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The Effect of Four Commercially Available Steel Decontamination Processes on the Performance of External Coatings

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ABSTRACT

External coatings used for corrosion protection often have to perform under severely corrosive environments. One major concern regarding coating performance is the negative effect of soluble salts on the steel substrate at the time of coating application, particularly for marine maintenance coating applications. These salts impact the ability of the applied coating systems to protect the steel in several ways including osmotic coating blistering, promotion of under-film metallic corrosion and coating disbondment. This paper focuses on removal of soluble salts contamination by commercially available decontamination processes in relation to external coating systems. We directly compare the effectiveness of four cleaning methods with the performance of ten coating systems. The methodology of surface contamination and preparation of test panels is discussed. After cleaning, sample evaluation for chloride ion contamination levels was carried out using Field method (commercial chloride ion test kit for surfaces) and Ion Chromatography method. Additionally, Scanning Electron Microscopy / Energy Dispersive X-ray Spectroscopy (SEM/EDX) and elemental surface mapping analysis were carried out. Laboratory testing of coating systems included Adhesion, Porosity, Electrochemical Impedance Spectroscopy (EIS) analysis and cyclic UV/Salt Fog exposure.

The performance of the ten coatings on all the substrates was good, but there were differences in gloss retention and on the degree of checking of the different coatings. The only significant difference in performance of the coatings compared to the method used for cleaning the substrate was higher undercreep observed for most of the coatings applied to the ultra-high pressure water jetted system. This shows the importance of substrate preparation due to the sensitivity of the coatings to even low levels of salt. Two coatings did not show increased undercreep and these may be more applicable for offshore maintenance applications where dry abrasive blasting is sometimes not used. The chemical treatment cleaning method used prior to coating application did not show any significant positive or negative effect on the performance of the applied coatings. The fact that the only differences in performance for the coatings applied to the differently prepared substrates was seen for undercreep suggests that the difference may be exacerbated for immersion situations. A follow up study to this one will examine the performance of internal coatings using immersion tests, and it will be interesting to see if these show increased effect on coating performance.

Key words: Protective Coating, Industrial Maintenance Coatings, Laboratory Testing, Surface Contamination, Salts, Decontamination, Cyclic Test, UV Exposure, Salt Fog, Electrochemical Impedance Spectroscopy (EIS), Porosity.

INTRODUCTION

An adequate surface preparation is a major requirement for reliable performance of any coating system. Surface quality prior to application is one of the primary controlling factors responsible for a coating's service life. Even the best coating will fail if it is applied to a contaminated surface. Coating systems used for corrosion protection in external service frequently have to perform under severely corrosive environments. In such instances, it is important that these coatings must be able to withstand the effect of weather, ultraviolet light and industrial or marine atmospheres - and still maintain their protective qualities as well as an acceptable appearance. According to International Organization for Standardization (ISO) ⁽¹⁾ 8502, ¹ "The behavior of protective coating systems is affected mainly by the condition of the substrate immediately before the coating system is applied". This behavior is controlled by: (i) rust and mill scale; (ii) soluble salts; (iii) surface profile; and (iv) debris contamination. ²

There is a general trend towards advising very low salt concentration values by coating manufacturers and standard issuing organizations, e.g. International Maritime Organization (IMO) ⁽²⁾ prescribes maximum chloride levels at 3µg/cm. ^{2,3} These low values seem to be very conservative as they must represent different coating systems and different operational conditions. However, it has been highlighted that cost benefits of applying extensive surface cleaning procedures should be assessed on an individual basis so that the most economical surface preparation method is selected. ⁴

Industrial maintenance coatings are an important part of both onshore and offshore oil and gas production facilities worldwide. Chevron Energy Technology Company's[†] (end user) offshore facilities have been addressing challenges of maintenance coating issues for a number of years. The service life of previous maintenance coating systems has not met expectations and there have been instances where they do not survive through to the next maintenance cycle. One major concern regarding coating performance is the negative effect of soluble salts on the steel substrate at the time of coating application. These salts impact the ability of the applied coating systems to protect the steel in several ways including osmotic coating blistering and promotion of under-film metallic and coating disbondment.

The end user in conjunction with Chevron Gulf of Mexico Business Unit[†] has contracted Charter Coatings Service (2000) Ltd.[†] to help design a test protocol to determine the effectiveness of three commonly used surface pre-cleaning methods along with a post blast chemical cleaning method. This

⁽¹⁾ International Organization for Standardization (ISO), 1 ch. de la Voie-Creuse, CP 56 – CH – 1211, Geneva 20, Switzerland.

⁽²⁾ International Maritime Organization, 202 Lambeth Road, London, SE1 7JW.

[†] Trade name.

testing program is to help determine both advantages and disadvantages of the four procedures and their relative benefits on exterior coating performance. This is important when considering the extra cost, which may be incurred in surface preparation prior to the coating application.

The focus of this study is on the effectiveness of soluble salts contamination removal by different commercially available decontamination processes. Subsequently, the aim of this study is to directly compare the effectiveness of four cleaning methods on the performance of ten commercial external service coating systems. Methodologies for surface contamination prior to surface cleaning and surface decontamination procedures are presented. The degree of chloride ion decontamination was determined using Ion Chromatography method, Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM/EDX) and elemental surface mapping analysis. All ten coatings were applied by the same applicator under controlled and supervised conditions. Tests examined Adhesion, Porosity, Electrochemical Impedance Spectroscopy (EIS) analysis and cyclic Ultra Violet (UV)/Salt Fog exposure. This paper discusses the findings of this study.

It is anticipated that the results of this study will assist the end user in selecting cleaning methods and coatings in order to improve the service life of external coating systems. A third party inspection group has overseen all phases of this study.

SURFACE CONTAMINATION

Since chlorides are the major contaminant on most exterior steel structures located near shore and offshore, this study investigated chloride contamination and decontamination prior to coating application. Steel contamination was carried out in the laboratory using test panels cut from cold-rolled carbon steel sheets to the specified dimensions prior to contamination. All panels were degreased with acetone and sweep blasted on both sides and all edges. To simulate offshore chloride contaminations, the sweep blasted clean panels were exposed to a salt fog atmosphere in a salt spray cabinet conducted as per ASTM International (ASTM) ⁽³⁾ B117-11. ⁵ The exposure time required to achieve a contamination level of 300 Cl⁻ ($\mu\text{g}/\text{cm}^2$) was 48 hours and produced heavily corroded substrates which showed general and localized corrosion attack. After brush cleaning the substrate to remove the loose corrosion products, the level of chloride contamination was 64 Cl⁻ ($\mu\text{g}/\text{cm}^2$). Chloride analysis was conducted using a laboratory extraction process conducted according to Mayne's approach, ⁶ and subsequent Ion Chromatography measurement.

