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**Non-destructive testing — Penetrant  
testing —**

Part 2:  
**Testing of penetrant materials**

*Essais non destructifs — Examen par ressuage —*

*Partie 2: Essais des produits de ressuage*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3452-2 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 138, *Non-destructive testing*, in collaboration with Technical Committee ISO/TC 135, *Non-destructive testing*, Subcommittee SC 2, *Surface methods*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 3452-2:2000), which has been technically revised.

ISO 3452 consists of the following parts, under the general title *Non-destructive testing — Penetrant testing*:

- *General principles*
- *Part 2: Testing of penetrant materials*
- *Part 3: Reference test blocks*
- *Part 4: Equipment*

# Non-destructive testing — Penetrant testing —

## Part 2: Testing of penetrant materials

**SAFETY PRECAUTIONS** — The materials required by this part of ISO 3452 include chemicals which may be harmful, flammable and/or volatile. All necessary precautions shall be observed. All relevant International, national and local regulations pertaining to health and safety, environmental requirements, etc. shall be observed.

### 1 Scope

This part of ISO 3452 specifies the technical requirements and test procedures for penetrant materials for their type testing and batch testing. It also details on-site control tests and methods.

### 2 Normative references

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

ISO 3059, *Non-destructive testing — Penetrant testing and magnetic particle testing — Viewing conditions*

ISO 3452-3, *Non-destructive testing — Penetrant testing — Part 3: Reference test blocks*

ISO 12706, *Non-destructive testing — Terminology — Terms used in penetrant testing*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

EN 571-1, *Non-destructive testing — Penetrant testing — Part 1: General principles*

### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12706, EN 571-1 and the following apply.

#### 3.1

##### **batch**

quantity of material produced at one operation having uniform properties throughout and with a unique identifying number or mark

#### 3.2

##### **candidate**

sample of the testing product submitted for evaluation in accordance with this part of ISO 3452

## 4 Classification

### 4.1 Testing products

Penetrant testing products shall be classified by type, method and form in accordance with Table 1.

**Table 1 — Testing products**

Penetrant		Excess penetrant remover		Developer	
Type	Denomination	Method	Denomination	Form	Denomination
I	Fluorescent penetrant	A	Water	a	Dry
II	Colour contrast penetrant	B	Lipophilic emulsifier: 1 Oil-based emulsifier 2 Rinsing with running water	b	Water soluble
III	Dual-purpose (fluorescent colour contrast penetrant)	C	Solvent (liquid): Class 1 Halogenated Class 2 Non-halogenated Class 3 Special application	c	Water suspendible
		D	Hydrophilic emulsifier: 1 Optional pre-rinse (water) 2 Emulsifier (water-diluted) 3 Final rise (water)	d	Solvent-based (non-aqueous for Type I)
				e	Solvent-based (non-aqueous for Types II and III)
		E	Water and solvent	f	Special application

### 4.2 Sensitivity levels

#### 4.2.1 General

Sensitivity levels shall be defined separately for penetrant, excess penetrant remover and developer, and for product families.

#### 4.2.2 Fluorescent product family

Sensitivity levels for this product family shall be defined by reference products:

- sensitivity level 1/2 (ultra-low);
- sensitivity level 1 (low);
- sensitivity level 2 (medium);
- sensitivity level 3 (high);
- sensitivity level 4 (ultra-high).

#### 4.2.3 Colour contrast product family

Sensitivity levels for this product family shall be defined using the type 1 reference block in accordance with ISO 3452-3:

- sensitivity level 1 (normal);
- sensitivity level 2 (high).

#### 4.2.4 Dual-purpose product family

There are no sensitivity levels for dual-purpose penetrants. Classification may be carried out as for colour contrast systems (see 4.2.3).

### 5 Testing of penetrant materials

#### 5.1 Testing facilities

##### 5.1.1 Type testing

Type testing shall be carried out on penetrant materials according to EN 571-1 to ensure their conformance to the requirements of this part of ISO 3452.

Type testing shall be carried out by a laboratory accredited in accordance with ISO/IEC 17025 for type testing of penetrant materials.

##### 5.1.2 Batch testing

Batch testing to the requirements of this part of ISO 3452 shall be carried out on each production batch according to EN 571-1 to ensure the batch has the same properties as the corresponding type approval sample. In the case of penetrant material packed in spray cans, the content of sulfur and halogens shall be additionally determined according to 6.12.

Batch testing shall be carried out under a defined and maintained quality system. A system meeting the requirements of ISO 9001 is considered suitable.

##### 5.1.3 Process control testing

Process control testing shall be carried out or commissioned by the user in accordance with EN 571-1 and ISO 3452-3.

#### 5.2 Reporting

##### 5.2.1 Type testing

The testing laboratory (see 5.1.1) shall provide a certificate of compliance with this part of ISO 3452 and a report that details the results obtained.

If any changes are made to the penetrant material composition, then a new type test and product identity shall be required.

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### 5.2.2 Batch testing

Manufacturers of penetrant materials shall provide certificates of compliance with this part of ISO 3452 (for example, see EN 10204).

### 5.2.3 Process and control testing

Results obtained shall be recorded (see Annex B).

## 5.3 Tests

### 5.3.1 Penetrants

Type and batch testing of penetrant properties shall be carried out in accordance with Table 2.

Table 2 — Properties of penetrants and required tests

Property	Test type	Use test method according to
Appearance	Batch	6.1
Sensitivity	Type and batch	6.2
Density	Type and batch	6.3
Viscosity	Type and batch	6.4
Flashpoint	Type and batch	6.5
Washability (Method A penetrants only)	Batch	6.6
Fluorescent brightness (Type I penetrants)	Type and batch	6.7
UV stability (Type I penetrants)	Type	6.8
Thermal stability (Type I penetrants)	Type	6.9
Water tolerance (Method A penetrants only)	Type	6.10
Corrosive properties	Type and batch	6.11
Content of sulfur and halogens <sup>a</sup>	Type and batch	6.12
Water content (Methods A and E)	Batch	6.20
Other contaminants on request (as required)	Batch	

<sup>a</sup> Only required for products designated "low in sulfur and halogens".

