraviolet
3LPE	three-layer polyethylene coating
3LPO	three-layer polyolefin coating
3LPP	three-layer polypropylene coating

6 Information supplied by the purchaser

6.1 General information

The purchase order shall include the following information:

- number of this part of ISO 21809 and year of publication (ISO 21809-1:2011);
- pipe quantity, outside diameter, minimum wall thickness, minimum, maximum and nominal length, grade of steel;
- bare pipe standard or specification designation, e.g. ISO 3183;
- design temperature range in accordance with 7.2;
- coating class and coating thickness class in accordance with 7.2 and 7.3;
- cutback configuration and finish (length, angle, visible epoxy, temporary protection, etc.);
- type of certificate of compliance.

6.2 Additional information

The purchase order shall specify which of the following provisions apply for the specific item ordered:

- pipe tracking and traceability of pipes to coating materials;
- permissible number of coating repairs, if different from the one defined in Clause 12;
- marking of pipes;
- handling procedures;
- storage procedures;
- documentation and schedule for supply of documentation;
- purchaser approval of APS;
- inspection and testing plan and/or daily log;
- inspection of incoming pipes;
- pipe end protection;
- minimum thickness of epoxy layer and/or total coating thickness required, if exceeding those in Table 2 and Table 9, respectively;

- surface profile of PE/PP coating (e.g. prior to concrete weight coating or special laying methods);
- surface pretreatment;
- special requirements relative to supply of coating materials (e.g. FBE or liquid, manufacturer-specific products and certification);
- methods, frequency and acceptance criteria for inspection and testing differing from this part of ISO 21809;
- procedure qualification trial (PQT);
- protection against adverse ambient conditions during storage.

7 Coating classification

7.1 General

Coating class shall be selected based on the design temperature range and expected field duty.

Coating thickness class shall be selected based on transport, handling, laying conditions and the expected operating and environmental conditions.

7.2 Coating classes

The coating shall be capable of withstanding the temperature range required, as shown in Table 1. The coating class shall be specified in the purchase order.

Table 1 — Coating classes and design temperature ranges

Coating class	A	B	C ^a
Top layer material	LDPE	MDPE/HDPE	PP
Design temperature ranges (°C)	–20 to +60	–40 to +80	–20 to +110
^a Installation and transportation at temperatures below 0 °C can cause mechanical damage.			

Use of coating classes outside these guidelines shall be approved by the purchaser or end user.

7.3 Coating thickness classes

Coating thickness class shall be selected by the purchaser or end user based on installation and service conditions and pipe dimensions. The coating thickness class, as shown in Table 2 as a function of coating class and pipe weight, shall be specified in the purchase order.

Table 2 — Minimum total coating thickness

P_m kg/m	Total coating thickness ^a mm								
	Class A1 ^b	Class A2 ^c	Class A3 ^d	Class B1 ^b	Class B2 ^c	Class B3 ^d	Class C1 ^b	Class C2 ^c	Class C3 ^d
$P_m \leq 15$	1,8	2,1	2,6	1,3	1,8	2,3	1,3	1,7	2,1
$15 < P_m \leq 50$	2,0	2,4	3,0	1,5	2,1	2,7	1,5	1,9	2,4
$50 < P_m \leq 130$	2,4	2,8	3,5	1,8	2,5	3,1	1,8	2,3	2,8
$130 < P_m \leq 300$	2,6	3,2	3,9	2,2	2,8	3,5	2,2	2,5	3,2
$300 < P_m$	3,2	3,8	4,7	2,5	3,3	4,2	2,5	3,0	3,8
^a The required total coating thickness may be reduced by a maximum of 10 % on the weld seam for SAW-welded pipes. ^b Class 1 is for light duty (onshore sandy soil). ^c Class 2 is for moderate duty (clay soils, absence of backfill). ^d Class 3 is for heavy duty (rocky soil or offshore).									

Use of thickness classes outside of these guidelines shall be approved by the purchaser or end user.

8 Coating materials

8.1 Composition of the coating system

The coating system shall consist of three layers:

- 1st layer: epoxy;
- 2nd layer: adhesive;
- 3rd layer: PE/PP top layer.

8.2 Qualification of the coating materials

8.2.1 General requirements

The manufacturer shall qualify each type of coating material in compliance with the requirements of this part of ISO 21809. The qualification shall be repeated in case of changes in the material composition, changes in the production process which influence the material processing behaviour and change in production facility.

The manufacturer shall carry out and report the material qualification in accordance with the requirements of Clause 8 and Tables 3, 4 and 5. The test report shall contain the results of the qualification tests and the data required in Table 6.

The applicator receiving the manufacturer's test report shall verify that it meets the requirement of this part of ISO 21809.

8.2.2 Epoxy material

The applicator shall use epoxy material that is in accordance with Table 3.

Table 3 — Requirements for the epoxy material

Liquid mono-component ^a or bi-component epoxy				
Properties	Unit	Test method	Requirements	
			Class A and Class B	Class C
Density	g/l	ISO 2811 (all parts)	Within ±0,05 of the manufacturer's specified nominal value	
Solid content of base and hardener	% mass	ISO 3251	In accordance with manufacturer's specification	
Minimum glass transition temperature (<i>T</i> _{g2}) (DSC analysis ^b)	°C	ISO 11357 (all parts)	≥95 and within manufacturer's specification	At least 5 above maximum pipeline design temperature with a minimum of 95
Gel time at 205 °C ± 3 °C	s	Manufacturer's specification	Within the 20 % of the nominal values specified by the manufacturer	
Epoxy powder (FBE)				
Properties	Unit	Test method	Requirements	
			Class A and Class B	Class C
Moisture content	% mass	Annex K	≤0,6	
Minimum glass transition temperature (<i>T</i> _{g2}) (DSC analysis ^b)	°C	Annex D	≥95 and within manufacturer's specification	At least 5 above maximum pipeline design temperature with a minimum of 95
Gel time at 205 °C ± 3 °C	s	Annex J	Within 20 % of the nominal value specified by the manufacturer	
Density	g/l	Annex N	Within ±0,05 of the manufacturer's specified nominal value	
^a Liquid mono-component is limited to Class A only.				
^b Curing condition (procedure as per manufacturer's specification).				

8.2.3 Adhesive material

The applicator shall use adhesive material that is in accordance with Table 4.

Table 4 — Requirements for the adhesive material (copolymeric or grafted adhesive in pellet or powder form)

Properties	Unit	Test method	Requirements		
			Class A	Class B	Class C
Elongation at break at 23 °C $\pm$ 2 °C ^a	%	ISO 527-2 or ISO 527-3	≥ 600	≥ 600	≥ 400
Tensile yield strength at 23 °C $\pm$ 2 °C ^a	MPa	ISO 527-2 or ISO 527-3	≥ 5	≥ 8	≥ 12
Notched impact strength at minimum class temperature	kJ/m ²	ISO 179-1 or ISO 179-2	—	—	≥ 3
Vicat softening temperature A/50 (9,8 N)	°C	ISO 306	≥ 60	≥ 85	≥ 110
Water content	%	ISO 15512	$\leq 0,1$	$\leq 0,1$	$\leq 0,1$
^a 2 mm-thick compression-moulded sheet, test specimen type according to ISO 527-3, strained at 50 mm/min (for PP-based adhesives, the specimen can also be prepared by injection moulding according to ISO 1873-2).					

8.2.4 PE/PP top layer material

The applicator shall use PE/PP material that is in accordance with Table 5.

Table 5 — Minimum requirements for PE/PP top layer

Properties	Unit	Test method	Requirements		
			Class A	Class B	Class C
Density of the base resin	g/cm ³	ISO 1183 (all parts) or ASTM D792 or ASTM D1505	≥0,920	≥0,930	≥0,890
Elongation at break at 23 °C ± 2 °C ^a	%	ISO 527-2 or ISO 527-3	≥600	≥600	≥400
Tensile yield strength at 23 °C ± 2 °C ^a	MPa	ISO 527-2 or ISO 527-3	≥10	≥15	≥20
Charpy impact strength, notched at minimum class temperature	kJ/m ²	ISO 179-1	—	—	≥3
Vicat softening temperature A/50 (9,8 N)	°C	ISO 306	≥95	≥110	≥130
Water content	%	ISO 15512	≤0,05	≤0,05	≤0,05
Hardness Shore D	—	ISO 868	≥50	≥55	≥60
ESCR (50 °C, F50, Cond. A: 10 % Igepal CO630, or Cond. B: 100 % Igepal CO630) ^{b,c}	H	ASTM D1693	≥300 Cond. A	≥1 000 Cond. A or ≥300 Cond. B if density >0,955	N.A.
Oxidation induction time (intercept in the tangent method)	min	ISO 11357 (all parts)	≥30 at 210 °C	≥30 at 210 °C or ≥10 at 220 °C	≥30 at 220 °C
UV resistance and thermal ageing ^c	%	Annex G	ΔMFR ≤ 35	ΔMFR ≤ 35	ΔMFR ≤ 35

^a Preparation of the test specimen in accordance with ISO 1872-2 for PE and ISO 1873-2 for PP (2 mm thickness), strained at 50 mm/min.

^b In order to validate the ESCR values, data shall be provided by manufacturers to define the limit in density (MFR) to pass that requirement. This limit shall not be wider than what is commonly known as fit-for-purpose PE grades for pipe coating.

^c Choice of test condition should be with approval of end user.

If the compounding is done during the application process, the applicator shall perform the qualification tests for the product in accordance with Table 5. The batch certificate, produced in accordance with Table 6, shall be issued.

NOTE The applicator sometimes performs compounding with additives against UV and thermal ageing or other purposes.

8.3 Batch certificate

8.3.1 The applicator shall provide batch certificates supplied by the manufacturer of each material and shall contain the information given in Table 6. The batch certificate shall state test methods and acceptance criteria.

