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

**Part 2:
Single layer fusion-bonded epoxy
coatings**

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

*Partie 2: Revêtements monocouche à base de résine époxydique
appliquée par fusion*



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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 2, *Pipeline transportation systems*.

This second edition cancels and replaces the first edition (ISO 21809-2:2007), which has been technically revised. It also includes the Technical corrigendum ISO 21809-2:2007/Cor.1:2008.

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: Single layer fusion-bonded epoxy coatings*
- *Part 3: Field joint coatings*
- *Part 4: Polyethylene coatings (2-layer PE)*
- *Part 5: External concrete coatings*

The following parts are under preparation:

- *Part 6: Multilayer fusion-bonded epoxy coatings (FBE)*

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 if there is innovative or developing technology. If 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 2: Single layer fusion-bonded epoxy coatings

1 Scope

This part of ISO 21809 specifies the requirements for qualification, application, testing and handling of materials for plant application of single layer fusion-bonded epoxy (FBE) coatings applied externally for the corrosion protection of bare steel pipe for use in pipeline transportation systems for the petroleum and natural gas industries as defined in ISO 13623.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2815, *Paints and varnishes — Buchholz indentation test*

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

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

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

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

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

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

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

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

ISO 10474:2013, *Steel and steel products — Inspection documents*

ISO 21809-2:2014(E)

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

ISO 11126 (all parts), *Preparation of steel substrates before application of paints and related products — Specifications for non-metallic blast-cleaning abrasives*

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

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

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

ISO 80000-1:2009, *Quantities and units — Part 1: General*

EN 10204:2004,¹⁾ *Metallic products — Types of inspection documents*

AS 3894.6,²⁾ *Site testing of protective coatings — Determination of residual contaminants*

ASTM D4060,³⁾ *Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser*

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

SSPC-AB 1,⁴⁾ *Mineral and Slag Abrasives*

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

SSPC-AB 3, *Ferrous Metallic Abrasive*

SSPC-SP 1, *Solvent cleaning*

3 Terms and definitions

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

3.1 application procedure specification

APS

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

3.2

applicator

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

3.3

batch

quantity of epoxy powder produced using the same formulation and raw materials of the same source during a continuous production run of not more than 8 h

3.4

batch certificate

certificate of analysis issued by the manufacturer

1) European Committee for Standardization, Management Centre, Avenue Marnix 17, B-1000, Brussels, Belgium.

2) Standards Australia, GPO Box 476, Sydney, NSW 2001, Australia.

3) American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA.

4) SSPC: The Society for Protective Coatings, 40 24th Street, 6th Floor, Pittsburg, PA 15222-4656, USA.

3.5**cutback**

length of pipe left uncoated at each end for joining purposes

3.6**glass transition**

reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one

[SOURCE: ISO 11357-2:2013, 3.1]

3.7**glass transition temperature** T_g

characteristic value of the temperature range over which the glass transition takes place

Note 1 to entry: Note to entry: The assigned glass transition temperature, T_g , can vary, depending on the specific property and on the method and conditions selected to measure it.

[SOURCE: ISO 11357-2:2013, 3.2]

3.8**holiday**

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

3.9**laboratory-coated test specimen**

specimen taken from a laboratory-prepared panel

3.10**manufacturer**

company responsible for the manufacture of coating material(s)

3.11**manufacturer's specification**

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

3.12**powder shipment**

amount of powder transported in one container

3.13**procedure qualification trial****PQT**

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

3.14**purchaser**

company responsible for providing the product order requirements

3.15**test report**

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

3.16**test ring**

sample taken from production-coated pipe

4 Symbols and abbreviated terms

4.1 Symbols

C	percentage conversion of FBE coating, expressed as a percentage
d	thickness, expressed in millimetres
ΔH	exothermic heat of reaction, expressed in joule per grams
M	mass, expressed in grams
R	mandrel radius, expressed in millimetres
T_g	glass transition temperature, expressed in degrees Celsius
ΔT_g	variation of the glass transition temperature, expressed in degrees Celsius
w_{ep}	mass fraction of the epoxy powder retained on a sieve, expressed as a percentage of total sample
w_m	mass fraction of moisture, expressed as a percentage

4.2 Abbreviated terms

d.c.	direct current
DSC	differential scanning calorimetry
FBE	fusion-bonded epoxy
HRC	Rockwell C scale hardness
ID	inner diameter
ITP	inspection and testing plan
NPS	nominal pipe size
OD	outer diameter
ppd	per pipe diameter

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 80000-1:2009, Annex B, Rule A.

NOTE For the purposes of this provision, the rounding method of ASTM E29 is equivalent to ISO 80000-1:2009, Annex B, Rule A.

5.2 Compliance to standard

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

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

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

6 Information supplied by the purchaser

6.1 General information

The purchase order shall include the following information:

- a) reference to this part of ISO 21809 and year of publication, i.e. ISO 21809-2:2014;
- b) pipe quantity, outside diameter, minimum wall thickness, minimum, maximum and nominal length, steel grade;
- c) bare pipe standard or specification designation, e.g. ISO 3183;
- d) minimum thickness and maximum permissible thickness of the coating;
- e) cutback and tolerances for both ends of pipe;
- f) minimum and maximum pipeline design temperatures (°C);
- g) type of certificate of compliance;
- h) pipe line installation methods for offshore (e.g. reel lay, S-lay, J-lay).

6.2 Additional information

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

- a) additional surface treatments;
- b) plant and process inspection by the purchaser;
- c) increased test ring length;
- d) test ring location;
- e) test frequency for additional test rings;
- f) additional markings;
- g) handling procedures;
- h) storage procedures;
- i) waiver of test reports;
- j) maximum allowable preheating temperature;
- k) applicator qualification requirements;
- l) other special requirements;

- m) pipe tracking and traceability of pipes to coating materials;
- n) permissible number coating repairs if different from [Clause 11](#);
- o) documentation and schedule for supply of documents;
- p) purchaser approval of APS;
- q) inspection and testing plan and/or daily log;
- r) inspection of incoming pipe;
- s) pipe end protection;
- t) surface pretreatments if any;
- u) PQT requirements;
- v) protection against adverse weather conditions during storage.

7 Coating materials

7.1 Epoxy powder

7.1.1 General

The applicator shall use epoxy powder that is

- a) certified by the manufacturer to be in accordance with the requirements of [7.1.2](#) and [8.1](#), and compatible with the requirements of [9.2](#), [9.3](#) and [9.4](#);
- b) identified by the manufacturer on each package with the following:
 - manufacturer's name;
 - product number/description;
 - product temperature range (maximum and minimum);
 - mass of material;
 - batch number/manufacturing identification number;
 - location of manufacture;
 - temperature requirements for transportation and storage;
 - year, month and day of manufacture;
 - expiry date, and
- c) handled, transported, and stored in accordance with the manufacturer's recommendations.

7.1.2 Properties

As a minimum, each batch of epoxy powder shall be tested by the manufacturer in accordance with the requirements of [Table 1](#). Test results shall be reported in accordance with ISO 10474 and a batch certificate with the test results shall be provided by the manufacturer to the applicator.

Table 1 — Minimum requirements for epoxy powder

Property	Unit	Test method	Requirements
Cure time	s	Clause A.2	Within the manufacturer's specification
Gel time	s	Clause A.3	Within the manufacturer's specification
Total volatile/moisture content mass fraction	%	Clause A.5	≤ 0,6 %
Particle size	%	Clause A.6	Maximum retained on 150 µm and 250 µm sieves within the manufacturer's specification
Density	g/cm ³	Clause A.7	Within the manufacturer's specification
Thermal characteristics	T_{g1} (°C) T_{g2} (°C) ΔH (J/g)	Clause A.8	Within the manufacturer's specification

7.1.3 Packaging

The powder shall be contained in packaging that is labelled to identify the items specified in [7.1.1 b](#)).

7.2 Repair materials

The applicator shall use repair materials that are certified by the powder manufacturer to be compatible with the epoxy powder.

Repair material batches shall be identified by the product manufacturer with the following:

- manufacturer's name;
- product number/description;
- product temperature range (maximum and minimum);
- mass of material;
- batch number/manufacturing identification number;
- location of manufacture;
- temperature requirements for transportation and storage;
- year, month and day of manufacture;
- expiry date.

Repair materials shall be handled, transported, and stored in accordance with the material manufacturer's recommendations.

8 Coating qualification

8.1 Qualification by manufacturer

8.1.1 Epoxy powder

8.1.1.1 General

The manufacturer shall qualify the epoxy powder in accordance with 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 the tests in accordance with the requirements of [8.1.1](#). Coatings designed for up to 95°C shall be qualified by the manufacturer through laboratory coated test specimens or plant applied coating for each of the applicable tests. The test results shall meet the acceptance criteria in [Table 2](#).

For coatings designed for services above 95°C, in addition to the requirements in [Table 2](#), the tests for flexibility, impact resistance, cathodic disbondment, hot water adhesion, tabor abrasion and thermal characteristics shall be repeated using samples that have been conditioned in an oven at a temperature of 5°C below T_g , for a minimum of 30 d followed by ambient temperature for 24 h before testing. Cathodic disbondment testing shall be performed using the method in [Clause A.10](#). Acceptance criteria for these tests shall be agreed between the manufacturer and the purchaser. Other temperature exposure conditions may be agreed based on the pipeline service conditions.

