# Multi-layer coating comprising a three-layer polyethylene coating and a polypropylene- or polyamide-based top coat – Requirements and tests

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**Multi-layer coating comprising a three-layer polyethylene coating and a polypropylene- or polyamide-based top coat – Requirements and tests**

## Preface

This Mill Standard has been issued for multi-layer coating systems used in conjunction with non-conventional pipe-laying techniques. Depending on project requirements, the polyethylene (PE) corrosion protection coating is supplemented with a top coat of polypropylene (PP) or polyamide (PA) for protection against mechanical damage. The system design does not provide for adhesion between the polyethylene coating and the polyamide top coat. The necessary shear strength is ensured by the rough surface of the polyethylene coating which provides for mechanical interlocking of the layers.

# Multi-layer coating comprising a three-layer polyethylene coating and a polypropylene- or polyamide-based top coat – Requirements and tests

**Previous editions**

None

# Multi-layer coating comprising a three-layer polyethylene coating and a polypropylene- or polyamide-based top coat – Requirements and tests

## 1 Scope

This Mill Standard defines the requirements on multi-layer coating systems used in conjunction with non-conventional pipe-laying projects. The coatings can be applied to longitudinally welded and spiral-weld as well as seamless steel tube & pipe and fittings used in the construction of pipelines for liquid and gaseous media. They can be used at service temperatures of between -40 °C and +80 °C.

## 2 Normative references

The following normatively referenced documents are indispensable in the application of this Mill Standard. In the case of dated references, only the edition cited applies. With undated references, the latest edition of the referenced document (including any amendments) applies.

DIN 30670, Polyethylene coatings of steel pipes and fittings — Requirements and tests

DIN EN 10204, Metallic products — Types of inspection certificates

DIN EN ISO 306, Plastics — Thermoplastic materials — Determination of the Vicat softening temperature (VST)

DIN EN ISO 307, Plastics — Polyamides — Determination of the viscosity number

DIN EN ISO 527-1, Plastics — Determination of tensile properties — Part 1: General principles

DIN EN ISO 527-2, Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion masses

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

DIN EN ISO 1133-1, Plastics — Determination of the mass-flow rate (MFR) and the melt volume flow rate (MVR) of thermoplastics — Part 1: Standard method

DIN ISO 1183-1 Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method

DIN EN ISO 2808, Paints and varnishes — Determination of film thickness

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

DIN EN ISO 9001, Quality management systems — Requirements

DIN EN ISO 11357-1, Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles

DIN EN ISO 11357-3, Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization

DIN EN ISO 11357-6, Plastics — Differential scanning calorimetry (DSC) — Part 6: Determination of oxidation induction time

E DIN EN ISO 15512, Plastics — Determination of water content

DIN EN ISO 21809-1, Petroleum and natural gas industries - External coatings for buried or submerged pipelines used in pipeline transportation systems - Part 1: Polyolefin coatings

## 3 Terminology

The terms and definitions used in this Mill Standard are in accordance with DIN 30670, DIN 30678 and DIN EN ISO 21809 1.

## 4 Symbols and abbreviations

| | |
|---|---|
| $A_0$ | Elongation at break in % |
| W | Impact energy in J |
| $L_0$, $L_1$ | Length values for the determination of elongation (same unit, i.e. m or mm) |
| DSC | Differential Scanning Calorimetry |
| MFR | Melt flow rate in g/(10 min) |
| OIT | Oxidation induction time to DIN EN ISO 11357-6 |
| S | Surface area in m² |

## 5 Multi-layer coating

The corrosion protection coating consists of three layers: an epoxy resin primer, an adhesive agent, and an extruded polyethylene coating to DIN 30670 or DIN EN ISO 21809-1. The epoxy resin primer is applied as a powder. The adhesive agent can either be applied as a powder or extruded over the primer. The polyethylene layer is applied by either blown film extrusion or the extrusion wrapping process.
Coarse PE granules, which are sprayed and fused onto the freshly extruded polyethylene coating while the pipe string is still hot, give the coating a rough surface (Rough Coat). In addition, a top coat of polypropylene or polyamide is extruded over the three-layer corrosion protection coating.

## 6 Ordering information

### 6.1 Mandatory information

The following information is mandatory:

- Number and dimensions of the pipes or fittings to be coated;
- Reference to this Mill Standard;
- Design temperature;
- Technical delivery conditions for the polyethylene coating;
- Type of top coat (polyamide or polypropylene).

