

SOLUBLE SALT CONTAMINATION: TESTING AND CLEANING BEFORE COATING

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ABSTRACT

This paper focuses on the often-overlooked issue of soluble salt contamination testing and removal during surface preparation prior to coating application. Soluble salts, including chlorides, sulfates, and nitrates, left on the surface prior to coating application can lead to osmotic blistering, corrosion, and coating failures. Several standards provide methods to test for soluble salts. However, there is no industry consensus on acceptable contamination levels given the diversity of coating systems and environments. This paper serves as a guide intended to shed light on the specific issue of soluble salt contamination, particularly on carbon steel surfaces. By addressing the nuances and challenges posed by soluble salt contaminants, this paper aims to equip engineers and professionals in the field with the knowledge and tools necessary to navigate the complexities of surface preparation and ensure the integrity and longevity of coating systems on carbon steel surfaces.

Key words: Soluble salts, surface preparation, delamination, blistering, corrosion, extraction, analysis, conductivity

INTRODUCTION

Surface preparation is known to be the most important factor influencing the life of a coating system applied to a metal surface. One of the critical aspects of surface preparation is removing contamination that may lead to blistering, delamination, or general failure of the coating systems. Both visible (grease, oil, or dirt) and non-visible (soluble salts) are equally deleterious to coating systems. Non-visible contaminants are often overlooked for two main reasons: the detrimental nature of the contaminant is not fully understood, and additional testing measures must be implemented to reveal the presence of the contamination.

This paper aims to provide engineers and professionals with the knowledge and tools needed to effectively address soluble salts during surface preparation for coating application on carbon steel surfaces through both testing and removal strategies. The discussion will involve understanding the origins and impacts of soluble salts, using reliable testing methods, specifying appropriate cleaning methods and salt thresholds, and tailoring project-specific requirements to ensure long-term protection and integrity of facilities.

WHY SOLUBLE SALTS ARE A PROBLEM: OSMOTIC BLISTERING

The most common coating failure due to salt contamination is osmotic blistering. Osmotic blistering occurs when water permeates through the coating and reacts with residual salts left under the coating system.¹ When residual soluble salts react with moisture, an aggressive corrosion environment is developed. As salts dissolve in the water that has been pulled through the coating system, the corrosion cell and growing solution causes an osmotic force on the coating film. As corrosion progresses and the osmotic force grows the coating system detaches from the surface of the steel, resulting in coating failure.

SOURCE OF SOLUBLE SALTS

The most common and damaging soluble salts found in steel surfaces are chlorides (Cl^-), sulfates (SO_4^{2-}),¹ and nitrates (NO_3^-),² collectively referred to as “CSN” throughout this report. CSNs can be present on new or used, rough or smooth steel surfaces. The sources of salt contamination are numerous. Contamination could occur during transportation, storage, and in service exposures.¹ The method of surface preparation can contribute to further contamination due to contaminated abrasive³ or water during blasting and washing. Salts can be deposited from the atmosphere in marine environments, during dew, fog, and rain. Some areas on facilities that are particularly susceptible to soluble salt contamination include, but are not limited to:

- Weld seams
- Corroded or pitted areas
- Areas with failed coatings

WHAT ASPECTS CONTRIBUTE TO THE RATE OF FAILURE

Predicting the exact reduction in coating lifespan due to the presence of soluble salts is complex. Soluble salt contamination issues are not resolved with time. Some conditions may promote coating failure in one year or less, while other conditions may cause the same result, but only after several years of service. Environmental factors that influence the rate of coating failures from soluble salt contamination include:

- Amount and type of contaminant on the surface⁴
 - Increasing salt concentration accelerates the rate of attack.
 - Chloride salt contamination has been found to be more critical than sulfate contamination.
- Coating system and thickness
 - Zinc-rich primers have been found to have more salt tolerance.⁴
 - Coatings with less water permeability will generally experience a slower rate of attack than more permeable coatings.
 - A thicker layer of coating will permit less water migration than thin layer of the same coating material over the same period of time.^{1,5}

- Temperature
 - Increasing temperature will increase the rate of water permeability through a coating and will increase the rate of corrosion.¹
- Service environments¹
 - Coating systems exposed in atmospheric environments are more tolerant to salt than immersion services.
 - Studies have found that linings used in gasoline and water immersions had lower thresholds for chloride than immersions in water only.

