

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

Part 6:

Extraction of water soluble contaminants for analysis (Bresle method)

1 Scope

This document specifies a method of extracting, for analysis, water soluble contaminants from a surface by use of flexible cells in the form of adhesive patches or sleeves 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 water soluble contaminants before painting or a similar treatment.

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

NOTE The extraction method might give a false negative or not take all the water-soluble material off the surface because of: a) Soluble materials hiding in the crevices or under folds of metal; b) Soluble materials under corrosion layers, passivation layers produced by corrosion inhibitors, oil, grease, or other non-visible thin films.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

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 overall removal of previous coatings*

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

ISO 8503-2, *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*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO 8502-6:2020(E)

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

This document covers two types of cells: adhesive patch and flexible sleeve.

With the first type, an adhesive patch with a central compartment designed to hold a solvent (in this case water) is attached to the surface from which water-soluble contaminants are to be removed. The water is injected into the compartment by means of a syringe, and then sucked back into the syringe. This operation is repeated, and the water is left to dissolve salts on the surface.

With the second type of cell, a sleeve containing water is attached to the surface and the water is left, under some agitation, to dissolve the salts. The water (now containing contaminants dissolved off the test surface) is then transferred to a suitable vessel for analysis.

5 Apparatus and materials**5.1 Adhesive cell**

An adhesive cell has a body which is made of ageing-resistant, flexible material with closed pores, (e.g. polyethylene foam, and with a hole punched in the centre. For the adhesive patch, the punched-out material is kept in the hole as reinforcement until the patch is used. With the adhesive sleeve, there is no punched-out material in the hole. The flexibility of the cell shall be sufficient to attach to rough surfaces and to areas with some angularity. For the flexible sleeve, it is essential that the foam adhesive ring maintains an exposed area of known size, 1 000 mm² for the standard S-1000 sleeve. One side of the cell is coated with a thin elastomer film or an extraction sleeve. The other side is coated with adhesive and covered by a removable protective sheet. The hole and the outer edge of the patch may be any shape, e.g. circular, rectangular, elliptical.

The thickness of the adhesive rim shall be 1,5 mm ± 0,3 mm and the width of the adhesive rim between the hole and the outer edge of the patch shall be at least 5 mm. The thickness of the adhesive rim of adhesive sleeves of size S-1000 (see [Table 1](#)) shall be 3 mm ± 0,3 mm and the width at least 7 mm.

Cells with one of the standard compartment sizes specified in [Table 1](#) are called standard cells.

It is essential that the adhesive cell is leak tight. An easily performed leak test for type testing shall be performed according to [Annex A](#). Twelve cells 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 17000.

The cell should not cause any noticeable variation in the contamination of the extraction liquid. To confirm accuracy, an initial cell blank test shall be required as described in [6.2](#).

Table 1 — Standard cells

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

Table 1 (continued)

Sleeve size	Compartment area mm ²
S-1000	1 000 ± 10

5.2 Reusable syringe

max. cylinder volume:	10 ml
max. needle diameter:	1 mm
max. needle length:	50 mm

5.3 Solvent (water), of at least grade 3 purity in accordance with ISO 3696.

NOTE 1 Usually, distilled or de-ionized water of conductivity less than 0,5 mS/m (5 µS/cm) meets this requirement.

NOTE 2 Only water is used in this method. Other acidic solvents are covered in ISO 8502-15.

5.4 Contact thermometer, accurate to 0,5 °C and graduated at 0,5 °C intervals.**5.5 Container**, suitable for the analysis to be performed.**6 Procedure****6.1 Selection of test surface**

It is important to select test areas where metal loss or pitting is observed, and on or around metal welds, as these are the areas where salts are most prevalently concentrated. Otherwise, choose test areas which are representative of the entire surface.

In case measurements before surface preparation are to be performed, it is recommended to scrape the surface with a metal spatula or knife until bright metal is exposed over at least 50 % of the test area, or replicates the surface condition specified immediately prior to coating.

6.2 Blank test

To check cleanliness of the cells, blank tests shall be performed in advance for each batch.

