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MARITIME SAFETY COMMITTEE
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Agenda item 7

MSC 88/INF.3
3 September 2010
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SHIP DESIGN AND EQUIPMENT

Revised standard for alternatives to testing the salt limit as related to resolution MSC.288(87)

Submitted by NACE International

SUMMARY

Executive summary:	This information document provides the revised NACE standard SP0508-2010 as requested by the Committee. This standard is an alternative for testing the salt limit in table 1 of Performance standards for protective coatings for cargo oil tanks – resolution MSC.288(87).
Strategic direction:	5.2
High-level action:	5.2.1
Planned output:	No related provisions
Action to be taken:	Paragraph 2
Related documents:	MSC 88/7/1; MSC 87/26 (paragraph 7.25); MSC 87/7/6 and DE 53/7/1

1 NACE International submits for the information of the Committee a standard that allows for alternative technologies to prove their equivalence to ISO 8502-9; to test for the salt limit on the substrate. As instructed by the Committee, NACE International has revised the standard by requiring evaluation at three salt levels (30, 50 and 85 mg/m^2). The standard, SP0508-2010 Item No. 21134, titled "Standard practice methods of validating equivalence to ISO 8502-9 on measurement of the levels of soluble salts" was revised by Task Group 393 and ratified by the Board of Directors on 19 August 2010.

Action requested of the Committee

2 The Committee is invited to consider the information provided and take action as appropriate.

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ANNEX

NACE SP0508-2010
Item No. 21134

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. 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/m^2, 50 mg/m^2, and 85 mg/m^2) 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 result 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.

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

NACE International
Standard practice

Methods of validating equivalence to ISO 8502-9 on measurement of the levels of soluble salts

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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 8502-6—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 (µS/cm), ISO 8502-9 provides a procedure to calculate the equivalent mass of the surface concentration as 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. The precision of a single ISO 8502-9 test result was determined to be ± 8.2 mg/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 (nonmandatory) 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.

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.

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:

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

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; and

2.2.1.3 Surface Condition C: new steel blasted to Sa 2½ in accordance with ISO 8501-1, using aluminum oxide to achieve a surface profile of 50 to 75 μm (2.0 to 3.0 mil).

2.2.2 Salt Level: Three target levels of salt contamination shall be tested:

2.2.2.1 30 mg/m^2;

2.2.2.2 50 mg/m^2; and

2.2.2.3 85 mg/m^2.

2.2.3 Test condition: A test condition is defined in this standard as a unique combination of surface condition and salt level. Laboratory tests shall be performed at the five test conditions listed in Table 1:

Table 1
Test Conditions

Test Condition	Surface Condition	Target Salt Level (mg/m^2)
1	A	50
2	B	50
3	C	50
4	C	30
5	C	85

2.2.4 Test panel area required: A sufficient number and area of test panels shall be prepared to accomplish all the testing required in this standard. The minimum test panel dimensions shall be 200 x 150 mm (8 x 6 in). No area of test panel may be measured twice for this protocol. Test panels should be adequately sized to allow performance of the test plan in paragraph 2.3 and the background contamination testing in paragraph 2.2.6, and to make allowances for the potential of failed tests and for test panels failing the criteria in Paragraph 2.4.1.

2.2.5 The procedures outlined in paragraphs 2.2.6 through 2.2.8 must be accomplished for each surface condition listed in paragraph 2.2.1.

2.2.6 Maximum background contamination of test panels: Test panels shall be washed with deionized water. After drying, five positions shall be randomly assigned for testing. These positions shall be tested in accordance with ISO 8502-9. If the maximum value of any one test is greater than 18 mg/m^2, this washing, drying, and testing step shall be repeated.

2.2.7 Salt composition: A solution with the following mass ratio of salts shall be used to dope all the test panels: 24.3% Na_2SO_4, 22.1% $NaNO_3$, and 53.6% NaCl. The solvent in the solution shall be exclusively type II water as defined by ASTM D1193.

2.2.8 Salt solution shall be distributed on the test panels in a way that will yield test results that comply with the criteria in paragraphs 2.4.1 and 2.4.2. Appendix C provides recommended methods for application of salt solution to test panels.

