INTERNATIONAL STANDARD

ISO 8502-2

Third edition
2017-01

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

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

Partie 2: Recherche en laboratoire des chlorures sur les surfaces nettoyées

Reference number
ISO 8502-2:2017(E)

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Foreword

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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 (see www.iso.org/directives).

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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document 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 third edition cancels and replaces the second edition (ISO 8502-2:2005), which has been technically revised with the following changes:

a) the analysis method has been deleted;

b) a selection of analysis methods from other standards have been added;

c) the coulometric method has been added and briefly described in an informative annex;

d) the formula in Clause 7 has been replaced by Formula (1) transforming chloride concentration in a solution to amount of chloride on the surface;

e) the document has been editorially revised.

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 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*

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 as follows:

a) presence of rust and mill scale;

b) presence of surface contaminants, including salts, dust, oils and greases;

c) surface profile.

The ISO 8501, ISO 8502 and ISO 8503 series of International Standards have been prepared to provide methods of assessing these factors, while the ISO 8504 series 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 series of 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, and

— within the capability of the cleaning procedure specified.

The four series of International Standards referred to above deal with the following aspects of preparation of steel substrates before application of paints and related products:

— ISO 8501 on visual assessment of surface cleanliness;

— ISO 8502 on tests for the assessment of surface cleanliness;

— ISO 8503 on surface roughness characteristics of blast-cleaned steel substrates;

— ISO 8504 on surface preparation methods.

Each of these International Standards is in turn divided into separate parts. This part of ISO 8502 describes a method for the assessment of chloride-containing salts that are readily soluble in water and are present on a steel surface. Rusted steel substrates, particularly of rust grades C or D (see ISO 8501-1), even when blast cleaned to preparation grade Sa 3 (see ISO 8501-1) can still be contaminated by soluble salts and corrosion products. These compounds are almost colourless and are localized at the lowest point of the rust pits. If they are not removed prior to painting, chemical reactions can result in large accumulations of rust that destroy the adhesion between the substrate and the applied protective coating. Even if the salt is readily soluble in water, it is often impossible to remove it completely from the surface by a simple washing such as that described in this procedure. The method does not therefore determine the total amount of chloride on the surface but gives an indication of the cleanliness level of the surface. Prolonging the washing time should remove a larger proportion of the salt.

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

1 Scope

This part of ISO 8502 describes a method for the determination of chloride-containing salts that are readily soluble in water and are present on a steel surface. The method is also applicable to previously coated surfaces. This part of ISO 8502 includes a method, applicable in the field or in the laboratory, for washing off the surface while several methods are referred to for chloride analyses.

NOTE 1 ISO 8502-5 describes a field test for the determination of chloride on a surface.

NOTE 2 The precision of the method is limited by both the accuracy of the selected method of analyses and by uncertainties in the sampling procedure. The extraction method might not take all the water soluble material off the surface due to

- soluble material hiding in crevices, under folds of metal or at the bottom of pits, and
- soluble material hiding under corrosion layers, passivation layers, inhibitors, oil, grease, or other non-visible thin films as these boundary layers can prevent contact with the underlying salt for its removal.

NOTE 3 The performance of a paint system is affected by the amount of soluble chloride remaining on the surface. The acceptable level of this contamination is related to the service conditions. For further information regarding levels of water-soluble salt contamination, see ISO/TR 15235.

2 Normative references

The following 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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

A defined area of the steel surface is washed with a known volume of water and assessment of chloride concentration in the wash water is then carried out using instrumental methods.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade.

4.1 Water, conforming to the requirements of grade 3 of ISO 3696.

4.2 Chalk, essentially chloride-free, **or other suitable means for marking the test area**.

4.3 Absorbent cotton pads (cotton wool), of mass 1 g to 1,5 g.

5 Apparatus

5.1 **Ruler**.

5.2 **Metal spatula** or **knife**.

5.3 **Gloves**, of plastics material.

5.4 **Beakers**, of capacity 250 ml.

5.5 **Small glass rod**.

5.6 **Filter funnel**.

5.7 **Filter paper**, approximately 120 mm diameter.

5.8 **Measuring cylinder**, of capacity 50 ml.

5.9 **Volumetric flasks**, of capacities 50 ml, 100 ml and 1 000 ml.

5.10 **Volumetric pipettes**, of capacities 1 ml and 20 ml.

6 Procedure

6.1 Washing of the surface

Carry out the test at least in duplicate. During the swabbing procedure, prevent accidental contamination of the wash water by using, for example, clean plastics gloves (5.3) each time.

