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Third edition 2013-11-15

Non-destructive testing — Penetrant testing —

Part 2: **Testing of penetrant materials**

Essais non destructifs — Examen par ressuage — Partie 2: Essai des produits de ressuage



Reference number ISO 3452-2:2013(E)



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Foreword

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

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 135, *Non-destructive testing*, Subcommittee SC 2, and by Technical Committee CEN/TC 138, *Non-destructive testing* in collaboration.

This third edition cancels and replaces the second edition (ISO 3452-2:2006), which has been technically revised.

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

- Part 1: General principles
- Part 2: Testing of penetrant materials
- Part 3: Reference test blocks
- Part 4: Equipment
- Part 5: Penetrant testing at temperatures higher than 50 °C
- Part 6: Penetrant testing at temperatures lower than 10 °C

The main changes with respects to the previous edition are listed below:

- a) The normative references were updated;
- b) <u>Tables 1</u>, <u>4</u>, <u>8</u>, <u>9</u> were corrected;
- c) A new <u>Clause 5.1</u> was inserted;
- d) <u>Clause 6.6</u> was revised;
- e) The former Annex B was deleted;
- f) Editorial changes were made.

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. This part of ISO 3452 covers the temperature range 10 $^{\circ}$ C to 50 $^{\circ}$ C. Additional tests in part 5 or part 6 of ISO 3452 may be required outside this range.

On-site control tests and methods are detailed in ISO 3452-1.

2 Normative references

The following referenced 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 3059, Non-destructive testing — Penetrant testing and magnetic particle testing - Viewing conditions

ISO 3452-1:2013, Non-destructive testing — Penetrant testing — Part 1: General principles

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

ISO 9712, Non-destructive testing — Qualification and certification of NDT personnel

ISO 10474, Steel and steel products — Inspection documents

ISO 12706, Non-destructive testing — Penetrant testing — Vocabulary

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12706, ISO 3452-1 and the following apply.

3.1

batch

quantity of material manufactured in one production 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 <u>Table 1</u>.

Table 1 — Testing products

	Penetrant	Ex	cess penetrant remover	Developer				
Туре	Denomination	Method	Denomination		Denomination			
I	Fluorescent penetrant	Α	Water	a	Dry			
II	Colour contrast pen-	В	I :	b	Water soluble			
	etrant		Lipophilic emulsifier	С	Water suspendable			
III	Dual-purpose (fluores-	С	Solvent (liquid):					
	cent colour contrast		Class 1 Halogenated	,	Solvent-based (non-			
	penetrant)		Class 2 Non-halogenated	d	aqueous for Type I)			
			Class 3 Special application					
		D	Hydrophilic emulsifier	e	Solvent-based (non- aqueous for Types II and III)			
		Ea	Water and solvent removable	f	Special application			
a E.	a Method E relates to application. Penetrant material qualified for method A are also considered qualified for method							

^{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. Sensitivity levels of the different types of penetrants are not comparable.

4.2.2 Fluorescent product family

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

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

Sensitivity level 1/2 applies to Type I method A only.

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 when used as a fluorescent system. However these products may be classified as colour contrast products (see 4.2.3).

5 Testing of penetrant materials

5.1 Personnel

Testing shall be carried out by proficient, suitably trained and qualified personnel and, where applicable, shall be supervised by competent personnel nominated by the employer or, by delegation of the employer, the inspection company in charge of testing. To demonstrate appropriate proficiency it is recommended that personnel be trained according to ISO 9712 or an equivalent formalized system. Operating authorization for personnel shall be issued by the employer in accordance with a written procedure. Non-destructive Testing (NDT) operations, unless otherwise agreed, shall be authorized by a competent and qualified NDT supervisory individual (Level 3 or equivalent) approved by the employer.

5.2 Testing facilities

5.2.1 Type testing

Type testing shall be carried out on penetrant materials according to ISO 3452-1 with exceptions as defined in this part of ISO 3452 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.

5.2.2 Batch testing

Batch testing to the requirements of this part of ISO 3452 shall be carried out on each production batch according to ISO 3452-1 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 $\underline{6.12}$.

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

5.2.3 Process and control testing

Process and control tests to monitor the implementation of the method are described in ISO 3452-1:2013, Annex B.

5.3 Reporting

5.3.1 Type testing

The testing laboratory (see <u>5.2.1</u>) 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.

5.3.2 **Batch testing**

Manufacturers of penetrant materials shall provide certificates of compliance with this part of ISO 3452 (for example, see EN 10204 or ISO 10474).