2.3 Test measurements

The following procedures shall be used for test measurements.

2.3.1 Develop a test plan that delivers the following test results, and then perform the tests in accordance with the test plan.

2.3.1.1 A minimum of 10 test results from the Bresle extraction test method (ISO 8502-9) at each test condition (see Table 1).

2.3.1.2 A minimum of three test results from the Bresle extraction test method (ISO 8502-9) for each test panel.

2.3.1.3 A minimum of 20 test results from the candidate method at each test condition (see Table 1).

2.3.2 Assign positions on the test panels in such a way that the ISO 8502-9 test results are widely and uniformly distributed across the entire surface of each test panel. Randomly assign the remaining positions on the test panels to the candidate method.

2.3.3 For each test panel, the first two tests and the last test shall be done using ISO 8502-9. Any remaining ISO 8502-9 tests, and the candidate method, shall be done in random order.

2.4 Data analysis

2.4.1 Validate the ISO 8502-9 measurement capability and uniformity of salt distribution for each test panel.

2.4.1.1 Apply the criteria in paragraphs 2.4.1 through 2.4.3 to the ISO 8502-9 test results of each test panel, one test panel at a time. If a test panel fails any one of the criteria in paragraphs 2.4.1 through 2.4.3, all test results from that test panel must be deleted from the data analysis;

2.4.1.2 The difference between the highest and lowest test results shall be less than 12 mg/m^2;

2.4.1.3 The average of the test results shall be within ± 5 mg/m^2 of the target salt level for that test condition (see Table 1); and

2.4.1.4 If test results are deleted because a test panel failed the criteria in paragraphs 2.4.1 through 2.4.3, the test results used for the data analysis must still include a minimum of 10 test results from ISO 8502-9 and 20 test results from the candidate method for each test condition (see Table 1). If the number of test results falls below the minimum number, an appropriate number of new test panels must be prepared and tested to add to the data set.

2.4.2 Validate the ISO 8502-9 measurement capability and uniformity of salt distribution for the data set.

2.4.2.1 The standard deviation of the ISO 8502-9 test results at 50 mg/m^2 (combining test conditions 1, 2, and 3 in Table 1) shall be less than 5.1 mg/m^2;

2.4.2.2 The standard deviation of the ISO 8502-9 test results at 30 mg/m^2 (test condition 4 in Table 1) shall be less than 5.3 mg/m^2;

2.4.2.3 The standard deviation of the ISO 8502-9 test results at 85 mg/m^2 (test condition 5 in Table 1) shall be less than 5.3 mg/m^2;

2.4.2.4 If the test results from just a few test panels are causing the standard deviation to be above the limit in paragraphs 2.4.2.1, 2.4.2.2, or 2.4.2.3, all the test results from those test panels may be removed from the data set. It is not permissible to delete only single test results to meet this criterion; and

2.4.2.5 If test results are deleted because of the criteria in paragraphs 2.4.1 through 2.4.3, the set of test results used for the data analysis must still include a minimum of 10 test results from ISO 8502-9 and 20 test results from the candidate method for each test condition (see Table 1). If the number of test results falls below the minimum number, an appropriate number of new test panels must be prepared and tested to add to the data set.

2.4.3 Validate the Candidate Method

2.4.3.1 The candidate method shall be deemed equivalent to ISO 8502-9 if it meets all of the following criteria (see Appendix B for basis for variability and bias requirements):

2.4.3.1.1 The standard deviation of the candidate method test results at 50 mg/m^2 (combining test conditions 1, 2, and 3 in Table 1) shall be less than 4.8 mg/m^2;

2.4.3.1.2 The standard deviation of the candidate method test results at 30 mg/m^2 (test condition 4 in Table 1) shall be less than 5.3 mg/m^2;

2.4.3.1.3 The standard deviation of the candidate method test results at 85 mg/m^2 (test condition 5 in Table 1) shall be less than 5.3 mg/m^2; and

2.4.3.1.4 The bias of the test method at each test condition shall be less than ± 4.2 mg/m^2 as shown in equation (1).

$$\text{bias}_i = \left\{ \bar{y}_{B,i} - \bar{y}_{C,i} \right\} \qquad (1)$$

where:

$\bar{y}_{B,i}$ = average of ISO 8502-9 method test results at test condition *i* = 1, 2, 3, 4, 5 (see Table 1).

