

Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness —

Part 6: Extraction of soluble contaminants for analysis — The Bresle method

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National foreword

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A list of organizations represented on STI/21 can be obtained on request to its secretary.

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Vorbereitung von Stahloberflächen vor dem Auftragen von Beschichtungsstoffen - Prüfungen zum Bewerten der Oberflächenreinheit - Teil 6: Lösen von wasserlöslichen Verunreinigungen zur Analyse; Bresle-Verfahren (ISO 8502-6:2006)

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Foreword

This document (EN ISO 8502-6:2006) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2007, and conflicting national standards shall be withdrawn at the latest by January 2007.

This document supersedes EN ISO 8502-6:1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 8502-6:2006 has been approved by CEN as EN ISO 8502-6:2006 without any modifications.

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application of paints and related
products — Tests for the assessment
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**Part 6:
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*Partie 6: Extraction des contaminants solubles en vue de l'analyse —
Méthode de Bresle*



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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 8502-6 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 12, *Preparation of steel substrates before application of paints and related products*.

This second edition cancels and replaces the first edition (ISO 8502-6:1995), which has been revised to clarify the procedure (see 5.6 and 5.7).

ISO 8502 consists of the following parts, under the general title *Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness*:

- *Part 2: Laboratory determination of chloride on cleaned surfaces*
- *Part 3: Assessment of dust on steel surfaces prepared for painting (pressure-sensitive tape method)*
- *Part 4: Guidance on the estimation of the probability of condensation prior to paint application*
- *Part 5: Measurement of chloride on steel surfaces prepared for painting (ion detection tube method)*
- *Part 6: Extraction of soluble contaminants for analysis — The Bresle method*
- *Part 8: Field method for the refractometric determination of moisture*
- *Part 9: Field method for the conductometric determination of water-soluble salts*
- *Part 11: Field method for the turbidimetric determination of water-soluble sulfate*
- *Part 12: Field method for the titrimetric determination of water-soluble ferrous ions*

Parts 1 and 10 have been withdrawn. Part 7 (*Field method for the determination of oil and grease*) is in preparation.

Introduction

The performance of protective coatings of paint and related products applied to steel is significantly affected by the state of the steel surface immediately prior to painting. The principal factors that are known to influence this performance are:

- a) the presence of rust and mill scale;
- b) the presence of surface contaminants, including salts, dust, oils and greases;
- c) the surface profile.

International Standards ISO 8501, ISO 8502 and ISO 8503 have been prepared to provide methods of assessing these factors, while ISO 8504 provides guidance on the preparation methods that are available for cleaning steel substrates, indicating the capabilities of each in attaining specified levels of cleanliness.

These International Standards do not contain recommendations for the protective coating systems to be applied to the steel surface. Neither do they contain recommendations for the surface quality requirements for specific situations, even though surface quality can have a direct influence on the choice of protective coating to be applied and on its performance. Such recommendations are found in other documents such as national standards and codes of practice. It will be necessary for the users of these International Standards to ensure that the qualities specified are:

- compatible and appropriate both for the environmental conditions to which the steel will be exposed and for the protective coating system to be used;
- within the capability of the cleaning procedure specified.

The four International Standards referred to above deal with the following aspects of preparation of steel substrates:

ISO 8501 — *Visual assessment of surface cleanliness*;

ISO 8502 — *Tests for the assessment of surface cleanliness*;

ISO 8503 — *Surface roughness characteristics of blast-cleaned steel substrates*;

ISO 8504 — *Surface preparation methods*.

Each of these International Standards is in turn divided into separate parts.

This is one of a number of parts of ISO 8502 that specify tests for the assessment of surface cleanliness. In connection with such tests, there are several methods for the extraction, for analysis, of soluble contaminants on surfaces to be painted. Some of these methods are based on the swabbing of comparatively large test surfaces. This technique provides average values of the contamination present, but it might conceal localized concentrations of contaminants. Also, swabbing might not ensure sufficient penetration to dissolve all of the deep-seated contamination such as ferrous salts.

There are other methods, however, which use small cells for the liquid used to remove and collect the surface contaminants. The cells (rigid or flexible) are attached to test surfaces where soluble contaminants could be expected, e.g. where pitting has occurred. This technique usually provides more accurate, point values of the contamination present.

This part of ISO 8502 describes a simple, inexpensive field test using flexible cells in the form of adhesive patches designed to be filled with solvent. The method was originally developed by a Swedish scientist, Dr. A. Bresle.

Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness —

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1 Scope

This part of ISO 8502 describes a method of extracting, for analysis, soluble contaminants from a surface by use of flexible cells in the form of adhesive patches which can be attached to any surface, regardless of its shape (flat or curved) and its orientation (facing in any direction, including downwards).

The method described is suitable for use in the field to determine the presence of soluble contaminants before painting or a similar treatment.

