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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 et immergées utilisées dans les systèmes de transport par conduites —

Partie 1: Revêtements à base de polyoléfines (PE tri couche et PP tri couche)

ICS 75.200

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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, *Petroleum 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*

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Introduction

Users of this part of ISO 21809 should be aware that further or differing requirements might 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, the vendor should identify any variations from this part of ISO 21809 and provide details.

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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 extruded 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 as defined in ISO 13623.

NOTE Pipes coated in accordance with this part of ISO 21809 are considered suitable to further 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, *Quantities and units – Part 0: General principles*

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 1: 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 and relative density of non-cellular plastics*

ISO 1872, *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*

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

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

ISO 3183 (all parts)*, Petroleum and natural gas industries- steel pipe for pipelines*

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

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

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

ISO 6964, *Polyolefin pipes and fittings - Determination of carbon black content by calcinations and pyrolysis; test method and basic specification*

ISO 8501(all parts), *Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness*

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

ISO 8503 (all parts), *Preparation of steel substrates before application of paints and related products — Surface roughness characteristics of blast-cleaned steel substrates*

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

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

ISO 11126, *Preparation of steel substrates before application of paints and related products – Specification 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 calorimetric (DSC) — Part 2: Determination of glass transition temperature*

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

ISO 18533, *Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds*

ISO 15512, *Plastics – Determination of water content*

ASTM D 1693, *Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics*

ASTM E-29-02, *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*

ASTM D 4940, *Standard Test method for conductimetric analysis of water soluble ionic contamination of blasting abrasives*

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3 Terms and definitions

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

3.1
applicator
company which 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
application procedure specification
APS
document describing procedures, methods, equipment and tools to be used for coating application

3.3
batch
quantity of material produced in a continuous manufacturing operation using raw materials of the same source and grade

3.4
batch certificate
certificate of analysis issued by the manufacturer

3.5
certificate of compliance
one of the types of documents defined by ISO 10474 to be issued in accordance with the purchasing requirements

3.6
cut-back
length of pipe left uncoated at each end for joining purposes (e.g. welding)

3.7
design temperature range
temperature range including maximum and minimum temperature likely to be reached during transport, handling, installation and operation

3.8
end user
company operating the pipeline system

3.9
holiday
coating discontinuity that exhibits electrical conductivity when exposed to a specific voltage

3.10
inspection and testing plan
(ITP)
document providing an overview of the sequence of inspections and tests, including appropriate resources and procedures Symbols and abbreviated terms

3.11
manufacturer
company responsible for the manufacture of coating material(s)

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3.12
manufacturer's specification
document which specifies the characteristics, test requirements and application recommendations for the coating materials.

3.13
operating temperature
temperature experienced by the pipe or pipeline system during operation which should not exceed the design temperature.

3.14
pipeline
those facilities through which fluids are conveyed, including pipe, pig traps, components and appurtenances, up to and including the isolating valves

[ISO 13623:2000]

3.15
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

[ISO 13623:2000]

3.16
procedure qualification trial
PQT
application of a coating and subsequent inspection/testing of its properties to confirm that the APS is adequate to produce coating of specified properties

3.17
purchaser
company responsible for providing the product order requirements

3.18
test report
document that provides the quantitative test results for tests conducted in accordance with the requirements of this part of ISO 21809

3.19
total coating thickness
sum of all three layers as defined in 8.1

4 Symbols and abbreviated terms

4.1 Symbols

ΔT_g	variation of the glass transition temperature (T_{g4} - T_{g3}) in °C
T_g	glass transition temperature in °C

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4.2 Abbreviations

DSC	differential scanning calorimetry
ESC	Environmental stress cracking resistance
FBE	fusion bonded epoxy
HDPE	high density polyethylene
LDPE	low density polyethylene
M	nominal pipe mass per metre (kg/m)
MDPE	medium density polyethylene
MFR	melt flow rate
PE	Polyethylene
PP	Polypropylene
SAW	submerged arc welding
3LPE	three layer polyethylene coating
3LPP	three layer polypropylene 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, Annex B; Rule A.

NOTE For the purpose of this provision, the rounding method of ASTM E 29 is equivalent to ISO 31-0, Annex B, Rule A

5.2 Compliance to standard

A quality system should be applied to assist compliance with the requirements of this document.

NOTE ISO/TS 29001[1] gives sector-specific guidance on quality management systems.

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 investigations necessary in order to be assured of compliance by the applicator and to reject any material that does not comply.

6 Coating classification

6.1 General

Coating class shall be selected based on the expected field duty. The transport, handling, laying conditions, operating conditions, soil conditions and the expected environmental conditions shall be considered in the selection of the coating thickness.

6.2 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.

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Table 1 — Coating classes and design temperatures

Coating class	A	B	C
Top layer material	LDPE	MDPE/HDPE	PP
Design temperature (°C)	-20 °C to + 60°C	-40 °C to + 80°C	-20 °C to + 110 °C

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

6.3 Coating thickness classes

Coating thickness class shall be selected based on the soil or ground, laying method on land and offshore, service conditions and pipe dimensions. The coating thickness class, as shown in Table 2 as a function of expected field duty and pipe weight, shall be specified in the purchase order.

Table 2 — Minimum total coating thickness

M kg/m	**Coating thickness** mm								
	Class A1	**Class A2**	**Class A3**	**Class B1**	**Class B2**	**Class B3**	**Class C1**	**Class C2**	**Class C3**
$M \leq 15$	1,8	2,1	2,6	1,3	1,8	2,3	1,3	1,7	2,1
$15 < M \leq 50$	2,0	2,4	3,0	1,5	2,1	2,7	1,5	1,9	2,4
$50 < M \leq 130$	2,4	2,8	3,5	1,8	2,5	3,1	1,8	2,3	2,8
$130 < M \leq 300$	2,6	3,2	3,9	2,2	2,8	3,5	2,2	2,5	3,2
$300 < M$	3,2	3,8	4,7	2,5	3,3	4,2	2,5	3,0	3,8

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

Classes A1, B1, C1 are for light duty; e.g. when the water/humidity barrier is not a major issue or when an anti rock system is adopted (special precautions are required in the project specification during storage, transport and handling; trench bottom is prepared by a partial or total backfilling with sand or very fine soil –absence of stones – etc).