### 6.2 Ordering options

The following information may be requested as optional information:

- Deviating layer thicknesses;

— Test certificate, e.g. test certificate 2.2 to DIN EN 10204 or other test certificates to DIN EN 10204;

— Deviating test frequencies.

# 7 Coating application

## 7.1 General

The coating materials shall be selected by the coating manufacturer, because they can judge best which materials are optimally suited in conjunction with their equipment and processes to meet the requirements specified in the applicable standards. While the three-layer corrosion protection coating calls for due consideration of the epoxy resin primer, the adhesive agent and the extruded polyethylene layer, the multi-layer coating system dealt with in the present Mill Standard features an additional top coat of polypropylene or polyamide top coat whose requirements are described in this Mill Standard. The color of the top coat should be clearly distinguishable from that of the polyethylene coating.

## 7.2 Coating structure

### 7.2.1 Polyethylene coating

The three-layer polyethylene coating is subject to the requirements of the agreed-upon technical delivery conditions.

### 7.2.2 Mechanical protection

Table 1 below provides an overview of the technical data specified in the delivery documents and the technical datasheets for the polypropylene or polyamide top coat. The minimum layer thickness of the mechanical protection is 2 mm. For particularly high mechanical loads, a greater thickness can be agreed upon. The layer thickness check is carried out as specified in 0.

Table 1 — Mandatory information in the product manufacturer's datasheets and delivery documents

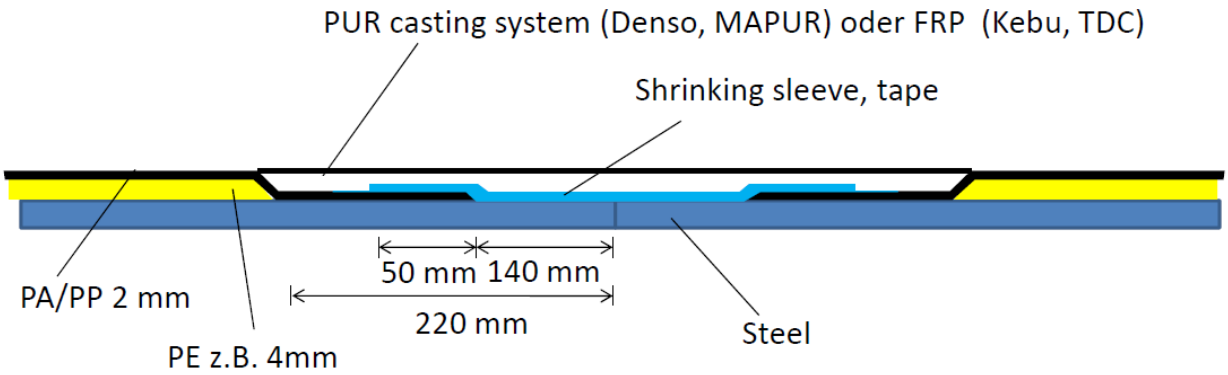
| Material | Property | Test basis | Mill certificate: Certificate of conformity (COC) or batch certificate (BC) | Technical datasheets |
|---|---|---|---|---|
| **General information** | Material designation | - | K, C | yes |
| | Product manufacturer | - | K, C | yes |
| | Storage conditions | - | - | yes |
| | Batch number | - | C | - |
| **Polypropylene** | OIT (220 °C, min 30 | DIN EN ISO 11357-6 | K, C | yes |

**Multi-layer coating comprising a three-layer polyethylene coating and a polypropylene- or polyamide-based top coat – Requirements and tests**

| Material | Property | Test basis | Mill certificate: Certificate of conformity (COC) or batch certificate (BC) | Technical datasheets |
|---|---|---|---|---|
| | min) | | | |
| | MFR (230 °C/2.16 kg) | DIN EN ISO 1133-1 | C | yes |
| | Yield stress | DIN EN ISO 527 | - | yes |
| | Elongation at break | DIN EN ISO 527 | - | yes |
| | Ultimate stress | DIN EN ISO 527 | - | yes |
| | Softening point A/50 (9.8 N) | DIN EN ISO 306 | - | yes |
| | Shore hardness D | DIN EN ISO 868 | - | yes |
| | Density | DIN EN ISO 1183-1 | C | yes |
| **Polyamide** | Melting point DSC ($\Delta T_{g2}$) | DIN EN ISO 11357-1/3 | C | yes |
| | Yield stress | DIN EN ISO 527 | - | yes |
| | Elongation at break | DIN EN ISO 527 | - | yes |
| | Ultimate stress | DIN EN ISO 527 | - | yes |
| | Shore hardness D | DIN EN ISO 868 | - | yes |
| | Density | DIN EN ISO 1183-1 | C | yes |
| | Water content | E DIN EN ISO 15512 | C | yes |