Laboratory test specimens shall be prepared in accordance with [8.1.1.2](#).

These test results shall be reported in accordance with ISO 10474 and shall be available to the applicator upon request.

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Table 2 — Requirements for coating qualification

Properties	Acceptance criteria	Number of test specimens	Test method
Thermal characteristics	Meets the manufacturer's specification	1	Clause A.8
Cathodic disbondment: 24 h, 65 °C ± 3 °C, -3,5 V	≤5 mm disbondment	3	Clause A.9
24 h hot-water adhesion 75 °C ± 3 °C	Rating of 1 to 2	3	Clause A.16
28 d hot-water adhesion 75 °C ± 3 °C	Rating of 1 to 3	3	Clause A.16
Cathodic disbondment: 28 d, 20 °C ± 3 °C, -1,5 V	≤8 mm disbondment	3	Clause A.9
Cathodic disbondment: 28 d, 65 °C ± 3 °C, -1,5 V	≤18 mm disbondment	3	Clause A.9
Cathodic disbondment: 28 d, max design temperature (if above 65 °C) ± 3 °C, -1,5 V	≤18 mm disbondment	3	Clause A.9
Cross-section porosity	Pass (compared with Figure A.11)	1	Clause A.12
Interface porosity	Pass (compared with Figure A.12)	1	Clause A.12
Flexibility at 0 °C for coating thickness of 350 µm to 500 µm	No cracking at 2,5° ppd	5	Clause A.13
Flexibility at -30 °C for coating thickness of 350 µm to 500 µm	No cracking at 2° ppd	3	Clause A.13
Flexibility at minimum design temperature for coating thickness of 350 µm to 500 µm	No cracking at 2° ppd	3	Clause A.13
Impact at minimum design temperature	≥2 J	3	Clause A.14
Strained coating, cathodic disbondment 28 d, 20 °C ± 3 °C, -1,5 V	No cracking	3	Clause A.15
Hardness test at maximum design temperature	Buchholz value min 80	3	ISO 2815
Taber abrasion	100 mg	3	ASTM D4060 ASTM CS-17 wheel, 1 000 cycles, 1kg mass

8.1.1.2 Preparation of the laboratory coated test specimens

Test specimens shall be mild steel and shall have dimensions in accordance with the applicable test method (see [Annex A](#)). The substrate shall be abrasively blast cleaned with steel grit, in accordance with ISO 11124-3, to provide a cleanliness in accordance with the requirements of ISO 8501-1:2007, grade Sa 2 1/2.

The surface shall have a peak-to-trough height of between 50 µm and 100 µm as measured in accordance with ISO 8503-4 (Stylus method), or ISO 8503-5 (Replica tape method).

Coating shall be applied in accordance with the product (data sheet) application guidelines.

The thickness of the coating on the completed test specimen shall be 350 µm to 500 µm. This should be measured by a calibrated coating thickness gauge verified to ± 5 % of full scale range.

8.1.2 Repair material

Qualification requirements for the repair material shall be agreed between repair material manufacturer and purchaser.

8.2 Qualification by applicator

8.2.1 General

The production coating shall be qualified by the applicator for each coating line, unless purchaser waives this requirement as indicated in 6.2. Applicator shall use coating materials qualified in accordance with the requirements of 8.1 and prepare coated samples in accordance with a documented APS.

Qualification shall be achieved by successful evaluation of pipe coated samples using the specified coating line and the coating qualification test results shall be reported in accordance with the requirements of this part of ISO 21809. The minimum requirements for plant qualification and production are given in 8.2.2.

The qualification shall be carried out in accordance with APS (see 8.2.3) and repeated in case of essential modifications of the coating line, coating materials and coating procedures.

8.2.2 Minimum requirements for plant qualification and production

The requirements for testing pipe surface preparation are given in Table 3. Requirements of plant applied coating (quality tests) are given in Table 4.

Table 3 — Requirements for inspection of surface preparation-production and plant qualification

Properties	Unit	Test method	Requirements	Frequency qualification	Frequency production
Incoming FBE powder	-	see 10.2	see 10.2	each batch	each shipment
Surface condition before blasting	-	visual inspection	free of contaminations	each pipe	each pipe
Environmental conditions	-	calculation	as determined at time of measurement	once	every 4 h
Pipe temperature before blasting	°C	thermocouple	minimum 3 °C above the dew point	once	every 4 h
Size, shape and properties of abrasive	-	visual and certification ISO 11124 (all parts) (metallic) ISO 11126 (all parts) (non metallic)	conformity to certificate, compliance to manufacturing/working procedures	once	1/day
Water soluble contamination of abrasives	µS/cm	ASTM D4940	conductivity max. 60	once	1/shift
Soluble salt after blasting	mg/m ²	Potassium ferricyanide indicator in accordance with AS 3894.6 and following conductive measurement, ISO 8502-9 and ISO 11127-6	salt content (as NaCl) max. 20	each pipe	every 4 h every 4 h if salt presence is indicated
Surface roughness of blasted surface (R _z /R _{ys})	µm	ISO 8503-4 or ISO 8503-5	50 to 100	5 pipes	every 1 h

Table 3 (continued)

Properties	Unit	Test method	Requirements	Frequency qualification	Frequency production
Visual inspection of blasted surface	-	ISO 8501-1	grade Sa 2 1/2	each pipe	each pipe
Presence of dust after dust removal	-	ISO 8502-3	max. class 2 (for both size and quantity)	5 pipes	every 1 h
Visual inspection of pipe prior to introduction to coating line	-	visual	no rust	each pipe	each pipe
Preheating temperature before coating	°C	pyrometer	compliance to APS	each pipe	each pipe

Table 4 — Requirements for plant applied coating: production and plant qualification

Properties	Acceptance Criteria	Test Method	Frequency for qualification	Frequency for production
Degree of cure DSC - ΔT_g	meets manufacturers specification	Clause A.8	first pipe	first pipe of the order and then 1/shift
Porosity	less than or equal to that illustrated in Figures A.11 and A.12	Clause A.12	first pipe	first pipe of the order and then 1/shift
Dry adhesion	rating of 1 to 2	Clause A.4	5 pipes	every 4 h
Impact	≥ 2 J	Clause A.14	3	1/shift
Flexibility at minimum design temperature for coating thickness of 350 μm to 500 μm	no cracking at 2° ppd	Clause A.13	3	1/shift
Hot-water adhesion; 24 h, 75°C \pm 3°C	rating of 1 to 2	Clause A.16	1	1/shift
Hot-water adhesion; 28 d, 75°C \pm 3°C	rating of 1 to 3	Clause A.16	1	In accordance with APS
Cathodic disbondment: 24 h, 65°C \pm 3°C - 3,5 V	≤ 5 mm disbondment	Clause A.9	1	1/shift
Interface contamination	30 % maximum	Clause A.11	1	1/shift
Cathodic disbondment: 28 d, at max. design temperature per APS (if design temperature is above 65°C) \pm 3°C - 1,4 V (Ag/AgCl reference electrode)	≤ 18 mm disbondment	Clause A.10	1	In accordance with APS
Cathodic disbondment, 28 d 65°C \pm 3°C - 1,5 V	≤ 8 mm disbondment	Clause A.9	1	In accordance with APS
Coating thickness	350 μm to 500 μm	-	In accordance with APS	In accordance with APS
Cut back	-	-	In accordance with APS	In accordance with APS
Continuity	no holidays	Subclause 10.3.2.2	each pipe	each pipe

8.2.3 Application procedure specification (APS)

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

- incoming inspection of the pipe and pipe tracking;
- data sheets for coating materials, including any materials to be used for coating repairs;
- data sheets for abrasive blast materials;
- certification, receipt, handling and storage of materials for coating and abrasive blasting;
- procedure for cleaning of all application equipment;
- preparation of steel surface including monitoring of environmental parameters, methods and tools for inspection, grinding of pipe surface defects and testing of surface preparation;
- coating application, including tools/equipment for control of process parameters essential for 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 pipe;
- any special condition for despatch of coated pipes, including protection of pipe ends;
- documentation.

The APS (see [Annex B](#)) 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 (see [Annex B](#)) including any revisions shall be approved by the purchaser prior to the start of production and any specified PQT.

The applicator shall prepare an ITP and a daily log to record quality control data in accordance with [B.3](#) or agreed similar.

8.2.4 Procedure qualification trial (PQT)

If specified by the purchaser, the APS shall be verified by a PQT in accordance with [Annex B](#).

9 Application of coating

9.1 General

The coating to be applied during production shall have been previously qualified in accordance with the requirements of [8.2](#) and should have a test report from the coating manufacturer according to [Table 2](#). The applicator receiving manufacturer's test report shall verify that it meets the requirement of this part of ISO 21809.

9.2 Surface preparation

9.2.1 Initial evaluation and surface preparation

All pipes surface shall be examined in accordance with the visual inspection section of [Table 3](#).

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-SP 1.

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

All pipes 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.

9.2.2 Abrasive blast cleaning

The abrasives used in the coating plant shall be in accordance with ISO 11124 (all parts) and sections of [Table 3](#).

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

The abrasives (including recycled materials) shall be maintained clean, dry and free from contaminants in accordance with SSPC-AB 1, SSPC-AB 2 and SSPC-AB 3 or ASTM D4940 so as not to contaminate the substrate.