5.4 Tests

5.4.1 Penetrants

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

Table 2 — Properties of penetrants and required tests

Property	Test type	Reference
Appearance	Batch	<u>6.1</u>
Sensitivity	Type and batch	<u>6.2</u>
Density	Type and batch	<u>6.3</u>
Viscosity	Type and batch	<u>6.4</u>
Flashpoint	Type and batch	<u>6.5</u>
Washability (Method A penetrants only)	Batch	<u>6.6</u>
Fluorescent brightness (Type I penetrants)	Type and batch	<u>6.7</u>
UV stability (Type I penetrants)	Туре	<u>6.8</u>
Thermal stability (Type I penetrants)	Туре	<u>6.9</u>
Water tolerance (Method A penetrants only)	Туре	<u>6.10</u>
Corrosive properties	Type and batch	<u>6.11</u>
Content of sulfur and halogensa	Type and batch	<u>6.12</u>
Water content (Methods A and E)	Batch	<u>6.20</u>
a Only required for products designated "low in s	ulfur and halogens".	

Excess penetrant removers (excluding method A)

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

Table 3 — Properties of excess penetrant removers and required tests

Property	Test type	Reference
Appearance	Batch	<u>6.1</u>
Sensitivity	Type and batch	<u>6.2</u>
Density	Type and batch	<u>6.3</u>
Viscosity (for Methods B and D only)	Type and batch	<u>6.4</u>
Flashpoint	Type and batch	<u>6.5</u>
Water tolerance (Method B only)	Type and batch	<u>6.10</u>
Corrosive properties	Type and batch	6.11
Content of sulfur and halogensa	Type and batch	6.12
Residue on evaporation/solid content (Method C only)	Type and batch	6.13
Penetrant tolerance (Methods B and D only)	Туре	<u>6.14</u>
a Only required for products designated "low in su	lfur and halogens".	

 Table 3 (continued)

Property	Test type	Reference				
Water content (Method B only)	Batch	<u>6.20</u>				
Other contaminants on request (as required)	Batch					
a Only required for products designated "low in sulfur and halogens".						

5.4.3 Developers

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

Table 4 — Properties of developers and required tests

Property		Form					Took borns	Reference	
		b	С	d	e	f	Test type	Reference	
Appearance	Х	Х	Х	Х	х	Х	Batch	<u>6.1</u>	
Sensitivity	Х	X	X	X	х	Х	Type and batch	<u>6.2</u>	
Flashpoint				X	Х	хb	Type and batch	<u>6.5</u>	
Corrosive properties		X	X	X	Х	X	Type and batch	<u>6.11</u>	
Content of sulfur and halogensa	Х	X	X	X	Х	Х	Type and batch	<u>6.12</u>	
Solid content				Х	х	хb	Type and batch	<u>6.13</u>	
Developer performance	Х	Х	Х	Х		Х	Type and batch	<u>6.15</u>	
Re-dispersability			Х	Х	Х	хb	Type and batch	<u>6.16</u>	
Density (of carrier liquid)				Х	Х	хb	Type and batch	<u>6.17</u>	
Particle size distribution	Х		Х	Х	Х	хb	Туре	<u>6.19</u>	
Other contaminants on request (as required)		X	Х	Х	Х	Х	Batch		
a Only required for products designated "low in sulphur and halogens".									
b If applicable.									

5.4.4 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, 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 <u>Table 5</u>).

Table 5 — Reference material designation

D.C	Designation				
Reference material	Method A	Methods B, C and D			
Penetrant, Type I, Level 1/2	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					
W water-washable					
PE post emulsifiable					
FE emulsifier for fluorescent penetrant					
VP visible penetrant					
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 <u>Table 6</u>). Form f developers shall be qualified in accordance with <u>6.2.1.1.4</u>.

A reference sample of each product shall be retained for comparison purposes and designated in accordance with <u>Tables 5</u> and <u>6</u>. 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 <u>6.2.1.1.4</u>.

