

SSPC: The Society for Protective Coatings Technology Guide 15

Field Methods for Extraction and Analysis of Soluble Salts on Steel and Other Nonporous Substrates

1. Scope

1.1 This Guide describes the most commonly used field methods for the extraction and analysis of soluble salts on steel and other nonporous substrates. Laboratory methods are only included for situations where more complete salt extractions are desired through rigorous laboratory retrieval and analysis protocols.

2. Description and Use

2.1 INTRODUCTION: Coatings applied on surfaces contaminated with soluble salts exceeding a certain concentration exhibit diminished performance. Likewise, self-passivating metals (not intended for coating such as stainless steel, aluminum and copper) can be compromised from elevated concentrations of residual soluble salts in corrosive environments. Soluble salt testing involves two basic steps: 1) extraction of salts from the surface into a solution and 2) the analysis of the solution. This Guide is intended to assist the user in selecting specific procedures for extracting and performing qualitative and/or quantitative soluble salts from steel and nonporous surfaces. It includes field methods for measuring total conductivity (fully automated or multi-step) and specific ions. It also offers guidance for destructive laboratory soluble salt extraction and testing. An overview of available field and laboratory techniques, methods of salt extraction and surface concentration calculations are provided in Tables 1 and 2 respectively. A discussion and sources on the efficiency of salt removal (also called extraction) from the metal surfaces of interest are provided in Appendix C.

2.1.1 Abbreviations:

Cl-1 chloride ion

cm³ cubic centimeters

cm² square centimeters

Fe⁺² ferrous ion

μS microSiemens

μS/cm microSiemens per centimeter

mg milligram ml milliliter

ppm parts per million NaCl sodium chloride

NO₃-1 nitrate ion SO₄-2 sulfate ion

2.2 SALT EXTRACTION SOLUTIONS

All of the reagents used to extract salts from metal surfaces in this document fall into two categories: reagent water and proprietary aqueous solutions. They will be designated herein as reagent water or proprietary extraction solution.

2.2.1 Reagent Water: Reagent water used for salt extraction should have a maximum conductivity of 5 μ S/cm. Distilled water may be purchased at grocery stores but verification of the conductivity is recommended. Alternatively, a portable demineralizer may be used to make reagent water on site. Pour tap water into the plastic bottle, attach the demineralizer cartridge in the direction indicated, invert, and squeeze out the desired amount of water (for many of the tests described below, at least 25 ml will be required). The cartridge can be used until the blue color turns brown, as indicated on the side of the cartridge. Once this occurs, replace the cartridge. Each cartridge should deionize approximately 3000 ml of water.

2.2.2 Proprietary Solutions: Proprietary extraction solutions may be included with some commercial test kits. These solutions should only be used for the soluble salts described in the kit instructions. Proprietary solutions are not suitable for measuring conductivity of extracted solutions.

3. Referenced Standards

The standards listed below are updated on a periodic basis. The user of this guide should consult the most recent revision.

3.1 SSPC STANDARDS AND JOINT STANDARDS:

SP 5/NACE No. 1 White Metal Blast Cleaning

3.2 ASTM INTERNATIONAL STANDARDS1

ASTM D4327 Standard Test Method for Anions in

Water by Chemically Suppressed Ion

Chromatography

ASTM D512 Standard Test Methods for Chloride Ion

In Water

ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959. For referenced ASTM standards, visit the ASTM website, http://www.astm.org, or contact ASTM Customer Service at service.gastm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

Extraction Fiber Strip. Additional product-specific information is available in each manufacturer's website (refer to Appendix D).

Note: As of May 2013, NAVSEA Standard Item 009-32 FY 12 (Change 1)⁴ requirement for solution conductivity for a Bresle Patch is V/A = 3 ml/1,250 mm². If NAVSEA compliance is required, the reader must verify that the automated unit being employed calculates surface salt concentrations as specified by the version of NAVSEA Standard Item 009-32 invoked by the construction contract.

4.1.1 Soluble Salt Meter

4.1.1.1 Overview of the method: The soluble salt meter⁵ is a hand-held device that is fully automated to perform both sample extraction and analysis. The device comes with a self-contained fluid dispenser that delivers accurate pre-measured volumes of reagent water for soluble salt measurements and post-measurement flushing. The device includes interchangeable silicone-rubber lined magnetic heads of different curvatures, and provides on-screen instructions for use. The meter includes an interface for connection to external peripheral equipment. Soluble salt is measured as conductance $(0-100~\mu\text{S/cm})$ or mg/m^2 . Additional specifications are available on manufacturer's website.