The cleanliness achieved at entry to the application line shall be in accordance with ISO 8501-1:2007, grade Sa 21/2 ("very thorough blast-cleaning").

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

If grinding is required after blast cleaning, the maximum allowable area of grinding shall be 10 cm² per metre of pipe length or 0,5 % of the pipe surface area whichever is lowest. If the area of grinding required exceeds these limits, the pipe shall be re-blasted provided grinding does not reduce the wall thickness below the specified minimum wall thickness of the pipe. Pipe with wall thickness below the specified minimum shall be rejected or repaired at the purchaser's option.

Surface finish shall be monitored and recorded according to [Table 3](#).

9.2.3 Surface dust contamination

The dust level shall be measured in accordance with ISO 8502-3. The in process inspection and acceptance requirements shall be in accordance with [Table 3](#). The maximum allowable level shall be class 2 (for both size and quantity).

9.2.4 Surface cleanliness and pretreatment

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

If surface pretreatment is used for qualification, it shall be used for production.

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

If levels of soluble salts above 20 mg/m² are measured, a surface pretreatment cleaning process shall be agreed between applicator and purchaser to bring the level to below 20 mg/m².

9.3 Coating application and curing temperature

9.3.1 General

Following surface preparation, the surface being coated shall not be exposed for a length of time which could result in flash rust. Exposure time shall not exceed 4 h.

Application and curing temperatures of the external pipe surface shall be as selected by the applicator within the manufacturer's recommendation and shall not exceed 275 °C. The coating shall be allowed to cure in accordance with the qualified coating procedure before being quenched in water, to permit handling if required.

CAUTION — For some steel grades, the maximum preheat temperature may be reduced by the purchaser to prevent changes to the pipe steel properties.

The coating shall be applied in accordance with the APS and coating manufacturer's recommendation. During the application of the coating, the preheating temperature of the pipe shall be monitored and recorded using optical pyrometers or contact thermometers. Temperature measuring crayons may be used to measure temperature only if agreed upon prior to coating, shall be non-oily and shall be validated for temperature control during qualification and production of the coating system. Once the coating temperature is established, the coating temperature shall be continuously monitored and recorded for each pipe unless otherwise agreed with the purchaser.

The pipe temperature prior to and during epoxy application shall be in accordance with the APS. In process monitoring of the application temperature shall be in accordance with [Table 3](#).

9.3.2 Powder recycle

The use of recycled powder shall be permitted if the recycle system automatically and continuously conveys the powder through a sieving system not coarser than 60 mesh size. The recycled powder may be continuously blended with new powder in the delivery system. This shall be stated in the APS.

Use of reclaimed powder, for example powder that has escaped the powder coating booth or has been spilled on the floor, shall not be permitted.

9.4 Coating thickness

The minimum and maximum permissible thickness of the coating shall be as specified in the purchase order.

CAUTION — Flexibility of the coating may be reduced with increasing thickness higher than 500 µm.

The coating thickness shall be measured at three random locations along each pipe length using a coating thickness gauge that is calibrated at least once every working shift (to a maximum of 12 h) against a thickness standard that is within the range of coating thickness specified in the purchase order. Such measured thickness values shall be recorded.

If no minimum thickness is specified in the purchase order, individual measured thickness values shall be greater than 350 µm.

For a specified minimum thickness of 350 µm, if one of individual measured thickness values is outside of the requirements, the coating thickness of the affected pipes shall be measured along the pipe length at intervals not exceeding 1 m. The average of such measured values for each pipe shall be at least 350 µm and no individual value shall be less than 300 µm.

If the coated pipe does not meet the requirements of this subclause, it shall be stripped and recoated in accordance with the requirements of [11.3](#).

Coating thickness measurements: frequency and acceptance shall be in accordance with [Table 3](#) and [Table 4](#).

9.5 Cutback

The cutback and tolerances for the FBE coating on both ends of the pipe shall be as specified in the purchase order and/or in the APS.

10 Inspection and testing

10.1 General

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

Inspection and testing shall be carried out in accordance with the APS, and ITP if applicable, and shall meet the requirements of [Table 3](#), [Table 4](#), and [Table 5](#).

10.2 Testing of incoming epoxy powder

Each powder shipment shall be tested for gel time in accordance with [A.3](#), prior to its use for production coating.

If the average gel time fails to conform to the specified requirements, the gel test shall be repeated using two additional samples taken from the batch. If both retests conform to the specified gel time requirement, the powder batch shall be accepted. If both retests fail to conform to the specified requirements, the powder batch shall be rejected. If only one of the two gel time tests passes, this batch may be accepted provided it passes additional tests in accordance with [Table 5](#).

Table 5 — Incoming epoxy powder — Additional tests

Property	Unit	Test method	Requirements	Frequency
Total volatile/moisture content mass fraction	%	Clause A.5	≤ 0,6 %	1/batch
Thermal characteristics	T_{g1} (°C) T_{g2} (°C) ΔH (J/g)	Clause A.8	Within the manufacturer's specification	1/batch

Any batch of powder stored or transported outside the manufacturer's specified storage conditions shall be retested to the requirements of [8.1.1](#) prior to being used for production.

10.3 In-process and finished product testing requirements

10.3.1 General

In-process and finished product inspection and testing shall be in accordance with [Clause 9](#), [10.3.2](#) and [10.3.3](#).

10.3.2 Holiday inspection

10.3.2.1 General

The entire coated surface of each length of pipe shall be inspected with a holiday detector consisting of:

- adjustable high-voltage holiday detector with ± 10 % reading accuracy, equipped with a sound and/or light signal;

- scanning electrode in the form of a metal brush, coiled spring or phosphor bronze wire designed so that the entire coated surface is contacted during holiday inspection;
- conductors which are used to connect the pipe to an earth electrode.

For inspection, the direct current potential of the detector shall be set to 5 V for each micrometre of minimum specified coating thickness. The detector shall be calibrated at least once every working shift (to a maximum of 12 h).

The instrument (holiday detector) and earth shall be connected to the coated pipe. The scanning electrode shall be passed over the surface of the coating with a continuous relative movement not exceeding 300 mm/s.

Inspection shall be performed when the temperature of the coating is less than 100 °C or as agreed between purchaser and applicator.

10.3.2.2 Acceptance criteria

Coated pipe having holidays shall be repaired in accordance with [11.2](#), provided the number of holidays does not exceed the following:

- for pipe smaller than 355,6 mm OD, 1,0 per metre length, determined by dividing the total number of holidays by the total pipe length, expressed in metres, for the individual pipe tested; or
- for pipe 355,6 mm OD or larger, 0,7 per square metre, determined by dividing the total number of holidays by the total outside surface area, expressed in square metres, for the individual pipe tested.

If the quantity of holidays exceeds the limit as specified above, or if the area of an individual holiday is equal to or greater than 250 cm², the affected pipe shall be stripped and recoated in accordance with [11.3](#).

No holidays shall be permitted in finished coating.

10.3.3 Production test rings

10.3.3.1 Facilities

The applicator shall have facilities available for the preparation, testing and evaluation of test ring samples for the tests required in [Table 4](#).

10.3.3.2 Test rings

Test rings shall be obtained from locations at least 300 mm from the pipe end and shall be of sufficient size to provide the mandatory tests as described in [Table 4](#).

Heat-affected or cold-hardened zones shall be excluded when preparing test specimens.

10.3.3.3 Testing requirements

The minimum test frequency, for each production line, shall be one test ring per pipe diameter and specified wall thickness every working shift (to a maximum of 12 h).

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

For each test ring, the tests conducted, the number of test specimens used, the test method used and the acceptance criteria shall be as given in [Table 4](#).

10.3.3.4 Retests

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

- a) the test that fails shall be repeated using two additional test samples taken from the originally tested end of the affected pipe, or
- b) all pipe coated after the previous acceptable test and prior to the next acceptable test shall be stripped and recoated in accordance with [11.3](#).

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

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

- all pipe coated after the previous acceptable test and prior to the next acceptable test shall be stripped and recoated in accordance with [11.3](#), or
- subject to the approval of the purchaser, further retesting may be performed to determine which pipes coated after the previous acceptable test are acceptable. Pipes that are not acceptable shall be stripped and recoated in accordance with [11.3](#).

If interface contamination test results are inaccurate due to the influence of pretreatment (e.g. colour), the coating may be deemed acceptable by agreement between the purchaser and applicator.

10.4 Test results

Regardless of any waiver of test reports specified in the purchase order, the results of all tests required in [Tables 3](#) and [4](#) shall be available to the purchaser on request.

11 Repair of coated pipe

11.1 General

If required in [Clause 9](#) or [Clause 10](#), coated pipe shall be repaired in accordance with [11.2](#) or by stripping or recoating in accordance with [11.3](#), whichever is applicable.

11.2 Repair of holidays

The repair of holidays shall conform to the manufacturer's recommended procedures and the following requirements:

- a) pipelines with operating temperatures above 60 °C shall not be repaired with hot melt patch stick;
- b) pinholes and areas of 2 mm in diameter or smaller shall be repaired with the manufacturer recommended hot-melt patch stick, two-part coating or the purchaser-approved equivalent;
- c) areas greater than 2 mm in diameter and less than 250 cm² in area shall be repaired with the manufacturer recommended two-part liquid coating, or the purchaser-approved equivalent;
- d) the minimum thickness of the repaired coating shall be in accordance with [9.4](#);
- e) all repairs shall be holiday-tested in accordance with [10.3.2](#);
- f) the number of repairs per length of pipe shall be recorded.