For the blank test, a piece of clean glass, to which the adhesive patch will adhere, shall be used and water of the same type as to be used for testing. The same procedure as above, [6.3](#) or [6.4](#), shall be followed. In case the background interference is significant, the cause shall be investigated and, if possible, eliminated.

In case the background interference introduced by the adhesive cell is significant, reproducible and cannot be eliminated, a blank test shall be performed in parallel to each test and the background subtracted from the field quantitative measurements in order to report a net surface contamination value. If this procedure is followed, it shall be stated in the report.

NOTE For the blank test, 2 patches can be stacked together.

6.3 Extraction by patch or sleeve by injection

6.3.1 Take an adhesive patch, "Bresle patch", or sleeve ([5.1](#)) of a suitable size (see [Table 1](#)). Remove the protective paper for the patch remove the punched-out material (see [Figure 1](#)).

ISO 8502-6:2020(E)

6.3.2 Press the adhesive side of the patch or sleeve against the test surface (see [Figure 2](#) and [Figure 7](#)) in such a way that the minimum amount of air is trapped in the compartment of the patch.

6.3.3 Fill the syringe ([5.2](#)) with water ([5.3](#)) (see [Figure 3](#)).

NOTE The volume of water needed to fill the patch compartment is proportional to the compartment area, and normally amounts to $2,6 \times 10^{-3} \text{ ml/mm}^2 \pm 0,6 \times 10^{-3} \text{ ml/mm}^2$. The volume of water needed to fill the sleeve compartment is proportional to the compartment area, and normally amounts to $10,0 \times 10^{-3} \text{ ml/mm}^2 \pm 1,0 \times 10^{-3} \text{ ml/mm}^2$ (10 ml for S-1000 sleeve).

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

6.3.5 If the patch or sleeve is in a position which makes access to the patch compartment difficult, bend the syringe needle as required. Inject the water, ensuring that it wets the whole of the test surface (see [Figure 4](#)).

If necessary to avoid air remaining trapped in the cell compartment, carry out the injection as follows: Inject half of the water. Evacuate the air through the needle by reverse operation of the syringe. Remove the needle from the patch or sleeve. Holding the syringe with the needle pointing upwards, expel the air. Re-insert the needle into the compartment and inject the remainder of the water.

6.3.6 Agitate the solution in the patch or sleeve. Agitation of the water in the patch can be accomplished by sucking the water back into the syringe (see [Figure 5](#)) and re-injecting it at least four times and/or by massaging to create water movement over the test surface. The solution in the flexible sleeve is to be agitated by massaging the sleeve. After a minimum of 10 min extraction time, suck the water back into the syringe (see [Figure 5](#)).

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

6.3.7 Transfer the water to a suitable vessel for analysis (see [Figure 6](#)), as per ISO 8502-9.

6.3.8 During steps [6.3.3](#) to [6.3.6](#), it is essential that no water be lost from the cell or syringe, due for instance to inferior-quality materials or improper handling of the materials. If any water is lost, the solution obtained shall be rejected. Some loss of solution during the final transfer of solution, step [6.3.7](#), is acceptable if the extracted volume is not further diluted. When diluted, no more than 5 % of the volume shall be lost.

6.3.9 On completion of step [6.3.7](#), 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.

NOTE In most cases, approximately 95 % of the soluble surface contaminants can be dissolved by carrying out steps [6.3.3](#) to [6.3.9](#) only once. By repeating these steps with fresh water, almost all of the remaining 5 % can also be dissolved.

6.3.10 Record the volume of water used, V_{extr} , i.e. the total amount of water injected into the extraction cell or the total volume of water used for the analysis as described in ISO 8502-9.

6.3.11 Record the temperature of the steel surface to the nearest $0,5^\circ\text{C}$ using the contact thermometer ([5.4](#)). The temperature shall be over $+5^\circ\text{C}$ to avoid ice on the surface.

6.4 Extraction by sleeve by pre-filling

6.4.1 Take an adhesive sleeve (5.1) of a suitable size (see Table 1). Remove the protective paper and straighten the sleeve.

6.4.2 Prefill the sleeve with the required volume of water (5.3) and adhere the sleeve to the surface without loss of water, see Figure 7.