REMOVAL OF SOLUBLE SALTS

Soluble salts are relatively easily removed from smooth surfaces.⁶ Blasted steel, however, has a porous surface (as shown in Figure 1) and offers a substantial surface area for the adsorption or absorption of various ions.³ This characteristic makes salt removal challenging even on visibly clean and abraded surfaces. The removal of soluble salts on surfaces with compromised coatings, corrosion products, and pitting poses an even more complex problem. The salt contaminants can remain in the recesses of corrosion pits, often beneath the corrosion product. To adequately remove salts from pitted areas, not only must the corrosion product be removed, but the contaminants must also be flushed from within the corrosion pits. After washing, the aggressive solution that was inside the pits may deposit on the surface, contributing to flash rusting, which also needs to be removed prior to coating. Challenging geometries will always pose challenges to establishing contaminate free surfaces. For example, the industry has not developed a remediation method to remove salts in tight crevices.⁶

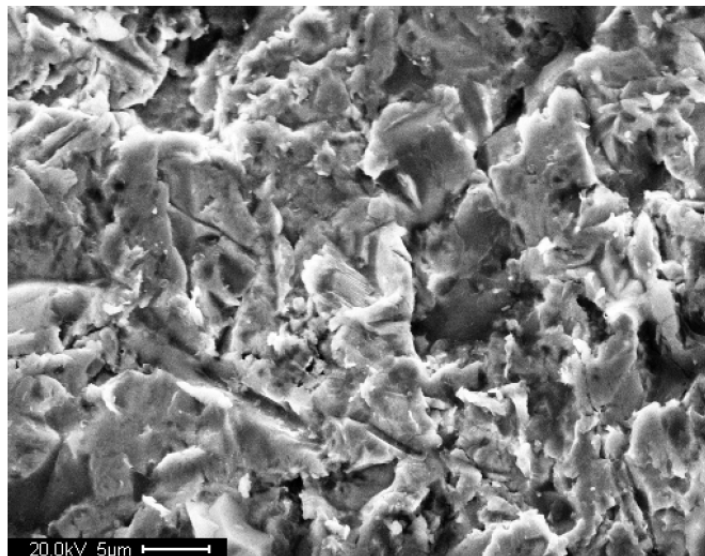


Figure 1. Reproduction of SEM image of virgin A588 steel surface after blast cleaning.⁸

Some of the most effective approaches for removing soluble salts involve wet methods with abrasives, such as ultra-high pressure waterjetting (UHP-WJ) with abrasive, high-pressure waterjetting (HP-WJ) with abrasive, and wet abrasive blast cleaning.⁷ Salt remover or chemical additives can also be used.⁷ Care must be used in selecting additives, because some have been found to be ineffective.^{8,9} Results of conductivity testing and chloride testing on steel plate surfaces before and after UHP-WJ with abrasive are shown in Figure 2.⁷ Each of the conductivity results presented are averages from three measurements. In this study, corroded surfaces had a higher deviation between the three values than blasted surfaces, suggesting that salts may be unevenly distributed in corrosion products.⁷

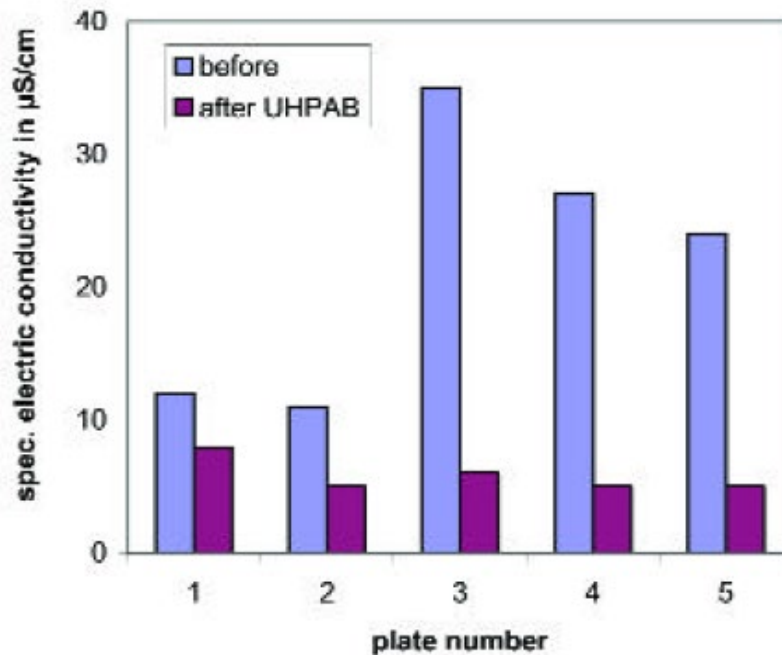


Figure 2. Reproduction of results of conductivity measurements before and after ultra-high pressure waterjetting with abrasive.⁷