### 5.3.2 Excess penetrant removers (excluding method A)

Type and batch testing of penetrant remover properties shall be carried out in accordance with Table 3.

**Table 3 — Properties of excess penetrant removers and required tests**

Property	Test type	Use test method according to
Appearance	Batch	6.1
Sensitivity	Type and batch	6.2
Density	Type and batch	6.3
Viscosity (for Methods B and D only)	Type and batch	6.4
Flashpoint	Type and batch	6.5
Water tolerance (Method B only)	Type and batch	6.10
Corrosive properties	Type and batch	6.11
Content of sulfur and halogens <sup>a</sup>	Type and batch	6.12
Residue on evaporation/solid content	Type and batch	6.13
Penetrant tolerance (Methods B and D only)	Type	6.14
Water content (Method B only)	Batch	6.20
Other contaminants on request (as required)	Batch	

<sup>a</sup> Only required for products designated "low in sulfur and halogens".

### 5.3.3 Developers

Type and batch testing of developer properties shall be carried out in accordance with Table 4.

**Table 4 — Properties of developers and required tests**

Property	Test type	Use test method according to
Appearance	Batch	6.1
Sensitivity	Type and batch	6.2
Flashpoint (Form d only)	Type and batch	6.5
Corrosive properties (except Form a)	Type and batch	6.11
Content of sulfur and halogens <sup>a</sup>	Type and batch	6.12
Solid content (Form d only)	Type and batch	6.13
Developer performance (except Form e)	Type and batch	6.15
Re-dispersability (Forms c and d only)	Type and batch	6.16
Density (of carrier liquid) (Form d only)	Type and batch	6.17
Particle size distribution	Type	6.19
Other contaminants on request (as required)	Batch	

<sup>a</sup> Only required for products designated "low in sulfur and halogens".

### 5.3.4 Batch tests for spray cans

Batch testing shall be carried out in accordance with the product performance test given in 6.18.

The first and last containers, and a container from the middle, of the batch shall be tested. Where testing for content of sulfur and halogens in accordance with 6.12 is appropriate, only the first container need be tested.

## 6 Test methods and requirements

### 6.1 Appearance

The appearance of the sample shall be the same as that of the type test sample.

### 6.2 Penetrant system sensitivity

#### 6.2.1 Fluorescent penetrants (Type I)

##### 6.2.1.1 Qualification provisions

##### 6.2.1.1.1 Penetrants (Type I)

Method A (water washable) penetrants and Methods B and D (post-emulsifiable) penetrants/emulsifiers shall be qualified with the appropriate reference dry developer D-1. Method C penetrants shall be qualified either on the basis of their performance as Method A, B, or D materials, or, alternatively, with the appropriate reference remover R-1 and reference dry developer D-1 (see Table 5).

**Table 5 — Reference material designation**

Reference material	Designation	
	Method A	Methods B, C and D
Penetrant, Type I, Level ½	FP-1/2	
Penetrant, Type I, Level 1	FP-1W	FP-1PE
Penetrant, Type I, Level 2	FP-2W	FP-2PE
Penetrant, Type I, Level 3	FP-3W	FP-3PE
Penetrant, Type I, Level 4	FP-4W	FP-4PE
Penetrant, Type II, Level 1	VP-1W	VP-1PE
Penetrant, Type II, Level 2	VP-2W	VP-2PE
Emulsifier, Type I, Method B		FE-B
Emulsifier, Type I, Method D		FE-D
Emulsifier, Type II, Method B		VE-B
Removers, Class 1, Method C	R-1	R-1
Removers, Class 2, Method C	R-2	R-2
Developer, Form a	D-1	D-1
Developer, Form e	D-2	D-2
FP fluorescent penetrant	FE emulsifier for fluorescent penetrant	
W water-washable	VP visible penetrant	
PE post emulsifiable	VE emulsifier for visible penetrant	

#### 6.2.1.1.2 Developers

All developers, except Form f (specific application), intended for use with Type I (fluorescent) penetrant materials, shall be qualified with the reference level 4, Method B penetrant/emulsifier system FP-4PE/FE-B (see Table 6). Form f developers shall be qualified in accordance with 6.2.1.1.4.

A reference sample of each product shall be retained for comparison purposes and designated in accordance with Tables 5 and 6. The manufacturer, manufacturer's reference and the batch number shall be recorded.

NOTE A list of reference products is available from qualified laboratories (e.g. MPA-Hannover, Germany).

#### 6.2.1.1.3 Solvent removers

Classes 1 and 2 solvent removers shall be qualified with reference penetrant FP-4PE and reference developer D-1. Class 3 solvent remover shall be qualified in accordance with 6.2.1.1.4.

#### 6.2.1.1.4 Specific application — Developer/removers

Form f developers and Class 3 removers shall be qualified with materials as specified by the manufacturer and approval shall be specific to those materials.

#### 6.2.1.1.5 Product family

While individual testing products are to be qualified, product families as defined by the manufacturer may be specified as meeting the requirements defined in this part of ISO 3452 (e.g. Type I, Level 2, Method D; Form a).

#### 6.2.1.2 Sensitivity

##### 6.2.1.2.1 General

Sensitivity of type I penetrant systems shall be determined by comparing results of candidate materials, and standard reference products using a set of test panels.

##### 6.2.1.2.2 Test panels

A suitable test panel should be used, e. g. a type 1 reference block, see ISO 3452-3.

Test panels according to ISO 3452-3 have chromium-nickel plating with thicknesses of 10  $\mu\text{m}$ , 20  $\mu\text{m}$ , 30  $\mu\text{m}$  and 50  $\mu\text{m}$ . For each thickness there is a pair of panels with similar cracks. The test panels should be used either for fluorescent or for colour contrast penetrants. The same panels should not be used for the two systems.