CLEANING METHODS

Four cleaning methods (denoted A, B, C and D) were used in this study on contaminated test panels with an initial contamination level of 64 Cl⁻ ($\mu\text{g}/\text{cm}^2$). The main goal of this study was to compare all four cleaning methods in terms of their efficiency and performance. Cleaning Method A is a common method in the industry, currently used by the end user; Cleaning Method B is a procedure that is an equivalent to Cleaning Method A except that a 25% commercial cleaning solution is used for the initial low pressure wash. Cleaning Method C is based on surface blasting followed by chemical treatment. Finally, Cleaning Method D is routinely used by the end user for offshore applications where abrasive blasting is not feasible nor the most appropriate; this method is based on ultra-high pressure (UHP) water jetting. Water used for all cleaning methods was fresh tap water with average chloride contamination of ≤ 10 mg/L (as reported by the water provider at the time of cleaning process). The details of the four cleaning methods are given below and shown in Figure 1.

Cleaning Method A: Pressure Wash with Fresh Water Followed by Blast Clean

Washing procedure of contaminated panels was performed using a low pressure wash (3000 psi) with fresh tap water. Each wash was for 2 minutes which is equivalent to 1 sq ft/min. Panels of 24x24x0.25

⁽³⁾ ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

in (4 sq ft) were washed four times and then blast cleaned to NACE No. 2/SSPC⁽⁴⁾-SP 10⁷ (Near White Metal Blast Cleaning) using fresh Garnet abrasives. Chloride tests were conducted for each coating system, after each wash and after the final blasting, using commercial chloride ion detection kit.

Cleaning Method B: Pressure Wash with 25% Commercial Cleaning Solution Diluted with Fresh Water Followed by Blast Clean

Washing procedure of contaminated panels was performed using a low pressure wash (3000 psi) with 25% commercial cleaning solution for 1 minute. This was followed by a rinse using fresh tap water at the same pressure for an additional 1 minute. Panels were washed three times and thereafter blast cleaned to NACE No. 2/SSPC-SP 10 (Near White Metal Blast Cleaning) using fresh Garnet abrasives. Chloride level tests were conducted after each wash and after the final blasting, using commercial chloride ion detection kit.

Cleaning Method C: Blast Clean Followed by Chemical Cleaning

Contaminated panels were first brush cleaned to remove the loose corrosion products, followed by blast cleaning to NACE No. 2/SSPC-SP 10 (Near White Metal Blast Cleaning) using fresh Garnet abrasives, then chemically treated by applying a commercial cleaning solution for 45 minutes and thereafter washed with a proprietary wash solution. Using the commercial chloride ion detection kit, two chloride density tests were conducted before and after applying the chemical treatment.

Cleaning Method D: Ultra-high Pressure Water Jetting (36000 psi) with Fresh Water

Panels used for this cleaning method, were initially blast cleaned to NACE No. 2/SSPC-SP 10 (Near White Metal Blast Cleaning) prior to the salt fog contaminations to establish an appropriate anchor pattern. Panels were then exposed to salt fog contamination for 48 hours to create a rust layer, which mimics the surface condition of the steel in service. Washing procedure of contaminated panels was performed using an ultra-high pressure water jetting (36000 psi) with fresh tap water for 2 minutes for only one time, resulting in SSPC-SP12/NACE No. 5 WJ-2/L cleanliness.⁸ Using the commercial chloride ion detection kit, only one chloride density test for each external coating type was conducted and that was immediately after the washing. This method simulates the end user's offshore maintenance procedure where surface blasting is not carried out. When maintenance coating application is required for flat deck or other areas where abrasive blasting is not feasible, nor the most appropriate cleaning method, the failed and the non-failed section of the decks are usually cleaned with ultra-high pressure water jetting to remove existing coatings and corrosion products prior to coating application.

Chloride Analysis

Two methods for measuring the chloride contamination level were used in this study, namely Laboratory Ion Chromatography (Dionex DX-120 Ion Chromatography system)[†] and a Field method (commercial chloride ion detection kit). The results obtained by both analytical methods are reported in Table 1. Based on results obtained, the following observations have been made:

- The level of chlorides was decreased significantly by one level of magnitude for all four cleaning methods.
- The Field Method detected Zero (0.00 µg/cm²) Cl⁻ level for the last step of each cleaning method prior to coating applications, indicating that the cleaning methods used removed chloride contamination below detectable levels for the Field Method.
- The Field Method did not detect chloride levels up to 2.7 µg/cm² which was detected by the Laboratory Extraction Method. No comparison was made for readings higher than 2.7 µg/cm².

⁽⁴⁾ The Society for Protective Coatings, 40 24th Street, 6th Floor, Pittsburgh, PA 15222.

[†]Trade name.



Figure 1: Diagram Showing Individual Steps Involved in the Contamination and Four Cleaning Methods

Table 1
Results of Chloride Ion Concentration

Cleaning Method	Laboratory Method Ion Chromatography Cl ⁻ (µg/cm ²)	Field Method Cl ⁻ (µg/cm ²)
Cleaning Method A: Pressure Wash with Fresh Water followed by Blast Clean	0.8 - 2.33	0
Cleaning Method B: Pressure Wash with Fresh 25% Aqueous Cleaning Solution followed by rinse using fresh tap water at the same pressure, then Blast Clean	1.4-1.9	0
Cleaning Method C: Blast Clean followed by Chemical Cleaning	1.4 - 2.13	0
Cleaning Method D: Ultra-high Pressure Water Jetting (36000 psi) with Fresh Water	1.2 - 2.7	0

SEM/EDX Analysis

Figure 2 shows representative results of the SEM/EDX analysis carried out on contaminated panels before and after cleaning using the four individual cleaning methods. The aim of the SEM/EDX analysis was to identify microscopically (through SEM images) and analytically (through EDX analysis) any presence of contaminants in the form of chlorides on sample panels after each of the four cleaning procedures.

