Advances in Technology and Standards for Mitigating the Effects of Soluble Salts

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Chlorides and other soluble salts can cause significant reductions in coating life and accelerated corrosion of metals if not removed prior to coating application. In the last several years, industry has developed new techniques for extracting salts using devices such as sleeves, magnetic cells, and filter paper. Also available are improved field methods for analyzing conductivity, chlorides, and sulfates. In addition, organizations such as SSPC and the International Organization for Standardization (ISO) have issued standard methods to guide the specifiers, applicators, and inspectors in selecting and utilizing the methods. Following a review of recent studies on the effect of soluble salts on coating performance, this article reviews the most commonly used procedures for extraction, analysis, and removal of salts from steel substrates. In addition, the article provides guidance for specifiers to assure that the levels of salt remaining on the surface will not be detrimental to the coating service life.

Background

Soluble salts are very widespread on government and industrial substrates. A major source is sea salt, which affects ships, offshore structures, waterfront structures, and inland facilities within several miles of the sea. Soluble salts can also arise from chemical processes, cooling towers, and burning of sulfur-containing coal.

Effects of Soluble Salts on Coating Life

General Effects

Soluble salts can affect the ability of coatings to protect steel in several ways. Salts that remain on the substrate can result in blistering of the coating. This phenomenon, known as osmotic blistering, can produce pressures of several thousand psi, which is enough to cause the coating to disbond. Osmotic blistering occurs because the salt on the substrate in the presence of moisture forms a highly concentrated solution. The water on the exterior of the film is at a much lower concentration. The water is then drawn through the coating film, which behaves like a semi-permeable membrane. Figure 1 illustrates osmotic blistering.

In addition, salts, particularly chloride or sulfate, can increase the rate of corrosion of metals. These salts act as catalysts, accelerating the anodic reaction.

Atmospheric and Immersion Studies

Several studies have been undertaken demonstrating the influence of soluble salts on the lifetimes of coatings. Morcillo[1] evaluated a series of conventional coatings over three to four levels of sodium chloride and ferrous sulfate. He exposed the coatings for 4.5 years in
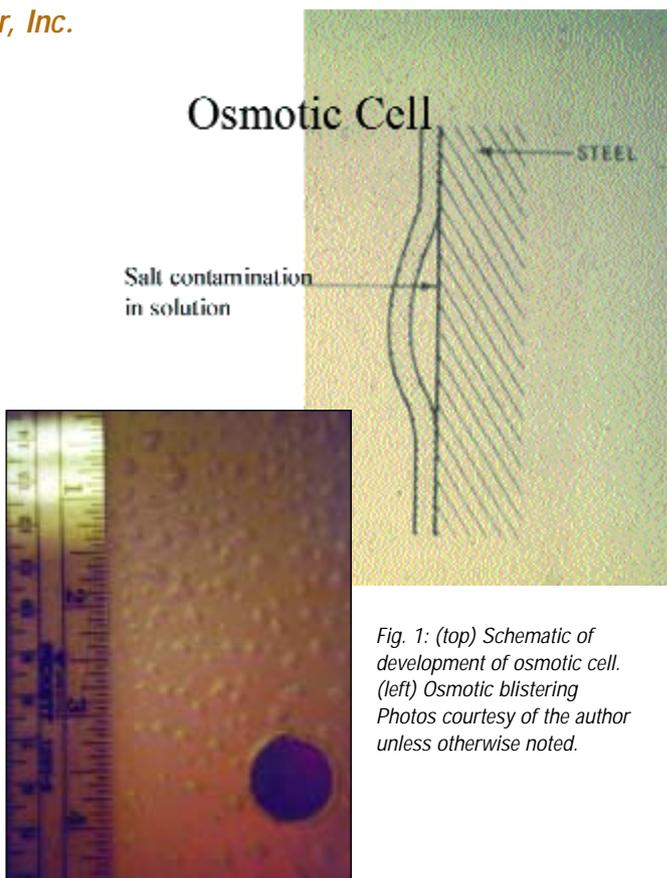

Fig. 1: (top) Schematic of development of osmotic cell. (left) Osmotic blistering
Photos courtesy of the author unless otherwise noted.