Classes A2, B2, C2 are for moderate duty

Classes A3, B3, C3 are for heavy duty such as rocky or clay soil, e. g. a good water/humidity barrier is required (submerged pipeline), an anti rock system is not adopted or when storage, transport, handling, laying and service conditions are very severe (high temperature, harsh conditions, etc.).

7 Information to be supplied by the purchaser

7.1 General information

The purchase order shall include the following information:

a) ISO Standard designation and year of publication (ISO 21809-1:xxxx);

b) bare pipe standard or specification designation (e.g. ISO 3183)

c) pipe quantity, outside diameter, minimum wall thickness, minimum, maximum and nominal length, grade of steel;

d) coating class and coating thickness class as per Tables 1 and 2;

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e) design temperature range;

f) type of certificate of compliance.

7.2 Additional information

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

a) pipe tracking and traceability of pipes to coating materials;

b) permissible number of coating repairs, if different from the one defined in Clause 12;

c) marking of pipes;

d) handling procedures;

e) storage procedures;

f) documentation and schedule for supply of documentation;

g) purchaser approval of APS;

h) inspection and testing plan and/or daily log;

i) inspection of incoming pipes;

j) pipe end protection;

k) minimum thickness of epoxy layer and/or total coating thickness required if exceeding those in Table 9 and Table 2 respectively;

l) surface design of PE/PP coating (e.g. concrete weight coating (CWC) or special laying methods);

m) special requirements to supply of coating materials (e.g. supplier specific products and certification);

n) cutback configuration (length, angle and visible epoxy) other than specified in this part of ISO 21809;

o) methods, frequency and acceptance criteria for inspection and testing differing to this part of ISO 21809;

p) procedure qualification trial (PQT).

8 Coating materials

8.1 Composition of the coating system

The coating system shall consist of three layers:

— 1st layer: Epoxy (liquid or powder)

— 2nd layer: Adhesive (powder sprayed or extruded)

— 3rd layer: PE/PP top layer (applied by extrusion)

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

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8.2 Qualification of the coating materials

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 basic requirements of Clause 8 and Table 3, Table 4 and Table 5. The test report shall contain the results of the qualification tests and the data required in Table 6, Table 7 and Table 8.Epoxy material

8.3 Epoxy material

The applicator shall use epoxy material that is in compliance 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/cm^3	ISO 2811	As per manufacturer's specification ± 0,05	
Solid content of base & hardener	% mass	ISO 3251	≥ 95	
Minimum glass transition temperature (DSC Analysis)[b]	°C	ISO 11357	≥ 95 Manufacturer's specification	At least 5 above design temperature with a minimum of 95
Gel time (s) at 205 °C		Manufacturer's specification	Within the manufacturer's specification	
Epoxy powder (FBE)				
Properties	**Unit**	**Test Method**	**Requirements**	
			Class A and Class B	**Class C**
Moisture content	% by mass	Annex K	≤ 0,6	
Minimum glass transition temperature (Tg2) (DSC Analysis)	°C	Annex D	≥ 95°C	At least 5 above design temperature with a minimum of 95 °C
Gel time	%	Annex J ISO 8130-2 or 3	Within 20 per manufacturer's specification	
Density	g/cm^3		As per manufacturer's specification ± 0,05	

[a] Liquid mono-component is limited to class A only

[b] Curing condition (procedure as manufacturer's specification)

8.4 Adhesive material

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

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

Properties	Unit	Test Method	Requirements		
			Class A	Class B	Class C
Elongation at break at (23 ± 2) °C [a]	%	ISO 527	≥ 600	≥ 600	≥ 400
Tensile yield strength at (23 ± 2) °C	MPa	ISO 527	≥ 5	≥ 8	≥ 12
Notched impact strength at minimum class temperature	kJ/m^2	ISO 179	--	--	≥ 3
Vicat softening temperature A/50 (9.8N)	°C	ISO 306	≥ 60	≥ 85	≥ 110
Water content	%	ISO 15512	≤ 0,1	≤ 0,1	≤ 0,1
[a] 2 mm thick compression moulded sheet, test specimen type according to ISO 527-2, strained at 50 mm/min (for PP based adhesives the specimen can be prepared also by injection moulding according to ISO 1873-2)					

8.5 PE/PP top layer material

The applicator shall use PE/PP material that is in compliance 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 [a]	g/cm^3	ISO 1183	≥ 0,920	≥ 0,930	≥ 0,890
Elongation at break at (23 ± 2) °C	%	ISO 527	≥ 600	≥ 600	≥ 400
Table 6 — Tensile Yield strength at (23 ± 2) °C [b]	MPa	ISO 527	≥ 10	≥ 15	≥ 20
Notched impact strength at minimum class temperature	KJ/m^2	ISO 179	--	--	≥ 3
Vicat softening temperature A/50 (9.8N)	°C	ISO 306	≥ 95	≥ 110	≥ 130
Water content	%	ISO 15512	≤ 0,05	≤ 0,05	≤ 0,05
Hardness Shore D	--	ISO 868	≥ 45	≥ 55	≥ 60
ESCR (50 °C, F50, Cond. A or B, 10% Igepal CO 630) [c]	h	ASTM D-1693	≥ 300 Cond.A	≥ 1000 Or ≥ 300 if density >0.955 Cond.B	n.a.

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Properties	Unit	Test Method	Requirements		
			Class A	Class B	Class C
Oxidation induction time (intercept in the tangent method)	min	ISO 11357	≥ 30 at 210 °C	≥ 30 at 210 °C	≥ 30 at 220 °C
UV Resistance and thermal ageing [b]	%	Annex G	ΔMFR ≤ 35	ΔMFR ≤ 35	ΔMFR ≤ 35

[a] Preparation of the test specimen compression moulded sheet annealed for 30 min at 100 °C.

[b] 2 mm thick compression moulded sheet, specimen ISO 527-2, strained at 50 mm/min (for PP the sample can be prepared also by injection moulding according to ISO 1873-2).