SEM images of a panel sample analyzed before any cleaning procedure revealed highly contaminated steel surface with many deposit patches. An EDX analysis of one of these patches confirmed the presence of chloride contamination (1.88% wt.) on the steel surface. SEM characterization of panels after the four individual cleaning methods showed highly deformed, rough surface morphologies resulting from blasting. However, no significant contamination deposits were observed. Further EDX analysis of cleaned panels revealed traces of chloride. There were some precipitates observed on the surface of the panel after Cleaning Method D (marked on Figure 2), which were identified as iron oxide. This was observed visually as a slight tarnish of the steel substrate after the ultra-high pressure water jetting cleaning which could not be avoided and which would most likely be observed in the field.

An additional elemental mapping analysis of panels after the four individual cleaning methods showed no localized concentration of chloride.

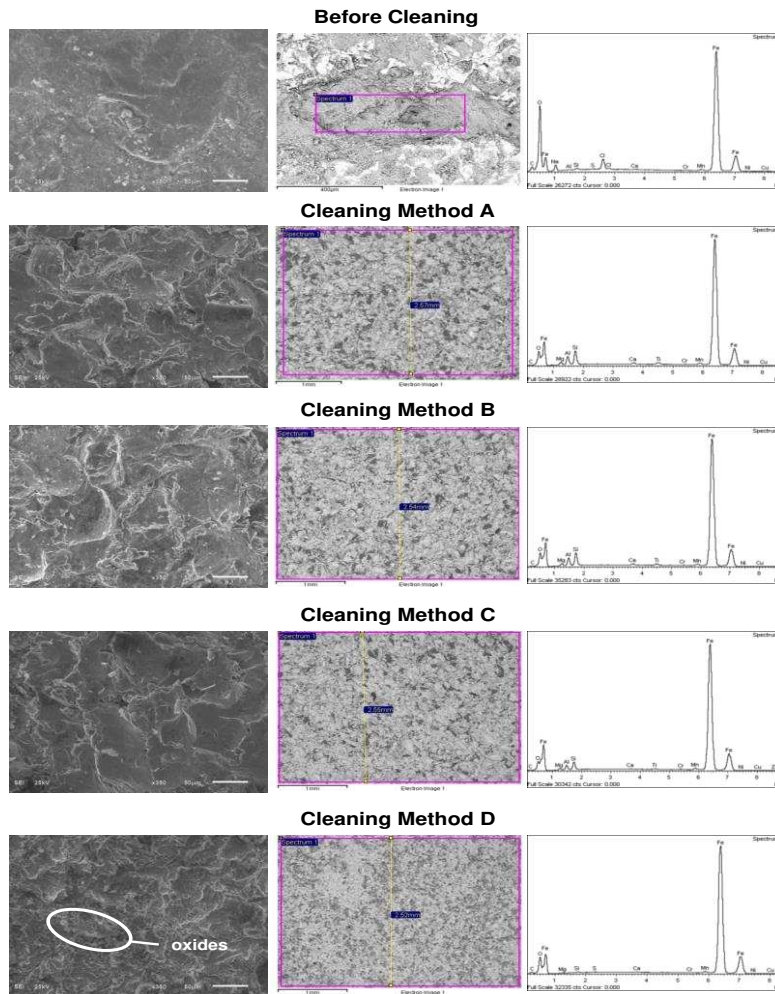


Figure 2: SEM Images with corresponding EDX Spectra for Panels before Cleaning and after Cleaning Methods A, B, C and D

COATING SYSTEMS

A total of ten commercially available maintenance coating systems suitable for exterior service were used in this study. The coating systems were supplied by five different coating manufacturers. The coating systems varied in their chemistry and number of coating layers. Protective coatings can be divided into several classes according to the basic chemical reactions involved in the film forming process. Table 2 shows the coating types used in this study and the generic resin materials in each coating as reported in the manufacturer's product data sheet along with the volume solids. Note that for simplicity, no differentiation of products is made according to hardener, though this has a very real effect on the coating characteristics. The coatings were grouped according to the number of layers in the coating system, the chemistry of the first and mid-coat(s) and top coat. A summary analysis of the different coating systems is given below and shown graphically in Figure 3.

Coating systems 1-8 have been previously approved by the end user for field application, while systems 9 (zinc silicate primer and siloxane epoxy) and 10 (epoxy primer and elastomeric pure polyurea) are new coatings. Data analysis of the three pie charts of Figure 3 indicates the following:

- **Number of coating layers:** 60% of the coating systems were two-coat, 20% of the coating systems were three-coat and 20% of the coating systems were four-coat (second and third layers of the four-coat system were identical).
- **Chemistry of the first and mid-coat(s):** The first layer of the three-coat systems was aluminum-pigmented high-solids mastic applied as a single coat or three coats. The first and mid-coat(s) of the two-coat systems were polymeric epoxy amine-aluminum-pigmented high-solids mastic. The one-coat system had organic zinc-rich epoxy primer and aluminum-pigmented high-solids mastic mid-coat. Two-coat systems had epoxy/surface tolerance epoxy first coat. Two-coat systems

had polyamide epoxy (containing Zn phosphate) as a first coat, 10% had zinc silicate primer and the one-coat system had aluminum pure epoxy first coat.

- **Chemistry of the top coat:** The top coat of two-coat systems was based cycloaliphatic amine epoxy. The top coat of seven coating systems was based on the modified siloxane hybrid, or modified epoxy, high solids glass flake epoxy, or acrylic polysiloxane, or siloxane epoxy. The top coat of one coating system was elastomeric pure polyurea.

COATING SYSTEMS APPLICATION

In order to achieve conformity of application conditions, all the coatings were applied at the same location by the same applicator. The applications were observed by a coating inspector and witnessed by a representative of each coating supplier. This allowed for continuity of analysis of the application characteristics, while ensuring that the supplier’s standard application procedures were properly followed.

Two coated plates were prepared for each coating system. Tests were conducted for wet and dry film thickness. All the coatings were successfully applied under similar application conditions of temperature and humidity using standard equipment that is recommended by the manufacturer and would be utilized in a field application. The coated plates were then delivered to our laboratory facilities where they were visually inspected and photographed. Test panels of the appropriate sizes were prepared using water-jet cutting.