### 7.2.3 Pipe end design

The requirements for the polyethylene coating at the pipe ends are defined as part of the agreed-upon technical delivery conditions. The polypropylene or polyamide top coat on the polyethylene coating is cut back over a length of at least 50 mm. Beveling of the polypropylene or polyamide top coat is not required.




## 8 Requirements

### 8.1 General

The requirements on coatings in accordance with this Mill Standard apply at the time of delivery and serve for quality assurance of the coating materials used and for monitoring of the coating process.

### 8.2 Material properties

The requirements on the three-layer coating for corrosion protection based on polyethylene are defined in the agreed-upon delivery conditions.

The properties of the top coats and the related tests are compiled in Tables 2 and 3. MFR measurements on the polypropylene and DSC measurements on the polyamide are performed on granules as part of the incoming inspection. The melt index (MFR) and melting point (DSC) shall be compared with the data given in the delivery documents.

Table 2 — Properties of polypropylene granules and polypropylene top coats

| Property | Specified requirement | Test according to: | Notes |
|---|---|---|---|
| MFR<br>Incoming inspection | | Appendix A | Incoming inspection of raw material (polypropylene granules). The MFR must match the specifications given in the delivery documents. |
| Layer thickness | min. 2 mm | Appendix C | |
| Shore hardness D | min. 60 | DIN EN ISO 868 | Shore hardness is determined on the pipe coating in accordance with ISO 868. |
| Elongation at break (23 °C ± 2 °C) | min. 400 % | Appendix D | |
| Low-temperature impact resistance (0 °C ± 2 °C) | min. 5 J/mm | Appendix E | 25 kV test: there must be no crack formation. |
| Indentation resistance (23 °C ± 2 °C) | max. 0.1 mm | Appendix F | |
| UV resistance | Δ MFR ± 35 % | Appendix G | Testing of MFR |
| Thermal ageing resistance | Δ MFR ± 35 % | Appendix H | Testing of MFR |
| Shear strength | min. 100 N/cm | Appendix I | |

Table 3 — Properties of polyamide granules and polyamide top coats

| Property | Specified requirements | Test according to: | Notes |
|---|---|---|---|
| Melting point<br>DSC ($\Delta T_{g2}$)<br>Incoming inspection | | DIN EN ISO 11357-1/3<br>Appendix B | Incoming inspection of raw material (polyamide granules). The melting point must match the specifications given in the delivery documents |
| Layer thickness | min. 2 mm | Appendix C | |
| Shore hardness D | min. 65 | DIN EN ISO 868 | Shore hardness is determined on the pipe coating in accordance with ISO 868. |
| Elongation at break (23 °C ± 2 °C) | min. 200 % | Appendix D | |

| Property | Specified requirements | Test according to: | Notes |
|---|---|---|---|
| Low-temperature impact resistance (-40 °C ± 2 °C) | min. 10 J/mm | Appendix E | 25 kV test. There must be no crack formation. |
| Indentation resistance (23 °C ± 2 °C) | max. 0.075 mm | Appendix F | |
| UV resistance | min 150 % | Appendix G | Testing of elongation at break |
| Thermal ageing resistance | min 150 % | Appendix H | Testing of elongation at break |
| Shear strength | min. 100 N/cm | Appendix I | |

### 8.3 Production monitoring

#### 8.3.1 Polyethylene coating

Before applying the polyamide top coat over the polyethylene coating, it must be ensured that the latter complies with the customer's order specifications. Where applicable, the surface roughness (rough coat) shall be subjected to visual inspection.

#### 8.3.2 Mechanical protection

The relevant parameters of the extrusion line and process and of the appearance of the top coat must be regularly checked. The top coat must be uniform in color and surface appearance. It must also be free from pores and other defects that affect the quality of the coating system.

