SSPC: The Society for Protective Coatings

TECHNOLOGY GUIDE 15

Field Methods for Retrieval 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 retrieval and analysis of soluble salts on steel and other nonporous substrates. Laboratory methods are only included for situations where laboratory control is desired. Much of this information was contained in SSPC-TU 4, Field Methods for the Retrieval and Analysis of Soluble Salts on Substrates, which will be withdrawn after publication of this Guide.[1]

2. Description and Use

2.1 Coatings applied on surfaces contaminated with soluble salts exceeding a certain concentration exhibit diminished performance. This Guide is intended to assist the user in selecting specific procedures for retrieving and analyzing soluble salts. Section 4 of the Guide discusses the various methods for retrieving salts from a surface. Section 5 discusses the analytical methods used to determine the concentration of the soluble salts in the extracted solution. See Appendix E for links to information on sources for testing equipment and materials.

3. Referenced Standards

3.1 SSPC STANDARDS AND JOINT STANDARDS:[1]

SP 5/NACE No. 1	White Metal Blast Cleaning

3.2 INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO) STANDARDS:[2]

ISO 8502	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 (ISO 8502-2:1992)
Part 5	Measurement of chloride on steel surfaces prepared for painting– Ion detection tube method (ISO 8502-5:1998)
Part 6	Extraction of soluble contaminants for analysis–The Bresle method (ISO 8502-6:1995)
Part 9	Field method for conductimetric determination of water-soluble salts (ISO 8502-9:1998)
Part 10:	Field method for the titrimetric determination of water-soluble chloride (ISO 8502-10:1999)
Part 12:	Field method for the titrimetric determination of water-soluble ferrous ions (ISO 8502-12:2003)

4. Retrieval Methods

4.1 CLASSES OF RETRIEVAL METHODS: Salt retrieval methods employed to help determine surface concentrations of salt on substrates fall into three general classes, which can be further subdivided. (See Appendix E for links to information on testing equipment and materials.)

4.1.1 Class A: Class A retrieval involves a methodology for containing a liquid that is held in contact with a surface of predetermined area. Turbulence within the contacting liquid enhances the dissolution of the salt contamination into the solution.

Method A1: Patch Cell Retrieval Method: This method utilizes a small adhesive patch covered with a latex film, which attaches to the structure forming a cell cavity. Self-contained adhesive edges allow the cell to adhere to the surface. Distilled or deionized water or a proprietary extraction liquid is then injected into its center with a hypodermic needle. The patch fills up like a large paint blister. The liquid is massaged against the surface being tested, retrieved from the patch using the hypodermic needle, and tested for concentration of ions.

Method A2:Sleeve Retrieval Method: This method uses a small flexible chloride-free latex sleeve (sock) with a self-contained adhesive edge that is attached to the structure being

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[1] Single copies of withdrawn standards may be obtained from SSPC upon request.

[2] International Organization for Standardization (ISO), Case Postale 56, Geneva CH-1211, Switzerland. ISO standards may be obtained through the American National Standards Institute, 1819 L Street, NW, Suite 600, Washington, DC 20036 (www.ansi.org).

tested, forming a cavity. A salt retrieval solution is dosed into the sleeve prior to attachment. The solution is massaged against the surface being tested for a specified period of time and is then removed. The sleeve then is removed and the solution tested for levels of chloride and/or nitrate. A kit is available for this method, with operating instructions and a pre-measured (fixed volume) proprietary solution.

4.1.2 Class B: Class B retrieval involves a methodology for containing a known volume of liquid within a measured area that is in contact with the surface. There may or may not be mechanical rubbing of the surface.

Method B1: Swabbing or Washing Methods: A low conductivity liquid such as deionized water and cotton swabs are used to extract salts from a surface. The method requires that the operator wear non-chlorinated latex rubber gloves to prevent cross contamination of the surface or the retrieved sample by salts naturally present on the surface of the skin. After swabbing the surface, the liquid is tested for concentration of ions.

Method B2: Filter Paper Extraction Method: A pre-wetted absorbent filter paper is placed on the surface from which the salt is to be extracted. The paper wets the surface and extracts soluble salts. After a pre-determined time, the paper is removed from the surface and placed over the electrodes of a resistivity meter. The meter indicates the conductivity of the wetted paper. The conductivity is proportional to the total dissolved salts.

4.1.3 Class C: Class C retrieval is used only in a laboratory setting and involves immersing the entire surface in boiling water. This method uses a predetermined volume of extraction liquid and a predetermined surface area.