[c] In order to validate the ESCR values, data shall be provided by manufacturers as to know 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.

The polyolefin shall be compounded and stabilized by additives against UV and thermal ageing. If the compounding is done during the application process the applicator shall qualify the process and carry out all the relevant tests versus the requirements of this part of ISO 21809 and shall certify that the compound is in compliance with this part of ISO 21809.

8.6 Batch certificate

Batch certificates shall be 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.

Table 6 — Batch certificate

Content	Coating material			
	Liquid primer	FBE primer	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
Pigment dispersion				x
Thermal characteristic	x	x		
Reactive site content			X	

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Technical information concerning the epoxy materials provided by the manufacturer(s) shall be in accordance with the following:

— trade name;

— generic type;

— mix ratio (if any);

— batch certificate;

— maximum thickness (mm), see Annex A;

— storage temperature min and max (°C);

— shelf life at storage temperature (months);

— technical properties (as per Table 3).

Technical information concerning the adhesive and PE/PP materials provided by the manufacturer(s) shall be in accordance with the following:

— trade name;

— description of the adhesive;

— colour;

— storage conditions;

— shelf life (months);

— technical properties (as per Table 4 and 5).

8.7 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 to meet the requirements of Table 7. Qualification shall be carried out separately for each coating line.

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:

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- incoming inspection of pipes and pipe tracking
- data sheets for coating materials, including any materials to be used for coating repairs
- data sheets for abrasive blasting materials
- certification, receipt, handling and storage of materials for coating and abrasive blasting
- preparation of the steel surface including control of environmental parameters, methods and tools for inspection 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.

If a preliminary APS is issued for a PQT (see 9.3), a revised APS shall be issued, if required to meet the project specifications based on the results of the PQT.

Coating work and associated inspection and testing shall be carried out in accordance with the APS.

9.3 Procedure qualification trial

If specified, the coating application procedure 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.

Table 7 — Properties of the applied coating

Properties	Unit	Test Method	Class A	Class B	Class C
Appearance and continuity	-	Annex B	Uniform colour, free of defects and discontinuities, delaminations, separations and holidays.		
Minimum impact strength at 23 °C [f]	J/mm	Annex E	> 5	> 7	> 10
Indentation at 23 °C at maximum class temperature	mm	Annex F	≤ 0,3 ≤ 0,4	≤ 0,2 ≤ 0,4	≤ 0,1 ≤ 0,4
Elongation at break of the PE/PP (23 ± 3) °C	%	ISO 527	≥ 400	≥ 400	≥ 400

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Properties	Unit	Test Method	Class A	Class B	Class C
Peel strength[a]	N/mm	Annex C	≥ 10 at 23 °C ≥ 2 at 60 °C[b]	≥ 15 at 23 °C ≥ 3 at 80 °C[c]	≥ 25 at 23 °C [e] ≥ 4 at 90 °C or at maximum operating temperature if above 90 °C[c]
Degree of cure of the epoxy (1st layer)	°C	Annex D	ΔT_g : ≤ 3 T[d T] Scratch test for liquid epoxy		
Product stability during extrusion of the PE/PP top layer process	%	ISO 1133	≤ 20 Δ MFR for classes A and B; ≤ 35 for class C (virgin compounded granulate before extrusion/extruded foil after extrusion of the same batch)		
Average radius of cathodic disbondment at: 23 °C/28 days; -1,5 V or 65 °C/ 24h;-3,5 V Maximum operation temperature/28days/ –1.5 V	mm	Annex H	≤ 7 ≤ 7 ≤ 15		
Flexibility at (0 ± 3) °C	%	Annex I	No cracking at 2,0 angle per pipe diameter length		

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

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

c No disbonding between steel and epoxy.

d For FBE with a glass transition temperature above 120 °C the material manufacturer shall recommend the test method.

e In case of break at 23 °C the maximum peel force shall be recorded. The minimum requirements at 23 °C must be fulfilled and the test shall be carried out at the maximum design temperature.

f Lower test temperature maybe agreed upon using the same acceptance criteria.

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, 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.

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10.1.2 Abrasive blast cleaning

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

NOTE If the pipe to be coated is of a high strength grade e.g. X80, X100, X120, harder abrasives might be needed to provide the required cleanliness and surface profile.

The abrasives shall be maintained clean, dry and free from contaminants in accordance to SSPC-AB 1, SSPC-AB 2 and SSPC-AB 3 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 grade Sa 2½ ('near white metal').

The surface profile attained shall be within the height of between 50 µm and 100 µm as measured in accordance with the requirements of ISO 8503-4 (Stylus) or ISO 8503-5 (Replica Tape).

If grinding is required after blast cleaning, the maximum allowable area of grinding shall be 10 cm² per 1 m pipe length.

10.1.3 Surface cleanliness

The surface cleanliness shall be measured in accordance with the requirements of ISO 8502-3. The maximum allowable level shall be Class 2.

10.1.4 Surface pre-treatment

If the applicator chooses a surface pre-treatment (e.g. deionised 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 pre-qualification it must 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/m^2 after blasting

If high levels of soluble salts are measured above 20 mg/m^2 a surface pre-treatment cleaning process shall be agreed upon 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. The use of tempil sticks shall be agreed and verified during qualification of the coating system.

Once the coating temperature is established, the temperature of every fifth pipe shall be taken and recorded.

10.2.2 Epoxy application

Following surface preparation, the surface to be coated shall not be exposed for a length of time which could result in flash rust detrimental to the quality and adhesion of the coating.

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.

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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 method shall be lateral or cross-head extrusion.

The extrusion conditions and the mass temperature of the extruded material shall be in accordance with the APS.

The applied coating shall be cooled to a temperature which 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 over a length unless otherwise specified.

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

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

Precautions shall be taken to avoid disbondment on the cutback.