$\bar{y}_{C,i}$ = average of candidate method test results at test condition *i* = 1, 2, 3, 4, 5 (see Table 1).

SECTION 3: REPORTING

On completion of testing, a report that documents the testing process and the test results obtained shall be prepared. It shall, as a minimum, contain the following information:

.1 Dates of test performance;

.2 Location(s) of testing;

.3 Number of tests performed and results of each test;

.4 Calculations used to demonstrate equivalence between candidate methods when applicable and Appendix A (ISO 8502-9 method);

.5 Conversion factor (if any) used to achieve equivalent results;

.6 Description of conductivity meter used for the ISO 8502-9 method, including make and model number, and the calibration log and calibration verification log for the meter as required in Appendix A; and

.7 Description of candidate method used, including any relevant make and model number, and the results log of any calibration verifications required by the manufacturer of the candidate method.

REFERENCES

1 ISO 8502-9 (latest revision), "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" (Geneva, Switzerland: ISO).

2 ISO 8502-6 (latest revision), "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" (Geneva, Switzerland: ISO).

3 "Bresle Patch Evaluation Report," Corrosion Control Consultants and Labs, Inc., May 8, 2008. This report is available from the NACE Technical Activities Division upon request.

4 H. Mitschke, "Effects of Chloride Contamination on the Performance of Tank and Vessel Linings," JPCL 18, 3 (2001): pp. 49-56.

5 G.C. Soltz, "The Effects of Substrate Contaminants on the Life of Epoxy Coatings Submerged in Seawater" (San Diego, CA: National Shipbuilding Research Program (NSRP) Report, Task 3-84-2, March 1991).

6 ASTM A36/A36M (latest revision), "Standard Specification for Carbon Structural Steel" (West Conshohocken, PA: ASTM).

7 ISO 8501-1 (latest revision), "Preparation of steel substrates before application of paints and related products – Visual assessment of surface cleanliness – Part 1: Rust grades and preparation grades of uncoated steel substrates and of steel substrates after overall removal of previous coatings" (Geneva, Switzerland: ISO).

8 ASTM D1193 (latest revision), "Standard Specification for Reagent Water" (West Conshohocken, PA: ASTM).

9 ASTM E177 (latest revision), "Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods" (West Conshohocken, PA: ASTM).

10 ASTM D4940 (latest revision), "Standard Test Method for Conductimetric Analysis of Water Soluble Ionic Contamination of Blasting Abrasives" (West Conshohocken, PA: ASTM).

Appendix A

Standard method for performance of soluble salts testing in accordance with ISO 8502-9 (Mandatory)

This test protocol is based on ISO 8502-6 and 8502-9 methods of conductivity evaluation, and provides a detailed and standardized process to be used for determination of conductivity using a flexible adhesive sample patch (e.g., Bresle patch) and direct reading conductivity meter with a sensor that can accurately measure conductivity of small quantities of fluid (i.e., < 0.5 mL).

This standard method for performance (ISO 8502-9) has been tested in the field, and is suitable for use when a standardized method is required (e.g., in specifications). The method is essentially similar to that widely used by some major industry users.

A1 Bresle patch background contamination

Bresle patches from different manufacturers or different manufacturing lots have different levels of background ionic contamination. The average background Bresle patch contamination for each manufacturing lot of Bresle patches used for the equivalence demonstration in this standard shall be determined as follows:

A1.1 Use a clean, flat, plate glass panel with enough area for 10 Bresle patch measurements.

A1.2 Rinse the glass panel a minimum of three times with deionized water. Allow to air dry completely.

A1.3 Randomly select 10 Bresle patches from the manufacturing lot that will be used to perform the equivalence validation in this standard.

A1.4 Perform the ISO 8502-9 method in accordance with paragraphs A2.1 through A2.9 for each Bresle patch.