Method C: Boiling Extraction Method: This method involves the use of boiling deionized water to extract salts from a sample coupon. This method is intended to be used for the extraction of salts from sample substrates in a laboratory setting. It may be used as a reference method to derive retrieval rates for the patch cell, sleeve, swabbing, and filter paper extraction methods described above. It may also be used for field samples cut from a structure or test panels exposed in a field or lot or cabinet. This method is described in Appendix A.

4.1.4 The first four methods can be used to help characterize surfaces encountered in either laboratory or field settings. In general, boiling extraction methods are only useful under laboratory conditions. No field or laboratory method is believed capable of retrieving all the soluble salt from a surface. The proportion of salt retrieved by field methods depends on the method used, the roughness of the examined surface, the degree of rusting, and the ambient conditions. The presence of pits or deep craters on the surface can lead to grossly inaccurate measurement of salt contamination, because salts in the bottoms of deep pits may escape detection.

4.2 EXTRACT SOLUTION: All the retrieval procedures described in this document use deionized or distilled water (designated as reagent water) or a proprietary solution.

4.2.1 Reagent Water: Reagent water used for salt retrieval should have a maximum conductivity of 5 microsiemens per centimeter (μS/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 deionized 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.

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

4.3 PATCH CELL RETRIEVAL METHOD: The patch cell sampling procedure is described in detail in ISO 8502-6.

4.3.1 Sample Acquisition Procedure

1. Remove the backing and the foam insert from the test cell and apply the cell firmly and tightly to a dry test surface. All orientations, including vertical, horizontal, or overhead are acceptable.
2. Insert the needle attached to the 5 mL syringe into the cell through its spongy foam perimeter, taking care not to inject beneath the foam or into the latex film. Evacuate the air from the test area by pulling back on the plunger. Expel the air from the syringe. Fill the syringe with 3 mL of the extraction liquid.
3. Inject 3 mL or other designated quantity of extraction liquid into the above cell taking care to keep air bubbles out of the syringe. Hold the cell perimeter firmly during this operation to prevent water leakage.
4. Remove the needle from the cell center (but not the spongy foam perimeter) and gently rub the top of the cell for 10 to 15 seconds to encourage dissolution of soluble salts. Increasing the massage time may increase the efficiency of the extraction liquid.
5. Withdraw and re-inject the extraction liquid a minimum of three times, each time gently rubbing the top of the cell for 10 to 15 seconds. Then, remove as much of the extraction liquid as possible and place it in a clean vial or other container. A new clean container should be

used for each test, or if reused, the container should be rinsed two or more times with reagent water.

6. If additional testing requires a higher volume of extraction liquid than that afforded by the extraction procedure, add extraction liquid to raise the sample volume to the required level and note the new sample volume and dilution percentage.
7. Use the extraction liquid from step 6 to determine chloride ion concentration, ferrous ion concentration, or other ion concentrations using one of the methods described in Section 5 (see Section 4.2.2).
8. If additional samples are to be taken, always use a fresh cell and clean syringe and needle; this will avoid cross contamination between samples.

4.3.2 Advantages of the Patch Cell Method:

1. The adhesively attached cells can conform to curved and irregular surfaces.
2. Cells such as these are commercially available in a variety of sizes; the most commonly used size retrieves salt from a surface of 12.5 cm^2. Smaller cell sizes permit assays of salt levels to be made at local corrosion sites such as craters or pits.
3. If reagent water has been used as the extraction liquid, conductivity can be determined using commercially available conductivity meters.
4. Acidic extraction liquids (such as the one furnished in the proprietary kit for this procedure) normally provide better extraction efficiency than deionized water.

4.3.3 Limitations of the Patch Cell Method:

1. The adhesively attached cells only accommodate a small amount of retrieval liquid. With the most commonly used cell size (12.5 cm^2 surface area), the actual quantity of cell liquid contacting the surface is 3 mL. This can limit the range of analyses that can be performed.
2. No in-line determination of conductivity can be performed with these cells.
3. The cell may not adhere well to rusted surfaces, but it may adhere so well to abrasive blast cleaned surfaces that it is difficult to remove.
4. The cell may leak through the hole introduced by the syringe.
5. The cells are consumable and can be used only once.

4.3.4 Retrieval Efficiency: see Appendix D.

4.4 SLEEVE RETRIEVAL METHOD

4.4.1 Sample Acquisition Procedure

1. Remove the cap from the bottle of premeasured extract and pour the entire contents into the sleeve.
2. Remove the pressure sensitive backing from the sleeve adhesive ring.
3. Remove most of the air from within the sleeve by squeezing the sleeve between fingers and thumb. Do not spill any extraction solution from the sleeve when evacuating air.
4. Firmly apply the sleeve to the test surface. Lift and hold the free end of sleeve upright to allow the extraction liquid to come into contact with the surface
5. Use the other hand to massage the solution through the sleeve against the surface for 2 minutes. It should be noted that increasing the massage time (e.g., up to 6 minutes) will increase the extent of salt removal. When the massage is complete, remove the sleeve and solution from the surface. For vertical or overhead surfaces, the extract solution will return to the lowered area in test sleeve. For horizontal surfaces press and slide a finger across the sleeve to move the solution to the closed end of the sleeve prior to removal.