## 9 Testing

### 9.1 General

The tests for the determination of the coating and coating material properties are described in the Appendices to this Mill Standard. A distinction must be made between production tests and system tests.

In the system test, the material properties and functional requirements of the coating components are verified and documented in line with this Mill Standard. Coating producers who comply with the requirements of DIN EN ISO 9001 or an equivalent management system can carry out the system test themselves.

Production monitoring is ensured by the coating producer or an authorized material testing institute. If the requirements specified in this Mill Standard are not met during these in-process inspections, the test shall be repeated on twice the number of samples from the same batch. If these also fail to meet the requirements, the whole batch shall be rejected.

## 9.2 Test certificates

A test certificate 2.2 to DIN EN 10204:2005-01 shall be issued as verification that all the specified requirements have been met. Other certificate types are subject to special agreement.

## 9.3 Testing type and frequency

The testing type and frequency for the three-layer polyethylene coating is defined in the agreed-upon technical delivery conditions. The testing type and frequency for the top coats are listed in Table 4. Other test procedures are subject to special agreement.

Table 4 — Testing type and frequency for the top coats

| Testing type | Frequency | In-process test | System test |
|---|---|---|---|
| Melting point DSC ($\Delta T_{g2}$)[a] | 1/batch | X | X (1 sample) |
| MFR[b] | 1/ batch | X | — |
| Layer thickness | 3/shift | X | — |
| Shore hardness | 1/ batch | X | X (5 samples) |
| Elongation at break | 1/ batch | X | X (5 samples) |
| Low-temperature impact resistance | 1/ batch | X | X (1 sample) |
| Indentation resistance | 1/ batch | X | X (3 samples) |
| Shear strength | — | — | X (1 sample) |
| UV resistance | — | — | X (1 set of samples) |
| Thermal ageing resistance | — | — | X (1 set of samples) |

[a] for polyamide only
[b] for polypropylene only

## 10 Marking

For identification purposes, a label is affixed to each pipe. It shall contain the following information:

— Name of pipe manufacturer;

— Reference to the applicable pipe standard;

— Reference to the standard applicable to the corrosion protection coating;

— Reference to this Mill Standard.

# Appendix A
(normative)

# Measurement of melt flow rate (MFR)

## A.1 General

The incoming polypropylen granulate is inspected by MFR measurements as described in DIN EN ISO 1133-1.

## A.2 Test equipment

The test equipment shall comply with DIN EN ISO 1133-1.

## A.3 Process description

### A.3.1 Sample preparation

The measurement is performed on granulate taken from the delivered batch of polypropylene

### A.3.2 Measuring procedure

The measurement shall be performed in accordance with DIN EN ISO 1133-1, Method A, at 230 °C (cf. Table 2).

### A.3.3 Evaluation

Evaluation shall be performed in accordance with DIN EN ISO 1133-1, 8.5.

# Appendix B
(normative)

# DSC measurements

## B.1 General

The incoming polyamide granulate is inspected by differential scanning calorimetry (DSC) thermal analysis as described in DIN EN ISO 11357-1 and -3. The measurements shall be performed in accordance with these standards, unless otherwise specified in the present Appendix.

## B.2 Test equipment

The test equipment consists of the following components:

— DSC tester with cooling device;

— Aluminum sample carrier with lid;

— If necessary, nitrogen gas supply, dry, analytical grade.

## B.3 Process description

### B.3.1 Measuring procedure

The measurement, performed on a granule taken from the delivered batch of polyamide, comprises the following steps:

— Heating of the sample from 25 °C to 230 °C at a rate of 20 K/min, followed by rapid cooling to 25 °C and an isothermal phase of 1.5 minutes;

— Heating of the sample from 25 °C to 230 °C at a rate of 20 K/min.

### B.3.2 Evaluation

The first heating step serves primarily to remove residual moisture from the sample. In the second step, the melting point of the polyamide / polypropylene is determined as shown in Figure B.1.