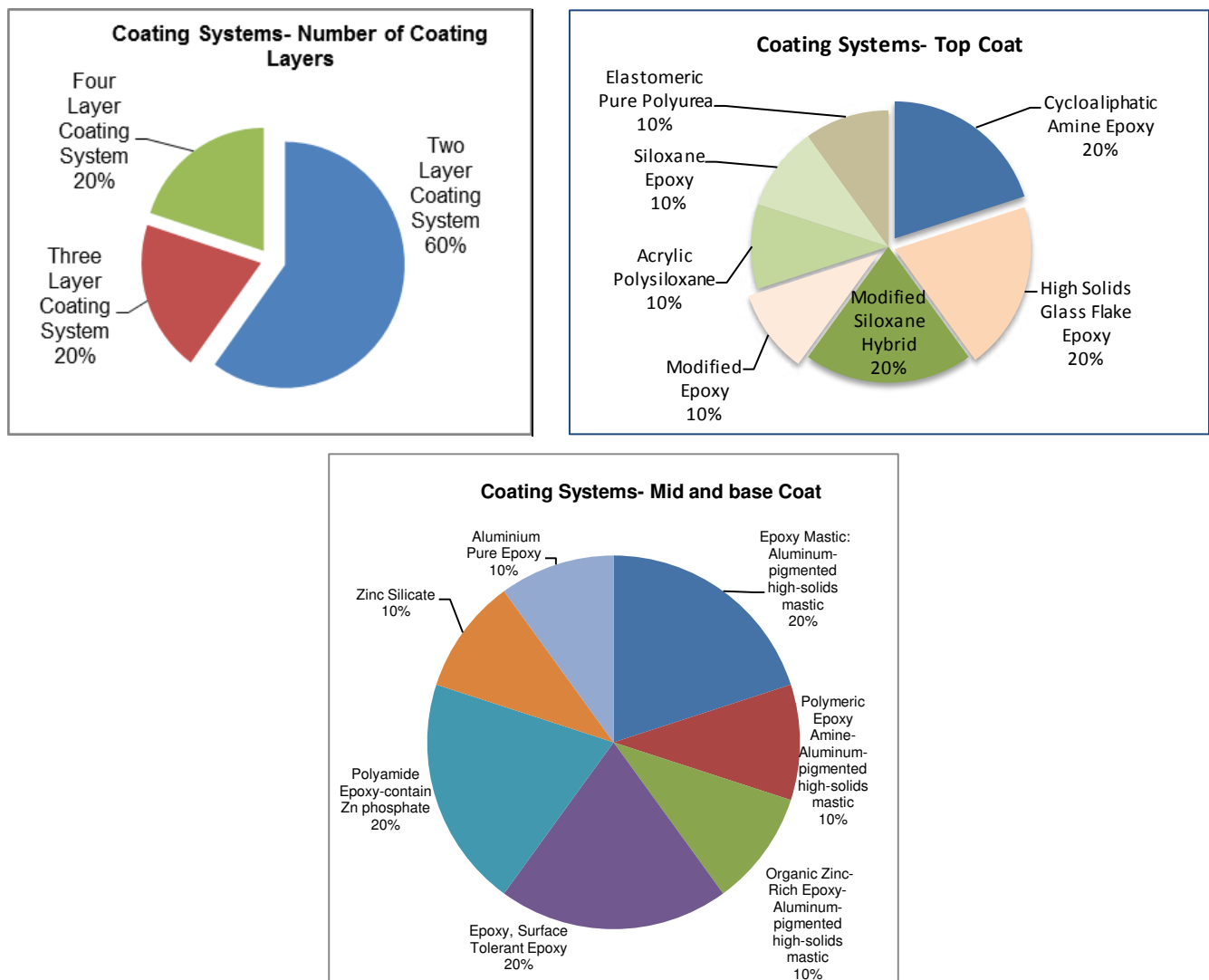


Figure 3: Breakdown of a number of Layers and their Chemistry in the Coating Systems

Table 2
Specification of External Coatings Used in this Study

Coating System	Number of Coating Layers & Application	Coating Type	Recommended Total Dry Film Thickness (DFT) mils / micron	% Solids (Volume)
1	Two-coat system applied by airless equipment after <u>cleaning methods A, B and C</u>	Epoxy Mastic (aluminum-pigmented high-solids mastic)	12-15 / 305-381	90
		Cycloaliphatic Amine Epoxy		75
2	Three-coat variant of coating 1 with extra barrier layer applied by airless equipment after <u>cleaning method D only</u>	Polymeric Epoxy Amine	13-17 / 330-431	90
		Epoxy Mastic		90
		Cycloaliphatic Amine Epoxy		75
3	Four-coat system applied by airless equipment after <u>cleaning methods A, B and C</u>	Solvent Based Organic Zinc-Rich Epoxy	16-24 / 406-610	61
		Epoxy Mastic		90
		Epoxy Mastic		90
		Modified Siloxane Hybrid		75
4	Four-coat variant of coating 3 with different barrier layer applied by airless equipment after <u>cleaning method D only</u>	Epoxy Mastic	18-26 / 457-660	90
		Epoxy Mastic		90
		Epoxy Mastic		90
		Modified Siloxane Hybrid		75
5	Two-coat system applied by airless equipment after <u>cleaning methods A, B, C and D</u>	Surface Tolerant Epoxy	16-20 / 406-508	82
		Modified Epoxy		85
6	Three-coat system applied by airless equipment after <u>cleaning methods A, B, C and D</u>	Aluminum Pure Epoxy	12-17 / 305-432	63
		Aluminum Pure Epoxy		63
		Acrylic Polysiloxane		72
7	Two-coat system applied by airless equipment after <u>cleaning methods A, B, C and D</u>	Polyamide Epoxy (containing Zn phosphate)	12-15 / 305-368	68
		High Solids Glass Flake Epoxy		87
8	Two-coat system applied by airless equipment after <u>cleaning methods A, B, C and D</u>	Polyamide Epoxy (containing Zn phosphate)	25-27 / 635-686	68
		High Solids Glass Flake Epoxy		87
9	Two-coat system applied by conventional and airless equipment after <u>cleaning methods A, B, C and D</u>	Zinc Silicate	6-9 / 152-227	40
		Siloxane Epoxy		90
10	Two-coat system applied by airless and plural component equipment after <u>cleaning methods A, B, C and D</u>	Epoxy Primer/Sealer	22 / 559	48
		Elastomeric Pure Polyurea		100

LABORATORY TESTING METHODOLOGY

In external service, coatings must be able to withstand the effects of weather, UV light and industrial or marine atmospheres and still maintain their protective qualities and an acceptable level of protection. The laboratory testing program was designed to simulate field conditions, specifically cyclic testing exposure to fluorescent UV light and salt fog environments. The adhesion assessment by pull-off and X-scribe methods, cross-section porosity and EIS were used to determine the effect of different surface cleaning methods on the coating adhesion and protectiveness of the coating systems.