Mark out a test area measuring approximately 25 000 mm^2 (for example, 250 mm × 100 mm) using the ruler (5.1) and chalk or other suitable means (4.2). Record the actual area in square millimetres.

Mark two beakers (5.4) A and B. Pour 45 ml of water (4.1) into beaker A, using the measuring cylinder (5.8). Soak an absorbent cotton pad (4.3) in the water and thoroughly swab the test area with the pad. Do not allow water to drip from the pad or run off the test area. Remove the water from the surface with the absorbent cotton pad and squeeze the washings into beaker B.

In the case of bare steel, scrape the surface with the metal spatula or knife (5.2) until bright metal is exposed and is of sufficient size to conduct a representative surface retrieval or replicates the surface condition specified immediately prior to coating.

Repeat the swabbing procedure with several portions of water until all the water in beaker A has been used. If the cotton pad becomes worn out, use a fresh one and keep the used absorbent cotton pads. The swabbing procedure should take at least 10 min.

Filter the washings, using the filter paper (5.7) and the filter funnel (5.6) and collect the filtrate in the volumetric flask of capacity 50 ml (5.9). Wash the used absorbent cotton pads and the filter paper with small portions of water (total 5 ml), using beaker B and the small glass rod (5.5). Squeeze the pads and collect the washings in the flask. Fill up to the mark with water.

NOTE An alternative method for sampling is described in ISO 8502-6.

6.2 Wash-water analysis

For the assessment of chloride concentration in the wash-water, different methods can be used depending on equipment available and accuracy required. Some examples are as follows:

- ion chromatography (ISO 10304-1);
- detection method of the ion detection tube method (ISO 8502-5);
- ion selective electrode (ISO 9517);
- coulometric titration, see Annex A.

NOTE Both the ion detection tube method (ISO 8502-5) and the ion selective electrode (ISO 9517) require conditioning of the wash-water before analyses.

If all salts present in the wash-water are considered to be containing the anion chloride, conductivity measurement can be used and converted to an assumed non-specific result using ISO 8502-9.

Other methods than those listed above might be appropriate. The measurement uncertainty of the method used shall always be taken into account and reported.

Titration including mercury or chromate containing substances should be avoided due to health and environmental hazards

7 Expression of results

Calculate the amount of chloride per unit surface area, $\rho_A(\text{Cl})$, expressed in milligrams per square metre, using Formula (1):

$$\rho_A\left(\text{Cl}\right) = \frac{c \times V \cdot 10^3}{A} \tag{1}$$

where

c is the chloride concentration in the wash-water, expressed in milligrams per litre;

A is the surface area washed, expressed in square millimetres.

V is the sampling volume of water, expressed in millilitres (50 ml used in the procedure of 6.1).

Dilution, e.g. by adding acid or ion strength adjustor or from adding a small amount of the sample to a larger volume of buffer solution (required for the ion detection tube, the ion selective electrode and for the coulometric titration methods, respectively), shall be taken into account when calculating the volume to be used in Formula (1).

8 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the surface tested, including details of shape and attitude (e.g. horizontal, vertical, at an angle);

b) a reference to this part of ISO 8502, i.e. ISO 8502-2;

c) the method used for assessing the chloride concentration in the wash-water;

d) the results of the test, including the results of all individual determinations and their mean, expressed as indicated in Clause 7;

e) any deviations from the test method specified;

f) any unusual features (anomalies) observed during the test;

g) date(s) of the test.

Annex A
(informative)

Determination of chloride by coulometric titration

Principle for the method: A current is passed between a cathode and a silver-containing anode placed in a buffer solution to which a portion of a reference solution with known chloride contents is added followed by the sample. The chloride ions will react with the produced silver ions forming a precipitate. The end point can be detected by changes in solution conductivity due to the presence of excess silver ions in solution or by potential changes.

Accuracy of the method is dependent on the system used for controlling current and the end point, the buffer solution and electrodes used (affecting the current efficiency and solubility of species formed), the accuracy of the reference solution, the difference in chloride concentration between the reference and the sample and the use of the instrumentation.

If commercial instruments are used, it is vital to follow the instrument manual, using proper buffer solutions etc.

Bibliography

[1] ISO 9517, *Iron ores — Determination of water-soluble chloride — Ion-selective electrode method*

[2] ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

[3] ISO 8502-5, *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)*

[4] ISO 8502-6, *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*

[5] 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*

[6] ISO/TR 15235, *Preparation of steel substrates before application of paints and related products — Collected information on the effect of levels of water-soluble salt contamination*

ICS 25.220.10

Price based on 6 pages