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

Part 9:

Field method for the conductometric determination of water-soluble salts

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

*Partie 9: Méthode in situ pour la détermination des sels solubles dans l'eau
par conductimétrie*

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Reference number
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Foreword

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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 8502-9 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*

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- *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 determination of oil and grease*
- *Part 8: Field method for 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 chloride*

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.

This part of ISO 8502 describes a field method for the assessment of the total amount of water-soluble salts, the salts being regarded as forming one single contaminant. The more aggressive contaminants causing corrosion and blistering (the ionic species) can easily be dissolved off and determined rapidly by this method. Consequently, the less aggressive and not so easily dissolved minor part of contaminant will remain un-assessed. For additional information on the test method, its potential and its limitations, see BRESLE, Å., Conductometric determination of salts on steel surfaces, *MP (Materials Performance)*, June 1995, Vol. 34, No. 6, pp. 35-37, NACE International, Houston TX, USA.

Rusty steel substrates, particularly those 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 water-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 blister formation and accumulations of rust that destroy the adhesion between the substrate and the applied protective coating.

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

Part 9:

Field method for the conductometric determination of water-soluble salts

1 Scope

This part of ISO 8502 describes a field method for the assessment of the total surface density of various water-soluble salts (mostly chlorides and sulfates) on steel surfaces before and/or after surface preparation.

The individual surface densities of chlorides, sulfates, etc., cannot be determined by this method.

This method assesses ionic contaminants only. These represent the greater part of the contamination.

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 agreement 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 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 salts on the given area of the steel surface are dissolved by the Bresle method (see ISO 8502-6), using water as solvent. The conductivity of the solution thus obtained is measured. Finally, the total surface density of the salts in this area is calculated by a simple but sufficiently accurate equation.

4 Solvent

Water of at least grade 3 purity in accordance with ISO 3696.

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

5 Apparatus and materials

5.1 Conductometer, with temperature compensation and sufficient range, e.g. from 0 mS/m (0 μ S/cm) to 200 mS/m (2000 μ S/cm).

5.2 Glass beaker, of convenient size and shape for housing the electrode end of the conductometer (5.1) during measurement.

5.3 Standard adhesive patch, as specified in 4.1 of ISO 8502-6:1995, e.g. of size A-1250.

NOTE — The patch should not cause any noticeable contamination of the extraction liquid. Certain patches are available today which are guaranteed to cause an ionic contamination of less than 7 mg/m², which is generally satisfactory. If there is no guarantee or if improved accuracy is required, then a blank test is recommended.

5.4 Syringe, as specified in 4.2 of ISO 8502-6:1995.

6 Procedure

6.1 Preparation of water and blank test

6.1.1 Pour into the beaker (5.2) an amount of water (clause 4) that is just large enough for the operation of the conductometer (5.1). Usually a volume between 10 ml and 20 ml is needed. To prevent foreign matter inside the beaker and syringe, and on the conductometer probe, from influencing the result, carry out the following blank test.

6.1.2 Completely fill the syringe (5.4) with water from the beaker. Then empty the syringe back into the beaker.

6.1.3 Immerse the electrodes of the conductometer fully in the water in the beaker and agitate gently. Record the conductivity (γ_1) and the units in which it is expressed, for example μ S/cm.

6.2 Removal of salts from the steel surface

6.2.1 Follow the procedure specified in clause 5 of ISO 8502-6:1995, subject to the following specific requirements.

6.2.2 Fill the syringe with about one-quarter of the water contained in the beaker.

6.2.3 After 1 min, suck the water back into the syringe cylinder (see 5.6 of ISO 8502-6:1995).

6.2.4 Without removing the syringe needle from the patch, re-inject the water into the patch compartment and then suck the water back into the syringe cylinder. Repeat until 10 cycles of injection and sucking have been completed (see 5.7 of ISO 8502-6:1995).

