

The Effect of Substrate Contaminates on the Life of Epoxy Coatings Submerged in Sea Water

**U.S. DEPARTMENT OF TRANSPORTATION
Maritime Administration and the U.S. Navy**

in cooperation with

**National Steel and Shipbuilding Company
San Diego, California**

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The Effects Of Substrate Contaminants On The Life Of Epoxy Coatings Submerged In Sea Water

**NATIONAL SHIPBUILDING
RESEARCH PROGRAM**

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ABSTRACT

This research was conducted to study the effects that contaminants, commonly occurring in the marine environment, have on epoxy coating systems. Three standard ballast tank coatings plus a clear epoxy coating were tested. Three chemicals and various size blasting grit particles were used as contaminants. The two chloride contaminants (sea salt and sodium chloride) were found to be much more detrimental to epoxy coatings, than was ferrous sulphate or the grit particles.

It could be seen through the clear epoxy coating, that chloride surface contamination levels even as low as $0.25 \mu\text{g Cl /CM}$ ($.014 \text{ oz sodium chloride/1000 square feet}$), could induce slight steel substrate corrosion. When chloride levels were $1 \mu\text{g Cl}$ ($.054 \text{ oz NaCl/1000Ft.}^2$) or higher, considerable under film corrosion was observable under the clear epoxy. Visible surface roughening (micro-blistering) of both the clear and coal tar epoxy coatings occurred when ever contamination was over $5 \mu\text{g Cl /CM}$ ($.28 \text{ oz NaCl/1000Ft.}$). Osmotic type blistering (ASTM size 8 and larger) occurred after the chloride ion levels had exceeded a level of $10 \mu\text{g Cl /CM}$ ($0.56 \text{ oz NaCl/1000Ft.}$) in the case of coal tar and clear epoxies. Slightly higher chloride levels were needed to cause osmotic blistering in the other coatings. The original micro-blisters coalesced to form the larger osmotic blisters. Where this coalescing occurred, under film corrosion stopped.

The larger size grit particles were found to cause some coating failures, if they were left on surfaces and then painted over. Small amounts of very fine grit dust did not cause obvious coating problems unless they were from grit that was highly contaminated with chloride.

The research found, that there is a critical need to carefully check blasted surfaces for chloride contamination, before applying coatings. Therefore several guides were included in the report (see appendix B), to help shipyards and the coating industry, to better understand, detect and correct problems arising from excess surface contamination.

FOREWORD

This research project was preformed for the Maritime Administration, under its National Shipbuilding Research Program. This program is a cooperative cost shared effort, between the US Navy, the Maritime Administration and the National Steel and Shipbuilding Company. The program has been administered by National Steel and Shipbuilding Company, with Mr. James R. Ruecker and later Mr. Lynwood P. Haumschilt acting as Program Manager. Note: "Mr. John Peart provided technical assistance to Mr. Ruecker and Mr. Haumschilt on the project.

ACKNOWLEDGEMENTS: Dave Bloodgood-Devoe Paints
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All of the experimental work described in this report took place at GCS Corrosion Consultants Inc's. laboratory in Howell, New Jersey. The project research work and this report were prepared by Dr. Gerald C. Soltz and Mrs. Rose-Marie Soltz, for GCS Corrosion Consultants Inc..

EXECUTIVE SUMMARY

The purpose of the research work, was to examine how environmental contaminants, common in marine coating operations, can adversely affect the service life of epoxy coatings, typically used to protect ship's salt water ballast tanks. The research examined how steel surfaces contaminated with: sodium chloride, sea salt, ferrous sulphate or blasting grit dust particles, affect a coating's performance. Four epoxy paint systems were tested over controlled amounts of these contaminants: coal tar, SovaPon, Mare Island and Aquapon. Aquapon is a clear (unpigmented) polyamide epoxy coating. While Aquapon is not normally used for immersion service, it was included in the testing program, to allow observation of subtle under film corrosion reactions that are obscured by pigmented coatings. It was found that the Aquapon and coal tar coatings performed similarly, and blistered to the same extent, at the contamination levels used in the test program. The Sovapon and Mare Island coatings were slightly more resistive to blistering when compared to Aquapon or coal tar but they also suffered from under film corrosion at contamination levels well below that required to cause obvious surface blisters.

The research work found that the widely accepted argument that, HIGH HUMIDITY IS THE CAUSE OF "STEEL RUST BACK", IS FALSE. In fact, PROPERLY CLEANED STEEL WILL NOT RUST FOR THOUSANDS OF HOURS, EVEN AT 100% RELATIVE HUMIDITY. Problems with Surface rerusting after blasting, indicates that there is still undesirable contamination on the steel's surface.

The very common practice of lowering the humidity to stop blasted steel surfaces from rapidly turning, does not correct the basic cause of the problem, it only hides it. Dehumidification only retards the flash rusting process temporarily.

DAMAGING UNDER FILM CORROSION WILL QUICKLY FORM UNDER THE PAINT FILM, EVEN IF THE CONTAMINATED SURFACES WERE TOTALLY RUST FREE WHEN IT WAS BEING COATED.

Under film rust (brownish color), was observed to quickly develop under clear coatings applied over steel that was contaminated. This surface turning, started during the air curing period. The rust color then turned black, after the first few days of submerged testing. This brown to black color change indicated, that water had diffused through the coating, reached the contaminated steel, and aqueous under film reactions were now occurring.

NOTE: These under film corrosion reactions did not occur where ever the metal surface was contamination free, even after 4500 hours of submerged testing. An electrolyte is needed for corrosion to occur and it does not diffuse through a sound coating system.

The research work also determined that:

1. Chloride ion surface contamination levels below $.25 \text{ ug. Cl}^-/\text{cm}^2$ ($0.014 \text{ oz. NaCl}/1000 \text{ Ft.}^2$) of steel surface, caused little or no visual steel substrate corrosion under clear epoxy, even after 4500 hours immersion in pressurized sea water.
2. Increasing the chloride ion surface contamination levels, to between 0.25 & $8 \text{ ug. Cl}^-/\text{cm}^2$ (0.014 oz. & $0.44 \text{ oz. NaCl}/1000 \text{ Ft.}^2$) of surface area, caused visible steel substrate corrosion reactions under clear epoxy in pressurized seawater immersion. The amount of under film corrosion that occurred was proportional to the amount of contamination placed on the substrate.
Note: Within these levels of under film contamination, neither the clear or the opaque epoxies had physical surface blistering. However, slight film surface roughening (micro-blistering) due to under film corrosion, could be observed in both the clear Aquapon and the coal tar epoxy coating when contamination exceeded $5 \text{ ug. Cl}^-/\text{cm}^2$ ($0.28 \text{ oz. NaCl}/1000 \text{ Ft.}^2$), or in the case of Sovapon and Mare Island epoxy, when it exceeded $10 \text{ ug. Cl}^-/\text{cm}^2$ ($0.58 \text{ oz. NaCl}/1000 \text{ Ft.}^2$).
3. Chloride ion surface contamination levels between 8 - $16 \text{ ug Cl}^-/\text{cm}^2$ ($.44$ - $.88 \text{ oz NaCl}/1000 \text{ Ft.}^2$) of steel surface caused steel substrate corrosion micro-blisters, which could be seen under clear epoxy. The micro-blisters coalesced to form the larger osmotic blisters, and under film corrosion stopped when this occurred. The same size osmotic blisters occurred in clear and coal tar epoxy. A slightly higher level of surface contamination 20 - $32 \text{ ug. Cl}^-/\text{cm}^2$ (1 - $1.7 \text{ oz NaCl}/1000 \text{ Ft.}^2$) was needed to cause blistering in the other coating systems. However, even at the $10 \text{ ug. Cl}^-/\text{cm}^2$ ($0.55 \text{ oz. NaCl}/1000 \text{ Ft.}^2$) level, these systems had very slight surface roughening, indicating considerable under film corrosion was in progress.
4. Much higher levels of sulphate contamination ($>250 \text{ ug./cm}^2$) ($8.5 \text{ oz}/1000 \text{ ft.}^2$) are required to cause coating blistering compared to chloride. While sea water does contain sulphate, it is at only 20% of the chloride level. Therefore, blistering from sea water sulphate contamination does not appear to be a primary problem.
Note: Sulphate in high sulphur coal slag grit is also a possible sulphate source.

5. High chloride levels in blasting grit may also cause problems, if they transfer more than a critical amount of contamination to the metal substrate, during blasting. It is therefore advisable to check, that the grit being used has a low enough contamination level, to produce the level of surface cleanliness specified. The level of grit contamination can be quickly determined in the field, by using distilled water and a low cost pocket conductivity meter.
6. painting over medium or large, loose grit dust particles (+40 mesh), can cause pin point rusting failures. Small amounts of very fine grit dust did not cause coating failures unless they also contained chloride contamination.

EXECUTIVE SUMMARY CONCLUSION

At the present time there are no industry standards, to define the safe levels of chloride contamination for steel surfaces being coated. This study has determined that certain commonly occurring substances will cause surface contamination, that is very detrimental to an organic coating's life. The study has generated information, that helps to define the levels of chloride contamination, that are detrimental to the service life of epoxy coatings. These are preliminary contamination danger levels, that need to be field confirmed and refined over the next few years. This conformation should be done, by making it standard practice in the coatg industry, to take measurements of final surface contamination levels prior to any coating application. By routinely taking these measurements, many potential coating problems will be caught and corrected before coating is applied, and simultaneously it will build up a record, of the actual levels of contamination, coatings were applied over. These field generated numbers, can be used in the future to develop practical contamination danger levels, for various types of coatings and service conditions. This new information will help to change coating application work from art to engineering.

However until sufficient field numbers are generated, and confirmed, industry can still start engineering coating surface preparation. This can be done by using the contamination danger levels found in the present study, as preliminary guide numbers. By using these numbers and some of the equipment listed in appendix B- GUIDE III, coating inspectors can measure surface contamination levels and decide if the steel surface is clean enough to coat.

It should be stressed that the critical contamination level may not be a single value, but a range, that is partly dependant upon the intended end service use. Under severe service conditions or when using less tolerant coatings, the lower end of the contamination range should be observed. However, under mild service conditions and/or if more tolerant coatings are being used, the higher end of the contamination range may still be acceptable. Each coating manufacturer must help to determine what the allowable contamination range is for their specific products, when used under specified service conditions. They should be willing to fully guarantee their products performance for the intended service conditions, when applied over the contamination levels they claim are safe.

The grit used for surface preparation can transfer contamination to the surface, if it contains too much chloride or sulphate. Therefore, the conductivity of the grit being used should be monitored, particularly if low surface contamination levels are being specified.

In order to help stimulate and accelerate the coating industry into doing routine contamination testing of grit and blasted surfaces, prior to coating application, we have also included in this report, a number of guides that can be used to better understand, measure and control surface contamination problems:

1. GUIDE I:
THE PRIMARY CAUSES OF COATING FAILURE.
2. GUIDE II:
THE EFFECT'S OF SURFACE CONTAMINATION.
3. GUIDE III
METHOD'S AVAILABLE FOR DETECTING & MEASURING
SURFACE AND GRIT CONTAMINATION.
4. GUIDE IV
SELECTING THE BEST SURFACE PREPARATION METHOD
TO REMOVE SUBSTRATE CONTAMINATION PROBLEMS.

NOTE: These 4 guides, are in appendix B.

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GUIDE III: METHOD'S AVAILABLE FOR DETECTING 5 MEASURING
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6. **OBJECTIVES OF THE PRESENT RESEARCH PROGRAM**

The purpose of this research project, was to review the technical literature and determine the key types of marine surface contamination that causes blistering in organic coating systems.

The initial literature search, turned up a number of references, that suggested that there were two important contaminants that stimulated steel corrosion and induced coating blister problems. The two contaminants were:

A. Chloride (reference#s 2,9,11,13,14,15,18,20,24,27,32,33)

AND

B. Sulphate (reference#s 10,15,20,23,27,31,32,33)

The above references did not define the level at which these contaminants become dangerous. The following references suggested some danger levels for chloride and sulphate in micrograms of contamination per square centimeter:

A. Chloride (reference #16(2ug), #38(10ug),#40(2ug))

B. Sulphate (reference #16(10ug),#38(50ug),#40(65ug))

Since the start up of the present research program, there have been a number of additional papers published, that have confirmed the validity choosing these two contaminants for study in our project:

A. Chloride (reference#s 1,3,5,6,18,28,29,30,35,36)

B. Sulphate (reference#s 1,3,5,6,28,29,30,35,36).

The level at which chloride and sulphate contamination damage coatings was not determined in the above references, but it was suggested in the following papers:

A. Chloride (ref.#4(5-10ug), #8(3ug),#25&26(10ug), 39(10ug))

B. Sulphate (reference# 25 & 26 (100ug), 39 (50ug))

In addition to investigating the effects of chemical contaminants, the program also tested for the effects that small quantities of spent grit had, if painted over. No similar testing work was reported in the literature. This can be a major contaminant, if the clean up after blasting, is not done completely.

These contaminants, were then placed under standard epoxy tank coatings and tested at environmental conditions, that simulated those found in salt water ballast tanks. The research program tested these contaminants, at many different concentration levels, to see what effects each level caused. The project's original goal, was to determine the maximum concentration level, for each type contaminant, that could be tolerated by three standard ballast tank coatings, before visible film blistering occurred.

This testing approach is a common one, in the paint industry. However, this method over looks the fact, that considerable paint system damage can occur, well before visible blisters are formed. Therefore, in addition to testing with standard (opaque) epoxy tank coatings, a clear epoxy coating system was included in the test program. This clear coating, allowed us to observe the normally hidden under film micro-corrosion and micro-blister reactions, along with the readily seen osmotic blisters.

NOTE: It is very important to point out, that our research work found a very close correlation, between the extent of blistering in the coal tar epoxy and the clear Aquapon epoxy, at the levels of contamination tested. This correlation, helps to legitimize the use of this clear epoxy, for the observation of substrate corrosion and micro-blistering. These subtle substrate reactions occur, long before any osmotic blistering is evident in the coating's surface. The use of a high performance, clear epoxy coating, to study these substrate corrosion reactions, turned up many facts that are obscured by the normally used opaque coatings.

RESEARCH DESIGN

New hot rolled steel panels, were carefully cleaned of all soluble contaminants, corrosion products and oil. These clean panels, were then air blasted with new steel grit. This blasting removed all roil-scale, leaving a clean steel surface with a anchor pattern of 1.5-2 roils. These ultra clean steel test panels, were then carefully doped with controlled amounts of contaminants. Various levels of contamination were applied, in amounts that were measured in micro-grams (ug) of contamination, per square centimeter (CM²) of surface area.

The contaminants used during this study were; analar sodium chloride, ASTM grade sea salt mixture, analar ferrous sulphate and various types of grit. After the controlled application of contamination was completed, the panels were painted by airless spray. The coatings used were; standard epoxy tank coating systems (three types tested) and a clear epoxy coating. The standard tank coating systems were applied at their manufacturers recommended film thickness of (12-16 mils)(300-400 microns) in 2 or 3 coats. The clear epoxy system was built up in 5 coats, to a typical tank lining thickness of, 12-14 roils (300-350 microns).

After the coatings were fully cured, the test panels were tested submerged, in a pressurized salt water test chamber, under the harshest conditions that would normally be expected in a ship's saltwater ballast tank, ie. 900F and 50 PSI pressure (115 ft head).

The panels were examined daily during the first week of testing, and then once every few days, for the next 300 hours of exposure. Testing was then continued further until 1900 to 4500 hours of submerged exposure had occurred.

The ASTM D714-5 Degree of Blistering Scale was initially used to record the extent of the coating's blistering. The ASTM D714-5 scale was then converted to a newer SSPC blister rating system, which produces single numbers from 0 (total failure) to 10 (no blisters), and is much easier to graph.

8. PROJECT EXPERIMENTAL RESULTS

OVERVIEW:

There are three parts to the experimental results.

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2. Testing using blasting grit particles and dust as surface contaminants under standard tank coatings.-----PAGES 45-57
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8.1 CHEMICAL SURFACE CONTAMINANTS:

There are eleven test series in this part of the experimental work. The testing program used Sodium Chloride, Sea Salt or Ferrous Sulphate as the chemical surface contaminants. These contaminants were used under:

- A. Coal Tar Epoxy- Series 1 through 5
- B. Sovapon Epoxy- Series 7 through 9
- C. Mare Island Epoxy- Series 10 and 11

The experimental results from of each of these test series, are shown as both tables and graphs. Also, some comments on each test series are included under each graph.

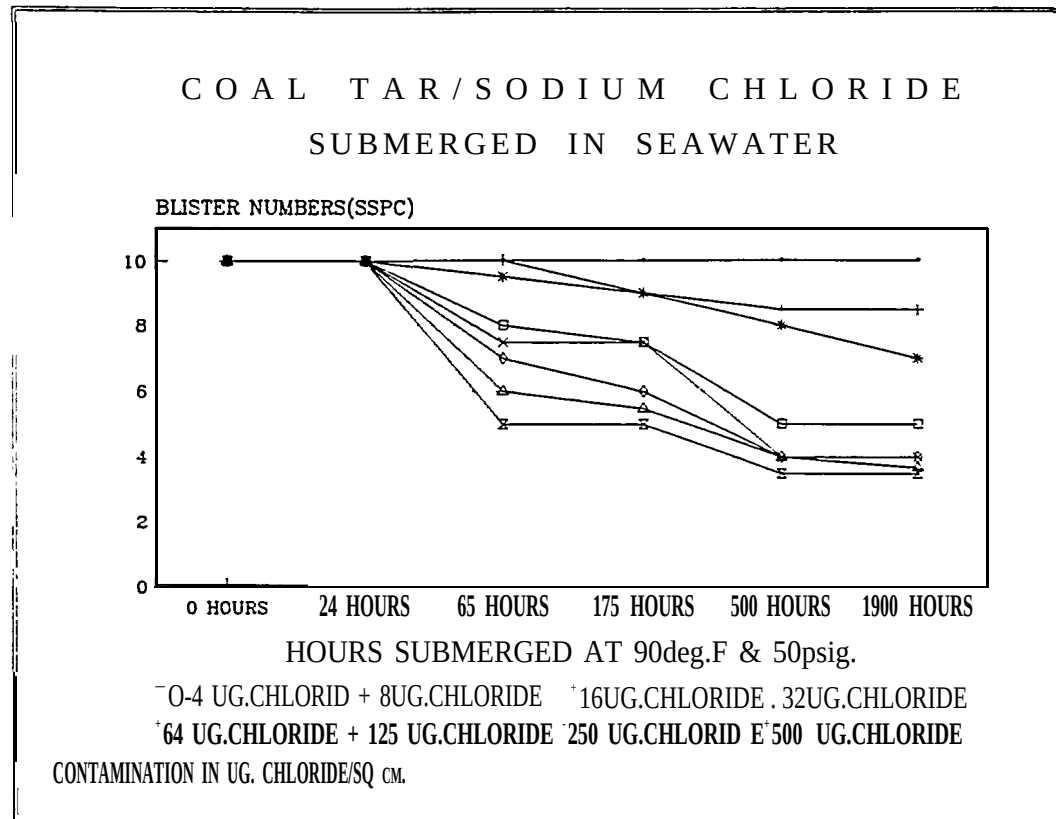
NOTE: The ASTM D714 blister evaluation method and the SSPC conversion method used for evaluating these tests, are listed in Appendix A, pages A11 & A12.

PANEL TEST SERIES # 1
 COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS
 LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING
 THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F

LEVEL OF CHLORIDE ION $\text{ug. Cl}_2 /$	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR				
			24 HOURS	65 HOURS	175 HOURS	500 HOURS	1900 HOURS
0	12A	15	10	10	10	10	10
0	13A	15	10	10	10	10	10
*(0.00)							
1	12B	16	10	10	10	10	10
1	13B	14	10	10	10	10	10
*(0.054)							
2	12C	10	10	10	10	10	10
2	13C	14	10	10	10	10	10
*(0.11)							
4	12D	17	10	10	10	10	10
4	13D	15	10	10	10	10	10
*(0.22)							
8	10A	17	10	10	9	8	8
8	11A	16	10	10	9+	9	9
*(0.44)							
1 6	10B	14	10	9+	9	9	9
16	11B	16	10	10	9+	7	5
*(0.88)							
32	10C	16	10	8	7	5	5
32	11C	16	10	8	8	5	5
*(1.7)							
64	10D	16	10	7+	7	4	4
64	11D	21	10	8	8	4	4
*(3.5)							
125	6A	16	10	7	7	5	5
125	7A	15	10	7	5	3	4
*(7)							
250	6B	18	10	6	6	5	5
250	7B	15	10	6	5	3	3
*(14)							
500	6C	18	10	5	5	4	4
500	7C	15	10	5	5	3	3
*(28)							
1000	6D	17	10	4	4	4	4
1000	7D	16	10	4	4	3	3
*(56)							

INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM () OUNCES
 OF SODIUM CHLORIDE SPREAD EVENLY OVER 1000 SQ.FT. OF SURFACE
 NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION
 OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND
 ANY RATINGS 7 OR LOWER ARE JUDGED TO BE BELOW THE ACCEPTABLE
 FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PROJECTS EXPERIMENTAL RESULTS



PANEL TEST SERIES #1- This was the first test series performed. 24 tests were run using sodium chloride as the contaminant and coal-tar epoxy as the coating. The coal tar epoxy was applied at an average thickness of 16 mils (400 microns) in two coats as per manufacturers instructions. The contamination levels ran from 0-1000 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 1900 hours. Periodic examinations were made to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

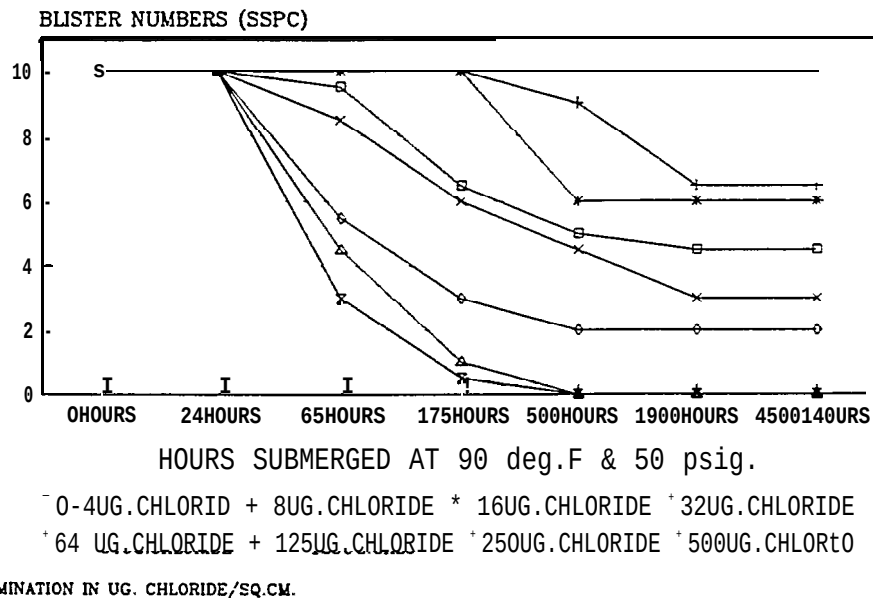
The tests found that no blistering occurred if the contamination was below 4 micrograms per square centimeter. At 8 micrograms per square centimeter blistering slowly increased with time, and the coating's condition after 1900 hours of testing was just passable (#7 by SSPC standards). Above this level of contamination the coating failed within 500 hours. The induction period before the onset of visible blistering varied between less than 65 hours (heavy contamination) to about 175 hours (light contamination) .

PANEL TEST SERIES # 2
 COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS
 LEVELS OF SEA SALT. TESTING WAS DONE BY SUBMERGING THE
 PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION $\text{ug. Cl}^- / \text{cm.}^2$	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. OF PAINT	SSPC THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	65 HOURS	175 HOURS	500 HOURS	1000 HOURS	4500 HOURS
0	19A	16	10	10	10	10	10	10
	20A	15	10	10	10	10	10	10
* (0.00)								
1	19B	17	10	10	10	10	10	10
	20B	14	10	10	10	10	10	10
* (0.054)								
2	19C	19	10	10	10	10	10	10
	20C	15	10	10	10	10	10	10
* (0.11)								
4	19D	20	10	10	10	10	10	10
	20D	18	10	10	10	10	10	10
* (0.22)								
8	17A	16	10	10	10	10	7	7
	18A	13	10	10	10	8	6	6
* (0.44)								
16	17B	16	10	10	10	6	6	6
	18B	16	10	10	10	6	6	6
* (0.88)								
32	17C	13	10	9+	7	5	4	4
	18C	15	10	10	6	5	5	5
* (1.7)								
64	17D	14	10	8	6	5	2	2
	18D	16	10	9	6	4	4	4
* (3.5)								
125	15A	14	10	5	2	2	2	2
	16A	13	10	6	4	2	2	2
* (7)								
250	15B	14	10	4	1	0	0	0
	16B	15	10	5	1	0	0	0
* (14)								
500	15C	14	10	3	1	0	0	0
	16C	16	10	3	1	0	0	0
* (28)								
1000	15D	18	10	3	1	0	0	0
	16D	18	10	3	1	0	0	0
* (56)								

INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM () OUNCES
 OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.
 NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION
 OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND
 ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE
 FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

COAL-TAR EPOXY/SEA SALT SUBMERGED IN SEAWATER



PANEL TEST SERIES #2- 24 tests were run using sea salt as the contaminant and coal-tar epoxy as the coating. The coal tar epoxy was applied at an average thickness of 16 mils (400 microns) in two coats as per manufacturers instructions. The contamination levels ran from 0-1000 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. c.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

The tests found that no blistering occurred if the contamination was below 4 micrograms per square centimeter. At 8 micrograms per square centimeter blistering slowly increased with time, and the coating's condition after 1900 hours was not passable (#6.5 SSPC). However after another 2600 hours testing no further deterioration occurred. Above this level of contamination the coating failed within 175-500 hours. The induction period before the onset of visible blistering varied between 65 hours (heavy contamination) to less than 500 hours (light contamination).