Table 6 — Sensitivity and removability matrix

Candidate material	Materials for processing candidates			Reference materials		
<b>Penetrant systems</b>						
Type I, Method A, Level ½			D-1	FP-1/2		D-1
Type I, Method A, Level 1			D-1	FP-1W		D-1
Type I, Method B, Level 1			D-1	FP-1PE	FE-B	D-1
Type I, Method C, Level 1			D-1	FP-1PE	R-1	D-1
Type I, Method D, Level 1			D-1	FP-1PE	FE-D	D-1
Type I, Method A, Level 2			D-1	FP-2W		D-1
Type I, Method B, Level 2			D-1	FP-2PE	FE-B	D-1
Type I, Method C, Level 2			D-1	FP-2PE	R-1	D-1
Type I, Method D, Level 2			D-1	FP-2PE	FE-D	D-1
Type I, Method A, Level 3			D-1	FP-3W		D-1
Type I, Method B, Level 3			D-1	FP-3W	FE-B	D-1
Type I, Method C, Level 3			D-1	FP-3PE	R-1	D-1
Type I, Method D, Level 3			D-1	FP-3PE	FE-D	D-1
Type I, Method A, Level 4			D-1	FP-4W		D-1
Type I, Method B, Level 4			D-1	FP-4PE	FE-B	D-1
Type I, Method C, Level 4			D-1	FP-4PE	R-1	D-1
Type I, Method D, Level 4			D-1	FP-4PE	FE-D	D-1
Type II, Method A, Level 1			D-2	VP-1PE	VE-B	D-2
Type II, Method B, Level 1			D-2	VP-1PE	VE-B	D-2
Type II, Method C, Level 1			D-2	VP-1PE	R-2	D-2
Type II, Method D, Level 1			D-2	VP-1PE	VE-B	D-2
Type II, Method A, Level 2			D-2	VP-2PE	VE-B	D-2
Type II, Method B, Level 2			D-2	VP-2PE	VE-B	D-2
Type II, Method C, Level 2			D-2	VP-2PE	R-2	D-2
Type II, Method D, Level 2			D-2	VP-2PE	VE-B	D-2
<b>Removers</b>						
Class 1	FP-4PE		D-1	FP-4PE	R-1	D-1
Class 2	FP-4PE		D-1	FP-4PE	R-2	D-1
<b>Developers</b>						
Form a	FP-4PE	FE-B		FP-4PE	FE-B	D-1
Form b	FP-4PE	FE-B		FP-4PE	FE-B	D-1
Form c	FP-4PE	FE-B		FP-4PE	FE-B	D-1
Form d	FP-4PE	FE-B		FP-4PE	FE-B	D-1
Form e	VP-2PE	VE-B		VP-2PE	VE-B	D-2

### 6.2.1.2.3 Test procedures

The same defined procedure shall be used for testing the candidate and the reference penetrant. The reference penetrant shall be of the same sensitivity level as the candidate. Table 7 gives an example of parameters. Each procedure shall be repeated at least three times and the results averaged.

**Table 7 — Type I sensitivity test parameters**

<b>Penetrant dwell</b>	All methods	Dip and then drain for 5 min at 5° to 10° from vertical.
<b>Pre-wash</b>	Method D	Spray for 1 min (160 kPa ± 10 % at 20 °C ± 5 °C).
<b>Emulsification</b>	Method B	Dip and then drain for 2 min.
	Method D	Immerse for 5 min, no agitation: for reference system, 20 % concentration; for candidate system, manufacturer's recommended concentration.
<b>Wash</b>	Method A	Spray for 1 min.
	Method B	Spray under UV-A radiation until fluorescent background is gone. If not achieved within 2 min, the test has failed.
	Method D	Plunge into water to stop emulsification, followed by spray for 2 min.
		For the three methods: 160 kPa ± 10 % in the water pipe nearest to the spray nozzle, at 20 °C ± 5 °C.
<b>Solvent wipe</b>	Method C	Wipe with a clean rag dampened with solvent; then wipe with clean, dry rag to remove excess solvent.
<b>Dry</b>	Methods A; B, D	Dry for 5 min in oven. The temperature in the oven should not be higher than 50 °C. Dry after developer application when testing Forms b and c.
	Method C	Dry for 5 min at room temperature.
<b>Developer</b>	All methods	Dip for 5 s max. in Form a (dry) developer and allow a minimum dwell of 5 min.

### 6.2.1.2.4 Equipment

Suitable equipment shall be used to compare the indications. An example is given in Annex C.

### 6.2.1.2.5 Interpretation of results

Indications shall be evaluated for visibility. The method of assessing visibility shall be defined by the testing laboratory. For visual assessment, viewing conditions shall be in accordance with ISO 3059. Where other assessment is carried out, the viewing conditions shall be stated in the report.

The results shall demonstrate similar or better performance than the reference product. Quantitative assessments, where used, shall show the candidate material giving a result of at least 90 % of the reference product.

6.2.2 Colour contrast penetrants (Type II)

6.2.2.1 Quality provisions

Methods A, B, C and D penetrants and associated removers (if any) shall be qualified with the reference nonaqueous wet developer D-2. Method C (solvent removable) penetrants may also be qualified with the reference solvent remover R-2 and the reference non-aqueous developer D-2 (see Table 6).

All developers, except Form f, intended for use with Type II (visible dye) penetrant materials shall be qualified with a Type II reference penetrant and a Method B emulsifier VP-PE/VE-B.

6.2.2.2 Test panels

The 30 µm and 50 µm test panels from the type 1 reference block in accordance with ISO 3452-3 shall be used.

6.2.2.3 Method of testing

The panels shall be initially calibrated using a Type I (fluorescent), Level 3 penetrant system. The number of clearly visible indications extending across at least 80 % of the panel width shall be recorded. The panels shall then be thoroughly cleaned to remove all traces of the fluorescent materials and reserved for use with Type II penetrants.

Using the candidate material, the panels shall be processed in accordance with a defined procedure. Table 8 gives an example of parameters.

Each procedure shall be repeated at least three times and the results averaged.

