INTERNATIONAL STANDARD

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

Part 10:
Field method for the titrimetric determination of water-soluble chloride

Préparation des subjectiles d'acier avant application de peintures et de produits assimilés — Essais pour apprécier la propreté d'une surface — Partie 10: Méthode in situ pour la détermination titrimétrique du chlorure hydrosoluble

Reference number
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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 3.

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 8502-10 was prepared by Technical Committee ISO/TC35, *Paints and varnishes,* Subcommittee SC 12, *Preparation of steel substrates before the application of paints and related products*.

ISO 8502 consists of the following parts, under the general title *Preparation of steel substrates before the 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 7: Field method for the determination of oil and grease*

— *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*

— *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*

— *Part 13: Field method for the determination of soluble salts by conductometric measurement*

At the time of publication of this part of ISO 8502, parts 7, 8, 11, 12 and 13 were in course of preparation.

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 system 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.

There are several methods for the analysis of chloride in solution. At least one of them is already an International Standard, ISO 8502-2. However, most of these methods are for laboratory use and very few are suitable for field use, i.e. in conjunction with sampling in workshops, at building sites, on board ships, etc, often under severe environmental conditions.

The proposed field method for chloride and the corresponding methods of analysis that have been developed for other contaminants (such as sulfate, soluble iron corrosion products, and oil and grease) are intended to be used in conjunction with the Bresle method for the extraction of contaminants from a surface, ISO 8502-6. These methods of analysis provide results which, after application of a simple conversion factor, indicate directly the amount of contaminant per unit surface area, usually expressed in mg/m^2. Of course, the same methods of analysis can also be used in conjunction with other contaminant extraction methods.

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

Part 10:
Field method for the titrimetric determination of water-soluble chloride

CAUTION — The method described in this part of ISO 8502 involves drop titration with mercuric nitrate solution. The small quantity of titrant solution and its low concentration are not likely to make it a hazard (e.g. by ingestion). From a legal point of view, however, the solution might represent an environmental pollution hazard. To avoid this, this part of ISO 8502 specifies requirements for the safe disposal and destruction of the solution (see 7.5).

The method also involves the use of hydrogen fluoride, and particular care should be taken when handling this reagent due to its hazardous nature.

1 Scope

This part of ISO 8502 specifies a field method for the determination of water-soluble chloride by drop titration.

The method is intended mainly for use in the assessment of contaminants on a surface. It is easy for unskilled personnel to carry out and is sufficiently accurate for most practical purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 8502. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 8502 are encouraged to investigate the possibility of applying the most recent editions of the normative documents listed below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 8502-2:1992, *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.*

ISO 8502-6:1995, *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.*

3 Principle

The water-soluble contaminants are removed from the test surface by the Bresle method (see ISO 8502-6) or any other convenient method. Water, containing sodium fluoride and hydrogen fluoride, is used as the solvent. The chloride concentration in the solution is determined by drop titration with mercuric nitrate solution, using diphenylcarbazone and bromophenol blue as indicators.

The concentration of the titrant, the size of the titrant drops and the area of the test surface (normally 1 250 mm^2) are chosen so that the number of drops required for the titration multiplied by a simple conversion factor gives the surface density of chloride.

4 Reactions

The titration is carried out at a pH of 3,0 ± 0,3. When the mercuric ions in the titrant are added to the test solution, they react with the chloride ions and undissociated mercuric chloride is formed. When all the chloride ions have been consumed, excess mercuric ions react with the diphenylcarbazone indicator to produce an intense violet colour, the appearance of which indicates the end point of the titration.

5 Reagents and materials

These reagents and materials are commercially available in kit form.

5.1 Plastic beaker, of suitable size, usually about 20 ml.

5.2 Solvent, consisting of distilled or de-ionized water containing 1 g/l of sodium fluoride (NaF) and 0,8 g/l of hydrogen fluoride (HF).

CAUTION — When handling hydrogen fluoride, particular care should be taken due to the hazardous nature of this reagent.