4.1.1.2 Extraction and Testing Protocol

- Step 1. Attach the meter to test surface (if not magnetic, press unit against surface during testing).
- Step 2. Inject reagent water into measurement chamber with one press of the dose bottle.
- Step 3. Meter automatically agitates solution.
- Step 4. Meter automatically takes readings (displays reading on LCD screen and stores values electronically).
- Step 5. Meter indicates completion of test cycle.
- Step 6. Wipe remaining reagent water from surface after removing SSM and flush meter by turning over and injecting water from dose bottle.

Unit is calibrated by the manufacturer.

4.1.1.3 Automatic Calculations reported as:

conductivity μ S/cm mg/m² (ISO 8502-9) mg/m² as NaCl (IMO PSPC)^{6,7}

4.1.2 Surface Salinity Meter

4.1.2.1 Overview of the method: The surface salinity meter⁸ (SSM-21P®), is another hand-held device that is fully

- NAVSEA Standard Item 009-32, "Cleaning and Painting Requirements" is available as a .pdf from the National Surface Treatment Center website http://www.nstcenter.biz>.
- The SSM Model RPCT-07-001®, manufactured by HEDON Equipment [see Appendix D] is the only meter meeting requirements of Section 4.1.1.1 known to the committee at this time. If you are aware of alternate manufacturers, please provide this information to SSPC Standards Development staff.
- The results of converting conductivity to any level of chloride, even under laboratory controls, have been found to be of questionable accuracy.
- IMO PSPC refers to the Performance Standard for Protective Coatings developed by the International Maritime Organization, International Maritime Organization 4, Albert Embankment, London SE1 7SR United Kingdom. A list of distributors of IMO publications is available at https://www.imo.org
- available at available at http://www.imo.org
 The Surface Salinity Meter (SSM-21P®) manufactured by DKK-TOA Corporation [see Appendix D] is the only meter meeting requirements of Section 4.1.2.1 known to the committee at this time. If you are aware of alternate manufacturers, please provide this information to SSPC Standards Development staff

automated to perform both sample extraction and analysis. Surface salinity (mg/m²) can be directly read in four formats: soluble salt in IMO PSPC and normal modes; sodium chloride concentration, and conductivity of solution. The measuring cell is magnetically or manually held against steel or metal surface to be tested. The cell measurement area is 1,250 mm². Pure (reagent) water (10 ml) is injected into the measurement cell using a plastic disposal syringe. The solution is agitated and the salinity measurement is automatically calculated, reported, and stored. The meter includes an interface for connection to external peripheral equipment. Additional specifications are available on the manufacturer's website (Appendix D).

4.1.2.2 Sample Extraction and Testing Protocol

- Step 1. Attach measurement cell onto the steel surface
- Step 2. Inject 10 ml of reagent water using the plastic syringe and press the start key on the meter
- Step 3. Press the solution stirrer button
- Step 4. Measurement result is displayed on the meter and also stored in the meter's internal data logger
- Step 5. Wipe off water from test area and flush measurement cell with additional water

Unit is calibrated by the manufacturer,

4.1.2.3 Automatic Calculations reported as:

conductivity μ S/cm mg/m² (ISO 8502-9) μ g/cm² as chlorides⁶

4.1.3 Continuous Flow Extraction Fiber Strip

4.1.3.1 Overview of the method: This method9 requires use of a pre-measured solvent ampoule that is attached to a disposable sensor, and a reusable meter to analyze the extracted solution. Surface soluble salts are extracted by the flow of solvent across an engineered fiber in the sensor. The extracted salt solution is collected in the sensor's reservoir. A pair of electrodes extends out of the body of the sensor. When these electrodes are placed in the accompanying meter, a direct reading of electrical conductivity proportional to concentration of soluble salt ion is displayed on the meter. This measurement process does not require any manual manipulation. The contact area of the unit is approximately 1.5 cm x 2.0 cm and does not seal to the test surface. The method permits evaluation of curved, irregular, and highly constrained test areas. Many units can be used in series for higher sampling coverage.

4.1.3.2 Sample Extraction and Testing Protocol

- Step 1. Squeeze the neck of the pre-measured ampoule containing solvent to break the seal, and push onto the sensor neck.
- Step 2. Affix the disposable sensor to the surface under test using standard non-residue painter's tape.

The SaltSmart® meter model 2000 manufactured by Louisville Solutions,Inc [see Appendix D] is the only meter meeting requirements of Section 4.1.3.1 known to the committee at this time. If you are aware of alternate manufacturers, please provide this information to SSPC Standards Development staff.