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

Part 5:

Measurement of chloride on steel surfaces
prepared for painting (ion detection tube
method)

*Préparation des subjectiles d'acier avant application de peintures et de
produits assimilés — Essais pour apprécier la propreté d'une surface —*

*Partie 5: Mesurage des chlorures sur les surfaces d'acier préparées pour la
mise en peinture (méthode du tube détecteur d'ions)*



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.

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.

International Standard ISO 8502-5 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*.

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 1: Field test for soluble iron corrosion products [Technical Report]*
- *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 refractometric determination of moisture*
- *Part 9: Field method for the conductometric determination of water-soluble salts.*
- *Part 10: Field method for the titrimetric determination of chloride*

Further parts are planned.

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

Part 5:

Measurement of chloride on steel surfaces prepared for painting (ion detection tube method)

1 Scope

This part of ISO 8502 describes a field test for the measurement of chloride ions using special detection tubes.

With suitable surface sampling techniques, the test is applicable to steel surfaces before and after cleaning, as well as to painted surfaces between applications of coats.

NOTE – ISO 8502-2, 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, describes a laboratory method for the determination of chloride on a surface.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 8502. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8502 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use – Specification and test methods.*

3 Principle

A proportion of the water-soluble chlorides on the test substrate is removed by controlled washing or immersion in water. The collected washings are analysed to determine the chloride ion concentration, which is indicated by a colour change in a detection tube containing silver chromate.

4 Apparatus and materials

4.1 Water, of at least grade 3 purity in accordance with ISO 3696.

4.2 Chloride ion detection tube

4.2.1 Design

The detection tube shall comprise a borosilicate glass tube, 140 mm to 150 mm in length and 2 mm to 3 mm inside diameter, containing 5 % (*m/m*) analytical reagent grade silver chromate in a silica sand carrier. The silica sand shall have a particle size range from 177 μm to 250 μm . The mixture of sand and chromate shall be retained in the tube by plugs of cotton wool in both ends and glass powder control material between the carrier and the plugs. Both ends of the glass tube shall be sealed by fusing the glass.

NOTE – If the tube is properly designed, the cotton wool will not be burnt.

4.2.1 Calibration

After making a batch of test tubes, break off both ends of several of them with the glass cutter (4.3) and immerse one end of a tube vertically into each of various solutions containing different chloride ion concentrations. Note the top of the colour change in each tube and mark all the tubes in the batch appropriately to give each a scale in mg/l.

NOTE – Commercial versions of the tubes are available for determining the chloride concentration over various ranges. A suitable tube is chosen for each test.

Unused detection tubes shall be stored in a refrigerator for no longer than two years.

4.3 Cutter, for cutting off the ends of the detection tube.

4.4 Measuring cylinder, of 250 ml capacity.

4.5 Beakers, one of 100 ml capacity, two of 250 ml capacity.

4.6 Glass stirring rod.

4.7 Cotton gauze, chloride-free, measuring approximately 300 mm x 300 mm.

4.8 Gloves, made of plastic or rubber.

4.9 Adhesive tape.

4.10 Tape measure.

5 Procedure

5.1 Washing the test area

While the following test describes a particular swabbing procedure for washing the test area, any of several suitable controlled-washing procedures available may be used if desired. During the washing procedure, ensure, by wearing clean plastic or rubber gloves (4.8), that the wash water is not accidentally contaminated.

Before starting the washing procedure, an area of the steel surface which has been prepared for painting shall be identified by the interested parties as suitable for the test. Mark out an actual test area measuring 0,5 m x 0,5 m, or 0,25 m² in total area, the shape depending on the size and shape of the steel substrate, using the tape measure (4.10) and adhesive tape (4.9).

Pour 130 ml of water (4.1) into one of the 250 ml beakers and 20 ml into the 100 ml beaker (see 4.5). Soak the cotton gauze (4.7) in the water in the larger beaker and squeeze some of the water out gently by hand. As the gloves must be washed at the very end of the washing procedure using only a limited quantity of water, users are advised to hold the gauze with the fingertips only. Thoroughly swab the whole of the test area with the gauze. Allow as little water as possible to drip from the gauze or to run off the test area. Remove the water from the surface with the gauze, squeezing the washings into the other 250 ml beaker. Repeat the swabbing procedure four times (i.e. for a total of five times), changing the direction of wiping each time.

On completion of the fifth swabbing procedure, place the gauze in the larger beaker. With great care rinse the glove fingertips in the water in the small beaker and subsequently pour this rinse water into the larger beaker. Mix the contents of the larger beaker by stirring with the glass stirring rod (4.6).

NOTE – The Bresle method specified in ISO 8502-6 may be used for extraction of the chloride in combination with analysis by the ion detection tube method if the chloride surface density to be determined is higher than 100 mg/m² (10 µg/cm²).

5.2 Determination of the chloride concentration in the test solution

Cut both ends of the detection tube with the cutter (4.3). Immerse one end of tube in the test solution. Hold the tube vertically with the end at or near the bottom of the beaker. When the solution reaches the top plug, read off the chloride concentration in the test solution, in mg/l, from the scale, as indicated by a slight change in the colour where the chromate has reacted with the chloride in the solution.

If the solution fails to reach the top of the tube within 5 min, discard the tube and repeat the procedure with a fresh tube.

Carry out a total of five determinations using five fresh tubes. Note each individual measurement and the average of the three middle values obtained.

No temperature correction is required if the test is carried out between 5 °C and 80 °C. The test shall not be carried out below 5 °C.

NOTE – The presence of bromide, iodide or cyanide ions will give slightly higher readings. Sulfate, nitrate or iron ions can affect the readings by up to 10 %. Variations in pH of the solution within the range 4-13 will not affect the readings.

During the procedure, avoid contaminating the solution with chloride from hands, gloves and the cotton used for swabbing. A blank test may be carried out to determine the amount of such contamination.

5.3 Verification test

To verify tube calibration, take three tubes per production lot and use them to test a solution of known chloride concentration close to that of the test solution already analysed. Take the average of the two closest values as the result of the verification test. The accuracy of the average obtained for the test solution concentration shall be within ± 15 %.

The chloride ion concentration in the test solution shall be within the range 50 mg/l to 100 mg/l.

6 Expression of results

Calculate the amount of chloride extracted N , expressed as milligrams of sodium chloride per square metre of surface, using the following equation:

$$N = V \times \rho_{\text{Cl}} \times \frac{M_r(\text{NaCl})}{A_r(\text{Cl})} \times \frac{1}{A}$$

where

V is the volume, in litres, of water used;

ρ_{Cl} is the chloride ion concentration, in milligrammes per litre, in the larger beaker;

$M_r(\text{NaCl})$ is the molecular mass of sodium chloride (= 58,5);

$A_r(\text{Cl})$ is the atomic mass of chlorine (= 35,5);

A is the test area, in square metres (= 0,25 m²).

7 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the steel surface tested;
- b) a reference to this part of ISO 8502 (ISO 8502-5);
- c) the manufacturer and batch number of the detection tubes used, and the type of tube;
- d) the results of the verification test on the detection tubes;
- e) the chloride ion concentration in the test solution, expressed as:
 - 1) the individual values for all five tubes,
 - 2) the average of the three middle values;
- f) the result of the test as indicated in clause 6;
- g) any deviation from the test method specified, in particular details of any other washing method used;
- h) the date of the test.

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Descriptors: paints, varnishes, substrates, steel products, tests, field tests, determination, surface condition, chlorides, cleanliness checks.

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