Table 8 — Type II sensitivity test parameters

<b>Penetrant dwell</b>	All methods	Dip and then drain for 5 min at 5° to 10° from vertical.
<b>Pre-wash</b>	Method D	Pre-wash for 30 s.
<b>Emulsification</b>	Method B	Emulsify for 30 s.
	Method D	Emulsify for 1,5 min.
<b>Wash</b>	Method A	Spray for 1 min.
	Method B	Spray under UV-A radiation until fluorescent background is gone. If not achieved within 2 min, the test has failed.
	Method D	Plunge into water to stop emulsification, followed by spray for 2 min.
		For the three methods: 160 kPa ± 10 % in water pipe nearest to the spray nozzle, at 20 °C ± 5 °C.
<b>Solvent wipe</b>	Method C	Wipe with a clean rag dampened with solvent; then wipe with clean, dry rag to remove excess solvent.
<b>Dry</b>	Methods A, B, D	Dry for 5 min in oven 50 °C ± 3 °C max.
	Method C	Dry for 5 min at room temperature.
<b>Developer</b>	All methods	Spray with reference developer D-2 of Table 5 and allow a minimum dwell of 5 minutes.

#### 6.2.2.4 Interpretation of results

For visual assessment, viewing conditions shall be in accordance with ISO 3059. Where other assessment is carried out, the viewing conditions shall be stated in the report.

A sensitivity percentage is achieved by the ratio of two figures:

- the number of unbroken indications covering at least 80 % of the panel width, clearly visible to the unaided eye (with glasses if usually worn);
- the number of indications seen when the panel has been first calibrated as per 6.2.2.3.

This ratio is multiplied by 100 to achieve a percentage value.

#### 6.2.2.5 Requirements

The sensitivity level shall be determined in accordance with Table 9.

**Table 9 — Determination of sensitivity level for colour contrast penetrants**

Sensitivity level	Percentage of discontinuities found	
	30 µm	50 µm
1	< 75	90 to 99
2	≥ 75	100

### 6.3 Density

#### 6.3.1 Test method

Density at 20 °C shall be determined using a method with an accuracy better than  $\pm 1$  %.

#### 6.3.2 Requirements

This result shall be reported for type testing (nominal value). For batch testing, a tolerance of  $\pm 5$  % of the nominal value shall be permitted.

### 6.4 Viscosity

#### 6.4.1 Test method

Viscosity shall be determined by a suitable method with an accuracy of better than  $\pm 1$  %. The result for a defined temperature shall be recorded. Batch testing shall be carried out at the specified temperature.

#### 6.4.2 Requirements

This result shall be reported for type testing (nominal value). For batch testing, a tolerance of  $\pm 10$  % of the nominal value shall be permitted.

## 6.5 Flashpoint

**WARNING** — Attention is drawn to the hazards involved in testing materials with flashpoints < 25 °C.

### 6.5.1 Test method

The flashpoint shall be determined by an appropriate stated method with an accuracy of better than  $\pm 2$  °C for materials with a flashpoint of less than 100 °C, or better than  $\pm 5$  °C for materials with a flashpoint greater than or equal to 100 °C.

Flashpoint measurement shall only be required for batch testing if the nominal flashpoint is within the range 20 °C to 110 °C. The flashpoint shall be determined by an appropriate method.

### 6.5.2 Requirements

The result shall be reported for type testing (nominal value). The flashpoint for batch testing shall be no more than 5 °C below the nominal value.

## 6.6 Washability (Method A penetrants)

When removed with a gentle water spray at  $(20 \pm 5)$  °C the sample penetrant shall not leave more residue on the surface roughness areas  $R_a = 5 \mu\text{m}$  and  $R_a = 10 \mu\text{m}$  of reference test block type 2 in accordance with ISO 3452-3 than the type test sample of the same penetrant rinsed under identical conditions. For fluorescent penetrants, this test shall be carried out under UV-A irradiance in excess of  $3 \text{ W/m}^2$ .

## 6.7 Fluorescent brightness

### 6.7.1 Test method

The fluorescent brightness of Type I penetrants shall be tested in accordance with Annex A.

### 6.7.2 Requirements

For type testing, the fluorescent brightness of the candidate shall not be less than the following percentages of the brightness of the reference FP-4PE (see Table 5):

Sensitivity level 1/2	penetrant	50 %
Sensitivity level 1	penetrant	65 %
Sensitivity level 2	penetrant	80 %
Sensitivity level 3	penetrant	90 %
Sensitivity level 4	penetrant	95 %

Batch testing shall be carried out compared with the type test sample. The tolerance shall be  $\pm 10$  %, but the fluorescent brightness shall not be less than the type test requirement.

## 6.8 UV stability

### 6.8.1 Test method

Prepare 10 filter paper specimens using the candidate penetrant and method in accordance with Annex A. Protect five from heat, light and air currents, while exposing the other 5 specimens to UV-A irradiation (365 nm) of  $(10 \pm 1) \text{ W/m}^2$  for 1 h while protecting them from heat and air currents. The fluorescent brightness of each specimen is determined using the method given in Annex A.

### 6.8.2 Requirements

The average fluorescent brightness of the UV-A irradiated specimens shall be greater than the following percentages of non-irradiated specimens:

Sensitivity level 1/2	penetrant	50 %
Sensitivity level 1	penetrant	50 %
Sensitivity level 2	penetrant	50 %
Sensitivity level 3	penetrant	70 %
Sensitivity level 4	penetrant	70 %

## 6.9 Thermal stability of fluorescent brightness

### 6.9.1 Test method

Prepare 10 filter paper specimens using the candidate penetrant and method in accordance with Annex A. Protect five from heat, light and air currents, while placing the other 5 specimens on a clean metal plate in a dead air oven at  $(115 \pm 2) \text{ }^\circ\text{C}$  for 1 h. The fluorescent brightness of each specimen is determined using the method given in Annex A.

### 6.9.2 Requirements

The average fluorescent brightness of the heated specimens shall be greater than the following percentages of unheated specimens:

Sensitivity level 1/2	penetrant	60%
Sensitivity level 1	penetrant	60%
Sensitivity level 2	penetrant	60%
Sensitivity level 3	penetrant	80%
Sensitivity level 4	penetrant	80%

## 6.10 Water tolerance

### 6.10.1 Test method

Water tolerance shall be determined by adding water accurately to a constantly stirred, accurately measured quantity of the candidate material (typically 20 ml) until the candidate material turns cloudy, thickens or separates. This test shall be carried out at  $(15 \pm 0,5) \text{ }^\circ\text{C}$ .

