

SSPC: The Society for Protective Coatings

TECHNOLOGY UPDATE NO. 4

Field Methods for Retrieval and Analysis of Soluble Salts on Substrates

NOTE: SSPC-TU 4 has been superseded by the new SSPC-Guide 15, and will soon be withdrawn. This version is provided for reference purposes.

1. Scope

1.1 This Technology Update describes the most commonly used field methods for the retrieval and analysis of soluble salts on substrates.

2. Description and Use

2.1 Coatings applied on surfaces contaminated with soluble salts over a certain concentration exhibit diminished performance. This Technology Update is intended to assist in retrieving soluble salts from a substrate and determining their concentration. This information will aid in deciding whether additional cleaning is required before the substrate can be coated. Section 3 of the Technology Update discusses the various methods for retrieving salts from a surface. Section 4 discusses the analytical methods used to determine the concentration of the soluble salts on the substrate.

3. Retrieval Methods

3.1 CLASSES OF RETRIEVAL METHODS

3.1.1 Salt retrieval methods employed to help determine surface concentrations of salt on ferrous metals fall into three general classes:

A. Cell Retrieval Methods. A Bresle™ cell is a small adhesive patch which attaches to the structure forming a cell cavity. This cell is stuck onto the surface being checked by its self-contained adhesive edges. Distilled water is then injected into its center with a hypodermic needle. The patch fills up like a large paint blister. The liquid is then retrieved from the patch and tested for concentration of salts.

B. Swabbing or Washing Methods. A low conductivity fluid such as deionized water is used to extract salts from a steel surface. The method requires that the operator wear surgical latex rubber gloves to prevent cross contamination of the surface or the retrieved sample by salts naturally present on the surface of the skin.

C. "Total" Extraction Methods. The total extraction method involves the use of boiling deionized water to extract

salts from a steel sample. This method is intended to be used to determine salt levels on steel in a laboratory setting. It may be used as a reference method to derive retrieval rates for the cell and swabbing methods described above. It may also be used for field samples cut from a sample or test panels exposed in a field or lot.

The first two methods can be used to help characterize surfaces encountered in either laboratory or field settings. In general, "total" extraction methods are only useful under laboratory conditions. No field 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.

3.2 REAGENT WATER: All the retrieval procedures described in this document use deionized or distilled water as a retrieval fluid. The quality requirements for this reagent water are defined as follows:

The reagent water is distilled or deionized water with a specific conductance of about 1 μ S/cm. Distilled water may be purchased at grocery stores. 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.

3.3 ADHESIVELY BONDED CELL (BRESLE CELL): The Bresle sampling procedure is described in detail in ISO 8502-6, "Bresle Sampling Method."

3.3.1 Sample Acquisition Procedure

1. Remove the backing (with Bresle logo) and the foam circle from the test cell and apply the cell firmly and tightly to a dry test surface, either vertical or horizontal.
2. Insert the 5 mL syringe needle into the cell through its spongy perimeter, taking care not to inject beneath the cell or into the semitransparent test area. Evacuate the air from the test area by pulling back on the plunger.
3. Inject 3 mL of distilled or deionized water into the 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 syringe from the cell center (but not the spongy perimeter) and gently rub the top of the cell for 10 to 15 seconds to encourage dissolution of soluble salts.
5. Withdraw and reinject the water 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 water as possible and place it in a clean vial or other container.
6. If additional testing requires a higher volume of solution than that afforded by the extraction procedure, add deionized water to raise the sample volume up to the required level and note the new sample volume.
7. Use the water from step 6 to determine chloride ion concentration, ferrous ion concentration, or pH using one of the methods described in Section 4.
8. If additional samples are to be taken always use a fresh Bresle cell and clean syringe and needle; this will avoid cross contamination between samples.