NOTE The volume of water needed to fill the sleeve compartment is proportional to the compartment area, and normally amounts to $10,0 \times 10^{-3} \text{ ml/mm}^2 \pm 1,0 \times 10^{-3} \text{ ml/mm}^2$.

6.4.3 Position the sleeve such that all water in the sleeve is in contact with the surface.

6.4.4 After a suitable period of time, to be agreed between the interested parties, however a minimum of 10 minutes, let the sleeve hang down in order for the water to flow into the compartment of the sleeve. During this period of time, agitate the sleeve several times by massaging the water on the surface.

6.4.5 Carefully remove the sleeve from the surface. Maintain the water for further analysis.

6.4.6 During steps 6.4.2 to 6.4.4, it is essential that no water be lost from the sleeve, due for instance to inferior-quality materials or improper handling of the materials. If any water is lost, the solution obtained shall be rejected. Some loss of solution during the final transfer of solution, step 6.4.5 is acceptable as long as the remaining volume is sufficient for the analyses to be performed, however no more than 30 % of the volume added.

6.4.7 Record the volume of water used, V_{extr} , i.e. the total amount of water injected into the extraction cell or the total volume of water used for the analysis as described in ISO 8502-9.

6.4.8 Record the temperature of the steel surface to the nearest 0,5 °C using the contact thermometer (5.4). The temperature shall be over +5 °C to avoid ice on the surface.

NOTE In most cases, approximately 95 % of the soluble surface contaminants can be dissolved by carrying out steps 6.4.1 to 6.4.5 only once. By repeating these steps with fresh water, almost all the remaining 5 % can also be dissolved.

7 Analyses

After sampling the solution is analysed as agreed between the interested parties. Suitable methods are conductometric determination of water-soluble salts according to ISO 8502-9 or other methods of chemical analyses.

NOTE Methods of analyses are not covered by this document.

8 Test report

The test report shall contain at least the following information:

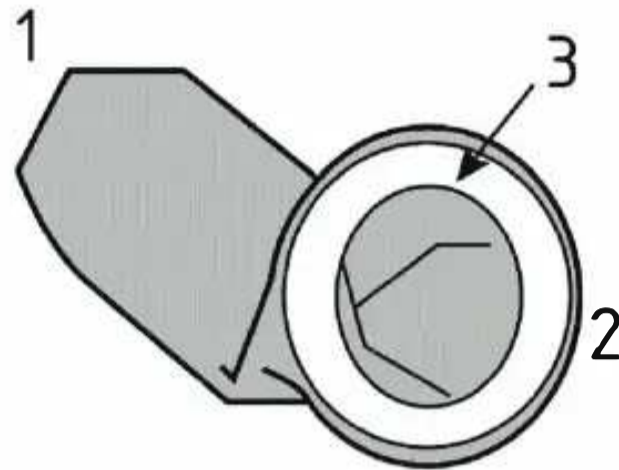
- a) a reference to this document, i.e. ISO 8502-6:2020;
- b) the type and size of extraction cell used: patch or sleeve;
- c) the type of water used as solvent;
- d) the total volume of water used for extraction, V_{extr} ;
- e) the total time of contact between the water and substrate;

ISO 8502-6:2020(E)

- f) the temperature during steps [6.3.1](#) to [6.3.11](#) or [6.4.1](#) to [6.4.8](#) of the procedure;
- g) the manufacturer's batch number of the patch or sleeve used;
- h) the date of the test.



a) Adhesive patch with protective paper and punched-out material



b) Adhesive sleeve

Key

- 1 flexible sleeve-closed end
- 2 flexible sleeve- open end
- 3 adhesive ring with protective paper