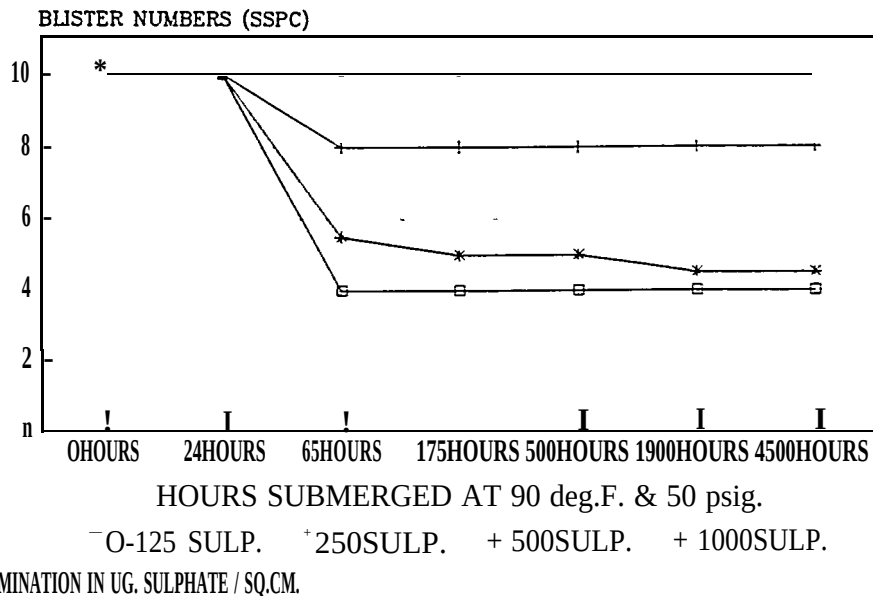
PANEL TEST SERIES #3

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF FERROUS SULPHATE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF SULPHATE ION ug. SO_4^{--} /Cm.	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	SSPC THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	65 HOURS	175 HOURS	500 HOURS	1900 HOURS	4500 HOURS
2	26C	14	10	10	10	10	10	10
2	27C	15	10	10	10	10	10	10
4	26D	15	10	10	10	10	10	10
4	27D	15	10	10	10	10	10	10
8	23A	16	10	10	10	10	10	10
8	25A	13	10	10	10	10	10	10
16	23B	16	10	10	10	10	10	10
16	25B	14	10	10	10	10	10	10
32	23C	14	10	10	10	10	10	10
32	25C	10	10	10	10	10	10	10
64	23D	15	10	10	10	10	10	10
64	25D	13	10	10	10	10	10	10
125	21A	18	10	10	10	10	10	10
125	22A	12	10	10	10	10	10	10
250	21B	18	10	10	10	10	10	10
250	22B	17	10	10	10	10	10	10
250	27A	16	10	4	4	4	4	4
500	21C	13	10	5	5	5	5	5
500	22C	16	10	3	3	3	3	3
500	26A	15	10	10	10	9+	6	6
500	27B	16	10	4	4	4	4	4
750	26B	16	10	3	3	3	3	3
1000	21D	16	10	5	5	5	5	5
1000	22D	17	10	3	3	3	3	3

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

COAL TAR/FERROUS SULPHATE SUBMERGED IN SEAWATER



PANEL TEST SERIES #3- 24 tests were run using ferrous sulphate as the contaminant and coal-tar epoxy as the coating. The coal tar epoxy was applied at an average thickness of 16 mils (400 microns) in two coats as per manufacturers instructions. The contamination levels ran from 2-1000 MICRO-GRAMS SULPHATE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

The tests found that no blistering Occurred if the contamination was below 125 micrograms per square centimeter. At 250 micrograms per square centimeter blistering rapidly increased with time, and the coating's condition after 65 hours of testing was failing by SSPC standards. The induction period was less than 65 hours.

PANEL TEST SERIES #4-PART A

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION $\frac{\text{ug. Cl}^-}{\text{cm.}}$	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
1.25	29A	9	10	10	10	10	10	10
1.25	30A	11	10	10	10	10	10	10
* (0.067)								
2.5	29B	10	10	10	10	10	10	10
2.5	30B	12	10	10	10	10	10	10
* (0.135)								
5	29D	11	10	10	10	10	9	9
5	30D	11	10	10	10	10	9	9
* (0.27)								
10	29C	9	10	9+	7	7	7	7
10	30C	11	10	9+	7	7	7	7
* (0.54)								
16	32A	12	10	9	9	6	6	6
16	33A	13	10	10	10	6	6	6
16	38B	16	10	9	9	7	7	7
16	39B	15	10	9	9	6	6	6
* (0.88)								
20	32B	14	10	9	7	6	6	6
20	33B	13	10	7	7	3	3	3
20	38A	13	10	9	9	6	4	4
20	39A	13	10	7	8	5	5	5
* (1.1)								
32	32C	12	10	5	4	5	5	4
32	33C	14	10	7	7	5	5	5
32	38C	11	10	4	4	5	5	5
32	39C	13	10	7	7	5	5	5
* (1.7)								
40	32D	13	10	4	3	5	5	5
40	33D	15	10	6	6	5	5	5
40	38D	13	10	8	8	5	5	5
40	39D	15	10	5	5	5	5	5
* (2.2)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #4 PART B

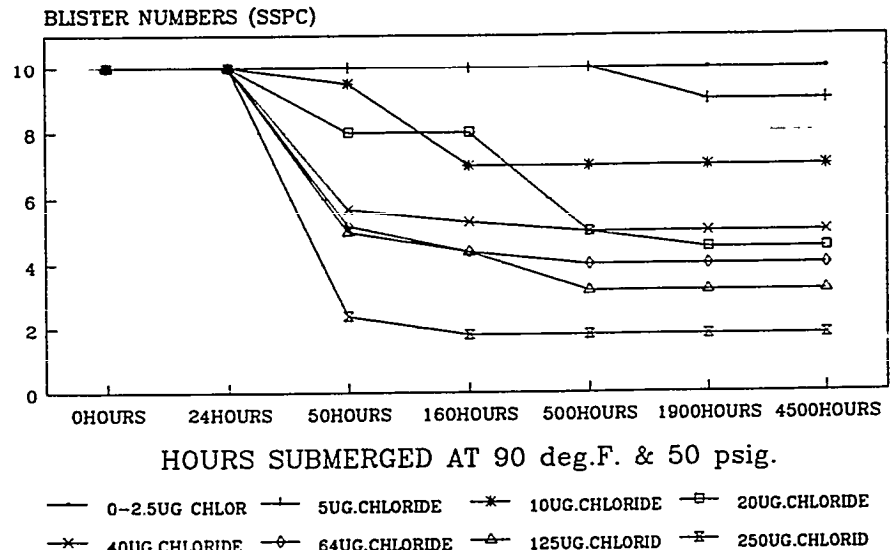
COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION ug. Cl ⁻ / Cm.	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	SSPC THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
64	34A	13	10	3	2	2	2	2
64	35A	13	10	3	2	2	2	2
64	37A	14	10	6	6	5	5	5
64	58A	12	10	7	6	5	5	5
64	59A	13	10	7	6	6	6	6
* (3.5)								
125	34B	14	10	2	2	2	2	2
125	35B	13	10	3	2	2	2	2
125	37B	16	10	6	6	2	2	2
125	58B	13	10	7	7	5	5	5
125	59B	13	10	7	7	5	5	5
* (7)								
250	34C	14	10	2	1	1	1	1
250	35C	12	10	3	2	2	2	2
250	37C	12	10	1	1	1	1	1
250	58C	13	10	3	2	2	2	2
250	59C	13	10	3	3	3	3	3
* (14)								
500	34D	16	10	2	1	1	1	1
500	35D	14	10	1	1	1	1	1
500	37D	13	10	1	1	1	1	1
500	58D	14	10	2	2	2	2	2
500	59D	10	10	2	2	2	2	2
* (28)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF-SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED By THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

COAL TAR/SODIUM CHLORIDE SUBMERGED IN SEA WATER

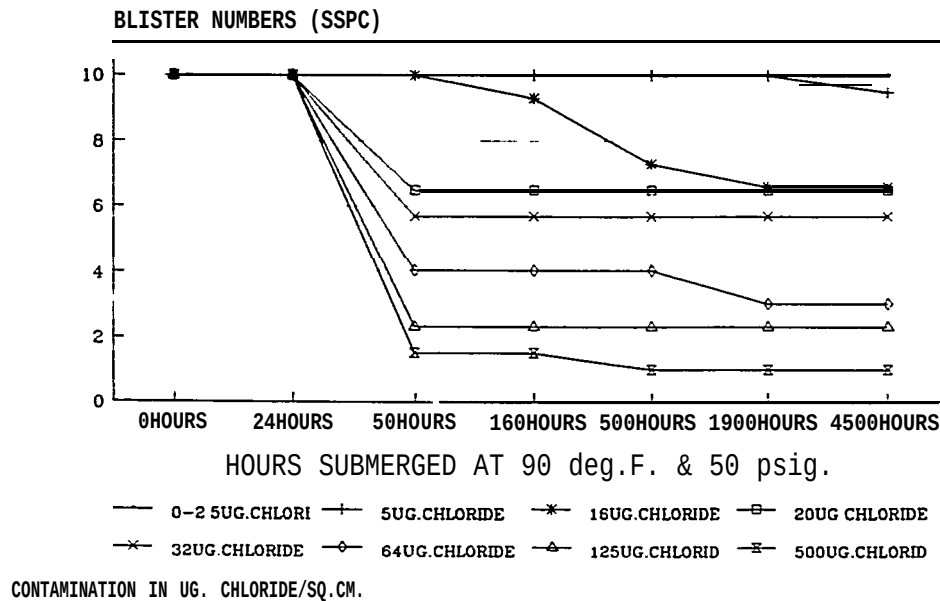


CONTAMINATION IN UG. CHLORIDE/SQ.CM.

PANEL TEST SERIES #4- This was the second test series (44 tests) run with sodium chloride as the contaminant and coal tar epoxy. The epoxy was applied at an average thickness of 14 mils (350 microns) in two coats as per manufacturers instructions. The contamination levels ran from 1.25-500 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F. (33 deg. C.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714. For grafting purposes this scale was converted to the SSPC NUMBERS.

The tests found that no blistering occurred if the chloride contamination was below 2.5 micrograms per square centimeter. Under film corrosion (ASTM S9) without osmotic blisters, was observed at the 5 microgram level after 1900 hours of testing. At 16 micrograms per square centimeter blistering slowly increased with time, and the coating's condition after 4500 hours of testing was just passable (#7 by SSPC standards). At the 32 microgram level of contamination the coating failed within 175 hours. The induction period before the onset of visible blistering varied between less than 50 hours (heavy contamination) to about 500 hours (light contamination).

COAL TAR EPOXY\ SEA SALT SUBMERGED IN SEA WATER



PANEL TEST SERIES #5- This was the second test series (32 tests) run with sea salt as the contaminant and coal tar epoxy. The epoxy was applied at an average thickness of 14 mils (350 microns) in two coats as per manufacturers instructions. The contamination levels ran from 0-500 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. c.) ran 4500 hours. Periodic examinations were made to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

The tests found that no blistering occurred if the chloride contamination was below 5 micrograms per square centimeter. Under film corrosion (ASTM S9-) without osmotic blisters, was just observed at the 5 microgram level after 1900 hours of testing. At 16 micrograms per square centimeter contamination, the blistering slowly increased with time, and the coating's condition after 4500 hours of testing was just below passable (#6.5 by SSPC standards). At the 20 microgram level of contamination, the coating failed within 175 hours. The induction period before the onset of visible blistering varied between less than 50 hours (heavy contamination) to over 500 hours (light contamination).

PANEL TEST SERIES #5-PART A

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SEA SALT. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF TEST CHLORIDE ION ug.Cl ₂ ⁻ / Cm. DISTILLED WATER ONLY	PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. OF PAINT	SSPC THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
0	60A&B	12	10	10	10	10	10	10
METHYL ALCHOL ONLY								
0	60C&D	11	10	10	10	10	10	10
*(0.00)								
1.25	46A	13	10	10	10	10	10	10
1.25	48A	13	10	10	10	10	10	10
*(0.067)								
2.5	46B	14	10	10	10	10	10	10
2.5	48B	13	10	10	10	10	10	10
*(0.135)								
5	46C	14	10	10	10	10	10	10-
5	48C	14	10	10	10	10	10	10-
*(0.27)								
10	46D	14	10	10	10	10	9	9
10	48D	14	10	10	10	10	9	9
*(0.54)								
16	43A	13	10	10	10	7	7	7
16	45A	15	10	10	10	9	7	7
16	114A	13	10	10	8	6	6	6
*(0.88)								
20	43B	14	10	6	6	6	6	6
20	45B	14	10	7	7	7	7	7
*(1.1)								
32	43C	13	10	6	6	6	6	6
32	45C	14	10	7	7	7	7	7
32	114B	13	10	4	4	4	4	4
*(1.7)								
40	43D	14	10	4	4	4	4	4
40	45D	14	10	3	3	3	3	3
*(2.2)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #5- PART B

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SEA SALT. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF TEST CHLORIDE 10N ug.Cl ₂ ⁻ / Cm. ²	PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D. F.T. OF PAINT	SSPC THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
64	41A	11	10	4	4	4	4	4
64	42A	13	10	4	4	4	4	4
64	114C	16	10	4	4	4	1	1
*(3.5)								
125	41B	13	10	2	2	2	2	2
125	42B	13	10	3	3	3	3	3
125	114D	16	10	2	2	2	2	2
*(7)								
250	41C	10	10	1	1	1	1	1
250	42C	14	10	3	3	1	1	1
*(14)								
500	41D	12	10	0	0	0	0	0
500	42D	16	10	2	2	1	1	1
*(28)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL Blistering WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #6

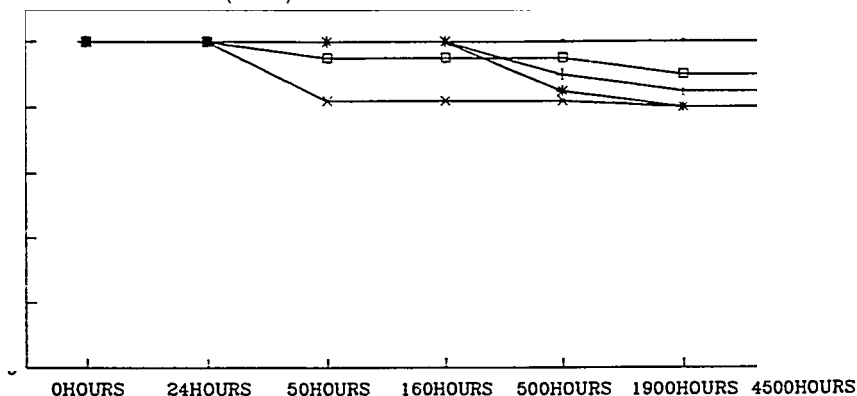
COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF FERROUS SULPHATE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF SULPHATE ION ug. SO_4^{2-} Cm.	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
1.25	55A	13	10	10	10	10	10	10
1.25	56A	14	10	10	10	10	10	10
2.5	56B	13	10	10	10	10	10	10
5	55B	14	10	10	10	10	10	10
5	55C	14	10	10	10	10	10	10
5	56C	12	10	10	10	10	10	10
10	55D	12	10	10	10	10	10	10
10	56D	12	10	10	10	10	10	10
16	52A	13	10	10	10	10	10	10
16	53A	14	10	10	10	10	10	10
20	52B	14	10	10	10	10	10	10
20	53B	14	10	10	10	10	10	10
32	52C	12	10	10	10	10	10	10
32	53C	14	10	10	10	10	10	10
40	52D	13	10	10	10	10	10	10
40	53D	12	10	10	10	10	10	10
64	49A	13	10	10	10	10	9	9
64	51A	15	10	10	10	8	8	8
125	34B	14	10	10	10	10	9	9
125	35B	13	10	10	10	7	7	7
250	34C	14	10	9	9	9	9	9
250	35C	12	10	10	10	10	9	9
500	34D	16	10	9+	9+	9+	9	9
500	35D	14	10	7	7	7	7	7

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

COAL TAR/FERROUS SULPHATE SUBMERGED IN SEA WATER

BLISTER NUMBERS (SSPC)



HOURS SUBMERGED AT 90 deg.F. & 50 psig.

— 0-40UG. SO4 + 64UG. SO4 * 125UG. SO4 □ 250UG. SO4
—x— 500UG. SO4

CONTAMINATION IN UG.SO4(SULPHATE)/SQ.CM.

PANEL TEST SERIES #6- This was the second test series (24 tests) run with ferrous sulphate as the contaminant and coal tar epoxy. The epoxy coating was applied at an average thickness of 13 mils (325 microns) in two coats as per manufacturers instructions. The contamination levels ran from 1.25-500 MICRO-GRAMS sulphate PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714. For grafting purposes this scale was converted to the SSPC NUMBERS.

The tests found that no blistering occurred if the sulphate contamination was 40 micrograms per square centimeter or lower. Under film corrosion (ASTM 9) without osmotic blisters, was observed at the 64 microgram level after 1900 hours of testing. Even at 500 micrograms per square centimeter contamination, the blistering only slowly increased with time, and the coating's condition after 4500 hours of testing was still passable (#8) by SSPC standards. The induction period before the onset of visible blistering varied between less than 50 hours (heavy contamination) to 500 hours (light contamination).

PANEL TEST SERIES #7- PART A

SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION ug. Cl ₂ ⁻ / Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
1.25	62A	9	10	10	10	10	10	10
1.25	63A	10	10	10	10	10	10	10
1.25	64A	11	10	10	10	10	10	10
*(0.067)								
2.5	62B	8	10	10	10	10	10	10
2.5	63B	10	10	10	10	10	10	10
2.5	64B	10	10	10	10	10	10	10
*(0.135)								
5	62C	10	10	10	10	10	10	10
5	63C	10	10	10	10	10	10	10
5	64C	11	10	10	10	10	10	10
*(0.27)								
10	62D	11	10	10	10	10	10	10
10	63D	10	10	10	10	10	10	10
10	64D	11	10	10	10	10	10	10
*(0.54)								
16	65A	12	10	10	10	10	10	9
16	66A	11	10	10	10	10	10	9
16	67B	11	10	10	10	10	10	10
*(0.88)								
20	65B	11	10	10	10	10	10	9
20	66B	9	10	10	10	10	10	9
20	67B	11	10	10	10	10	10	10
*(1.1)								
32	65C	11	10	10	10	9	9	9
32	66C	11	10	10	10	9	8	7
32	67C	10	10	10	10	9	9	9
*(1.7)								
40	65D	10	10	10	10	7	7	6
40	66D	10	10	10	10	7	7	6
40	67D	10	10	10	10	7	7	7

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.
NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #7-PART B

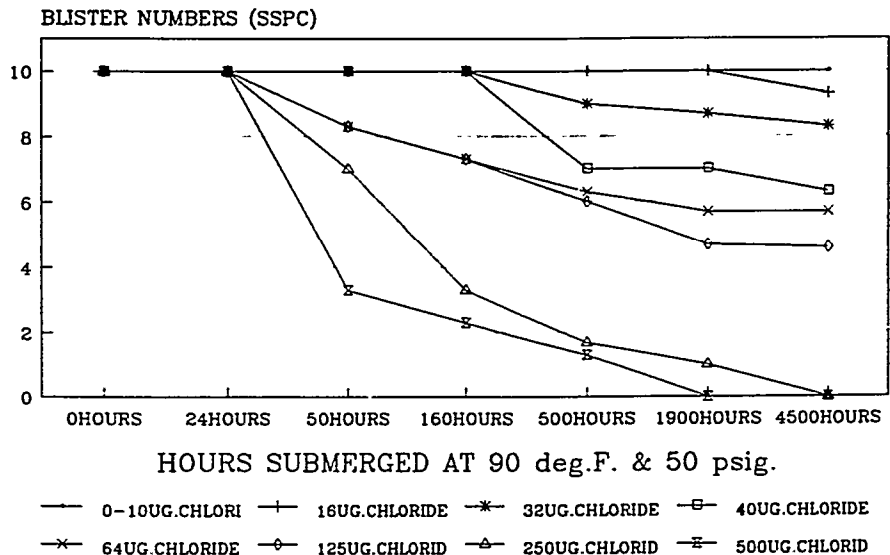
SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION ug.Cl ⁻ / Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
64	68A	10	10	8	8	6	5	5
64	69A	11	10	10	7	7	7	7
64	70A	11	10	7	7	6	5	5
* (3.5)								
125	68B	11	10	8	7	5	4	3
125	69B	12	10	10	8	8	6	6
125	70B	11	10	7	7	5	4	4
* (7)								
250	68C	10	10	8	3	0	0	0
250	69C	11	10	5	2	2	1	0
250	70C	11	10	8	5	3	2	1
* (14)								
500	68D	11	10	3	2	0	0	0
500	69D	10	10	5	3	2	0	0
500	70D	11	10	2	2	2	0	0
* (28)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

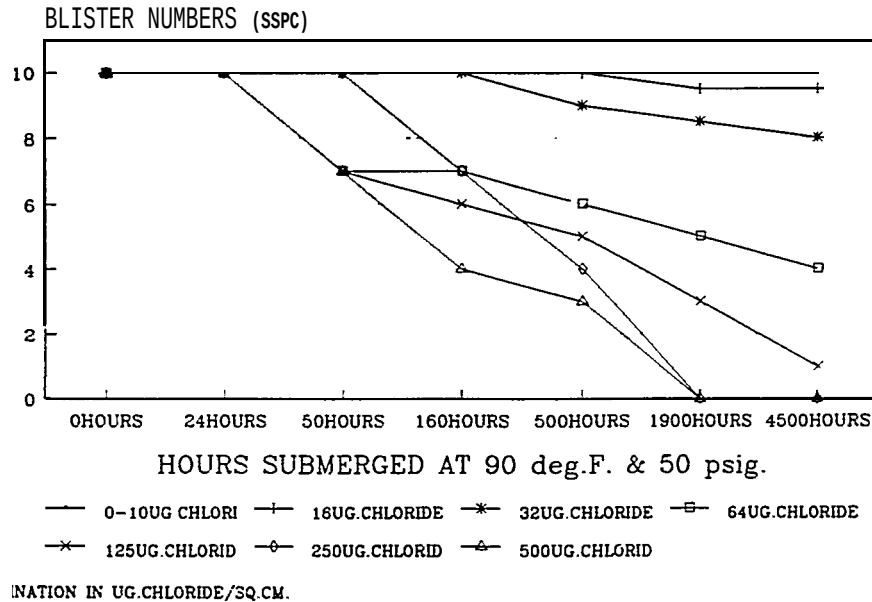
SOVAPON/SODIUM CHLORIDE SUBMERGED IN SEA WATER



PANEL TEST SERIES #7- This test series (36 tests) run with sodium chloride as the contaminant and sovapon epoxy. The epoxy was applied at an average thickness of 11 mils (275 microns) in two coats as per manufacturers instructions. The contamination levels ran from 0 to 500 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

The tests found that no blistering occurred if the chloride contamination was below 10 micrograms per square centimeter. Under film corrosion (ASTM S9) without osmotic blisters, was observed at the 10 microgram level after 4500 hours of testing. At 32 micrograms per square centimeter blistering slowly increased with time, and the coating's condition after 4500 hours of testing was still passable (#8 by SSPC standards). At the 64 microgram level of contamination the coating failed within 500 hours. The induction period before the onset of visible blistering varied between less than 50 hours (heavy Contamination) to about 500 hours (light contamination).