6.2.5 At the end of the 10th cycle, retrieve as much as possible of the water from the patch compartment and transfer to the beaker (5.2), thus restoring its content to nearly the original volume in 6.1.1 (see 5.8 of 8502-6:1995).

6.3 Conductometric measurement

Immerse the electrodes of the conductometer fully in the now contaminated water in the beaker, and record the conductivity (γ_2) expressed in the same units as in 6.1.3.

7 Expression of results

The total surface density ρ_A of the salts is given by the equation

$$\rho_A = \frac{m}{A} \quad \dots(1)$$

where

m is the mass of salts dissolved from that part of the surface which is covered by the patch compartment;

A is the area of that part of the surface.

In this case, m is given by

$$m = c \cdot V \cdot \Delta\gamma \quad \dots(2)$$

where

c is an empirical constant approximately equal to $5 \text{ kg} \cdot \text{m}^{-2} \cdot \text{S}^{-1}$ (see also clause 8);

V is the original volume of water in the beaker (see 6.1.1);

$\Delta\gamma$ is the change in conductivity, i.e. the difference between the conductivity measured in 6.3 (γ_2) and the conductivity measured in 6.1.3 (γ_1).

From (1) and (2), it follows that

$$\rho_A = \frac{c \cdot V \cdot \Delta\gamma}{A} \quad \dots(3)$$

Since $c = 5 \text{ kg} \cdot \text{m}^{-2} \cdot \text{S}^{-1}$, and if for example $V = 10 \text{ ml}$ (6.1.1) and $A = 1250 \text{ mm}^2$ (5.3), it follows that

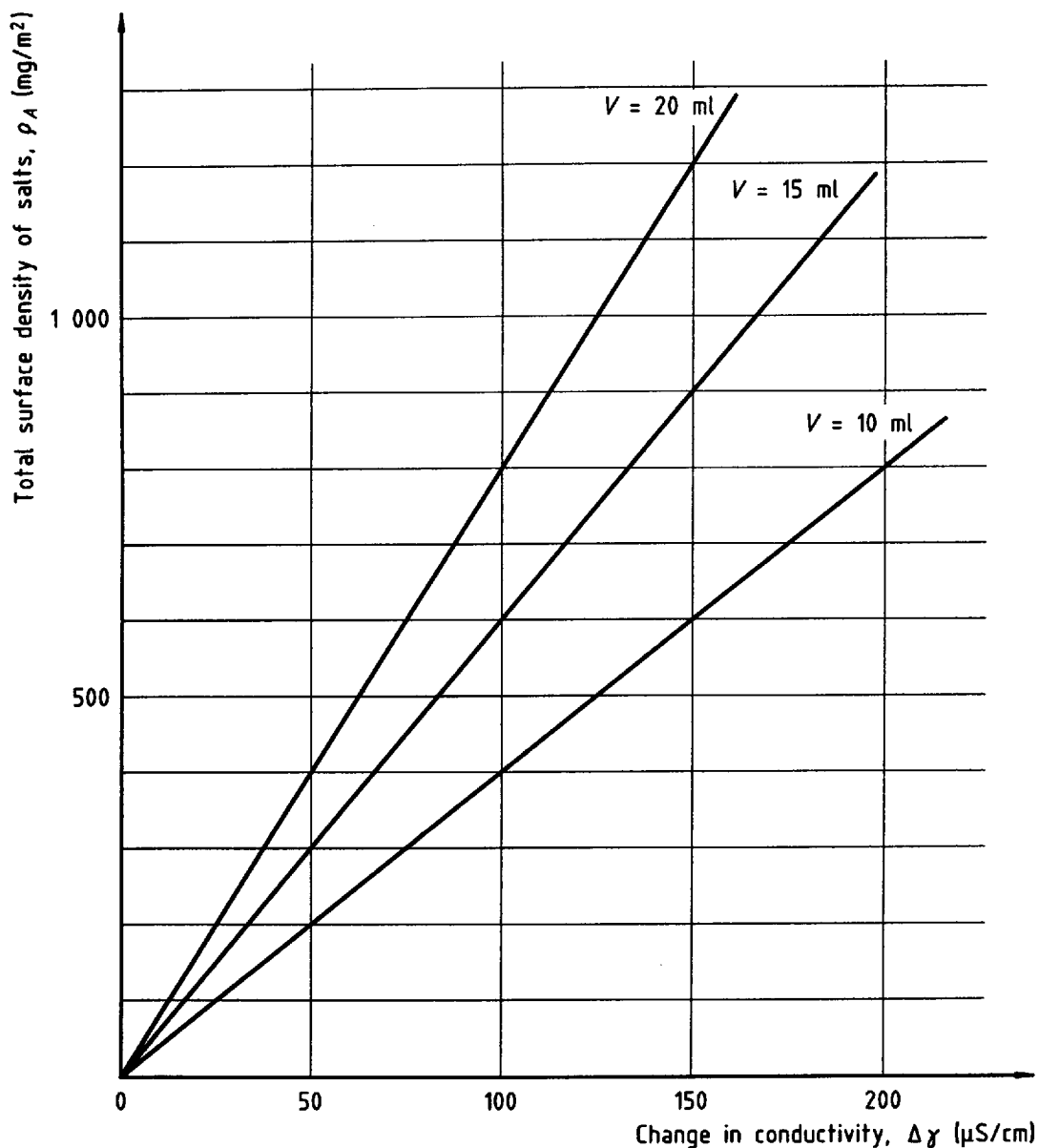
$$\rho_A = \Delta\gamma \cdot 40 \cdot 10^{-3} \text{ kg} \cdot \text{m}^{-1} \cdot \text{S}^{-1} \quad \dots(4)$$