11.3 Stripping and recoating

The pipe surface shall be cleaned by a combination of heating to a temperature not to exceed 275 °C, scraping, brushing and/or abrasive blast cleaning. All previous coating shall be removed prior to the

normal cleaning and coating process. Recoating shall be performed in accordance with [Clauses 9](#) and [10](#). The identity of each stripped pipe shall be recorded.

CAUTION — For some steel grades, the maximum stripping temperature may be reduced by the purchaser, to prevent changes to the pipe steel properties.

12 Markings

12.1 General

Coated pipe shall be marked in accordance with [12.2](#) and with any additional markings specified in the purchase order. Additional markings by the applicator shall be by agreement.

12.2 Required markings

The following markings shall be placed on the coating:

- applicator's name or mark;
- reference to this part of ISO 21809 and year of publication, i.e. ISO 21809-2:2014;
- coating manufacturer, product name;
- product temperature range (minimum and maximum);
- markings required by the applicable pipe specification or standard;
- date of coating application;
- pipe manufacturer, pipe number, pipe OD and wall thickness.

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

13 Handling and storage in the coating area

13.1 Handling

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

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

Coating that is damaged after the holiday inspection (see [10.3.2](#)) shall be repaired in accordance with [Clause 11](#).

Coated pipe shall have full encirclement separators around each length. Such separators shall be sized and located in order to prevent damage to the coating.

13.2 Storage

If specified in the purchase order, the applicator shall submit details of the facilities and the methods to be used for yard storage.

14 Test reports and inspection documents

Unless specified otherwise in the purchase order, an Inspection certificate 3.1 in accordance with ISO 10474:2013 (or type 3.1 in accordance with EN 10204:2004) shall be issued by the applicator, which provides the results from the inspection and testing of the coated pipes in accordance with the requirements of this part of ISO 21809 and any other requirements specified in the purchase order. If, however, in the purchase order, the purchaser does waive the requirement for an Inspection certificate, then the applicator should provide a declaration of compliance with the order “2.1” in accordance with ISO 10474:2013 (or a declaration of compliance with the order “type 2.1” in accordance with EN 10204:2004).

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Annex A (normative)

Test methods

A.1 General

This annex contains the test methods referenced in this part of ISO 21809.

A.2 Cure time of the epoxy powder

A.2.1 Equipment

The equipment shall consist of the following:

A.2.1.1 Hotplate, controllable to ± 3 °C.

A.2.1.2 Metal plate, approximately 25 mm \times 150 mm \times 150 mm.

A.2.1.3 Contact thermometer.

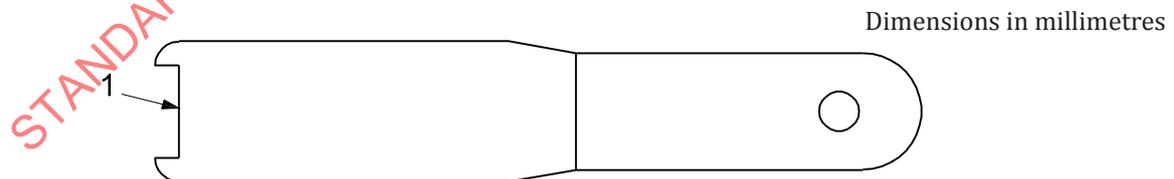
A.2.1.4 Timing device.

A.2.1.5 Draw-down tool (see [Figure A.1](#)).

A.2.1.6 Spatula.

A.2.1.7 Differential scanning calorimeter (DSC), with cooling accessory.

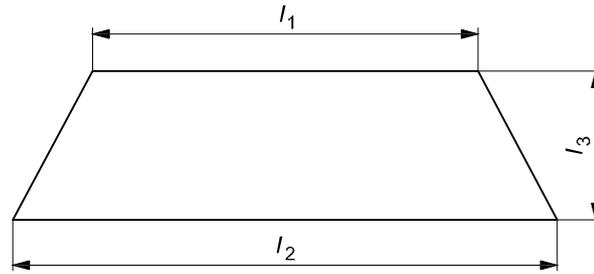
A.2.1.8 Utility knife, having a length, without the blade, of 135 mm \pm 20 mm and a one-piece metal blade, 0,65 m \pm 0,1 m thick, with an exposed cutting edge of 25 mm \pm 5 mm with other dimensions as shown in [Figure A.2](#).



Key

1 notch: (25 \pm 1) mm \times (0,9 \pm 0,1) mm

Figure A.1 — Draw-down tool



l_1 30 mm \pm 5 mm

l_2 57 mm \pm 5 mm

l_3 18 mm \pm 3 mm

Figure A.2 — Utility knife blade

A.2.2 Procedure

A.2.2.1 Heat and maintain the metal plate temperature at 232 °C \pm 3 °C.

A.2.2.2 Use the draw-down tool to deposit a film of epoxy powder on the metal plate to achieve a film thickness of 350 μ m to 500 μ m. Start the timing device at the instant of powder deposition on the hotplate surface.

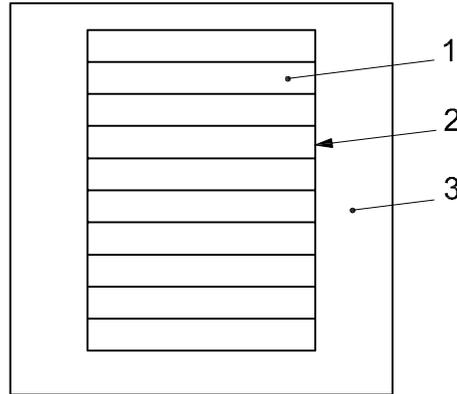
A.2.2.3 Before the film has gelled completely, scribe the film generally as shown in [Figure A.3](#), using the utility knife or spatula to produce 10 strips of coating.

A.2.2.4 Following 30 s \pm 1 s after the timing device has started, using the utility knife, remove a strip of coating and immediately quench it in cold water.

A.2.2.5 For each additional 30 s \pm 1 s of elapsed time, repeat the operation required by [A.2.2.4](#). Remove the coating strips in sequential order following the direction of film drawn, starting at the beginning of the draw.

A.2.2.6 Using the differential scanning calorimeter, determine the change in glass transition value, ΔT_g , or the percentage conversion, C , in accordance with the requirements of [A.8.4.3.1](#) or [A.8.4.3.2](#), respectively.

A.2.2.7 As specified by the manufacturer, plot time versus ΔT_g or time versus the percentage conversion.



Key

- 1 coating
- 2 division made with knife or spatula
- 3 metal plate

Figure A.3 — Cure film

A.2.3 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- time, in seconds, corresponding to a ΔT_g of 2 °C, or the time in seconds corresponding to a conversion, C , of 99 %.

A.3 Gel time of the epoxy powder

A.3.1 Equipment

The equipment shall consist of the following:

- A.3.1.1 Hotplate**, controllable to ± 3 °C.
- A.3.1.2 Metal plate**, for placing on top of the hotplate.
- A.3.1.3 Stopwatch** or **electric timing device**, capable of measuring 0,1 s intervals.
- A.3.1.4 Draw down tool** (see [Figure A.1](#)).

A.3.2 Procedure

- A.3.2.1** Conduct three tests and average the results.
- A.3.2.2** Heat and maintain the temperature of the metal plate surface that will be in contact with the powder at a temperature of 205 °C \pm 3 °C.
- A.3.2.3** Cover the bottom 25 mm of the draw-down tool with epoxy powder.

A.3.2.4 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 350 µm to 500 µm.

A.3.2.5 Start the timing device at the instant of powder deposition on the metal plate.

A.3.2.6 The draw-down tool is held at an angle of approximately 45° to the hot plate surface, in a manner that allows most of the tool's mass (weight) to be borne on the plate. Repeatedly draw the edge of the tool through the melted epoxy powder. Stop the timing device when the tool rides up on the gelled powder and no longer contacts the metal plate, as illustrated in [Figure A.4](#).

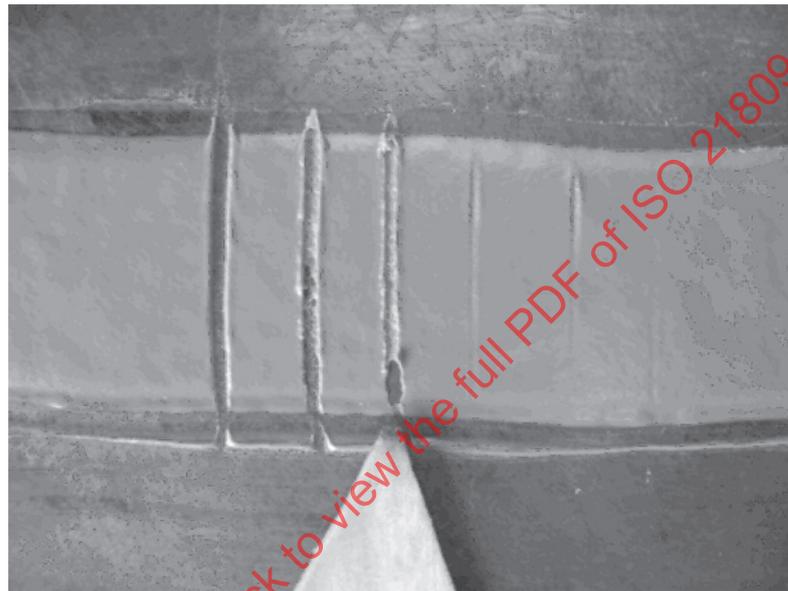


Figure A.4 — Gel time assessment

A.3.3 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- gel time, expressed in seconds.