SOVAPON/SEA SALT SUBMERGED IN SEA WATER



PANEL TEST SERIES #8- This test series (48 tests) run with sea salt as the contaminant and sovapon epoxy. The epoxy was applied at an average thickness of 11 mils (275 microns) in two coats as per manufacturers instructions. The contamination levels ran from 0 to 500 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 4500 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

PANEL TEST SERIES #8- PART A

SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SEA SALT. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION $\text{ug. Cl}_2^- / \text{Cm.}^2$	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24	50	160	500	1900	4500
			HOURS	HOURS	HOURS	HOURS	HOURS	HOURS
NOTE: (S)=SINGLE COAT								
1.25	71A	10(S)	10	10	10	10	10	10
1.25	72A	10(S)	10	10	10	10	7	5
1.25	73A	10	10	10	10	10	10	10
*(0.067)								
2.5	71B	10(S)	10	10	10	10	8	7
2.5	72B	11	10	10	10	10	7	5
2.5	64B	10	10	10	10	10	10	10
*(0.135)								
5	71C	8(S)	10	10	10	10	8	6
5	72C	11(S)	10	10	10	10	7	6
5	73C	11	10	10	10	10	10	10
*(0.27)								
10	71D	9(S)	10	10	10	10	8	6
10	72D	7(S)	10	10	10	10	6	4
10	73D	10	10	10	10	10	10	10
*(0.54)								
16	74A	9(S)	10	10	10	10	8	6
16	75A	11	10	10	10	10	10	10
16	76A	9(S)	10	10	10	7	7	5
16	115A	15	10	10	10	10	9	9
16	116A	12	10	10	10	10	6	6
16	117A	13	10	10	10	10	9	7
*(0.88)								
20	74B	9(S)	10	10	10	10	10	7
20	75B	11	10	10	10	10	10	10
20	76B	10(S)	10	10	10	8	7	6
*(1.1)								
32	74C	10(S)	10	10	10	10	7	4
32	75C	9(SQ)	10	10	10	10	7	5
32	76C	10(S)	10	10	10	7	6	4
32	115B	15	10	10	10	10	10 -	9
32	116B	12	10	10	9	7	6	5
32	117B	13	10	10	10 -	10 -	10 -	10 -
*(1.7)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.
(S) AFTER DFT INDICATES ONLY A SINGLE HEAVY COAT OF PAINT INSTEAD OF THE RECOMMENDED TWO COAT SOVAPON SYSTEM.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE Acceptable FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #8- PART B
 SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS
 LEVELS OF SEA SALT. TESTING WAS DONE BY submerging THE
 PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE 10N ug.Cl ₂ / Cm. ²	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT NOTE: (S)=SINGLE COAT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
40	74D	10(S)	10	10	10	10	7	5
40	75D	11(S)	10	10	10	10	9	8
40	76D	10(S)	10	10	10	8	7	6
* (2.2)								
64	77A	10	10	10	10-	6	6	6
64	78A	9(S)	10	9	9	6	6	4
64	79A	10	10	10	10	10	7	6
64	115C	15	10	4	4	4	3	2
64	116C	13	10	4	4	4	4	2
64	117C	13	10	6	6	6	5	4
* (3.5)								
125	77B	11	10	7	6	5	4	3
125	78B	10(S)	10	6	6	3	1	0
125	79B	11	10	10	10-	8	3	0
125	115D	15	10	7	4	4	3	1
125	116D	13	10	7	6	4	2	0
125	117D	13	10	7	4	4	3	2
* (7)								
250	77C	10(S)	10	4	4	4	0	0
250	78C	11(S)	10	4	4	2	0	0
250	79C	11	10	10	7	4	0	0
* (14)								
500	77D	11(S)	10	2	2	2	0	0
500	78D	11(S)	10	3	2	2	0	0
500	79D	10	10	7	4	3	0	0
* (28)								

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES
 OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

(S) AFTER DFT INDICATES ONLY A SINGLE HEAVY COAT OF PAINT INSTEAD
 OF THE RECOMMENDED TWO COAT SOVAPON SYSTEM.

NOTE: THE PANEL BLISTERING WAS RATED By THE SSPC MODIFICATION OF
 THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY
 RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE
 LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #9- PART A

SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF FERROUS SULPHATE. TESTING WAS DONE BY SUBMERGING THE PANELS IN Artificial SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF SULPHATE ION Ug . SO ₂ /Cm.	TEST PANEL 'S NUMBER & SECTION	PANEL 'S AVERAGE D.F.T. OF PAINT NOTE: (S)=SINGLE COAT	THE BLISTER CONDITIONS					
			OBSERVED AFTER TESTING FOR					
			24	50	160	500	1900	4500
			HOURS	HOURS	HOURS	HOURS	HOURS	HOURS
0	8 9A&B	11(S)	10	10	10	10	6	6
0	89C&D	11	10	10	10	10	10	10
0	90A&B	9(S)	10	10	10	6	6	6
0	90C&D	11	10	10	10	10	10	10
1.25	80A	9(S)	10	10	10	10	7	7
1.25	81A	9(S)	10	10	10	10	9	9
1.25	82A	9(S)	10	10	10	10	10	6
2.5	80B	10(S)	10	10	10	10	7	7
2.5	81B	10(S)	10	10	10	10	9	9
2.5	82B	11(S)	10	10	10	10	10	8
5	80C	9(S)	10	10	10	10	7	7
5	81C	7(S)	10	10	10	7	5	4
5	82C	9(S)	10	10	10	10	10	7
10	80D	9(S)	10	10	10	10	6	6
10	81D	9(S)	10	10	10	7	6	5
10	64D	11(S)	10	10	10	10	10	6
16	83A	11	10	10	10	10	10	10
16	84A	11	10	10	10	10	10	10
16	85B	13	10	10	10	10	10	10
20	83B	11	10	10	10	10	10	10
20	84B	11	10	10	10	10	10	10
20	85B	12	10	10	10	10	10	10
32	83C	12(S)	10	10	10	10	8	8
32	84C	11	10	10	10	10	10	10
32	85C	14	10	10	10	10	10	10

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE Acceptable FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

PANEL TEST SERIES #9-PART B

SOVAPON EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF FERROUS SULPHATE. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF TEST SULPHATE ION Ug./Cm. ²	PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. OF PAINT NOTE:(S)=SINGLE COAT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR					
			24 HOURS	50 HOURS	160 HOURS	500 HOURS	1900 HOURS	4500 HOURS
40	83D	12(S)	10	10	10	10	9	9
40	84D	10	10	10	10	10	10	10
40	85D	15	10	10	10	10	10	10
64	86A	12	10	10	10	10	10	10
64	87A	10(S)	10	10	10	10	10	6
64	88A	10(S)	10	10	10	10	7	6
125	86B	11	10	10	10	10	10	10
125	87B	9(S)	10	10	10	10	7	3
125	88B	10(S)	10	10	10	9	8	7
250	86C	11	10	10	10	10	10	10
250	87C	10(S)	10	10	6	6	2	0
250	88C	11(S)	10	10	9	9	9	9
500	86D	11	10	10	10	10	10	10
500	87D	10(S)	10	10	6	6	2	0
500	88D	7(S)	10	5	5	3	0	0

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW THE ACCEPTABLE FAILURE LEVEL FOR SUBMERGED BALLAST SERVICE.

COMMENTS ON SERIES # 9 (Note: No graph was done for this series.)

This test series (36 tests) was run with various thickness applications of Sovapon over Ferrous Sulphate contamination. Where the coating was applied according to manufacturers thickness specification and in the proper number of coats, there was no blistering from sulphate even at 500 ug per square centimeter.

Severe coating blistering did occur in this series from solvent entrapment, when the coating was not applied according manufacturer's recommended thickness and number of coats. At low contamination levels, this type of blistering did not have active corrosion under still sound blisters. Many of these blisters broke open during testing and then active corrosion occurred.

PANEL TEST SERIES #10

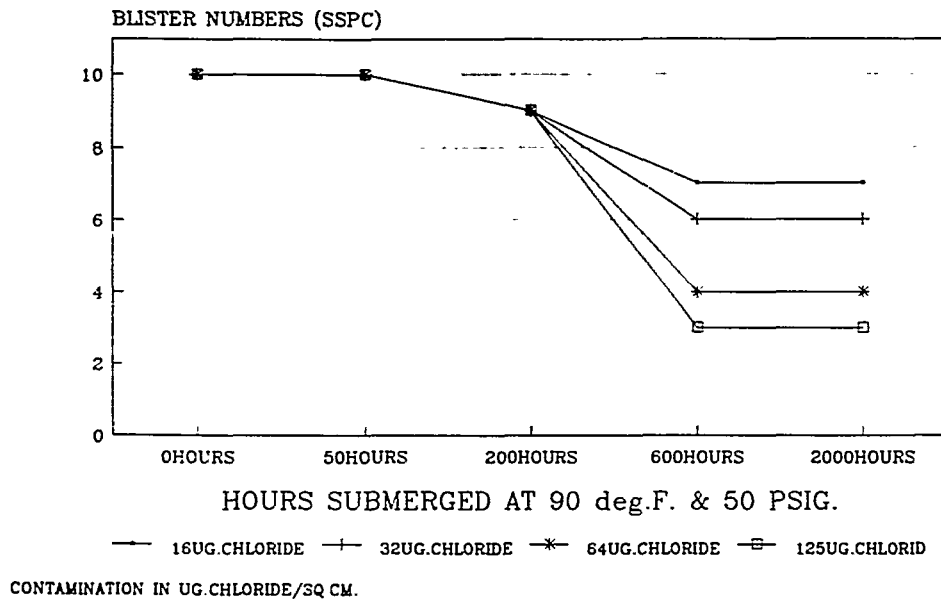
MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SODIUM CHLORIDE. TESTING WAS DONE BY SUBMERGING THE PANELS IN Artificial SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF TEST CHLORIDE ION ug.C½ / Cm.	PANEL'S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR				
			24 HOURS	50 HOURS	200 HOURS	600 HOURS	2000 HOURS
16	127A	12	10	9	9	7	7
16	128A	12	10	9	9	7	7
16	129A	13	10	9	9	7	7
* (0.88)							
32	127B	12	10	9	9	6	6
32	128B	12	10	9	9	6	6
	129B	13	10	9	9	6	6
* (1.7)							
64	127C	14	10	9	9	4	4
64	128C	12	10	9	9	4	4
64	129C	13	10	9	9	4	4
* (3.5)							
125	127D	14	10	9	9	3	3
125	128D	12	10	9	9	3	3
125	129D	13	10	9	9	3	3
* (7)							

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (*) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN Acceptable FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

MARE ISLAND EPOXY/SALT SUBMERGED IN SEA WATER



PANEL TEST SERIES #10- This test series (12 tests) run with sodium chloride as the contaminant and Mare Island epoxy. The epoxy was applied at an average thickness of 13 mils (325 microns) in three coats as per manufacturers instructions. The contamination levels ran from 16 to 125 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 2000 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

PANEL TEST SERIES #11

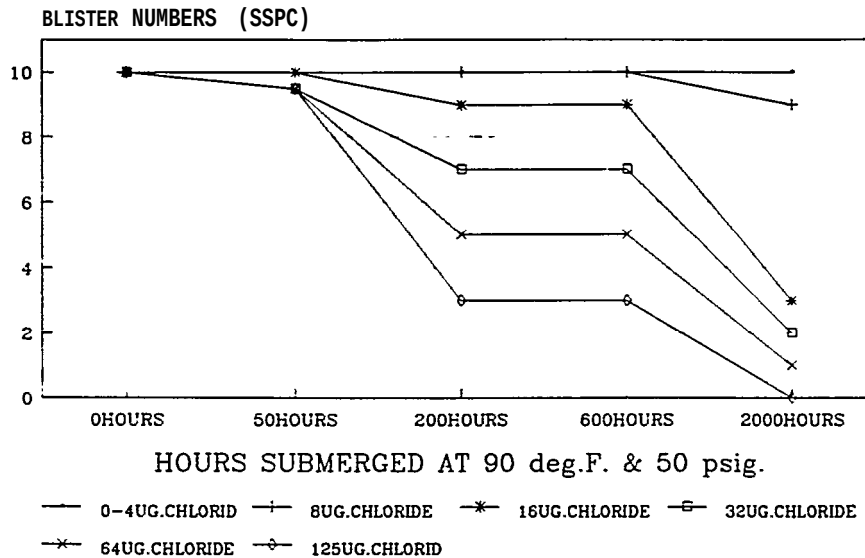
MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH VARIOUS LEVELS OF SEA SALT. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

LEVEL OF CHLORIDE ION $\text{ug. Cl}^- /$ Cm.^2	TEST PANEL ' S NUMBER & SECTION	PANEL ' S AVERAGE D.F.T. OF PAINT	THE BLISTER CONDITIONS OBSERVED AFTER TESTING FOR				
			24 HOURS	50 HOURS	200 HOURS	600 HOURS	2000 HOURS
0	202	14	10	10	10	10	10
0	203	14	10	10	10	10	10
*(0.00)							
1	130A	13	10	10	10	10	10
1	131A	12	10	10	10	10	10
*(0.054)							
2	130B	13	10	10	10	10	10
2	131B	12	10	10	10	10	10
*(0.11)							
4	130C	13	10	10	10	10	10
4	131C	13	10	10	10	10	10
*(0.22)							
8	130D	13	10	10	10	10	9
8	131D	13	10	10	10	10	10
*(0.44)							
16	134A	12	10	10	9	9	3
16	135A	13	10	10	9	9	3
16	136A	18	10	10	9.5	9	7
*(0.88)							
32	134B	12	10	9.5	7	7	2
32	135B	13	10	9.5	7	7	2
32	136B	18	10	10	8	8	7
*(1.7)							
64	134C	13	10	9.5	5	5	1
64	135C	12	10	9.5	5	5	1
64	136C	14	10	9.5	6	6	1
*(3.5)							
125	134D	13	10	9.5	3	3	0
125	135D	12	10	9.5	3	3	0
125	136D	14	10	9.5	4	4	0
*(7)							

* INDICATES THE EQUIVALENT LEVEL OF CHLORIDE ION FROM (.) OUNCES OF SODIUM CHLORIDE EVENLY SPREAD OVER 1000 SQ.FT. OF SURFACE.

NOTE: THE PANEL BLISTERING WAS RATED BY THE SSPC MODIFICATION OF THE ASTM D714 BLISTER SCALE. A 10 RATING IS PERFECT AND ANY RATINGS 7 OR BELOW ARE JUDGED TO BE BELOW AN ACCEPTABLE FAILURE LEVEL FOR COATINGS USED IN SUBMERGED BALLAST SERVICE.

MARE ISLAND EPOXY/SEA SALT SUBMERGED IN SEA WATER



PANEL TEST SERIES #11- This test series (22 tests) run with sea salt as the contaminant and Mare Island epoxy. The epoxy was applied at an average thickness of 13 mils (325 microns) in three coats as per manufacturers instructions. The contamination levels ran from 1 to 125 MICRO-GRAMS CHLORIDE PER. SQUARE CENTIMETER. The submerged testing in seawater at 90 deg. F.(33 deg. C.) ran 2000 hours. Periodic examinations were made, to observe changes in the coating such as rusting or blistering. The blisters were rated according to ASTM D714, however for grafting purposes this scale was converted to the SSPC BLISTER NUMBERS.

8.2 GRIT PARTICLE SURFACE CONTAMINANTS:

The most abundant potential contaminant during blasting is the grit itself. Usually considerable effort is expended to insure the blasted surfaces are cleaned free of all grit particles and the fine dust produced during blasting. The most effective method for grit removal is by final vacuuming over the entire surface. However, even with full attention to details, it is very difficult to keep the horizontal surfaces totally grit free. Grit particles tend to get stuck between staging boards, inside pipe poles, in plastic covers, and it is a hard job to get every particle up. The question is, how clean do we have to be, to get good coating results. Three series of tests were run using:

- 1.) Mare Island Epoxy- Series 12A-D
- 2.) Coal Tar Epoxy- Series 13A-D
- 3.) Polyamide Epoxy- Series 14A-D

Four types of grit contamination were used in each series:

- 1.) Copper slag, with an ASTM conductivity of 100uS
- 2.) Coal Slag with a ASTM conductivity of 210uS
- 3.) Coal Slag with a ASTM conductivity of 1400uS.

Note: This grit was found to contain a large amount of chloride contamination and it was causing the high conductivity.

4.) Coal Slag with a ASTM conductivity of 100uS. This is the same coal slag as number 3, but it had been washed free of chloride with distilled water. After washing, it was dried.

The grits were sieved to four sizes:

- 1.) Larger than 20 mesh- +20
- 2.) Between 20 and 40 mesh- +40
- 3.) Between 40 and 80 mesh- +80
- 4.) Less than 80 mesh- -80

The testing found that it is very important to clean up the grit properly. The larger size particles and the more contaminated grit had the highest tendency to cause problems. The three coat Mare Island system performed better than the two coat systems. This better performance, may be due to the better wetting action of the primer in Mare Island's system.

PANEL TEST SERIES #12A

MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH APACHE COPPER SLAG WITH A ASTM CONDUCTIVITY OF 100 uS. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 900F.

SIZE MESH	LEVEL OF GRIT	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
	ug. Cm.2			OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 500 HOURS	2000 HOURS
+20	3000	260A	15	10	10	10	10	10
+20	3000	262A	16	10	10	10	10	10
+40	3000	260B	15	10	10	10	10	10
+40	2000	262B	16	10	10	10	10	10
+80	3000	260C	17	10	10	10	10	10
+80	3000	262C	15	10	10	10	10	10
-80	2000	260D	17	10	10	10	10	10
-80	2000	262D	15	10	10	10	10	10

This test series using Mare Island epoxy over Apache copper slag as the contaminant looked very good. There was no sign of break down in way of the grit particles. This paint performed better than the other coatings in these tests, but it is a three coat system. The better wetting of primer coat in this coating system, may have helped it to pass this test.

PANEL TEST SERIES #12B

MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH
POLYGRIT COAL SLAG WITH A ASTM CONDUCTIVITY OF 210 uS.
TESTING WAS DONE BY SUBMERGING THE PANELS IN
ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
	ug. Cm.2			OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	263A	15	10	10	10	10	10
+20	2000	264A	14	10	10	10	10	10
+40	2000	263B	15	10	10	10	10	10
+40	2000	264B	14	10	20	10	10	10
+80	3000	263C	16	10	10	10	10	10
+80	3000	264C	16	10	10	10	10	10
-80	2000	263D	16	10	10	10	10	10
-80	2000	264D	16	10	10	10	10	10

This test series using Mare Island epoxy over Polygrit Coal Slag as the contaminant looked very good. There was no sign of break down in way of the grit particles. This paint performed better than the other coatings in these tests, but it is a three coat system. The better wetting of primer coat in this coating system, may have helped it to pass this test.

PANEL TEST SERIES #12C

MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH COAL SLAG WITH A ASTM CONDUCTIVITY OF 1400uS (FROM CHLORIDE). TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 900F₀

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	265A	15	10	10	10	10	8*
+20	2000	267A	17	10	10	10	10	9*
+40	2000	265B	15	10	10	10	10	10
+40	2000	267B	17	10	10	10	20	8*
+80	2000	265C	15	10	10	10	10	10
+80	3000	267C	17	10	10	10	10	10
-80	2000	265D	15	10	10	10	10	10
-80	2000	267D	17	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using Mare Island epoxy over a poor quality Coal Slag as the contaminant had some failures at larger size particles. There was no visible sign of break down in way of the finer grit particles but there may have been hidden underfilm corrosion. This paint still performed better than the other coatings in these tests, but it is a three coat system. The better wetting of primer coat in this coating system, may have helped it in this test.

PANEL TEST SERIES #12D

MARE ISLAND EPOXY COATING OVER STEEL CONTAMINATED WITH COAL SLAG A ASTM CONDUCTIVITY OF 100 uS. (WASHED)
TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 900F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	200A	14	10	10	10	10	10
+20	3000	201A	15	10	10	10	10	10
+40	3000	200B	14	10	10	10	10	10
+40	2000	201B	15	10	10	10	10	10
+80	3000	200C	14	10	10	10	10	10
+80	3000	201C	15	10	10	10	10	10
-80	2000	200D	14	10	10	10	10	10
-80	2000	202D	15	10	10	10	10	10

This test series using Mare Island epoxy over the same Coal Slag as used for Series 12C. However, it had been cleaned of all chloride contamination. There was now no sign of break down in way of the grit particles. This helps to confirm the danger of using contaminated grit. This paint performed better than the other coatings in these tests, but it is a three coat system. The better wetting of primer coat in this coating system, may have helped it to pass this test.

PANEL TEST SERIES #13A

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH
APACHE COPPER SLAG WITH A ASTM CONDUCTIVITY OF 100 uS.
TESTING WAS DONE BY SUBMERGING THE PANELS IN
ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
	ug.			OBSERVED	AFTER	TESTING	FOR	
	Cm. ²			24 HOURS	50 HOURS	200 HOURS	600 HOURS	2000 HOURS
+20	3000	106A	12	10	10	7*	7*	7*
+20	2000	107A	15	10	10	7*	7*	7*
+40	3000	106B	13	10	10	10	10	10
+40	3000	107B	15	10	10	10	10	10
+80	3000	106C	15	10	10	10	10	10
+80	3000	107C	16	10	10	10	10	10
-80	2000	106D	15	10	10	10	10	10
-80	2000	107D	16	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using Coal Tar Epoxy over Apache copper slag as the contaminant looked good except at the larger grit particles where failures occurred rapidly. There was no sign of break down in way of the small grit particles. This two coat system did not perform as well as the three coat Mare Island System.

PANEL TEST SERIES #13B

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH
POLYGRIT COAL SLAG WITH A ASTM CONDUCTIVITY OF 210 uS.
TESTING WAS DONE BY SUBMERGING THE PANELS IN
ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	108A	17	10	10	10	10	10
+20	3000	109A	14	10	10	8*	8*	8*
+40	3000	108B	17	10	10	10	10	10
+40	3000	109B	14	10	10	10	10	10
+80	3000	108C	14	10	10	10	10	10
+80	3000	109C	13	10	10	10	10	10
-80	2000	108D	14	10	10	10	10	10
-80	2000	109D	13	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using Coal Tar Epoxy over Polygrit coal slag as the contaminant looked good except at the larger grit particles where failures occurred rapidly. There was no sign of break down in way of the small grit particles. This two coat system did not perform as well as the three coat Mare Island System.

PANEL TEST SERIES #13C

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH COAL SLAG WITH A ASTM CONDUCTIVITY OF 1400uS (FROM CHLORIDE). TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 900F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	110A	14	10	10	10	8*	6*
+20	3000	111A	14	10	10	9*	8*	9*
+40	3000	110B	14	10	10	10	10	4*
+40	3000	111B	14	10	10	10	10	4*
+80	3000	110C	17	10	10	10	10	7*
+80	3000	111C	15	10	10	10	10	10
-80	2000	110D	17	10	10	10	10	10
-80	2000	111D	15	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using Coal Tar Epoxy over a poor quality Coal Slag as the contaminant had some failures at all size particles except the very fine ones. While there was no visible sign of breakdown in way of these very fine grit particles but there may have been hidden underfilm corrosion. This two coat system did not performed as well as the three coat Mare Island system.

PANEL TEST SERIES #13D

COAL TAR EPOXY COATING OVER STEEL CONTAMINATED WITH
COAL SLAG A ASTM CONDUCTIVITY OF 100 uS. (WASHED)

TESTING WAS DONE BY SUBMERGING THE PANELS IN
ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	112A	27	10	10	10	10	10
+20	3000	113A	15	10	10	9*	9*	9*
+40	3000	112B	17	10	10	10	10	10
+40	3000	113B	15	10	10	10	10	10
+80	3000	112C	16	10	10	10	10	10
+80	2000	112C	16	10	10	10	10	10
-80	2000	112D	16	10	10	10	10	10
-80	2000	113D	16	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series used Coal Tar Epoxy over the same Coal Slag as used for Series 13C. However, it had been cleaned of all chloride contamination. There was now no sign of break down in way of the smaller grit particles. This two coat system did not performed as well as the three coat Mare Island system.

PANEL TEST SERIES #14A

POLYAMIDE EPOXY COATING OVER STEEL CONTAMINATED WITH APACHE COPPER SLAG WITH A ASTM CONDUCTIVITY OF 100 uS. TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS					FOR
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	600 HOURS	2000 HOURS	
+20	3000	118A	14	10	10	10	10	5*	
+20	3000	119A	14	10	10	8*	8*	5*	
+40	3000	118B	14	10	10	10	10	5*	
+40	3000	119B	14	10	10	10	10	6*	
+80	3000	118C	14	10	10	10	10	10	
+80	3000	119C	14	10	10	10	10	9*	
-80	2000	118D	14	10	10	10	10	10	
-80	2000	119D	14	10	10	10	10	10	

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using polyamide epoxy over Apache Copper Slag as the contaminant, looked poor except at the smaller grit particles. There was no sign of break down in way of the very fine grit particles. This two coat system did not perform as well as the three coat Mare Island System in this test.

PANEL TEST SERIES #14B
 POLYAMIDE EPOXY COATING OVER STEEL CONTAMINATED WITH
 POLYGRIT COAL SLAG WITH A ASTM CONDUCTIVITY OF 210 uS.
 TESTING WAS DONE BY SUBMERGING THE PANELS IN
 ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

SIZE MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	50 HOURS	AFTER TESTING 200 HOURS	600 HOURS	FOR 2000 HOURS
+20	3000	120A	14	10	10	10	7*	4*
+20	3000	121A	15	10	10	10	7*	5*
+40	3000	120B	14	10	10	10	7*	3*
+40	3000	121B	15	10	10	10	8*	5*
+80	3000	120C	13	10	10	10	10	5*
+80	3000	121C	15	10	10	10	10	6*
-80	2000	120D	12	10	10	10	10	10
-80	2000	121D	15	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using polyamide epoxy over Polygrit coal Slag as the contaminant, looked poor except at the smaller grit particles. There was no sign of break down in way of the very fine grit particles. This two coat system did not perform as well as the three coat Mare Island System in this test.