Figure 1 — Adhesive patch

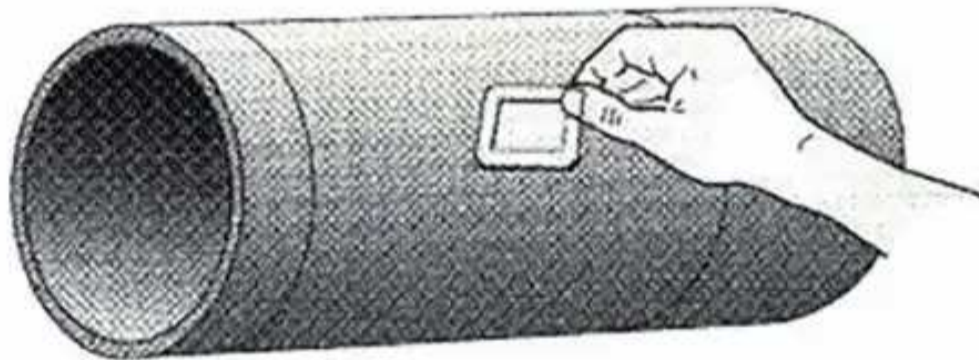


Figure 2 — Adhesive patch being attached to the test surface

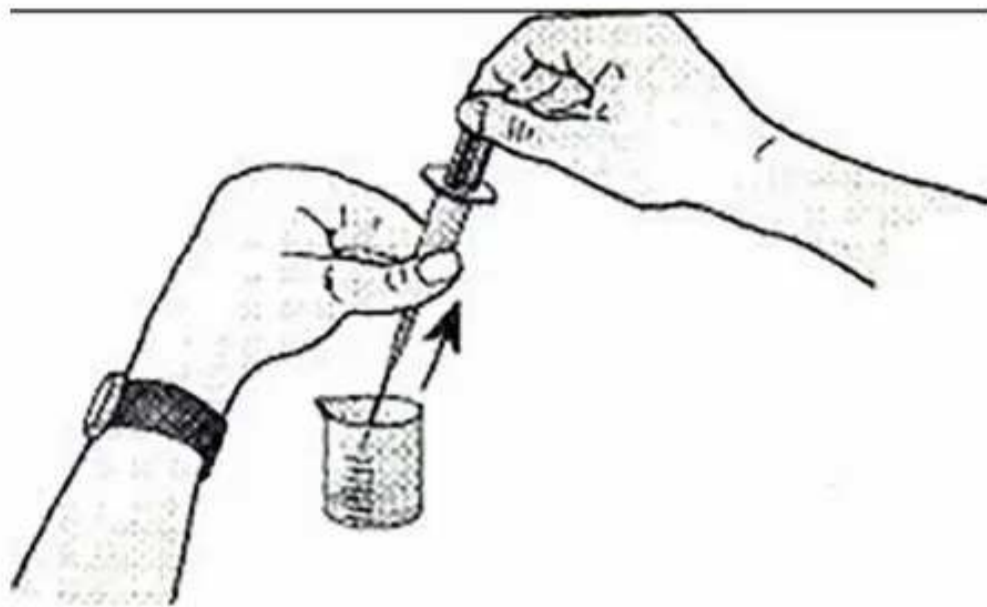


Figure 3 — Syringe being filled with water

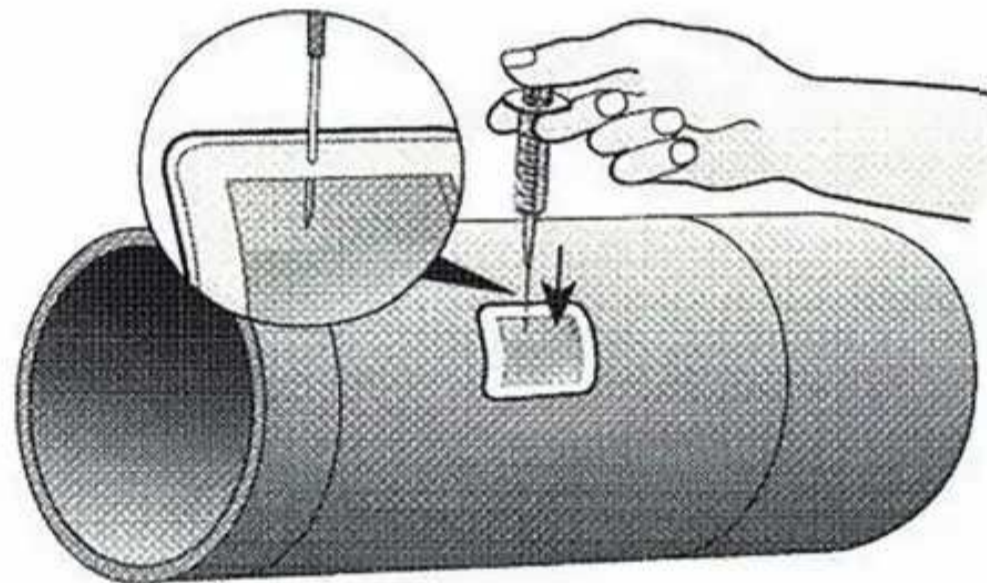


Figure 4 — Water being injected into the compartment in the patch (carefully following the procedure described in [6.3.4](#) and [6.3.5](#))