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

Table 6 — Batch certificate

Content	Coating material			
	Liquid epoxy	FBE	Adhesive	PE/PP top coat
Identification of the manufacturer	x	x	x	x
Product identification	x	x	x	x
Batch identification	x	x	x	x
Date and place of manufacturing	x	x	x	x
Density	x	x	x	x
Viscosity of base and hardener	x			
Solid content of base and hardener	x			
Melt flow rate			x	x
Gel time	x	x		
Particle size		x		
Moisture/water content		x	x	x
Carbon black content				Only PE ^a
Pigment dispersion				x ^a
Thermal characteristic	x	x		
Reactive site content			x ^b	
^a If applicable.				
^b Direct and indirect methods can be proposed by the manufacturer.				

8.3.2 The manufacturer's specification for epoxy materials shall include the following:

- trade name;
- generic type;
- mix ratio (if any);
- maximum thickness, expressed in millimetres or micrometres (see Annex A);
- maximum and minimum storage temperatures, expressed in degrees Celsius;
- shelf-life at storage temperature, expressed in months;
- technical properties, in accordance with Table 3.

8.3.3 The manufacturer's specification for adhesive and PE/PP materials shall include the following:

- trade name;
- description of the adhesive;
- colour;
- maximum and minimum storage temperatures, expressed in degrees Celsius;
- shelf-life at storage temperature, expressed in months;
- technical properties, in accordance with Tables 4 and 5.

8.4 Storage and handling of coating materials

Storage and handling of coating materials shall be in accordance with the manufacturer's specification.

9 Coating system qualification

9.1 General

Each coating system shall be qualified by the applicator. Qualification shall be carried out separately for each coating line. The applicator shall carry out and report the coatings qualification in accordance with the requirements of this part of ISO 21809. The test report shall contain the results of the qualification tests and data required in Tables 6, 7 and 8. The applicator shall apply coating materials qualified in accordance with the requirements of 8.2.

Qualification shall be repeated in case of modifications to the coating line, coating materials or application procedures.

9.2 Application procedure specification

Prior to the start of coating production and any specified PQT, the applicator shall prepare an APS, including:

- incoming inspection of pipes and pipe tracking;
- data sheets for coating materials, including any materials used for coating repairs;
- data sheets for abrasive blasting materials;
- certification, receipt, handling and storage of materials for coating and abrasive blasting;
- cleaning procedure for all application equipment;
- preparation of the steel surface including monitoring of environmental parameters, methods and tools for inspection, grinding of pipe surface defects and testing of surface preparation;
- coating application, including tools/equipment for control of process parameters essential for the quality of the coating;
- lay-out sketch or flow diagram for the coating plant;
- methods and tools/equipment for inspection and testing of the applied coating;
- repairs of coating defects and any associated inspection and testing;
- stripping of defective coating;
- preparation of coating cutback areas;
- marking and traceability;
- handling and storage of pipes;
- any special conditions for dispatch of coated pipes, including protection of pipe ends;
- documentation.

The APS shall cover all items associated with quality control as defined in this part of ISO 21809 and any agreed amendments. It shall be available to the purchaser on request at any time during production.

If specified, the APS, including any revisions, shall be approved by the purchaser prior to the start of production and any specified PQT.

Table 7 — Properties of the applied coating

Properties		Unit	Test method	Class A	Class B	Class C
Continuity		—	Annex B	Free of defects and discontinuities, delaminations, separations and holidays		
Impact strength at 23 °C ± 3 °C ^a		J/mm	Annex E	>5	>7	>10
Indentation	at 23 °C ± 3 °C	mm	Annex F	≤0,3	≤0,2	≤0,1
	at maximum design temperature			≤0,4	≤0,4	≤0,4
Elongation at break of PE/PP at 23 °C ± 3 °C ^b		%	ISO 527-3	≥400	≥400	≥400
Peel strength ^{b,c,d,h}		N/mm	Annex C C.5 (hanging mass)	≥10 at ≥23 °C ≥2 at ≥60 °C	≥15 at ≥23 °C ≥3 at ≥80 °C	≥25 at ≥23 °C ≥4 at ≥90 °C or at maximum operating temperature if above 90 °C
Degree of cure of the epoxy (1st layer) ^e ΔT_g		°C	Annex D	According to the manufacturer's specification and ≤5 °C		
Product stability during application of the PE/PP top layer process		%	ISO 1133	≤20 ΔMFR for Classes A and B; ≤35 for Class C (virgin compounded granulate before application/coating after application of the same batch)		
Average radius of cathodic disbondment ^f	at 23 °C/28 d; –1,5 V	mm	Annex H	≤7		
	at 65 °C/24 h; –3,5 V			≤7		
	Maximum operation ^g temperature/28; –1,5 V			≤15		
Flexibility		—	Annex I	No cracking at an angle of 2,0° per pipe diameter length		
Hot water immersion test		mm	Annex M	Average ≤2 and maximum ≤3		

^a Lower test temperature may be agreed upon using the same acceptance criteria.

^b No single recorded peeling value shall be lower than 30 % of the requirements.

^c Cohesive failure mode in the adhesive if a non-grafted adhesive is used, no disbonding between steel and epoxy.

^d In case of break at 23 °C, the maximum peel force shall be recorded. The minimum requirements at 23 °C shall be fulfilled and the test shall then be carried out only at 90 °C (or at maximum operating temperature if higher than 90 °C).

^e For FBE with a glass transition temperature above 120 °C, testing shall be according to the manufacturer's recommendation.

^f In production, test only at 65 °C/24h/–3,5 V condition is required.

^g Maximum test temperature shall not be greater than 90 °C.

^h Hanging weight method may be used if agreed with end user.

9.3 Procedure qualification trial

If specified, the APS shall be verified by a PQT in accordance with Annex L.

9.4 Inspection and testing plan

If specified, the applicator shall prepare an ITP and a daily log to record quality control data in accordance with Annex L.

10 Application of the coating system

10.1 Surface preparation

10.1.1 Initial preparation

All dirt, deleterious matter and contaminants, such as oil and grease, shall be removed from the pipe prior to coating. If necessary, the 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 in accordance with the APS. 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.

10.1.2 Abrasive blast cleaning

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

NOTE If the pipe being coated is of a high-strength grade, e.g. X80, X100 or X120, harder abrasives can be required 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 SSPC-AB 1, SSPC-AB 2 and SSPC-AB 3 or ASTM D4940 so as not to contaminate the substrate.

The cleanliness achieved at entry to the application line shall be in accordance with the requirements of ISO 8501-1:2007, grade Sa 2 1/2 ("near-white metal").

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 (Stylus method) or ISO 8503-5 (Replica tape method). The Stylus method shall be used for verification. The Replica tape method may be used when a correlation is established with the Stylus (Profilometer) method with a cut-off length of 2,5 mm.

If grinding is required after blast cleaning, the maximum allowable area of grinding shall be 10 cm² per metre of pipe length or 0,5 % of the pipe surface area, whichever is lower. If the grinding area required exceeds these limits, the pipe shall be reblasted.

10.1.3 Surface dust contamination

The dust level shall be measured in accordance with the requirements of ISO 8502-3. The maximum allowable level shall be Class 2 (for both size and quantity).

10.1.4 Surface cleanliness and pre-treatment

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

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

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

If 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.

10.2 Coating application

10.2.1 General

The coating shall be applied in accordance with the APS. During the application of the coating components, the preheating temperature of the pipe shall be monitored and recorded using optical pyrometers or contact thermometers. Temperature-measuring crayons may be used to measure temperature only if agreed upon prior to coating, and shall be validated for temperature control during qualification and production of the coating system.

Throughout production, the coating temperature shall be continuously monitored and recorded once every half hour unless otherwise agreed with the end user/purchaser.

10.2.2 Epoxy application

Following surface preparation, the surface being coated shall not be exposed for a length of time that can result in flash rust and under no circumstance shall the time be more than 4 h.

The pipe temperature prior to and during epoxy application shall be in accordance with the APS.

The thickness of the epoxy layer after curing shall be in accordance with Table 9. The degree of cure shall be in accordance with Table 7.

10.2.3 Adhesive application

The elapsed time between epoxy application and adhesive application shall be in accordance with the APS.

10.2.4 PE/PP application

The application of the PE/PP layer shall be in accordance with the APS.

The applied coating shall be cooled to a temperature that prevents handling damage during finishing and final inspection.

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

10.3 Cutback

The coating at the pipe ends shall be removed to expose a length of bare metal as specified in the purchase order.

The polyolefin shall be bevelled to an angle not exceeding 30° measured in the direction of the pipe axis.

The cutback length shall be measured from the root face of the pipe to the beginning of the coating bevel.

11 Inspection and testing

11.1 General

Inspection and testing shall be carried out in accordance with the APS, and ITP if applicable, and meet the requirements of Tables 8 and 9.