TESTING FOR SOLUBLE SALTS

Soluble salts are not visible and must be tested for using chemical means. There are several standards that provide methods to measure the concentration of soluble salts on substrate surfaces in the field:

- SSPC Guide 15, *“Field Methods for Retrieval and Analysis of Soluble Salts on Steel and Other Nonporous Surfaces.”*¹¹
- ISO 8502, *“Preparation of steel substrates before application of paints and related products – Tests for the assessment of surface cleanliness (Parts 5, 6, and 9)”*¹²
- ANSI/NACE SP0508-2017, *“Methods of Validating Equivalence to ISO 8502-9 on Measurement of the Levels of Soluble Salts”*¹³

Quantitative field-testing methods require two steps: salt extraction and extraction analysis. Each test method differs by either (or both) the method of extraction or the method of analysis.

Extraction can be performed using an extraction solution that is exposed to the testing surface using a swab,¹¹ extraction sleeve (ISO 8502-5¹⁴), or patch (ISO 8502-6¹⁵) as shown in Figure 3. An extraction solution will be exposed to the surface using these methods and will dissolve salts present on the surface. Each standard specifies the length of time and technique used to expose the solution to the surface. The methods and standards consider the surface area tested, the volume of the fluid, solution contact time, solution agitation, and the susceptibility of the test solution to contamination.

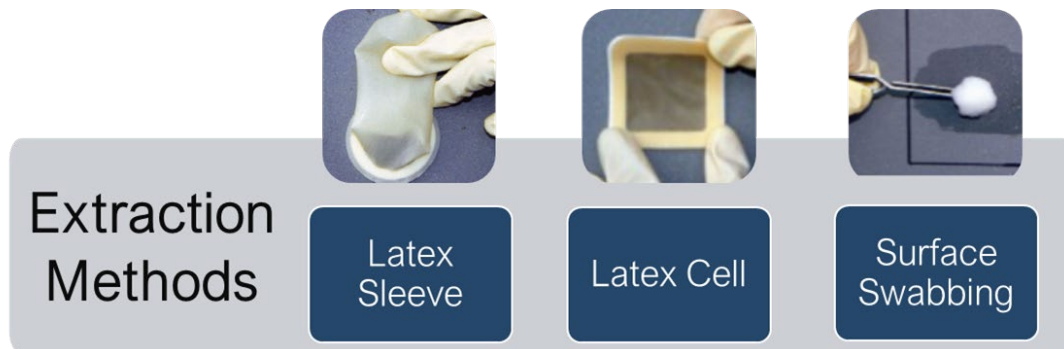


Figure 3 Common Extraction Methods.

Extraction efficiency is one of the largest differentiators for each testing method. Extraction efficiency is defined as the quantity of salt retrieved from the surface as a percentage of the total amount originally on the surface. Some studies found that the efficiency for the latex sleeve, latex cell, and surface swabbing were high for high chloride concentrations.² The efficiency decreased significantly for chloride concentrations at 3 and 5 $\mu\text{g}/\text{cm}^2$ with the sleeve test, but remained consistent with the other test methods. Extraction efficiency was found to be less effective for several methods when used on rusted and pitted steel panels.³ Literature has also found that the longer salt contamination sits on the surface, the lower the efficiency of extraction.⁶ No extraction method is completely efficient, meaning no test will collect all the salt that is on the surface.³

Once an extraction is made, the solution is then analyzed for salt concentration. Analysis can be performed to provide the conductivity of the solution (a collective number of all conductive ions in solution)¹⁶ or an ion specific concentration (the concentration of chlorides, sulfates, or nitrates).¹¹ Conductivity and sulfate concentration can be measured using a meter. Chloride and Nitrate ion concentration can be measured with a titration tube. Figure 4 provides a pictorial sequence of extraction and analysis performed using ion specific testing on a steel surface following the sleeve method in SSPC Guide 15¹¹ and ISO 8502-5.¹⁴

Conductivity measurements are provided in units of micro siemens per centimeter ($\mu\text{S}/\text{cm}$). Ion specific measurements are typically reported in the form of weight of salt per a unit area or micrograms per square centimeter ($\mu\text{g}/\text{cm}^2$). Titration results are often read as parts per million (ppm). When salts are extracted and analyzed in a solution taken from a known volume of liquid and a known surface area, it is easy to convert between ppm and $\mu\text{g}/\text{cm}^2$. In fact, some testing kits are pre-sized so that 1 ppm is equivalent to 1 $\mu\text{g}/\text{cm}^2$.