A1.5 Calculate the increase in conductivity attributable to patch contamination using Equation (A1):

$$\gamma \text{ (patch contamination)} = \gamma \text{ (sample)} - \gamma \text{ (extraction solution)} \qquad \text{(A1)}$$

where:

γ (patch contamination) = conductivity attributable to patch contamination in µS/cm
γ (sample) = conductivity of the sample solution in µS/cm
γ (extraction solution) = conductivity of the extraction solution in µS/cm

A1.6 Calculate the conductivity attributable to the average patch contamination, γ (patch contamination average), for the 10 Bresle patches. This is the value used for the calculation in paragraph A2.10.

A2 Standard test method

A2.1 Before each measurement or series of measurements, and at a frequency of no less than once every four hours, use appropriate calibration solution to verify that the conductivity meter is accurate. Adjust the conductivity meter as necessary to achieve accurate measurement. Refer to the manufacturer's operating instructions for guidance.

A2.2 Rinse conductivity cell prior to use with deionized water (i.e., fresh test solution) three times.

A2.3 Measure conductivity of this test solution and record the value.

A2.4 Use a standard adhesive patch of size A-1250 as specified in Clause 4.1 of ISO 8502-6. Remove the protective backing and foam center from the Bresle patch as shown in Figure A1.

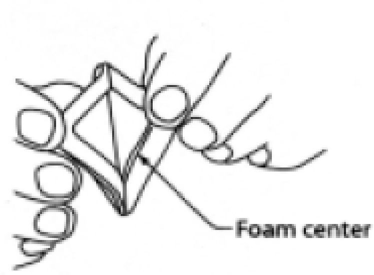

Figure A1 - Peeling Bresle patch

A2.5 Apply Bresle patch to surface. Press firmly around the perimeter of the patch as shown in Figure A2 to ensure a complete seal.

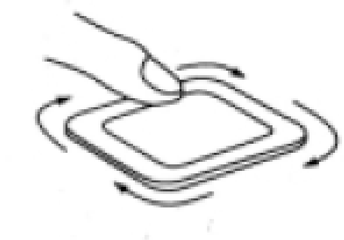

Figure A2 - Pressing Bresle patch

A2.6 Fill syringe (Figure A3) completely with deionized water, then discard the water. Perform this rinse activity three times.

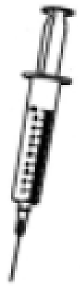

Figure A3 - Filled syringe

A2.7 Insert syringe needle into Bresle patch at an angle of approximately 30° to the test surface near the outer edge of the patch (see Figure A4) so that it passes through the adhesive foam body into the test compartment and remove air from the patch. Remove syringe needle from the patch, and fill the syringe with 3 mL of deionized water. Replace syringe needle through foam into patch and inject 3 mL of deionized water into the patch.

Figure A4 - Inserting syringe needle into patch

A2.8 Remove the syringe needle from the patch and immediately gently rub the surface of the Bresle patch, as shown in Figure A5, for 90 seconds to allow the water to dissolve surface contaminants. Extract the sample solution from patch with syringe within 15 seconds of completion of rubbing.

Figure A5: Rubbing Bresle patch

A2.9 Inject sample solution directly into conductivity cell as shown in Figure A6. Rinse cell three times with the same test solution to be measured before taking nine readings. Record the conductivity value of the sample solution as γ (sample).

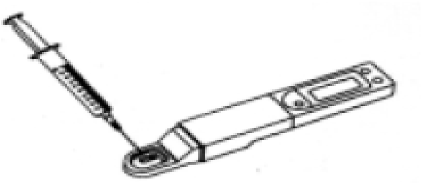

Figure A6: Reading conductivity

A2.10 Calculate the increase in conductivity in the test solution (Δγ) using Equation (A2). This value is corrected for the contribution of ionic contamination from the Bresle patch and the extraction solution. An offset factor is then added back in. The offset must be added because historically salt contamination specifications have been developed to account for the ionic contamination offset introduced by the Bresle method itself. The offset factor of 6 μS/cm used in Equation (A2) is consistent with ISO 8502-9 and confirmed by laboratory tests of Bresle patches from multiple sources.

$$\Delta\gamma = \gamma\ (\text{sample}) - \gamma\ (\text{extraction solution}) - \gamma\ (\text{patch contamination average}) + 6\ \mu\text{S/cm} \qquad \text{(A2)}$$

where:

γ (sample) = conductivity of the sample solution in μS/cm
γ (extraction solution) = conductivity of the extraction solution in μS/cm
γ (patch contamination average) = conductivity attributable to the average patch contamination in μS/cm (from paragraph A1.6)

A2.11 Calculate the surface density of the salts using equation (A3):

$$\rho_A = c \times \frac{V}{A} \times \Delta\gamma \qquad \text{(A3)}$$

where:

ρA is the surface density of the salts in mg/m^2
c = 5.0 kg/m^2 · S (constant)
A = 1,250 mm^2 (surface area)
V = 3.0 mL (volume of test solution)
$\Delta\gamma$ is the increase in conductivity in the test solution in µS/cm

* * *

Appendix B

Rationale for Specification Limits
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions therein.

An independent test laboratory evaluated the variance of the ISO 8502-9 test method under NACE supervision. The laboratory performed 150 measurements at three levels of salt contamination using five operators. The levels of salt contamination were 30, 50, and 80 mg/m^2. Each operator performed 10 tests at each level. The standard deviations are listed in Table B1.

Table B1 - Standard deviations

Salt Surface Density	Standard Deviation
30 mg/m^2	4.2 mg/m^2
50 mg/m^2	4.4 mg/m^2
80 mg/m^2	4.0 mg/m^2
Pooled Standard Deviation	4.2 mg/m^2

These tests demonstrate that the absolute variance, and not the relative percentage variance, is essentially constant in the range of 30 mg/m^2 to 80 mg/m^2. The standard deviation of the test method is 4.2 mg/m^2. This value is considered to be the repeatability standard deviation, s_r (as defined in ASTM E177), that can be achieved by one laboratory over a short time. This value (s_r = 4.2 mg/m^2) has been adopted for the purpose of determining equivalence. The results of these tests are available from NACE for review by interested parties.

For paragraph 1.1.5, the precision of a single ISO 8502-9 test result is computed as the 95% confidence interval based on this standard deviation (1.96 x s_r = 8.2 mg/m^2).

In paragraph 2.4.1.2, the test panel uniformity criterion is the repeatability limit for two individual test results as defined in ASTM E177 and shown in equation (B1):

$$r = 1.96 \times \sqrt{2} \times s_r = 1.96 \times \sqrt{2} \times 4.2 \text{ mg/m}^2 = 11.6 \text{ mg/m}^2 \qquad \text{(B1)}$$

where:

r is the repeatability limit; and
s_r is the repeatability standard deviation determined by the NACE task group.

In paragraph 2.4.1.3, the test panel average criterion is at the 95% confidence interval for the average of a sample of three test results from a test panel with an ISO 8502-9 standard deviation of s_r.

In paragraph 2.4.2.1, the standard deviation criterion is the one-tailed test at 95% confidence level that the standard deviation of a random sample of 30 test results comes from a population (i.e., operator and laboratory) with a true standard deviation of s_r or less, illustrated in equation (B2):

$$s_{sample} < \sqrt{\frac{X^2_{(0.05,n-1)}}{(n-1)}} \times s_r = \sqrt{\frac{42.6}{29}} \times 4.2\,\text{mg/m}^2 = 5.1\,\text{mg/m}^2 \qquad \text{(B2)}$$

where:

s_{sample} is the standard deviation of the test results;
n is the number of test results; and
χ^2 is the critical value of the chi-squared distribution.

In paragraphs 2.4.2.2 and 2.4.2.3, the standard deviation criterion is the same test applied to a random sample of 10 test results, illustrated in equation (B3):

$$s_{sample} < \sqrt{\frac{X^2_{(0.05,n-1)}}{(n-1)}} \times s_r = \sqrt{\frac{16.9}{9}} \times 4.2\,\text{mg/m}^2 = 5.8\,\text{mg/m}^2 \qquad \text{(B3)}$$

This relaxed standard deviation criterion is required to compensate for the smaller sample sizes at test conditions 4 and 5 (10 ISO 8502-9 tests vs. 30 at the combined test conditions 1, 2, and 3).