Table 1:
ISO Summary on Acceptable Chloride Levels

Coating System	"Safe" Level of Cl*	Test Exposure
Epoxy phenolic (1 coat)	1	100% humidity, 40 C
Epoxy polyamide (3 coats)	5	condensing humidity
Coal tar epoxy (10 mils)	50	500 hrs immersion, deionized water
Fusion-bonded epoxy	<3	48 hrs immersion, 65 C water, 1.5 volts cathodic disbondment
Tank lining epoxy	10–20	pressure immersion (90 F, 50 psi)**
Epoxy mastic (2 coats)	7	pressure immersion (90 F, 50 psi)**

* units of micrograms per square centimeters   ** 50 psi = 3.4 bar; 90 F = 32 C

a marine atmosphere and up to 14 years in industrial, rural, and urban atmospheres. SSPC[2] evaluated typical bridge coating systems in accelerated laboratory tests and on test bridges over chloride- and sulfate-contaminated steel.

Each of these atmospheric studies determined that coating failure was more pronounced at chloride levels starting at about 10 $\mu g/cm^2$, but that zinc-rich coatings had a greater tolerance for chloride. Also, coatings had a higher tolerance for sulfate ions than for chloride ions.

In a more recent study, Mitschke[3] studied immersion coatings. He evaluated a series of nine epoxy, epoxy novolac, and epoxy phenolic tank linings over different levels of chloride immersed in tap water at different temperatures. He examined the panels periodically, defining failure when 20% of the surface was blistered. At 75 F (24 C) after 13 months, the threshold chloride levels (concentrations which could be tolerated) ranged from 4 $\mu g/cm^2$ to 20 $\mu g/cm^2$. As the temperature was raised, the threshold levels decreased. At 190 F (88

C), an epoxy phenolic and an epoxy novolac exhibited thresholds of 17.5 $\mu g/cm^2$ and 7.4 $\mu g/cm^2$, respectively. All the other coatings failed even with no measurable chloride on the surface. The author concluded that even very small quantities of chloride on the surface reduced the service life of these coatings.

Determining the Maximum Acceptable Levels
A subcommittee from ISO reviewed published data on the performance of various coatings in different exposures when applied over chloride.[4] Some of the data are shown in Table 1.

Most of the above tests were based on applying the coating over a pre-measured level of chloride or sulfate. This procedure provides the researcher with a relatively accurate measure of the level of salt on the surface.

To assess the potential impact of salts deposited on a surface from the environment, one requires methods for detecting the salt after the deposition.

Detection is typically a two-step process: first the salt is extracted from the surface. Then, the extracted solution is analyzed for the type and quantity of salt.

The next section describes the most common methods used by the coatings industry for extraction and analysis.

Extracting Salts from Surfaces
The industry has developed several field methods for extracting soluble salts from surfaces to be painted. The most prominent are swabbing and adhesive patch (Bresle [Fig. 2 and 3]). Others include the adhesive sleeve (Fig. 4) and wetted filter paper. (See discussion below on conductivity.)
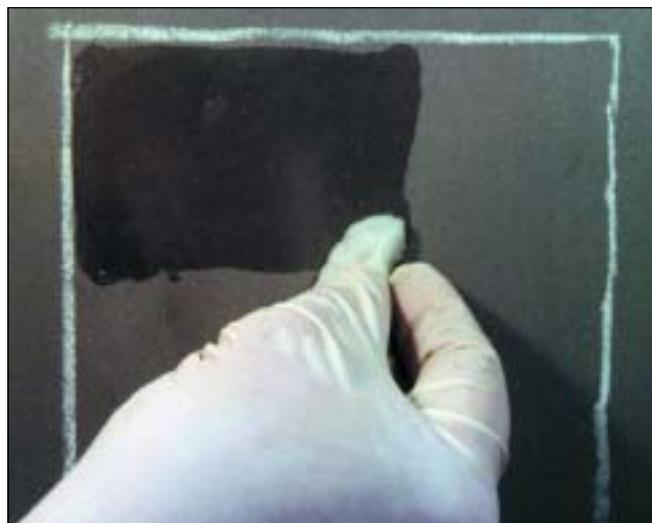
Fig. 2: Swabbing in progress for extraction of soluble salts from a steel surface