Water tolerance is the percentage of added water in the final volume (water and test material at which cloudiness/thickening occurred).

### 6.10.2 Requirements

Water tolerance shall be greater than 5 %.

### 6.11 Corrosive properties

#### 6.11.1 General

The compatibility of the penetrant material and the materials to be inspected shall be confirmed by the following methods.

#### 6.11.2 Type testing

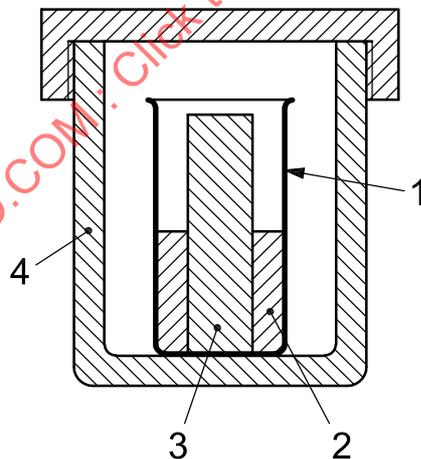
##### 6.11.2.1 Moderate temperature corrosion

###### 6.11.2.1.1 Test procedure

For penetrant materials intended for use on metallic components, the test shall be carried out on bare aluminium alloy EN AW 7075, in the metallurgical state T6 or equivalent, AZ-31B magnesium alloy or equivalent, and on 30 CrMo4 steel or equivalent. Test panels of each of these materials shall be prepared by polishing the surface with silicon carbide paper (240 grit) and rinsing with a volatile, sulfur-free hydrocarbon solvent (e.g. analytical grade acetone) immediately prior to use.

The test panels shall be immersed to half their length in the penetrant material under test, and placed in a glass beaker of sufficient size inside a Parr bomb calorimeter (or equivalent container capable of withstanding an internal pressure of 700 kPa) as shown in Figure 1.

The sealed calorimeter shall then be placed in an oven, or hot water bath, maintained at  $(50 \pm 1)^\circ\text{C}$  for  $2\text{ h} \pm 5\text{ min}$ . The test panel shall then be removed and rinsed briefly under distilled water or organic solvent, as appropriate, to remove all residues of penetrant materials, and inspected.



#### Key

- 1 beaker
- 2 penetrant material
- 3 test panel
- 4 calorimeter

Figure 1 — Parr bomb calorimeter

### 6.11.2.1.2 Requirements

There shall be no evidence of staining, pitting or any other corrosion when examined under 10 × magnification.

### 6.11.2.2 Compatibility with other materials

#### 6.11.2.2.1 Test procedure

The procedure according to 6.11.2.1.1 may be adapted for use with any other materials with which the penetrant material is to be used, by replacing the metal test panel with a panel of that material.

#### 6.11.2.2.2 Requirements

There shall be no evidence of degradation of the test panel surface.

### 6.11.2.3 High temperature titanium stress corrosion

#### 6.11.2.3.1 Test panels

The test panels shall be Ti-8Al-1Mo-1V (also denominated Ti 811), duplex annealed.

#### 6.11.2.3.2 Specimen preparation

The test specimen shall be as shown in Figure 2 with the longitudinal grain direction parallel to the length dimension. The surfaces of the panels shall be prepared to a surface finish of  $R_a = 20 \mu\text{m}$ . Brake form panels over a  $(7,11 \pm 0,25)$  mm radius mandrel to produce an unrestrained angle of  $65^\circ \pm 5$  (see Figure 2).

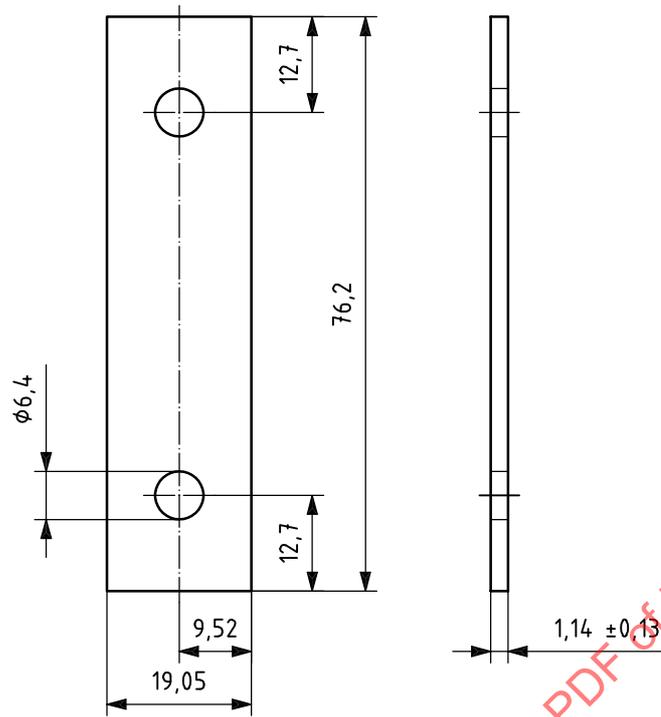
#### 6.11.2.3.3 Test procedure

Four test specimen shall be used with each sample to be tested. Prior to stressing, the specimens shall be cleaned by solvent wiping or soak, and lightly etched in 40 % nitric acid ( $\text{HNO}_3$ ), 3,5 % hydrofluoric acid (HF) solution. After etching, panels shall be rinsed to ensure acid removal and dried. Stress the specimens with a 6,4 mm bolt as shown in Figure 2 c). One specimen shall remain uncoated, one specimen shall be coated with 3,5 % sodium chloride (NaCl) solution and the remaining specimens shall be coated with the test sample. Coating shall be performed by immersing the stressed panels in the sample with the open end up. Drain the stressed panels for between 8 h and 11 h. Place the stressed specimens in an oven at  $(540 \pm 10)^\circ\text{C}$  for  $(4,5 \pm 0,9)$  h.