This same principle governs the standard deviation criteria in paragraph 2.4.3. In paragraph 2.4.3.1.1, the overall standard deviation criterion is the one-tailed test at 95% confidence level that the standard deviation of a random sample of 60 test results (the combined number of candidate test results at 50 mg/m^2 from test conditions 1, 2, and 3) comes from a population (i.e., operator and laboratory) with a true standard deviation of s_r or less, as shown in equation (B4):

$$s_{sample} < \sqrt{\frac{X^2_{(0.05,n-1)}}{(n-1)}} \times s_r = \sqrt{\frac{77.9}{59}} \times 4.2\,\text{mg/m}^2 = 4.8\,\text{mg/m}^2 \qquad \text{(B4)}$$

Whereas, in paragraph 2.4.3.1.2 and 2.4.3.1.3, the standard deviation criterion is applied to a random sample of 20 candidate method test results, as shown in equation (B5):

$$s_{sample} < \sqrt{\frac{X^2_{(0.05,n-1)}}{(n-1)}} \times s_r = \sqrt{\frac{30.1}{19}} \times 4.2\,\text{mg/m}^2 = 5.3\,\text{mg/m}^2 \qquad \text{(B5)}$$

For paragraph 2.4.3.1.4, this protocol is designed to reliably detect a bias of one standard deviation of the ISO 8502-9 test method at each test condition.

Appendix C

Recommended preparation of test panels and methods for applying salt solution to test panels
(Non-mandatory)

This appendix is considered non-mandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions therein.

Preparation of test panels

Use abrasive that, when tested in accordance with ASTM D4940, has a conductivity value no greater than 100 µS/cm.

Surface profile on the blasted surfaces of test panels is important because it dramatically influences the surface area under test (i.e., the surface area to which solvent is applied). Because, however, the Appendix A (ISO 8502-9) method and the candidate method are tested side by side, this aspect is not regarded as of great importance. A surface profile of 50 to 75 µm (2.0 to 3.0 mil) is required (see paragraph 2.2.1.3).

Drawdown method

In this method, a salt solution is applied to test panels and spread across the entire test panel surface as evenly as possible. The goal is to establish a uniform concentration of salts. Test areas are then selected at random.

Preparing the doping solution

The composition of the doping solution shall be in accordance with paragraph 2.2.7. The volume of the doping solution to be applied to each test panel shall be appropriate to allow uniform spreading of the doping solution to all corners of the test panel. The concentration of the doping solution shall be adjusted such that, when it is spread uniformly across the test panel surface and tested in accordance with paragraph 2.2.8, the results meet the stated requirements.

Test panel doping

Apply an appropriate volume of the doping solution in a column down the center of the test panel using a pipette (1 mL has been found appropriate for 200 x 150 mm [8 x 6 in] test panels). Immediately spread the doping solution evenly over the entire test panel with an approximately 125 mm (5 in) in length by 3 mm (0.125 in) outside diameter (OD) glass rod. The glass rod shall be continuously wiped (but not rotated) over the test panel to keep the doping solution uniformly distributed until the liquid dries. A gentle stream of dry, compressed air may then be blown over the test panel to accelerate drying.

Drop method

In this method, discrete areas of each test panel are doped with a known concentration of salts. Prior to doping, the boundary of each test area is marked with a permanent marker. The marked areas should be large enough as to not interfere with testing. Doping solution is dropped into the center of the marked areas and tested randomly.

Preparing the doping solution

The composition of the doping solution shall be in accordance with paragraph 2.2.7. The volume of each drop shall be determined such that when spread on the test panel surface a single test is capable of measuring the entirety of deposited salts. An appropriate volume for use with the Bresle patch size of 1,250 mm^2 has been found to be 50 µL. The concentration of the doping solution shall be adjusted such that when it is spread applied in discrete locations and tested in accordance with paragraph 2.2.8 the results meet the stated requirements.

Test panel doping

Place a single drop of the doping solution in the center of each marked area on the test panel using a pipette. Allow the doping solution to spread and begin to evaporate. Once the majority of the doping solution has evaporated, a gentle stream of dry, compressed air may be blown over each spot to remove residual moisture.