Table 2:
Summary of Published Data on Extraction Efficiency

Method	Salt/Concentration	Efficiency	Source
Swabbing	Cl: 20, 200, 250 & 500 $\mu g/cm^2$	43% to 78% avg: 56%	Reference 2
Swabbing	SO_4: 20 & 200 $\mu g/cm^2$	27% to 42% avg: 34%	Reference 2
Swabbing	NH_4: 10 & 100 $\mu g/cm^2$	24% to 86% avg: 55%	Reference 2
Swabbing	Cl: 5, 15, 50, 100 $\mu g/cm^2$	82% to 120% avg: 100%	Reference 6
Swabbing	SO_4: 100, 150, 200 $\mu g/cm^2$	80% to 86% avg: 83%	Reference 6
Bresle	Cl: 15, 50 $\mu g/cm^2$	133% to 166% avg: 150%	Reference 6
Bresle	SO_4: 100, 150 $\mu g/cm^2$	57% to 126% avg: 92%	Reference 6
Bresle	Cl: 10, 25, 50 $\mu g/cm^2$	42% to 90% avg: 62%	Reference 7
Bresle	Cl: 10, 25, 50 $\mu g/cm^2$	17% to 28% avg: 22%	Reference 7
Bresle	Cl: 10, 25, 50 $\mu g/cm^2$	26% to 53% avg: 35%	Reference 7
Bresle	Cl: 1.5, 3.0, 6.0, 18 $\mu g/cm^2$	20% to 80% avg: ~60%	Reference 8

Fig. 3: (left) Adhesive patch (Bresle method) for extracting soluble salts

Fig. 4: (right) Adhesive sleeve for soluble salt extraction
Courtesy of Chlor*Rid International

the swabbing method gave extraction efficiencies in the range of 30–40% while the Bresle cell efficiencies were in the range of 40–60%.[5] More recent data and reassessment of earlier data have shown this to be an over-simplification. For the swabbing method, extraction efficiencies have ranged from 20–90% while those for the Bresle cell have ranged from 20% to over 100%. Measured extraction efficiencies of greater than 100% can arise from inaccuracies (e.g., non-uniformity) in the initial deposition of the salt or in the extraction or analytical procedures. For the newer methods identified above, there is little if any objective data available. Table 2 presents a summary of the published data on extraction efficiency.

Analyzing Soluble Salts
The most common species analyzed is chloride ion. There

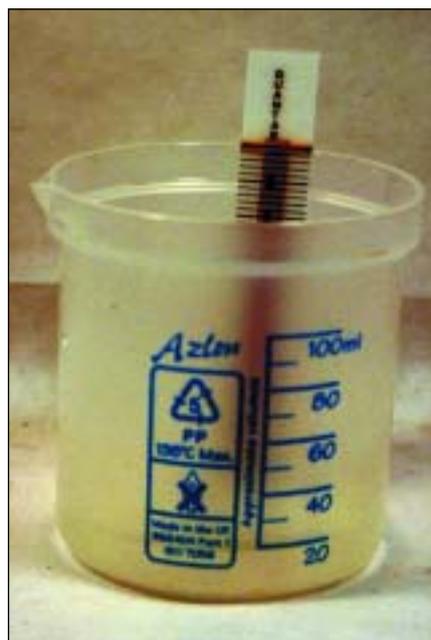Fig. 5: (above) Ion detection tubes

Fig. 6: (left) Paper chromatography strips placid in extracted liquid to determine chloride concentration

These are described in SSPC-TU 4, "Field Methods for Retrieval and Analysis of Soluble Salts on Substrates," and other publications.

Unfortunately, the efficiency of extraction varies quite significantly among these methods and within a given method. Earlier data published by SSPC had indicated that

are three common field methods for analyzing chloride. These include ion detection tubes (Fig. 5), paper chromatography strips (Fig. 6), and titration. Each of these is sufficiently accurate and precise for determining the concentration of chloride in the extracted liquid. The ion detection tube is the most sensitive. (Of the three methods, it

Fig. 7: Pocket conductivity meter

Fig. 8: (below) Meter measures conductivity of pre-wetted filter paper placed on substrate
Courtesy of Elcometer Ltd.

can measure the lowest level of chloride.)