4.4.2 Advantages of the Sleeve Retrieval Method:

1. This method is very simple to perform, as all components are pre-measured.
2. The adhesive sleeve can conform to curved and irregular surfaces. Tests can be performed on vertical, horizontal, and overhead surfaces.
3. For extremely rough or pitted surfaces, the seal ring may be doubled, thereby allowing testing to be performed.
4. The kit form of this method provides a pre-measured volume of extraction solution and a fixed area of the sleeve opening. These features are designed to provide a direct final reading in micrograms per square centimeter (μg/cm^2).
5. All components are one-time usage, eliminating cross contamination from test to test.
6. In hot weather or on hot surfaces, the encapsulated extract solution will not evaporate.
7. The extractions also provide sufficient sample size for analyses to be performed for different ions.
8. Acidic extraction liquids (such as the one furnished in the proprietary kit for this procedure) normally provide better extraction efficiency than deionized water.

4.4.3 Limitations of the Sleeve Retrieval Method:

1. The adhesive sleeve may not adhere well to rusted surfaces, but it may adhere so well to abrasive blast cleaned surfaces that it is difficult to remove.
3. No in-line conductivity testing can be performed with the sleeves.
4. The sleeves are consumable and can be used only once.

4.4.4 Retrieval Efficiency: see Appendix D.

4.5 SWABBING OR WASHING METHOD

The materials, procedures, and advantages and limitations are described below. A similar procedure is described in ISO 8502-2, Sections 5 and 6.

4.5.1 Procedure for Swabbing Method:

1. Use a ruler and chloride free marker to outline a representative surface area of specific size (e.g., 15 by 15 cm [6 by 6 inch]).
2. From a graduated cylinder, pour a measured volume (e.g., 22.5 mL) of reagent water into one of two plastic beakers (beaker A.) (**Note:** The suggested sample volume and area computes to 1 mL per 10 cm^2. This can simplify later calculations of salt contamination levels.)
3. Repeat step 2 with the second beaker (beaker B).
4. Using a pair of tweezers or chloride free latex or rubber gloves, dampen a sterile cotton ball or chloride free small sponge in the water in beaker A. Thoroughly swab the area measured in step 1, taking care to avoid dripping the liquid on the surface. After swabbing, swirl the applicator (cotton ball or sponge) in the water and then squeeze it against the inside of beaker A to extract as much water as possible from the applicator.
5. Repeat this swabbing, swirling, and squeezing operation four times with fresh applicators and then leave the applicators in the water in beaker A.
6. Use an applicator to dry the measured test area and place it in beaker A.
7. Stir the water and applicators for two minutes to achieve thorough mixing and to extract salts from the cotton swabs or sponge.
8. Record the final volume.
9. Take the same number of fresh applicators, identical to those used in steps 4 through 7 above, and immerse them in beaker B. Then stir as in Steps 4 and 5, and let sit, covered, for at least three minutes. This will provide the control sample.

4.5.2 Retrieval Efficiency: see Appendix D:

4.5.3 Advantages of Swabbing Method:

1. The swabbing retrieval method provides a means for acquiring samples of salt from steel or other non-porous surfaces using readily available materials.
2. Retrievals can be conducted on a range of surfaces without regard to surface irregularities or condition.
3. The swabbing method can be used on large areas to indicate general surface contamination by salts.
4. The extractions also provide sufficient sample size for several analyses to be performed for different ions.

4.5.4 Limitations of Swabbing Method:

1. Swabbing methods are difficult to perform in an overhead or vertical position. Extracted liquid may be lost dripping from the swabs.
2. Swabbing is not well suited for measuring salt levels of small, localized contamination such as craters or pits.
3. There is a risk of contamination of a sample by the operator if gloves or any other equipment used for these procedures become damaged.
4. In hot weather or on hot surfaces the extraction liquid may evaporate on the surface prior to its removal.