Unfortunately, it is not feasible to establish an empirical relationship between field measured conductivity and ion-specific measurements due to the unknown variation of ions present in the extraction solution. Several theoretical conversions have been formulated to facilitate comparisons between conductivity and chloride concentrations. These conversions assume that various concentrations of chloride ions contribute to the solution conductivity. Figure 5 shows the theoretical relationship for a typical patch cell compared to three salt solutions.⁶

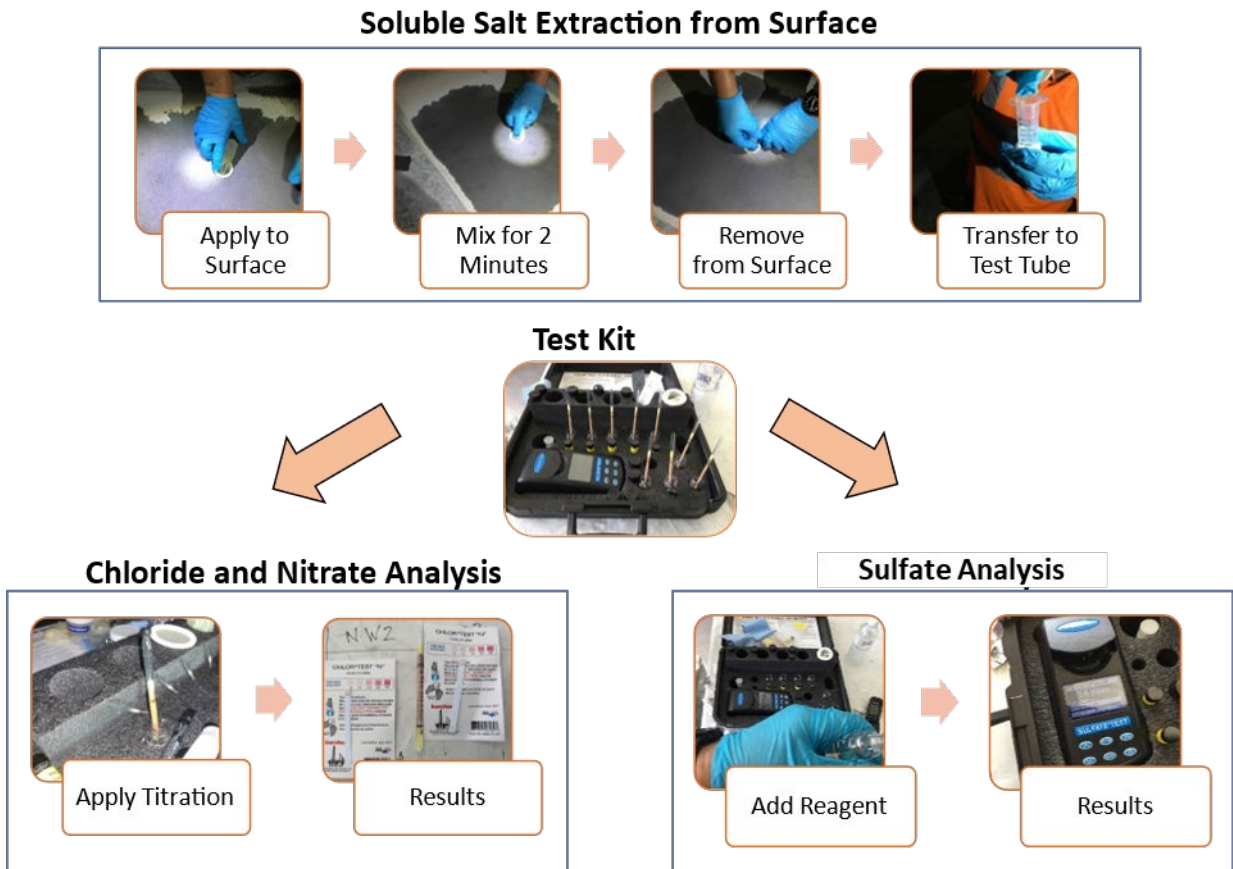


Figure 4: Pictorial sequence of events during ion-specific measurements of soluble salts using the latex sleeve methodology using SSPC Guide 15¹¹ and ISO 8502-5.¹⁴