Conductivity measures the ability of the extracted liquid to carry an electric current. It is a measure of the total dissolved salts, but does not provide direct information on the specific chemical ions. There are several types of field conductivity meters available. One is the pocket conductivity meter shown in Fig. 7. A relatively new device is a meter that measures the conductivity of pre-wetted filter paper placed on the substrate (Fig. 8). The units are furnished with calibration solutions. They are sensitive to conductivities of 2–3 microsiemens/cm. (This concentration is approximately equivalent to 6–10 parts per million [ppm] chloride if one assumes that chloride is the only soluble ion.) These instruments are also sufficiently accurate and precise for measuring the soluble salts at levels that can affect coating performance.

Field test kits are also available for measuring the levels of soluble sulfate and ferrous ions as well as nitrate ions. These salts are much less frequently specified than are

chloride and conductivity.

Methods for Removing Soluble Salts

Traditionally, the most productive and effective method for preparing steel for application of a coating is dry abrasive blasting. Dry blasting, however, is most suitable for mechanically breaking up layers of rust, mill scale, and coating, and for eroding the steel to produce a profile. It is not intended to remove water-soluble salt or grease and oil. For more effective removal of salts, some form of water in the surface preparation is desirable.

Waterjetting at high-and ultra-high-pressures of 10,000 to 35,000 psi (666 to 2,333 bar) is generally effective at dissolving and removing any salts the waterjet can reach. However, these jets by themselves (i.e., without abrasive) are relatively ineffective at removing tight rust or mill scale. In addition, water alone cannot produce a surface profile normally recommended for high-performance coatings. These systems are, however, suitable for maintenance painting where the surface previously had a profile that can be restored.

Another approach is wet abrasive blasting, in which water and abrasive are utilized. One version is to inject water, typically at 1,000–3,000 psi (66 to 200 bar), into a conventional air abrasive blasting nozzle. Alternatively, one can inject abrasive into a waterjetting system. These techniques are described in SSPC-TR 2/NACE 6G198, Wet Abrasive Blast Cleaning. Some recent data on the effectiveness of wet and dry methods in removing chlorides are given in Table 3.[9–12]

Table 3:
Comparison of Salts from Wet and Dry Cleaning Methods

Method	Remaining Salt ($\mu g/cm^2$)	% Extracted	Source
Wet blasting	0–3.2	avg*: 96.2%	Reference 9[a]
Waterjetting (35 ksi)	0–2.4	avg*: 95.9%	Reference 9
Hand tool (SP 2)	160–288	avg*: 43.8%	Reference 9
Power tool (SP 3)	212–296	avg*: 35.4%	Reference 9
Blast (SP 6)	44–68	avg*: 83.0%	Reference 9
UHP waterjet	1.6–1.8	avg: 93.5%	Reference 10[b]
Blast (SP 10)	3.3	84%	Reference 10
Needle gun (SP 3)	11.4	3%	Reference 10
Wire brush (SP 2)	15.2	9%	Reference 10
Blast (SP 5)	<3.2–3.4	avg: 90.2%	Reference 11[c]
Power tool (SP 3)	16.2–24.1	avg: 43.5%	Reference 11
SP 3 + steam	8.6–12.9	avg: 69.9%	Reference 11
Power tool (SP 11)	7.0–13.9	avg: 72.1%	Reference 11
SP 11 + steam	3.9–7.7	avg: 84.5%	Reference 11
Power tool (SP 3)	22–97	avg: 45.4%	Reference 12[d]
Power tool (SP 11)	41–124	avg: 17.2%	Reference 12

[a] Salts extracted by Bresle and analyzed by conductivity
[b] Methods not included in paper
[c] Salts extracted by boiling and analyzed by selective ion electrode
[d] Salts extracted by swabbing and analyzed by conductivity