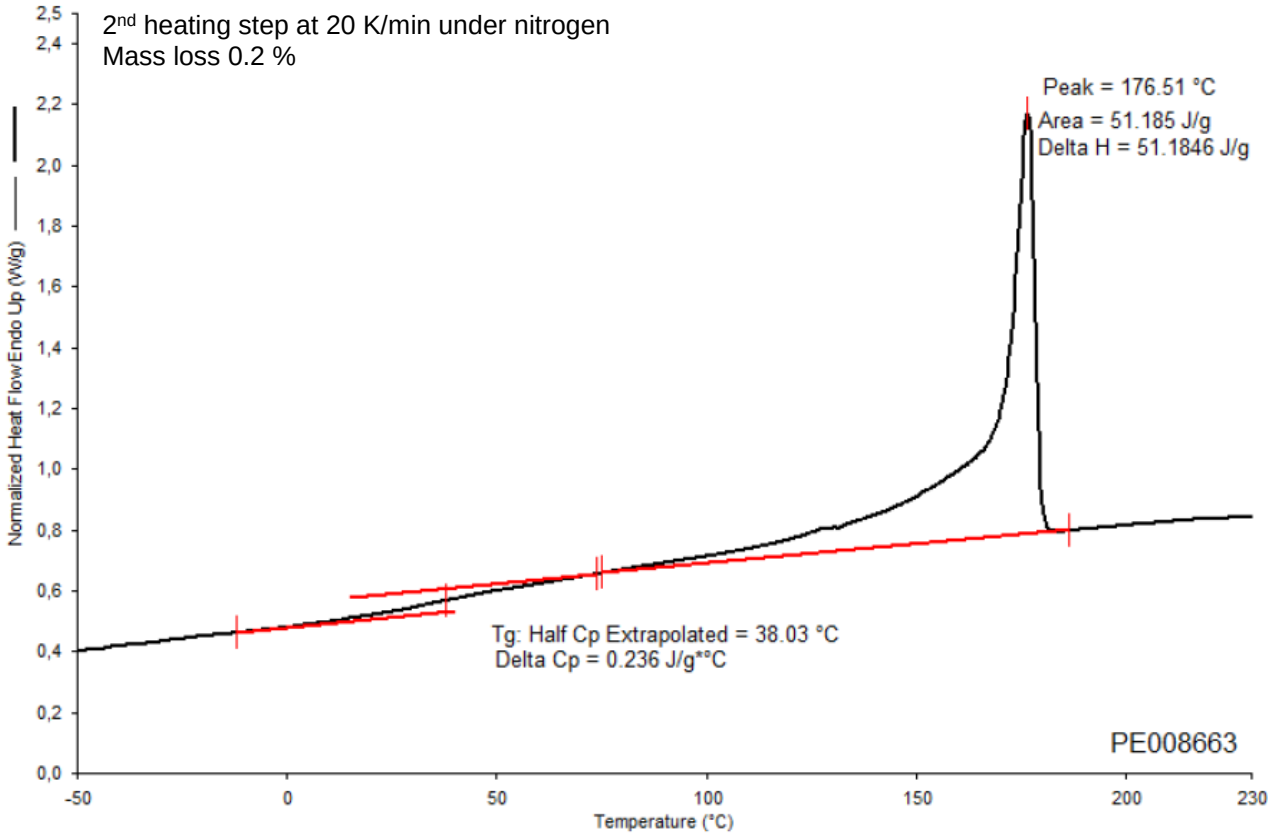



Figure B.1: Determination of the melting point during the second heating step

# Appendix C
(normative)

# Thickness check

The thickness of the polypropylene or polyamide top coat is equal to the difference between the total coating thickness and the thickness of the corrosion protection coating. Alternatively, it can be measured directly when cutting it back at the pipe ends. The thickness of the corrosion protection coating is measured at the pipe ends.

Procedures for coating thickness checks are described in DIN EN ISO 2808. The thickness of the complete coating system and of the corrosion protection coating is checked using a nondestructive method in accordance with DIN EN ISO 2808.

This nondestructive check is performed with the aid of an electro-magnetic, eddy current or ultrasonic measuring device with an accuracy of ±10 %. The device must be designed and calibrated for the thickness range concerned. To determine the total coating thickness, a minimum of twelve measurements must be performed on each pipe to be examined.

# Appendix D
(normative)

# Elongation at break

## D.1 General

Elongation at break is tested in accordance with DIN EN ISO 527-1. The test samples are produced and selected in accordance with DIN EN ISO 527-2.

## D.2 Test equipment

The test shall be performed using a tension device that allows the recording of a force-path diagram.