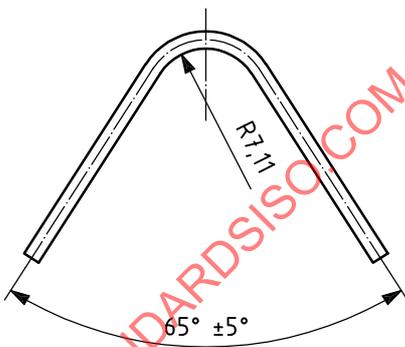
#### 6.11.2.3.4 Interpretation

The specimens shall be viewed for cracks while stressed. If the panel coated with the NaCl solution does not show cracks, remove the bolt, and clean the coated surface by soaking in a 50 % sodium hydroxide (NaOH) solution at  $140^\circ\text{C} \pm 5^\circ\text{C}$  for 30 min followed by a water rinse. Etch in a 40 %  $\text{HNO}_3$ , 3,5 % HF solution for 3 min to 4 min. Examine the etched surface under 10 × magnification. If no pitting or cracks can be observed on the remaining specimens while they are still in the holders, they shall also be cleaned, etched and examined as above. If the NaCl coated specimen has no pitting or cracks or the uncoated specimen has cracks, the test is invalid and shall be repeated. Test specimens shall not be reused. If the test is determined to be valid, the specimen coated with the test sample shall show no evidence of cracks.

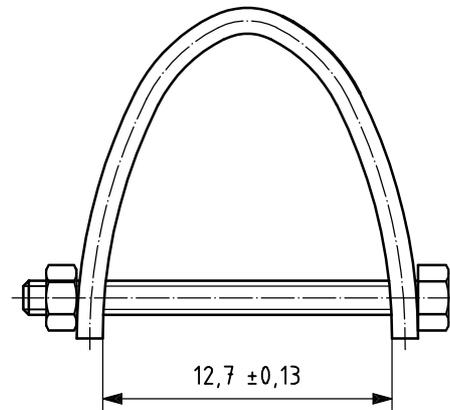
Dimensions in millimetres  
 $\pm 0,5$  tolerance except thickness



a) Dimension details



b) Bend details



c) Stressing details (not to scale)

NOTE The drawings have been derived using imperial values (inches).

Figure 2 — Test specimen for high-temperature titanium stress corrosion

#### 6.11.2.4 High-temperature corrosion of cast nickel alloys

##### 6.11.2.4.1 Specimen preparation

The test specimens shall be Alloy 713LC cut to approximately 25 mm × 13 mm × 2,5 mm. The surface shall be polished with 600 grit abrasive paper to produce a smooth and uniform finish.

##### 6.11.2.4.2 Test procedure

Four test specimens shall be used with the sample to be tested. Immerse or coat two test specimens with the material being tested. Place the two coated and two uncoated specimens in an oven maintained at  $(1\,000 \pm 50)$  °C for  $(100 \pm 4)$ h. Remove the test specimens from the oven and allow them to cool to room temperature. Section, mount, and polish the specimens.

##### 6.11.2.4.3 Interpretation

Examine the cross section of each specimen under  $200 \times$  magnification for evidence of corrosion or oxidation. Coated specimens shall show no more corrosion, oxidation, intergranular, or preferential attack than uncoated specimens.

#### 6.11.3 Batch testing

##### 6.11.3.1 Compatibility with metals

Batch testing shall be carried out on magnesium alloy panels only in accordance with 6.11.2.1.1 except that the panels shall be left for a period of 24 h at room temperature, after which time they shall be cleaned and inspected in accordance with 6.11.2.1.1.

##### 6.11.3.1.1 Requirements

There shall be no evidence of staining, pitting or any other corrosion compared with the untreated half.

##### 6.11.3.2 Compatibility with other materials

The procedure according to 6.11.2.1.1 may be adapted for use with any other materials with which the penetrant material is to be used, by replacing the metal test panel with a panel of the other material.

##### 6.11.3.2.1 Requirements

There shall be no evidence of degradation of the material under test.

#### 6.12 Content of sulfur and halogens (for products designated low in sulfur and halogens)

##### 6.12.1 Test method

The sulfur and halogen content shall be determined by a suitable stated method. The accuracy for liquids shall be  $\pm 10 \times 10^{-6}$  (10 parts per million) by mass for values of less than  $200 \times 10^{-6}$  (200 parts per million) by mass. The accuracy for solids shall be  $\pm 50 \times 10^{-6}$  (50 parts per million) by mass for values of less than  $200 \times 10^{-6}$  (200 parts per million) by mass.

Spray cans shall be purged for 5 s before sampling. Immediately after weighing, spray the content of the can into a 100 ml beaker and transfer immediately into the platinum boat. The operation shall not last more than 2 min from the start of sampling to closing of the bomb calorimeter.

##### 6.12.2 Requirements

Total sulfur content without evaporation shall be less than  $200 \times 10^{-6}$  (200 parts per million) by mass. Total halogen content (chloride and fluoride), without evaporation, shall be less than  $200 \times 10^{-6}$  (200 parts per million) by mass.

## 6.13 Residue on evaporation/solid content

### 6.13.1 Solvent removers

#### 6.13.1.1 Test procedure

A sample having an initial volume of  $(100 \pm 1)$  ml shall be evaporated for 1 h on a  $(15 \pm 1)$  cm Petri dish over a water bath or in an oven at a temperature of  $(15 \pm 1)$  °C above final boiling point of the product. After this time, the mass of the residue shall be measured.

#### 6.13.1.2 Requirements

The mass shall be less than 5 mg.

### 6.13.2 Form d and e developers

#### 6.13.2.1 Test procedure

A sample having an initial mass of  $(100 \pm 1)$  g shall be evaporated for 1 h on a  $(15 \pm 1)$  cm Petri dish over a water bath or in an oven at a temperature of  $(15 \pm 1)$  °C above final boiling point of the product. After this time, the mass of the residue shall be measured and recorded as a percentage of the initial mass.

#### 6.13.2.2 Requirements

The result shall be reported for type testing (nominal value). For batch testing a tolerance  $\pm 10$  % of the nominal value shall be permitted.

