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

Part 12:

**Field method for the titrimetric  
determination of water-soluble ferrous ions**

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

*Partie 12: Méthode in situ pour la détermination des ions ferreux  
hydrosolubles par titrimétrie*



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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.

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 part of ISO 8502 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8502-12 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 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, 11 and 13 were in 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 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 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 a number of methods for the analysis of ferrous ions in solution. However, most of these 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 ferrous ions and the corresponding methods of analysis that have been developed for other contaminants (such as sulfate, chlorides, and oil and grease) are intended to be used in conjunction with the Bresle method for the removal 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 contaminants per unit surface area, usually expressed in mg/m<sup>2</sup>. The same method 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 12:

### Field method for the titrimetric determination of water-soluble ferrous ions

**WARNING** — The method described in this part of ISO 8502 involves drop titration with a solution of potassium dichromate. The small quantity of titrant solution and its low concentration are not likely to make it a hazard (e.g. by ingestion). Attention is drawn, however, to national and/or local legislation regarding possible environmental pollution hazards.

## 1 Scope

This part of ISO 8502 describes a field method for the determination, by drop titration, of soluble ferrous ions on steel surfaces before and/or after surface preparation.

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 it 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 indicated 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.

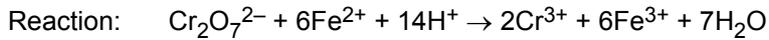
ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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 surface contaminants to be assessed are removed by the Bresle method (ISO 8502-6), or any other convenient method, using water as the solvent. After removal of contaminants and acidifying the solvent with phosphoric acid, the ferrous ion concentration of the solvent is determined by drop titration with a dichromate solution as titrant, using diphenylamine sulfonate as indicator.

The concentration of the titrant, the size of the drops and the area of the test surface (normally 1 250 mm<sup>2</sup>) are chosen so that the number of drops required for the titration multiplied by a simple conversion factor gives the surface density of water-soluble ferrous ions.



## 4 Reagents

During the analysis, use only water conforming to the requirements of at least grade 3 of ISO 3696.

NOTE Usually, distilled or de-ionized water of conductivity less than 0,5 mS/m (5 µS/cm) meets this requirement.

**4.1 Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) solution**, 85 %, in de-ionized water, the ratio by volume being 1:2 (one volume of phosphoric acid to two volumes of water).

**4.2 Indicator solution**: a colourless 0,5 % solution of the sodium salt of diphenylamine sulfonate (C<sub>6</sub>H<sub>5</sub>NHC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>Na) in de-ionized water, kept in bottle A (5.2).

Check once a year that ageing has not destroyed the indicator, by using the solution to titrate a sulfate-containing solution, e.g. ordinary tap water.

**4.3 Potassium dichromate solution**,  $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,002 \text{ mol/l}$ , for use as titrant, kept in bottle B (5.3).

## 5 Apparatus

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

**5.2 Bottle A**, volume about 30 ml, having a device for dropwise release of indicator solution (4.2), about 0,05 ml being released with each drop.

**5.3 Bottle B**, volume about 30 ml, having a device for dropwise release of titrant solution (4.3), (0,050 ± 0,002) ml being released with each drop.

## 6 Removal of water-soluble contaminants from the surface

Remove the water-soluble contaminants from the steel surface using the Bresle method given in ISO 8502-6 or any other convenient method.

When using the Bresle method, use a patch of size A-1250 (compartment area 1 250 mm<sup>2</sup>), unless another size proves to be necessary. Whatever the patch size, use a volume of solvent water which is proportional to the compartment area of the patch, i.e. (2,5 ± 0,5) µl per square mm of surface area.

## 7 Procedure

Using the procedure given in clause 5 of ISO 8502-6:1995, collect the solution containing the ferrous ions to be analysed in the plastic beaker (5.1).

Add approximately 1 ml of indicator solution (4.2) and mix by carefully swirling the plastic beaker so that the solution becomes homogeneous.

Determine the ferrous ion concentration by drop titration as follows:

Add 4 ml of phosphoric acid solution (4.1) and mix by carefully swirling the plastic beaker so that the solution becomes homogeneous.

Add 1 drop [(0,050 ± 0,002) ml] of the titrant solution (4.3) as blank.

Add further drops of the titrant solution slowly one by one, briefly swirling the solution in the plastic beaker after each addition, until the colour of the solution turns from colourless through greyish blue to violet. Note the number of drops required for the colour change (not counting the first drop added as blank).

NOTE The colour change is not very pronounced. The addition of more drops does not give any further change.

## 8 Expression of results

If a Bresle patch of size A-1250 is used, the relationship between the number of drops required for the titration in clause 7 and the surface density of the ferrous ions is as given in Table 1. The conversion factor is 27 mg/m<sup>2</sup> per drop.

Table 1 — Result of titration

Number of drops required for titration in clause 7	Surface density of ferrous ions mg/m <sup>2</sup>	
	min.	max.
1	0	27
2	27	54
3	54	81
4	81	108
5	108	135
6	135	162
7	162	189

If the compartment area of the Bresle patch is not 1 250 mm<sup>2</sup>, multiply the result by 1 250 and divide by the numerical value of the actual compartment area in mm<sup>2</sup>.

Express the result as the range of the surface density of ferrous ions, in mg/m<sup>2</sup>.

## 9 Precision

The precision of this method is independent of the surface density of the ferrous ions (see Table 1).

## 10 Test report

The test report shall contain at least the following information:

- a reference to this part of ISO 8502 (i.e. ISO 8502-12:2003);
- all details necessary for complete identification of the surface tested;
- the method used to remove soluble contaminants from the steel surface;
- the area of the test surface;

- e) the results of the test, expressed as the surface density of ferrous ions and determined in accordance with clause 8;
- f) the date of the test.