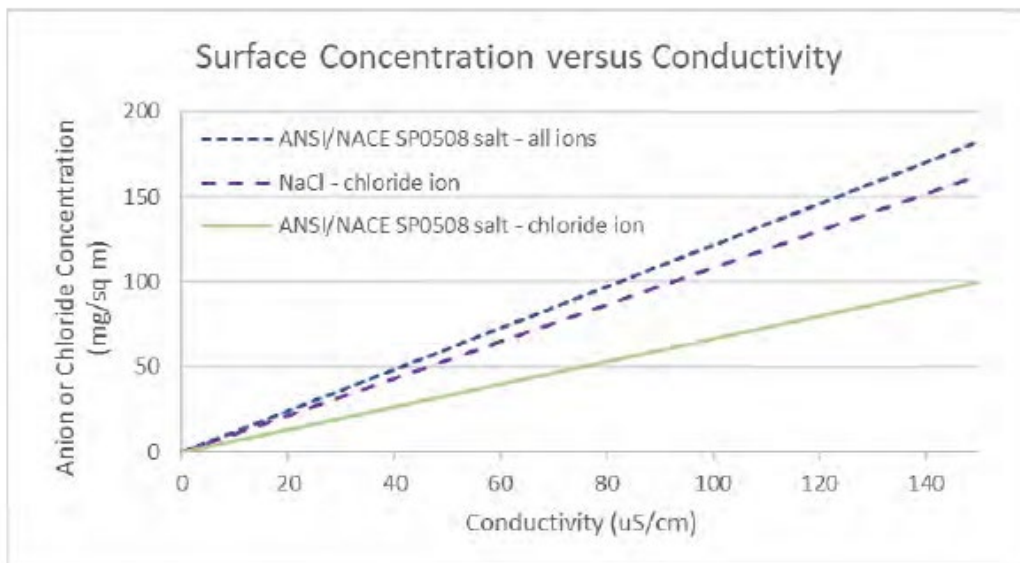


Figure 5: Reproduction of the relationship between surface ion concentration and conductivity for three known solutions.⁶

Positive results from conductivity testing should be treated with caution if the source of contamination is not confirmed. In one case study, positive conductivity results were found using the patch method.¹⁷ When similar conductivity results were measured after the additional washes, and investigation was initiated and found the contamination to be caused by dissolution of zinc salts from the original primer, not chloride ions on the surface. After ion specific testing for chlorides, the surface was found to be satisfactory and compliant for coating. Had the ion specific testing been performed initially, cost savings could have been made by eliminating additional and ineffective washes.

The primary differences between field-testing methods for salt contamination are the degree of accuracy, lower limit of detection, ease of use, and the degree of potential operator error. Testing errors can occur due to procedure complexity, operator measuring accuracy, and cross-contamination from reuse of components.¹¹

LOCATION AND TEST FREQUENCY

Salt contamination is typically non-uniformly distributed on steel surfaces in the field. The contamination can also be redistributed after in-service use. For this reason, the location and frequency of testing is an essential part of contamination testing. Two references are used to help specifiers and owners determine the frequency and location of testing:

- NACE SP0716-2016, "*Soluble Salt Testing Frequency and Locations on Previously Coated Surfaces*"¹⁸
- SSPC Guide 24, "*Soluble Salt Testing Frequency and Locations on New Steel Surfaces*."¹⁹

Both references provide guidance on the number and location of tests that should be performed based on the shape and complexity of the structure, locations where water can collect, condition of the surface, and transportation variables. A minimum of two initial tests are recommended for all structural components discussed. The number of tests increase with complexity and size of the component. Additional testing may recommend based on results of the initial testing.

ACCEPTABLE SOLUBLE SALT CONCENTRATION

Currently, there is no industry consensus on an acceptable soluble salt level for steel surfaces before applying coatings.⁴ Given the diversity of coating systems and operating environments, a standardized consensus is unlikely to emerge.