## D.3 Process description

### D.3.1 Sample preparation

The test shall be performed on samples taken from the top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating

### D.3.2 Measuring procedure

Elongation at break is tested on sample types 1B, 5A or 5B (cf. Figs. 1 and A.2, DIN EN ISO 527-2) at a temperature of 23 °C ± 2 °C.

The following test speeds according to Table D.1 below shall be observed.

Table D.1 — Test speeds

| **Sample type to DIN EN ISO 527-2** | **Test speed to DIN EN ISO 527-1** mm/min |
|---|---|
| 1B | 50 |
| 5A | 25 |
| 5B | 10 |

### D.3.3 Evaluation

Elongation at break ($A_0$ in %) is determined according to the following equation:

$$A_0 = \frac{L_1 - L_0}{L_0} \cdot 100$$

where

*$A_0$ = Elongation at break in %;*

*$L_0$ = Initial length (distance) between measuring points;*

*$L_1$ = Distance between measuring points at sample break.*

The test result is calculated as the arithmetic mean of five samples.

# Appendix E
(normative)

# Low-temperature impact resistance

## E.1 General

Impact resistance is determined with the aid of a weight dropped with a defined impact force (cf. Tables 2 and 3). The polypropylene top coat is tested at 0 °C, the polyamide top coat at -40 °C.

## E.2 Test equipment

The impact resistance test is carried out using a device with a drop weight that impinges vertically onto the polyamide top coat removed from the pipe. The drop weight device shall be guided to ensure minimum friction. The accuracy of the test should not be impaired by any air build-up. The front face of the drop weight is part of the surface of a sphere with a diameter of 25 mm. The drop height of the weight is about 1 m.

## E.3 Process description

### E.3.1 Sample preparation

The test shall be performed on samples from the top coat. After a circumferential incision, the top coat can be easily separated from the polyethylene coating.

### E.3.2 Measuring procedure and evaluation

The sample is subjected to five impacts. The distance between two impact points should be at least 30 mm.

Using a high-voltage test device, the sample is then checked for defects. Punctures are not permissible.

# Appendix F
(normative)

# Indentation resistance

## F.1 General

This test consists of measuring the indentation of a punch in the coating under defined conditions in terms of temperature and the load applied.

## F.2 Test equipment

The test equipment comprises a 250-g metal rod with an additional weight. A metal pin with a flat face shall be fitted at the center of the lower end of the rod. The diameter of the flat face shall be 1.80 mm ± 0.05 mm, corresponding to an indentation area of 2.5 mm². The total mass shall be 2.5 kg, resulting in a surface pressure of 10 N/mm². Furthermore, a penetrometer with a dial gauge having an accuracy of 0.1 mm is required. The test shall be conducted at room temperature.

## F.3 Process description

### F.3.1 Sample preparation

The test shall be performed on samples from the top coat. After a circumferential incision, the top coat can be easily separated from the polyethylene coating

### F.3.2 Measuring procedure and evaluation

After conditioning the test sample at test temperature (RT) for about 1 h, the punch (without the additional weight) shall be lowered slowly and carefully onto the sample surface, and the zero value set at the penetrometer within 5 s. Then the additional weight shall be attached to the punch and, after a loading time of 24 h, the depth of penetration read from the penetrometer.

# Appendix G
(normative)

# UV resistance

## G.1 General

To test their resistance to UV ageing, samples taken from the polyamide or polypropylene top coat are exposed to continuous irradiation under xenon light at defined temperature and humidity conditions. Ageing-related changes are assessed on the basis of the MFR variations according to Appendix A and variations in elongation at break according to Appendix D.

## G.2 Test equipment

The test equipment shall enable the samples to be aged under the specified test conditions and shall consist of an irradiation chamber fitted with a xenon arc lamp (cf. DIN EN ISO 4892-2, Method A).

## G.3 Process description

### G.3.1 Sample preparation

The test shall be performed on samples taken from the top coat. After a circumferential incision, the top coat can be easily separated from the polyethylene coating.

### G.3.2 Measuring procedure

Measurements shall be carried out in accordance with DIN EN ISO 4892-2, Method A, Exposure Cycle no. 1. The requirements specified in DIN EN ISO 21809-1 shall be observed. Samples for determining the MFR according to Appendix A (PP) or elongation at break according to Appendix D (PA) shall be taken both before exposure to irradiation (initial value) with 5 GJ/m² (PP) or 7 GJ/m² (PA) and afterwards.