Equation (4) gives ρ_A expressed in the SI unit kg/m^2 .

Multiplying the numerical value obtained from equation (4) by 10^6 gives ρ_A in mg/m^2 .

Multiplying the numerical value obtained from equation (4) by 10^5 gives ρ_A in $\mu\text{g/cm}^2$.

Inserting different values of V in equation (3) for other volumes of water 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 water in the beaker. In each case, the patch size is A-1250, in accordance with ISO 8502-6.

Figure 1 — The total surface density ρ_A of the salts as a function of the change in conductivity $\Delta\gamma$

8 Accuracy

The accuracy of the method depends on the accuracy of the empirical constant c in equation (3), and on the accuracy to which the variables in that equation can be determined. But other factors will also have an influence on the accuracy, such as variations in temperature. Very little has been published on these matters. Practical experience has shown, however, that most variations have negligible influence on the overall accuracy, except that in the constant c , which is dependent upon the types of salt dissolved in the water in 6.2.

In the majority of cases, the following types of ion are pre-dominant in the water: Cl^- , SO_4^{2-} , HCO_3^- , Na^+ , Ca^{2+} and Fe^{2+} . Other types of ion may also be present. Usually, however, their contribution to the variation in the constant c is only marginal.

The value of the constant c ($5 \text{ kg} \cdot \text{m}^{-2} \text{S}^{-1}$) given in clause 7 is representative of what can be called normal conditions. It is based on calculations of the total conductivity from the specific conductivities of the individual ions extracted from ten rusty steel specimens. Should the conditions be more extreme, e.g. due to some particular industry in the vicinity, the variation in the constant c may amount to about $\pm 12 \%$. This conclusion is drawn from calculations that are based on the known specific conductance of each type of ion.

For more information about the method, see the introduction.

9 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 8502 (i.e. ISO 8502-9);
- b) the conductometer measurement range used in 5.1;
- c) the conductivities measured in 6.1.3 and 6.3;
- d) the total surface density of the salts;
- e) any deviations from the procedure specified in 6.2;
- f) the date of the test.

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