5.3 Bottle A, volume about 20 ml, containing a 1 % solution of diphenylcarbazone plus bromophenol blue in alcohol, prepared as described in ISO 8502-2, and with a device for dropwise release of this solution (indicator A), about 0,04 ml being released with each drop. Make sure that indicator A has not been affected by ageing; check once a year by titration of chloride-containing solutions, e.g. ordinary tap water.

5.4 Bottle B, volume about 20 ml, containing a 0,5 mol/l solution of nitric acid, and with a device for dropwise release of this solution (buffer B), about 0,05 ml being released with each drop.

5.5 Bottle C, volume about 20 ml, containing a 0,036 mol/l solution of mercuric nitrate, and with a device for dropwise release of this solution (titrant C), (0,050 ± 0,002) ml being released with each drop.

5.6 Bottle D, volume about 20 ml, containing a 0,007 2 mol/l solution of mercuric nitrate, and with a device for dropwise release of this solution (titrant D), (0,050 ± 0,002) ml being released with each drop.

NOTE The concentration of titrant C is 5 times that of titrant D.

5.7 Container for liquid wastes, volume about 250 ml, for waste solutions, and containing a small piece of pure zinc, e.g. a strip weighing about 5 g, to absorb the mercury in the waste solutions by amalgamation.

6 Removal of water-soluble contaminants from the steel surface

6.1 To remove the water-soluble contaminants from the steel surface, use the Bresle method (see ISO 8502-6) or any other convenient method.

6.2 When using the Bresle method, use patch size A-1250 (compartment area 1 250 mm^2), unless another size proves to be necessary. Whatever the patch size, use a volume of solvent (5.2) which is proportional to the compartment area of the patch and equal to (3,5 ± 0,5) µl/mm^2.

7 Determination by drop titration

7.1 Collect in the plastic beaker (5.1) the solution containing the soluble contaminants to be analysed.

7.2 Add 2 drops of indicator A (see 5.3) and mix by carefully swirling the plastic beaker until the colour of the solution becomes a homogeneous blue.

7.3 Add 2 drops of buffer B (see 5.4) or as many drops as are needed to turn the solution yellow.

7.4 Add drops of titrant C (see 5.5) slowly one by one, briefly swirling the solution in the plastic beaker after each addition, until the colour of the solution turns from yellow to blue. Note the number of drops required for the colour change.

If only one drop of titrant C is required and a more accurate result is needed, replace titrant C by titrant D (see 5.6) and repeat 7.1 to 7.4.

7.5 On completion of the titration, discard the contents of the plastic beaker into the waste container (5.7).

CAUTION — When the waste container is full, the solution it contains will be mercury-free and may be disposed of without any restrictions, but the zinc strip, with which the mercury from titrant C and D will have amalgamated, cannot. Depending on how often the analysis kit (5.1 to 5.7) is used, the zinc strip (5.7) has to be replaced every 1 to 10 years. When replacing the zinc strip, the old strip shall be disposed of in accordance with legal requirements and in accordance with current practice, usually that applicable to the disposal of dentist's waste containing mercury.

8 Expression of results

8.1 If a patch of size A-1250 is used, the relationship between the number of drops required for the titration in 7.4 and the surface density of chloride is as given in Table 1. If the titration is carried out with titrant C, the conversion factor is 100. If it is carried out with titrant D, the conversion factor is 20.

Table 1 — Result of titration

Number of drops required for titration	Surface density of chloride mg/m^2			
	Solution C		Solution D	
	min.	max.	min.	max.
1	0	100	0	20
2	100	200	20	40
3	200	300	40	60
4	300	400	60	80
5	400	500	80	100

8.2 Dividing the numerical values in Table 1 by 10 gives the surface density in µg/cm^2.

If the actual compartment area of the patch is not 1 250 mm^2, multiply the result obtained in 8.1 by 1 250 divided by the numerical value of the actual compartment area in mm^2.

9 Precision

The precision of the method is determined by the difference between the max. and min. values. As can be seen from Table 1 it is independent of the surface density of chloride but depends on the concentration of the titrant.

10 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 8502 (i.e. ISO 8502-10);

b) the method used to remove the soluble contaminants from the steel surface;

c) the area of the test surface;

d) the titrant used (C or D);

e) the surface density of chloride as determined in accordance with 8.1 and 8.2;

f) the date of the test.