Table 8 — Requirements for inspection of surface preparation

Properties	Unit	Test method	Requirements	Frequency qualification	Frequency production
Surface condition before blasting	—	Visual inspection	Free of contaminations	Each pipe	Each pipe
Environmental conditions	—	Calculation and direct measurement	As determined at time of measurement	Once	Every 4 h
Pipe temperature before blasting	°C	Thermocouple	Minimum 3 above the dew point	Once	Every 4 h
Size, shape and properties of virgin abrasive	—	Visual + certification ISO 11124 (all parts) (metallic) ISO 11126 (all parts) (non-metallic)	Conformity to certificate and compliance with manufacturing/working procedures	Once	Every batch
Water-soluble contamination of abrasives	mS/cm	ASTM D4940	Conductivity max. 60	Once	1/shift
Soluble salt after blasting	—	Potassium ferricyanide indicator in accordance with AS 3894-6	—	Each pipe	Every 4 h
Soluble salt after blasting	mg/m ²	Conductive measurement, ISO 8502-9 and ISO 11127-6	Salt content (as NaCl) max. 20	Each pipe	Every 4 h, if salt presence is indicated
Surface roughness of blasted surface (R_z/R_{y5})	µm	ISO 8503-4 or ISO 8503-5	50 to 100	5 pipes	Every 1 h
Visual inspection of blasted surface	—	ISO 8501-1	Grade Sa 2 1/2	Each pipe	Each pipe
Presence of dust after dust removal	—	ISO 8502-3	Max. Class 2	5 pipes	Every 1 h
Visual inspection of pipe prior to introduction to coating line	—	Visual	No rust	Each pipe	Each pipe
Temperature of extruded adhesive and polyolefin	°C	Thermometer	Compliance to APS	Once	Every 1 h
Preheating temperature before coating	°C	Pyrometer	Compliance to APS	Each pipe	Every 0,5 h

Table 9 — Requirements for inspection and testing of applied coating

Properties	Unit	Test method	Requirements	Frequency qualification	Frequency production
Minimum epoxy thickness ^a	µm	ISO 2808 or ASTM D4138	Liquid epoxy: 25 FBE: 125	1st pipe	1/shift
Minimum adhesive thickness ^a	µm	ISO 2808 or ASTM D4138	150 on pipe body	1st pipe	1/shift
Degree of cure	°C	Annex D	Table 7	1st pipe	1st pipe and 1/shift
Continuity	—	Annex B	Table 7	Each pipe	Each pipe
Total thickness of coating	mm	Annex A	Table 2	5 pipes	Every 10 pipes
Impact resistance	J/mm	Annex E	Table 7	3 pipes	Once per PE/PP batch
Peel strength ^b	N/mm	Annex C	Table 7	5 pipes	Every 4 h
Indentation	mm	Annex F	Table 7	Once	Each PE/PP batch
Elongation at break	%	ISO 527-2 or ISO 527-3	Table 7	Once	Each PE/PP batch
Cathodic disbondment	mm	Annex H	65 °C/24H/–3.5 V	Once	1/day
Hot water immersion test	mm	Annex M	Table 7	Once	1/day
Flexibility	Degrees per pipe length diameter	Annex I	Table 7	Once	—
In process degradation of PE/PP)	%	ISO 1133	ΔMFR: max. 35 for PP; max. 20 for PE between raw and applied material	Once	1st pipe per shift
Cutback	mm	Measuring	subclause 10.3	Each pipe	Recorded once per hour
Coating repairs	—	Visual/Annex B	No holidays	Once for validation	Each defect
^a For FBE and adhesive thickness, it is measured on part-coated pipes over the length of pipe partially coated at start-up. ^b Each pipe shall be tested at 23 °C at both ends with an additional test at high temperature.					

11.2 Retesting

Pipes that fail to meet the requirements of Table 8 shall not be coated until the cause has been identified and corrected. Pipes that have been coated since the last acceptable test shall be accepted if they meet the requirements of Table 9.

Pipes that fail to meet the requirements of Table 9 shall be retested for the parameter(s) found to be out of specification. If the retest also fails to meet the requirements in Table 9, two pipes in the coating sequence prior to the pipe that failed and two pipes in the sequence after that pipe shall be retested.

If the results of all retested pipes are satisfactory, the coating shall be considered acceptable on all pipes except the pipe that failed. If any of the retests also fail, the total production back to the last test passed shall be blocked and further testing shall be initiated by the applicator and agreed with the purchaser. In the case of confirmed failure, the coating shall be rejected.

12 Coating repairs

Defects in the finished coating caused by the application process, transportation, handling and storage in the coating plant or in the storage area, as well as those that have been subjected to destructive testing, shall be repaired.

The defect area shall not exceed 10 cm² and the total number of defects being repaired shall not exceed 1 defect per metre length of pipe, unless otherwise specified. If the size or number of defects exceeds these limits, the affected pipe shall be stripped and recoated in accordance with the APS.

Defects shall be repaired and inspected using materials and procedures in accordance with the APS and any PQT. Repair materials shall be compatible with the applied coating.

If it is required to strip a pipe, it shall be carried out in accordance with the APS.

13 Marking

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 code;
- marking required by the applicable pipe specification or standard;
- ISO International Standard designation and year of publication (ISO 21809-1:2011);
- coating thickness class;
- specified total coating thickness from 7.3;
- maximum design temperature (for Class C only).

EXAMPLE Applicator - ISO 21809-1 2011 Class B2 -2,5 mm.

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

14 Handling and storage in the applicators facilities

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 (see Table 9) shall be repaired in accordance with the requirements of Clause 12.

14.2 Storage

If specified in the purchase order, the applicator shall specify in the APS details of the facilities and the methods being used for yard storage.

15 Test reports and certificate of compliance

Unless otherwise specified in the purchase order, an Inspection Certificate of type 3.1.B in accordance with ISO 10474:1991 (or type 3.1 in accordance with EN 10204:2004) shall be issued by the applicator, which provides the results from the inspection and testing of the coating materials and coated pipes in accordance with the requirements of this part of ISO 21809 and any other requirements specified in the purchase order. If, however, in the purchase order, the purchaser does waive the requirement for an inspection certificate, then the applicator should provide a certificate of compliance.

Annex A (normative)

Inspection of thickness

A.1 General

The test shall consist of measuring the thickness of the applied coating by means of a non-destructive process.

A.2 Equipment

A.2.1 Thickness-measuring instrument, with ± 10 % reading accuracy.

The instrument shall be calibrated for the range of coating thicknesses being measured.

A.3 Procedure

A.3.1 At the start of each shift, the instrument readings shall be checked using calibrated plates and shims that are ± 20 % of the thickness being measured. Calibration shall be carried out at the same temperature as the samples being measured. The surface roughness and cleanliness of the plates shall be representative of the production pipe.

NOTE When measuring thicknesses of more than 1 mm, the surface roughness of the pipe is not relevant.

A.3.2 On each pipe being tested, carry out a total of 12 single readings in accordance with ISO 2808. If any of these 12 readings is below the minimum coating thickness, carry out an additional four readings around this area. The average of the additional four readings and the initial reading shall be higher than the minimum thickness.

For submerged arc welded pipes, four thickness measurements shall be taken on the weld area.

A.4 Results and test reports

The results shall consist of all individual measurements and the calculated arithmetic average of all measurements.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex B (normative)

Holiday detection test

B.1 General

This test shall consist of detecting any porosity of the coating using a scanning electrode energized by high-arc-voltage. Defects shall be detected by a spark occurring between the steel and the electrode at the defect accompanied by a sound and/or light signal.

B.2 Equipment

B.2.1 High-voltage holiday detector, adjustable, calibrated to within 10 % of the required voltage, equipped with a sound and/or light signal.

B.2.2 Scanning electrode, in the form of a metal brush, coil spring or conductive rubber. The electrode shall conform to the shape of the pipe to ensure full coverage.

B.2.3 Conductors, which are used to complete the circuit.

B.3 Procedure

B.3.1 The test shall be performed only on a coating that is free from surface moisture.

B.3.2 At the start of each shift, the instrument shall be verified by a certified voltmeter and adjusted if necessary.

B.3.3 The instrument (holiday detector) shall be connected to the pipe, completing the circuit, and switched on.

B.3.4 Set the voltage at 10 kV/mm based on the minimum total coating thickness. The voltage shall not exceed 25 kV.

B.3.5 The scanning electrode shall be passed over the surface of the coating being inspected with a continuous movement. The rate of the relative movement of the electrode shall not be limited, but it shall be demonstrated that a defect of 1 mm in diameter can be detected.

B.4 Results and test reports

The results shall consist of recording the number of holidays detected. Each holiday shall be marked for repair.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex C (normative)

Peel strength test

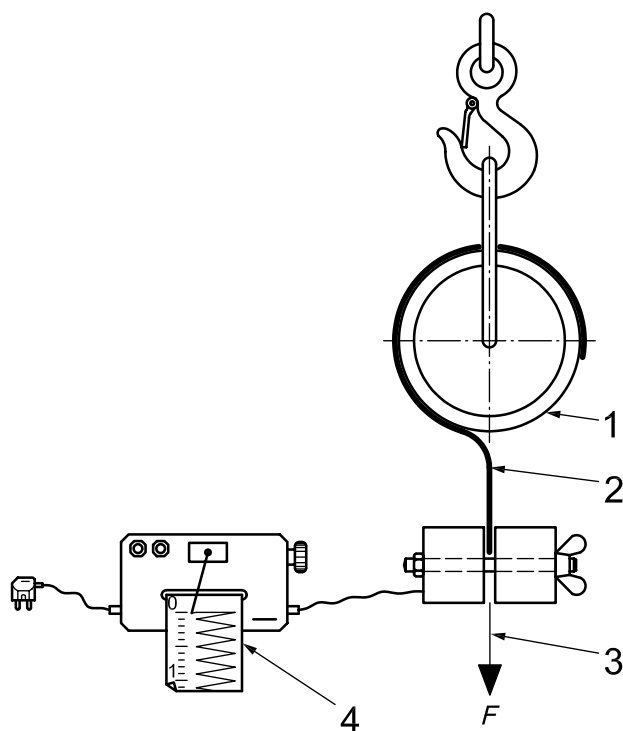
C.1 General

The test shall consist of measuring the force required for peeling the coating from the metal substrate of the pipe at a constant rate of pull.

C.2 Equipment

C.2.1 Tensile testing machine, capable of recording the peel force with a 5 % reading accuracy, that operates at a rate of pull of 10 mm/min, as shown in Figure C.1 for small diameters or in Figure C.2 for large diameters.