### G.3.3 Evaluation

The variations of the arithmetic mean values shall be documented and evaluated after exposure to 5 GJ/m² (PP) and 7 GJ/m² (PA).

# Appendix H
(normative)

# Thermal ageing resistance

## H.1 General

To test their resistance to thermal ageing, samples taken from the polyamide or polypropylene top coat are exposed to dry heat in a in a circulating-air oven. Ageing-related changes are assessed on the basis of the MFR variations according to Appendix A and variations in elongation at break according to Appendix D.

## H.2 Test equipment

The test shall be performed on samples that have been aged in a thermostatically controlled circulating-air oven capable of maintaining a test temperature constant within ± 2 °C.

## H.3 Process description

### H.3.1 Sample preparation

The test shall be performed on samples from the polyamide or polypropylene top coat. After a circumferential incision, the top coat can be easily separated from the polyethylene coating.

### H.3.2 Measuring procedure

The samples are exposed to a temperature of 100 °C for 100 days (2,400 h) in the case of PP and 200 days (4,800 h) in the case of PA. Before the start of exposure (initial value) and after every 800 h of exposure to heat, PP samples are taken for determining the MFR according to Appendix, and PA samples for measuring elongation at break according to Appendix D.

### H.3.3 Evaluation

The variations of the arithmetic mean values shall be documented. The final evaluation shall be performed after the exposure time specified above for each type of coating.

# Appendix I
(normative)

# Shear strength

## I.1 General

Shear strength shall be tested according to the corresponding measurement on cement mortar coatings as per DVGW Worksheet GW 340. This test determines the shear strength by removing the top coat from a suitably prepared pipe section under defined load conditions at room temperature.

## I.2 Test equipment

The test equipment consists of a press capable of removing the polyamide or polypropylene top coat by exerting a load that rises at a uniform rate of 80 (±10) N/s throughout the duration of the test.

## I.3 Process description

### I.3.1 Sample preparation

The test shall be performed on a pipe section on which the top coat has been cut back by about 2 cm. The width of the top coat to be removed should be at least 25 mm. A second pipe section is prepared so that its inside diameter can be pushed over the PE corrosion protection coating. The test set-up is shown in Figure I.1 below.

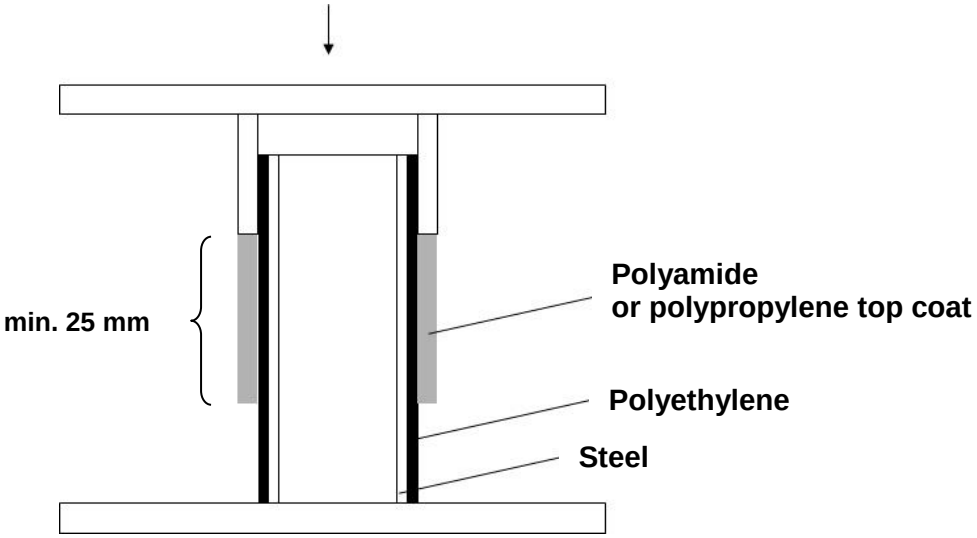



Figure I.1 – Test set-up for shear strength testing

### I.3.2 Measuring procedure and evaluation

In the press used for the shear strength test, the top coat is removed from the first pipe section with the aid of the second pipe section. The maximum force measured before removal of the top coat, related to the top coat's surface area, is recorded as shear strength in N/cm².