A.4 Dry adhesion test

A.4.1 Equipment

The following equipment is required

A.4.1.1 Utility knife, see [A.2.1.8](#).

A.4.2 Test specimens

Test can be carried out *in situ* on pipe or on a laboratory sample/test ring.

A.4.3 Procedure

A.4.3.1 Inscribe a V-cut with two 20 mm lines intersecting at approximately 5 mm from their ends at 30° to 45°.

A.4.3.2 Insert the blade of the knife at the point of the V-cut, 45° to the surface, then with an upward flicking action attempt to dislodge the coating within the V. If little or no coating is removed, repeat this action within the V at least four times to confirm the integrity of the coating.

A.4.3.3 Examine the appearance of the disbonded surface and compare with the following ratings:

- Rating 1: coating is not removed cleanly at any point in the V-cut. The entire profile contains some adherent coating.
- Rating 2: coating is removed in small chips with some difficulty. Substantial coating remains adhered to the surface profile.
- Rating 3: coating is removed in chips that are slightly larger in area than the knife point inserted under the coating. Some of the coating remains adhered to the surface of the steel within the V-cut.
- Rating 4: coating is removed with no chipping of the coating and minimal resistance. No coating remains adhered to the steel within the V-cut.

A.4.4 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- adhesion rating.

Testing of production coating requires pipe number or identification.

A.5 Total volatile/moisture content of the epoxy powder — Mass loss

A.5.1 Procedure A (manual procedure)

A.5.1.1 Equipment

The equipment shall consist of the following:

A.5.1.1.1 Oven, controllable to ± 3 °C.

A.5.1.1.2 Balance, accurate to $\pm 0,001$ g.

A.5.1.1.3 Desiccator.

A.5.1.1.4 Sample container.

A.5.1.2 Procedure

A.5.1.2.1 Weigh the sample container to the nearest 0,001 g. Transfer approximately 10 g of epoxy powder into the sample container and spread evenly. Weigh the sample container and epoxy powder to the nearest 0,001 g.

A.5.1.2.2 Place the sample container with the epoxy powder into the oven for a maximum of 2 h at $105\text{ °C} \pm 3\text{ °C}$.

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

A.5.1.2.3 Calculate the percentage moisture, w_m , expressed as a percentage, using Formula (A.1):

$$w_m = \frac{M_i - M_f}{M_i - M_c} \times 100 \quad (\text{A.1})$$

where

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

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

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

A.5.2 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.

A.5.3 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- procedure used;
- percentage of moisture content.

A.6 Particle size of the epoxy powder

A.6.1 Equipment

The equipment required shall consist of the following:

A.6.1.1 Air-jet sieving unit, with vacuum cleaner attachment and 150 μm and 250 μm screens.

A.6.1.2 Balance, accurate to $\pm 0,01\text{ g}$.

A.6.2 Procedure

A.6.2.1 Weigh the sieve and one screen to the nearest 0,01 g. Place approximately 20 g of epoxy powder onto the top of the screen, and record the mass of the powder to the nearest 0,01 g.

A.6.2.2 Place the sieve into the sieving unit, cover the unit, and secure it. Operate the sieving unit for 3 min and remove the cover.

A.6.2.3 Remove the sieve, and weigh it to the nearest 0,01 g.

A.6.2.4 Calculate the percentage of epoxy powder, w_{ep} , retained on the screen as given in Formula (A.2):

$$w_{ep} = \frac{M_r - M_s}{M_p} \times 100 \quad (\text{A.2})$$

where

M_p is the initial mass of powder placed on screen, expressed in grams;

M_r is the final mass of sieve, screen and retained powder, expressed in grams;

M_s is the initial mass of sieve and screen, expressed in grams.

A.6.2.5 Repeat, using the other screen.

A.6.3 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- percentage of powder retained for each screen size.

A.7 Density of the epoxy powder

A.7.1 General

At the option of the applicator, the density of the epoxy powder shall be determined using the procedure described in either [A.7.2](#) or [A.7.3](#). The test temperature shall be $20\text{ °C} \pm 3\text{ °C}$.

A.7.2 Procedure A (manual procedure)

A.7.2.1 Equipment

The equipment shall consist of the following:

A.7.2.1.1 Balance, accurate to $\pm 0,01\text{ g}$.

A.7.2.1.2 Flask, 100 ml volumetric.

A.7.2.1.3 Mineral spirits.

A.7.2.2 Procedure

A.7.2.2.1 Weigh the flask to the nearest 0,01 g.

A.7.2.2.2 Add approximately 20 g of epoxy powder to the flask and weigh the flask plus epoxy powder to the nearest 0,01 g.

A.7.2.2.3 Add sufficient mineral spirits to cover and wet the epoxy powder. Stopper the flask, and agitate it for several minutes, ensuring that neither air pockets nor lumps of powder exist. Wash the stopper and walls of the flask with mineral spirits until they are free of powder and the flask is filled to the 100 ml level. Weigh the flask plus epoxy powder and mineral spirits to the nearest 0,01 g.

A.7.2.2.4 Empty the flask. Clean and dry the flask, add 100 ml of mineral spirits and weigh the flask plus mineral spirits to the nearest 0,01 g.

A.7.2.2.5 Calculate the density of the mineral spirits, ρ_s , expressed in grams per litre, using Formula (A.3):

$$\rho_s = \frac{(M_{fs} - M_f)}{0,1} \quad (\text{A.3})$$

where

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

M_f is the mass of flask, expressed in grams.

A.7.2.2.6 Calculate the density of the epoxy powder, ρ_p , expressed as grams per litre, using Formula (A.4):

$$\rho_p = \frac{M_{fp} - M_f}{0,1 - (M_{fps} - M_{fp}) / \rho_s} \quad (\text{A.4})$$

where

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

M_f is the mass of flask, expressed in grams;

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

ρ_s is the density of mineral spirits, expressed in grams per litre.

A.7.3 Procedure B (automatic procedure)

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

A.7.4 Results

The following information shall be recorded:

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

A.8 Thermal analysis of epoxy powder and cured coating film

A.8.1 General

Thermal analysis is used to characterize the uncured epoxy powder and the cured coating film.

The method that shall be used is 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 otherwise stated in this part of ISO 21809.

A.8.2 Equipment

The equipment shall consist of the following:

A.8.2.1 Differential scanning calorimeter (DSC), with cooling accessory.

A.8.2.2 Balance, accurate to $\pm 0,1$ mg.

A.8.2.3 Press, for sample encapsulation.

A.8.2.4 Aluminium pans, with covers.

A.8.2.5 Supply of dry N₂, analytical grade.

A.8.3 Procedure and measurement for epoxy powder

A.8.3.1 Measurement

The following heating cycles, starting with run (a) as the conditioning run, shall be performed:

- run (a): Heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to $70\text{ °C} \pm 5\text{ °C}$ at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$;
- run (b): Heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to $250\text{ °C} \pm 5\text{ °C}$ or to the maximum temperature specified by the manufacturer at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$;
- run (c): Heat the sample from $25\text{ °C} \pm 5\text{ °C}$ to expected $T_g + 40\text{ °C}$ at a rate of 20 °C/min , then immediately cool the sample to $25\text{ °C} \pm 5\text{ °C}$.

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

A.8.3.2 Evaluation of results

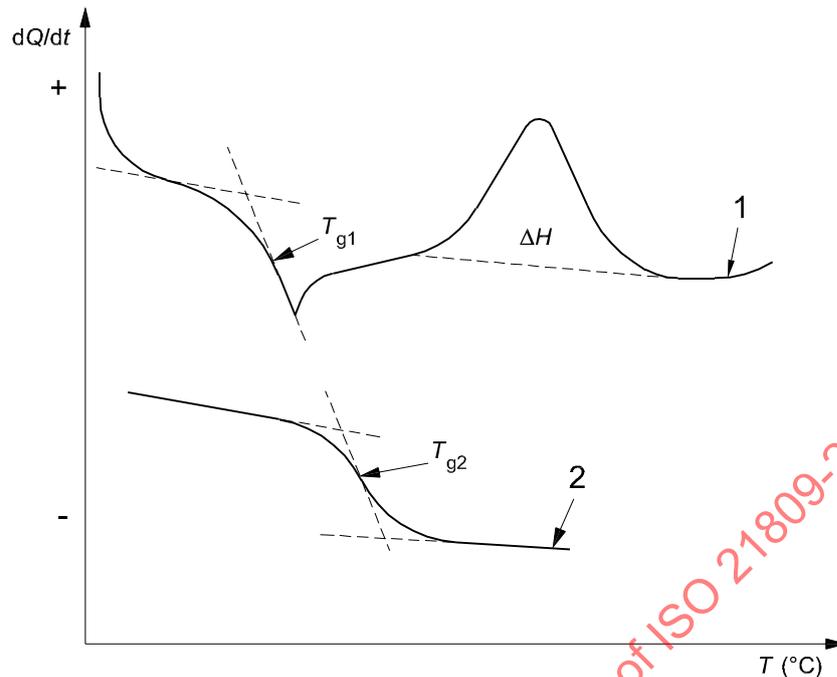
A.8.3.2.1 Glass transition temperature, T_g

The T_g of the epoxy powder is calculated at the point of inflection intersection (see [Figure A.5](#)).