This part of ISO 8502 does not cover the subsequent analysis of the contaminants that have been dissolved off. Methods of analysis suitable for field use are described in other parts of ISO 8502.

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 554, *Standard atmospheres for conditioning and/or testing — Specifications*

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

ISO 8503-2, *Preparation of steel substrates before application of paints and related products — Surface roughness characteristics of blast-cleaned steel substrates — Part 2: Method for the grading of surface profile of abrasive blast-cleaned steel — Comparator procedure*

ISO/IEC Guide 2, *Standardization and related activities — General vocabulary*

3 Principle

An adhesive patch with a central compartment designed to hold a solvent is attached to the surface from which soluble contaminants are to be removed. The solvent is injected into the compartment by means of a syringe, and then sucked back into the syringe. This operation is repeated a number of times. The solvent (now containing contaminants dissolved off the test surface) is then transferred to a suitable vessel for analysis.

4 Apparatus and materials

4.1 Adhesive patch, the body of which is made of ageing-resistant, flexible material with closed pores, e.g. polyethylene foam, and with a hole punched in the centre. The punched-out material is kept in the hole as a reinforcement until the patch is used. One side of the patch is coated with a thin elastomer film. The other side is coated with adhesive and covered by a removable protective sheet made of paper.

NOTE The hole and the outer edge of the patch may be any shape, e.g. circular, rectangular, elliptical, etc.

The thickness of the patch shall be $1,5 \text{ mm} \pm 0,3 \text{ mm}$. The width of the adhesive rim between the hole and the outer edge of the patch shall be at least 5 mm. Patches with one of the standard compartment sizes specified in Table 1 are called standard patches.

It is essential that the adhesive patch is leaktight. An easily performed leak test has therefore been developed for type testing (see Annex A). Twelve patches of the same size shall be tested, and at least eight of them shall pass the test. The leak test shall be carried out by an accredited laboratory and the result stated in a test report. For terms and definitions in this context, see ISO/IEC Guide 2.

Table 1 — Standard patches

Patch size	Compartment area (mm ²)
A-0155	155 ± 2
A-0310	310 ± 3
A-0625	625 ± 6
A-1250	$1\,250 \pm 13$
A-2500	$2\,500 \pm 25$

4.2 Reusable syringe:

max. cylinder volume: 8 ml
 max. needle diameter: 1 mm
 max. needle length: 50 mm

4.3 Solvent, chosen as a function of the surface contaminants to be determined. For the determination of water-soluble salts or other water-soluble contaminants, use distilled or deionized water.

4.4 Contact thermometer, accurate to $0,5 \text{ }^\circ\text{C}$ and graduated at $0,5 \text{ }^\circ\text{C}$ intervals.

5 Procedure

5.1 Take an adhesive patch (4.1) of a suitable size (see Table 1). Remove the protective paper and the punched-out material (see Figure 1).

5.2 Press the adhesive side of the patch against the test surface (see Figure 2) in such a way that the minimum amount of air is trapped in the compartment in the patch.

5.3 Fill the syringe (4.2) with solvent (4.3) (see Figure 3).

NOTE The volume of solvent needed to fill the patch compartment is proportional to the compartment area, and normally amounts to $2,6 \times 10^{-3} \text{ ml/mm}^2 \pm 0,6 \times 10^{-3} \text{ ml/mm}^2$.

5.4 Insert the syringe needle at an angle of about 30° to the test surface near the outer edge of the patch, so that it passes through the adhesive foam body of the patch into the compartment formed between the elastomer film and the test surface (see Figure 4).

If the patch is in a position which makes access to the patch compartment difficult, bend the syringe needle as required.

5.5 Inject the solvent, ensuring that it wets the whole of the test surface (see Figure 4).

If necessary to avoid air remaining trapped in the patch compartment, carry out the injection in two steps as follows:

Inject half of the solvent. Evacuate the air through the needle by reverse operation of the syringe. Remove the needle from the patch. Holding the syringe with the needle pointing upwards, expel the air. Re-insert the needle into the compartment and inject the remainder of the solvent.

5.6 After a suitable period of time, to be agreed between the interested parties, suck the solvent back into the syringe (see Figure 5). During this period of time, without removing the syringe needle from the patch, re-inject the solvent into the compartment and then suck the solvent back into the syringe cylinder at least four times.

NOTE On unpitted blast-cleaned areas, a period of 10 min has been found satisfactory, as by then more than 90 % of the soluble salts have usually been dissolved.

5.7 Transfer the solvent to a suitable vessel for analysis (see Figure 6).

NOTE In most cases, approximately 95 % of the soluble surface contaminants can be dissolved off by carrying out steps 5.3 to 5.7 only once. By repeating these steps with fresh solvent, almost all of the remaining 5 % can also be dissolved off.

