INTERNATIONAL STANDARD

ISO 8502-2

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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 des chlorures sur les surfaces nettoyées

Reference number
ISO 8502-2:1992(E)

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 8502-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee 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 detector tube method*
- *Part 6: Sampling of soluble impurities on surfaces to be painted — Bresle method*
- *Part 7: Analysis of soluble impurities on surfaces to be painted — Analysis methods for field use for chloride*

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- *Part 8: Analysis of soluble impurities on surfaces to be painted — Analysis methods for fields use for sulfate*
- *Part 9: Analysis of soluble impurities on surfaces to be painted — Analysis methods for field use for ferrous salts*
- *Part 10: Analysis of soluble impurities on surfaces to be painted — Analysis methods for field use for oil and grease*
- *Part 11: Analysis of soluble impurities on surfaces to be painted — Analysis methods for field use for moisture*

Users should note that the titles to future parts 5 to 11 are working titles only and that, while it is at present planned to publish all the parts listed above, one or more may nevertheless be deleted from the work programme before publication, which may, in turn, lead to renumbering of the remaining parts.

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 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 and ISO 8501-2), may 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. Careful scraping of the surface with a metal spatula or knife during the washing, and prolonging the washing time, should remove a larger proportion of the salt.

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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

WARNING — The procedure described in this part of ISO 8502 is intended to be carried out by qualified chemists or by other suitably trained and/or supervised personnel. The substances and procedures used in this method may be injurious to health if adequate precautions are not taken. Attention is drawn in the text (see 4.4) to certain specific hazards. This part of ISO 8502 refers only to its technical suitability and does not absolve the user from statutory obligations relating to health and safety.

1 Scope

This part of ISO 8502 describes a method for assessment 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. It is normally to be used in a laboratory using washings sampled from surfaces on site.

The method is applicable to the assessment of salts that have been introduced by the cleaning procedure, or that have deposited on the surface before or after the cleaning.

NOTE 1 Although the procedure for chloride determination is generally accurate, the overall precision of the method is limited by uncertainties in the sampling procedure. In addition, traces of iron chloride at the bottom of pits are difficult to extract into the sample.

The test method cannot be used on surfaces treated with chromate or nitrite, which are commonly used as inhibitors in the water when wet blast-cleaning. This is because a chromate concentration of 10 mg/l or greater or a nitrite concentration of 20 mg/l or greater in the wash water interferes with the determination of the chloride.

An iron(III) ion concentration of 10 mg/l also interferes with the determination, but the iron(III) ions are concentrated in the rust deposits, which are removed from the test solution by filtration.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8502. At the time of publication, the editions indicated were 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 editions of the standards 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.*

ISO 8501-1:1988, *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 8501-2:—[1], *Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness — Part 2: Preparation grades of previously coated steel substrates after localized removal of previous coatings.*

3 Principle

A defined area of the steel surface is washed with a known volume of water and the chloride in the wash water is titrated with mercury(II) nitrate using a diphenylcarbazone/bromophenol blue mixed-indicator titration method developed by Clarke[2]. In the titration, mercury ions react with free chloride ions to form $HgCl_2$ which dissociates only slightly. When the chloride ions are consumed, the excess mercury ions give an intense violet colour with diphenylcarbazone, thus indicating the end-point of the titration.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water of at least grade 3 purity in accordance with ISO 3696.

4.1 Nitric acid, $c(\mathrm{HNO_3})$ about 0,05 mol/l.

Add 3,5 ml of concentrated nitric acid (ρ = 1,40 g/ml) to some water, and make up to 1 000 ml with water.

4.2 Sodium hydroxide solution, $c(\mathrm{NaOH})$ about 0,025 mol/l.

Dissolve 1,0 g of sodium hydroxide in water, transfer to a 1 000 ml volumetric flask and make up to the mark with water.

4.3 Potassium chloride, standard solution, $\rho(\mathrm{Cl})$ = 100 mg/l.

Dissolve 0,210 3 g of potassium chloride in water, transfer to a 1 000 ml volumetric flask and make up to the mark with water.

1 ml of this solution contains 0,1 mg of Cl.

4.4 Mercury(II) nitrate, standard volumetric solution, $c[\mathrm{Hg(NO_3)_2}]$ = 0,012 5 mol/l.

WARNING — Mercury(II) nitrate is toxic. Avoid contact with eyes and skin.

4.4.1 Preparation

Dissolve 4,171 g of mercury(II) nitrate hemihydrate, $[\mathrm{Hg(NO_3)_2 \cdot 0{,}5H_2O}]$ in water, transfer to a 1 000 ml volumetric flask and make up to the mark with water.