PANEL TEST SERIES #14C

POLYAMIDE EPOXY COATING OVER STEEL CONTAMINATED WITH COAL SLAG WITH A ASTM CONDUCTIVITY OF 1400uS (FROM CHLORIDE). TESTING WAS DONE BY SUBMERGING THE PANELS IN ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

S I Z E MESH	LEVEL	TEST	PANEL 'S	CONDITIONS				
	OF	PANEL 'S	AVERAGE					
	GRIT	NUMBER	D.F.T.	OBSERVED	AFTER	TESTING	FOR	
	ug. Cm. ²	& SECTION	& PAINT	24 HOURS	50 HOURS	200 HOURS	600 HOURS	2000 HOURS
+20	3000	122A	13	10	10	10	7*	5*
+20	3000	123A	14	10	10	10	5*	3*
+40	3000	122B	12	10	10	10	7*	3*
+40	3000	122B	14	10	10	10	5*	1*
+80	3000	122C	14	10	10	10	10	0*
+80	2000	123C	15	10	10	10	5	0*
-80	2000	122D	14	10	10	10	10	10
	2000	123D	15	10	10	10	10	10

*HAS RUST SPOTS AT GRIT PARTICLES.

This test series using polyamide epoxy over a poor quality Coal Slag as the contaminant had some failures at all size particles except the very fine ones. While there was no visible sign of breakdown in way of these very fine grit particles but there may have been hidden underfilm corrosion. This two coat system did not performed as well as the three coat Mare Island system.

PANEL TEST SERIES #14D

POLYAMIDE EPOXY COATING OVER STEEL CONTAMINATED WITH
COAL SLAG A ASTM CONDUCTIVITY OF 100 Us. (WASHED)

TESTING WAS DONE BY SUBMERGING THE PANELS IN

ARTIFICIAL SEA WATER AT 50 PSI PRESSURE & 90°F.

S I Z E MESH	LEVEL OF GRIT ug. Cm. ²	TEST PANEL'S NUMBER & SECTION	PANEL'S AVERAGE D.F.T. & PAINT	CONDITIONS				
				OBSERVED 24 HOURS	AFTER 50 HOURS	TESTING 200 HOURS	FOR 600 HOURS	2000 HOURS
+20	3000	124A	15	10	10	10	6*	0*
+20	3000	125A	14	10	10	10	7*	0*
+40	3000	124B	15	10	10	10	7*	0*
+40	3000	125B	14	10	10	10	7*	0*
+80	3000	124C	14	10	10	10	10	0*
+80	3000	125C	13	10	10	10	7*	0*
-80	2000	124D	14	10	10	10	10	10
-80	2000	125D	13	10	10	10	10	10

HAS RUST SPOTS AT GRIT PARTICLES.

This test series used polyamide epoxy over the same Coal Slag as used for Series 14C. However, it had been cleaned of all chloride contamination. There was still considerable break down in way of all but the smallest grit particles. This two coat system 'did not performed as well under this test.

8.3 CLEAR EPOXY CONTAMINATION STUDIES

1. INTRODUCTION:

A number of tests were run with clear epoxy, to observe the subtle changes that occur underneath the paint films at the metal surface, prior to and during osmotic blistering. These observations were done with Aquapon polyamide epoxy, manufactured by Pittsburgh Paints. While this clear coating was not designed specifically for use in ballast tanks, it was found to be equal in performance to standard coal tar epoxy of similar thickness.

The ability to observe changes at the metal surface, during the slow corrosion and diffusion processes, was very important, as it showed how the microscopic corrosion effects, originating from surface contamination lead first to macroscopic subsurface blisters, and ultimately to the obvious osmotic surface blisters. The initial microscopic substrate reactions, are obscured by opaque paint films and go unnoticed.

The clean steel was contaminated with various levels of chloride and sulphate salts, similar to those used under the standard opaque coating. Contamination was applied only to the central part of the panels, leaving about a 1 inch clear border. This approach was used, as it left a contamination free metal standard, adjacent to contaminated metal for comparison purposes. This configuration also eliminated the tendency for coating failure at edges.

2. OBSERVATIONS WITH CHLORIDE CONTAMINATION:

A. INITIAL EFFECTS OF CONTAMINATION ON BARE BLASTED STEEL

As soon as the contamination application work started, it was observed that relatively low levels of contamination, would change the color of the steel surface slightly, if applied by water solution. Therefore, some initial work was done using methanol as a non aqueous solvent for dissolving the contaminants. The methanol worked well, and eliminated the initial surface turning problem. However after application, the methanol applied chlorides induced greater surface corrosion activity in air, than occurred from similar levels of chloride applied by water solutions.

This greater surface reactivity, may be due to the fact that methanol wets the steel better and this gets the chlorides onto the surface better than from water. Also the salt crystals deposited from methanol solutions are finer and more numerous.

It was therefore decided, to use only water solutions for application of the contaminants. A hot air blower was used to force dry the panels immediately after applying contamination, to minimize the degree of surface turning.

The presence of a contamination free border helped to highlight any surface color changes. Without this border, a higher level of contamination could have been required before any color changes could be observed.

In order to cause any immediate (30 seconds) surface turning (rust back), the surface contamination level had to be at least $\text{lug Cl}^-/\text{cm}^2$ ($.0540\text{Z NaCl}/1000\text{Ft}^2$). At higher levels of chloride, the immediate surface discoloration became more obvious, but never very dark.

When viewed closely, or with slight magnification (2X), it could be clearly seen that the surface turning in the case of chloride was not a smooth even color, but a distinct pattern of light brown micro spots (anodes), surrounded by still white metal areas (cathodes) . This uneven color pattern occurred even at the highest levels of contamination used.

This spotty corrosion pattern is not surprising. It occurs, because steel surfaces are not electro-chemically homogeneous. In fact, a steel surface is made up of dozens of microscopic potential micro-corrosion cells per square inch (6.5cm^2). These potential micro-corrosion cells, are activated when ever an electrolyte covers them. The dark spots, are the corrosion by-products from the micro-anodes, where corrosion (metal loss) is occurring. It is the corrosion by-products that are creating the observed color change. There is no corrosion or color change in way of cathodes (bright metal) that surrounds each anode area.

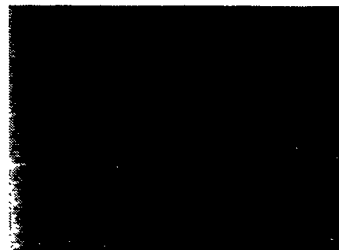
An immediate corrosion reaction starts up at each anode, when ever the solution covering it and the cathode, is an electrolyte. When the solution is alcohol or distilled water no reactions occur, because they are not electrolytes. However, when chloride contamination is added to distilled water, it's conductivity rapidly increases and it becomes an electrolyte and then surface corrosion can occur.

If no contamination is present to create an electrolyte corrosion can not occur. This is why a very clean surface does not turn in air, even at 100% relative humidity (see Photo Series A). Also see Appendix B, guide 11-2.2.1-3, for more details on these basic corrosion reactions.

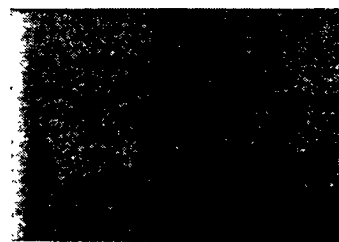
The slight surface turning, that accompanied the application of the intermediate and higher levels of surface contamination, was stopped from proceeding any further by immediately placing the contaminated panel into an oven at $130^{\circ}\text{F}(55^{\circ}\text{C})$.

PHOTO SERIES A
EFFECTS OF HUMIDITY AND CONTAMINATION
ON UNCOATED STEEL

CLEAN NEW STEEL
NO EXPOSURE
TO HUMIDITY or
CONTAMINATION
White metal



CLEAN STEEL after
1200 HOURS EXPOSURE
100% RELATIVE HUMIDITY
NO CONTAMINATION
Still white metal



CONTAMINATED STEEL
1UG. Chloride /CM²
500 HOURS EXPOSURE
100% RELATIVE HUMIDITY
Even brown turning over
about 20% of the surface



PHOTO SERIES A- Testing of clean steel in a humidity chamber at 100% non-condensing relative humidity found that clean steel did not rust, even after 1200 hours in this test (3500 hours in other tests). However, if small amounts of chloride were put on similarly cleaned steel surfaces, turning would quickly occur. The amount of corrosion caused by small amounts of chloride is relatively large, as the chloride is not directly consumed in the primary corrosion reactions that are taking place under the paint films. Therefore it is important to try to get the contamination levels as low as is practical, if a long coating life is required.

This heating of the panels, has the same effect as lowering the relative humidity to about 15%. It dries up the electrolyte on the surface, and halts further electrochemical reactions, until surface moisture returns. This procedure held the panels at the same level of slight discoloration for days, so that a complete test series could be contaminated in preparation for spraying.

B. SUBSTRATE CHANGES OCCURRING IN AIR AFTER COATING:

Obviously, surfaces painted with normal epoxy can not be observed once they are coated. This fact has led many coating inspectors to be satisfied, so long as the coated surfaces were white, or only slightly turned at the time of coating application. In fact, when turning is a problem, the Relative Humidity is often lowered and painting is done as quickly as possible.

This is not a solution to the contamination problem. The contaminated steel may look all right at the time of coating application, but it will still turn later under the coating. The myth about the power of dehumidification, to solve the problem of rust back, must be dispelled. If a surface tends to rust back, it must be tested by swab or limpet cell, to determine the level of contamination. If the contamination level tests too high, additional surface preparation should be done.

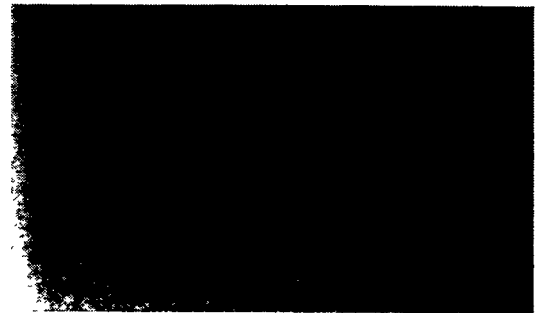
When contaminated surfaces were painted with clear epoxy, the metal surfaces were observed to noticeably darken over the week long cure period. The amount of darkening occurring under the coating in air, was greater at the higher levels of contamination. Note, no darkening could be perceived below the $\text{lug Cl}^-/\text{cm}^2$ level. The darkening in air, was always brownish in color, not black. These surface changes can be seen in the Photo Series B.

NOTE: Unused contaminated panels are still only dark brown after several years in air, but the brown is deeper in color now, then it was after the first month or two.

PHOTO SERIES B
EFFECTS OF CHLORIDE CONTAMINATION
UNDER CLEAR EPOXY COATING AFTER
AIR CURING BUT PRIOR TO
ANY SUBMERGED TESTING



1 UG. Cl^-/CM^2



2 UG. Cl^-/CM^2



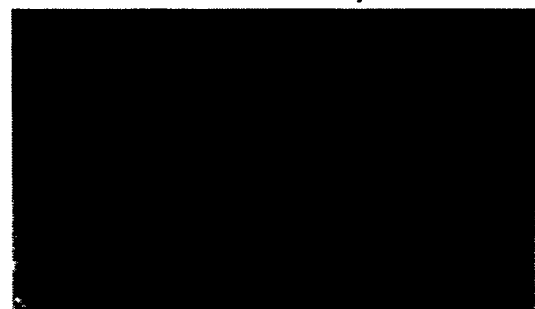
4 UG. Cl^-/CM^2



8 UG. Cl^-/CM^2



16UG. Cl^-/CM^2



32 UG. Cl^-/CM^2

PHOTO SERIES B- Shows the rust back effects that increasing levels of chloride caused under clear epoxy in air. At the time of coating application there was no discoloration on panels contaminated with less than 1UG Cl^-/CM^2 . Above this level there was very slight brownish discoloration which darkened noticeably during the week long air curing period. This under film rust back increases progressively with higher levels of chloride, until most of the surface is covered with brownish colored rust back. This brownish under film corrosion continues to get darker with time in air, but it does not get black until the panel is submerged. Note: The photographs shown in series B-E were taken at different times, but on the same panels. The photographed surfaces are printed at full scale size in series B-E.

C. INITIAL SUBMERGED TESTING OBSERVATIONS (INDUCTION STAGE)

After proper curing, the panels were subjected to submerged testing in seawater at 90°F(33°C) and a head pressure of 115 feet. Examination of the panels after 24 and 48 hours, found them to be unchanged at this time and the surface discoloration was still brown (THE MOISTURE INDUCTION PERIOD).

At the 70 hour inspection the fact that water had permeated through the 12 mil(300 micron) thick, semi-permeable coating was clearly shown, by a marked color change at the metal substrate, from light rust brown to black. The density of the black substrate discoloration, increased with each higher level of contamination. However in spite of this darkening, there was still the distinct anodic micro-spots of corrosion by-product which had turned from brown to black. These anodic spots were surrounded by white metal areas (cathodes).

Photo Series C shows the surface corrosion pattern occurring after seventy hours.

Note: Under submerged corrosion conditions, it is usual for the cathode area to surround the anode area. This is a typical way pits are formed. The area of pitting (anode) is shielded from oxygen, by corrosion by-products and it becomes lower in oxygen. The open, corrosion free area surrounding the anode, receives more oxygen, and it becomes the cathode area. At the cathode, dissolved oxygen is reduced to OH⁻ ions (alkaline), no corrosion occurs and the metal in this area stays white. In fact, the white areas are being protected from corrosion at the expense of the anode areas.

NOTE: This type of anode stabilization process (pitting) does not occur in atmospheric corrosion. Under atmospheric conditions there is a constant shifting of anodic and cathodic sites with time. This shifting leads to a more uniform pattern of surface corrosion, with an even rust film forming over the entire metal surface.

PHOTO SERIES C
EFFECTS OF CHLORIDE CONTAMINATION
UNDER CLEAR EPOXY COATING AFTER
70 HOURS OF SUBMERGED TESTING

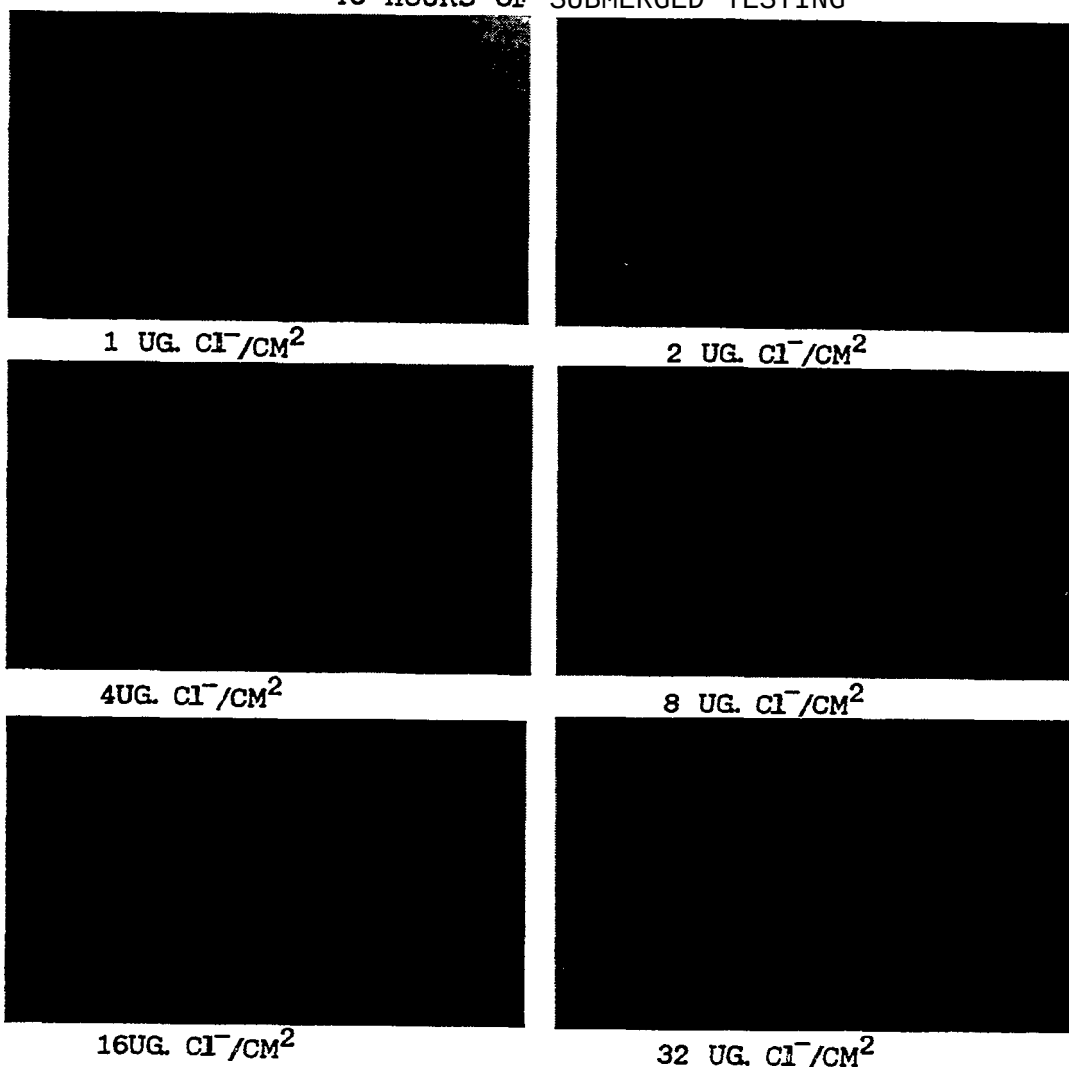


PHOTO SERIES C- shows the corrosion effects that increasing levels of chloride caused under clear epoxy after 70 hours submergence in sea water at 90°F (33°C) at 50 psig (3.3 BAR). At the time of coating application there was no discoloration on panels contaminated with less than 1UG Cl-/CM-. Above this level there was very slight discoloration which darkened noticeably during the week long air curing period. This under film rust increases progressively with higher levels of chloride, until most of the contaminated surface is covered with brownish colored rust. This brownish under film corrosion continues to get darker with time in air, but it did not get black until the panels were submerged. This transformation can be seen in this photographic series and indicates that moisture has permeated through the semi-permeable coating. Also slight discoloration could now be seen at the 0.5 UG Cl/cm² contamination level (not shown).

D. EFFECTS OF LONGER TERM SUBMERSION (EARLY GROWTH STAGE)

After the dramatic color change occurs at the metal substrate, then slow aqueous corrosion occurs. Both water and oxygen slowly diffuse through the semi-permeable film and this allows a slow micro-corrosion pitting process to go on underneath the sound paint film. During this micro-pitting process there is a slow buildup of corrosion by-products between the micro-anode and the surrounding cathode. After hundred of hours of these slow electro-chemical reactions, there is a noticeable buildup in the volume of corrosion by-product. The amount of corrosion by-product build up, is relative to the amount of contamination originally present, but factors such as the oxygen diffusion rate through the paint film may limit the rate of the reaction. At the higher levels of contamination, some small osmotic blisters (liquid filled) are also beginning to form. These osmotic blisters caused noticeable surface blistering. Note, where these larger blisters occur, there appears to be coalescing of many micro-corrosion cells into the larger osmotic type blisters. These micro-corrosion cells do not raise the surface of the coating. Also at this time, very small amounts of corrosion by-product can be observed, starting to building up on surfaces contaminated at only $0.5 \mu\text{g Cl}^-/\text{cm}^2$.

The surface of the coating is still very smooth except in way of the small number of osmotic type blisters that have occurred only on the heavily contaminated surfaces.

PHOTO SERIES D
EFFECTS OF CHLORIDE CONTAMINATION
UNDER CLEAR EPOXY COATING AFTER
800 HOURS OF SUBMERGED TESTING

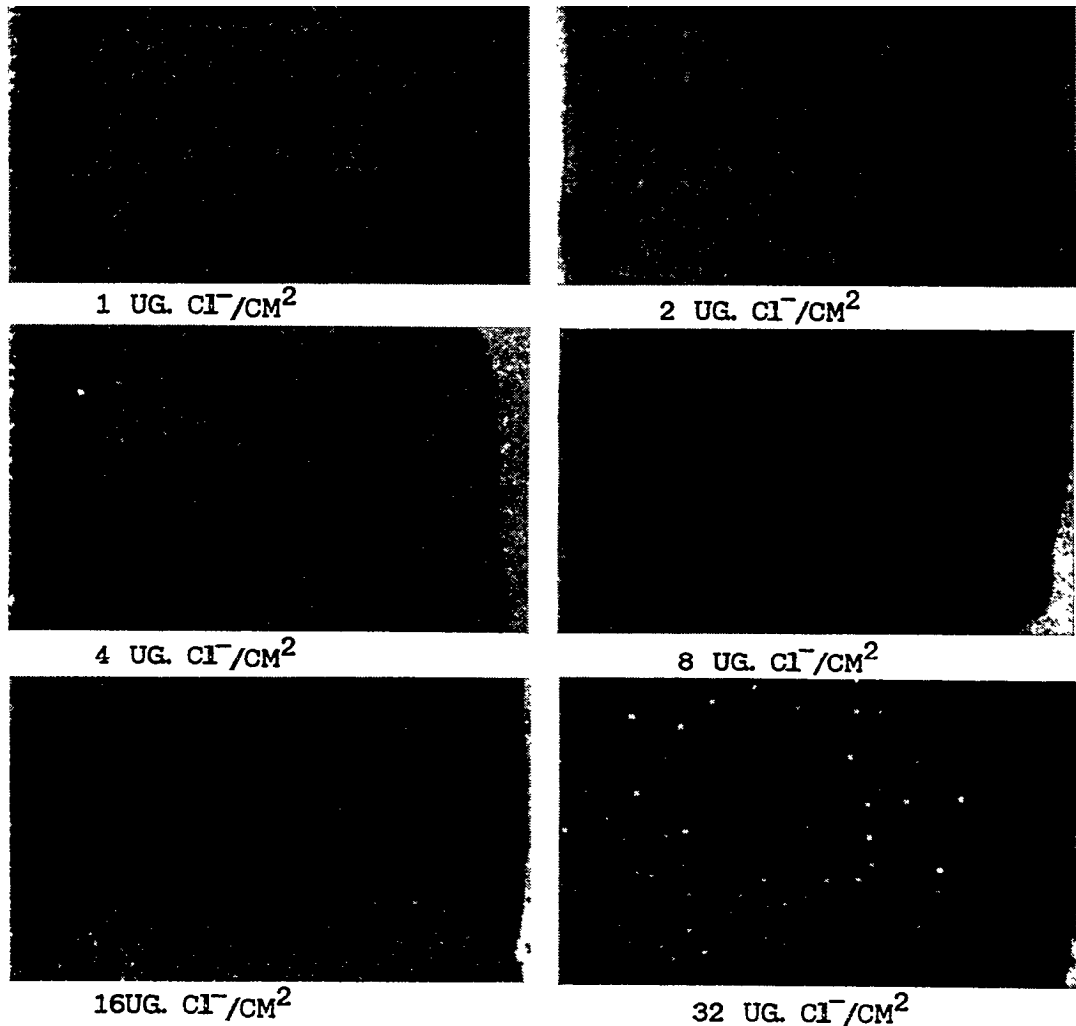


PHOTO SERIES D- shows the corrosion effects that increasing levels of chloride, caused under clear epoxy after 800 hours submergence in sea water at 90°F (33°C) at 50 psig (3.3 BAR). At the time of coating application there was no discoloration on panels contaminated with less than 1UG Cl^-/CM^2 . Above this level there was very slight discoloration which darkened noticeably during the week long air curing period. This brownish under film corrosion continued to get darker in air, and then turned black when the panels were submerged. This transformation indicated that water had reached the contaminated substrate. This water has now taken part in the slow under film corrosion reactions for over 700 hours. All the contaminated surfaces showed more corrosion than seen in Series C. Also blisters were occurring at the higher contamination levels and now some discoloration could be seen, even at the 0.25 UG Cl^- contamination levels.

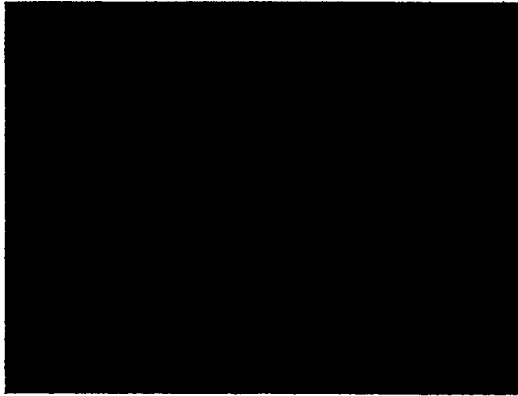
E. EFFECTS OF LONG TERM SUBMERSION (LATE GROWTH STAGE)

The term late growth stage, is only a relative one. These tests were not intentionally run in a very accelerated manner. Therefore our 4500 hour test (longest test run) may still be only an early test by practical service life standards. In fact, ballast tank coatings are expected to last about 100,000 hours, under conditions only slightly less severe than the testing procedure used. It is estimated that the submerged pressure test accelerated the under film corrosion reactions by a factor of 3 times due to the 50 PSIG air pressure used.