Adhesion: Adhesion of coating systems was assessed using Pull-off and X-scribe methods. The Pull-off test was conducted in compliance with ASTM D4541-02 using a self-aligning pull-off tester.⁹ X-scribe adhesion test examines adhesion of a coating to the substrate by applying a force using the levering action of a knife tip. The knife adhesion test was conducted according to ASTM D6677-07.¹⁰

Cross-section Porosity: The integrity of the coating is an important part of its ability to adhere to and protect a substrate. If the coating film is porous, the strength and integrity of the coating can be significantly reduced and could cause premature failure. The porosity present in the coating system was rated in accordance with the rating scale given in CSA⁽⁵⁾ Z245.20, section 12.10.¹¹

EIS Study: EIS is a laboratory method for evaluating the protectiveness of organic coatings. The EIS test was conducted according to ISO 16773-2: 2007.¹² EIS was conducted on untested samples and the measurements were made using a designed electrolytic cell method, employing a beaker filled with 3% NaCl solution at 73°F/23°C for 48 hours. Impedance measurements were carried out in the frequency range of 100 kHz to 0.01 Hz.

Cyclic Testing: The outdoor corrosion of coated metals is influenced by many factors, including: corrosive atmospheres, rain, condensation, UV light, wet/dry cycling and temperature cycling. These factors frequently have a synergistic effect on one another. Hence, cyclic testing was intended to provide a more realistic simulation of the interaction of these factors than is found in traditional tests with continuous exposure to a static set of corrosive conditions. The test specimens were exposed to alternating periods of one week in a fluorescent UV/condensation chamber (4h UV light at 60°C and 4h condensation at 50°C) and one week in a cyclic salt fog/dry chamber according to ASTM D5894¹³ Standard for 1008 hours (6 weeks) in total (1h fog at ambient temperature and 1h dry-off at 35°C). The fluorescent UV/condensation exposure was carried out in compliance with Cycle 2 of ASTM D4587¹⁴ Standard, while the cyclic salt fog/dry exposure was carried out according to ASTM G85¹⁵. Post-test evaluations included assessment of blistering as per ASTM D714¹⁶, checking rating as per ASTM D 660¹⁷, color change, gloss, chalking, rusting and undercreep rating as per ASTM D1654¹⁸.

RESULTS AND DISCUSSION

The Pull-off Adhesion: The pull-off adhesion test results indicate that the effect of cleaning methods on the coating systems' adhesion was identical for each coating system. No adhesive failure at the coating system/substrate interface has been reported for all coating systems and the cleaning methods indicate good coating application process leading to high interface strength. The pull-off strength of the coating system was found to be a factor of the coating type and not the cleaning method.

X-Scribe Adhesion Test: All coating systems showed no sign of any adhesive or brittle breakaway from the substrate indicating excellent coating to substrate adhesion. For the same type of coating, the variation in the substrate cleaning method did not affect the coating adhesion.

Cross-section Porosity: Both cross-section porosity and interface porosity were very low for all tested coating systems. No porosity results are reported for coating system 10, as the coating was too flexible to allow for clean coating removal from the substrate.

EIS Study: Data was collected from all of the untested coating systems. The objective of the EIS study was to compare the barrier properties (water permeability) of the coating systems and to determine if variation in substrate cleaning methods used prior to coating application has an effect on the coatings' barrier properties. In general, EIS data on the coatings indicated good/excellent barrier properties (Log Z above 9-10) for all coating systems and cleaning methods.

The Cyclic Testing: Results are summarized in Table 3 and shown in Figures 4-6. The results are presented and discussed below for each individual coating system:

1. Epoxy Mastic (Aluminum-pigmented high-solids mastic) / Cycloaliphatic Amine Epoxy – Two-coat System: The cyclic testing results indicate that the three cleaning methods had similar effect on the coating performance; the coating showed no change in DFT, no blistering, no rusting and no

⁽⁵⁾ Canadian Standards Association (CSA), 178 Rexdale Blvd., Toronto, Ontario M9W 1R9.

cracking. Loss of gloss, slight change in color and very slight tendency to undercreep (≤ 0.5 mm) were noted for all tested samples. Epoxy mastics, over the past decade have proven to be a tremendous success in the coatings industry – especially for simplifying the maintenance procedure. The epoxy resins are modified with a refined hydrocarbon resin to enhance the moisture resistance, flexibility and the wetting properties of epoxy coatings. Aluminum pigments have been widely used in engineering paints and protective coatings for many years. The aluminum pigments improve the barrier properties of protective paints.^{19, 20} From a corrosion protection point of view, their ability to impede the transport of water and oxygen is the most important property. The pigments are impermeable to water and oxygen as the diffusion path is directed around the particles, which decreases the permeability through the paint.⁶ In addition the pigments also act as barriers to radiation, as they reflect both, infrared, UV and visible light.⁶ This increases the stability of the paint and may also reduce heat radiation from the substrate.