T_{g1} , the glass transition temperature of the uncured powder, is obtained by evaluating run (b) and T_{g2} , the glass transition temperature of the cured material, is obtained by evaluating run (c).

A.8.3.2.2 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 A.5 — Examples of thermal scans of epoxy powder

A.8.4 Procedure and measurement for coating sample

A.8.4.1 General

A representative sample of the cured film is taken.

Weigh in $10 \text{ mg} \pm 3 \text{ mg}$ to an accuracy of $0,1 \text{ mg}$ into a preweighed pan with lid. The pan is sealed with the cover and weighed. Determine the final mass of the sample after subtracting the mass of the pan with lid.

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

A.8.4.2 Measurement

The following heating cycles, starting with run (a) as the conditioning run, shall be performed:

- run (a): heat the sample from $25 \text{ °C} \pm 5 \text{ °C}$ to $110 \text{ °C} \pm 5 \text{ °C}$ at a rate of 20 °C/min and hold for 1,5 min, then cool the sample to $25 \text{ °C} \pm 5 \text{ °C}$;
- run (b): heat the sample from $25 \text{ °C} \pm 5 \text{ °C}$ to $250 \text{ °C} \pm 5 \text{ °C}$ or to a maximum temperature recommended by the manufacturer at a rate of 20 °C/min , then cool the sample to $25 \text{ °C} \pm 5 \text{ °C}$;
- run (c) heat the sample from $25 \text{ °C} \pm 5 \text{ °C}$ to expected $T_g + 40 \text{ °C}$ at a rate of 20 °C/min , then cool the sample to $25 \text{ °C} \pm 5 \text{ °C}$.

For certain epoxy-powders, different heating cycles may be necessary according to instructions of the epoxy manufacturer.

Samples taken from pipes that have been stored or buried shall be dried in an oven for one hour at $110 \text{ °C} \pm 3 \text{ °C}$ before testing.

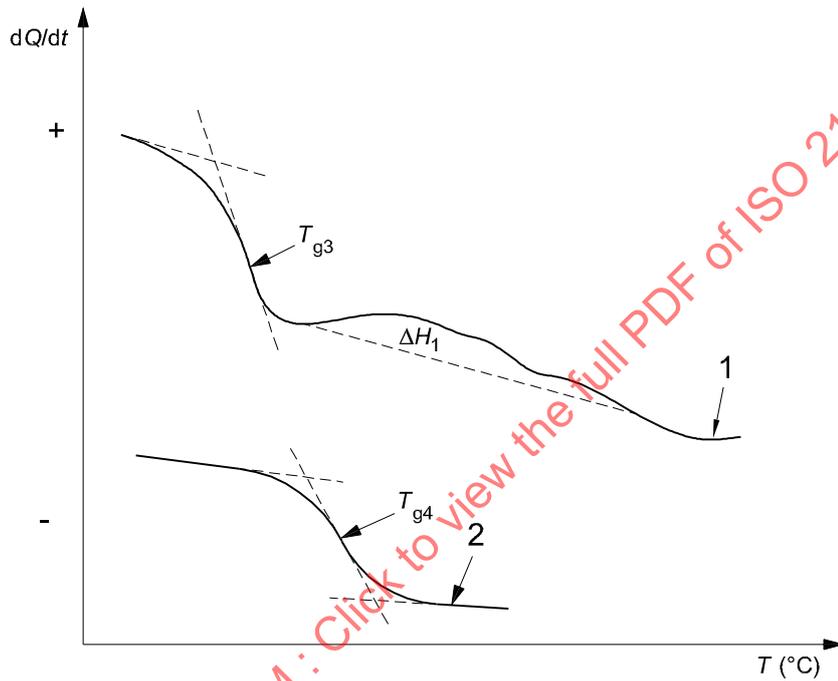
A.8.4.3 Evaluation of results

A.8.4.3.1 Glass transition temperature, T_g

The T_g for the coating is calculated as the inflection point (see [Figure A.6](#)), where T_{g3} is the glass transition temperature, expressed in degrees Celsius, of run (b) in [A.8.4.2](#) and T_{g4} is the glass transition temperature, expressed in degrees Celsius, of run (c) in [A.8.4.2](#).

For coatings, determine ΔT_g , the change in the glass transition temperature, expressed in degrees Celsius, value using Formula (A.5):

$$\Delta T_g = T_{g4} - T_{g3} \tag{A.5}$$



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

Figure A.6 — Examples of thermal scans on coating

A.8.4.3.2 Residual heat of reaction of cured coating

The exothermic heat, ΔH_1 , of reaction is obtained by integrating the exothermic peak of the DSC scan run (b) in [A.8.4.2](#).

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

The degree of conversion, C , can be calculated from Formula (A.6):

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

where

ΔH is the exothermic heat of reaction of the powder; run (b) in [A.8.3.1](#);

ΔH_1 is the exothermic heat of reaction of the powder; run (b) in [A.8.4.2](#).

A.8.5 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.

A.9 Cathodic disbondment of coatings for standard temperatures up to 95°C

A.9.1 Equipment

A.9.1.1 Procedure A — Nominal pipe diameter size 4" (101,6 mm) and larger

A.9.1.1.1 Power supply, rectified d.c., with controlled voltage output.

A.9.1.1.2 Hotplate, with a steel tray containing sand or steel grit/shot controllable to ± 3 °C, or oven, controllable to ± 3 °C.

A.9.1.1.3 Calomel reference electrode.

A.9.1.1.4 Platinum wire electrode, 0,8 mm.

A.9.1.1.5 Plastic cylinder, 75 mm \pm 3 mm ID.

A.9.1.1.6 Sodium chloride, 3 % solution in distilled water.

A.9.1.1.7 Utility knife (see [A.2.1.8](#)).

A.9.1.1.8 Drill bit, 3 mm to 6 mm (see [Figure A.7](#)). For test consistency, use the same size drill bit for the entire purchase order.

A.9.1.2 Procedure B — Nominal pipe diameter size smaller than 4" (101,6 mm)

A.9.1.2.1 Power supply, rectified d.c., with controlled voltage output.

A.9.1.2.2 Oven, controllable to $\pm 3^{\circ}\text{C}$, or a magnetic stirrer with either a hotplate tray controllable to $\pm 3^{\circ}\text{C}$ or immersion heater controllable to $\pm 3^{\circ}\text{C}$

A.9.1.2.3 Calomel reference electrode.

A.9.1.2.4 Platinum or equivalent wire electrode.

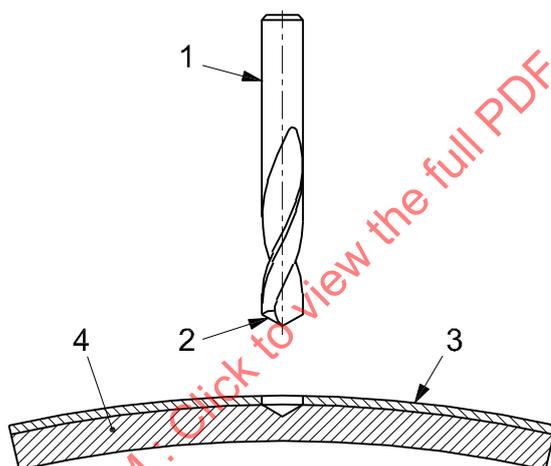
A.9.1.2.5 Non-conductive vessel, capable of withstanding test temperature ranges.

A.9.1.2.6 Alcohol thermometer or thermocouple.

A.9.1.2.7 Sodium chloride (NaCl), 3 % solution in distilled water.

A.9.1.2.8 Utility knife (see [A.2.1.8](#)).

A.9.1.2.9 Drill bit, 3 mm to 6 mm (see [Figure A.7](#)). For test consistency, use the same size drill bit for the entire purchase order.



Key

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

Figure A.7 — Production of an artificial defect

A.9.2 Test specimens

A.9.2.1 Nominal pipe diameter size 4" (101,6 mm) and larger (Procedure A)

A.9.2.1.1 For laboratory-coated test panels, specimen dimensions shall be approximately 100 mm \times 100 mm \times 7 mm.

A.9.2.1.2 For samples extracted from the pipe, specimens from test rings shall be approximately 100 mm \times 100 mm \times pipe wall thickness (reference [Figure A.8](#)).

A.9.2.2 Nominal pipe diameter size smaller than 4" (101,6 mm) (Procedure B)

A.9.2.2.1 Specimens shall have a length of approximately 20" (508 mm). Set dimensions similar to (Figure A.9).

A.9.3 Procedure

A.9.3.1 Procedure A — Nominal pipe diameter size 4" (101,6 mm) and larger

A.9.3.1.1 Use only test specimens that are confirmed to be holiday-free with a holiday detector set at a minimum of 1 800 V.

A.9.3.1.2 Drill a 3 mm to 6 mm diameter holiday in the centre of the test specimen through the coating to expose the steel substrate (see [Figure A.7](#) above).

A.9.3.1.3 Centre the plastic cylinder over the holiday and apply a sealant to form a water-resistant seal.

A.9.3.1.4 Fill the cylinder to a height of at least 70 mm with the sodium chloride solution that has been preheated to the test temperature. Mark the solution level on the cylinder.