At the end of 4500 hours of testing the following was observed:

- a.) Uncontaminated steel areas were still white metal.
- b.) The lowest chloride contamination test level was $0.25 \text{ug Cl}^-/\text{cm}^2$ ($0.014 \text{oz NaCl}/1000 \text{Ft}^2$). Very small black (anode) spots could be observed. The percent surface area covered by micro-anodes was about 5%.
- c.) A doubling of the amount of chloride contamination to $0.5 \text{ug Cl}^-/\text{cm}^2$ ($0.027 \text{oz NaCl}/1000 \text{Ft}^2$, caused about double the amount of black (anode) area, so that about 10% of the surface was covered with micro-anodes.
- d.) Another doubling of the chloride contamination to $1 \text{ug Cl}^-/\text{cm}^2$ ($0.054 \text{oz NaCl}/1000 \text{Ft}^2$) again caused approximately double the amount of black area, and about 20% of the surface was covered with micro-anodes.
- e.) Another doubling of the chloride contamination to $2 \text{ug Cl}^-/\text{cm}^2$ ($0.11 \text{oz NaCl}/1000 \text{Ft}^2$) increased the volume of under film corrosion noticeably, but the area covered by micro-anodes did not quite double (only about 33%). It must be remembered that the anode area and the cathode area must coexist for under film pitting corrosion to continue. This slowing down of the growth of the anode area, indicates that the anode to cathode area ratio is reaching limits. The cathodic oxygen reduction reactions usually need a larger surface area than the anodic area they are receiving electrons from.

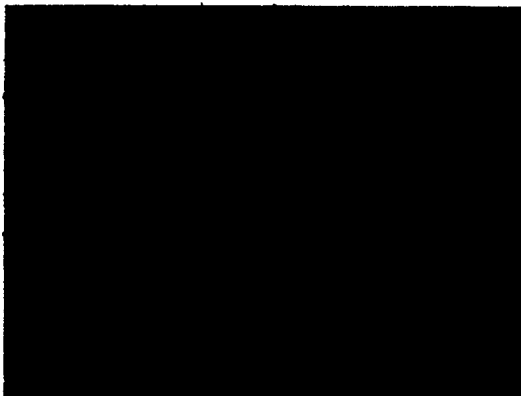
PHOTO SERIES E (part 1)
EFFECTS OF CHLORIDE CONTAMINATION
UNDER CLEAR EPOXY COATING AFTER
4500 HOURS OF SUBMERGED TESTING



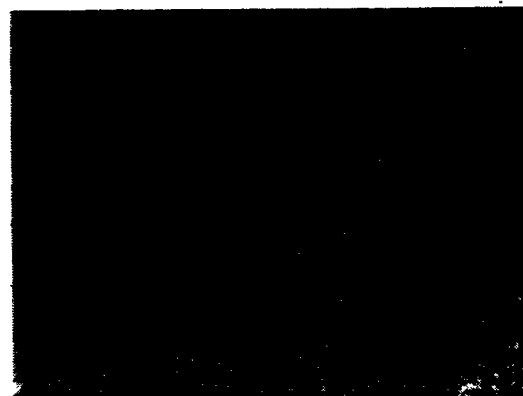
0.25 UG. Cl^-/CM^2



0.5 UG. Cl^-/CM^2



1 UG. Cl^-/CM^2



2 UG. Cl^-/CM^2

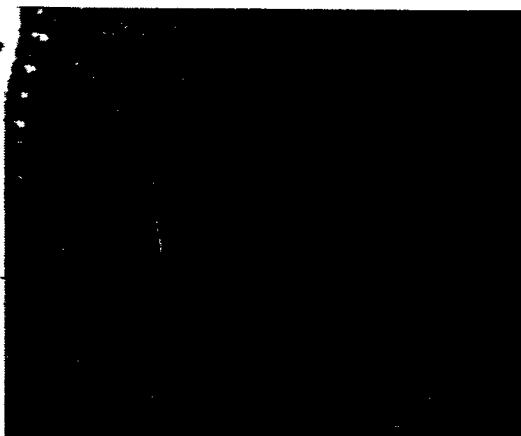
PHOTO SERIES E(parts 1 & 2)- show the corrosion effects that increasing levels of chloride caused under clear epoxy, after 4500 hours of submergence in sea water at 90°F (33°C) at 50 psig (3.3 BAR). At the time of coating application there was no discoloration on panels contaminated with less than 1UG Cl^-/CM^2 . Above this level there was very slight discoloration which darkened noticeably during the air curing period. This brownish under film corrosion turned black when the panel was submerged. This transformation indicated that water had reached the contaminated substrate. This water has now taken part in the slow under film corrosion reactions for over 4400 hours. All the contaminated surfaces showed much more corrosion than seen in Series D, many blisters were occurring at contamination levels above 8-16 UG. Cl^-/CM^2 . Some black discoloration could clearly be seen at the 0.25 UG Cl^- contamination levels, but, NO UNDER FILM CORROSION WAS OCCURRING IN WAY OF CONTAMINATION FREE AREAS EVEN AFTER 4500 HOURS SUBMERGENCE.

- f.) Another doubling of the chloride contamination to $4\mu\text{g Cl}^-/\text{cm}^2$ ($0.22\text{oz NaCl}/1000\text{Ft}^2$) increased the volume of under film corrosion to an extent where it slightly roughened the surface. The anode cathode area was now about 1/1 with about 50% of the surface covered by black corrosion reaction by-products. No osmotic blisters were formed at this level.
- g.) Another doubling of the chloride contamination to $8\mu\text{g Cl}^-/\text{cm}^2$ ($0.44\text{oz NaCl}/1000\text{Ft}^2$) increased the volume of under film corrosion to an extent where it slightly roughened the surface. The anode cathode area ratio was still about 1/1 with about 50% of the surface covered by black corrosion reaction by-products. A few osmotic blisters were formed at this level. Where they occurred, there was indications of corrosion cell coalescing.
- h.) Another doubling of the chloride contamination to $16\mu\text{g Cl}^-/\text{cm}^2$ ($.88\text{oz NaCl}/1000\text{Ft}^2$) increased the volume of under film corrosion to an extent where it noticeably roughened the surface. The anode cathode area ratio was still about 1/1 with about 50% of the surface covered by black corrosion reaction by-products, in areas where there was no osmotic blistering occurring. However, in areas where osmotic blisters had formed there was coalescing of the corrosion by-products from numerous micro-corrosion cells into a larger osmotic blisters.
- i.) Another doubling of the chloride contamination to $32\mu\text{g Cl}^-/\text{cm}^2$ ($1.8\text{oz NaCl}/1000\text{Ft}^2$) reduced the area of active under film corrosion to a few small areas. In most areas, there were large scattered osmotic blisters that had developed from the coalesced micro-blisters. The original micro-corrosion pattern could be seen under the osmotic blisters, but it appeared to be inactive. The micro-anodes surface color had changed from black to a rust brown. The whiter cathode areas also were still visible under the osmotic blister.

PHOTO SERIES E (part 2)
EFFECTS OF CHLORIDE CONTAMINATION
UNDER CLEAR EPOXY COATING AFTER
4500 HOURS OF SUBMERGED TESTING



4 UG. Cl^-/CM^2



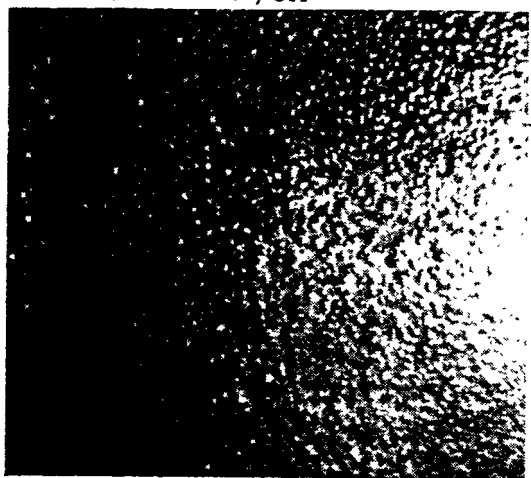
8 UG. Cl^-/CM^2



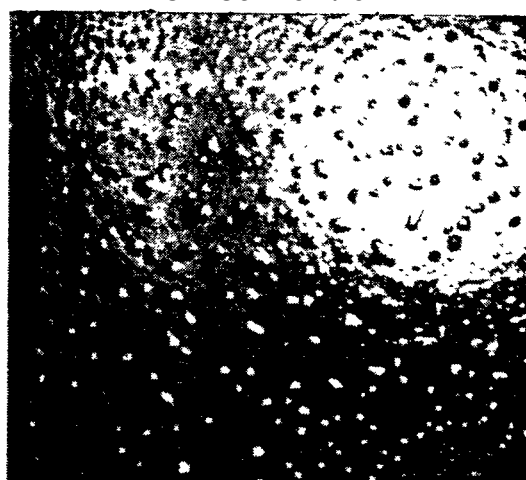
16UG. Cl^-/CM^2



32 UG. Cl^-/CM^2



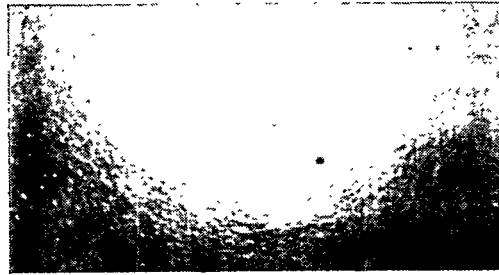
16UG. Cl^-/CM^2



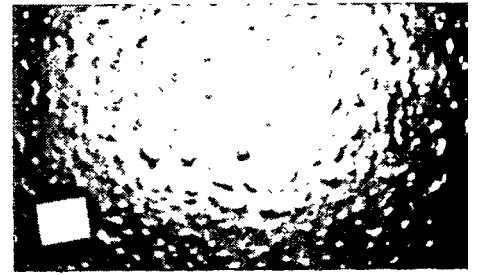
32 UG. Cl^-/CM^2

NOTE:(oblique lighting used to highlight the blisters)

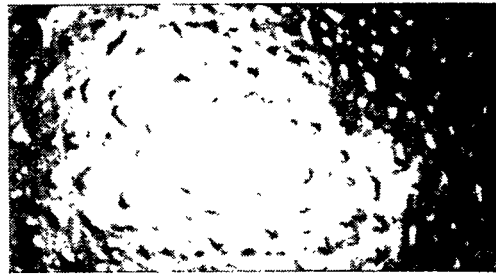
PHOTO SERIES F
OSMOTIC SURFACE BLISTERING FROM CHLORIDE
CONTAMINATION UNDER CLEAR EPOXY COATING



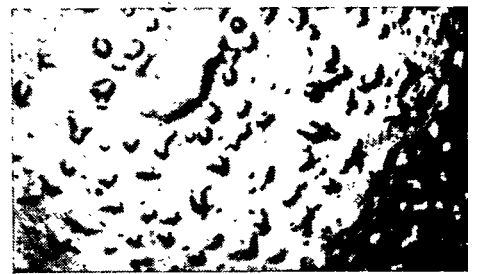
8 UG. Cl^-/CM^2



16 UG. Cl^-/CM^2



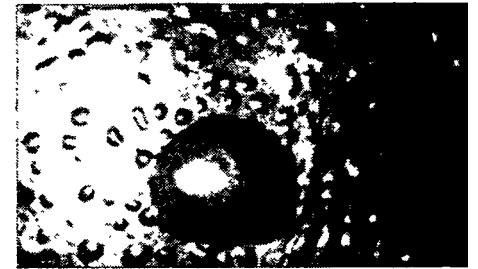
32 UG. Cl^-/CM^2



64 UG. Cl^-/CM^2



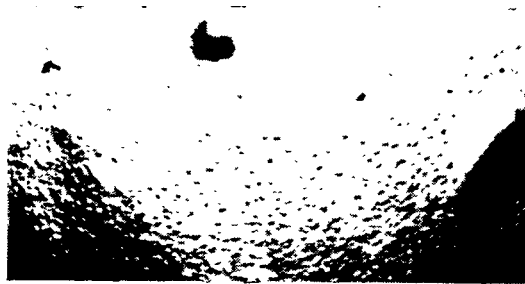
125 UG. Cl^-/CM^2



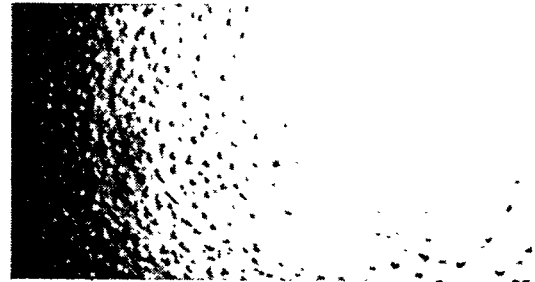
500 UG. Cl^-/CM^2

PHOTO SERIES F- Shows the blistering effects that increasing levels of chloride (sea salt), caused under clear epoxy after 1900 hours submergence in sea water at 90°F (33°C) at 50psig. (3.3 BAR). At the time of coating application there was slight surface discoloration in way of the contamination. This darkened during the curing period and turned black soon after the panels were submerged. This color change indicated that water had reached the contaminated substrate. Slow under film corrosion reactions and osmotic blistering could now take place. These pictures were taken with oblique lighting to highlight the surface changes, and not the substrate corrosion. At the lowest contamination level (8 UG. Cl^-/CM^2) shown, there is only slight surface roughening and no ASTM size 8 blisters. We have rated this visible roughening as a ASTM size 9 blister, however, this is not an official ASTM size. The extent of under film corrosion, that can be seen under clear epoxy with ASTM size 9 blisters is about 100%. It is very important to recognize that the same ASTM size 9 blisters also occur at this contamination levels under coal tar epoxy and at slightly higher levels under Sovapon or Mare Island epoxy.

PHOTO SERIES G
OSMOTIC SURFACE BLISTERING FROM SODIUM CHLORIDE
CONTAMINATION UNDER COAL TAR EPOXY



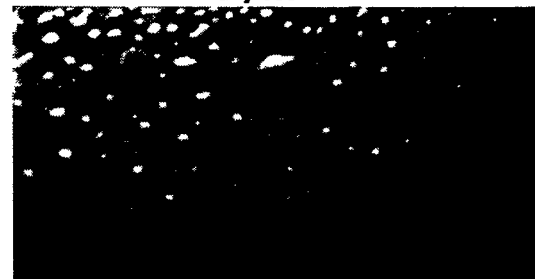
5 UG. Cl^-/CM^2



10 UG. Cl^-/CM^2



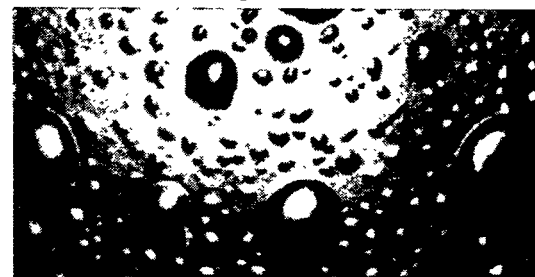
32 UG. Cl^-/CM^2



64 UG. Cl^-/CM^2



125 UG. Cl^-/CM^2



250 UG. Cl^-/CM^2

PHOTO SERIES G- Shows the blistering effects that increasing levels of chloride (sodium chloride), caused under clear epoxy after 1900 hours submergence in sea water at 90°F (33°C) at 50 psig (3.3 BAR). At the time of coating application there was only slight surface discoloration in way of the contamination. Slow under film corrosion reactions and osmotic blistering occurred in way of this contamination. These pictures were taken with oblique lighting to highlight the surface changes. At the lowest contamination level (5 UG. Cl^-/CM^2) shown, there is only very slight surface roughening and no ASTM size 8 blisters. We have rated this just visible roughening as a ASTM size 9+ blister, however, this is not an official ASTM size. The extent of under film corrosion, that can be seen under clear epoxy with ASTM size 9 blisters is about 100%. It is very important to recognize that the ASTM size 9 blisters occur at similar levels of contamination under coal tar epoxy and at only slightly higher levels under Sovapon or the Mare Island type epoxy.

PHOTO SERIES H
OSMOTIC SURFACE BLISTERING FROM CHLORIDE
(SEA SALT) CONTAMINATION UNDER COAL TAR EPOXY

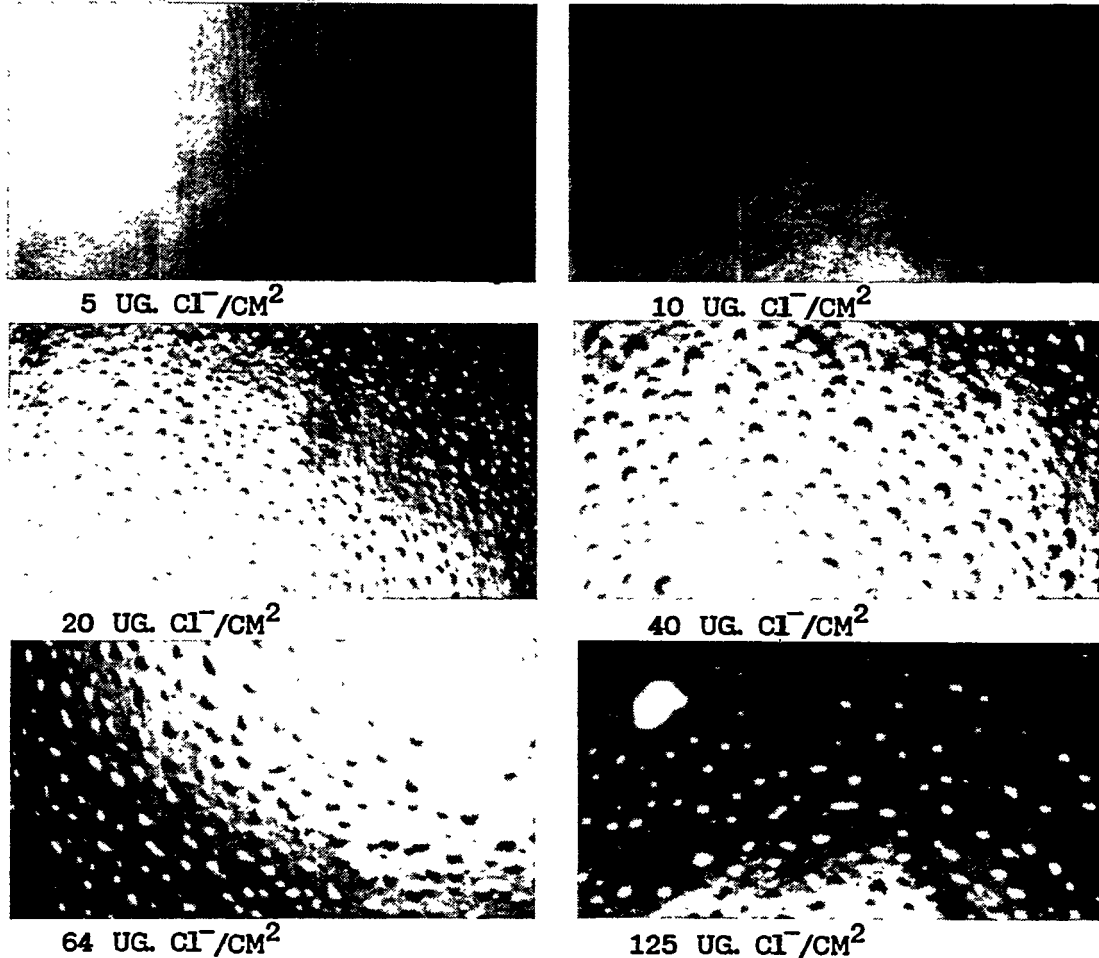


PHOTO SERIES H- Shows the blistering effects that increasing levels of chloride (from sea salt), caused under clear epoxy after 1900 hours submergence in sea water at 90°F (33°C) at 50psig. (3.3 BAR). At the time of coating application there was only slight surface discoloration in way of the contamination. Slow under film corrosion reactions and osmotic blistering occurred in way of this contamination. These pictures were taken with oblique lighting to highlight the surface changes. At the lowest contamination level (5 UG. CL⁻/CM²) shown there was no surface roughening. At the next contamination level shown (10UG. CL⁻/CM²) there was slight surface roughening and no ASTM size 8 blisters, We have rated this just visible roughening as a ASTM size 9+ blister, however, this is not an official ASTM size. The extent of under film corrosion, that can be seen under clear epoxy with ASTM size 9 blisters is about 100%. It is very important to recognize that the ASTM size 9 blisters occur at the same contamination levels under coal tar epoxy and at only slightly higher levels under Sovapon or the Mare Island type epoxy.

9. DISCUSSION OF EXPERIMENTAL FINDINGS:

THE PROCESSES INVOLVED IN COATING BLISTERING:

The 12-14 mil thick clear Aquapon epoxy, blistered the same as coal tar epoxy of similar thickness. By using clear epoxy, over contaminated surfaces, under film reactions and corrosion by-product color changes, could be observed in situ. These under film processes are normally obscured, by conventional opaque coatings. The following coating failure scenario was observed:

9.1 DISCUSSION BACKGROUND NOTES

These 4 background notes are useful, for better understanding the parts that osmosis and electro-chemistry both play in the coating blistering process.

NOTE: For more information, see appendix B, guide-II, sections 2.1.2 (osmosis) & 2.2.(1,2&3) (corrosion & electrochemistry).

1. The surface of steel is not electro-chemically homogeneous. It is composed of dozens of microscopic anodes and cathodes per square inch (6.5 cm²), that are potential micro-corrosion cells.
2. These microscopic anodes and cathodes are instantly activated, into electro-chemical cells, when they are covered with a suitable electrolyte.
3. The presence of a soluble, hygroscopic salt on the surface of steel, can create electrolyte from water submersion or moisture in the air. When this occurs, it activates any micro-corrosion cells it is in contact with, and corrosion occurs. This is the normal cause of rustback, on contaminated steel.
4. The presence of a soluble salt under the coating will also create osmotic diffusion. This diffusion can drive water through the coating and pressurize the underside of the coating and create a blister.

9.2 OBSERVATIONS AND COMMENTS ON COATING BLISTERING:

A.) During the application of chloride contamination, the solutions used for the research caused slight surface rusting. This was due to the fact, that the contamination solution was an electrolyte. Note, no rusting occurred when distilled water or very dilute test solutions were used, since they were not electrolytes.

B.) A distinct corrosion pattern could be seen on the steel panel, after it was contaminated. There were small light brown spots (anode sites), surrounded by larger white metal (cathode sites). The cathode sites were interconnected to each other, and the anode spots were isolated from each other. The anodes spots became larger, at higher levels of surface contamination, but they never exceeded about 50% of the total surface area.

Note: There must always be both anodes and cathode areas present, for corrosion to occur.

C.) Immediately after contamination, the surface corrosion reactions were temporarily stopped. This was done, by storing the panels under very low humidity conditions. This removed the water from the electrolyte solution, and left dry salt on the surface. Dry salt is not an electrolyte by itself, but it can readily turn distilled water or moisture from the air into an electrolyte. Also since dry salt is hygroscopic, it tends to absorb water from the atmosphere, except at low relative humidities. Salt also increases the tendency for osmotic diffusion through the semi-permeable coating when it is submerged. Therefore having soluble salt on a metal surface usually causes active corrosion, unless the humidity is kept very low.

D.) After coating application, the contaminated surfaces absorbed enough moisture from the air (during the curing period) to turn them a darker brown than they were at the time of coating. See upper photograph on page 78.

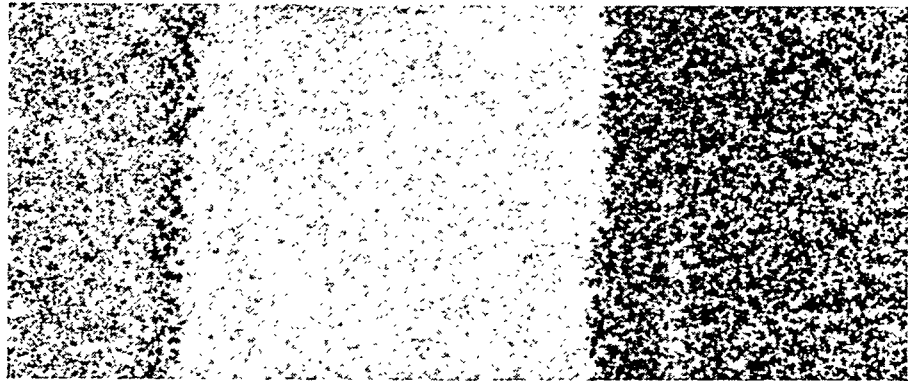
Note, Even a heavy duty coating system, is not a total barrier to moisture or oxygen transmission, as it is a semi-permeable membrane. However, it is a barrier to chloride diffusion. This semi-permeable membrane will therefore protect a clean metal substrate from the chloride in seawater. However if a contaminated surface is coated, this prevents the chloride from leaving the surface, even if it is submerged in distilled water.

