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Standard Practice

Methods of Validating Equivalence to ISO 8502-9 on Measurement of the Levels of Soluble Salts

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Foreword

The purpose of this standard practice is to define a method that shows equivalence of other methods for measuring the level of contamination of soluble salts on surfaces to the Bresle patch method defined by ISO⁽¹⁾ 8502-9.¹ This standard practice provides a way to establish equivalence by testing and comparing results of the tests to meet established criteria that would be achieved using the method specified in ISO 8502-9. Equivalence is evaluated at three salt levels (30 mg/m², 50 mg/m², and 85 mg/m²) on three surface conditions (grit blasted steel, zinc silicate preconstruction primer on steel, and rusted steel).

This standard is intended for use by engineers, specification writers, test equipment suppliers, contractors, and anyone testing for soluble salts on surfaces using ISO 8502-9 and considering use of equivalent methods for this purpose. It provides a standard method to show such equivalence to the results obtained using ISO 8502-9.

The definition of equivalence as used in this standard is “an alternative tool, method, or procedure that predictably and reliably provides the same measurement values as testing in full accordance with ISO 8502-9 would provide under the same circumstances, i.e., that measures the total salt contamination amount at an extraction rate equal to the method given in ISO 8502-9, and presents the results as total surface density of the salts as described in ISO 8502-9, expression of results, and accuracy of the determination.”

The validation of the equivalence of a method must be made in a laboratory.

This standard was originally issued in 2008 and revised in 2010 by Task Group (TG) 392, “Measurement of Soluble Salts on Marine Structures.” TG 392 is administered by Specific Technology Group (STG) 44, “Marine Corrosion: Ships and Structures,” and sponsored by STG 04, “Coatings and Linings, Protective: Surface Preparation.” It is issued by NACE International under the auspices of STG 44.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*. The terms *shall* and *must* are used to state a requirement and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

⁽¹⁾ International Organization for Standardization (ISO), 1 ch. de la Voie-Creuse, Case postale 56, CH-1211 Geneva 20, Switzerland.

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Section 1: General

1.1 ISO 8502-9 Range of Variance

1.1.1 The assessment and determination of surface contamination (by salts) prior to application of protective coatings is critical to their service life expectancy. Determination of the level of surface cleanliness is performed using a field method for the conductometric determination of water-soluble salts in accordance with ISO 8502-9. As a part of that method, the extraction of soluble salt contaminants for analysis is performed in accordance with ISO 8502-6²—the Bresle method. The field execution of this method involves using a syringe to inject deionized water into the Bresle patch, washing the substrate surface inside the patch, then extracting the test water for direct measurement of conductivity. Details of this procedure are provided in Appendix A (mandatory). Once a conductivity value is determined by the conductivity meter, expressed as microsiemens per centimeter ($\mu\text{S}/\text{cm}$), ISO 8502-9 provides a procedure to calculate the equivalent mass of the surface concentration or total surface density of the salts.

1.1.2 Step-by-step execution of the ISO 8502-6 and ISO 8502-9 test methods introduces a number of potential variances during the field evaluation. Some examples include background salt contamination in the Bresle patch itself, the sensitivity and resolution of the conductivity meter, cycle time of the test water inside the Bresle patch, human errors in measuring and injecting the test water, and general operator experience. These individual process variances combine to create an overall variance in the ISO 8502-9 test method.

1.1.3 The test method defined in ISO 8502-9 allows certain execution parameters of the test (e.g., size of adhesive patch, volume of test solution, and time of dissolution of salts inside the adhesive patch—the dwell time) to vary. This standard adopted best practices and has set the parameters in Appendix A. With a dwell time at a minimum of 90 seconds, the variance from operator error was found to be reduced dramatically when the operator timing is not perfect. Hence, a dwell time of 90 seconds was adopted in Appendix A. For the purpose of defining equivalence, the procedure in Appendix A shall be used to create the reference values to which the candidate method will be validated.