4.6 FILTER PAPER EXTRACTION METHOD

4.6.1 Sample Acquisition Procedure:

1. Put on a pair of clean chloride free latex or rubber gloves.
2. Fill a syringe with the specified level (about 2 mL) of reagent water (see Section 4.2.1).
3. Eject the water from the syringe onto the paper surface taking care to retain as much water as possible on the paper. **Note:** Use only sample paper recommended by the manufacturer as soluble salt free. Commercial filter papers are not suitable as they can contain excessive salt levels.
4. Place the wetted paper on the area to be sampled, pressing it firmly into its contours and surface irregularities.
5. Press out as much entrapped air as possible from beneath the paper.
6. When 2 minutes have elapsed, remove the sample paper from the surface for analysis.
   Refer to Section 5.3 for analysis procedure.

4.6.2 Advantage of Filter Paper Extraction Method: The filter paper procedure is relatively simple and is less subject to operator error.

4.6.3 Limitations of Filter Paper Extraction Method:

1. The instrument measures total soluble salts, rather than a specific ion such as chloride or nitrate.
2. There is no independent data on the accuracy or precision of this method.
3. The water is subject to evaporation loss under conditions of high temperature and/or low humidity.

5. Analytical Methods

5.1 QUANTITATIVE ANALYSIS: This section discusses the most common analytical methods used to determine the amount of soluble salt contamination in the extracted solution.

The following substances and quantities are described:

- Conductivity
- Soluble Chloride Ion
- Soluble Ferrous Ion
- Soluble Sulfate Ion
- Soluble Nitrate Ion

5.1.1 Precision and Accuracy of Quantitative Measurements: In response to requests from SSPC, some suppliers of proprietary equipment have provided data on precision and accuracy of analytical methods. If this information was provided, it is given in the section describing the method. *Information provided by suppliers has not been verified by third-party testing.* Users are advised to contact the manufacturers directly to obtain additional information that is not provided in the guide. See Appendix E for links to equipment suppliers and manufacturers.

5.2 FIELD MEASUREMENT OF CONDUCTIVITY (TOTAL SOLUBLE SALTS)

5.2.1 This method provides a measurement of solution conductivity. Conductivity is a measure of the total dissolved salts.

5.2.2 Types of conductivity meters: Commercially available portable conductivity meters available include “pocket” type, “cup” type, and meters with specific features. The “pocket” types are operated by placing the probe into the liquid to be analyzed. For the “cup” type, the liquid is placed in a cup that forms part of the meter. One special meter is that described in Section 5.3 for analyzing the filter paper. Each meter has its own degree of accuracy.

5.2.3 Test Procedure

5.2.3.1 Procedure for pocket-type conductivity meters: This procedure entails two measurements: the first of the control solution (typically reagent water), which is Reading One, and the second of the extraction liquid, which is Reading Two. Wash the probe end of conductivity meter with reagent water prior to each reading to prevent cross contamination. Place the probe end of a calibrated conductivity meter into the reagent water and record the reading on the meter (Reading One). Then repeat the procedure for the extracted liquid (Reading Two). Subtract Reading One from Reading Two. The resulting number is the corrected conductivity of the extracted liquid.

5.2.3.2 Procedure for cup-type conductivity meters: If an external cup conductivity meter is used, transfer about 10 milliliters of the reagent water into the cup of the calibrated conductivity meter. Select the appropriate range and record the conductivity reading in μS/cm (Reading One). Transfer some of the extracted liquid into the cup and record conductivity (Reading Two).

Subtract Reading One from ReadingTwo. The resulting number is the corrected conductivity of the extracted liquid. A similar method is also described in ISO 8502-9.

See Appendix C for a procedure to estimate equivalent chloride ion surface concentrations from conductivity of extract.

5.3 FIELD PROCEDURE FOR ANALYZING FILTER PAPER FOR SOLUBLE SALTS: Place the sample paper over the concentric copper electrodes of the resistivity meter, ensuring that the outer ring is completely covered. All air must be excluded from beneath the paper. The lid is closed, and after seconds, the reading is displayed in μg/cm^2, based on sodium chloride. The manufacturer of one device has prepared charts showing the variation in the salt level readings due to simulated marine and simulated urban salt concentrations and due to temperature.

5.4 FIELD DETECTION OF CHLORIDE ION BY ION DETECTION TUBE: This method uses sealed vacuum tubes with crystals impregnated with silver dichromate (pink). The ends of the tubes are snapped off, opening the tube much like a straw. When one end of the tube is immersed in the extract solution, capillary action wicks the solution to the top of the tube. On contact with the chloride ion the silver dichromate converts to silver chloride (white). When the solution reaches the top of the tube, the white cotton at the top changes color to amber. This indicates the titration is complete. Graduations on the side of the tube provide the level of chloride ions present in the solution. This method, described in ISO 8502-5, can detect chloride levels from 1 to 2000 ppm, using tubes with varying ranges of detection. The tube most commonly used for surface testing of chlorides has a detection range of +0 to 60 ppm. One supplier provided the following data on standard deviation (sd) at different concentrations (C): C of 1.0 ppm, sd of 0.38 ppm; C of 3.0 ppm, sd of 0.17 ppm; C of 5.0 ppm, sd of 0.30 ppm; C of 10 ppm, sd of 0.48 ppm; C of 30 ppm, sd of 0.50 ppm. See Appendix B for information on converting from a solution concentration in ppm to a surface concentration in μg/cm^2.