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 $\mu m,\,20~\mu m,$ $30 \, \mu m$ and $50 \, \mu m$. 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 fo	or processing	candidates	Reference materials			
Penetrant systems							
Type I, Method A, Level 1/2			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-3PE	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	
		Rer	novers				
Class 1	FP-4PE		D-1	FP-4PE	R-1	D-1	
Class 2	FP-4PE		D-1	FP-4PE	R-2	D-1	
,		Dev	elopers				
Form a	FP-4PE	FE-B		FP-4PE	FE-B	D-1	
From b	FP-4PE	FE-B		FP-4PE	FE-B	D-1	
Form c	FP-4PE	FE-B		FP-4PE	FE-B	D-1	

Table 6 (continued)

Candidate material	Materials fo	or processing	candidates	Ref	erence mater	ials
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. <u>Table 7</u> 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

Penetrant dwell	All methods	Dip and then drain for 5 min at 5° to 10° from vertical.			
Pre-wash	Method D	Spray with water for 1 min (160 \pm 16) kPa at (20 \pm 5) °C).			
Emulsification	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. 			
Wash	Method A	Spray with water for 1 min.			
Method B		Spray with water 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 with water for 2 min.			
		For the three methods: pressure at (160 ± 16) kPa in the water pipe nearest to the spray nozzle, temperature at (20 ± 5) °C.			
Solvent wipe	Method C	Wipe with a clean rag dampened with solvent; then wipe with clean, dry rag to remove excess solvent.			
Dry Methods A, B, D Dry for 5 min than 50 °C.		Dry for 5 min in oven. The temperature in the oven should not be higher than 50 $^{\circ}\text{C}.$			
		Dry after developer application when testing Forms b and c.			
	Method C	Dry for 5 min at room temperature.			
Developer	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 B.

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 non-aqueous 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 <u>Table 6</u>).

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 <u>Table 8</u>.

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

Penetrant dwell	All methods	Dip and then drain for 5 min at 5° to 10° from vertical.
Pre-wash	Method D	Pre-wash with water for 30 s.
Emulsification	Method B	Emulsify for 30 s.
	Method D	Emulsify for 1,5 min.
Wash	Method A	Spray with water for 1 min.
	Method B	Spray with water 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 with water for 2 min.
		For the three methods: pressure at (160 \pm 16) kPa in water pipe nearest to the spray nozzle, temperature at (20 \pm 5) °C.
Solvent wipe	Method C	Wipe with a clean rag dampened with solvent.
Dry	Methods A, B, D	Dry for 5 min in oven (50 ± 3) °C max.
	Method C	Dry for 5 min at room temperature.
Developer	All methods	Spray with reference developer D-2 of <u>Table 5</u> and allow a minimum dwell of 5 min.

Table 8 — Type II sensitivity test parameters

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 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

Consitivity lovel	Percentage of discontinuities found					
Sensitivity level	30 μm	50 μm				
1	not applicable	≥ 90				
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 ± 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 ± 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

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 $5\,^{\circ}\text{C}$ below the nominal value.

6.6 Washability (Method A penetrants)

When removed with a gentle water spray at (20 ± 5) °C the sample penetrant shall not leave more background on the surface roughness areas R_a = 5 µm and R_a = 10 µm of reference test block type 2 in accordance with ISO 3452-3 than the type test sample of the same penetrant processed under identical conditions. For Type I penetrants, this test shall be carried out under UV-A irradiance in excess of 10 W/m² and white light maximum 20 lx. For Type II penetrants this test shall be carried out under visible light minimum 500 lx. For Type III penetrants both tests shall be carried out. Visually compare new batches with the retained sample of an acceptable batch of same penetrant system.

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 <u>Table 5</u>):

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) W/m² 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 %

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 ± 2) °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 shows a different visual appearance, e.g. cloudiness, thickening or separation. This test shall be carried out at (15 ± 0.5) °C.

Water tolerance is the percentage of added water in the final volume.

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

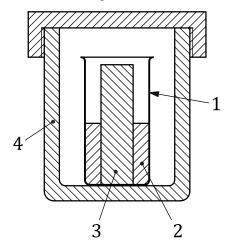
6.11.2.1 Moderate temperature corrosion

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 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 <u>Figure 1</u>.

The sealed calorimeter shall then be placed in an oven, or hot water bath, maintained at (50 ± 1) °C for 2 h \pm 5 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	colourimeter

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 <u>6.11.2.1.1</u> 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 Ti8Al1Mo1V (also denominated Ti 811), duplex annealed.