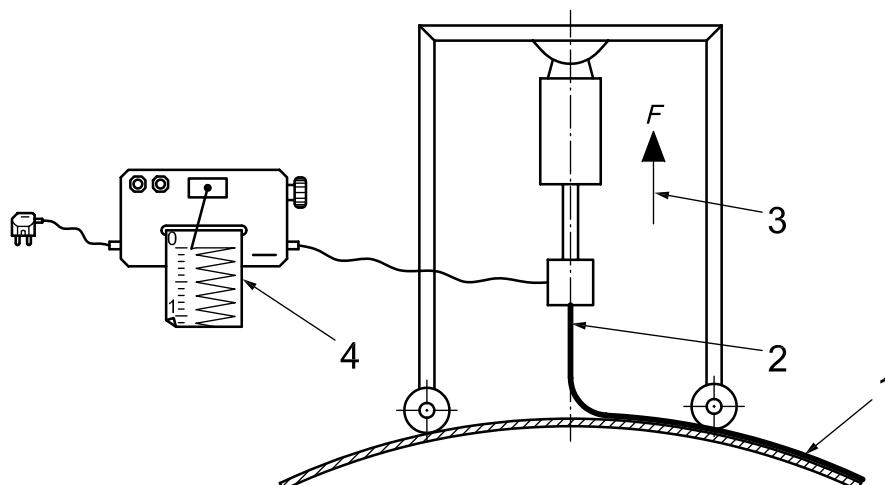
C.2.2 Cutting tool (e.g. knife).



Key

- 1 pipe ring
- 2 coating strip
- 3 peel force, F
- 4 registration unit

Figure C.1 — Peel strength test equipment for a small-diameter joint



Key

- 1 coated pipe surface
- 2 coating strip
- 3 peel force, F
- 4 registration unit

Figure C.2 — Peel strength test equipment for a large-diameter joint

C.3 Procedure

C.3.1 General

C.3.1.1 The coating thickness may be reduced to the minimum thickness of the coating class to facilitate peel testing.

C.3.1.2 The peel test shall be performed at the temperatures specified in Table 7.

C.3.1.3 The temperature shall be measured by means of an adapted probe on the external surface of the pipe at the root of the peeled strip.

C.3.1.4 The peel force shall be graphically recorded over 140 mm using a constant peeling speed of 10 mm/min.

C.3.2 Small-diameter pipes

C.3.2.1 A pipe ring of 200 mm in length shall be cut from the pipe.

C.3.2.2 A sample coating strip shall be cut in the circumferential direction of the pipe ring, measuring a minimum of 160 mm long and 20 mm to 50 mm wide.

C.3.2.3 The pipe ring shall be free to rotate about its axis as shown in Figure C.1.

C.3.2.4 The cut end of the coating strip shall be secured to one of the gripping jaws of the testing machine and peeled perpendicular to the pipe axis.

C.3.3 Large-diameter pipes

C.3.3.1 The pipe shall be supported during the test to prevent movement.

C.3.3.2 A sample coating strip shall be cut in the circumferential direction of the pipe, measuring a minimum of 160 mm long and 20 mm to 50 mm wide.

C.3.3.3 The cut end of the coating strip shall be secured to one of the gripping jaws of the testing machine and peeled perpendicular to the pipe axis.

C.3.3.4 Ring pipe or a cut sample can be used for measurement at high temperature instead of the pipe.

C.4 Results and test reports

The results shall be calculated by dividing the peel force data for 140 mm of peeling into seven intervals of 20 mm, discarding the first and last intervals. The peel strength shall be calculated from the remaining data.

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

The average peel strength shall meet the requirements of Table 7 and no single recorded peeling value shall be 30 % below the specified value.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- temperature of test;
- date of test;
- test results.

C.5 Measurement of the peel strength with a hanging mass

C.5.1 General

The method shall consist of measuring the rate at which the coating is peeled from the metal substrate of the pipe at constant load (see Figure C.3).

C.5.2 Equipment

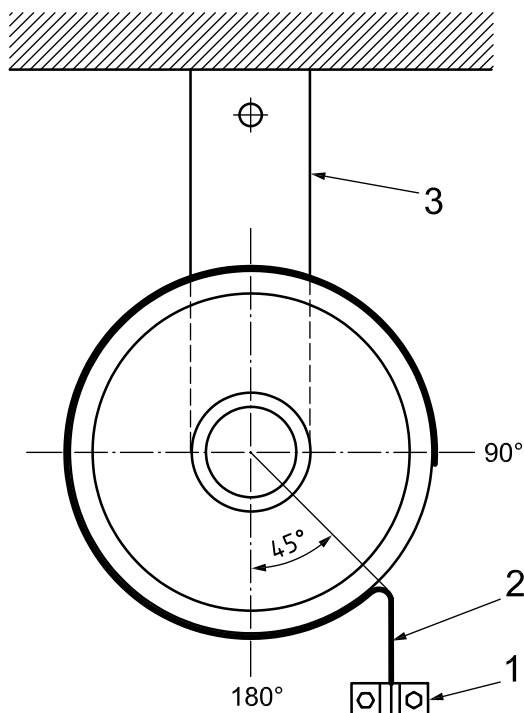
C.5.2.1 **Mass**, of 25 kg (for system A), 38 kg (for system B) or 63 kg (for system C) for $23\text{ °C} \pm 3\text{ °C}$.

C.5.2.2 **Mass**, of 5 kg (for system A), 7,5 kg (for system B) or 10 kg (for system C) for high temperature conditions.

C.5.2.3 **Cutting tool** (e.g. knife).

C.5.2.4 **Timing device**.

C.5.2.5 **Attaching device**.



Key

- 1 mass
- 2 peel strip
- 3 attaching device

Figure C.3 — Peel strength test equipment with a hanging mass

C.5.3 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.

C.5.4 Procedure

C.5.4.1 The test shall be performed at temperatures in accordance with Table 7.

C.5.4.2 The peel strip shall be 25 mm ± 1 mm wide.

C.5.4.3 The peel orientation shall be circumferential.

C.5.4.4 If a test ring is used, it shall be conditioned at the test temperature for at least 1 h before starting the test.

C.5.4.5 Two cuts shall be made 25 mm ± 1 mm apart through the coating to the steel substrate with the cutting tool, from the 90° position to the 180° position. The coating shall be cut at a right angle at the 90° position and it shall be peeled off down to the 135° position, as shown in Figure C.3.

C.5.4.6 The temperature shall be measured by means of an adapted probe on the external surface of the pipe at the root of the peeled strip.

C.5.4.7 Attach the appropriate mass to the free end of the strip, as shown in Figure C.3.

C.5.4.8 Peeling time shall be measured in minutes from the 135° position to the 180° position. Alternatively, terminate peeling when the test time, t , expressed in minutes, calculated in accordance with Equation (C.1), has been exceeded:

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

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

C.5.4.9 The distance peeled shall be measured in millimetres from the time the test is initiated until it is terminated.

C.5.4.10 The peeling speed, v_p , expressed in millimetres per minute, shall be calculated as given by Equation (C.2):

$$v_p = l/t \quad (\text{C.2})$$

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.

C.5.5 Report and test report

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

- date of test;
- identification of test specimen;
- specified pipe outside diameter;
- mass, expressed in kilograms;
- distance peeled, expressed in millimetres;
- peeling time, expressed in minutes;
- peeling speed, expressed in millimetres per minute;
- temperature of test.

Annex D (normative)

Thermal analysis of epoxy and cured epoxy coating film

D.1 General

Thermal analysis shall be used to characterize the uncured epoxy (powder, one-component liquid or two-component liquid) and the cured coating film.

Differential scanning calorimetry (DSC) shall be used. Reference can be made to ISO 11357-2 for a description of the general procedure and definitions. General handling and calibration shall be performed as in ISO 11357-2 unless stated otherwise in this part of ISO 21809.

D.2 Equipment

D.2.1 Differential scanning calorimeter (DSC), with cooling accessory.

D.2.2 Balance, accurate to 0,1 mg.

D.2.3 Sample-encapsulating press.

D.2.4 Aluminium pans, with covers.

D.2.5 Nitrogen gas supply, dry, analytical grade.

D.3 Procedure and measurement for epoxy

D.3.1 Procedure

For two-component epoxy liquid, taking into consideration the supplier's recommendations, accurately and separately homogenize each component before use and mix them together in the exact mixing ratio; then continue to homogenize completely the mix of base and hardener for about 5 min. The minimum quantity to be mixed is 100 g to avoid mixing mistakes.

For one-component epoxy liquid, taking into consideration the supplier's recommendations, completely homogenize 100 g of the sample before use.

D.3.2 Measurement

Perform the following heating cycles, starting with run (a) as the conditioning run for powder epoxy only.

- Run (a): heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to $70\text{ °C} \pm 5\text{ °C}$ at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$.
- Run (b): heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to $275\text{ °C} \pm 5\text{ °C}$ at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$. Hold for 3 min at $25\text{ °C} \pm 5\text{ °C}$.
- Run (c): heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to $T_g + 40\text{ °C}$ (typically 150 °C) at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$.

For certain epoxies, different heating cycles can be required according to the instructions of the epoxy manufacturer.