Several studies have offered failure rate results for different coating systems exposed to various environments with salt contamination on their surfaces.^{1,4,5,20} For example, one study showed that failure occurred in a system with three (3) coats of epoxy in 100% humidity at 104°F with as little as 1 µg/cm² chloride on the surface.⁵ While another study showed that a tank lining epoxy withstood immersion at 90°F under pressure of 50 psi with 40 µg/cm² chloride and 125 µg/cm² sulfate on the surface.⁵ Unfortunately, the results in numerous studies are highly variable, and therefore cannot be used to generate a standard threshold.

Laboratory testing shows that "*clean uncontaminated steel will not rust, even in 100% humidity, for thousands of hours.*"²¹ Without some form of contamination, there will not be rust. Though non-detectable (ND) results are desired, ND surfaces are not always feasible in the field. It is nearly impossible to achieve an absence of contamination in certain environments (marine, for example). In all cases, it is important to understand that a coating applied to a surface with ND soluble salt contaminants will perform better than a surface with 5 µg/cm² of contaminants, which will perform better than a surface with 10 µg/cm² of contaminants.⁵

Engineers and specifiers must also understand that a ND limit can be misleading because the testing equipment industry is always evolving. Detection limits of testing equipment are continuously falling (giving higher resolution measurements). A machine that once had a detection limit of 5 µg/cm², may now have a detection limit of less than 1 µg/cm² due to advances in the testing technology. Because the accuracy of the machines is generally greater than 1 µg/cm², a ND limit will require a cleaner surface to achieve with newer testing equipment than with older testing equipment. This important to understand as a specifier.

Government, coating manufacturers, and testing manufacturers have developed guidelines for an acceptable maximum level of salt contamination for under various exposures. Several industry guidelines are also currently being developed. The authors' compilation of maximum allowable soluble salts on surfaces used for immersion services (fuel and chemical) at ambient temperatures is provided in Table 1.

Table 1. A compilation of maximum allowable soluble salt limits for epoxy coatings used in immersion service (fuel and chemical) at ambient temperatures from various sources.

	Mnf 1 (µg/cm ²)	Mnf 2 (µg/cm ²)	Mnf 3 (µg/cm ²)	NAVSEA Spec 009-32 (µS/cm)	DoD UFGS 09 97 13.15 09 97 13.17
Chlorides: Cl ⁻	3	5	5	NA	ND
Sulfates: SO ₄ ⁻²	10	10	10	NA	ND
Nitrates: NO ₃ ⁻	5	15	10	NA	ND
Total	NA	NA	15	30	ND
Test Method	Patch/Cell Sleeve	Sleeve	Not Specified	Conductivity	Sleeve

Mnf: Manufacturer
 µg/cm²: micrograms per centimeter squared
 NA: Not available
 NAVSEA: Naval Sea Systems Command
 Spec: Specification
 µS/cm: microsiemens per centimeter
 DoD: Department of Defense
 UFGS: United Facilities Guide Specifications
 ND: Non-detectable

SPECIFICATION BEST PRACTICES

To address the variability in testing methods, acceptable contamination levels, salt exposure, and coating systems, every specification should include requirements for acceptable salt levels, testing methodology, testing frequently, and testing location requirements that are specific to the project. Testing methods must be communicated in inspection testing plans, prior to start of work. Testing locations and frequency may need to be tailored to address areas where salt may accumulate (areas of apparent contamination, rough or textured areas, areas where water accumulates, and welds). Without such requirements, projects risk specification misinterpretation, expensive change orders, and premature coating failures.

The project specific soluble salt threshold levels should correlate with manufacturer's recommendations for the coating in use and the anticipated environmental conditions. Coatings manufacturers understand the unique characteristics of their coatings systems and will be able to provide the best guidance on what thresholds should be used and which testing methods would be acceptable to determine that threshold. Manufacturers should also be able to provide specifiers guidance on the best methods to clean the surface if soluble salts are anticipated or detected.

CONCLUSIONS

Understanding the detrimental effects of soluble salts on coating performance, employing reliable testing methods, and implementing effective mitigation procedures are crucial steps in ensuring the long-term protection of steel surfaces. Ensuring that the specification addresses each of these aspects is vital for both corrosion mitigation and project success. The cost of coating failure far exceeds the expenses associated with identifying and removing soluble salts. By following the principles outlined in this paper, industry professionals can mitigate the risk of catastrophic failures, reduce maintenance costs, and enhance the durability of protective coatings on steel structures and equipment.

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