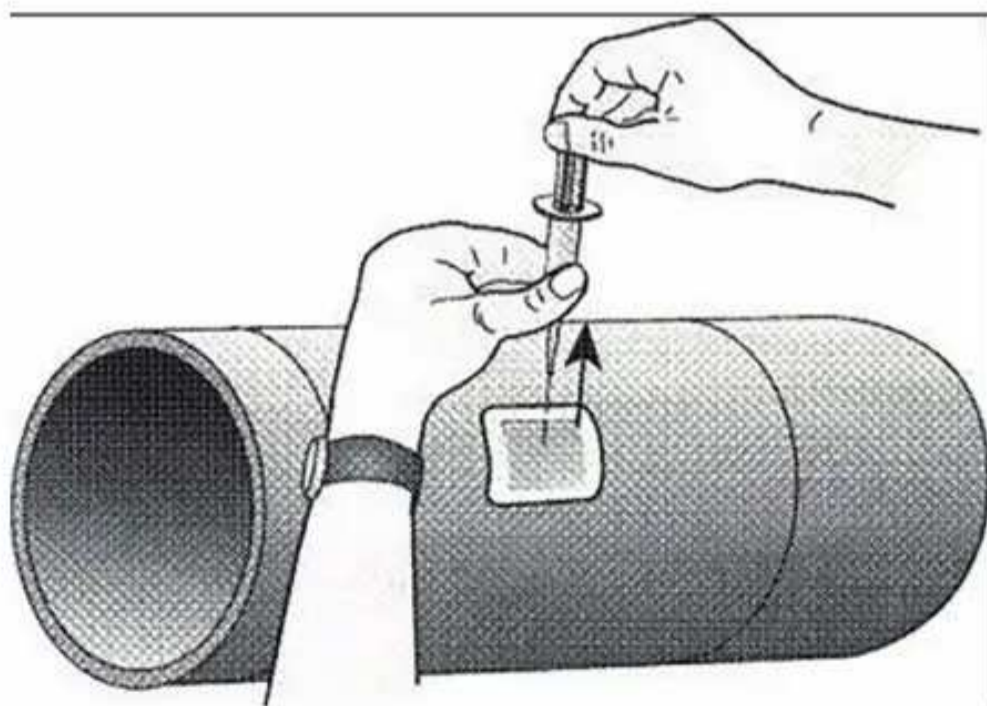


Figure 5 — Water being retrieved from the compartment in the patch

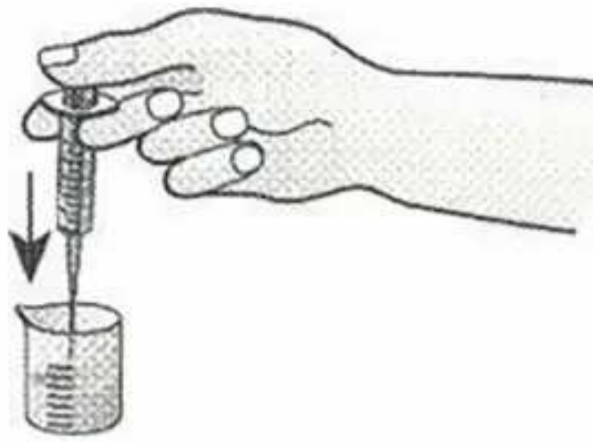


Figure 6 — Water being transferred to a suitable vessel for subsequent analysis

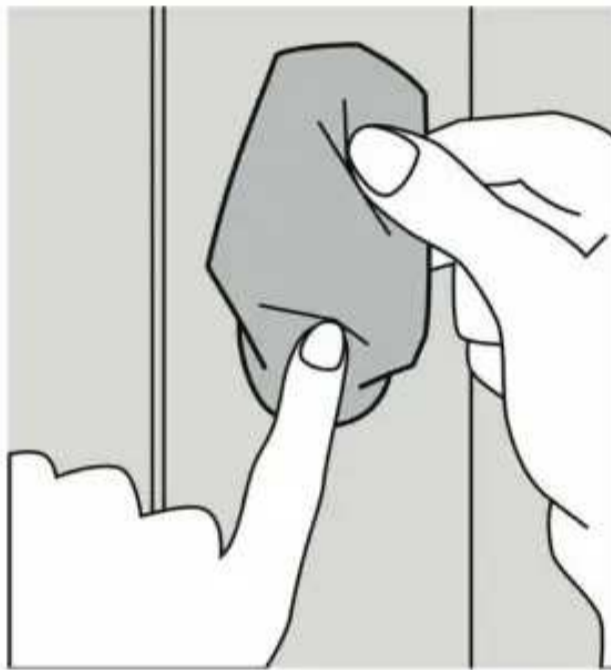


Figure 7 — Flexible sleeve being adhered to a metal surface

Annex A (normative)

Leak test for type testing of adhesive cells (patches and sleeves)

A.1 General

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

Leakage of the water 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.

The test method may also be used for production testing, as well as checking differences of such patches or sleeves. Finally, it may be used for comparisons and to predict possible differences in the contaminant-dissolution procedure (see [Clause 6](#)). However, passing the test is no guarantee that a patch will function satisfactorily in practice under all circumstances.

A.2 Principle

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

A.3 Apparatus and material

A.3.1 Adhesive patch or sleeve, as in [5.1](#).

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 1/2 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 [5.2](#).

A.3.4 Water, distilled or deionized.

A.3.5 Stopwatch.

A.4 Procedure

Perform the test at the temperature $(23 \pm 2)^\circ\text{C}$ and relative humidity $(50 \pm 5)\%$ (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.

ISO 8502-6:2020(E)