6.11.2.3.2 Specimen preparation

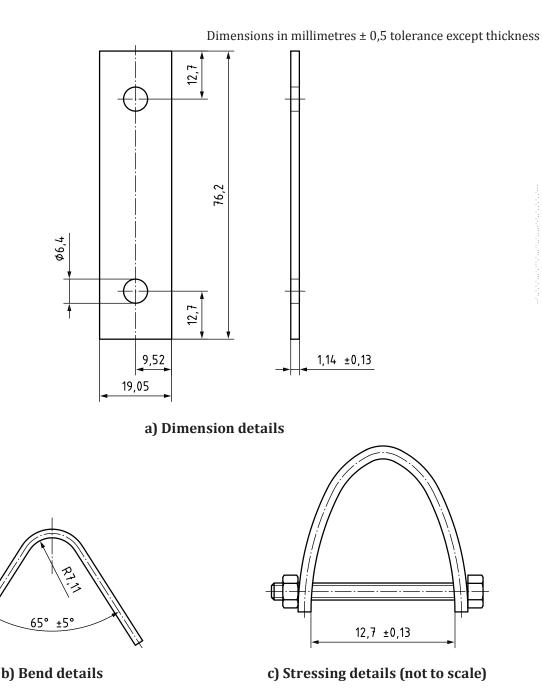
The test specimen shall be as shown in Figure 2 with the longitudinal grain direction parallel to the length dimension. The surfaces of the panels shall be prepared to a surface finish of R_a = 0,5 μ m. Brake form panels over a (7,11 ± 0,25) mm radius mandrel to produce an unrestrained angle of (65 ± 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 (HNO₃), 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 ± 10) °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 ± 5) °C for 30 min followed by a water rinse. Etch in a 40 % HNO₃, 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.



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 \times 13 mm \times 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 $(1\,000\pm50)$ °C for (100 ± 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 × 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

6.11.3.1.1 General

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.

6.11.3.1.2 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

6.11.3.2.1 General

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.

6.11.3.2.2 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 200×10^{-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 200×10^{-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 200×10^{-6} (200 parts per million) by mass. Total halogen content (chloride and fluoride), without evaporation, shall be less than 200×10^{-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 Re-dispersability

6.16.1 Water-suspendable developers

The solids shall be readily suspended when stirred or agitated.

6.16.2 Solvent based developers (non-aqueous)

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 ± 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 pressurized 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 characterized by the following parameters:

lower diameter. d_1 10 % of the particles smaller than d_1

50 % of the particles larger and 50 % smaller than d_a average diameter, d_{a}

10 % of the particles larger than d_{11} upper diameter, 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.

Lipophilic emulsifiers shall contain less than 5 % of water.

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 ± 10) 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^{1}). 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 ($\underline{A.1.4}$).
- **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 (approximately 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.

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 an does not constitute an endorsement by ISO of the product named.

ISO 3452-2:2013(E)

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).
- Fluorescent brightness of sample under test = $T/S \times 100 \%$. A.4.3

Annex B

(informative)

Equipment for determination of the visibility of fluorescent indications²⁾

B.1 General configuration

The equipment consists of a table for the panel whose surface is illuminated by 2 UV-A-lamps from both sides under 45° against the surface. A video camera with suitable resolution can be used to produce images.

The equipment should be sensitive to wavelengths between 450 nm and 650 nm only.

B.2 Image processing

The images of the indications are introduced to an image processing system run on a PC. In a defined area of the panel, the indications over a luminance threshold (grey value) are indicated and the main parameters (light stream, length) listed.

B.3 Evaluation

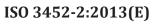
For the relative comparison of the penetrant candidate and the reference, the ability to see the corresponding indications is compared. This may be the visible length or the light stream (luminance multiplied by area of the indication).

When the same crack for the candidate and the reference is used, the procedure shall be carried out one after the other under the same conditions. In case of equivalent panel pairs, the procedure shall be carried out simultaneously and the corresponding indications compared.

²⁾ Also applicable for colour contrast indications, if modified.

Bibliography

- [1] ISO 760, Determination of water — Karl Fischer method (General method)
- [2] ISO 6296, Petroleum products — Determination of water — Potentiometric Karl Fischer titration method
- [3] ISO 9001, Quality management systems — Requirements
- [4] ISO 10336, Crude petroleum — Determination of water — Potentiometric Karl Fischer titration method
- ISO 10337, Crude petroleum Determination of water Coulometric Karl Fischer titration method [5]
- [6] ISO 12937:2000, Petroleum products — Determination of water — Coulometric Karl Fischer titration method
- [7] EN 10204, Metallic products — Types of inspection documents
- [8] EN 13267, Surface active agents — Determination of water content — Karl Fischer method
- ISO 3452-5, Non-destructive testing Penetrant testing Part 5: Penetrant testing at temperatures [9] higher than 50 degrees C
- ISO 3452-6, Non-destructive testing Penetrant testing Part 6: Penetrant testing at temperatures [10] lower than 10 degrees C



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