11 Inspection and testing

11.1 General

Inspection and testing shall be carried out in accordance with the APS and meet the requirements of Table 8 and Table 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
Surface condition after blasting	mg/m^2	conductive measurement, ISO 8502-9	salt content max. 20	each pipe	5 pipes at start of production and 1 pipe/shift
Environmental conditions		Calculation	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

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Properties	Unit	Test Method	Requirements	Frequency Qualification	Frequency Production
Size, shape and properties of abrasive		Visual + certification ISO 11124 resp. ISO 11126	conformity to certificate, compliance to manufacturing/ working procedures	once	1/day
Water soluble contamination of abrasives	mS/cm	ASTM 4940	conductivity max. 60	once	1/shift
Surface roughness of blasted surface (R_z/R_{y5})		ISO 8503-4	50 µm to 100 µm	5 pipes	every 1 h
Visual inspection of blasted surface		ISO 8501-1	grade Sa 2 ½	each pipe	each pipe
Presence of dust after dust removal		ISO 8502-3	max. class 2	5 pipes	every 1 h
Pipe condition prior to coating	°C	monitoring	no rust, pipe temperature at least 3 above the dew point	continuously	continuously
Temperature of extruded adhesive and polyolefin		thermometer	compliance to APS	once	every 1/h
Preheating temperature before coating		thermometer	compliance to APS	each pipe	every 5th pipe

Table 9 – Requirements for inspection and testing of applied coating

Properties	Unit	Test Method	Requirements	Frequency Qualification	Frequency Production
Minimum epoxy thickness	µm	ISO 2808	liquid epoxy: 25 FBE: 125	at start up	each start up
Minimum adhesive thickness	µm		150 on pipe body	at start up	each start up
Degree of cure	°C	Annex D ISO 11357-2	$\Delta T_g \leq +3$ for FBE Scratch test for liquid epoxy	at start up	each start up and every 8 h of production
Appearance and continuity		visual Annex B	Table 7	continuously	continuously
Total thickness of coating		Annex A	Table 2	5 pipe	every 10 pipes
Impact resistance		Annex E	Table 7	3 pipe	once per PE/PP batch
Peel strength		Annex C	Table 7	5 pipes [a]	every 4 h

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Properties	Unit	Test Method	Requirements	Frequency Qualification	Frequency Production
Indentation		Annex F	Table 7	once	each PE/PP batch
Elongation at break		ISO 527	Table 7	once	Each PE/PP batch
Cathodic disbondment		Annex H	Table 7	once	1/day
Flexibility		Annex I	Table 7	once	-
In process degradation of PE/PP)	%	ISO 1133	ΔMFR max. 35 for PP; max. for 20 for PE between raw and extruded material	once	at start up
Cutback		Measuring	10.3	each pipe	each pipe
Coating repairs		Visual/Holiday detection	no holidays	once for demonstration	each defect

11.2 Acceptance criteria

Pipes which 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 which fail to meet the requirements of Table 9 shall be re-tested. If the retest also fails to meet the requirements in Table 9, two pipes in the coating sequence prior to the pipe which 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 excluding the pipe that failed. If the tests fail, the total production back to the last passed test shall be blocked and further testing shall be initiated by the applicator.

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 which have been subjected to destructive testing shall be repaired.

The defect area shall not exceed 10 cm² and the total number of defects to be repaired shall not exceed 1 defect/m² of the coated surface 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 in accordance with the APS. Repair procedures and materials shall be compatible with the applied coating.

13 Marking

The following markings shall be placed on the coating:

— applicator's name or code;

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— ISO Standard designation and year of publication (ISO 21809-1, xxxx);coating thickness class;

— coating thickness;

— design temperature for class C only;

EXAMPLE Applicator - ISO 21809-1 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, transportation and storage in the coating area

Coated pipe shall be handled in a manner that minimises damage to the pipe and coating. If specified, the applicator shall submit details of the handling and storage procedures including loading requirements if the applicator is responsible for loading.

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Annex A
(normative)

Inspection of thickness

A.1 General

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

A.2 Instrument

A magnetic, electromagnetic or ultrasonic measuring instrument with ± 10 % accuracy shall be used. The instrument shall be calibrated for the range of coating thickness to be measured.

A.3 Procedure

The instrument reading shall be verified on a steel pipe surface using a calibrated shim of the same thickness range as the coating. The surface profile and cleanliness of the steel pipe surface shall be representative of the production pipe.

NOTE For measuring thickness of more than 1 mm, the surface profile of the pipe is not relevant.

On each pipe to be tested, a total of 12 single readings shall be carried out in accordance with ISO 2808. If any of these 12 readings is below the minimum coating thickness, an additional 4 readings shall be taken around this area. The average of the additional 4 readings and the initial reading shall be higher than the minimum thickness.

The measurements shall be taken at points distributed along 4 equally spaced longitudinal lines at the pipe length with three equally circumferential lines on the middle of the pipe at a distance of at least 300 mm from the end of the coating.

For submerged arc welded pipes, 4 thickness measurements shall be undertaken on the weld area.

A.4 Results

The arithmetic average of all measurements shall be calculated and recorded.

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Annex B
(normative)

Holiday detection test

B.1 General

This test consists of identifying coating discontinuities by electrical conductivity.

B.2 Equipment

The equipment shall consist of an adjustable high-voltage holiday detector, equipped with a sound and light signal and a scanning electrode in the form of a metal brush or conductive rubber conforming to the shape of the pipe

B.3 Procedure

The instrument shall be connected to the pipe, completing the circuit, switched on, and the scanning electrode passed over the surface of the coating to be 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 diameter will be detected.

The voltage shall be set at 10 kV/mm based on the minimum coating thickness of coating and shall not exceed 25 kV.

B.4 Results

The number of holidays detected shall be recorded.

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Annex C
(normative)

Peel strength test

C.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.

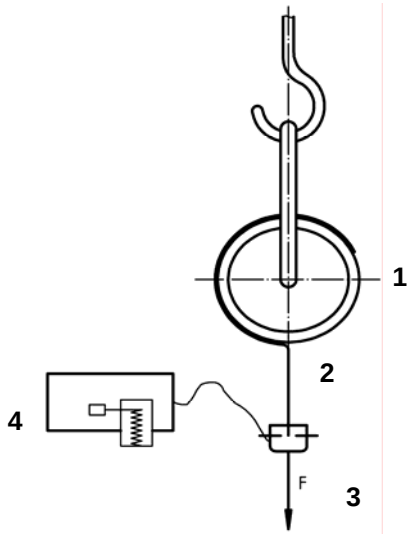
C.2 Equipment

The equipment shall consist of:

— tensile testing machine with which it is possible to record the peel force and which operates at a rate of pull of 10 mm/min, as in Figure C.1 or C.2;

— cutting tool (e. g. knife).