A.4.1 Attach the adhesive patch or sleeve (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. The volume is determined as 3 times the maximum injectable volume for each extraction cell as described in 6.3.3.

Table A.1 — Injection Volumes

Patch size	Volume of water injected ml
A-0155	1,5 ± 0,1
A-0310	3,0 ± 0,1
A-0625	6,0 ± 0,1
A-1250	12,0 ± 0,1
A-2500	24,0 ± 0,1
Sleeve size	Volume of water injected ml
S-1000	31,8 ± 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 or sleeve 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;
- g) a reference to this document, i.e. ISO 8502-6:2020.

Bibliography

- [1] ISO/IEC Guide 2, *Standardization and related activities — General vocabulary*
- [2] ISO 8502-15¹⁾, *Preparation of steel substrates before application of paints and related products — Tests for the assessment of surface cleanliness — Part 15: Extraction of soluble contaminants for analysis by acid extraction*
- [3] ISO 8504 (all parts), *Preparation of steel substrates before application of paints and related products — Surface preparation methods*
- [4] BRESLE Å., Conductometric determination of salts on steel surfaces, MP (*Materials Performance*), June 1995, Vol. 34, No. 6, pp. 35-37, NACE International, Houston TX, USA
- [5] CRC Handbook of Chemistry and Physics, 83rd edition, CRC Press
- [6] FRANKHUIZEN N.H., Measuring NaCl, Salt and Soluble Contaminants with Bresle Patches — Part 1 & 2, *Materials Performance* December 2009 pp. 34-36, NACE International, Houston TX, USA. (General information on salts given Part 1, Materials)

1) Under preparation. Stage at the date of publication. ISO/DIS 8502-15:2020.