5.5 FIELD DETECTION OF CHLORIDE ION BY PAPER STRIP METHOD: To determine the chloride level, place the lower end of a test strip into the extracted solution. Allow the solution to wick up and saturate the test strip, as indicated by the yellow band across the top of the strip turning blue (about 5 minutes). Then, record the scale number at the top edge of the white column (chloride ion causes the existing tan color on the strip to turn white) and compare it with the conversion chart enclosed with the test strip bottle. The range of concentration over which this method is useful is from 30 to 600+ ppm chloride ion. The precision reported by one manufacturer is ± 10% chloride. **Note:** The reading from the strip must be converted to ppm using the supplied conversion chart corresponding to the batch of test strips used for the analysis. See Appendix B for information on converting from a solution concentration in ppm to a surface concentration in μg/cm^2.

5.6 FIELD DETECTION OF CHLORIDE ION BY FIELD TITRATION METHOD

5.6.1 Test Procedure: A commercially available test kit is used to analyze the solution collected from the surface. The titration (sometimes referred to as "drop titration") is performed on a small sample (2 to 3 mL) from the extracted solution. The kit includes four solutions contained in separate reagent bottles. The procedure is as follows:

1. Using Reagent Bottle 1, press (squeeze) out 2 drops of red indicator liquid into a plastic vial containing the sample solution. Carefully agitate the liquid until it is homogeneous in color (purple).

2. Using Reagent Bottle 2, squeeze out 2 drops into the vial. The sample liquid should be yellow in color. If not, add drop-by-drop additional Reagent 2 until sample turns yellow, agitating between each drop addition.

3. At this point, a judgment should be made as to how much chloride is anticipated. If low surface concentrations (0 to 10 μg/cm^2) are expected, proceed using Reagent Bottle 4. If higher concentrations are expected, proceed using Reagent Bottle 3.

4. For low concentrations, add, drop by drop, the contents of Reagent Bottle 4. For high concentrations, add, drop by drop, the contents of Reagent Bottle 3. In either case, thoroughly agitate the solution after the addition of each drop. Count the number of drops required to turn the solution from yellow to blue, thoroughly agitating the solution after the addition of each drop. The procedure is described in ISO 8502-10.

5.6.2 Determining surface concentration: Each drop from Reagent Bottle 4 is equivalent to approximately 25 μg of chloride recovered from the surface. Each drop from Reagent Bottle 3 is equivalent to approximately 125 μg of chloride recovered from the surface. Use the following formulas to determine the maximum surface concentration in μg/cm^2 (see note below) knowing the surface area (cm^2) and the number of drops.

For Reagent Bottle 4:

$$\text{maximum surface concentration } (\mu\text{g/cm}^2) = \frac{25 \cdot (\#\text{ drops})}{\text{surface area (cm}^2)}$$

For Reagent Bottle 3:

$$\text{maximum surface concentration } (\mu\text{g/cm}^2) = \frac{125 \cdot (\#\text{ drops})}{\text{surface area (cm}^2)}$$

Note: Because it is impossible to determine whether the entire drop or a fraction of the drop was needed to turn the solution from yellow to blue, the concentrations derived from the above formulas represent the maximum concentrations. The actual concentration may range from that computed from the preceding drop to the concentration computed from formulas 1 and 2 (e.g., for 4 drops from Reagent Bottle 4 for a solution extracted from a surface area of 12.5 cm^2, the concentration range would be 6 to 8 μg/cm^2). (Commonly used patch cells have a surface area of 12.5 cm^2.) This method does not determine a specific surface concentration of chloride ion, but rather the results are reported as a range, e.g., greater than 6 and less than 8 μg/cm^2 chloride ion. One manufacturer reports a sampling accuracy of 1 to 2 μg/cm^2 or 5 to 10 μg/cm^2 depending on the specific titration chemicals used. No information on precision was provided.

5.7 LABORATORY REFERENCE METHOD FOR DETECTION OF CHLORIDE ION BY TITRATION: ISO 8502-2, "Laboratory determination of chloride on cleaned surfaces," describes a titration method based on the reaction of chloride ion with mercuric nitrate to form insoluble mercuric chloride. The indicator is a solution of diphenylcarbazole/bromophenol blue, which turns to an intense violet color to indicate the completion of the titration.