Table 4:
Selected Contents of SSPC-TU 4

Section	Description
3.3	Adhesively Bonded Cell (Bresle Cell)
3.4	Swabbing or Washing Method
3.5	Total Extraction Method
4.2	Field Measurement of Conductivity (Total Soluble Salts)
4.3	Field Detection of Chloride Ion by Kitigawa Tube
4.4	Field Detection of Chloride Ion by Quantab Method
4.5/4.6	Field Detection of Chloride Ion by Titration Methods 1 and 2
4.7	Laboratory Reference Method for Detection of Chloride Ion by Titration
4.8	Qualitative Field Detection of Ferrous Ion
4.9	Quantitative Field Detection of Ferrous Ion
4.10	Field Detection of Sulfate Ion
4.11	General Method for Estimating Surface Concentration of Salts
Appendix C	Estimating Surface Salt Concentration

Other surface preparation methods have also been evaluated. Power and hand tool cleaning methods are very ineffective in removing salts. Several proprietary treatments have been developed. These entail spraying a water solution of a proprietary chemical to the surface. The concentrations and the pressure vary, depending on the particular product. Several owners and specifiers have rated these products very highly. However, there is little published data to corroborate these claims.

Specifying Soluble Salt Removal

This section provides guidance to specifiers and owners in preparing and implementing a specification to assure adequate salt removal. It is essential that the specification include clear language for

- sampling (where to measure the surface),
- extraction,
- analysis, and
- acceptance level.

Sampling

Presently, there is little consensus within the industry regarding the number or location of samples. The U.S. Naval Facilities Engineering Command (NAVFAC) guide specification, Interior Coating of Welded Steel Petroleum Fuel Tanks, stipulates three tests for the first 100 m^2 (1,100 ft^2) plus one additional test for each additional 200 m^2 (2,200 ft^2). The specification also instructs the inspector to concentrate testing at areas of coating failure, pitting, and welds. Others have also suggested selecting the areas with the greatest likelihood of high salt levels (e.g., under bridge expansion joints or at the base of ballast tanks). SSPC and NACE are drafting procedures for sampling.

Extraction and Analysis

SSPC-TU 4 includes specific methods for extraction and analysis for most of the techniques described above. (A revised edition is expected in 2002 that will include more definitive descriptions of methods.) The relevant contents of TU 4 are shown in Table 4.

The ISO subcommittee identified above also contacted coating manufacturers from around the world for recommendations on the acceptance level for chloride.[4] Their recommendations along with those of the U.S. Navy and a Norwegian classification society[13] are shown in Table 5. For each source, the original report provides recommendations for the specific methods of extraction and analysis. These requirements are primarily based on marine exposures such as ocean-going ships and offshore structures. Studies by Morcillo, Appleman, and others have demonstrated that the susceptibility of coatings to early degradation from salts is strongly dependent on the type of coat-

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List of ISO Standards on Soluble Salts

ISO 8502-2:1992 Preparation of Steel Substrates before Application of Paints and Related Products—Tests for the Assessment of Surface Cleanliness

Part 1: Field Test for Soluble Iron Corrosion Products (ISO/TR 8502-1:1991)

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 the Conductometric 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 11: Field Method for the Turbidimetric Determination of Water-Soluble Sulfate (ISO/AWI 8502-11)

Part 12: Field Method for the Titrimetric Determination of Water-Soluble Ferrous Ions (ISO/DIS 8502-12)

Part 13: Field Method for the Determination of Soluble Salts by Conductometric Measurement (ISO/WD 8502-13)

Table 5: ISO Survey of Manufacturers' Recommended Chloride Limits

Source	Level for Immersion	Level for Atmospheric
Coating suppliers	3–10	5–25
U.S. Navy	3	5
Det Norske Veritas[10]	2	—

units = $\mu g/cm^2$

ing, its thickness, and the service environment.

Specification Language

Listed below are several examples of clauses that could be incorporated into procurement documents.

• Extraction by Patch Cell—"Extract salts from the surface in a minimum of 3 test areas within a 100 sq ft (9 sq m) section at representative units of the structure or vessel in accordance with Section 3.3 (Patch Cell) from SSPC-TU 4."

