INTERNATIONAL STANDARD

ISO 8502-11

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

Part 11:
Field method for the turbidimetric determination of water-soluble sulfate

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

Partie 11: Méthode in situ pour la détermination turbidimétrique des sulfates hydrosolubles

Reference number
ISO 8502-11:2006(E)

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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 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 8502-11 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 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 11: Field method for the turbidimetric determination of water-soluble sulfate*
- *Part 12: Field method for the titrimetric determination of water-soluble ferrous ions*

Parts 1 and 10 have been withdrawn. A field method for the determination of oil and grease is under consideration as Part 7.

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 sulfates 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.

This field method for sulfates, and the corresponding methods of analysis that have been developed for other contaminants (such as ferrous ions, 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^2. The same method of analysis can also be used in conjunction with other contaminant extraction methods.

INTERNATIONAL STANDARD **ISO 8502-11:2006(E)**

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

Part 11:
Field method for the turbidimetric determination of water-soluble sulfate

1 Scope

This part of ISO 8502 describes a field method for the assessment of the surface density of water-soluble sulfate on steel surfaces before and/or after surface preparation.

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*

ISO 8502-6:2006, *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

Water-soluble sulfate and other salts are removed from a specific part of a steel surface, using the Bresle method, using water conforming to the requirements of grade 3 of ISO 3696:1987 as solvent (see ISO 8502-6).

A precipitation reagent, $Ba(NO_3)_2$ or $BaCl_2$, and a flocculant are added to the solution of removed sulfate, to produce a colloidal solution of $BaSO_4$. The spectral absorbance of the dispersion depends on the sulfate content and can be used to determine the mass concentration of the removed sulfate in the solution. The corresponding surface density of the sulfate is then calculated by a simple formula.

4 Reagents

4.1 **Water**, conforming to the requirements of grade 3 of ISO 3696:1987.

4.2 **Precipitation reagent**, $Ba(NO_3)_2$ or $BaCl_2$, as specified by the manufacturer of the turbidimeter (5.1).

4.3 **Flocculant**, as specified by the manufacturer of the turbidimeter (5.1).

5 Apparatus

5.1 Turbidimeter, for measurement of the spectral absorbance of a colloidal solution. It shall be calibrated to indicate on a scale or a digital display the mass concentration of SO_4^{2-} in that solution within a range from 0 mg/l to at least 80 mg/l.

NOTE Turbidimeters are commercially available and usually delivered in kits also containing the required reagents (4.2 and 4.3), the latter sometimes already mixed together in convenient amounts for instant use.

5.2 Two measurement cells, designed for and belonging to the turbidimeter (5.1), designated the first cell and the second cell.

5.3 Plastics beaker.

5.4 Standard adhesive patch, conforming to the requirements of 4.1 in ISO 8502-6:2006, e.g. of size A-1250.

5.5 Syringe, conforming to the requirements of 4.2 in ISO 8502-6:2006.

5.6 Thermometer for measuring ambient air temperature, capable of being read to the nearest 0,5 °C and graduated at 0,5 °C intervals.

6 Procedure

6.1 Preparation of solvent

Pour as much solvent (water) (4.1) into the plastics beaker (5.3) as required for two measurements with the turbidimeter (5.1). Record the volume, e.g. 20 ml, to the nearest 0,5 ml.

6.2 Removal of water-soluble contaminants from the surface

Use the procedure given in Clause 5 of ISO 8502-6:2006 with the following specific additional requirements:

a) Fill the syringe (5.5) with as much as required of the solvent prepared in 6.1 (see 5.3 of ISO 8502-6:2006).

b) The total extraction time shall be 10 min ± 1 min. During that time, repeat the following 10 times:

   Without removing the syringe needle from the adhesive patch, inject the solvent into the patch compartment and then suck it back into the syringe cylinder (see 5.6 of ISO 8502-6:2006).

At the end of the 10th cycle, retrieve and transfer as much as possible of the solution containing the contaminants removed from the surface covered by the patch compartment to the beaker (5.3), thus restoring its content to the original volume (see 6.1) (e.g. 20 ml) as described in 5.7 of ISO 8502-6:2006. See also 6.4, second paragraph.

6.3 Blank test and preparation of the colloidal solution

6.3.1 Pour half of the solution prepared in 6.2, e.g. 10 ml, into the first cell (see 5.2) and carry out the following blank test.

6.3.2 Place the first cell in the turbidimeter and record the value indicated, which represents zero mass concentration of SO_4^{2-}.

6.3.3 To the remaining half of the solution prepared in 6.2, add amounts of precipitation reagent and flocculant (4.2 and 4.3) as specified by the manufacturer of the turbidimeter. This will make the solution colloidal and spectrally absorbant. See also 6.4, second paragraph.

6.3.4 Pour this colloidal solution into the second cell (see 5.2), and install the second cell in the turbidimeter.

6.4 Turbidimetric measurement

Record the value indicated by the turbidimeter for the colloidal dispersion in the second cell. Subtract the blank test value (see 6.3.2) and record the difference, which represents the mass concentration of SO_4^{2-} in the solution prepared in 6.2.

If the measurement range of the turbidimeter is too narrow, dilute the solution prepared in 6.2 by adding as much fresh solvent as necessary to reduce sufficiently the mass concentration, e.g. 10 ml or 20 ml. Record the resulting total volume of solution in the beaker, e.g. 30 ml or 40 ml. Do this before adding the precipitation reagent and flocculant.

7 Calculation and expression of results

Calculate the surface density of water-soluble sulfate, ρ_A, in kg/m², using the equation:

$$\rho_A = \frac{m}{A} \tag{1}$$

where

m is the mass of water-soluble sulfate removed from that part of the surface covered by the patch compartment, in kg;

A is the area of that part of the surface, in m².