5.8 QUALITATIVE FIELD DETECTION OF FERROUS ION: In this method (described in ISO 8502-12) blotting paper is treated with potassium ferricyanide solution. The blotting paper is moistened and placed in contact with the steel surface to be tested. On contact with ferrous ions, the paper shows blue spots. The sensitivity of the method is less than 1 ppm ferrous ion. No information on precision is available, as this is a qualitative test.

Potassium ferricyanide test paper may be used as an economical screening test for active corrosion sites. It is specific to soluble ferrous ion. When used properly, it will not give false negatives, but may produce false positives. If soluble salt concentration is suspected due to a positive indication using potassium ferricyanide paper, then confirming tests utilizing another ion-specific test method may be required.

5.9 QUANTITATIVE FIELD DETECTION OF FERROUS ION: To determine the ferrous ion concentration in parts per million, moisten a ferrous ion test strip with the solution being tested and compare the resulting color to the color chart on the container label. A complex is formed between 1,10-phenanthroline and ferrous ion that has a vivid red color. Color changes are seen even at ferrous ion concentrations below 1 ppm. Typical concentration ranges for test strips are between 0.5 and 10 ppm ferrous ion. Iron test strips from one manufacturer are graduated in unequal steps: 0-3-10-15-50-100-250-500 ppm. No data is available on the precision of this technique. See Appendix B for information on converting from a solution concentration in ppm of solution to a surface concentration in μg/cm^2. Note that a proprietary kit provides the readings directly in μg/cm^2.

5.10 FIELD DETECTION OF SULFATE ION: This method works on the principle that if sulfate is present in the solution, it becomes turbid (cloudy) when barium chloride is added. The simplest instrument for measuring the degree of turbidity in the field is the optical comparator. Barium chloride is available as powder or pre-measured tablets. The tablets are more convenient to use but take a little longer to dissolve. Panes of plastic with a known degree of cloudiness are compared side by side with the sample prepared using the kit. The pane closest in cloudiness to that of the sample is taken as the sample sulfate level. The interval between each pane value has to be quite large, because the eye is not as discriminating as a well-calibrated spectrophotometer in the laboratory. Also, because the eye is not sensitive to very low levels of turbidity, the minimum level of sulfate that can be detected by this method is around 20 ppm. No data is available on the precision of this technique. See Appendix B for information on converting from a solution concentration in ppm of solution to a surface concentration in $\mu g/cm^2$.

5.11 FIELD DETECTION OF SULFATE ION BY PHOTOELECTRIC COLORIMETER: This proprietary method uses an electronic microprocessor, factory programmed, that measures the turbidity of a solution after barium chloride powder has been mixed with the test solution. This method is more sensitive than the visual method (Section 5.10). The colorimeter readings are in parts per million (ppm), and the range is from 1 to 100 ppm. The supplier reports that the accuracy of this unit over the full photometric range is ± 2%. See Appendix B for information on converting from ppm of the test solution to $\mu g/cm^2$ of the surface concentration. **Note:** the proprietary kit provides the readings directly in $\mu g/cm^2$.

5.12 FIELD DETECTION OF NITRATE ION BY PAPER STRIP: To determine the nitrate concentration, place the lower end of a test strip into the extract for two seconds. Allow the strip to stand for one minute. Observe the color and compare to the color shown on the strip. The technique measures the solution concentration in ppm. The detection range is up to 50 ppm nitrate. No data is available on the precision of this technique. See Appendix B1 for information on converting a solution concentration in ppm of solution to a surface concentration in $\mu g/cm^2$. **Note:** a proprietary kit provides the readings directly in ppm.

6. Disclaimer

6.1 This guide is designed to describe, review, or analyze new or improved technology and does not meet the definition of a standard as defined by SSPC. A guide differs from a standard in that it is (a) a set of instructions or organized information based on a consensus of best industry practice, and (b) a set of directions provided to aid in preparing one's own modified specifications.

6.2 While every precaution is taken to ensure that all information furnished in SSPC guides is as accurate, complete, and useful as possible, SSPC cannot assume responsibility nor incur any obligation resulting from the use of any materials or methods described herein, or of the guide itself.

6.3 This guide does not attempt to address problems concerning safety associated with its use. The user of this guide, as well as the user of all products or practices described herein, is responsible for instituting appropriate health and safety practices and for ensuring compliance with all governmental regulations.