D.4 Evaluation of results

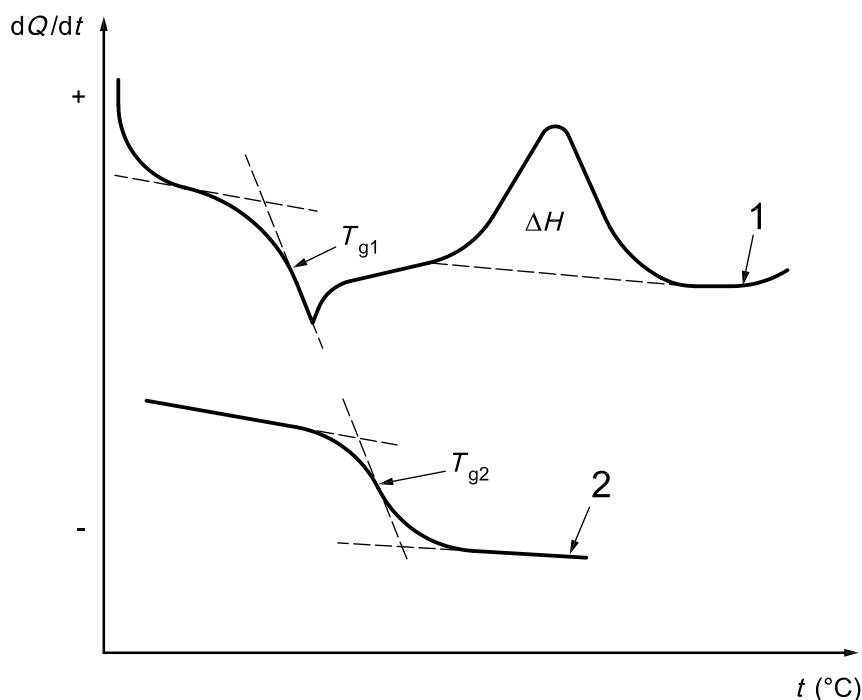
D.4.1 Glass transition temperature

The glass transition temperature, T_g , is calculated at the point of inflection intersection (see Figure D.1).

By evaluating run (b), the T_g of the uncured powder obtained is equal to T_{g1} . By evaluating run (c), the T_g of the cured material (powder and liquid) obtained is equal to T_{g2} .

D.4.2 Heat of reaction of the epoxy

The exothermic heat of reaction, ΔH , is obtained by integrating the exothermic peak of the DSC scan.



Key

- 1 run (b)
- 2 run (c)

Figure D.1 — Examples of thermal scans on epoxy powder

D.5 Procedures and measurement for coating sample

D.5.1 Sample preparation

D.5.1.1 Two-component epoxy liquid

Accurately and separately homogenize each component before use in accordance with the instructions of the manufacturer.

Mix them together in the exact ratio and completely homogenize the mix of base and hardener for about 5 min.

Apply the product at the thickness of 500 μm on an aluminium panel that has been completely degreased. The thickness of the panel is about 1 mm.

Let the film cure for at least 2 h at ambient temperature. Put the panel in a ventilated oven for 15 min at 170 °C, then take it out and let it cool at ambient temperature.

After about 2 h, bend the aluminium panel and take off one or more scales of the film in order to get the necessary mass of material to put in the test capsule.

As an alternative, a representative sample of cured film may be taken directly from the pipe.

D.5.1.2 One-component epoxy liquid

Homogenize the sample before use in accordance with the instructions of the manufacturer.

Apply the product at the thickness of 500 µm on an aluminium panel that has been completely degreased. The thickness of the panel is about 1 mm.

Let the film cure for at least 2 h at ambient temperature. Put the panel in ventilated oven for 15 min at 170 °C, then take it out and let it cool at ambient temperature.

After about 2 h, bend the aluminium panel and take off one or more scales of the film in order to get the necessary mass of material to put in the test capsule.

As an alternative, a representative sample of cured film may be taken directly from the pipe.

D.5.1.3 Epoxy powder

A representative sample of the cured film shall be taken directly from the pipe.

Weigh out 10 mg ± 3 mg to an accuracy of 0,1 mg. The pan is sealed with the cover. Determine the final mass after sealing.

Place the sample and the reference sample in the DSC cell and purge with dry, nitrogen gas.

Samples taken from pipes that have been stored or buried shall be dried before testing.

D.5.2 Measurement

The following heating cycles shall be performed, starting with run (a) as the conditioning run for the powder samples only.

Liquid epoxy samples shall start with run (b).

- Run (a): heat the sample from 25 °C ± 5 °C to 110 °C ± 5 °C at a rate of 20 °C/min and hold for 1,5 min, then cool the sample to 25 °C ± 5 °C.
- Run (b): heat the sample from 25 °C ± 5 °C to 275 °C ± 5 °C at a rate of 20 °C/min, then cool the sample to 25 °C ± 5 °C. Hold for 3 min at 25 °C ± 5 °C.
- Run (c): heat the sample from 25 °C ± 5 °C to $T_g + 40$ °C (typically 150 °C) at a rate of 20 °C/min, then cool the sample to 25 °C ± 5 °C.

For certain epoxies, different heating cycles can be necessary according to the instructions of the epoxy manufacturer.

Samples taken from pipes that have been stored or buried shall be dried before testing.

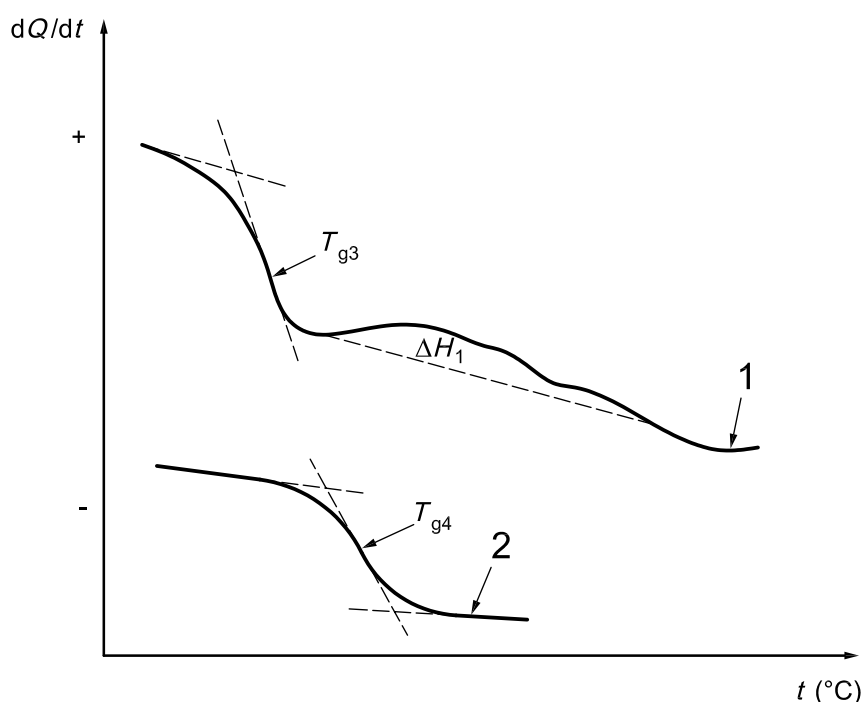
D.5.3 Evaluation of results

D.5.3.1 Glass transition temperature

The glass transition temperature, T_g , shall be calculated in the same way for the epoxy for run (b) and run (c) in Figure D.2 where T_{g3} is T_g of run (b) in D.5.2 and T_{g4} is T_g of run (c) in D.5.2, all expressed in degrees Celsius.

For coatings, determine ΔT_g , the change in T_g value, using Equation (D.1) and the T_g values defined in paragraph 1.

$$\Delta T_g = T_{g4} - T_{g3} \quad (D.1)$$



Key

- 1 run (b)
- 2 run (c)

Figure D.2 — Examples of thermal scans on coating

D.5.3.2 Residual heat of reaction of cured coating

The exothermic heat of reaction, ΔH_1 , shall be obtained by integrating the exothermic peak of the DSC scan run (b) in Figure D.2.

In a fully cured coating film, no residual heat of reaction should be observed.

The degree of conversion, C , expressed as a percentage, can be calculated as given in Equation (D.2):

$$C = \frac{\Delta H - \Delta H_1}{\Delta H} \times 100 \quad (D.2)$$

where

ΔH is the exothermic heat of reaction of the powder; run (b) in D.3.2;

ΔH_1 is the exothermic heat of reaction of the powder; run (b) in D.5.2.

D.6 Results and test report

Record the following information on uncured and cured material:

- reference to this part of ISO 21809;
- type of material and batch number;
- date of test;
- type of DSC equipment;
- for the epoxy powder: T_{g1} , T_{g2} , ΔH ;
- for the epoxy liquids: T_{g2} , ΔH ;
- for the cured coating film: T_{g3} , T_{g4} , ΔT_g , ΔH_1 and C .

Testing of production coating shall also require the pipe number or identification.

Annex E (normative)

Impact test

E.1 General

The test shall consist 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. Carry out the test on pipes or cut samples. Do not carry out this test on pipes with a diameter of less than 50 mm.

E.2 Equipment

E.2.1 Drop-weight testing machine, consisting of the following:

- Straight guide made of steel, aluminium or plastic, rigid and non-deformable, of inside diameter between 40 mm and 60 mm, at least as long as 1,30 m and containing a smooth and even inside surface. Provide the guide with
 - support and levelling devices (for example two spirit levels for the horizontal plane and a plumb line for the vertical plane),
 - graduated rod, which 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 $25 \text{ mm} \pm 1 \%$.
- Fix a small metal rod of 6 mm in diameter perpendicular to the flat face of the head and in its centre, where this rod shall be long enough to hold the additional weights required for the tests. Equip the punch with a suitable system for raising it to the required height; the mass of the assembly shall be appropriate to the energy being checked and shall be accurate $\pm 2 \%$.
- Weights, formed by 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 accurate to $\pm 2 \%$.

E.3 Procedure

E.3.1 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, adapt the method described, if necessary, to the agreement between the applicator and the purchaser.

E.3.2 The coated pipe shall be placed on a rigid and stable horizontal support and shall, if necessary, support the pipe interior to reduce its elastic response.

E.3.3 A holiday detection test shall be carried out prior to the impact test (see Annex B) to identify the defective points and avoid making the impact at these locations.

E.3.4 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. Ten impacts shall be

carried out, allowing the weight corresponding to the specified energy to fall from a height of 1 m. The points of impact shall be selected to avoid any protruding welds. Furthermore, the distance from the points of impact to the end of the pipe shall be at least $1,5D$ and at least 50 mm apart between the axes of the impacts.