4.4.2 Standardization

Introduce by means of a pipette (5.11) into a beaker (5.5) a 20 ml aliquot of the potassium chloride standard solution (4.3). Add 5 drops of the diphenylcarbazone/bromophenol blue mixed-indicator solution (4.5) and stir. If a blue-violet or red colour develops, add nitric acid (4.1) dropwise until the colour changes to yellow and then add 1 ml excess of nitric acid. If a yellow or orange colour forms immediately on the addition of the indicator, obtain the blue-violet colour by adding sodium hydroxide solution (4.2) dropwise. Then proceed with the acidification. Titrate the yellow acidified solution with the mercury(II) nitrate solution (4.4) until a blue-violet colour persists throughout the solution. Retain the titrated solution for comparison with the blank titrated solution obtained in 6.1. Calculate the concentration of the mercury(II) nitrate solution. Note that 1 mol of mercury(II) ions is equivalent to 2 mol of chloride ions.

4.5 Diphenylcarbazone/bromophenol blue, mixed-indicator solution.

Dissolve 0,5 g of crystalline diphenylcarbazone and 0,05 g of crystalline bromophenol blue in 75 ml of ethanol, 95 % (V/V), and make up to 100 ml with ethanol.

Store in a brown bottle.

5 Apparatus and materials

5.1 Ruler and **essentially chloride-free chalk**, or other suitable means for marking the test area.

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

5.3 Metal spatula or **knife**.

5.4 Gloves, of plastics material.

5.5 Beakers, of capacity 250 ml.

5.6 Small glass rod.

5.7 Funnel.

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[1] To be published.

[2] CLARKE, F.E. : Determination of chloride in water, *Analytical Chemistry*, 22 (1950), 4, pp. 553-555.

5.8 Filter paper, approximately 120 mm diameter.

5.9 Measuring cylinder, of capacity 50 ml.

5.10 Volumetric flasks, of capacity 50 ml, 100 ml and 1 000 ml.

5.11 Volumetric pipettes, of capacity 1 ml and 20 ml.

5.12 Device for titration, preferably a digital titrator.

6 Procedure

6.1 Blank titration

Carry out a blank titration each time on water, retaining the titrated solution for comparison of end-points. Follow the procedure described in 4.4.2 but using 20 ml of water instead of the potassium chloride solution.

6.2 Washing of the surface

Carry out the test at least in duplicate. During the swabbing procedure, ensure absence of accidental contamination of the wash water by using, for example, clean plastic gloves (5.4).

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

Mark two beakers (5.5) A and B. Pour 45 ml of water into beaker A, using the measuring cylinder (5.9). Soak an absorbent cotton pad (5.2) 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 (5.3) until bright metal is exposed over at least 50 % of the test area.

Repeat the swabbing procedure with several portions of water and, if the pad is worn out, use a fresh one, continuing in this way until all the water has been used up. Keep the used absorbent cotton pads. The swabbing procedure should take at least 5 min.

Filter the washings, using the filter paper (5.8) and the funnel (5.7), and collect the filtrate in the volumetric flask of capacity 50 ml (5.10). Wash the used absorbent cotton pads and the filter papers with small portions of water (total 5 ml), using beaker B and the small glass rod (5.6). Squeeze the pads and collect the washings in the flask. Make up to the mark with water.

6.3 Wash-water titration

Carefully shake the 50 ml volumetric flask. Using a pipette (5.11), transfer 20 ml of the wash water into a clean beaker (5.5). Determine the chloride content by following the procedure given in 4.4.2 but using the wash water.

7 Expression of results

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

$$\rho_A(\text{Cl}) = \frac{(V_1 - V_0)c \times 1{,}773 \times 10^8}{A}$$

where

V_1 is the volume, expressed in millilitres, of the mercury(II) nitrate solution used for the titration of the wash water (see 6.3);

V_0 is the volume, expressed in millilitres, of the mercury(II) nitrate solution used for the blank titration (see 6.1);

c is the actual concentration, expressed in moles of $Hg(NO_3)_2$ per litre, of the mercury(II) nitrate solution, as determined in 4.4.2;

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

Report each result to the nearest 10 mg/m^2.

NOTE 2 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. Experience in the use of this method is limited and more will be needed before limits can be specified in connection with its use.

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 (ISO 8502-2);

c) the results of the test, as indicated in clause 7;

d) any deviation from the test method specified;

e) the date(s) of the test.