APPENDIX A: Boiling Extraction Method

A.1 Materials Required: This laboratory reference procedure requires the following items (all apparatus and sample containers should be previously cleaned with deionized or distilled water):

1. Hot plate with thermostatic control
2. Reagent water, conductivity no greater than 5 $\mu S/cm$
3. Inert glass granules to prevent bumping of boiling water. (**Note:** the use of boiling stones or chips is not suggested, as these contribute ions to the water and buffer the pH of the extraction liquid on the alkaline side.)
4. Steel panels of known dimensions (e.g., 10 x 15 x 0.64 cm [4 x 6 x 1/4 inch]), previously cleaned to reflect the specification level of cleanliness used in the field, using the same abrasive as used in the field. If a level of cleanliness is not specified, then panels are cleaned to SSPC-SP 5/NACE No. 1.
5. Stainless steel or Pyrex pans of dimension no less than 15 x 20 x 5 cm (6 x 8 x 2 inch)
6. Test panels of dimensions no greater than 13 x 18 x 2.5 cm (5 x 7 x 1 inch)
7. 500 mL graduated cylinder
8. Stainless steel tongs
9. Conical funnel
10. 750 mL laboratory storage bottle

A.2 Sample Acquisition Procedure

1. Place the following items in the pan:
   - Approximately 350 mL of reagent water
   - Between 5 and 10 anti-bumping granules
   - A test panel (see item 4 of Section A.1).
2. Place the pan on the hot plate and raise water temperature to boiling over a period from 10 to 20 minutes. Maintain the temperature at boiling for 1 hour. The test panel must be kept completely submerged. If liquid is lost by boiling evaporation, it must be replenished during the test. If the panel is placed horizontally in the pan, turn the panel over after 30 minutes.
3. At the end of the test, turn off the hot plate, and remove the pan from the hot plate. Allow the liquid in the pan to cool for at least 30 minutes.

4. The steel test panel may be removed from the pan with stainless steel tongs either hot or after cooling. It should be rinsed with a small quantity of reagent water to remove any soluble salt ions from the surface, and drained over the pan. When the panel has drained dry, remove it from the work area.
5. Using the conical funnel, transfer the liquid in the pan to the graduated cylinder. Add sufficient reagent water to the graduated cylinder to bring the volume of material up to 500 ml.
6. Mix the total liquid thoroughly by transferring it between the storage bottle and the graduated cylinder.

A.3 Advantage of Boiling Extraction Method: This method provides a benchmark for determining the maximum retrieval efficiency of field retrieval methods.

A.4 Limitation of Boiling Extraction Method: This method is unsuited for use in the field. The use of laboratory-contaminated samples may provide some indication of relative extraction efficiencies, but because field conditions responsible for salt contamination are highly variable, it is best to use actual field samples where possible.

APPENDIX B: Conversions

B.1 Determining Surface Concentration from Solution Concentration: One can convert the solution concentration to an equivalent surface concentration as follows:

where:

$$E = C \bullet \frac{V}{A} \qquad \text{(Formula 1)}$$

C = solution concentration in ppm ($\mu g/cm^3$)
E = surface concentration in $\mu g/cm^2$
V = volume of extract solution in mL (1 mL = 1 cm^3)
A= area in cm^2

Example:
C = 42 ppm
A= 12.5 cm^2
V= 2 mL

$$E = 42 \bullet \frac{2}{12.5} = 6.7\ \mu g/cm^2$$

B.2 Determining Solution Concentration from Surface Concentration: One can convert the surface concentration to an equivalent solution concentration as follows:

$$C = E \bullet \frac{A}{V} \qquad \text{(Formula 2)}$$

where:
C = solution concentration in ppm ($\mu g/cm^3$)
E = surface concentration in $\mu g/cm^2$
V = volume of extract solution in mL
A= area in cm^2

Example:
E = 10 $\mu g/cm^2$
A= 12.5 cm^2
V= 2 mL

$$C = 10 \bullet \frac{12.5}{2} = 62.5 \text{ ppm}$$

B3. Unit Conversions:

1 ppm = 1 μg/cc of water
1 mL = 1 cc = 1 cm^3
1 $\mu g/cm^2$ = 10 mg/m^2

APPENDIX C: Determining Equivalent Surface Concentration From Conductivity

Note: The ability to remove all of the soluble salt ions from the surface of a structure that has been in service for extended periods of time and is severely corroded and or pitted has not yet been demonstrated. Therefore, all extraction tests remove less than 100% of the soluble salt ions from the structures, and the amount in the samples represents an unknown percentage of the amount actually existing on the structure. The test methods that are indicated in this document are therefore semi-quantitative and do not represent the actual amount of soluble salt ions existing on the structure.