E.3.5 The holiday detection test shall be performed at each location (see Annex B).

E.3.6 The hard steel punch shall be checked every 30 impacts. If damaged, it shall be replaced.

E.4 Results and test reports

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

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- weight used;
- date of test;
- test results.

Annex F (normative)

Indentation test

F.1 General

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

F.2 Equipment

F.2.1 Chamber, inside ventilated or a circulated bath, thermostatically controlled to ± 2 °C.

F.2.2 Penetrometer, comprised of

- a cylindrical indenter with a diameter of $1,8 \text{ mm} \pm 0,05 \text{ mm}$, with a cross-sectional area of $2,5 \text{ mm}^2$, on the top of which is mounted a weight. The assembly (indenter plus weight) shall produce a force of $25 \pm 0,5 \text{ N}$,
- a dial gauge, and
- another measurement system accurate to $\pm 0,01 \text{ mm}$.

F.3 Procedure

F.3.1 The test shall be performed three times on a coupon cut from the pipe or on strips of polyolefin removed from the pipe surface.

F.3.2 The test sample shall be held within the penetrometer assembly, in the thermostatically controlled chamber and set to the test temperature. The test sample shall be kept in the chamber for 1 h. The readings on the dial gauge shall be recorded.

F.3.3 The indenter shall be loaded with the mass of 2,5 kg in total into the equipment. A test duration of 24 h shall be used. The readings of the dial gauge shall be recorded.

F.4 Results and test reports

The resultant indentation shall be calculated as the difference between the dial gauge reading before and after the 24 h test duration. The average of the three samples shall be recorded.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex G (normative)

UV ageing test and thermal ageing test

G.1 UV ageing

G.1.1 General

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

Evaluation shall be performed by assessing the change in the material through the variation in its melt flow rate.

G.1.2 Equipment

G.1.2.1 Irradiation chamber, equipped with a xenon lamp (see ISO 4892-2).

G.1.2.2 Melt flow tester (see ISO 1133).

G.1.3 Sampling

The test shall be carried out on a sample of PE/PP in accordance with ISO 1872-2 for PE and ISO 527-2 for PP.

G.1.4 Procedure

G.1.4.1 The size of the sample shall be large enough to make ten test specimens of type 1B, 5A or 5B (see Figures 1 and A.2 in ISO 527-2).

G.1.4.2 The test sample shall be exposed under the following conditions:

- artificial weathering (see ISO 4892-2:2006, Table 1, Method A);
- black standard temperature ($65\text{ °C} \pm 3\text{ °C}$);
- relative humidity ($65\% \pm 5\%$);
- spray cycle: 18 min $\pm$ 0,5 min spray, 102 min $\pm$ 0,5 min dry;
- total radiant energy: 7 GJ/m² for Classes A and B; 5 GJ/m² for Class C;
- continuous exposure.

G.1.4.3 Three melt-flow-rate measurements shall be undertaken on the test sample in accordance with ISO 1133.

G.1.4.4 The melt flow rate, R_{MF1} , for the exposed samples shall be calculated as the arithmetic mean of the three results.

G.1.4.5 The melt flow rate, R_{MF0} , for an unexposed test sample of identical shape shall be calculated in accordance with ISO 1133.

G.1.5 Results and test report

The variation of the melt flow rate, ΔR_{MF} , expressed as the percentage variation after exposure, shall be calculated using Equation (G.1):

$$\Delta R_{MF} = \frac{R_{MF1} - R_{MF0}}{R_{MF0}} \times 100 \quad (G.1)$$

where

R_{MF0} is the melt flow rate measured before exposure;

R_{MF1} is the melt flow rate measured after exposure.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

G.2 Thermal ageing

G.2.1 General

The test shall consist of subjecting PE/PP material test samples to the effect of dry heat from a thermostatically controlled oven.

Evaluation shall assess the change in the material by the variation in its melt flow rate.

G.2.2 Equipment

G.2.2.1 Oven, thermostatically controlled, with air circulation maintaining a test temperature within ± 3 °C.

G.2.2.2 Melt-flow tester (see ISO 1133).

G.2.3 Sampling

The test shall be carried out on a sample of PE/PP material in accordance with ISO 1872-2 for PE and ISO 527-2 for PP.

G.2.4 Procedure

G.2.4.1 The test temperature and duration shall be in accordance with Table G.1.

Table G.1 — Temperature and duration of test

Coating class	Test duration h	Test temperature °C
A	2 400	100 °C ± 3 °C
B	4 800	100 °C ± 3 °C
C Design temperature, $T \leq 80$ °C	240	150 °C ± 3 °C
C Design temperature, $T > 80$ °C	$(T-70) \times 24$	150 °C ± 3 °C

G.2.4.2 Three melt-flow-rate measurements shall be undertaken on the test sample in accordance with ISO 1133.

G.2.4.3 The melt flow rate, R_{MF1} , for the exposed samples shall be calculated as the arithmetic mean of the three results.

G.2.4.4 The melt flow rate, R_{MF0} , for a test sample of identical shape that has not been placed in the oven shall be calculated in accordance with ISO 1133.

G.2.5 Results and test report

The results shall be expressed as the percentage variation of the melt flow rate, ΔR_{MF} , after exposure using Equation (G.1).

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex H (normative)

Cathodic disbondment test

H.1 General

The test shall consist of assessing the resistance to disbondment of damage to the coating when exposed to cathodic polarization.

The test shall be performed on test specimens taken from the coated components previously subjected to holiday detection (see Annex B), 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.

H.2 Equipment

H.2.1 Rectified d.c. power supply, with controlled voltage output.

H.2.2 Hotplate with a steel tray containing sand or steel grit/shot, controllable to within 3 °C, or an oven controllable to within 3 °C.

H.2.3 Calomel reference electrode.

H.2.4 Wire electrode, platinum, 0,8 mm to 1,0 mm.

H.2.5 Plastic cylinder, 75 mm $\pm$ 3 mm internal diameter.

H.2.6 Sodium chloride solution, 3 % in distilled water.

H.2.7 Utility knife.

H.2.8 Drill bit, 6 mm (see Figure H.1).

Key

- 1 fluted 6 mm drill bit
- 2 milled conic end
- a Steel.
- b Coating.

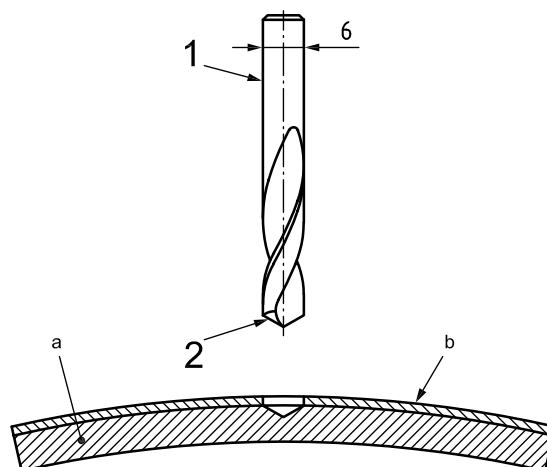


Figure H.1 — Production of artificial defect

H.3 Test specimens

Specimens from test rings shall be approximately 100 mm × 100 mm × the pipe wall thickness.

H.4 Procedure

H.4.1 Only test specimens that are confirmed to be holiday-free using a holiday detector as described in Annex B shall be used

H.4.2 A 6 mm diameter holiday shall be drilled in the centre of the test specimen through the coating to expose the steel substrate (see Figure H.1).

H.4.3 Centre the plastic cylinder over the holiday and apply a sealant to form a water-resistant seal.

H.4.4 The cylinder shall be filled to a height of at least 70 mm with the sodium chloride solution that has been preheated to the test temperature.

H.4.5 The solution level shall be marked on the cylinder.

H.4.6 The electrode shall be inserted into the solution and connected to the positive wire from the d.c. power supply.

H.4.7 The negative wire from the d.c. power supply shall be attached to a bare spot prepared on the test specimen.

H.4.8 Voltage shall be applied (negative with respect to the calomel reference electrode) to the test specimen, and maintained at a constant temperature under one or more of the following test conditions, as given in Tables 7 and 9. The test shall be carried out using one of the following sets of conditions once the temperature has been reached:

- a) -1,5 V, 20 °C ± 3 °C, for 28 d;
- b) -3,5 V, 65 °C ± 3 °C, for 24 h;
- c) -1,5 V, maximum operation temperature (maximum of 90 °C, see Table 7), for 28 d.

Maintain the solution level by the addition of distilled water as required (see Figures H.2 and H.3).

H.4.9 Upon test completion, the test cell shall be dismantled, the specimen air cooled to 20 °C ± 3 °C, and the cathodic disbondment characteristics of the test specimen evaluated within 1 h of the removal from heat.

H.4.10 Using the utility knife, 6 to 12 radial cuts through the coating shall be made to the substrate. Such cuts shall extend at least 20 mm from the centre of the holiday.

H.4.11 The tip of the blade of the utility knife shall be inserted under the coating at the holiday. Chip off the coating using a levering action. Continue until the coating demonstrates a definite resistance to the levering action.

H.4.12 The disbonded distance from the edge of the original holiday along each radial cut shall be measured, and the average of the measured values shall be calculated.