E.) Once the coated metal is submerged, there is an induction period of 50-100 hours. This is the time needed for the water to diffuse through the 12-16 mils (300-400 microns) of coating and reach the metal substrate. If there is soluble contamination, such as chlorides, on the metal's surface, they dissolve in this pure water, and create an electrolyte. This activates micro-corrosion cells under the coating. Aqueous under film corrosion then begins. This transition to aqueous corrosion, is indicated by a change in color, of the under film corrosion by-products, from brown to black. Note, the under film corrosion cell pattern does not change, only the color of the corrosion by-products does. See bottom photo page 78.

F.) The aqueous corrosion process goes on relatively slowly. Its reaction rate is controlled by two key factors:

1. The type and amount of contamination present.
2. Diffusion characteristics of the coating.

Another factor that must be considered is time. If the contamination and diffusion factors are low, then the time to failure is usually very long (100,000 HOURS +). However, if either or both of the key factors are high then the time to failure will be much shorter.



CONTAMINATED
16UG. CL-/CM²
(light brown)

NONE CONTAMINATED
WHITE METAL
(silver color)

CONTAMINATED
32UG. CL-/CM²
(darker brown)

Close up photograph of sodium chloride contaminated and non contaminated blasted steel, after coating with clear epoxy and allowing a one weeks time for air cure.
NOTE: Magnification is about 4 times original size.



CONTAMINATED
16UG. CL-/CM²
(black color)

NONE CONTAMINATED
WHITE METAL
(silver color)

CONTAMINATED
32UG. CL-/CM²
(denser black)

Close up photograph of the same sodium chloride contaminated and non contaminated blasted & coated steel shown above, after it has been submerged in sea water at 90° F. (33° C.) at 50 psig. for 500 hours.(4 X magnification)
NOTE: When the chloride contamination levels exceeds about 4UG./CM², then the general corrosion pattern covers 100% of the contaminated surface. However, this pattern is not a smooth even film of corrosion product, but one of many small corrosion cells. These small active cells buildup corrosion products under the coating, and this causes the initial film roughening (S9 size blisters). With chloride contamination, this usually occurs before larger osmotic blisters (liquid filled) of the standard ASTM D714 size, are formed.

6.) As the under film corrosion process continues (TIME), there is a slow build up in the volume of the micro-corrosion cells and their by-products. The higher the initial level of contamination, the greater will be the rate of this buildup.

a) At very low levels of contamination there was no visible surface discoloration during the first 1000 hours and only light discoloration, at the end of 4500 hours.

b) At $1 \mu\text{g Cl}^-/\text{cm}^2$, there was barely visible black surface discoloration after the induction period. After 4500 hours, there was a moderate buildup of micro-corrosion by-products on the metal surface (20% of the surface).

NOTE: A MAXIMUM LEVEL OF 1 TO 1.5 MICROGRAMS OF CHLORIDE PER SQUARE CENTIMETER (.054-.08oz NaCl/1000 SQUARE FEET) IS SUGGESTED FOR HIGH QUALITY COATING APPLICATIONS.

c) At $2 \mu\text{g Cl}^-/\text{cm}^2$, there was visible black surface discoloration after the induction period. By the end of 4500 hours there was considerable micro-corrosion buildup on the metal surface (33% of the surface).

d) At $4 \mu\text{g Cl}^-/\text{cm}^2$, there was a visible amount of black on the surface after the induction period. After 4500 hours, there was heavy micro-corrosion buildup on the surface (50%).

e) At $8 \mu\text{g Cl}^-/\text{cm}^2$ there was a very visible amount of black discoloration after the induction period. At the end of 4500 hours, there was very heavy micro-corrosion buildup, on the metal's surface. This buildup caused slight surface roughening of the coating, and also a few osmotic blisters.

Note: Osmotic blistering was not observed below $8\text{ug Cl}^-/\text{cm}^2$, but coating surface roughening from micro-blisters was observed as low as $4\text{ug Cl}^-/\text{cm}^2$.

f) At $16\text{ug Cl}^-/\text{cm}^2$, there was heavy black discoloration at the end of the induction period. At the end of 4500 hours there was under film corrosion still going on in some areas, while osmotic blistering was occurring in other areas.

g) At $32\text{ug Cl}^-/\text{cm}^2$, there was heavy black discoloration on the metal surface at the end of the induction period. At the end of 4500 hours, almost all micro-corrosion cells, had coalesced into osmotic type blisters.

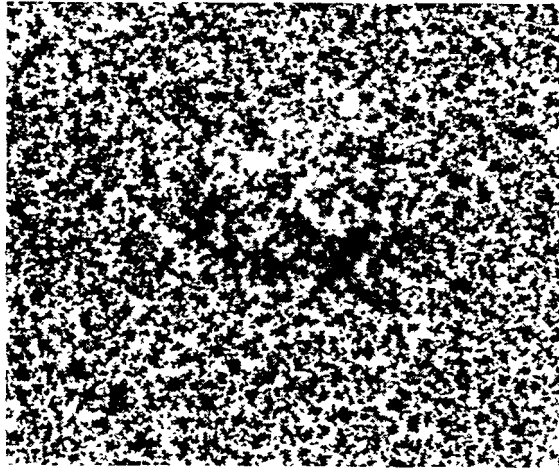
H.) There is a transformation process, from under film micro-cell corrosion into osmotic blistering.

a) The underfilm corrosion first builds up as distinct micro-cells, which can be seen through the clear epoxy. These cells/blisters, build up over each micro-anode.

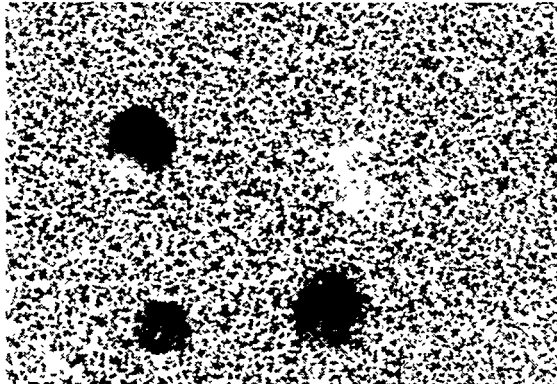
b) The total surface area of the micro anodes, may increase to about 50% of the total surface area, but not much more. The micro anode's area depends on the initial level of contamination and the length of time that corrosion has been in progress. White metal areas (cathodes), are also present surrounding the anode.

c) At a latter stage of their development the volume of the corrosion micro-cells, become large enough to roughen the coating originally smooth surface and become just visible to the eye (micro-blisters).

d) The micro-blisters finally reach a critical size and then rupture the coating's bond, to the metal substrate.



Close up photograph through a clear epoxy coated surface, with a medium size (S4), clear liquid filled blister on it. The steel surface was contaminated with 125 UG. chloride /CM² (sodium chloride used) and exposed for 1900 hours in seawater at 90° F. Note that the general corrosion pattern (darker colors) extends through the blister. However, the corrosion pattern color under the blister is a light brown color, while the rest of the corrosion pattern is black. In between the corrosion are areas that appear to be white metal. This situation is probably due to the many corrosion cells on the surface forming anodic (corrosion product areas) and protected cathodic areas (white metal). Picture magnification is about 3X.



Close up photograph through a clear epoxy coated surface, with several medium size (S6), dark clear blue black liquid filled blisters on it. The steel surface was contaminated with 64 UG. chloride /CM² (sea salt used) and exposed for 1900 hours in seawater at 90° F. Note that the general surface corrosion pattern (darker colors) extends through the blister. The corrosion pattern is black. In between the corrosion are areas that appear to be white metal. picture magnification is about 3X.

NOTE: With sea salt contamination all liquid filled blisters were clear/dark blue. With sodium chloride contamination some blisters were clear and some were the dark type. The cause of this difference was not investigated.

e) The micro-blisters then begin to coalesce into the bigger osmotic type blisters. This coalescing of the micro-blisters, is seen at the areas where the osmotic blisters have formed. In fact the pattern of the original micro-corrosion cells can still be seen under the osmotic blister. See photographs Page 81.

f) The micro-corrosion cell activity on the metal surface stops, once the coating detaches from the metal and starts to form into osmotic type blisters.

These see through epoxy tests, have increased our basic understanding of the negative effects, very small amounts of substrate contamination, have on organic coating systems. The testing found, that osmotic blistering will occur once the contamination exceeds a certain level.

Note: The osmotic blister initiation level, varied slightly with each coating tested. However, these differences may not be as great over a longer testing period.

9.3 THE OBSERVED CHANGES IN THE AMOUNTS OF UNDER FILM
SURFACE CORROSION (ANODE AREA)
AND COATING BLISTERING
FROM VARIOUS LEVELS OF CHLORIDE SURFACE CONTAMINATION.
ALSO, THE RELATIONSHIPS BETWEEN
DIFFERENT SURFACE CONTAMINATION MEASUREMENT SCALES

PERCENT OF METAL SURFACE COVERED WITH UNDER-FILM CORROSION	<THE SAME LEVELS OF CHLORIDE CONTAMINATION > < MEASURED ON 5 DIFFERENT SCALES A to E >				
	A ug.Cl- CM ²	B mg.Cl- M ²	C Us LIMPET	D oz.Cl- 1000FT ²	E oz.NaCl 1000F ²
NO SURFACE 0%CORROSION	0.00	0.00	0.00	0.00	0.00
SURFACE 5% CORROSION	0.25	2.50	1.0	0.008	0.014
SURFACE 10% CORROSION	0.50	5.00	2.0	0.016	0.027
SURFACE 20% CORROSION*	1.0	10.0	4.0	0.033	0.054
*MAXIMUM SUGGESTED CHLORIDE LEVEL FOR GOOD COATING LIFE					
SURFACE 33% CORROSION	2.0	20.0	8.0	0.066	0.11
SURFACE 50% CORROSION	4.0	40.0	16.0	0.13	0.22
50% SURFACE CORROSION TINY BLISTERS	8.0	80.0	32.0	0.26	0.44
SOME SURFACE CORROSION & MEDIUM BLISTERS	16.0	160.0	64.0	0.52	0.88
SCATTERED SURFACE CORROSION & LARGER BLISTERS	30.0	300.0	120.0	1.0	1.65
MEASUREMENT SCALES-A	B	C	D	E	

MEASUREMENT SCALES AND LIMPET CELL READINGS(CHART SCALES A-E)
A-Micrograms (ug) chloride (Cl⁻), per square centimeter(CM²)
OR Grams (g) chloride (Cl⁻) per 100 square meters (100M²)
B-Milligrams (mg) chloride (Cl⁻), per square meter (M²)
C-CONDUCTIVITY IN MICRO-SIEMENS (uS) OF SURFACE TEST SOLUTION
if obtained with 1X sensitivity LIMPET CELL.
D-Ounces (oz) chloride (Cl⁻) per 1000 square feet(1000Ft²)
E-Ounces (oz) Sodium Chloride (NaCl) per 1000 square feet

Note, % surface corrosion refers only to area of anodes.
The cathode area (white metal) will be at least as large
as the anode area, in a micro-corrosion cell.

The above chart shows the relationships observed between
under film corrosion, osmotic blistering and various levels of
sodium chloride contamination, under 12-14 roils of a clear
polyamid epoxy. Testing was done in sea water at 90°F.(350C.)
and 50 PSIG (115Ft.Head). This clear epoxy performed similarly
to standard coal tar epoxy. It can be seen from the chart, that
considerable under film corrosion can occur, without surface
blistering. The underfilm corrosion reactions stopped, when
osmotic blisters formed.

*NOTE: A suggested (preliminary) maximum level for chloride contamination, to insure a good coating system life under saltwater ballast conditions, is between 1 & 2 micrograms per square centimeter. Considerably higher levels may not cause short term blistering but they will increase under film corrosion and probably cause osmotic blistering ultimately.

The chart shows the numerical relationship between several different scales, that surface contamination levels can be described by. The Internationally correct way to describe the contamination level on a surface, is in micrograms of contamination, per square centimeter (see column A). However, it is hard to relate a large structure such as a tank, to these tiny scientific numbers. Therefore, the chart Lists some other possible non-standard scales to describe contamination levels.

*These are shown as columns A through E in the above chart.

Column A (micrograms per square centimeter)(STANDARD INTERNATIONAL SCALE) can also be labeled in an unorthodox way, that is much easier for a engineer to understand. Grams (1/28 of an ounce) of chloride per 100 square meters (about 1076 square feet). This is a number most people can at least picture in their mind. It may help you to picture the contamination problem in grams per 100 square meters, while taking measurements in micrograms per square centimeter, the numbers are the same.

Column B shows the equivalent number scale if contamination levels are measured in milligrams per square meter. In fact this measurement system is used by some researchers. Its use has cause confusion, since this scale reads 10 times higher than the scale for micrograms per square centimeter. This difference occurs, because the square meter is 10,000 square centimeters and the milligram is only 1000 micrograms. This ten to one ratio difference, can easily confused people reading literature that uses it.

Column C shows a theoretical LIMPET CELL'S SOLUTION READING in micro-Siemens, if the surface contamination was pure sodium chloride and the cell retrieved 100% of the chloride present. Note, laboratory experiments by SSPC have found that the various retrieval methods (LIMPET cells and swabbing), do not reclaim all the surface contaminants, and therefore the readings obtained, are always less than 100% of the contamination present. The Limpet cells were found to be more efficient, than swabbing. The Limpet cell readings shown in column C, are based on a cell that tests a surface area (in square centimeters) that is equal to the cell's volume in milliliters, this is considered a 1X type cell. Usually more sensitivity is recommended than 1X.

Limpet cells can be designed to have greater sensitivity, by increasing the surface area being tested and minimizing the amount of distilled water used. Limpet cells with a ratio up to about 8X can be made up. If an 8X cell were used to take the readings in column C, they would be 8 times higher. Higher sensitivity type cells are particularly desirable, when checking that surfaces are below the recommended 1-2 microgram MAXIMUM RECOMMENDED LEVEL of chloride contamination.

Column D shows contamination levels expressed in ounces (lounce=28 grams) of chloride per 1000 square feet (equal to about 93 square meters). While this is an unorthodox way to describe chloride contamination levels, it is much easier for an engineer to understand. You can see from the numbers in column D how careful we must be, to prevent leaving dangerous amounts of chloride contamination on surfaces being coated.

Column E shows contamination levels in ounces (28 grams) of Sodium Chloride per 1000 square feet (about 93 square meters). When investigating contamination it is usually easier to measure specifically for chloride ions and ignore the presence of other ions. However you can't get chloride ions alone. To get one ounce of chloride ions you need 1.65 ounces of salt. The numbers in column E are the same as Column D multiplied by a factor of 1.65.

9.4 GENERAL DISCUSSION:

THE EFFECTS THAT CHLORIDE CONTAMINATION HAS
ON SURFACES, DEPEND ON TWO KEY FACTORS

- 1) Diffusion rate of water and oxygen through the coating.
- 2) Level and type of surface contamination present.

1. Diffusion rate of water and oxygen through the coating.

Many people still believe, that a pinhole free coating prevents water and oxygen from reaching the metal surface. This belief is easy to come by, when one looks at a heavy duty coating system, that has been spark tested with several thousand volts and passed. The fact is, all organic coating systems are semi-permeable to water and many other fluids. They are also semi-permeable to oxygen transmission. Fortunately for the marine industry, they are impermeable to the chloride ion. These moisture and oxygen diffusion rates can vary from coating to coating. These diffusion rates can also vary in the same coating, when different type cargo is carried, or with changes in temperature and pressure..

In addition, osmotic diffusion forces can be induced in a coating when substrate cleanliness is neglected.

Semi-permeability is a basic characteristic of organic coatings that must be understood and planned for.

2. Level of surface contamination.

The level of chloride contamination on the metal has been shown to be a critical factor in the activation of surface micro-corrosion cells. Its presence is required to ionize the deionized water, that is naturally diffusing through the semi-permeable coating, to the metal substrate. If there is no contamination present, the water does not ionize and activate the potential corrosion cells, present on the steel's surface.

The clear epoxy experiments showed how little chloride contamination is need, to start up these micro-corrosion cells.

Under practical working conditions it will not be possible to prevent or remove all contamination, but it is very possible to eliminate most of it. During new construction it is relatively easy to keep contamination levels low. Levels well below $1 \mu\text{g Cl}^-/\text{cm}^2$ should be easy to achieve, by following good house keeping procedures.

The problems come, with getting older steel clean enough, particularly if it is badly corroded. For high performance coating work (ballast and cargo tanks, underwater hull etc.) the goal should be between 1 and $2 \mu\text{g Cl}^-/\text{cm}^2$ maximum and lower if practical.

Some paint manufacturers feel a higher level is ok, based on the fact that it usually takes about $10 \mu\text{g}/\text{cm}^2$ chloride contamination, to produce osmotic type blisters during testing programs. The problem with this approach is that an accelerated test programs usually can not anticipate what will happen with slow under film corrosion reactions, over the expected 100,000 hour working life of a epoxy coating system.

It is usually not too difficult to get contamination well below the $10 \mu\text{g Cl}^-/\text{cm}^2$ level, if good blasting practices are followed. If dry blasting alone can not achieve the proper level of contamination removal, then other, steps such as pre-blasting and fresh water washing may also be required.

TIME IN SERVICE

The experiments clearly showed, that with time there is a steady deterioration in a coating system. This is particularly true if there is contamination and active under film corrosion occurring. To achieve the desired 100,000+ hour service life, the final contamination level must be considerably lower than, a level that will cause blistering within a few thousand hours of testing. It should also be recognized, that the contamination salts are not rapidly consumed during the corrosion processes. The chloride and sulphate ions recycle and therefore very small amounts can keep the corrosion process going on for a very long time. This slow corrosion process creates more and more pressure under the paint and ultimately leads to its failure.

9.4 OBSERVATIONS WITH SULPHATE CONTAMINATION:

Only a limited number of tests were done with Ferrous Sulphate under clear epoxy. This contaminant requires about 10 times the level to induce blistering as the chloride ion does. In fact, to reach this level the panels became so discolored with ferrous sulphate that no inspector would pass the surface. The clear tests showed some differences and similarities between this contaminant and chloride.

- 1) Ferrous sulphate discolors the steel surface much more than sodium chloride *or* sea salt at all the levels of contamination tested.
- 2) The anode and cathode areas are not activated by this contaminant. The surface color changes are uniform at the time of contamination (like a coat of paint).
- 3) Similar to chloride contamination, the metal surface darkens slightly during the air curing period. This indicates some reactions are occurring, under the coating.
- 4) Similar to chloride there was an induction period of less than 100 hours for the moisture to diffuse through the 12 mil (300 micron) film and cause a large substrate color change.
- 5) The color change after induction was even dark grey to black, depending on the level of contamination. The higher levels of contamination caused the black color.
- 6) There we no indication of formation of anode cathode areas forming. This salt may passivate the surface.

NOTE: Without the mechanism of micro-corrosion cells to help buildup micro-blisters, only osmotic forces are available to create blistering. This may account in part for the very much higher levels of sulphate needed to induce blistering compared to chloride.

9.5 CONCLUSION:

The 4500 hour testing program found, that extensive under film corrosion from chloride contamination, can occur well before any osmotic blistering is observed in the coatings surface. In fact, the first signs of under film micro-cell corrosion, occurred at a contamination level that was only about 1/50 of that required to cause osmotic size blistering. When the contamination level was about 1/5 the level required to cause osmotic blistering, almost 25% of the substrate was covered with under film corrosion products. These under film corrosion reactions started after less than 100 hours of submerged testing.

The corrosion testing method used, was not unusually aggressive. 4500 hours of testing at 90°F and 50 pounds per square inch air pressure is probably equivalent to 2 years in normal ballast service. This 2 year estimate is based on the fact that the oxygen diffusion through the coating was increased by a factor of 3, by the air pressure used during the testing program.

One of the most common questions asked about coatings is how long will they last? This is a very difficult question, to get an answer for. The typical estimate of about 10 years effective life (88,000 hours), would be a rational number when discussing heavy duty ballast tank coatings. However, usually all the coating within a given tank, does not last the same amount of time. Coating failure usually occurs in three stages.

First: there is usually some very minor breakdown within the first 6 months to one year. This is normally due to human error. This type failure can be minimized by good inspection. However, considering the complexity of coating a tank, some minor faults may still occur, even with good inspections.

If the epoxy coating system is sound at the end of the first year, it is usually good for at least 4 or 5 more years of ballast service (40-50,000 hours).

Second: At the end of the first five year period, a few morw weak spots may show up, even if the coating was properly applied. Overall, the system should still be in very good condition at this time. However, if the coating was improperly applied, serious breakdown may be occurring and total replacement may be warranted. Poor surface preparation, can be a major cause of this type of premature coating failure.

If the coating is still in good condition at the first five year stage, then it should last another 5 years. At the end of ten years, even well applied epoxy systems start showing their age and a more general coating breakdown can be expected.

NOTE: AT THIS TIME IT IS NOT UNCOMMON TO FIND CONSIDERABLE UNDER FILM CORROSION HAS OCCURRED IN SOME AREAS, WHILE OTHER SIMILAR AREAS ARE STILL TIGHT AND FREE FROM UNDER FILM CORROSION. ONE OF THE MOST PLAUSIBLE EXPLANATIONS FOR THIS DIFFERENCE IN PERFORMANCE, FROM THE SAME COATING SYSTEM, USED UNDER THE SAME SERVICE CONDITIONS; IS THAT THE LEVEL OF SURFACE CONTAMINATION AT THE TIME OF COATING APPLICATION WAS DIFFERENT AT EACH AREA.

TO GET THE MAXIMUM SERVICE LIFE OUT OF A COATING SYSTEM IT IS IMPORTANT TO GET THE SURFACE AS CLEAN AND FREE OF SOLUBLE CONTAMINATION, AS IS PRACTICAL.

If a ten year PLUS service life is desired from a coating system, it is important to pay attention to surface contamination levels. If in the future a practical organic coating system can be developed, that has no permeability to water or oxygen, then the importance of good contamination removal may become past history. Note, the success of an organic coating to work over soluble contamination is doubtful, with anything less than zero permeability.

Until an impermeable coating is developed and proven, our best approach will be to do careful surface preparation.

9.5 GETTING GOOD QUALITY SURFACE PREPARATION

The practical goal of the present research project is not to make surface preparation more complicated, but to help the coating industry recognize and correct some common practices that are detrimental to good coating practice.

It should be stressed, that in most cases it is not that difficult to achieve the low levels of contamination needed to prevent serious coating problems. The fact is, that the majority of the coating work done in the past, has been relatively successful. This success is due to many factors, including the fact that the amount of contamination left on the surface had not been too high.

BLAST ACCORDING TO SURFACE PREPARATION WORK NEEDED:

In order to achieve a good surface preparation standard, the blasting work effort must be adjusted to correct the existing surface contamination problems. These problems vary greatly, according to the initial surface conditions such as:

a. Clean new steel or lightly corroded new steel.

This type surface is easy to dry blast clean. Usually there are no surface contamination problems after completion of mineral grit blasting. Steel grit can also be used if the surfaces to be blasted are free of oil and salt deposits. Steel grit or shot should never be used if oil or salt deposits are present, as they can be contaminated easily and then contaminate other areas. Always wash down and degrease contaminated surfaces before blasting.

b. Well coated old steel with limited coating breakdown.

This type surface is easy to dry blast properly. However, extra attention should be given to areas of coating breakdown and corrosion, to help bring them up to the same cleanliness standard as the smooth areas. This extra attention is needed, to get uniform coating performance.

c. Coated old steel with: considerable coating breakdown, under film corrosion, active corrosion and some scale covered pitting corrosion.

This type of surface is more difficult to dry blast free of all contamination, but it can usually be done to a satisfactory level, if the work is approached properly.

The first three conditions will usually respond well to dry blasting as long as the surface is relatively smooth and free from pitting. The pitted areas present the greatest challenge to obtaining good surface preparation.

It is difficult to get the grit particles into the bottom of pits. The blasters must be trained to work extra hard on rough or pitted areas. They must not move on as soon as it seems clean, but instead they must blast a little longer before continuing on. If they follow this procedure in the rough and pitted areas, much more of the surface will be properly cleaned on the first try. In spite of this extra effort, some pitted areas will still turn while the rest of the tank is being blasted. The most common approach to this re-rust problem, is a quick sand sweeping to brighten up the turned surfaces. This approach is totally wrong, as it only temporarily hides the surface contamination problem, without correcting it. It must be recognized, that if blasted areas turn, they are still badly contaminated. They need to be hard blasted, to remove the deeply imbedded corrosion products that are laden with chloride and sulphate contamination. The grit used for cleaning pitted steel should be a mix of fine and medium coarse size grit. The fine grit is needed to get into the bottoms of the pits and remove any contamination trapped there.

d. Old steel, that is badly pitted and heavily scaled.

This type of surface may not respond well to dry blasting alone. It may require pre-blasting, freshwater washing and then re-blasting before a properly cleaned surface is obtained. See GUIDE IV.