C.1 Equivalent Surface Concentration of Chloride from Conductivity of Chloride Solution: This procedure may be used when one suspects that the only (or major) soluble salt present on a surface is chloride. Assuming that all salt is present as chloride can provide a worst-case scenario, as chloride is generally agreed to be the most aggressive in accelerating corrosion and inducing osmotic blistering.

One can estimate the solution concentration from the conductivity if one knows or assumes the identity of the soluble salt. For example, if one assumes that the salt is sodium chloride only, the solution concentration is estimated as follows:

where:

$$E = (0.3) \bullet S \bullet \frac{V}{A} \qquad \text{(Formula 3*)}$$

E = surface concentration of equivalent chloride in $\mu g/cm^2$
S = conductivity in $\mu S/cm$
V = volume of extract solution in mL
A = area in cm^2

Example:
S = 70 $\mu S/cm$
V = 2 mL
A = 12.5 cm^2

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[*] This formula is valid only where sodium chloride is the only soluble salt and at low concentrations. In actual field samples, there are almost always other salts present.

$$E = 0.3 \cdot 70 \cdot \frac{2}{12.5} = 3.4\ \mu\text{g/cm}^2 \text{ of equivalent chloride}$$

C.2 Equivalent Surface Concentration of Total Salt from Conductivity: Based on measurement of the average conductivity from 12 representative samples of soluble salts, an ISO committee developed the following formula for converting conductivity to surface concentration of salts (ISO 8502-10).

$$E_1 = (0.5) \cdot S \cdot \frac{V}{A} \qquad \text{(Formula 4)}$$

Where:
E_1 = surface concentration of total chloride in μg/ cm^2
S = conductivity in μS/cm
V = volume of extract solution in mL
A = area in cm^2

Example:
S = 70 μS/cm
V = 2 mL
A = 12.5 cm^2

$$E_1 = 0.5 \cdot 70 \cdot \frac{2}{12.5} = 5.6\ \mu\text{g/cm}^2 \text{ of soluble salt}$$

C.3 Comparison of Methods C.1 and C.2

The methods C.1 and C.2 give very similar results. Method C.1 computes the concentration (E) of the chloride ion, whereas Method C.2 computes the concentration (E_1) of the chloride compound. Assuming that the salt is sodium chloride, and knowing the formula weight of sodium chloride to be 58.5 and the atomic weight of the chloride ion to be 35.5, these two concentrations are related by:

$$\begin{array}{c}\text{concentration of}\\ \text{sodium chloride}\end{array} = \begin{array}{c}\text{concentration}\\ \text{of chloride ion}\end{array} \cdot \frac{\text{formula wt. of NaCl}}{\text{atomic wt. of Cl}^-\text{ ion}}$$

Using the values from the numerical examples above, the similarity between methods C1 and C2 can be demonstrated.

$$E_1 = (3.4) \cdot \frac{58.5}{35.5} = 5.6$$

APPENDIX D: Discussion and Sources on Extraction Efficiency

Extraction efficiency is defined as the quantity of salt retrieved from the surface as a percentage of the total amount originally on that surface. The extraction efficiency varies significantly among different situations. Some of the significant variables are:

- The method of extraction
- The proficiency of the operator
- The degree of roughness of the surface
- The size of the area from which salt is extracted
- The type and concentration of the salt
- The degree of corrosion and pitting of the substrate
- The extraction time
- The method of contamination (i.e., by artificial or natural methods)

Several studies have been conducted to evaluate the efficiency of extraction. For several laboratory studies, the researchers applied specific quantities of salts over defined areas to provide an average concentration as a control. In a few studies, the steel substrates were exposed to the salts (e.g., in an accelerated laboratory chamber or in atmospheric exposures). For these tests, the control concentration was determined using the total extraction method (boiling). The researchers assumed that this technique would extract 100% of the soluble salts. Some studies have questioned the validity of using artificially doped test panels to establish extraction efficiency.

From a review of the published literature one concludes that there is not enough data to develop specific extraction efficiencies for the various extraction procedures. A list of sources for extraction efficiencies is given below.

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APPENDIX E: Sources of Testing Equipment and Supplies

Information on suppliers of testing and analytical equipment may be found in the Journal of Protective Coatings and Linings Buyer's Guide. The following web sites also provide contact information and links to manufacturers of coating testing equipment and supplies. http://www.sspc.org/links/equip.html; http://www.paintsquare.com/bg/buying_guide_equip.cfm.

Readers may also search the World Wide Web using keywords such as analytical testing equipment, Bresle, chloride ion, coating test kit, conductivity, ferrous ion, ion detection tube, inspection, laboratory testing supplies, nitrate ion, paint testing equipment, and sulfate ion.