3.3.2 Advantages of the Bresle Cell Method: The adhesively attached cells can conform to curved and irregular surfaces. Cells such as these are available in a variety of sizes; the most commonly used size retrieves salt from a surface of 12.5 cm². Smaller cell sizes permit assays of salt levels to be made at local corrosion sites such as craters or pits. The adhesively attached cells are commercially available.

3.3.3 Limitations of the Bresle Cell Method: The adhesively attached cells only accommodate a small amount of retrieval fluid. With the most commonly used cell size (12.5 cm² surface area), the actual quantity of cell fluid contacting the surface is a little over 3 mL. This can limit the range of analyses that can be performed to determine the surface salt levels. No in-line determination of conductivity can be performed with these cells. The Bresle cell will not adhere well to rusted surfaces, but it will adhere to clean or grit blasted surfaces so well there may be difficulty in removing the patch. The cell may leak through the hole introduced by the syringe.

3.3.4 Retrieval Efficiency: The efficiency of soluble salt retrieval is approximately 45% to 60% (see Appendix A).

3.3.5 Alternative Test Cell: A magnetically attached surface cell is described in Appendix B.

3.4 SWABBING OR WASHING METHOD

3.4.1 Materials and Equipment for Swabbing Method

1. Reagent Water: as defined in section 3.2.1.
2. Cotton: Use cotton balls of the highest quality, sterile, absorbent cotton such as found in first-aid kits. (Note:

chloride-free sponges of similar size may also be used.)

3. Gloves: Disposable vinyl or latex medical gloves must be worn to prevent contamination of the surface by salt found on the hands.
4. Two plastic beakers labeled A and B.
5. Graduated cylinder.

3.4.2 Procedure for Swabbing Method

1. Use a ruler and marker to outline a representative surface area of specific size (e.g., 15 cm by 15 cm).
2. Using distilled or deionized water and a graduated cylinder, pour a measured volume (e.g., 22.5 mL) of pure water into beaker A. (Note: The suggested sample area and volume give a 1:10 ratio of volume to area. This can simplify later calculations of salt contamination levels.)
3. Repeat step 2 with beaker B.
4. Using a pair of tweezers or plastic gloves, dampen a cotton ball in the water in beaker A and thoroughly swab the area measured in step 1. After swabbing, swirl the cotton ball in the water and then squeeze it against the inside of beaker A to extract most of the water from the cotton ball.
5. Repeat this swabbing, swirling, and squeezing operation four times with fresh cotton balls and then leave the cotton balls in the water in beaker A.
6. Use a fresh cotton ball to dry the measured test area and place it in beaker A.
7. Stir the water and cotton balls for two minutes to achieve thorough mixing.
8. Record the final volume. (If low, bring up to 22.5 mL with distilled water.)
9. Place a number of fresh cotton balls identical to those used in steps 4 through 7 above into beaker B. Let sit, covered, for at least three minutes. This will provide the control sample.

3.4.3 Retrieval Efficiency. The efficiency of soluble salt retrieval is variable, ranging between 25% and 35% (see Appendix A).

3.4.4 Advantages of Swabbing Method: The most pronounced benefit of the swabbing retrieval method is that it provides an inexpensive and simple method for acquiring samples of salt from any steel surface. Retrievals can be conducted on a range of surfaces without regard to surface irregularities or condition.

A second benefit of swabbing methods is that they can be used on large areas, giving reliable indications of general surface contamination by salts. They also provide sufficient sample size for several analyses to be performed on the retrieved contaminants for both species and level.

3.4.5 Limitations of Swabbing Method: The major limitations of swabbing retrieval are:



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1. Swabbing methods are difficult to perform in an overhead position. There is a distinct possibility that retrieved fluid is lost dripping from the swabs or running down the arms of the operator.
2. Swabbing methods have very low retrieval rates.
3. Swabbing is incapable of providing accurate levels of salt levels for small localized contamination such as that found in craters or pits.
4. There is a risk of contamination of a sample by the operator if the gloves become damaged. Check gloves for damage frequently. There is a risk of cross contamination from sample to sample if gloves are not cleaned between each sample.