A.9.3.1.5 Insert the electrode into the solution and connect it to the positive wire from the d.c. power supply. Attach the negative wire from the d.c. power supply to a bare spot prepared on the test specimen.

A.9.3.1.6 Apply voltage to the test specimen, and maintain constant temperature under one or more of the following test conditions, as given in [Tables 2, 3, 4](#) and [5](#):

- a) $-1,5 \text{ V} \pm 0,15 \text{ V}$, $20^\circ\text{C} \pm 3^\circ\text{C}$, for 28 d;
- b) $-3,5 \text{ V} \pm 0,15 \text{ V}$, $65^\circ\text{C} \pm 3^\circ\text{C}$, for 24 h;
- c) $-1,5 \text{ V} \pm 0,15 \text{ V}$, $65^\circ\text{C} \pm 3^\circ\text{C}$, for 28 d.

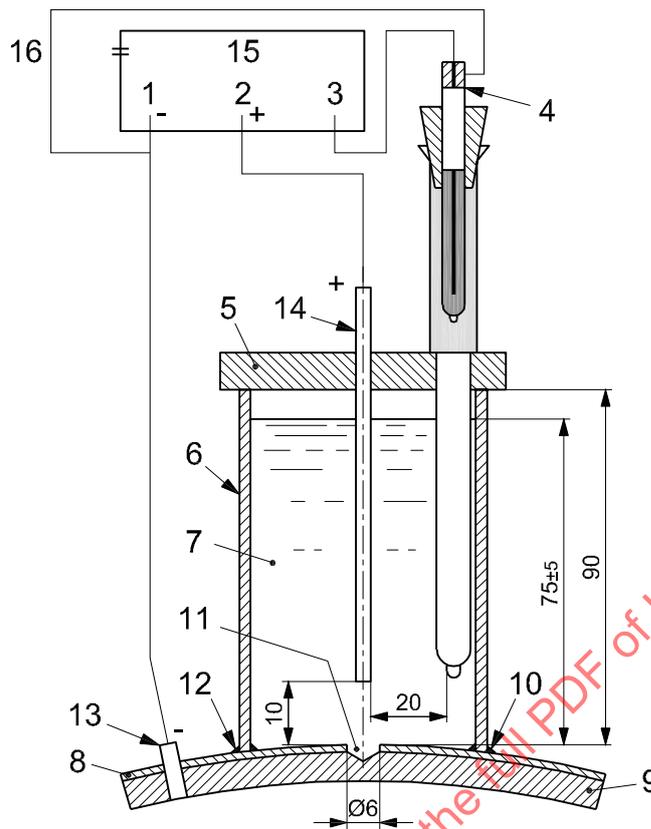
Maintain the solution level by the addition of distilled water as required (see [Figure A.8](#))

A.9.3.1.7 Upon test completion, dismantle the test cell. As soon as possible, make eight radial cuts through the coating to the substrate, using the utility knife. Such cuts shall extend at least 20 mm from the centre of the holiday.

A.9.3.1.8 Allow the specimen to air cool to $20^\circ\text{C} \pm 3^\circ\text{C}$, and evaluate the cathodic disbondment characteristics of the test specimen within 1 h of the removal from heat.

A.9.3.1.9 Insert the tip of the blade of the utility knife under the coating at the holiday. Using a levering action, chip off the coating. Continue until the coating demonstrates a definite resistance to the levering action.

A.9.3.1.10 Measure the disbonded distance from the edge of the original holiday along each radial cut, and average such measured values.



Key

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

Figure A.8 — Electrolytic cell for nominal pipe size 4" (101,6 mm) and larger diameter pipe

A.9.3.2 Procedure B — Nominal pipe size smaller than 4" (101,6mm)

A.9.3.2.1 Use only test specimens that are confirmed to be holiday-free with a holiday detector set at a minimum of 1 800 V.

A.9.3.2.2 Drill a 3 mm to 6 mm diameter holiday in the centre of the test specimen through the coating to expose the steel substrate (see [Figure A.7](#) above).

A.9.3.2.3 Fill the cylinder to a height of approximately 2" (50,8 mm) from the top of the vessel with the sodium chloride (NaCl) solution that has been preheated to the test temperature. Mark the solution level on the cylinder.

A.9.3.2.4 Attach the container lid with precut holes for a temperature measuring device, an anode wire, and the specimen(s) being tested. If more than one specimen is to be tested in the same vessel, samples shall be separated equidistant from each other and the anode.

A.9.3.2.5 Insert the temperature measuring device, the anode, and the specimen(s) into the solution.

A.9.3.2.6 Connect the anode to the positive wire from the d.c. power supply. Attach the negative wire from the d.c. power supply to a bare spot prepared on the test specimen(s). If multiple samples are being tested they can be connected in series by connecting the negative lead from one sample to the next.

A.9.3.2.7 Apply voltage to the test specimen, and maintain constant temperature under one or more of the following test conditions, as given in [Tables 2, 3, 4](#) and [5](#):

- a) $-1,5 \text{ V} \pm 0,15 \text{ V}$, $20 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$, for 28 d;
- b) $-3,5 \text{ V} \pm 0,15 \text{ V}$, $65 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$, for 24 h;
- c) $-1,5 \text{ V} \pm 0,15 \text{ V}$, $65 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$, for 28 d.

Maintain the solution level by the addition of distilled water as required (see [Figure A.9](#)).

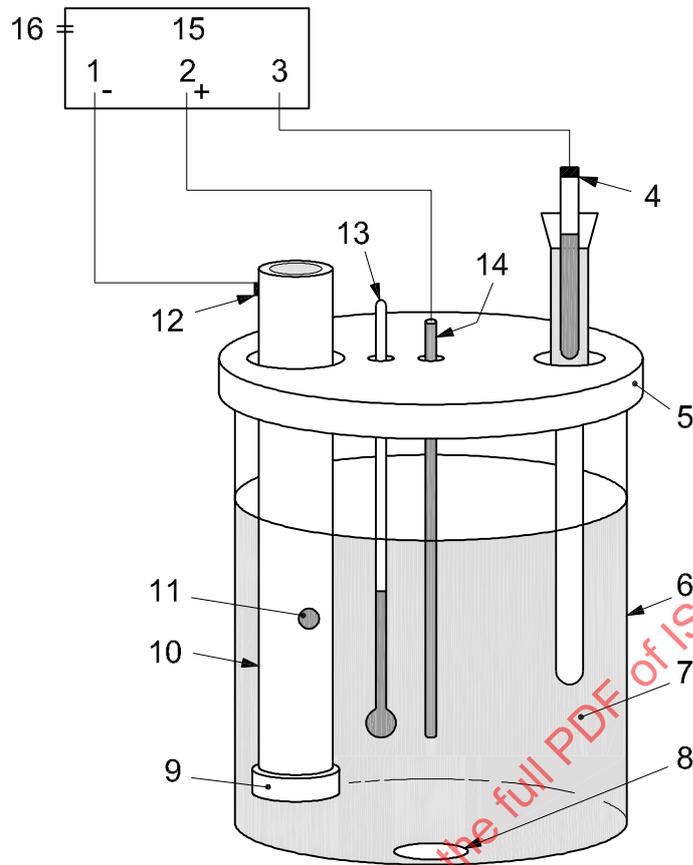
A.9.3.2.8 Upon test completion, dismantle the test cell. As soon as possible, make eight radial cuts through the coating to the substrate, using the utility knife. Such cuts shall extend at least 20 mm from the centre of the holiday.

A.9.3.2.9 Allow the specimen to air cool to $20 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$, then evaluate the cathodic disbondment characteristics of the test specimen within 1 h of the removal from heat.

A.9.3.2.10 Evaluate the cathodic disbondment by inserting the tip of the blade of the utility knife under the coating at the holiday.

A.9.3.2.11 Using a levering action, chip off the coating. Continue until the coating demonstrates a definite resistance to the levering action.

A.9.3.2.12 Measure the disbonded distance from the edge of the original holiday along each radial cut, and average such measured values.



Key

- | | | | |
|---|---|----|-----------------------------|
| 1 | working electrode | 9 | sealed end of pipe specimen |
| 2 | electrode (anode) | 10 | coated pipe |
| 3 | electrode (reference) | 11 | artificial defect |
| 4 | reference electrode | 12 | electrode (cathode) |
| 5 | plastic or polytetrafluoroethylene (PTFE) cover | 13 | thermometer or thermocouple |
| 6 | non-conducting material | 14 | platinum electrode (anode) |
| 7 | electrolyte ≥ 300 ml | 15 | rectified d.c. source |
| 8 | magnetic stirrer | 16 | power supply |

Figure A.9 — Electrolytic cell for pipe with nominal diameter smaller than 4" (101,6 mm)

A.9.4 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- average disbondment value, expressed in millimetres;
- pipe number or identification.

A.10 Cathodic disbondment of high temperature coatings (>95°C)

A.10.1 Equipment

The equipment shall consist of the following:

A.10.1.1 d.c. power supply, capable of supplying constant voltage up to 3,5 V. Anode shall be connected to the positive terminal of the d.c. power supply and the specimen to the negative terminal.

A.10.1.2 Hot plate, with a steel tray consisting of steel grit/shot, sand controllable to $\pm 3^\circ\text{C}$ or liquid heating circulation bath with built-in heater (silicone oil or equivalent) to produce control temperature inside the bath vessel where samples are placed. Other heating methods can be used by agreement.