## 6.14 Penetrant tolerance

### 6.14.1 Lipophilic emulsifier (Method B)

The addition of 20 % (volume fraction) of the penetrant(s) with which the emulsifier is to be used shall not result in an increase in the background.

### 6.14.2 Hydrophilic emulsifier (Method D)

At the qualification concentration of the emulsifier, the addition of 1 % (volume fraction) of the penetrant(s) with which the remover is certified shall not result in an increase in the background.

## 6.15 Developer performance

When applied according to the manufacturer's recommendations, the developer shall give a fine, even, non-reflective and non-fluorescent coating. When used in conjunction with the appropriate penetrant, the developer shall increase the visibility of the penetrant indications.

## 6.16 Redispersability

### 6.16.1 Water-suspendible developers

The solids shall be readily suspended when stirred or agitated.

### 6.16.2 Solvent based developers (nonaqueous)

The solids shall be readily dispersed when stirred or agitated. Aerosol solids contents shall be suspended after 30 s of shaking.

## 6.17 Density of carrier liquid

### 6.17.1 Test method

The density of the carrier liquid shall be determined by a method with an accuracy of better than  $\pm 1\%$ .

### 6.17.2 Requirements

For type testing, the result shall be reported (nominal value). For batch testing a tolerance of  $\pm 5\%$  of the nominal value shall be permitted.

## 6.18 Product performance (pressurized containers)

When used in accordance with the manufacturer's recommendations, the product sprayed from the pressurised container shall satisfy the requirements for that product and the requirements of 6.12.

## 6.19 Particle size distribution

The particle size distribution of dry developer powder and the solid content of the wet developer shall be determined by the diffraction method or an equivalent method.

The particle size distribution is characterised by the following parameters:

- lower diameter,  $d_l$ , 10 % of the particles smaller than  $d_l$
- average diameter,  $d_a$ , 50 % of the particles larger and 50 % smaller than  $d_a$
- upper diameter,  $d_u$ , 10 % of the particles larger than  $d_u$

## 6.20 Water content

### 6.20.1 Test method

Water content of Methods A and E penetrants, as well as Methods B and D (undiluted) emulsifiers, shall be accurately measured by a defined procedure. Examples of such procedures are listed in the Bibliography.

### 6.20.2 Requirements

Non-water-based penetrants shall contain less than 5 % of water. Water-based penetrants shall comply with the manufacturer's specification.

Hydrophilic emulsifiers shall contain less than 5 % of water.

## 7 Packaging and labelling

Packaging and labelling shall be in accordance with all applicable international, national and local regulations. Containers and their contents shall be compatible. Containers shall be marked with the batch number in order to ensure traceability and the use-by date.

## Annex A (normative)

### Comparison of fluorescent brightness

#### A.1 Apparatus

**A.1.1 Fluorimeter**, having the following characteristics, equipped to hold filter paper samples (see A.2) and having a lightproof sample compartment, an excitation wavelength of  $(365 \pm 20)$  nm, and measuring emitted light using a sensor with a response similar to the CEI response curve in photopic condition.

**A.1.2 Glassware**: pipettes and measuring cylinders (volumetric flasks) suitable for accurately preparing 4,0 % solutions; 50 ml beakers.

**A.1.3 Suitable, absorbent, non-fluorescent filter paper**, for example Whatman (R) No. 4<sup>1)</sup>. Cut to 2 cm × 2 cm or as required to fit the fluorimeter (A.1.1). These papers shall be kept dry before use, for example, in a desiccator (A.1.5).

**A.1.4 Filter paper drying stand** with “crocodile” clips or similar to hold paper specimens vertically.

**A.1.5 Desiccator**, suitable for holding the filter paper (A.1.4).

**A.1.6 Suitable desiccant**, e.g. silica gel for use in the desiccator (A.1.5).

**A.1.7 Solvent**, fast-drying, 100 % volatile, non-fluorescent, and fully miscible with the penetrant under test.

#### A.2 Preparation of filter paper specimens

**A.2.1** Accurately prepare separate 4,0 % (volume fraction) solutions of test and standard penetrants in an appropriate solvent.

**A.2.2** Pour each solution into a separate glass beaker, then, into each beaker, place, one at a time, five filter paper specimens for 5 s each.

**A.2.3** Allow each paper specimen to dry (approx. 5 min) by suspending them vertically in the “crocodile” clips or similar in the desiccator.

#### A.3 Measurement of fluorescent brightness

After allowing the fluorimeter to stabilize, zero the instrument and then introduce the filter paper specimens, one at a time, into the sample holder. Close the lightproof cover and measure the intensity of the emitted light when the specimen is illuminated in the fluorimeter.

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1) Whatman (R) No. 4 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 3452 and does not constitute an endorsement by ISO of the product named.

#### A.4 Calculation

- A.4.1 Calculate the average reading given by the five standard specimens (S).
- A.4.2 Calculate the average reading given by the five test specimens (T).
- A.4.3 Fluorescent brightness of sample under test =  $T/S \times 100 \%$ .

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## Annex B (normative)

### Process control tests

#### B.1 General

The process control tests given in this annex shall be used when testing in accordance with EN 571-1.

In order to maintain the integrity of a penetrant process, the process as a whole and the individual components of the system shall be regularly checked to ensure that they meet the required standards. This requirement is only applicable to process lines, since products supplied in aerosols or thixotropic penetrants will only be used for a single inspection. In addition, some materials used in process lines can be applied to the work piece either by conventional or electrostatic spraying. Since, once again, the material will only be used for one inspection, these tests are not applicable.

NOTE Where some elements of the process are sprayed it does not eliminate the need for control checks on other parts of the process.

#### B.2 Control tests

Table B.1 details the control tests to be carried out and their frequency. It is the responsibility of a level 3 person, according to ISO 9712 or EN 473, to decide which tests are applicable to a particular process line. The tests may be carried out at more frequent intervals or additional tests carried out if necessary to ensure correct processing conditions.