3.5 TOTAL EXTRACTION METHOD

3.5.1 Materials Required: To perform a "total retrieval" using this laboratory reference procedure, the following items are required (all apparatus and sample containers should be previously cleaned with deionized or distilled water):

1. Hot plate, with thermostatic control.
2. Deionized water, conductivity no greater than $3 \mu\text{S/cm}$.
3. Inert glass granules to prevent bumping of boiling water. (Note: the use of Boileezers is not suggested, as these contribute ions to the water and buffer the pH of the extraction fluid on the alkaline side.)
4. Steel panels of known dimensions (e.g., $10 \times 15 \times 0.64 \text{ cm}$ [$4 \times 6 \times 1/4 \text{ in}$]), 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 "White Metal Blast Cleaning" using metallic grit showing a conductivity rating less than or equal to $3 \mu\text{S/cm}$ when tested in accordance with ASTM D 4940.
5. Stainless steel or Pyrex pans of dimensions no less than $15 \times 20 \times 5 \text{ cm}$ ($6 \times 8 \times 2 \text{ in}$).
6. Test sections of interest of dimensions no greater than $13 \times 18 \times 2.5 \text{ cm}$ ($5 \times 7 \times 1 \text{ in}$).
7. 500 mL graduated cylinder.
8. Stainless steel tongs.
9. Conical funnel.
10. 750 mL laboratory storage bottle.

3.5.2 Sample Acquisition Procedure

1. The following items are placed in the pans:
 - a. Approximately 350 mL of deionized water;
 - b. Between 5 and 10 anti-bumping granules;
 - c. A test section.
2. The pan is placed on the hot plate and raised to boiling temperature over a period no less than ten minutes. The assembly is maintained at boiling for one hour. The fluid level in the pan shall be maintained so as to completely submerge the test section. If fluid is lost by boiling or evaporation it shall be replenished during the test.

3. At the end of the test the hot plate is turned off. The pan is removed from the hot plate and the fluid is allowed to cool for at least one half hour.
4. The steel test section is removed from the pan and drained over the pan by holding with a pair of stainless steel tongs. When the panel has drained dry, the test section is removed from the work area.
5. The fluid in the pan is transferred to the graduated cylinder via the conical funnel. Sufficient deionized water is added to the graduated cylinder to bring the volume of material up to 500 mL.
6. The total fluid is thoroughly mixed by transferring between the storage bottle and the graduated cylinder.

3.5.3 Retrieval Efficiency: The efficiency of soluble salt retrieval is at least 90%.

3.5.4 Advantages of Total Extraction Method: This method provides a benchmark for determining the relative retrieval efficiency of cell and swabbing retrieval methods.

3.5.5 Limitations of Total Extraction Method: This method is unsuited for use in the field.

4. Analytical Methods

4.1 QUANTITATIVE ANALYSIS: The second part of this Technology Update discusses the most common analytical methods used to determine the amount of soluble salt contamination on a steel substrate. The following substances and quantities are described:

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

4.1.1 Precision and Accuracy of Quantitative Measurements: The precision of each quantitative analytical procedure is given in the section describing each method. Accuracy of each method is given when available.

4.2 FIELD MEASUREMENT OF CONDUCTIVITY (TOTAL SOLUBLE SALTS): This provides an accurate measurement of solution conductivity ($\pm 1 \mu\text{S/cm}$). From this conductivity measurement an estimate of the total soluble salts can be made.

First, determine the conductivity of the extract. Place the lower end of a calibrated pocket-sized conductivity meter into the extracted water from Section 3.4.2, Step 8 or from Section 3.4.2, Step 6. This is Reading One. Record conductivity in $\mu\text{S/cm}$.

Note: If a conductivity meter is used, transfer about 10 milliliters of the extracted solution into the cup of the conductivity meter. Select the appropriate range and record the conductivity reading in $\mu\text{S/cm}$. Convert to other units as described in Section 4.2.1.