2. Polymeric Epoxy Amine / Epoxy Mastic / Cycloaliphatic Amine Epoxy – Three-coat System: This system was applied only on steel substrate cleaned with the ultra-high pressure water jetting - 36000 psi (Method D) as per the end user's offshore specifications. The coating showed no change in DFT, no blistering, rusting, cracking and only slight change in color. The coating showed loss of gloss and some tendency to undercreep (3.1 mm). The epoxy coating has the combined properties of high build, good cohesive strength and strong adhesion. These properties give the system impact and general damage resistance while maintaining flexibility of the system. Cycloaliphatic amines have been used as a curing agent for years. These low viscous and highly reactive hardeners are very favorable with regard to application properties and performance under ambient cure conditions. After cure, they result in systems that combine high chemical and temperature resistance with superior mechanical performance. Undercutting is a measure of adhesion. The term applies to the corrosion at a break in the coating, growing back underneath the surface of the coating away from the break. To have resistance to undercutting, a coating must be strongly adhesive and must maintain its adhesion even at a raw edge between the coating and the steel. Organic coatings show the greatest tendency to undercutting by corrosion because of their frequent variable adhesion to a surface. Also, organic materials produce a definite interface between the metal surface and the organic coating. The adhesion of the coating to the metal is one of a simple bond between the two materials. Part of this bond is physical, but the remainder has to do with compatibility and the ability of the coating to thoroughly wet the substrate surface.
3. Solvent Based Organic Zinc-Rich Epoxy / 2 Layers of Epoxy Mastic / Modified Siloxane Hybrid – Four-coat System: The coating showed excellent performance for the three cleaning methods as indicated by no change in DFT, no blistering, no rusting, no cracking, no change in color, no loss of gloss, and a resistance to undercreep (0.0 mm). The zinc-rich epoxy coating primer layer arrests corrosion of the steel through a cathodic protective system related to the activity of the zinc versus the steel substrate. The relative inertness of the zinc to atmospheric conditions combined with it being higher in the "reactivity table" than iron makes it excellent for this application. The epoxy component of this primer affords good adhesion properties to the steel. This coating is applied as a thin layer to help prevent corrosion at damage sites. The epoxy coating has the combined properties of high build, good cohesive strength and strong adhesion. These properties give the system impact and general damage resistance while maintaining flexibility of the system. The intermediate two layers of epoxy mastic have the combined properties of high build, good cohesive strength and strong adhesion. The top coat is based on polysiloxane chemistry. These products started to appear in the 1990s and are now an alternative to polyurethane products with arguably better resistance to deterioration under atmospheric conditions because of the stronger Si-O bond (about 108 Kcal/mole) versus the C-C bond (83 Kcal/mole) as well as the reduced tendency of the polysiloxanes to oxidation and consequent deterioration of color or coating integrity.
4. Three Layers of Epoxy Mastic / Modified Siloxane Hybrid – Four-coat System: The coating system was applied only on steel substrate cleaned with the ultra-high pressure water jetting - 36000 psi (Method D) as per the end user's offshore specifications. The cyclic testing results showed no change in DFT, no blistering, no rusting, no cracking, no change in color, no loss of gloss but a greater tendency to undercreep (1.7 mm). It is interesting to report that both coating system 1 and

this coating have similar first coat of epoxy mastic (aluminum-pigmented high-solids mastic) and, as reported above, when this was applied on steel substrates after cleaning methods A, B and C, the coating system showed very good resistance to undercreep (≤ 0.5 mm). This gives clear indication that the observed undercreep for this coating system is most likely due to the same phenomenon as resulted in the tarnish noted on the steel substrate after the ultra-high pressure water jetting prior to coating application. This indicates that effective surface cleaning and blasting is required for coating with epoxy mastic as a first coat. In addition, the retained glossy appearance of this coating system compared with coating system 1 above, which showed severe loss of gloss is due to use of the modified siloxane hybrid as a top coat.

5. Surface Tolerant Epoxy / Modified Epoxy – Two-coat System: All cleaning methods had similar effect on the coating performance with no change in DFT, no blistering, no rusting, no cracking, medium change in color and loss of gloss. Checking was observed on the test panels. Although undercreep was observed in all samples, the steel substrate cleaned by ultra-high pressure water jetting (Method D) showed lower resistance to undercreep (4.6 mm) than the other cleaning methods. This indicates that effective surface cleaning and blasting is required to improve coating performance with regard to undercreep.
6. Two Layers of Aluminum Pure Epoxy / Acrylic Polysiloxane – Three-coat System: All cleaning methods had similar effect on the coating performance with no change in DFT, no blistering, no rusting, no cracking, no change in color and no loss of gloss. There was a difference though in the undercreep test results. Although undercreep was observed in all samples, the steel substrate cleaned by ultra-high pressure water jetting (Method D) showed lower resistance to undercreep (3.3 mm) than the other cleaning methods.
7. Polyamide Epoxy (containing Zn phosphate) / High Solids Glass Flake Epoxy – Two-coat System: All cleaning methods had similar effect on the coating performance with no change in DFT, no blistering, no rusting, no cracking, slight change in color, loss of gloss and checking. The tendency to undercreep was observed (2.4 mm, 1.5 mm, 1.7 mm and 2.6 mm for cleaning Methods A, B, C and D, respectively). The steel substrate cleaned by ultra-high pressure water jetting (Method D) showed slightly lower resistance to undercreep (2.6 mm) than the other cleaning methods. Epoxy coatings, generally cross-linked with amines or polyamides, are widely used as heavy duty moisture- and chemical-resistant, well adhered, corrosion-resistant coatings and lining in various environments. ²¹ Zinc phosphate is a well-known pigment that shows anticorrosion properties by forming a protective layer when in contact with iron ions of mild steel. Glass flake pigments are also used in protective coatings to improve the barrier properties of the coating.
8. Polyamide Epoxy (containing Zn phosphate) / High Solids Glass Flake Epoxy – Two-coat System: The coating is the same as coating 7 but was applied at a higher DFT (24-25 mils) compared with 14 mils of coating system 7. The coating showed similar performance as reported above for coating system 7. All cleaning methods showed similar effect on the coating performance with no change in DFT, no blistering, no rusting, no cracking, slight change in color, loss of gloss, checking and a tendency to undercreep (1.5 mm, 2.3 mm, 2.6 and 4.8 mm for cleaning Methods A, B, C and D, respectively). Steel substrate cleaned by ultra-high pressure water jetting (Method D) showed lower resistance to undercreep (4.8 mm) compared to the other cleaning methods. This indicates effective surface cleaning and blasting are required to improve coating performance with regard to undercreep.
9. Zinc Silicate / Siloxane Epoxy – Two-coat System: The cyclic testing results indicate the four cleaning methods had similar effect on the coating performance; the coating showed very good performance as indicated by no change in DFT, no blistering, no rusting, no cracking, no change in color, no loss of gloss and very slight tendency to undercreep (≤ 0.8 mm). This is the only coating which has surface tolerant properties for tarnished substrate which is applicable for offshore coating application. The inorganic zinc primer has a chemical bond in addition to a physical bond between the coating and the steel. This combined bond is much more durable and is not subjected to undercutting.