• Chloride Analysis by Ion Tube—"Test each portion of liquid extracted for soluble chloride ion in accordance with Section 4.3 of SSPC-TU 4 (ion detection tubes); record the ppm."

Conclusion

Soluble salts, particularly chlorides, are widely prevalent in many industrial exposures where coatings are applied. If not removed prior to application of the coatings, the soluble salts can adversely affect coating lifetime, resulting in early degradation and failure. The most successful means of removing the salts is a combination of water and abrasive, but even this technique will not be 100% effective.

Detection of salt on the surface entails extraction, then analysis for conductivity or for a specific salt such as chloride. Standard extraction methods are available, but they give variable results which depend on the operator, the concentration of salt, the roughness of the steel, and other factors. Standard field analytical methods are available for analyzing chloride and conductivity. These methods provide consistent, accurate results.

The industry is moving toward consensus on acceptable levels of salt. There is, however, relatively little solid data to support these levels. Guidelines and consensus standards are available to assist specifiers in mitigating the effects of soluble salts.

References

1. Manuel Morcillo and Joaquín Simancas, "Water-Soluble Contaminants at the Steel/Paint Interface: Their Effect in Atmospheric and Marine Services," Paper 12 of the *Proceedings* of PCE 97, The Hague, The Netherlands, March 17–20, 1997 (Pittsburgh, PA: Technology Publishing Co., 1997).

2. B.R. Appleman, S.K. Boocock, R.E.F. Weaver, and G.C. Soltz, "Effect of Surface Contaminants on Coating Life," Report No. FHWA-RD-91-101, Federal Highway Administration (June 1991).

3. Howard Mitschke, "Effects of Chloride Contamination on Performance of Tank and Vessel Linings," *JPCL* (March 2001), pp. 49–56.

4. ISO/PDTR 15235 "Preparation of Steel Substrates before Application of Paint and Related Products—Collected Information on the Effect of Levels of Water Soluble Salt Contamination before Application of Paints and Related Products," prepared by ISO TC35/SC12 Working Group 5, International Organization for Standardization (November 18, 1999).

5. B.R. Appleman, J.A. Bruno, Jr., and R.E.F. Weaver, "Maintenance Coating of Weathering Steel: Interim Report," Publication No. FHWA-RD-91-087 (Washington, DC: Federal Highway Administration, January 1992), SSPC Publication 91-04.

6. S. Flores, J. Simancas, and M. Morcillo, "Methods for Sampling and Analyzing Soluble Salts on Steel Surfaces: A Comparative Study," *JPCL* (March 1994), pp. 76–83.

7. Simon K. Boocock, "SSPC Research and Performance Testing of Abrasives and Salt Retrieval Techniques," *JPCL* (March 1994), p. 28.

8. Unni Steinsmo and Sten B. Axelsen, "Assessment of Salt Contamination and Determination of Its Effect on Coating Performance," *Proceedings* of the PCE 98 Conference and Exhibition, The Hague, The Netherlands, April 1–3, 1998 (Pittsburgh, PA: Technology Publishing Co., 1998), pp. 71–85.

9. A. Forsgren and C. Appelgren, "Comparison of Chloride Levels Remaining on the Steel Surface After Various Pretreatments," *Proceedings* of PCE 2000, Genoa, Italy, March 8–10, 2000 (Pittsburgh, PA: Technology Publishing Co., 2000).

10. Bill Allen, "Evaluating UHP Waterjetting for Ballast Tank Coating Systems," *PCE* (October 1997), p. 38.

11. Kenneth A. Trimber, "An Investigation into the Removal of Soluble Salts Using Power Tools and Steam Cleaning," *Proceedings* of the 7th SSPC Technical Symposium, SSPC Publication 88-03 (1988), pp. 56–67.

12. John W. Peart, "The Effectiveness of Power Tool Cleaning as an Alternative to Abrasive Blasting," Report No. NSRP 0447, National Shipbuilding Research Program (June 1995).

13. E. Askheim, "Ballast Tank & Cargo Holds in DNV's Guidelines for Corrosion Protection of Ships," *PCE Guide to Marine Coatings* (Pittsburgh, PA: Technology Publishing Co., 2000), pp. 332–341.