Key

- 1 working electrode
- 2 electrode (anode)
- 3 electrode (reference)
- 4 reference electrode
- 5 plastic cover
- 6 plastic pipe (minimum internal $\varnothing 75$ mm)
- 7 electrolyte ≥ 300 ml
- 8 coating
- 9 steel test piece
- 10 sealing material
- 11 artificial defect
- 12 sealing material
- 13 electrode (cathode)
- 14 platinum electrode $\varnothing 0,8$ mm to 1,0 mm (anode)
- 15 rectified d.c. source
- 16 power supply

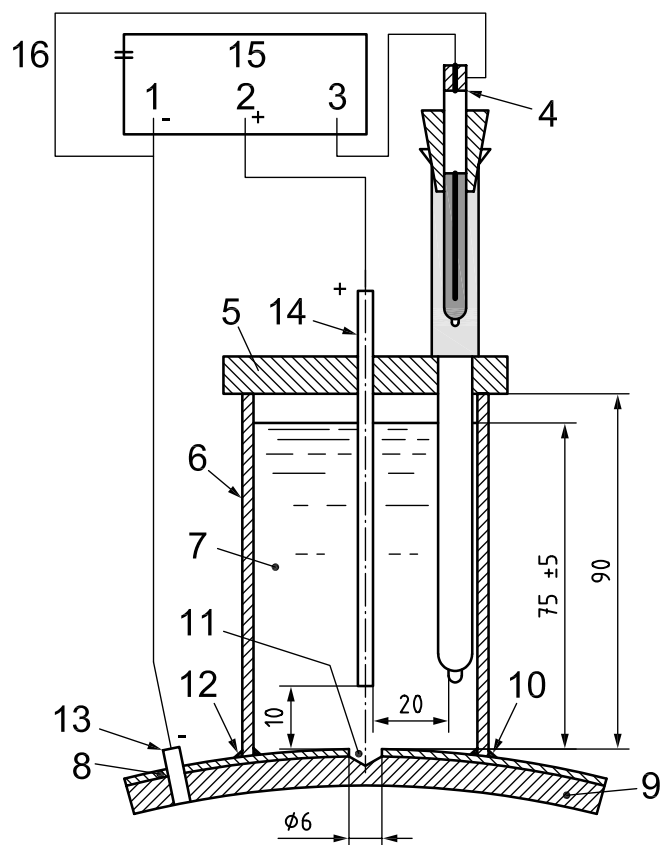


Figure H.2 — Electrolytic cell for NPS 4 and larger-diameter pipe

Key

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

- a To negative lead (–).
- b To positive lead (+).

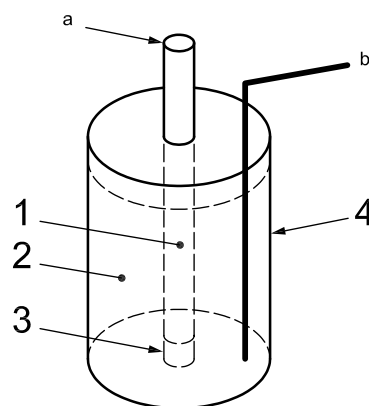


Figure H.3 — Electrolytic cell for pipe smaller than NPS 4 diameter pipe

H.5 Results and test report

The results shall consist of recording the average disbondment value, expressed in millimetres.

Testing of production coating requires pipe number or identification.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test voltage;
- test duration;
- test temperature;
- test results.

Annex I (normative)

Flexibility test

I.1 General

The test shall consist of assessing the flexibility of three-layer polyolefin coatings, applied to a bare steel substrate.

I.2 Equipment

I.2.1 Hydraulic press.

I.2.2 Bending mandrels, with fixed radii.

I.2.3 Freezer.

I.2.4 Strain gauges, if applicable.

I.2.5 Electric timing device, or stopwatch, capable of measuring 0,1 s intervals.

I.2.6 Thermometer.

I.3 Test specimen

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.

I.4 Procedure

I.4.1 The coating on the edge of the test sample shall be smoothed to remove any potential stress risers.

I.4.2 The test specimen shall be placed in the freezer, cooled to between $-2\text{ }^{\circ}\text{C}$ and $0\text{ }^{\circ}\text{C}$, and held at that temperature for a minimum of 1 h.

I.4.3 The effective sample thickness, d , which includes the actual sample thickness and any curvature, shall be determined by placing the specimen on a flat surface and measuring the effective thickness shown in Figure I.1.

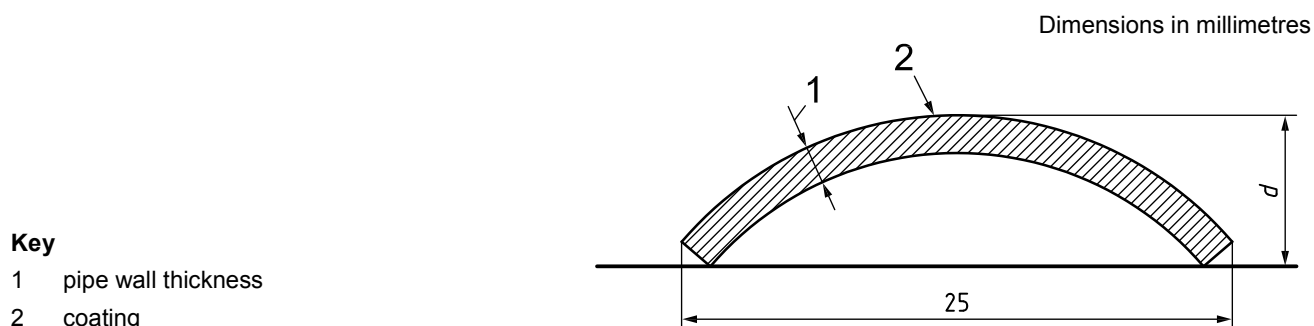


Figure I.1 — Effective strap thickness diagram

I.4.4 The mandrel radius, r , expressed in millimetres, which corresponds to an angle of deflection of 2° per pipe diameter length, shall be determined by using Equation (I.1):

$$r = 28,15 \times d \quad (\text{I.1})$$

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

I.4.5 The test sample shall be bent over a mandrel whose radius is not larger than that determined in accordance with Equation (I.1).

I.4.6 The specimen shall be bent 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.

I.4.7 The bent test sample shall be warmed to $23^\circ\text{C} \pm 3^\circ\text{C}$, and held within this temperature range for a minimum of 2 h. Within the next hour, visually inspect it for the presence of cracks.

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

I.5 Result and test report

The presence of cracking shall constitute a failure in accordance with Table 7.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- total coating thickness;
- date of test;
- test results.

Annex J (normative)

Gel time of the epoxy powder

J.1 General

The test shall consist of assessing the gel time of the epoxy powder used in the three-layer coating.

J.2 Equipment

J.2.1 Hotplate, controllable to within 3 °C.

J.2.2 Metal plate, placed on top of the hotplate.

J.2.3 Stopwatch or **electric timing device**, capable of measuring 0,1 s intervals.

J.2.4 Draw-down tool (see Figure J.1).

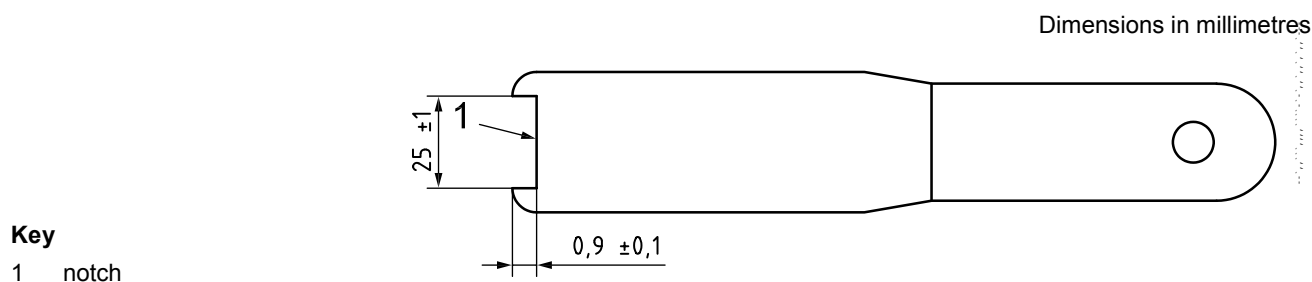


Figure J.1 — Draw-down tool

J.3 Procedure

J.3.1 Three tests shall be conducted and the results averaged.

J.3.2 The metal plate surface shall be heated and maintained so that the area in contact with the powder is at a temperature of $205\text{ °C} \pm 3\text{ °C}$.

J.3.3 The bottom 25 mm of the draw-down tool shall be covered with epoxy powder.

J.3.4 The epoxy powder shall be drawn across and deposited on the metal plate using a smooth motion while holding the tool at an angle of approximately 45° to the metal plate, thereby creating a tongue of epoxy powder approximately 25 mm wide.

NOTE The target thickness of the cured film is 300 µm to 400 µm.

J.3.5 Starting the timing device and deposition of epoxy powder on the metal plate surface shall be simultaneous.

J.3.6 The draw-down tool shall be held at an angle of approximately 45° to the hotplate surface, in a manner that allows most of the tool's weight to be borne on the plate. Repeatedly draw the edge of the tool

through the melted epoxy powder. The timing device shall be stopped when the tool rides up on the gelled powder and no longer contacts the metal plate, as illustrated in Figure J.2.

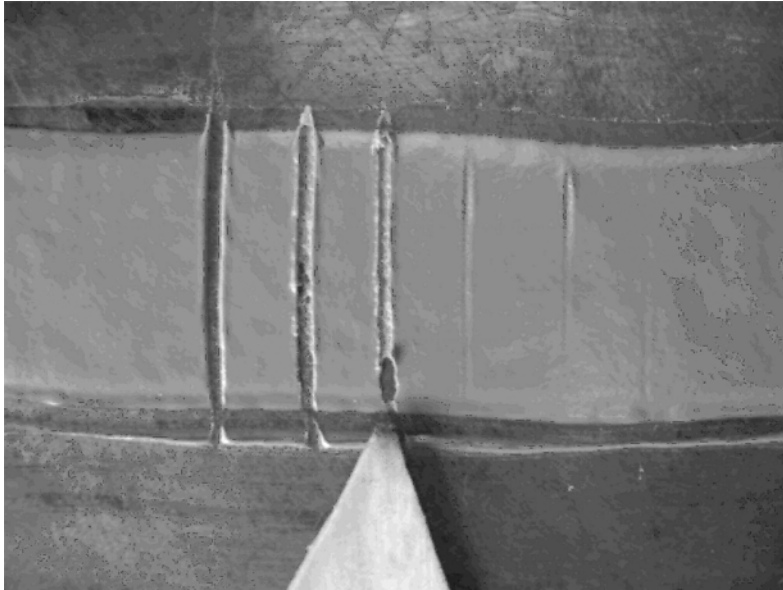


Figure J.2 — Gel time assessment

J.4 Results and test reports

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex K (normative)

Total volatile/moisture content of the epoxy powder — Mass loss

K.1 General

The test shall consist of assessing the loss of volatiles from the epoxy powder.