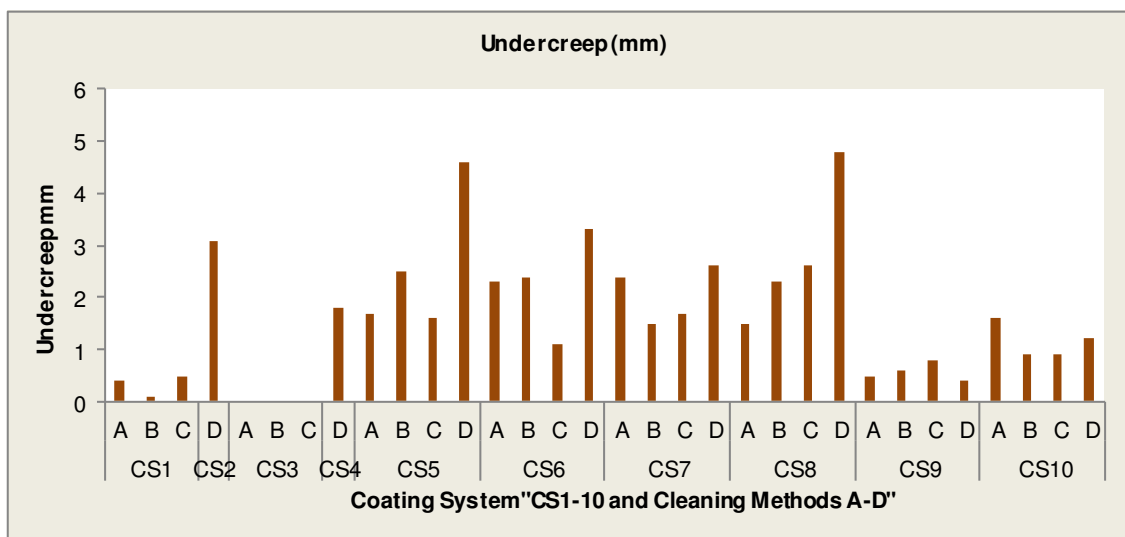
10. Epoxy Primer-sealer / Elastomeric Pure Polyurea – Two-coat System: All cleaning methods had similar effect on the coating performance with no change in DFT, no blistering, no rusting, no cracking and no change in color. Checking and some loss of gloss were noted for all test samples. The coating showed some tendency to undercreep (0.9 - 1.6 mm) and checking.

CONCLUSIONS

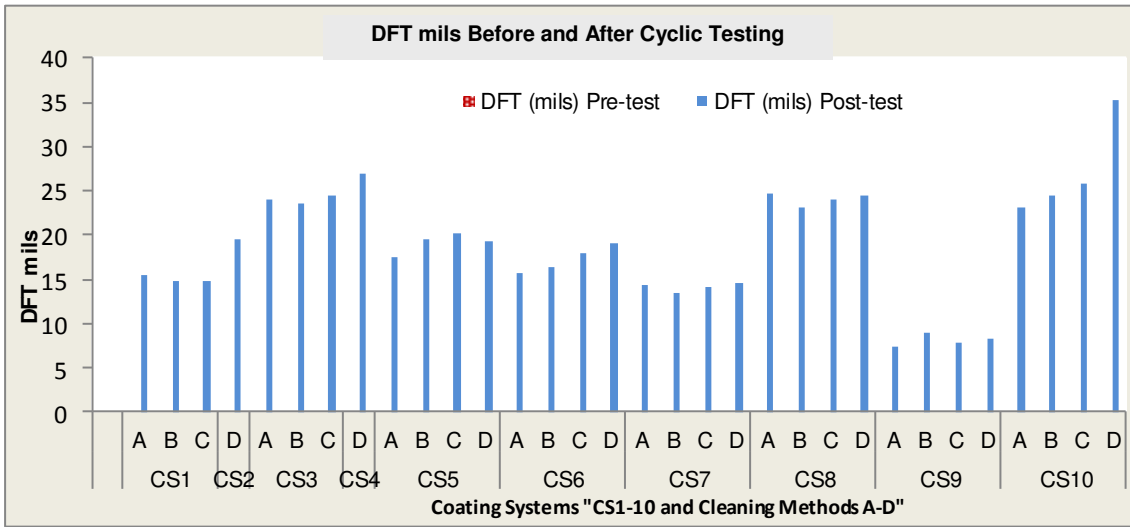
Examination of the samples prepared using the four cleaning methods showed low salt levels for all methods. However, the substrate preparation method, using ultra-high pressure wash with no blast conducted before or after the water jetting, resulted in a slightly tarnished substrate due to oxides on the steel. A field method used for determining chloride levels was shown to be less sensitive than a laboratory extraction method which is a concern given that chloride levels are critical for coating performance even at low levels.

The performance of all ten coatings on all the substrates in service environment simulated testing was good but there were differences in gloss change and degree of checking of the different coatings which are surface phenomena and formulation-related phenomena. The only significant difference in protective performance of the coatings versus the method used for cleaning the substrate was that there was higher undercreep observed for most of the coatings applied to the ultra-high pressure washed system which shows the coating systems' sensitivity to substrate preparation and to "tarnish." The coating system which is based on zinc silicate primers and a top coat of siloxane epoxy did not show increased undercreep, and this coating may be more applicable for offshore application where blasting is sometimes not used prior to coating application.

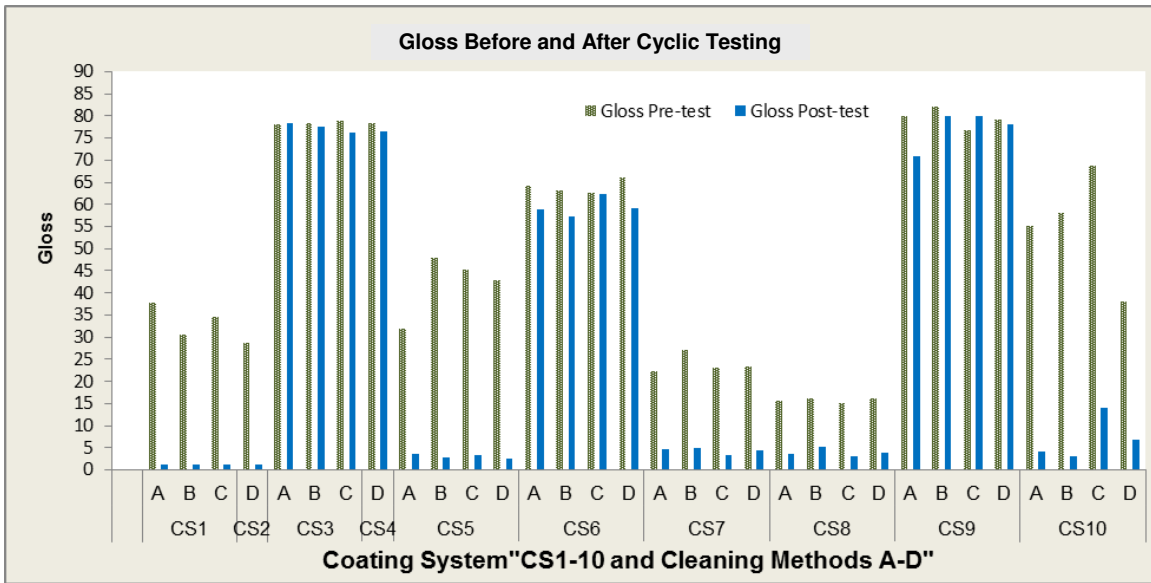
The chemical treatment included in this study (Cleaning Method C), did not show any significant positive or negative effect on the performance of the applied coatings. The fact that the only differences in performance for the coatings applied to the differently prepared substrates was seen for undercreep suggests that the difference may be exacerbated for immersion situations. A follow up study to this one will examine the performance of internal coatings using immersion tests and it will be interesting to see if these show increased effect on coating performance.