1.1.4 Bresle patches from different manufacturers or manufacturing lots may have different levels of background contamination. Specifications for salt contamination include the contribution of this background contamination so that the operator in the field does not have to subtract a background value to report the test results. Appendix A therefore includes a provision to correct the ISO 8502-9 test results to a consistent offset contributed by Bresle patch background contamination so that this standard gives consistent results when tests are performed with Bresle patches from different manufacturers or manufacturing lots.

1.1.5 The range of variance in ISO 8502-6 and 8502-9 has been demonstrated by extensive laboratory tests.³ The precision of a single ISO 8502-9 test result was determined to be $\pm 8.2 \text{ mg}/\text{m}^2$ in the salt level range of 30 to 80 mg/m^2 . The absolute variance, and not the relative or percent variance, was found to be constant in this range. See Appendix B (non-mandatory) for details about these tests and the data analysis.

1.1.6 Any equivalent tools, methods, or procedures must show that they meet the same criteria, thereby providing the same measures of soluble salt level, and be within the same range of variance, as would be produced by following the procedure in ISO 8502-9.

1.1.7 The objective of this standard is to determine whether methods other than the Bresle method are suitable alternatives for measuring salt contamination in the field. Although tests may be performed on flat, horizontal surfaces for ease of use, each method should also be capable of performing measurements on vertical and overhead surfaces. When such measurements cannot be performed on vertical or overhead surfaces, this limitation must be noted in the validation report (Section 3).

1.2 Reasons for Salt Measurement

1.2.1 It is well known that applying protective coatings over salt contamination may cause degradation of the coating/substrate interface. This may lead to corrosion of the substrate and detachment of the coating. These effects reduce the expected service life of the coating.⁴

1.2.2 Different salt ions influence the rate of corrosion under coatings differently. However, most common salts promote corrosion and have a negative effect on the service life of coatings.

1.2.3 Salts on a surface under a coating can create an osmotic cell, drawing water into and through the coating. The osmotic pressure generated can cause blisters to form in the coating.

1.2.4 The general relationship between the amount of salt contamination under the coating film and consequent deterioration of the performance may be considered to be linear.⁵

1.2.5 Although virtually unobtainable, a zero measure of salt contamination may be considered the only "safe" level before application of coating films. Therefore, the maximum quantity of salt contamination allowed in a specification should be based on a holistic risk/cost analysis.

Section 2: Test Methods

2.1 Test Considerations

2.1.1 For the purposes of this standard, all references to performing a test by ISO 8502-9 shall mean performing a test by following the test method in Appendix A.

2.1.2 Alternative test methods may produce raw test results with values that are different from those produced by ISO 8502-9. It is valid to use a single mathematical correction factor to produce equivalence of alternative test results to those produced by ISO 8502-9.

2.1.3 Failed measurements may be removed from the data sets that are used for data analysis in Paragraph 2.4.

2.1.3.1 In this standard, "failed measurement" is defined as a failure of the test method that can be verified independently of the final test result. For example, ISO 8502-9 failures can include leaking of water from the cell during the test, conductivity meter failing a conductivity calibration check using a known standard solution, or failure of the operator to comply with the test method.

2.1.3.2 Data removed from the analysis as a result of failures of this nature must be documented in the final report, including the reason for deeming them failed measurements.

2.2 Preparation of Test Panels

2.2.1 Surface Conditions of Test Panels: Laboratory tests shall be performed on carbon steel test panels (e.g., standard grade copper-free hot-rolled carbon steel in accordance with ASTM⁽²⁾ A366). Appendix C (nonmandatory) provides recommendations on the preparation of test panels. Three surface conditions shall be tested:

2.2.1.1 Surface Condition A: zinc shop-primer-coated new steel.

2.2.1.2 Surface Condition B: non-shop-primed steel rusted to rust grade C in accordance with ISO 8501-1.⁷

⁽²⁾ ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.