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 en laboratoire des chlorures sur les surfaces nettoyées

Reference number
ISO 8502-2:2005(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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Contents

Page

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 8502-2 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 8502-2:1992), which has been technically 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 1: Field test for soluble iron corrosion products* (withdrawn) *[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 the 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 water-soluble chloride* (withdrawn)
- *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*

This corrected version of ISO 8502-2:2005 incorporates the following corrections:

- in 4.5 “0,012 5 mg/l” has been changed to “0,012 5 mol/l”;
- in 4.5.2 the numerator in the equation has been changed from “$2{,}8 \times 10^{-5}$” to “$2{,}8 \times 10^{-2}$” and the volume unit has been added in the where clause, giving “V is the volume, in millilitres, of the mercury(II) nitrate solution consumed.”

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, and

c) the surface profile.

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, and
- 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 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

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 4.5) 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 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. It will normally be used in a laboratory using washings sampled from surfaces on site.

The method is applicable to the determination of salts that have been deposited on the steel surface before, during or after the cleaning operations.

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 is not applicable to surfaces treated with chromate, nitrite or amine, 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. Also the amine inhibitors can form a hydroxide boundary layer (not water soluble) over the substrate and prevent the water from contacting the underlying salt for its removal.

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.

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. For further information regarding levels of water-soluble salt contamination see ISO/TR 15235 [1].

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 3696:1987, *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 the chloride in the wash water is titrated with mercury(II) nitrate using a diphenylcarbazone/bromophenol blue mixed indicator titration method.

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, indicating the end-point of the titration.

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 Nitric acid, $c(HNO_3)$ 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.

4.3 Sodium hydroxide solution, $c(NaOH)$ 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 1 000 ml with water.

4.4 Potassium chloride standard solution, $c(Cl)$ = 0,002 8 mol/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 chloride.

4.5 Mercury(II) nitrate standard volumetric solution, $c[Hg(NO_3)_2 \cdot 0{,}5H_2O]$ about 0,012 5 mol/l.

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

4.5.1 Preparation

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

4.5.2 Standardization

Using a pipette (5.10), introduce into a beaker (5.4) a 20 ml aliquot portion of the potassium chloride standard solution (4.4).

Add 5 drops of the diphenylcarbazone/bromophenol blue mixed-indicator solution (4.6) and stir. If a blue-violet or red colour develops, add nitric acid (4.2) dropwise until the colour changes to yellow and then add 1 ml excess of nitric acid. If a yellow or orange colour forms immediately after the addition of the indicator, obtain the blue-violet colour by adding sodium hydroxide solution (4.3) dropwise. Then proceed with the acidification. Titrate the yellow acidified solution with the mercury(II) nitrate solution (4.5) 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, c, in moles per litre, of the mercury(II) nitrate solution.

$$c = \frac{2{,}8 \times 10^{-2}}{V}$$

where V is the volume, in millilitres, of the mercury(II) nitrate solution consumed.

NOTE 2 mol of chloride ions is equivalent to 1 mol of mercury(II) ions.

4.6 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, of a volume fraction of 95 %, and make up to 100 ml with ethanol.

Store in a brown bottle.

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

4.8 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.

5.11 Device for titration, preferably a digital titrator.

6 Procedure

6.1 Blank titration

In parallel with each wash-water titration, carry out a blank titration on water. Follow the procedure described in 4.5.2 but using 20 ml of water (4.1) instead of the potassium chloride standard solution (4.4), and using the titrated solution obtained in 4.5.2 as a reference for the colour at the endpoint.

6.2 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 (4.7). Record the actual area in square millimetres.