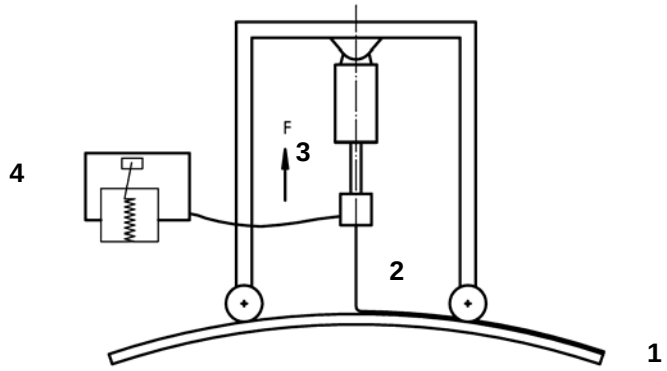
— Suggested equipment as in Figure C.1 or C.2



Key
1. pipe ring
2. coating strip
3. peel force
4. registration unit

Figure C.1 — Testing equipment (small diameters)

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Key
1. coated pipe surface
2. coating strip
3. peel force
4. registration unit

Figure C.2 – Testing equipment (large diameters)

C.3 Procedure

C.3.1 General

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

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.

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

A pipe ring 200 mm in length shall be cut from the pipe. A sample coating strip shall be cut in the circumferential direction of the pipe ring measuring a minimum of 160 mm in length and 20 mm to 50 mm in width.

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

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

A sample coating strip shall be cut in the circumferential direction of the pipe measuring a minimum of 160 mm in length and 20 mm to 50 mm in width. 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.4 Results

The peel force data for 140 mm of peeling shall be divided into seven intervals of 20 mm and the first and last intervals discarded. The peel strength shall be calculated from remaining data.

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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 point in the 100 mm length shall be more than 30 % below the specified value.

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Annex D
(normative)

Thermal analysis of epoxy powder and cured coating film (FBE)

D.1. General

The uncured epoxy powder and the cured coating film shall be characterized by Differential Scanning Calorimetry (DSC).

The general procedure and definitions are given in ISO 11357-1. General handling and calibration shall be performed as in ISO 11357-1 unless stated differently in this Annex.

D.2. Equipment

The equipment shall consist of the following:

— differential scanning calorimeter (DSC) with cooling accessory;

— balance accurate to 0,1 mg;

— sample encapsulating press;

— aluminium pans with covers;

— dry gas supply of N_2, analytical grade.

D.3. Procedures and measurement for epoxy powder

D.3.1. Measurement

The measurement shall consist of the following steps:

a) Heat the sample from 25 °C +/- 5 °C to 70 °C +/- 5 °C at a rate of 20 °C/min, then immediately cool the sample to 25 °C +/- 5 °C

b) Heat the sample from 25 °C ± 5 °C to 275 °C ± 5 °C at a rate of 20 °C/min, then immediately cool the sample to 25 °C ± 5 °C

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 immediately cool the sample to 25 °C ± 5 °C

NOTE For certain epoxy powders different heating cycles might be necessary according to the instructions of the manufacturer.

D.3.2. Evaluation of results

D.3.2.1. Glass transition temperature (T_g)

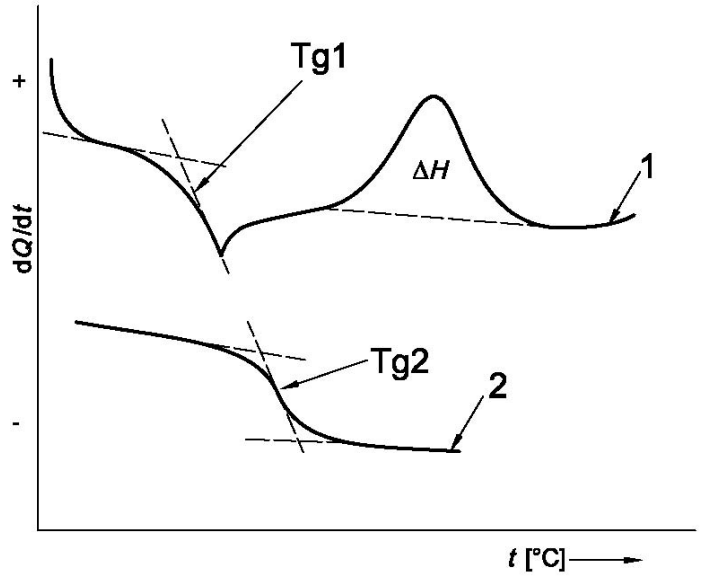
T_g is calculated at the point of inflection (Figure D.1).

By evaluating run (b) the T_g of the uncured powder is obtained = T_{g1}. By evaluating run (c) the T_g of the cured material is obtained = T_{g2}.

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D.3.2.2. Heat of reaction of epoxy powder

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.4. Procedures and measurement for coating sample

D.4.1. General

A representative sample of the cured film is taken.

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

Place the sample and the reference sample in the DSC cell and purge with dry, N_2 gas.

D.4.2. Measurement

The measurement shall consist of the following steps:

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.

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.

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.

NOTE For certain epoxy powders different heating cycles might be necessary according to instructions of the epoxy-powder manufacturer.