In this case, m is given by the equation:

$$m = V \times \rho_B \tag{2}$$

where

V is the volume of the solution prepared in 6.2, after possible dilution and before the blank test, in m³;

ρ_B is the mass concentration of SO_4^{2-} in that solution (see 6.4, second paragraph), in kg/m³.

From Equations (1) and (2) it follows that:

$$\rho_A = \frac{V \times \rho_B}{A} \tag{3}$$

EXAMPLE

If V = 20 ml (see 6.2) and A = 1 250 mm² (see 5.4), it follows that ρ_A is given by the equation:

$$\rho_A = \frac{20 \times 10^{-6}}{1\,250 \times 10^{-6}} \times \rho_B$$

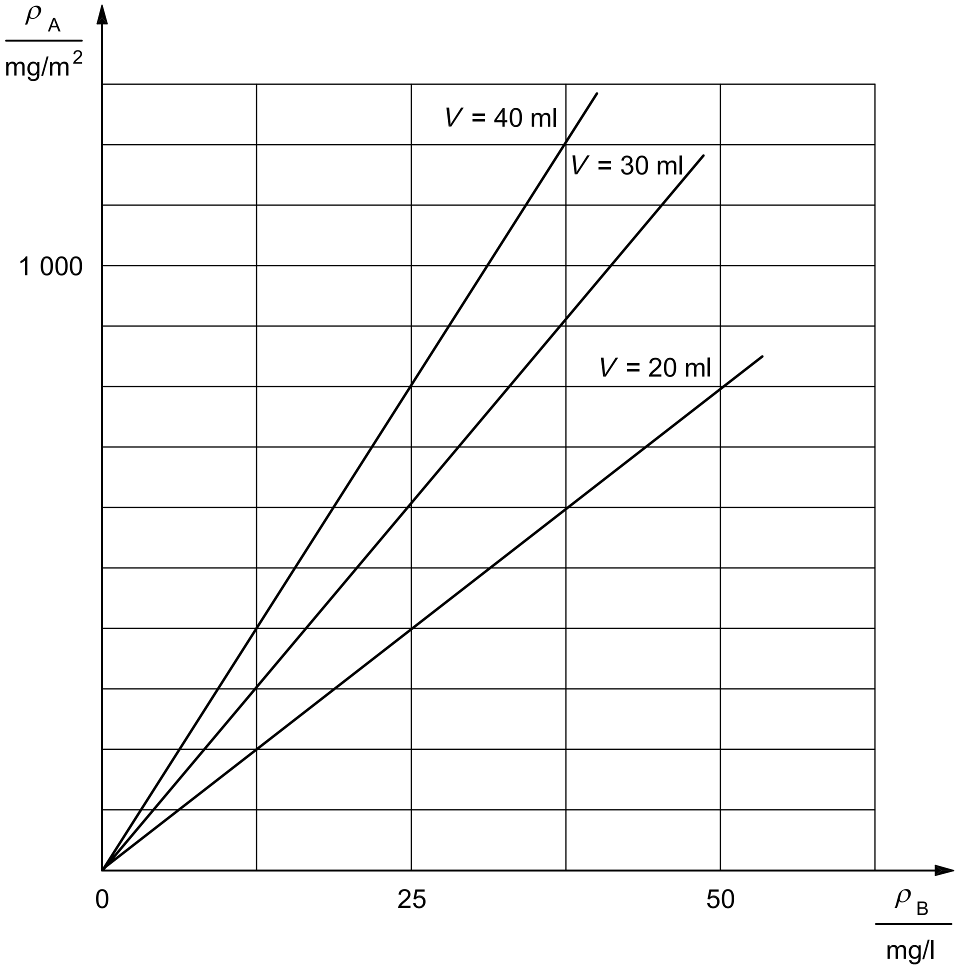
$$= 16 \times 10^{-3} \times \rho_B \tag{4}$$

Equation (4) gives ρ_A expressed in kg/m², provided that ρ_B is given in kg/m³ (1 kg/m³ = 1 000 mg/l).

Multiplying the numerical value obtained from Equation (4) by 10^6 gives ρ_A in mg/m².

Multiplying the numerical value obtained from Equation (4) by 10^5 gives ρ_A in µg/cm^2.

Inserting different values of V in Equation (3) for other volumes of the solution prepared in 6.2 gives equations similar to Equation (4). Three straight lines corresponding to three different equations are plotted in Figure 1.



NOTE Each straight line corresponds to a different volume of solution in the beaker. In each case, the patch size is A-1250 in accordance with ISO 8502-6.

Figure 1 — Surface density ρ_A of sulfate as a function of the mass concentration ρ_B

8 Precision

Various factors have an influence on the precision of the method, such as variations in temperature. Very little, if anything, has been published on these matters. Practical experience shows, however, that most variations have a negligible influence on the overall precision.

No collaborative tests have been carried out to generate precision data. Such data will be added when it is available.

9 Test report

The test report shall contain at least the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this part of ISO 8502 (i.e. ISO 8502-11:2006);

c) details of the test procedure, including:

   1) the measurement range of the turbidimeter (5.1),

   2) the area of the test surface (see 5.4),

   3) the volume of the solution prepared in 6.2;

d) the results of the test, including the results of any individual determinations and their mean, calculated as specified in Clause 7, including:

   1) the mass concentration of SO_4^{2-} in the solution (see 6.4),

   2) the surface density of water-soluble sulfate as determined by Equation (3) in Clause 7;

e) any deviations from the procedure given in Clause 6;

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

g) the date of the test.

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