K.2 Procedure A — Manual method

K.2.1 Equipment

K.2.1.1 Oven, controllable to within 3 °C.

K.2.1.2 Balance, accurate to 0,001 g.

K.2.1.3 Desiccator.

K.2.1.4 Sample container.

K.2.2 Procedure

K.2.2.1 The sample container shall be weighed to the nearest 0,001 g.

K.2.2.2 Approximately 10 g of epoxy powder shall be transferred in the sample container.

K.2.2.3 The sample container and epoxy powder shall be weighed to the nearest 0,001 g.

K.2.2.4 The sample container with the epoxy powder shall be placed in the oven for a maximum of 2 h at 105 °C ± 3 °C.

K.2.2.5 The container shall be removed from the oven and placed it in the desiccators to cool.

K.2.2.6 The sample container shall be weighed when it has cooled to 23 °C ± 3 °C.

K.2.2.7 The sample container shall be returned to the desiccators; repeat at intervals of 60 min ± 10 min until two consecutive mass determinations are within 0,001 g.

K.2.2.8 The percentage of moisture, w_m , shall be calculated using Equation (K.1):

$$w_m = \frac{M_I - M_F}{M_I - M_C} \times 100 \quad (\text{K.1})$$

where

M_I is the initial mass of the sample container and epoxy powder, expressed in grams;

M_F is the final mass of the sample container and epoxy powder, expressed in grams;

M_C is the mass of the sample container, expressed in grams.

K.3 Procedure B — Automatic procedure

The moisture content of the epoxy powder shall be determined using a machine that automatically determines moisture content by mass loss.

K.4 Results and test reports

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- instruments used;
- reference to this part of ISO 21809;
- date of test;
- test results.

Annex L (normative)

Procedure qualification trial, inspection and testing plan and daily log

L.1 General

If specified, a procedure qualification trial, PQT, shall be carried out in accordance with L.2 to verify the capability of the coating application procedure, coating materials, tools/equipment and personnel to produce pipeline coating with the required properties.

If specified, an inspection and testing plan, ITP, shall be produced in accordance with L.3.

L.2 Procedure qualification trial

L.2.1 All items in the APS, from surface preparation to preparation of pipe ends shall be performed and inspected/tested.

L.2.2 The specific process parameters shall be selected to be used during the PQT so that the suitability of the ranges and combinations specified in the APS can be verified (e.g. a maximum value for one parameter may be applied in combination with a minimum value for another, if deemed critical).

L.2.3 Requirements for the frequency of testing and inspection, methods and acceptance criteria are contained in Tables 8 and 9.

L.2.4 The results from the PQT shall be documented in a report, including the process parameters used, the APS and the coating material certificates. Any recommendations for revision of the APS that apply for production shall be highlighted in the report.

L.3 Inspection and testing plan and daily log

L.3.1 All coating application and quality control activities shall be identified in the ITP in a consecutive order, including recordings of the process parameters that are relevant to each activity.

L.3.2 For each process control, the ITP shall identify the following:

- applicable standards or applicator's procedures;
- frequencies;
- inspection/testing activity;
- acceptance criteria;
- actions in case of non-conformity.

L.3.3 If specified, a daily log shall be used to record all inspection and testing data, process parameters and calibrations of equipment for quality control.

Annex M **(normative)**

Hot water immersion test

M.1 General

The test shall consist of a hot water immersion procedure to test the resistance of factory-applied, three-layer polyolefin coating on pipe to loss of adhesion from a steel substrate in a wet environment.

The testing shall require that samples for testing be cut from line pipe, and is applicable to PQT/PPT and production testing, and to system qualification testing, where required.

It may be necessary to use a “dummy” pipe in some instances.

M.2 Equipment

M.2.1 Oven or heating bath, controllable to $80\text{ °C} \pm 3\text{ °C}$.

M.2.2 Glass vessel, of a suitable size to accommodate the samples, with a glass/plastic cover to minimize evaporation.

M.2.3 Utility knife.

M.3 Test specimens

M.3.1 For pipes with an outer diameter of up to 76 mm (3 in), test samples shall be cold-cut into 150 mm pipe rings with the applied factory coating.

M.3.2 For larger pipes, test samples shall be cold-cut into 150 mm × 100 mm segments, with the shorter side transverse to the pipe axis.

M.3.3 The exposed surfaces shall be prepared by wet grinding with 120-grit abrasive paper.

M.3.4 Three samples shall be prepared from the same pipe for each test.

M.4 Procedure

M.4.1 The specimens shall be placed in a glass vessel filled with distilled/deionized water already heated to 80 °C. Ensure that the test specimens are covered by at least 50 mm of water. Maintain the water and samples at 80 °C for 48 h.

M.4.2 After the conditioning period, the samples shall be removed from the bath and dried with paper or tissue.

M.4.3 The samples shall be examined after they have cooled to room temperature.

M.4.4 The samples shall be examined visually for loss of adhesion at the coating/substrate interface along all four sides. Do not take into account any coating disbondment for 5 mm on each side of the corners of a sample.

M.4.5 At any area where loss of adhesion to the substrate has occurred, a sharp knife at the coating/substrate interface shall be used to assess for loss of adhesion by pressing the blade into the interface and rotating it on the longitudinal axis, trying to lift off any of the coating that has lost adhesion.

M.4.6 If any loss of adhesion is found visually or by use of the knife, remove the non-adherent coating system in that area with a sharp knife, in order to expose the substrate under the disbonded coating.

M.4.7 The maximum and average depth of adhesion loss, expressed in millimetres, of the three-layer polyolefin coating system in the disbonded area shall be measured.

M.4.8 Take photographs of the overall sample tested and close-up photographs of any areas of loss of adhesion of the three-layer polyolefin coating system.

M.5 Results and test reports

The average and the maximum loss of adhesion of the coating in all areas where disbonding of the coating has occurred shall be recorded, except for a distance of 5 mm each side of the corners of the sample.

The photographs taken shall be enclosed with the results.

The test reports shall include at least the following:

- identification of test specimens;
- procedure used;
- reference to this part of ISO 21809;
- pictures;
- date of test;
- test results.

Annex N (normative)

Density of epoxy powder

N.1 General

The test shall consist of evaluating the density of the epoxy powder.

N.2 Procedure A — Manual procedure

N.2.1 Equipment

N.2.1.1 Balance, accurate to 0,01 g.

N.2.1.2 Flask, 100 ml volumetric.

N.2.1.3 Mineral spirit.

N.2.2 Procedure

N.2.2.1 The flask shall be weighted to the nearest 0,01 g.

N.2.2.2 Approximately 20 g of epoxy powder shall be added to the flask and the mass of the flask and powder shall be measured to an accuracy of 0,01 g.

N.2.2.3 Sufficient mineral spirit shall be added to cover and wet the epoxy powder. The flask shall be sealed with a stopper and agitated for several minutes to ensure that neither air pocket nor lumps of powder exist.

N.2.2.4 The stopper and walls of the flask shall be washed with mineral spirits until they are free of powder and the flask is filled to the 100 ml level.

N.2.2.5 The flask plus contents shall be weighed to an accuracy of 0,01 g.

N.2.2.6 The flask shall be emptied, cleaned and dried.

N.2.2.7 100 ml of mineral spirit shall be added to the flask and weighed to an accuracy of 0,01 g.

N.2.2.8 The density of the mineral spirit, ρ_s , expressed in grams per litre, shall be calculated using Equation (N.1):

$$\rho_s = \frac{(M_{fs} - M_f)}{0,1} \quad (N.1)$$

where

M_{fs} is the mass of the flask plus mineral spirit, expressed in grams;

M_f is the mass of the flask, expressed in grams.

N.2.2.9 The density of the epoxy powder, ρ_p , expressed in grams per litre, shall be calculated using Equation (N.2):

$$\rho_p = \frac{(M_{fp} - M_f)}{0,1 - \left(\frac{M_{fps} - M_{fp}}{\rho_{ps}} \right)} \quad (\text{N.2})$$

where

M_{fp} is the mass of the flask plus epoxy powder, expressed in grams;

M_f is the mass of the flask, expressed in grams;

M_{fps} is the mass of the flask plus epoxy powder and mineral spirits, expressed in grams;

ρ_{ps} is the density of the mineral spirits, expressed in grams per litre.

N.3 Procedure B — Automatic procedure

The density of the epoxy powder shall be determined using an air or helium pycnometer in accordance with ISO 8130-2 or ISO 8130-3.

N.4 Results and test reports

The test reports shall include at least the following:

- epoxy powder batch number;
- date of test;
- procedure used;
- type of pycnometer used for procedure B;
- density of epoxy powder, expressed in grams per litre.

Bibliography

- [1] ISO 14001, *Environmental management systems — Requirements with guidance for use*
- [2] ISO/TS 29001, *Petroleum, petrochemical and natural gas industries — Sector-specific quality management systems — Requirements for product and service supply organizations*
- [3] ISO 3183, *Petroleum and natural gas industries — Steel pipe for pipeline transportation systems*
- [4] ASTM E29, *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*

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