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

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D.4.3. Evaluation of results

D.4.3.1. Glass transition temperature (T_g)

T_g is calculated in the same way as for the epoxy powder for run 3 (b) and 3 (c); Figure D.2

where

T_{g3} is T_g of run D.4.2(b) (°C)

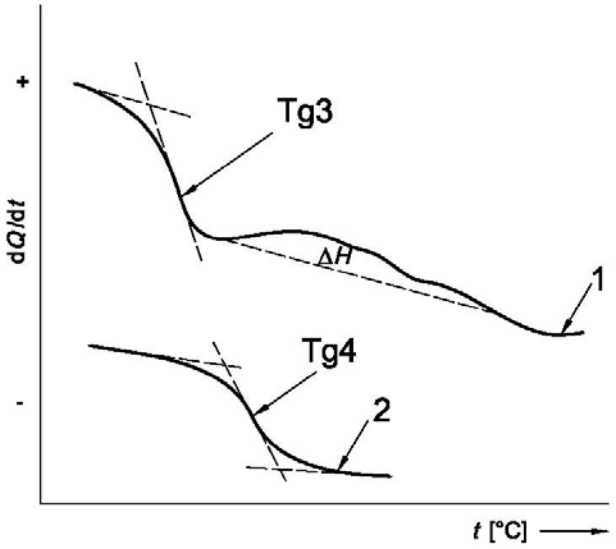
T_{g4} is T_g of run D.4.2(c) (°C)

For coatings, determine the change in T_g value using the following formula:

$$\Delta T_g = T_{g4} - T_{g3} \quad (5)$$

where

ΔT_g is change in T_g value (°C)



Key
1 run (b)
2 run (c)

Figure D.2 — Examples of thermal scans on coating

D.4.3.2. Residual heat of reaction of cured coating

The exothermic heat of reaction ($\Delta H1$) is obtained by integrating the exothermic peak of the DSC-scan according run D4.2.b.

NOTE In a fully cured coating film no residual heat of reaction is visible.

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The degree of conversion can be calculated by:

$$C = \frac{\Delta H - \Delta H_1}{\Delta H \times 100} \quad (6)$$

where

C is degree of conversion (%)

ΔH is exothermic heat of reaction of the powder (run D 3.1.b)

ΔH_1 is exothermic heat of reaction of the coating film (run D 4.2.b)

D.5. Results

The following information shall be recorded:

— type of material and batch number;

— date of testing;

— type of DSC equipment;

— for the epoxy-powder T_{g1}, T_{g2} , ΔH;

— for the cured coating film T_{g3}, T_{g4}, ΔT_g, ΔH_1 and C.

Testing of production coating requires pipe number or identification.

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Annex E
(normative)

Impact test

E.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.

E.2 Drop weight equipment

The equipment shall consist of a drop weight testing machine comprising:

— 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:

  a) support and levelling devices (for example, two spirit levels for the horizontal plane and a plumb line for the vertical plane);

  b) graduated rod which makes it possible to determine the drop height to an accuracy of 5 mm.

  NOTE 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, where 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 to the required height ; the mass of this assembly shall be 1 kg ± 0,005 kg;

— sufficient number of additional 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 with an accuracy of ± 5 g.

E.3 Procedure

The test shall be carried out at a temperature of 23 °C ± 3 °C. If provisions have been made to perform this test outside this temperature range, the method described shall be adapted, after agreement, if necessary, between the coater 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 (see Annex C) 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. Ten impacts shall be carried out allowing the weight corresponding to the energy laid down in 8.7 and Table 3 to fall from a height of 1 m. The points of impact shall be selected and shall avoid any protruding welds. Furthermore, the distance from the

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points of impact to the end of the pipe shall be at least 1,5 D (D is the outside diameter of the pipe and at least 50 mm apart between the axes of the impacts. The holiday detection test shall then be undertaken at each location (see Annex C).

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

E.4 Results

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

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Annex F
(normative)

Indentation test

F.1General

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

F.2 Indentation test equipment

The equipment shall consist of:

— inside ventilated oven or a circulated bath thermostatically controlled to ± 2 °C;

— penetrometer comprising:

a) cylindrical indenter with a diameter of 1,8 mm (cross-sectional area 2,5 mm^2) on the top of which is mounted a weight. The assembly (indenter plus weight) shall produce a force of 25 N;

b) dial gauge;

c) other measurement system accurate to ± 0,01 mm.

F.3 Procedure

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

The test sample, held within the penetrometer assembly, shall be placed 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.

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

F.4 Results

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

The average of the 3 samples shall be recorded.

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Annex G
(normative)

UV ageing test and thermal ageing test

G.1 UV Ageing

G.1.1 General

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

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

G.1.2 Equipment

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

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

The size of the sample shall be large enough to make 10 test specimens of type 1B, 5A or 5B (see Figures 1 and A.2 in ISO 527-2).

The test specimen shall be exposed under the following conditions:

— artificial weathering (see method A in Table 1 of ISO 4892-2) ;

— black standard temperature BST (65 °C ± 3 °C);

— relative humidity 65 % ± 5 %;

— spray cycle : 18 min ± 0,5 min spray / 102 min ± 0,5 min dry ;

— total radiant energy 7 GJ/ m² for classes A and B; 5 GJ/m² for class C;

— continuous exposure.

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

MFR_1 shall be calculated as the arithmetic mean of the three results.

The melt flow rate MFR_O shall be calculated in accordance with ISO 1133 on a test sample of identical shape which has not been exposed.

G.1.5 Results

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The variation of the melt flow rate (ΔMFR) after exposure shall be expressed as a percentage using equation (G.3):

$$\Delta MFR = \frac{MFR_1 - MFR_0}{MFR_0} x\,100 \quad (\%) \qquad (G.3)$$

where

MFR_O is the melt flow rate measured before exposure

MFR_1 is the melt flow rate measured after exposure

G.2 Thermal ageing

G.2.1 General

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

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

G.2.2 Equipment

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

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

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

Table G.1 – Test Temperature and Duration

Coating Class	Test Duration (h)	Test Temperature (°C)
A	2 400	100 °C ± 2 °C
B	4 800	100 °C ± 2 °C
C Operating temperature (*T*) ≤ 80°C	240	150 °C
C Operating temperature (*T*) > 80°C	(*T*-70) x 24	150 °C

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

MFR_1 shall be calculated as the arithmetic mean of the three results. The melt flow rate MFR_O shall be calculated in accordance with ISO 1133 on a test sample of identical shape which has not been placed in the oven.