**Figure 4: Cyclic Testing Results
Undercreep for Ten Coating Systems and Four Cleaning Methods (A-D)**



**Figure 5: Cyclic Testing Results
DFT for Ten Coating Systems and Four Cleaning Methods (A-D)**



**Figure 6: Cyclic Testing Results
Gloss for Ten Coating Systems and Four Cleaning Methods (A-D)**

Table 3: Laboratory Tests Results Summary

Coating System	Steel Substrate Cleaning Methods	Cyclic Testing Results									
		DFT mils		Gloss		Blisters	Color Change	Cracks	Checking*	Undercreep**	
		Before	After	Before	After					(mm)	rating
1 Epoxy Mastic (aluminum-pigmented high-solids mastic) Cycloaliphatic Amine Epoxy	Cleaning Method A	15.4	15.4	37.8	1.1	None	Slight	None	None	0.4	9
	Cleaning Method B	15.4	14.7	30.5	1.2	None	Slight	None	None	0.1	9
	Cleaning Method C	16.0	14.9	34.4	1.3	None	Slight	None	None	0.5	9
2 Polymeric Epoxy Amine/Epoxy Mastic/Cycloaliphatic Amine Epoxy	Cleaning Method D	16.3	19.6	28.6	1.2	None	Slight	None	None	3.1	5
3 Solvent Based Organic Zinc-Rich Epoxy/2 layers of Epoxy Mastic/ Modified Siloxane Hybrid	Cleaning Method A	24.2	23.9	78.0	78.3	None	None	None	None	0	10
	Cleaning Method B	24.2	23.5	78.2	77.6	None	None	None	None	0	10
	Cleaning Method C	26.9	24.5	78.9	76.3	None	None	None	None	0	10
4 3 Layers of Epoxy Mastic/Modified Siloxane Hybrid	Cleaning Method D	26.5	27.0	78.2	76.5	None	None	None	None	1.8	7
5 Surface Tolerant Epoxy/Modified Epoxy	Cleaning Method A	17.8	17.6	31.7	3.7	None	Medium	None	Yes	1.7	7
	Cleaning Method B	18.8	19.5	47.8	2.7	None	Medium	None	Yes	2.5	6
	Cleaning Method C	19.8	20.2	45.1	3.4	None	Medium	None	Yes	1.6	7
	Cleaning Method D	19.7	19.2	42.8	2.5	None	Medium	None	Yes	4.6	5
6 2 Layers of Aluminum Pure Epoxy/ Acrylic Polysiloxane	Cleaning Method A	15.3	15.7	64.0	58.9	None	None	None	None	2.3	6
	Cleaning Method B	16.8	16.4	63.0	57.3	None	None	None	None	2.4	6
	Cleaning Method C	17.5	17.9	62.5	62.4	None	None	None	None	1.1	7
	Cleaning Method D	19.2	19.0	66.1	59.0	None	None	None	None	3.3	5
7 Polyamide Epoxy (containing Zn phosphate)/ High Solids Glass Flake Epoxy	Cleaning Method A	14.2	14.4	22.3	4.6	None	Slight	None	Yes	2.4	6
	Cleaning Method B	13.7	13.4	26.9	4.9	None	Slight	None	Yes	1.5	7
	Cleaning Method C	14.2	14.1	22.9	3.3	None	Slight	None	Yes	1.7	7
	Cleaning Method D	24.6	24.5	23.2	4.4	None	Slight	None	Yes	2.6	6
8 (Same as 7 but higher DFT) Polyamide Epoxy (containing Zn phosphate)/High Solids Glass Flake Epoxy	Cleaning Method A	25.0	24.6	15.6	3.7	None	Slight	None	Yes	1.5	7
	Cleaning Method B	24.0	23.2	16.0	5.2	None	Slight	None	Yes	2.3	6
	Cleaning Method C	24.3	24.0	14.9	3.1	None	Slight	None	Yes	2.6	6
	Cleaning Method D	24.6	24.5	16.1	4.0	None	Slight	None	Yes	4.8	5
9 Zinc Silicate/Siloxane Epoxy	Cleaning Method A	7.4	7.3	79.9	71	None	None	None	None	0.5	9
	Cleaning Method B	9.3	9.0	82.1	80	None	None	None	None	0.6	8
	Cleaning Method C	8.2	7.9	76.7	80	None	None	None	None	0.8	8
	Cleaning Method D	9.3	8.2	79.1	78	None	None	None	None	0.4	9
10 Epoxy Primer/Sealer/Elastomeric Pure Polyurea	Cleaning Method A	25.0	23.1	55.1	4.1	None	None	None	Yes	1.6	7
	Cleaning Method B	27.0	24.5	57.9	3.1	None	None	None	Yes	0.9	8
	Cleaning Method C	27.0	25.9	68.6	14.0	None	None	None	Yes	0.9	8
	Cleaning Method D	36.0	35.2	37.9	6.8	None	None	None	Yes	1.2	7

- Cleaning Method A: Pressure Wash - Blast Clean
- Cleaning Method B: Pressure Wash 25% Aqueous Cleaning Solution - Blast Clean
- Cleaning Method C: Blast Clean - Chemical Cleaning
- Cleaning Method D: Ultra-high Pressure Water Jetting (36000 psi)

* Checking was observed. Rating as per ASTM D 660
 ** Mean Creepage from Scribe. Rating as per ASTM D1654

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