5.8 During steps 5.3 to 5.7, it is essential that no solvent be lost from the patch or syringe, due for instance to inferior-quality materials or improper handling of the materials. If any solvent is lost, the solution obtained shall be rejected.

5.9 On completion of step 5.7, clean and rinse the syringe so that it can be re-used. A bent needle is best left as it is until it becomes necessary to straighten it or bend it further.

5.10 Record the temperature of the steel surface to the nearest 0,5 °C using the contact thermometer (4.4).

6 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 8502 (i.e. ISO 8502-6);
- b) the solvent used;
- c) the volume of solvent injected;
- d) the total time of contact between the solvent and substrate, i.e. the time agreed in 5.6 multiplied by the total number of cycles carried out;
- e) the temperature during steps 5.3 to 5.7 of the procedure;
- f) the manufacturer's batch number of the patch used;
- g) the date of the test.

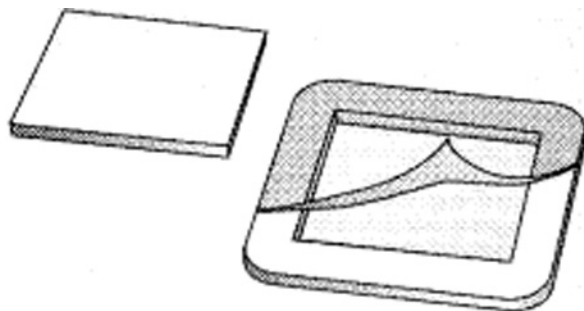


Figure 1 — Protective paper and punched-out material are removed

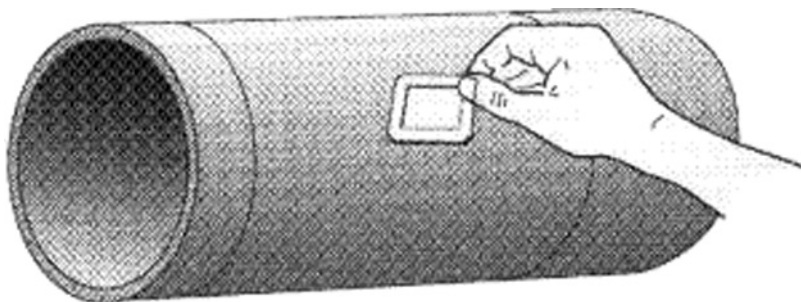


Figure 2 — The adhesive patch is attached to the test surface

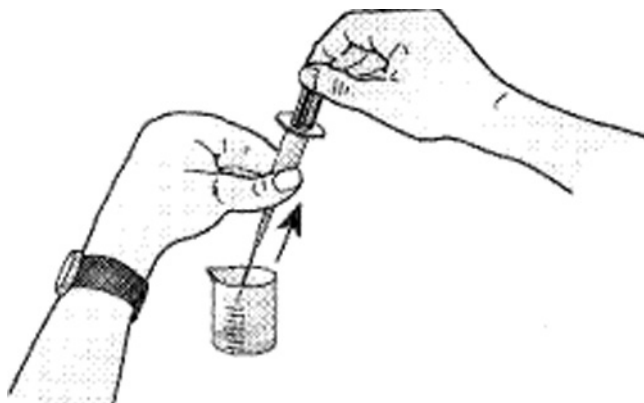


Figure 3 — The syringe is filled with solvent

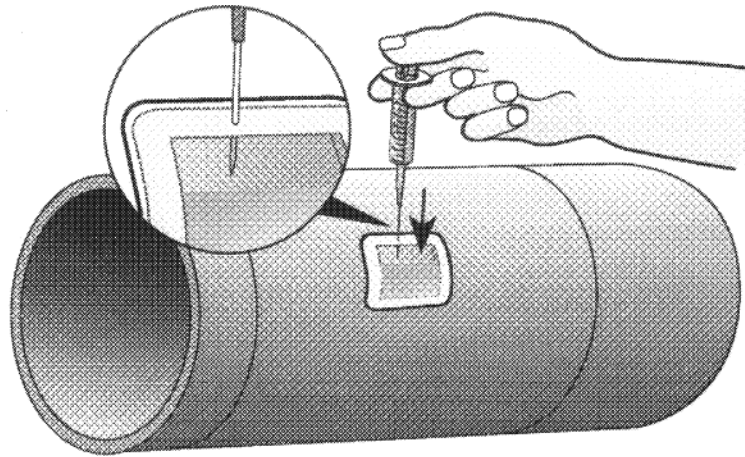


Figure 4 — The solvent is injected into the compartment in the patch
(follow carefully the procedure described in 5.4 and 5.5)