G.2.5 Results

The variation of the melt flow rate (ΔMFR) shall be expressed as a percentage using equation (G.3).

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Annex H
(normative)

Cathodic disbondment test

H.1 General

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

The test shall be performed on test specimen taken from the coated components previously subjected to holiday-detection (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 Electrical source

The source for the voltage and the current shall consist of a stabilised d.c. power unit. The current source shall be capable of supplying 20 mA to each test area simultaneously. A cathodic polarisation potential of –1 500 mV to a saturated calomel reference electrode (equivalent to UH = - 1 260 mV where UH is the potential of the standard hydrogen electrode) shall be maintained (see also H.2.3).

H.2.2 Electrolytic cell

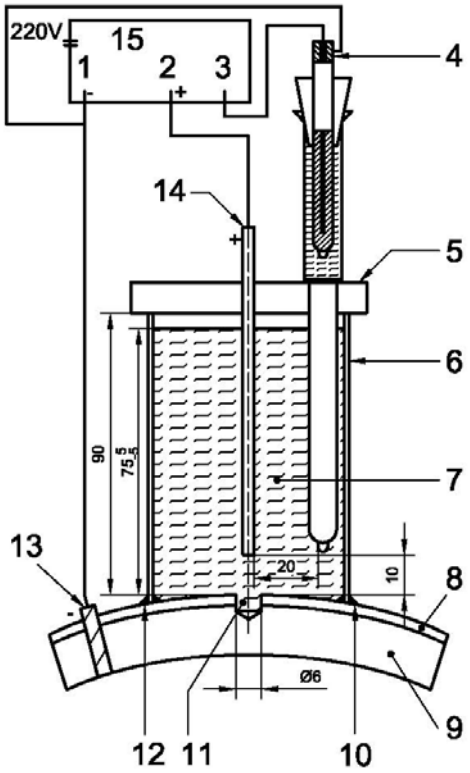
For tests, typical test cell configurations are shown in Figure H.1 for large diameter components and in Figure H.2 for small diameter components.

The electrolytic cell shall comprise of:

— rigid plastic pipe of an internal diameter of minimum 50 mm. The height shall be such that the total volume of the electrolyte is equal or greater than 150 ml with a minimum height of the electrolyte of 70 mm;

— rigid plastic cover in which holes shall be drilled to allow the passage of the electrodes and any other measuring instruments deemed necessary, and to allow the escape of hydrogen.

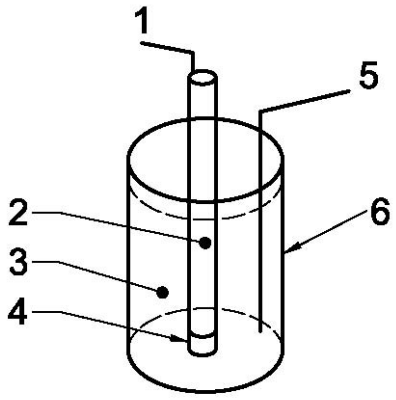
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Key

1 working electrode
2 electrode (anode)
3 electrode (reference)
4 reference electrode
5 Plastic cover
6 Plastic pipe (minimum internal ∅ 50 mm)
7 electrolyte ≥ 150 ml
8 coating
9 steel test piece
10 sealing material
11 artificial defect
12 sealing material
13 electrode (cathode)
14 Platinium electrode ∅ 0,8 mm to 1,0 mm (anode)
15 rectified d.c. source

Figure H.1 — Electrolytic cell for large diameter pipe



Key

1 to negative lead (–)
2 holiday
3 electrolyte
4 end cap
5 to positive lead (+)
6 beaker

Figure H.2 — Electrolytic cell for small diameter pipe

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H.2.3 Electrodes

H.2.3.1 Reference electrode

The reference electrode suitable to produce a potential (see H.2.1) shall be 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 (Figure H.1).

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

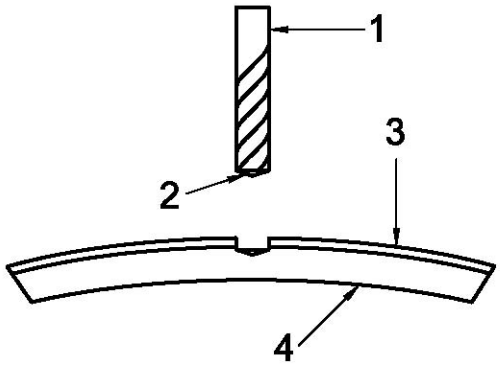
H.2.3.2 Auxiliary electrode (anode)

The auxiliary electrode shall consist of an inert material, e. g. platinum wire of 0,8 mm to 1,0 mm diameter. It shall be immersed in the electrolyte (approximately within 10 mm over the coating defect).

H.2.3.3 Working electrode (cathode)

The working electrode is 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 H.3 and Figure H.3).

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



Key

1 fluted and mill face mill ∅ 6 mm
2 conic end
3 coating
4 steel

Figure H.3 — Drilling of artificial defect

H.2.4 Electrolyte

The electrolyte shall consist of a solution of 3 % NaCl concentration in distilled or deionised water. The solution shall be made from annular grade sodium chloride.

The pH at the 23 °C ± 2 °C during the test shall be in the range of 6 to 9.

The height of the electrolyte in the cell shall be 75 mm ± 5 mm.

H.2.5 Heating equipment

Suitable heating equipment shall be used 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.

H.3 Sampling

The test samples shall be cold cut from a coated component and shall have a minimum size of 80 mm x 80 mm, unless the test is performed on the body of the coated component.

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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 (see Annex B).

A 6 mm diameter hole (see Figure H.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.

H.4 Procedure

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

The cell shall be filled with the NaCl electrolyte (see H.2.4).

A negative cathodic potential shall be applied between the reference and working electrodes (see Figure H.1 or H.2), with an accuracy of ± 10 mV. If a saturated calomel electrode is used the potential shall be - 1 500 mV.

The test shall be performed for the test period required. The level of the electrolyte shall be readjusted with distilled or deionised water, if necessary.