Table B.1 — Control test form

Control tests	ISO 3452-2:2006, subclause	Frequency				Recording		
		Start of each work period	Weekly	Monthly	Every 12 months	Other	Numerical value	Visual assessment (signature)
<b>System review</b>								
Materials levels (including spraying systems network)	B.4.1	X					Not applicable	
System performance using reference test block type 2	B.4.2	X					Not applicable	
<b>General review</b>								
Penetrant appearance	B.4.3	X					Not applicable	
Rinse water appearance	B.4.4	X					Not applicable	
Rinse water temperature	B.4.5	X					Not applicable	Not applicable
Oven temperature	B.4.6	X					Not applicable	Not applicable
Working area	B.4.7	X					Not applicable	
Compressed air filter(s)	B.4.8		X				Not applicable	
Integrity of UV-A filters (fluorescent system)	B.4.9	X					Not applicable	
UV-A irradiance (fluorescent system)	B.4.10			X				Not applicable
Visible light intensity in inspection booth (fluorescent systems)	B.4.11			X				Not applicable
Visible light intensity (colour contrast systems)	B.4.12			X				Not applicable
<b>Penetrants</b>								
Fluorescent intensity <sup>a</sup>	B.4.13			X			Not applicable	
Colour contrast intensity <sup>a</sup>	B.4.14			X			Not applicable	
Suppliers' overcheck	B.4.14				X		Not applicable	
<b>Emulsifiers</b>								
Concentration of freshly diluted hydrophilic remover	B.4.16					X		Not applicable

Table B.1 (continued)

Control tests	ISO 3452-2:2006, subclause	Frequency				Other	Recording	
		Start of each work period	Weekly	Monthly	Every 12 months		Numerical value	Visual assessment (signature)
<b>Developers</b>								
Appearance of dry powder	B.4.17.1	X					Not applicable	
Fluorescence of dry powder	B.4.17.2	X						Not applicable
Water soluble developer								
a) Concentration	B.4.17.3.1	X						Not applicable
b) Wetting test	B.4.17.3.2	X						Not applicable
c) Temperature	B.4.17.3.3	X						Not applicable
d) Fluorescence of solution	B.4.17.3.4	X					Not applicable	
Water suspendible developer								
a) Concentration	B.4.17.4.1	X						Not applicable
b) Temperature	B.4.17.4.2	X						Not applicable
c) Fluorescence of suspension	B.4.17.4.3	X					Not applicable	
<b>Calibration</b>								
UV-A radiometer	B.4.18					≤ 24 months	Not applicable	
Luxmeter	B.4.19					≤ 24 months	Not applicable	
Thermometers	B.4.20				X		Not applicable	
Pressure gauges	B.4.21				X			Not applicable
Test blocks	B.4.22					Recommended		
<sup>a</sup> Not applicable for aerosols.								

### B.3 Control test forms

The results of each control test shall be recorded on a control test form as shown in Table B.1. A separate form shall be used for each penetrant plant. Any deviations found shall be reported to the responsible person and appropriate corrective action must be taken.

The following shall be included:

- company and site;
- process line identity;
- date;
- shift;
- name and qualification;
- signature.

### B.4 Control test

#### B.4.1 Materials levels

The level of material in all process systems shall be visually examined to ensure that there is sufficient material to allow complete coverage of the components to be processed. If insufficient material is in the system, extra material shall be added and mixed before any other tests are carried out.

#### B.4.2 System performance

This test shall be carried out using a type 2 reference test block in accordance with ISO 3452-3. It is often advantageous to also use a component with known discontinuities typical of those normally expected.

A record in the form of a permanent replica, photograph or other suitable means showing the discontinuities including the level of background, shall be prepared using new, unused materials of the same family and processed using the same parameters normally in use and retained for reference. This record shall be used as a comparison for the practical results obtained using the same test for the daily system performance check. Indications from peelable developers are not the same as obtained using standard developers. The indications on the chrome-plated side of the type 2 reference test block or on the component with known discontinuities shall show the same number of indications and pattern with those of the record prepared using the same materials and process sequence. Similarly, the level of background shall appear the same as that shown on the record.

##### B.4.2.1 Reference test parts cleaning

To ensure that reference test parts are sensitive enough to a change in penetrant process parameters, it is necessary to draw out all of the penetrant, which remains entrapped in the discontinuities after testing. It is of the utmost importance not to physically modify the discontinuities.

The best way is to counteract the adsorption effect on the entrapped penetrant due to the discontinuity walls by a stronger capillary effect. A solvent-based (non-aqueous wet) developer is the most appropriate means.

The following procedure shall be used.

- a) Immediately after processing, remove developer with a water rinse.

- b) Dry, i.e. Not by wiping.
- c) Apply a thick coat of form d developer, with the coating arriving wet on the surface.
- d) Leave for 10 min to 15 min.
- e) Repeat stages a) to d), allowing the developer to remain for 30 min.
- f) Check for penetrant traces under adequate illumination and, if present, repeat stages a) to d) until all traces of penetrant have been removed.
- g) Finally, wash with water and dry.

#### **B.4.3 Penetrant appearance**

Check for any abnormal aspects of the penetrant (e.g. milky appearance, visible contamination, deposits of water at the bottom or top of the penetrant).

#### **B.4.4 Rinse water appearance**

When using recycled water, check for opacity, fluorescence, foam and coloration of the rinse water. Any of these suggests that the treatment system is not functioning effectively.

#### **B.4.5 Rinse water temperature**

Check that the rinse water temperature is within the specified limits.

#### **B.4.6 Oven temperature**

Check that the oven temperature is within the specified limits in the area of the work pieces.

#### **B.4.7 Working area**

Ensure that the working area is clean and tidy. When inspecting components processed with a fluorescent penetrant system, there shall be no reflective surface, e.g. white paper on the inspection bench or in the immediate vicinity of the inspection area. In addition, there shall be no stray white light sources near to the inspection area.

#### **B.4.8 Compressed air filter(s)**

Ensure that the trap(s) are free of contaminants.

#### **B.4.9 Integrity of UV-A filters**

Ensure that lamps with UV-A filters are in good condition.

#### **B.4.10 UV-A irradiance**

Measure UV-A irradiance in accordance with ISO 3059.

#### **B.4.11 Visible light intensity in inspection booth (fluorescent systems)**

Measure the maximum visible light intensity in the booth in accordance with ISO 3059.