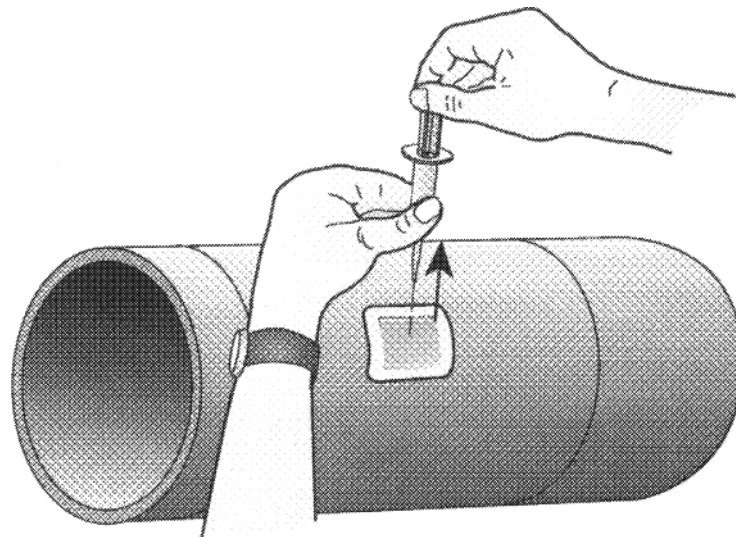


Figure 5 — The solvent is retrieved from the compartment in the patch

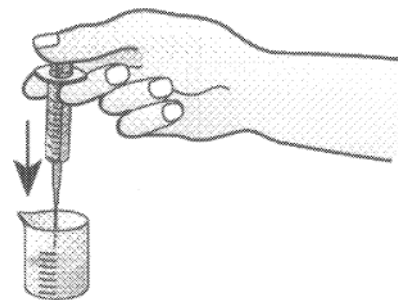


Figure 6 — The solvent is transferred to a suitable vessel for subsequent analysis

Annex A (normative)

Leak test for type testing of patches

A.1 General

The quantitative nature of the removal of the soluble contaminants from a steel surface largely depends on the leaktightness of the adhesive patch, including the adhesive bond between the patch body and the steel surface.

Leakage of the solvent is more likely to occur when the surface is unclean (e.g. covered by rust or moisture) or when it is rough (e.g. due to pits remaining after blast-cleaning).

Leakage is also more likely to occur when the internal pressure is high and of long duration.

These factors affecting leakage are intentionally exaggerated in the following leak test, which is intended for type testing of adhesive patches having an elastomer film made of rubber.

NOTE The test method may also be used for production testing, as well as checking deliveries of such patches. Finally, it may be used for comparisons and to predict possible difficulties in the contaminant-dissolution procedure (Clause 5). However, passing the test is no guarantee that a patch will function satisfactorily in practice under all circumstances.

A.2 Principle

A patch is attached to a clean steel plate of known roughness. Water is injected into the patch compartment in order to create an internal pressure and hence stress the patch. The patch is checked for leakage after a specified time.

A.3 Apparatus and materials

A.3.1 Adhesive patch, as in 4.1.

A.3.2 Visually clean steel plate, of convenient size, e.g. 150 mm × 150 mm, originally of rust grade D, prepared to D Sa 2 ½ as defined in ISO 8501-1, and its secondary profile having been identified as “angular” and graded as “coarse” as defined in ISO 8503-2.

A.3.3 Syringe, as in 4.2.

A.3.4 Water, distilled or deionized.

A.3.5 Stopwatch.

A.4 Procedure

Perform the test in standard atmosphere 23/50 with wide tolerances as defined in ISO 554.

NOTE The numerical values 23 and 50 refer to the temperature in degrees Celsius and the relative humidity in percent, respectively.

A.4.1 Attach the adhesive patch (A.3.1) to the steel plate (A.3.2) and use the syringe (A.3.3) to inject a volume of water (A.3.4) as specified in Table A.1.

Table A.1

Patch size	Volume of water injected (ml)
A-0155	$0,8 \pm 0,1$
A-0310	$3,7 \pm 0,1$
A-0625	$5,5 \pm 0,1$
A-1250	$14,9 \pm 0,1$
A-2500	$39,5 \pm 0,1$

A.4.2 Start the stopwatch (A.3.5).

A.4.3 Check that there is no leakage. Repeat this check at least every 5 min until 20 min have elapsed from the start of the test (A.4.2).

A.4.4 If leakage occurs before 20 min have elapsed, note the time at which it occurred and the point on the patch at which it started.

A.4.5 If leakage does not occur within 20 min, the adhesive patch has passed the leak test.

A.5 Test report

The test report shall contain at least the following information:

- a) the type of adhesive patch tested and its size;
- b) the manufacturer's batch number;
- c) the volume of water injected;
- d) the time required for leakage to occur, if less than 20 min;
- e) the point where leakage started;
- f) the date of the test.

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