H.5 Investigation procedure

After the test, the cell with the electrolyte shall be removed. The test sample shall be rinsed with water, cooled and dried. The examination must take place within one hour of removing the samples from the test equipment.

After drying, the area of the coating subjected to the test shall be examined 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 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, peel away slowly a radial section of coating continuing until firm adhesion is encountered. As loss of adhesion is not always obvious, carefully examine the substrate for sign of residual coatings which indicates that disbonding has not occurred.

— Repeat with each radial segment.

NOTE Different methodologies for removing the polyolefin can be agreed.

H.6 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.

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Annex I
(normative)

Flexibility test

I.1 General

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

I.2 Equipment

The equipment shall consist of the following:

— hydraulic press;

— bending mandrels with fixed radii;

— freezer;

— strain gauges, if applicable.

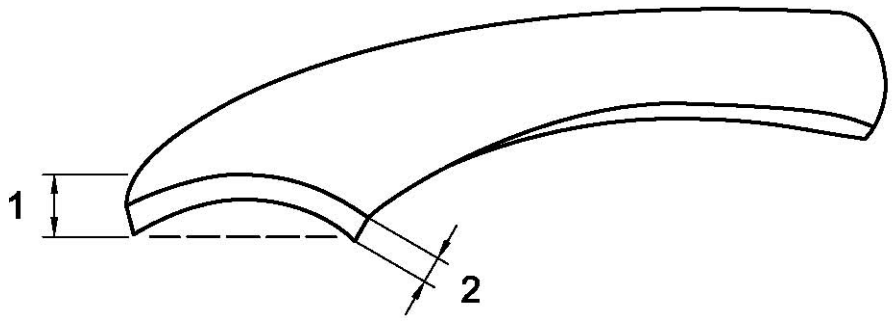
I.3 Specimen

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.

I.4 Procedure

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 0 °C, and hold it at that temperature for a minimum of 1 h.

Determine the effective sample thickness t 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 I.1.



Key
1 effective thickness
2 actual thickness

Figure I.1 — Effective strap thickness diagram

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Determine the mandrel radius that corresponds to an angle of deflection of 2° per pipe diameter length by using the following formula:

$$R = 28{,}15 \times t \qquad \text{(I.1)}$$

where

R is the mandrel radius (mm);

t is the effective sample thickness (mm).

Bend the test sample over a mandrel whose radius is not larger than that determined in accordance with the formula (I.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.

Warm the bent test sample to 20 °C ± 5 °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.

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

I.5 Test result

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

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Annex J
(normative)

Gel time of the epoxy powder

J.1 General

This procedure shall be used to assess the gel time of the epoxy powder used in the three layer coating.

J.2 Equipment

The equipment shall consist of the following:

— hotplate controllable to within 3 °C;

— metal plate placed on top of the hotplate;

— stopwatch or electric timing device capable of measuring 0.1 s intervals; and

— draw down tool

J.3 Procedure

J.3.1 Conduct three tests and average the results.

J.3.2 Heat and maintain the temperature of the metal plate surface that will be in contact with the powder at a temperature of 205 °C ± 3 °C.

J.3.3 Cover the bottom 25 mm of the draw down tool with epoxy powder.

J.3.4 In a smooth motion, deposit and draw the epoxy powder across the metal plate 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 Start the timing device and deposition of epoxy powder on the metal plate surface simultaneously.

J.3.6 Applying light pressure on the draw-down tool, repeatedly draw the edge of the tool through the melted epoxy powder. Stop the timing device when the tool rides up on the gelled epoxy powder and no longer contacts the metal plate.

J.4 Results

The following information shall be reported:

— epoxy powder batch number;

— date of testing; and

— gel time in seconds.

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Annex K
(normative)

Moisture content of the epoxy powder – mass loss

K.1 Procedure A (manual procedure)

K.1.1 Equipment

The equipment shall consist of the following:

a) oven controllable to within 3 °C;

b) balance accurate to 0,001 g;

c) desiccator;

d) sample container.

K.1.2 Procedures

Weigh the sample container to the nearest 0,001 g. Transfer approximately 10 g of epoxy powder into the sample container. Weigh the sample container and epoxy powder to the nearest 0,001 g.

Place the sample container with the epoxy powder into the oven for a maximum of 2 h at 105 °C ± 3 °C.

Remove the container from the oven, and place it in the desiccators to cool. Weigh the sample container when it has cooled to 20 °C ± 3 °C, and then return it to the desiccators; repeat at intervals of 60 min ± 10 min until two consecutive mass determinations are within 0,001 g.

Calculate the percentage of moisture using the following formula:

$$M = \frac{B - C}{B - A} \times 100 \qquad (1)$$

where

M is the percentage of moisture (%)

B is the initial mass of sample container and epoxy powder (g)

C is the final mass of sample container and epoxy powder (g)

A is the mass of sample container (g)

K.2 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.2.1 Results

The following information shall be recorded:

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a) epoxy powder batch number;

b) date of testing;

c) procedure used;

d) percentage of moisture content.

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Annex L
(informative)

Procedure qualification trial (PQT), Inspection and testing plan (ITP) and Daily log

L.1 General

If specified, a PQT shall be carried out in accordance with Clause 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 ITP shall be produced in accordance with Clause L.3

L.2 Procedure qualification trial (PQT)

All items in the APS from surface preparation to preparation of pipe ends shall be performed and inspected/tested. The specific process parameters to be used during the PQT shall be selected 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).

The PQT shall utilise pipes of the same dimensions as those to be coated during production. A minimum of 5 pipes shall be coated and tested; including 3 pipes with full 3-layer polyolefin coating system applied. Requirements for the frequency of testing and inspection, methods and acceptance criteria are contained in Table 9.

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 to be applied for production shall be highlighted in the report.

L.3 Inspection and testing plan (ITP) and daily log

The ITP shall identify all coating application and quality control activities in a consecutive order, including recordings of the process parameters that are relevant to each activity. Reference shall be made to the applicable standards or applicator's procedures, frequencies and acceptance criteria that shall apply for each process control and inspection/testing activity, and actions in case of non-conformities.

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

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