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APPENDIX: A EXPERIMENTAL DESIGN AND TESTING PROCEDURES

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APPENDIX A EXPERIMENTAL DESIGN AND TESTING PROCEDURES

A1 DESCRIPTION OF TEST PANELS:

The 14 GAUGE steel test panels were cut from A 37 grade hot rolled mild steel. The panels were cut by shearing. Two sizes were cut 6"X 12" and 6"x6".

A2 PRE-CLEANING PROCEDURES:

Prior to any grit blasting, the panels were cleaned of all oil and soil, by water washing with laboratory detergent, rinsing with fresh water and then drying. All the panels were then cleaned free of slight rust and corrosion by disc sanding and at the same time, all edges were ground smooth, to improve painting them. In order to remove all water soluble contaminants and other loose dirt picked up during disc sanding, the panels were rewashed with water and laboratory glassware detergent. They were then rinsed in fresh water and forced dried with hot air before any rusting could occur. The panels were finally dip rinsed in two acetone baths to remove any other soluble deposits. At this point the panels were packed in brown paper in preparation for transportation to be blasted. These pre cleaned panels remained in these brown paper packages about 6 months. When the packages were opened, they were found to be still free of any corrosion.

A3 BLAST CLEANING PROCEDURES:

The pre-cleaned hot rolled steel panels were blasted to SSPC SP5, white metal using new steel grit. Note prior to being blasted, most of the panels still had over 95% of their surface covered with intact mill scale. The blasted panels had a final surface profile of about 2 mils. During the blasting process the new steel grit was recycled. These ultra clean steel surfaces have shown no tendency to rust, even when exposed to very high relative humidity conditions (85-100% non-condensing) for months. This freedom from rusting, even under long term high humidity conditions, demonstrates the panels freedom from ionic surface contamination. However, when small amounts of contamination i.e. $\mu\text{g Chloride}/\text{CM}^2$ ($0.0540\text{z.NaCl}/1000\text{Ft}^2$.) are placed on these blasted surfaces rusting will rapidly occur at 75% relative humidity.

NOTE: The panels were handled with cotton gloves after blasting, to prevent chloride pick up from the hands.

A4 PANEL STORAGE AFTER BLAST CLEANING:

The ultra clean panels were individually wrapped in brown paper and then stored in sealed 5 gallon pails." Each pail held 20 panels, and also had a package of silica gel in it to insure dryness. No vapor phase inhibitor was used to protect these panels. A recent check of panels found them to be still in rust free condition after several years storage.

A5.1 CONTAMINANTS USED FOR TESTS:

Three types of chemical contamination was used during the testing work. Analar sodium chloride and a ASTM grade synthetic sea water salt mixture were used for the tests involving chloride ion levels. Analar ferrous sulphate was used during the sulphate tests. In addition to testing the effects of chemical, tests were also run using different types and sizes of blasting grit particles as sources of surface contamination.

A5.2 MIXING OF CHEMICAL CONTAMINATION SOLUTIONS:

The chemical contamination solutions used during the experimental work were made up by mixing a measured weight of the contaminant (± 0.01 gram) in 0.5 liter of distilled water. This initial base solution was then divided in half, and one half was then diluted with an equal quantity of distilled water to reach the next desired solution strength. This dilution process was repeated until the lowest required level of solution concentration was reached.

A5.3 PANEL CONTAMINATION PROCEDURES:

The panels were contaminated using water solutions of either sodium chloride, synthetic sea water or ferrous sulphate. The appropriate solution concentration was chosen to obtain the desired surface contamination levels. The contaminant was applied by evenly spreading 0.25 milliliter of solution over 150 cm.² (25 inch²) of surface area, and then drying the surface with a hot air blower for about 15 seconds. It was observed that, except when using distilled water or very dilute chloride solutions, the surface becomes slightly discolored. In an attempt to eliminate this surface discoloration, a number of samples were made up using only methanol to dissolve the sodium chloride. This method did eliminated the immediate surface turning, occurring from water/sodium chloride solutions. However, after 24 hours of air exposure at 85% relative humidity, the panels contaminated by solutions of sodium chloride in methanol, developed more rust than the panels contaminated at similar chloride levels in water solutions.

Note, the application of pure methanol over an area previously contaminated by a water/chloride mixture, also appears to increase the intensity of rusting. The use of methanol instead of water to distribute chloride over a surface, appears to make the chloride contamination more active. This increased activity, may be due to the fact that the methanol solutions wetted the steel surface better than water, and thereby gets the chlorides deeper into the surface roughness profile. Also the salt crystals deposited by methanol evaporation, appear to be smaller and more evenly distributed, than crystals deposited from water solutions.

A5.4 GRIT CONTAMINANTS USED FOR TESTS:

In addition to the chemical contamination tests, other tests were run to check what effect incomplete removal of blasting grit from blasted surfaces, would have on paint films sprayed over them. Three coal slag grits and one copper slag grit were used for contaminants. Each grit was sieved to obtain four different mesh sizes (+20, +40, +80 & -80). The four test coatings were painted over controlled amounts of each type and size grit, to see how they would be affected by this type of contamination.

A5.5 STORING OF CONTAMINATED PANELS PRIOR TO COATING THEM:

The chemically contaminated panels were held in an air convection oven at 130° F (55°C), until just a few minutes before paint application. The oven prevented the panels from having visible surface changes, for several days. On the other hand unheated contaminated panel surfaces quickly deteriorate due to rust back.

This heating procedure is very analogous to the standard practice of dehumidification, normally used to prevent the turning of blasted steel in tanks. The panels inside the convection oven *were* about 65F^u above the air's dew point. This is equivalent to about 15% relative humidity. IT IS IMPORTANT TO STRESS, THAT THE USE OF DEHUMIDIFICATION TO PREVENT SURFACE TURNING, INDICATES A MISUNDERSTANDING OF THE SURFACE CONTAMINATION PROBLEM. Dehumidification only temporarily stops the contamination from reacting with the surface. Under film corrosion starts up soon after the tank is put **back in service, wherever contamination** is present.

A6.1 TYPES OF PAINT USED DURING TESTING -

Four types of two component epoxy coatings were used for the test program:

1. Coal tar epoxy, a 2 coat system from Valspar.
2. Sovapon epoxy, a 2 coat system from Valspar.
3. Mare Island epoxy, this is a standard government 3 coat epoxy system, it was made by Devoe Paint.
4. Aquapon polyamide clear epoxy, Pittsburgh Paints.

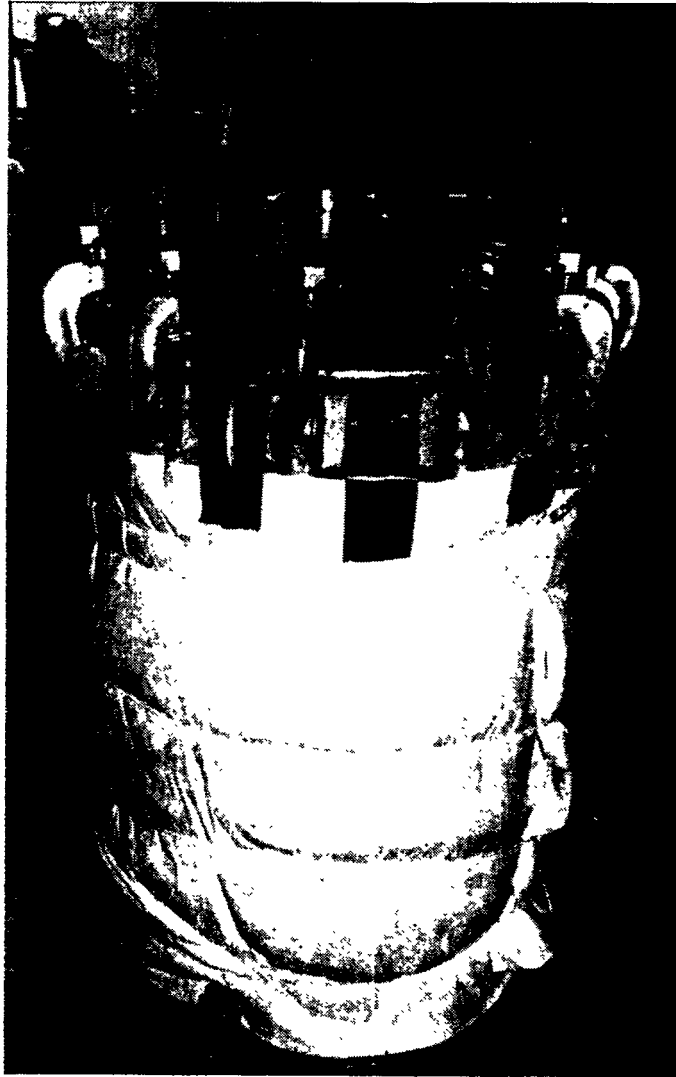
A6.2 PAINTING PROCEDURES -

The epoxy paints were applied by airless spray. No solvents were added to thin the paint. The panels were sprayed one at a time, and then hung to dry, between coats. Proper inter coat timing was followed based on the temperature conditions occurring during drying. After the final coat was applied, the system was allowed to cure for at least one week, before any submerged testing was done.

A7 DESCRIPTION OF TEST TANK -

The testing work was done in a modified 60 gallon pressure paint pot, manufactured by Devilbus. This pot can be pressurized up to 110 psi. The galvanized inside of the tank was coated with coal tar epoxy. The test racks for holding the painted panels were made of 1 inch thick plexiglas. The rack is secured to the tank's removable top cover. The cover and the test rack are raised and lowered by a 1/2 ton chain fall. The test rack holds up to 140 12 X 6 inch panels. The temperature of the tank can be raised above ambient by the 1600 watt electric heating band located on the outside of the tank. The heater can raise and hold the tank's temperature up to 100 degrees Fahrenheit above ambient. The tank and heater are covered with a fiberglass blanket to reduce heat losses.

PRESSURE TESTING TANK



VIEW OF CLOSED PRESSURE TESTING TANK

The tank is a modified 60 gallon Devilbus paint pot. It has been coated inside with coal tar epoxy. It has a 1600 watt band heater on the outside that permits heating to about 100° F (45° C) above the ambient temperature. Tests were run at 90° F (33° C) and 50 PSIG (3.3 Bar) (equivalent to about a 115 ft head of water). Compressed air was used to pressurize the tank.

PANEL TEST RACK
& SUB-TANK



VIEW OF TANK COVER FITTED WITH TEST RACK & SUB-TANK

The test rack is permanently attached to the cover of the tank via 4 support rods and a support plate. It consists of three 1 inch thick plexiglas plates that have been slotted to hold up to 144 - 12 x 6" test panels. The test setup can also hold less panels and a plexiglass sub-tank, 'which can be seen on the upper test rack. A different fluid is put into the sub-tank. This arrangement permits simultaneous testing of panels in two media such as salt water in the main tank and distilled water in the sub-tank. The sub-tank is partly submerged in the main tank's test liquid to keep temperatures constant.

A8 TESTING PROCEDURE

Panels were tested in the pressure tank after they were fully cured. The tank was filled with ASTM grade synthetic sea water. The testing conditions used for most tests was 90°F (35°C) and 50 PSIG (3.3 BAR) (115 foot head). This testing procedure was chosen, as it closely simulates real ballast tank conditions. It was not desirable to use accelerated type testing procedures for the present study, as the coating is already under stress from surface contamination.

To run a test, new test panels were inserted into the test rack. The temperature of the tanks salt water was checked and adjusted if necessary. When the water conditions were at the proper temperature, the tank's cover and test rack unit were lowered into the tank and the tanks closure clamps were secured. Air pressure was admitted to the tank via a pressure regulator, to build up the pressure inside the tank to 50 PSIG (3.3 BAR) (115 ft. water head). The tank was kept closed and pressurized for 24 hours periods at the start of a test. At the end of 24 hours the tank was opened and the panels were examined for signs of blistering, and in the case of clear coatings also for signs of under film corrosion. After inspection, the tank was closed and left pressurized for another 24-36 hours. A second examination was then made, to look for any new changes. After this inspection the tank was again closed and pressurized. A third inspection was made after several more days of submersion to check for additional changes. Several more inspections were made as the test continued until about 2000 hours of testing was completed. Because of the large capacity of the tank some panels were left in the tank for as much as 4500 hours while later test series were also being run.

A9 ASTM D714 METHOD FOR DETERMINING DEGREES OF BLISTERING
IN PAINTS.

Note: This visual ASTM evaluation method is not precise.
Judging the amount of blistering in a paint system is very
difficult. Different inspectors will perceive each level of
blistering slightly differently. Also, the ASTM method
measures only two factors: blister size and blister
frequency. The ASTM blister scale does not account for
the very important substrate corrosion reactions that occur
before and during blister formation.

ASTM PAINT BLISTER SIZES:

ASTM SIZE 10- NO CHANGES IN SURFACE TEXTURE OBSERVED.

*SEE NOTE 1 (SIZE 9) surface roughening (not an ASTM SIZE) .

ASTM SIZE 8- BLISTERS ARE PINPOINT SIZE.

ASTM SIZE 6- UP TO 1/16 INCH BLISTERS

ASTM SIZE 4- BETWEEN 1/16 & 1/8 INCH DIAMETER BLISTERS

ASTM SIZE 2- 3/8 INCH OR LARGER SIZE BLISTERS

ASTM PAINT BLISTER FREQUENCY NUMBERS:

ASTM FREQUENCY NUMBER 10- NO BLISTERS OBSERVED.

ASTM FREQUENCY NUMBER 8- FEW BLISTERS

(one or a few blisters per unit area)

ASTM FREQUENCY NUMBER 6- MEDIUM BLISTERS

(several blisters per unit area)

ASTM FREQUENCY NUMBER 4- MEDIUM DENSE BLISTERING

(many blisters but with some flat areas between them)

ASTM FREQUENCY NUMBER 2- DENSE BLISTERING

(continuously blistered over the entire surface)

Note 1: In the present research we also used some non ASTM
blister size numbers. Size 9 was used to represent
the roughening of a coating's surface by under film
corrosion, before the formation of ASTM size osmotic
blisters.

A10-SSPC CONVERSION OF ASTM D714 BLISTER RATING METHOD:

This chart was developed by the STEEL STRUCTURES PAINTING COUNCIL. It helps to simplify the double numbers of the ASTM blister rating method (BLISTER SIZE AND BLISTER FREQUENCY) into an easier to use single number scale. An ASTM or SSPC rating of 10 indicates there are no blisters.

A rating below 7 on the SSPC scale is usually considered failure, but this choice is arbitrary. The acceptable level of failure should depend more on the intended type of service the coating system is to be used in.

Note: This SSPC conversion method still does not address the fundamental limitations of the ASTM blister rating system. A better method is needed to properly study coating system failure mechanisms when under film corrosion is also occurring.

ASTM	<--ASTM D714 FREQUENCY SCALE-->			
D714	FEW	MEDIUM	MEDIUM	DENSE
BLISTER			DENSE	
SIZE	(F8)	(F6)	(F4)	(F2)
<u>SSPC CONVERSION NUMBERS FOR ASTM D714</u>				
S9	9	9	8	7
S8	9	8	7	6
S6	7	6	5	4
S4	5	4	3	2
S2 "	3	2	1	0

NOTE: THE FOLLOWING PATTERNS ARE USED TO CONVERT THE ASTM D714 MULTIPLE BLISTER RATINGS TO A SINGLE SSPC NUMBER.

1. IF THERE ARE TWO SIZES OF BLISTERS WITH SIMILAR FREQUENCIES THE AVERAGE RATING BETWEEN THE TWO IS USED.
2. IF THERE ARE DIFFERENT SIZES AND VERY DIFFERENT FREQUENCIES OF BLISTERS ON A PANEL, THE SITUATION THAT IS THE WORST, IS REPORTED.

APPENDIX B- FOUR GUIDES FOR BETTER UNDERSTANDING AND CORRECTING THE SURFACE CONTAMINATION PROBLEM

AN OVERVIEW OF APPENDIX B GUIDES:

The use of paint films to protect steel from the marine environment has a long history. In the early days surface preparation was usually very simple; just scrape, wire brush or grind off most rust and scale. Possibly, if the surface was initially very dirty, it may have also been washed off by hose (hopefully with fresh water). These limited types of surface preparation, were usually adequate for the more contamination tolerant oil base paints, used in the past. Also, the life expectancy of these earlier paints was short by today's standards. However, with the advent of larger marine structures and better coating systems, it has become very important, to improve both the productivity and the cleaning effectiveness of the surface preparation methods used by the modern coatings industry.

GUIDE I- is a general discussion of the three primary reasons for coating failures. These are; improper surface preparation, improper application procedures and choosing an improper type of coating system for the service environment in which it is to be used.

GUIDE II- Lists the main environments coatings are used in, and the types of contamination present, that can adversely affect coatings. The different physical and corrosion effects that the soluble contaminants can cause, are then reviewed.

Presently, the three most widely used surface preparation standards (Swedish, SSPC & NACE) all fail to address the fact, that nearly invisible surface contamination can be a potential cause of coating failure problems. It is even more surprising that at this time only one major paint manufacturer protects their products reputation and performance, by always checking that the surface chloride level is below the maximum allowed.

Also, in the past few years, the Steel Structures Painting Council, has been publishing more and more articles on the importance of properly removing substrate contamination, to help stimulate the paint industry into much more awareness of the problem. However, at the present time the painting industry is only just starting to look into the surface contamination problem.

During the next few years, it should become routine for coating inspectors to continuously take grit and surface contamination measurements during a coating project. However, to do this easier, we need to develop simple to use testing methods. We also need to standardize the methods used for detecting and measuring grit and surface contamination.

GUIDE III- Discusses most of the Presently used contamination detection and measuring methods. The purpose of this guide is to tell you what methods are available and where you can get more information on various pieces of equipment to do the testing.

GUIDE IV- Is a discussion on modern surface preparation methods and their limits, when removing surface contamination.

Presently dry blasting (using steel grit/shot or mineral grits), is the most common surface preparation method used for large structures. This method is very productive, and is usually effective when done properly. However, it's effectiveness in removing deeply imbedded corrosion product contamination is not always adequate. The surface contaminants that remain after blasting, are of prime interest to the present research. There is still much misunderstanding throughout the painting industry as to:

WHEN A SURFACE IS CLEAN ENOUGH FOR COATING APPLICATION

or

WHY CONTAMINATION IS SOMETIMES NOT EFFECTIVELY
REMOVED, BY DRY GRIT BLASTING PROCESSES ALONE

There is also wide spread denial in the paint industry, of the contamination problem, even when it becomes obvious. For example, it is a common practice to just ignore the presence of dangerous levels of contamination and solve a rapid re-rusting problem by:

PAINTING A BLASTED METAL SURFACE QUICKLY BEFORE IT CAN TURN
or by
USING DEHUMIDIFICATION TO HOLD THE BLAST IN A TANK.

These common industry practices, do not address or correct the basic cause of the steel turning problem.

Steel turning is normally due to incomplete removal of the deeply embedded surface contamination, by the surface preparation methods being used and NOT BECAUSE OF HUMIDITY. This contamination is almost invisible to the eye just after blasting. However, as the contamination is usually both hygroscopic and ionic, it rapidly makes it's presence known, by causing very visible surface corrosion by-products. It is these corrosion by-products that are being described as flash rusting or rustback. The usual cause that is argued for the rust back problem is HIGH HUMIDITY rather than the true cause of the problem HYGROSCOPIC SURFACE CONTAMINATION.

The present research program found that moisture will diffuse through 12-16 mils of epoxy coating in less than 100 hours after it is submerged in sea water. Once the moisture reaches the contaminated substrate, it then dissolves any soluble contaminants present. This dissolution process creates an ionic solution at the metal surface under the paint. The ionic solution causes osmotic diffusion reactions and corrosion processes to begin.

Note: The moisture passing through the coating does not carry chloride contamination with it from the salt water, to the metal surface. The chloride substrate problem is only due to the incomplete removal of corrosion contamination from the metal surface prior to coating application.

The coating industry must begin to routinely check for the presence of the invisible surface contamination, before applying any coatings. If this is done, many potential failure problems will be prevented. As this monitoring practice becomes routine, it will become more obvious where the serious contamination problems are. In many cases a contamination source can be easily eliminated once it is recognized to be a problem. All coating inspectors must learn to routinely check for contamination sources. This checking can be done easily, with low cost (\$50 and up) pocket conductivity meters that have only recently become widely available. These rugged meters soon will become a key coating inspection tool. A coating inspector using only a conductivity meter and distilled water, can quickly determine whether a contamination problem exists, and how serious it is. These inspections should be made during all stages of the coating application program. Inspectors must also learn how to measure the grit's specific chloride and sulphate levels, in case the preliminary conductivity readings indicate there is a serious contamination problem that needs to be further identified.

INDEXS OF GUIDES IN APPENDIX B:

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ORGANIC COATINGS.

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GUIDE III- A GUIDE TO THE METHODS AVAILABLE FOR DETECTING
AND MEASURING SURFACE AND GRIT CONTAMINATION

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surface contamination-----PAGE III-6

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prepare steel surfaces for coating.

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badly contaminated tank Steel-----PAGE IV-5

I-1.5 Chloride and water contamination can come from poorly dried blasting air. Blasting air may also contain oil. The ventilation air system also can pickup chloride contamination and transfer onto the blasted surfaces if it is not filtered properly. Note: The amounts of salt needed to cause problems are extremely small. Less than 1 ounce of salt contamination per 1000 square feet of steel surface can cause serious blistering problems. To get good coating performance, we must keep salt contamination below 1/20 of an ounce per 1000 square feet.

I-1.6 Horizontal surface contamination is very often caused by workers walking on horizontal steel surfaces such as blasted tank bottoms with dirty shoes. Contamination also occurs on vertical steel surfaces from sweaty hands or by body contact. All persons walking on blasted steel must wear clean shoes with plastic shoe covers. Also the painters must wear clean shoes and covers, to prevent inter-coat contamination problems.

I-1.7 Unusually rough areas, rough welds, weld splatter, very sharp edges etc should be ground or disked smooth before starting blasting work. All welding work in areas to be coated should be completed before blasting is started.

I-1.8 Improper clean up of all the heavy grit and fine dust and particles of spent grit left by the blasting work.

NOTE : Massive coating failure can also result from many of the factors listed in I-2 or I-3.

- I-2 FOLLOWING IMPROPER COATING APPLICATION PROCEDURES:
This is a common stage of a coating application program, to create serious coating problems. The careful workmanship and efforts of many people are required from the earliest steps in the manufacturing of the coating, until the coating system is applied and cured. Any breakdown in this long chain of events can lead to serious and expensive coating failure:
- I-2.1 Improper raw materials or improper manufacturing procedures.
 - I-2.2 Improper shipping, or improper storage of coatings prior to using, particularly in hot climates. Storage at high temperatures can greatly shorten a coating's useful shelf life. The use of an over aged epoxy, can create problems during application and during service.
 - I-2.3 Improper temperature control of the coating material just prior to mixing, can lead to a very short pot life if it is too high. On the other hand in cold weather there is a high tendency for painters to add excess thinners to improve spraying and this can lead to serious solvent entrapment problems. This solvent entrapment can cause coating blistering after the tank is put into service.
 - I-2.4 Improper or inadequate mixing of coating.
 - I-2.5 Wrong or excess solvent added to paint during mixing.
 - I-2.6 Improper induction time between mixing and spraying.

- I-2.7 Poor painter workmanship can cause uneven, inadequate or excessive film thickness, over spray, dry spray, runs, sags, pin holes etc. .
- I-2.8 Lack of proper stripe coat applications in way of edges, cutouts, corners, pitting and surface defects. Poor or no color between full coats and stripe coats.
- I-2.9 Improper ventilation during coating spraying and curing. Both adequate volumes of ventilation air and proper relative humidity must be maintained until the coating has released all entrapped solvents and is cured."
- I-2.10 Improper steel surface temperatures during spraying & curing. Too hot can cause paint system damage. Too cold will retard or even stop the curing process.
- I-2.11 Improper over coating times (temperature dependant). In hot weather, it is very important to insure inter-coat application times are correct. A delay in application can lead serious inter-coat adhesion problems. This factor is particularly important if the coating has a very short over-coating time limit.
- I-2.12 Improper testing of the total coating system for: proper thickness, runs and sags, pin holes, proper cure etc. A good final inspection includes visual, magnetic film thickness and electrical continuity testing.

I-3 USING A COATING SYSTEM IN THE WRONG ENVIRONMENT:

The inability of the coating system to withstand the environment it is being used in, will usually result in massive failure in a relatively short time if any of the following factors are true:

I-3.1 Inadequate solvent resistance.

I-3.2 Inadequate ultraviolet resistance.

I-3.3 Poor chemical resistance/saponification.

I-3.4 Inadequate temperature resistance.

I-3.5 Poor water resistance.

I-3.6 Inadequate electrical resistance for cathodic protection.

I-3.7 Using many inhibited coating systems under submerged conditions, usually leads to osmotic blistering.

GUIDE II- THE EFFECTS THAT SUBSTRATE CONTAMINATION HAS ON ORGANIC COATINGS

II-1 SOURCES OF ENVIRONMENTAL CONTAMINANTS:

The following summarizes the environments that coated steel is used in and the contaminants found in these environments, that are known to be detrimental to the service life of organic coating systems applied onto steel:

- a. Marine environment - Chlorides, sulphate and oil.
- b. Bridges - Ammonia, chlorides and sulphate.
- c. Industrial atmosphere - Ammonia, chlorides & sulphate.
- d. Industrial submersion - various chemicals & oils.
- e. Rural - there is usually no contamination problems unless de-icing salts are used on the structure.