A.10.1.3 Non-conductive vessel, with a 75 mm \pm 3 mm ID capable of withstanding specified test temperatures. A glass or borosilicate glass cylinder either fitted with a PTFE plate or glass enclosure with port for a thermometer/ thermocouple, platinum anode, reference electrode and a reflex condenser coil (to re-introduce the electrolyte evaporated from the vessel to minimize the loss of electrolyte).

A.10.1.4 Reference electrode: Ag/AgCl double junction reference electrode shall be used. The double junction reference electrode contains an Ag/AgCl wire in equilibrium with saturated KCl solution. The external reservoir contains a 1 M (molar) KNO_3 solution.

A.10.1.5 Anode: 0,8 mm platinum wire shall be used.

A.10.1.6 Anode isolation: an anode isolation shall be used as follows.

Insert the anode into a glass tube of proper size with a removable glass wool plug. The top opening at the glass tube should be small to reduce water evaporation but allow chlorine gas to escape. This isolation shall be 25 mm away from the holiday. The glass wool plug may not be reused.

A.10.1.7 Thermocouple.

A.10.1.8 Electrolyte: 3 % sodium chloride.

A.10.1.9 Drill bit, of 3 mm to 6 mm in diameter.

A.10.1.10 Utility knife (see [A.2.1.8](#)).

A.10.2 Test specimens

A.10.2.1 For laboratory-coated test specimen, dimensions shall be approximately 100 mm \times 100 mm \times 6 mm to 8 mm.

A.10.2.2 For samples extracted from the pipe, specimens shall be approximately 100 mm \times 100 mm \times pipe wall thickness (see [Figure A.10](#)).

A.10.2.3 Remove coating from one of the edges of the sample (bare spot) for the negative terminal wire to be connected for test. Alternatively, drill a hole through the specimen for metal to metal contact to attach the negative terminal.

A.10.3 Procedure

A.10.3.1 Use only test specimens that are confirmed to be holiday-free with a holiday detector set at a minimum of 1 800 V.

A.10.3.2 For temperatures ≥ 95 °C, the test temperature shall be the steel temperature of the specimen. A small hole shall be drilled for 10 mm deep into the steel in parallel to the steel surface. A thermocouple wire or other temperature measurement device shall be inserted into the steel to measure the steel temperature.

A.10.3.3 Drill a 3 mm to 6 mm diameter holiday in the centre of the test specimen through the coating to expose the steel substrate (see [Figure A.7](#) above).

A.10.3.4 Centre the non-conductive vessel over the holiday and apply a sealant to form a water-resistant seal. Silicone caulk has been found suitable. Alternatively, a clamping mechanism can be utilized for vessels with a lip at the bottom where a seal rated beyond the test temperature is used.

A.10.3.5 Fill the cylinder to a height of at least 70 mm with the 3 % sodium chloride electrolyte that has been preheated to the test temperature. Mark the solution level on the cylinder.

A.10.3.6 Change the solution weekly. The entire test solution shall be changed after 28 d in case the test duration is more than 28 d by agreement

A.10.3.7 Insert the electrode into the solution and connect it to the positive wire from the d.c. power supply.

A.10.3.8 Attach the negative wire from the d.c. power supply to a bare spot prepared on the test specimen.

A.10.3.9 Apply voltage to the test specimen, and maintain constant temperature under one or more of the test conditions below, as given in [Tables 2, 3, 4](#) and [5](#):

- a) $-1,4 \text{ V} \pm 0,15 \text{ V}$, $20^\circ\text{C} \pm 3^\circ\text{C}$, for $28 \text{ d} \pm 2 \text{ h}$;
- b) $-3,4 \text{ V} \pm 0,15 \text{ V}$, $65^\circ\text{C} \pm 3^\circ\text{C}$, for $24 \text{ h} \pm 30 \text{ min}$;
- c) $-1,4 \text{ V} \pm 0,15 \text{ V}$, $65^\circ\text{C} \pm 3^\circ\text{C}$, for $28 \text{ d} \pm 2 \text{ h}$.
- d) $-1,4 \text{ V} \pm 0,15 \text{ V}$, $95^\circ\text{C} \pm 3^\circ\text{C}$, for $28 \text{ d} \pm 2 \text{ h}$.
- e) $-1,4 \text{ V} \pm 0,15 \text{ V}$, at design temperature of the product, for $28 \text{ d} \pm 2 \text{ h}$.

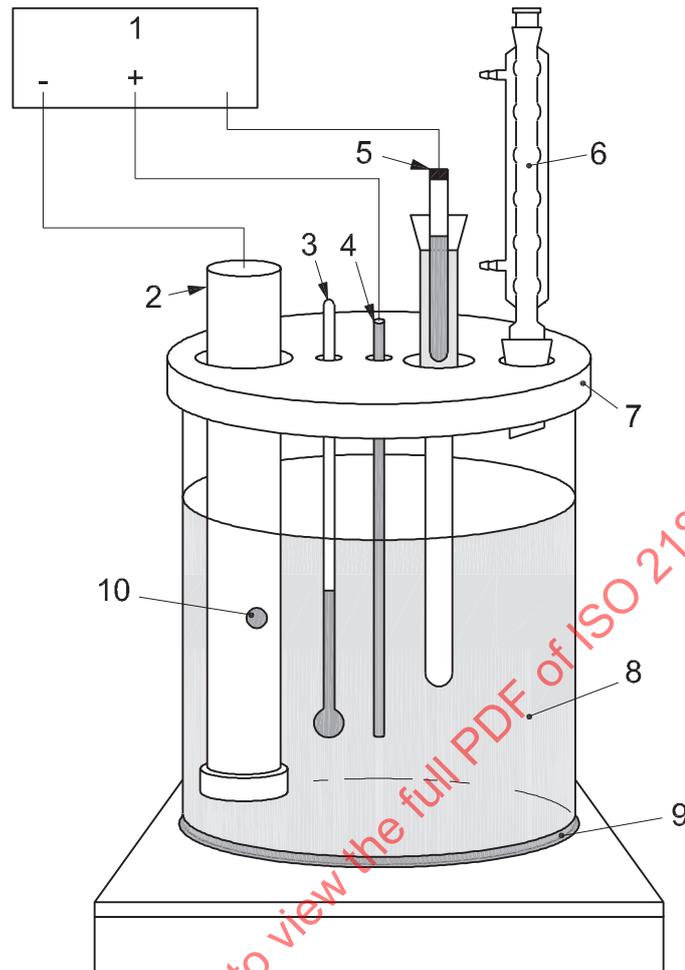
A.10.3.10 Maintain the solution level by adding the required amount of distilled/deionized water.

A.10.3.11 Upon completion, dismantle the test cell. As soon as possible, make eight radial cuts through the coating to the substrate, using the utility knife. Such cuts shall extend at least 20 mm from the centre of the holiday.

A.10.3.12 Allow the specimen to air cool to $20^\circ\text{C} \pm 3^\circ\text{C}$, and evaluate the cathodic disbondment characteristics of the test specimen within 1 h of the removal of the specimen from test set-up.

A.10.3.13 Insert the tip of the blade of the utility knife under the coating at the holiday. Using a levering action, chip off the coating. Continue until the coating demonstrates a definite resistance to the levering action.

A.10.3.14 Measure the disbonded distance from the edge of the original holiday along each radial cut, and average such measured values.

**Key**

1	rectified d.c. source	6	electrolyte condensing system
2	coated specimen	7	plastic or PTFE cover
3	thermometer or thermocouple	8	electrolyte
4	platinum electrode (anode)	9	heat source
5	reference electrode (Ag/AgCl)	10	artificial defect (holiday)

Figure A.10 — Electrolytic cell for high temperature coatings (>95°C)

A.10.4 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- average disbondment value, expressed in millimetres;
- testing of production coating requires pipe number or identification.

A.11 Interface contamination of the coating

A.11.1 Equipment

The equipment shall consist of the following:

A.11.1.1 Stereo microscope.

A.11.1.2 Utility knife (see [A.2.1.8](#)).

A.11.2 Test specimens

Test specimens shall be approximately 25 mm × 200 mm × pipe wall thickness, with the 200 mm dimension parallel to the axis of the pipe.

A.11.3 Procedure

A.11.3.1 Use the utility knife to remove a piece of coating approximately 3 mm × 20 mm from the test specimen bent in accordance with the requirements of [A.13](#).

A.11.3.2 Examine the metal interface side of the coating with the stereo microscope at 40× magnification. Estimate the percentage of interface contamination.

A.11.4 Results

The following information shall be recorded:

- epoxy-powder batch number;
- date of testing;
- percentage of interface contamination.

Testing of production coating requires pipe number or identification.

A.12 Porosity of the coating

A.12.1 Equipment

The equipment shall consist of the following:

A.12.1.1 Stereo microscope, magnification 40×.

A.12.1.2 Bench vise or guided-bend jig.

A.12.1.3 Dry ice or freezer.

A.12.1.4 Utility knife (see [A.2.1.8](#)).

A.12.2 Test specimens

Laboratory-coated test specimens shall be approximately 6 mm to 8 mm × 25 mm × 200 mm. Specimens from test rings shall be approximately 25 mm × 200 mm × pipe wall thickness, with the 200 mm dimension parallel to the axis of the pipe.