In addition to these every day environmental sources there are several other ways for contamination to get on metal surfaces prior to and during coating application:

- a. Contamination from wash down water - chlorides & oil.
- b. Contamination from the blasting grit - Chloride, sulphate, and fluoride (only in coal slags).
- c. Contamination from inhibited wash water - Inhibitor.
- d. Contamination from the coating material - primers can contain soluble chromates etc.
- e. Contamination from solvent entrapped in the cured films.
- f. Contamination from workers- Chloride from body sweat, hands, dirty shoes, sitting on steel, food, soft drinks, worker urination etc. .
- g* Contamination from recycled steel grit or shot. Once contamination gets into reusable grit it can be transferred from it onto otherwise clean steel.

II-2 COATING PROBLEMS ASSOCIATED WITH THE PRESENCE OF UNDER FILM CONTAMINATION

II-2.1 BLISTERING IN ORGANIC COATINGS

Blistering is the most widely recognized problem that is associated with the presence of substrate contamination. Blisters can vary in size from smaller than the head of a pin, to 6 inches (150mm) or more in diameter. Typically blisters are between 1/16" to 3/8" (2mm to 5mm) in diameter. They can have a frequency between a few blisters per square yard/sq.meter to many per sq.in./sq. cm.. Usually the larger the blisters, the lower their frequency. The large blisters are liquid filled and they are caused primarily by osmosis. The smaller fully developed blisters, usually contain very little liquid. These smaller size blisters are filled mainly with corrosion product buildup. The larger size liquid filled blisters usually are caused by the following factors:

II-2.1.1 DIFFUSION OF LIQUID THROUGH THE COATING

Liquids can diffuse through all organic paint films, because they ARE SEMI-PERMEABLE. Water, moisture and many (but not all) fluids *will slowly* diffuse through all organic coatings. During this

swelling of the coating, without any blistering. This swelling normally reverses, if the fluid is allowed to slowly diffuse out. This normal film diffusion should not permanently change or damage the coating, provided the coating is chemically and solvent resistant to the fluid it is immersed in. Since coatings are SEMI-PERMEABLE membranes, they are also subject to a special diffusion phenomenon called osmosis.

2.1.2 OSMOSIS

This is the term used to describe a special diffusion process, common to semi-permeable membranes. In the basic diffusion process, there is no net flow of solvent, once the membrane absorbs sufficient fluid to reach equilibrium.

HOWEVER, IF THERE IS A SOLUTION CONCENTRATION DIFFERENCE ON OPPOSITE SIDES OF A SEMI-PERMEABLE MEMBRANE, THEN THIS EQUILIBRIUM SITUATION (NO NET FLOW) CHANGES.

Note: by definition, a solution consists of a solvent such as water and a solute such as sodium chloride.

IDEALLY, ONLY THE SOLVENT PASSES THROUGH THE MEMBRANE. THE SODIUM CHLORIDE IN SEA WATER DOES NOT PASS THROUGH SOUND PAINT FILMS. THE SOLUBLE SUBSTRATE CONTAMINATION ALSO DOES NOT DIFFUSE OUT FROM THE METAL SURFACE INTO THE SEA WATER.

A differential force called osmotic pressure, is created whenever there are solvent/solute concentration differences on opposite sides of a semi-permeable membrane.

THE EFFECT OF THIS OSMOTIC PRESSURE IS TO CAUSE SOME OF THE SOLVENT FROM THE MORE DILUTE SOLUTION, TO DIFFUSE THROUGH THE SEMI-PERMEABLE MEMBRANE, TOWARD THE MORE CONCENTRATED SOLUTION SIDE AND SLOWLY DILUTE IT.

This one direction diffusion, will continue until the two solutions have the same concentration, or if the more concentrated solution is pressurized enough to physically oppose the osmotic diffusion process. In fact, if the pressure on the more concentrated side of a semi-permeable membrane is increased enough, reverse osmosis can occur and NET SOLVENT FLOW WILL REVERSE DIRECTION AND SOLVENT WILL BEGIN TO LEAVE THE MORE CONCENTRATED SOLUTION.

During the formation of coating blisters, osmosis causes pressure to build up at contamination sites. If this pressure exceeds the adhesion of the coating, it lifts the coating at that point and forms a blister. The blister then continues to grow until equilibrium is reached either by solution dilution, or the pressure builds up inside the blister, to resist further flow into it.

The pressure needed to stop osmotic diffusion from occurring through a semi-permeable membrane is called the osmotic pressure. The approximate osmotic pressure (Po) of a solution is usually expressed in atmospheres.

Note: 1 Atm.= 14.7Psi. (1 BAR)

Osmotic pressure can be calculated if the solution's OSMOLALITY (osmol) and also the solution's *absolute* temperature in degrees Kelvin (K) is known.

Note: 25°C (76°F) = 298° K (absolute).

The formula for calculating Osmotic Pressure (Po) is:

OSMOTIC PRESSURE $P_o = \text{OSMOL\#} \times 0.082 \times \text{TEMP. IN DEGREES K}$

OSMOLALITY (OSMOL)#S OF KEY CONTAMINANTS, IN AQUEOUS SOLUTIONS: (from Handbook of Chemistry and Physics)

<u>WT.%IN SOLUTION</u>	<u>0.5%</u>	<u>1.0%</u>	<u>2.0%</u>	<u>5.0%</u>	<u>10.0%</u>	<u>20.0%</u>	<u>40.0%</u>
SODIUM CHLORIDE	0.16	0.32	0.64	1.63	3.51	8.84	----
SEA WATER SALTS	0.14	0.28	0.56	1.45	-----	-----	-----
SODIUM SULPHATE	0.09	0.17	0.33	0.73	-----	-----	-----
METHYL ALCOHOL	0.15	0.30	0.61	1.62	3.53	8.09	20.1
FERRIC CHLORIDE	0.10	0.20	0.44	1.02	2.56	-----	-----

GII-2.1.3 EFFECTS OF SOLUBLE SALTS ON THE STEEL SUBSTRATE

When there are soluble contaminants on the metal surfaces, or within the coating itself, they can cause serious osmotic blister problems. Osmosis will occur when water, or any other solvent that is diffusing through the coating, comes in contact with a substance it can dissolve. If there is soluble contamination such as chloride on the metal's surface or a water soluble solvent entrapped within the coating itself, then a osmotic pressure gradient will develop. This osmotic pressure gradient then causes the solvent from the more dilute exterior solution, to diffuse through the coating and dilute the contaminant. This pressure/diffusion gradient will continue so long as any concentration difference remains. As the soluble substance continues to dissolve under (or within) the paint film, hydraulic pressure builds up, due to an increase in the solutions volume. If enough pressure builds up and overcomes the adhesion of the paint to the metal substrate, then a blister will develop.

NOTE: OSMOTIC PRESSURE NUMBERS ARE EXPRESSED OPPOSITE TO NORMALLY UNDERSTOOD PRESSURE NUMBERS. THE SOLVENT FLOWS FROM THE LOW OSMOTIC NUMBER SIDE OF THE SEMI-PERMABLE MEMBRANE TO THE HIGH OSMOTIC NUMBER SIDE.

For example the osmotic pressure from a 20% sodium chloride solution at 20 deg.C is about 212 atmospheres or 3125 PSIG. In comparison, the osmotic pressure of normal sea water is only 25 atmospheres (375psi) and distilled water has the lowest possible osmotic pressure 0. Distilled water is a pure solvent. The solvent flows through the semi-permeable membrane to dilute the more concentrated solution (higher osmotic number).

The total percentage of dissolved solids in sea water is about 3.5% solids (2.5% sodium chloride, 0.5% magnesium chloride & 0.4% sodium sulphate). This is usually lower than the initial solution concentration under the paint film, when a soluble substance first starts to dissolve. However, after this dilution process has continued for a period of time, the blister's solution concentration will be reduced toward the outside seawater's concentration.

However, if distilled water is on the outside of the blister, solution equilibrium will take much longer to occur. This is because of the negligible osmotic pressure of distilled water. To reach equilibrium in distilled water, all soluble products will have to react and form insoluble corrosion products, or the pressure inside the blister must increase enough to balance the blister solution's osmotic pressure.

Therefore, the tendency for coating blistering is less when it is submerged in salt water, compared to fresh water and the highest tendency for osmotic blistering will be in distilled or demineralized water service. This is why paint blister problems are more common in ship's distilled water storage tanks, than in saltwater ballast tanks.

GII-2.1.4 EFFECTS OF SOLVENT RETENTION IN COATINGS.

Solvent retention can occur in paint films for many reasons such as; excess film thickness, poor ventilation, insufficient curing between coats, putting the coating system into immersion service too quickly, poor coating formulation, use of improper solvents etc. If the retained solvent is miscible with water, it will create an osmotic pressure and probably induce film blistering. In chemical tankers, severe blistering can also occur, if prior solvent cargos are water soluble and not enough time is allowed for full release of the solvent that has diffused into the coating. This problem is very common with methanol. Note that the osmotic pressure for a 40% by weight methanol/water solution, is almost 500 atmospheres (over 7000 PSIG).

GII-2.1.5 EFFECTS OF SOLUBLE SALTS LEFT ON AN OLD COATING THAT IS BEING RECOATED

The presence of soluble salt on the surface of an old coating can cause inter-coat blister problems, for the same reasons explained for soluble contamination on the metal substrate (see page II-5). However, the blisters in this instance should not have any corrosion reactions with the metal surface. painters walking on the paint with dirty shoes can also cause this problem, they must wear clean shoe covers at all times.

GII-2.1.6 EFFECTS OF SOLUBLE PAINT COMPONENTS

Soluble inhibitor in primer formulations can help reduce under film corrosion in atmospheric exposures. However, the use of these inhibitors for submerged service conditions is not recommended, as their presence in the coating can create osmotic pressure and paint blistering. Note, if an inhibitor is to be used under submerged service conditions, it should have low osmolality number.

GII-2.1.7 EFFECT OF ENDO-OSMOSIS ON COATING BLISTERING

Endo-osmosis is a special form of osmosis. It is caused by electro-chemical potential differentials, created by cathodic protection or anodic corrosion processes and not solution concentration differences. The effect of these potential differences, may increase or decrease the rate of solution diffusion, depending on the potential direction and the electronic charge nature of the coating itself. The subtle effects of endo-osmosis, have not been well examined for various coatings. It is generally considered a problem only associated with cathodic protection. However, if this phenomenon was better understood, and the proper anode/coating system could be found, we may be able to use endo-osmosis to our advantage, and Prevent corrosion and blistering problems from contamination simultaneously.

GII-2.2.1 CORROSION REACTIONS AT THE METAL/COATING INTERFACE

The presence of chloride contamination under a coating, will cause underfilm corrosion to occur, even though the metal is visually free of corrosion at the time of coating application. This factor is not understood by many people in the coating industry. Therefore, it is common for people to want to paint surfaces quickly, before they can turn. This approach does not correct the problem.

Testing was done with a clear epoxy, that performed equally to conventional coal tar epoxy in submerged testing. The tests were run with both sodium chloride and sea salt surface contamination. Underfilm rusting" (brownish color) was observed developing under the clear coating, during the one week air curing period. This rusting was observed with contamination levels of $2\mu\text{g Cl}^-/\text{cm}^2$ ($0.11\text{ oz NaCl}/1000\text{Ft}^2$) and higher. This is about $1/5$ the level of contamination needed to cause osmotic blistering. After 70 hours of submerged testing, the brownish color was observed to turn black, and more corrosion was occurring. Even the lowest level of contamination tested, $0.25\mu\text{g Cl}^-/\text{cm}^2$, caused some surface corrosion after 4500 hours. However, contamination free surfaces were observed to be still corrosion free after 4500 hours of testing. The higher the level of surface contamination trapped under the coating, the greater the extent of underfilm corrosion observed.

The diffusion of moisture . . d oxygen through the organic coating, is a natural physical property of semi-permeable membranes. Until a practical impermeable organic coating system is developed, the most effective approach to the under film corrosion problem, is reduction of contamination by good surface preparation. The surface contamination provides one of the key elements needed for corrosion to take place. This element is the soluble salt required for making the corrosion cell's electrolyte.

GII-2.2.2 THE PARTS OF A CORROSION CELL AND THEIR FUNCTIONS

In order for corrosion to occur an electro-chemical cell must form. There are 6 key parts to this cell:

- 1) Cathode sites- This is where the dissolved oxygen (O_2) in the water and the water ($H^+ + OH^-$) itself, are electro-chemically reduced to form excess hydroxide ions (OH^-). Electrons are required in this process.
- 2) Electron path. The electrons needed for the cathodic site reactions come from anodic areas. The electrons travel to the cathodic sites via the electron path. This is the metal connecting the anodic area to the cathodic area. Electrons (e^-) can only travel via metallic or semi-metallic conductors.
- 3) Anodic sites - this is where the soluble metal ions (M^{++}) (oxidation) are released from the insoluble metal surface (M) and enter into the electrolyte.

Note: In order for reactions 1 & 3 to take place, electrons (e^-) must simultaneously leave the anodic sites via the metal (electron path). The same number of electrons are consumed during the formation of the OH^- ions at the cathodic sites. Dissolved oxygen must be present in the water and be reduced to OH^- ions for the corrosion process to continue.

4) Electrolyte-

This is the ionically conductive path between the anode and the cathode sites.

In the case of under film corrosion, the liquid for the electrolyte is transported to the sites via the coating, either by normal diffusion or by osmosis. This liquid is free of salt and can not act as an electrolyte. However when contamination is present at the steel surface, it changes this situation by mixing with the pure water, and making it an electrolyte.

The electrolyte can be liquid or semi-liquid. Electrons do not pass through the electrolyte, but ions do.

During the corrosion process, the electrolyte near the anode area is acidic (H^+), high in the corroding metal's ions (M^{++}) and low in dissolved oxygen (O_2), when compared to the electrolyte near the cathode.

The electrolyte near the cathode is more alkaline (OH^-), high in dissolved oxygen and low in metal ions (M^{++}).

These electrolyte conditions are unstable. The soluble metal ions and hydroxide ions diffuse towards each other, and meet at some intermediate zone along the electrolyte path that connects the anode and cathode. A stable precipitate forms at this point. This precipitate is typically called rust.

5) Cathodic reactant concentration-

This is the oxygen that is physically dissolved in the electrolyte. It is required for corrosion to occur. The coating does not permit rapid replenishment of this necessary element, therefore when it is in good condition, the corrosion reaction rates are very low.

6) Electrolyte ions- (Chloride)

The chloride ion is very effective in raising the electrolyte's conductivity. The higher the conductivity, the the greater the corrosion rates possible (all other factors remaining the same).

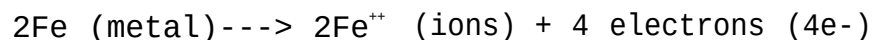
NOTE: The chloride ions do not get used Up in the primary anodic and cathodic reactions. Because of this, the limited chloride ion concentration in the substrate solution does not change rapidly. Therefore, even a small amount of chloride contamination can keep the underfilm corrosion reactions going a long time.

It has also been found that the chloride and sulphate ions tend to migrate and concentrate at metal surface beneath the bulk corrosion products. This is why it is so important to remove all the old corrosion from the metal's surface. A very small amount of corrosion by-product left on the surface, will still cause a considerable amount of underfilm corrosion.

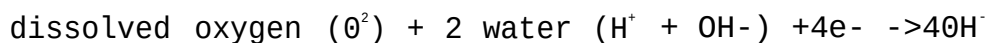
GII-2.2.3 BASIC ELECTRO-CHEMICAL REACTIONS

The excess electrons (e^-) from the anodic reaction electro-chemically combine with dissolved Oxygen (O_2) in the water and with the water ($H^+ + OH^-$) to form excess hydroxide ions (OH^-) ions, which later react with the excess metal ions produced at the anode area. During this electro-chemical reaction electrons from the anode are consumed.

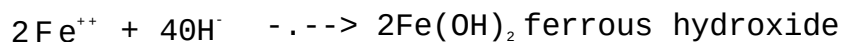
The anodic reactions for iron or steel are:



The cathodic reactions are:



A third reaction occurs later within the electrolyte:



GII-3 THE EFFECTS OF RELATIVE HUMIDITY CONDITIONS ON BLASTED STEEL SURFACES

It is well known in the coating industry that low humidity conditions will stop steel from turning. In fact this "LOWER THE HUMIDITY" approach is widely used when blasting tankage. While it is not as necessary to use this approach when blasting new steel, it is usually done out of force of habit for all tank work.

Low humidity is not required to prevent very clean steel surfaces from turning. Testing showed that clean steel does not turn for thousands of hours even if the humidity is at 100% (non-condensing). However, steel turning will rapidly occur under much lower humidity conditions, if hygroscopic salts are present on the surface. These hygroscopic salts absorb moisture from the air whenever the relative humidity is above a certain level. Different salts respond to different levels of relative humidity before becoming moist. The hygroscopic salts that come from the seawater are sodium chloride and magnesium chloride. Corrosion by-products can also be hygroscopic.

The use of excess dehumidification to hold the steel is not a good practice during the early stages of blasting. The natural turning of contaminated steel should be used to find out where the problems are. High dehumidification makes them more difficult to find. The practice of sweetening the surface by sweeping just before inspection, is also counterproductive as it only hides the problem temporarily.

The turning problem then shows up hours later, even with good dehumidification, after the tank is free of dust. In order to completely remove deeply embedded contamination, the areas of turning must be hard blasted.

NOTE: Good dehumidification must always be used to insure the clean steel surfaces are kept absolutely dry during the coating application and cure period.

GII-4 LOSS OF COATING ADHESION DUE TO SURFACE CONTAMINATION

- a) INITIAL LOSS - It is well known that many surface contaminants reduce the adhesion of paint films. An obvious example is grease and to a lesser extent oil. The better the initial adhesion of the paint system the more it can resist blistering.
- b) ONCE IN SERVICE - The presence of under film contamination can quickly reduce coating adhesion through blistering and under film corrosion. Heavy under film contamination can show up as blisters after a few weeks of submerged service. Loss of adhesion due to under film corrosion takes longer, but it is not uncommon to find *serious* coating breakdown after a few months of service.

Note: Initially very little osmotic blistering occurs in a coating system from contamination. The originally very smooth coated surface first becomes slightly rough. This roughening is due to slight film lifting from underfilm corrosion cell by-products that are physically much larger than the metal they come from.

It is very useful to carefully examine older coating systems for this type of surface roughening. The general presence of film roughening, can *indicates* that the coating system is reaching the end of its useful service life. If it is noted, then the coating's adhesion should also be checked. These adhesion tests will also expose the metal substrate so it can be examined for under film corrosion.

GII-5 SURFACE TOLERANT COATING SYSTEMS

Under ideal conditions the steel surface should be dry, clean and free of all contamination (VISIBLE & INVISIBLE) at the time of coating application. If this is done and the surface has the proper anchor pattern, then a maximum coating system life can be expected. However, from a production stand point, it would be desirable if we can develop coating systems that are tolerant to less perfect conditions. The development of a long life, contaminant tolerant coating system is the ultimate goal. Some coatings are more tolerant than others, but none of the organic coating systems seem to be fully immune to premature failure in immersion service when surface preparation is minimal. The forces of osmosis and under film corrosion are difficult to hold back by using a semi-permeable barrier coating. It would appear that an organic film alone is not the answer and some type of inhibitor is needed to stop at least the corrosion process.

In the past inhibited organic primers' have been used to counteract the corrosion, from surface contaminants. However, the inhibitor chemicals themselves have caused blistering problems under submerged conditions and this approach was abandoned in recent years. Even if the perfect corrosion inhibitor is found, it will not prevent the osmosis problems that can occur from contamination.

It should also be noted, that many *of the* inorganic zinc coatings appear to be more tolerant to chloride surface contamination than organic coatings. Also the combination of zinc anodes and inorganic zinc coatings, appears to be a very good ballast tank protection system.

GII-6 PROPER COATING APPLICATION

A well applied coating system designed for ballast tank service can last a long time. A good epoxy system should not have any blisters or loss of adhesion during the first 5 years of service. After this it is not unusual to have minor problems at inevitable weak spots in the system, but overall the system should remain in very good condition for at least 5 more years. It should be pointed out that this ten year projected life is not an absolute number but one of general experience in the marine industry when better epoxy coating systems are applied properly. However, there is good reason to expect that this life can be significantly extended by paying more attention to reducing invisible surface contamination presently ignored in most coating surface preparation and application specifications. It should also be pointed out that when a coating system begins to fail many parts are still in very good condition. If we can determine why these areas have lasted so much longer under the same service conditions, we will learn how to improve our coating application procedures even more.

GUIDE III- A GUIDE TO METHODS AVAILABLE FOR DETECTING AND MEASURING SURFACE AND GRIT CONTAMINATION

Note: part of the information given in this guide was researched for a FEDERAL HIGHWAY ADMINISTRATION PROJECT- "EFFECT OF SURFACE CONTAMINANTS ON COATING LIFE" (DTFH61-88-C-00027). This project was done jointly by Steel Structures Painting Council and GCS Corrosion Consultants Inc..

III-1.1 VISUAL DETECTION (THE TURNING OF BLASTED STEEL)

Salt and other forms of ionic contamination Usually are not visible. However their presence on freshly blasted surfaces, rapidly becomes apparent, when these contaminants cause visible surface corrosion products. The problem is there is a common misunderstanding, that the rapid re-rusting of recently blasted steel is due to high ambient humidity. Therefore, it is standard practice to reduce humidity to a level that temporarily interrupts the corrosion reaction. The general use of dehumidification, without also checking the steel for surface contamination, is dangerous. This common practice, can hide the fact that there is a contamination problem. In fact:
WHEN FRESHLY BLASTED STEEL TURNS QUICKLY, IT IS A VERY POSITIVE SIGN, THAT THERE IS A SURFACE CONTAMINATION PROBLEM

Clean steel will not turn for thousands of hours, even at 100% relative humidity.

Quickly painting the surface before flash rusting occurs, does not resolve the problem. It must be recognized that the surface contamination remaining, will still induce corrosion and osmotic reactions. These reactions occur, as soon as moisture passes through the semi-permeable organic coating system. Once the tank is put into ballast service, it takes only a few days for the moisture to pass through the coating and reach the steel.

The fact that the steel was still white at the time of coating application, does not stop the reaction, it only slows it down . The steel will still corrode underneath the coating if contamination is present. However, if you decide to coat over contamination, than it is better to do it while the surfaces are still dry and white, rather than after they have turned.

NOTE : This last statement is not meant as an endorsement, for painting over contamination. It is just acknowledging that initial coating adhesion is better, when a contaminated surface is painted over while it is still white, rather than after it has turned. Coating over contamination does not stop the corrosion reactions, it only slows them down and delays the failure.

DEHUMIDIFICATION SHOULD NOT BE USED TO HIDE THE PRESENCE
OF STEEL CONTAMINATION BY STOPPING IT FROM TURNING.

It should be used primarily
TO INSURE THE CLEAN STEEL SURFACES ARE
VERY DRY AT THE TIME OF COATING APPLICATION

The turning of steel after blasting, must be recognized as *the* symptom, not the problem. The steel surface must be properly cleaned of contamination, in order to insure good coating performance.

The actual level of contamination left on the surface, can not be judged by eye. Therefore, proper instrument testing of surfaces must also be done, to back up visual detection.

Properly trained inspectors, can spot signs of contamination at a fairly low level, once they know what to look for. The detection instrumentation helps the inspector make better technical evaluation, of what he is observing.

There are also other invisible contaminants, that are not corrosive. Oil is a good example of this type contamination. Note, the presence of oil on a chloride contaminated surface, could stop the flash rusting process and give an inspector a false sense of surface cleanness. However, instrument testing would detect this contamination in most cases.

III-1.2 LIMPET CONDUCTIVITY CELL MEASUREMENTS ON SURFACES

It is possible to detect the presence of ionic surface contamination, by means of Limpet cell measurements. The idea for this type of surface test cell was suggested over 30 years ago by J.E.O. Mayne²³, but very little work was done. Recently at least two instruments have been developed using this principle. They permit relatively simple testing for surface contaminants directly on painted or bare steel. These instruments incorporate a conductivity meter with the limpet cell. They are temporarily attached to the steel surface by strong magnets:

1. SOLUBLE SALTS DETECTOR by DATA ACQUISITION LIMITED
Electron House, Higher Hillgate, Stockport, Cheshire
SK13QD England, Tel#01-44-61-477 3888.
2. SOLTZ DETECTOR by GCS CORROSION CONSULTANTS
3 COOPER DRIVE, Howell, N.J. 07731, Tel#608-363 8820

These cells are first sealed against the metal surface, by a built in soft rubber O ring. Proper sealing can be a problem if the steel is very rough. Distilled water is then injected into the cell, to dissolve the soluble contamination from the metal's surface. This field test takes about 3 minutes. The cell's built in meter gives an immediate reading, which indicates the level of surface contamination by measuring solution conductivity.

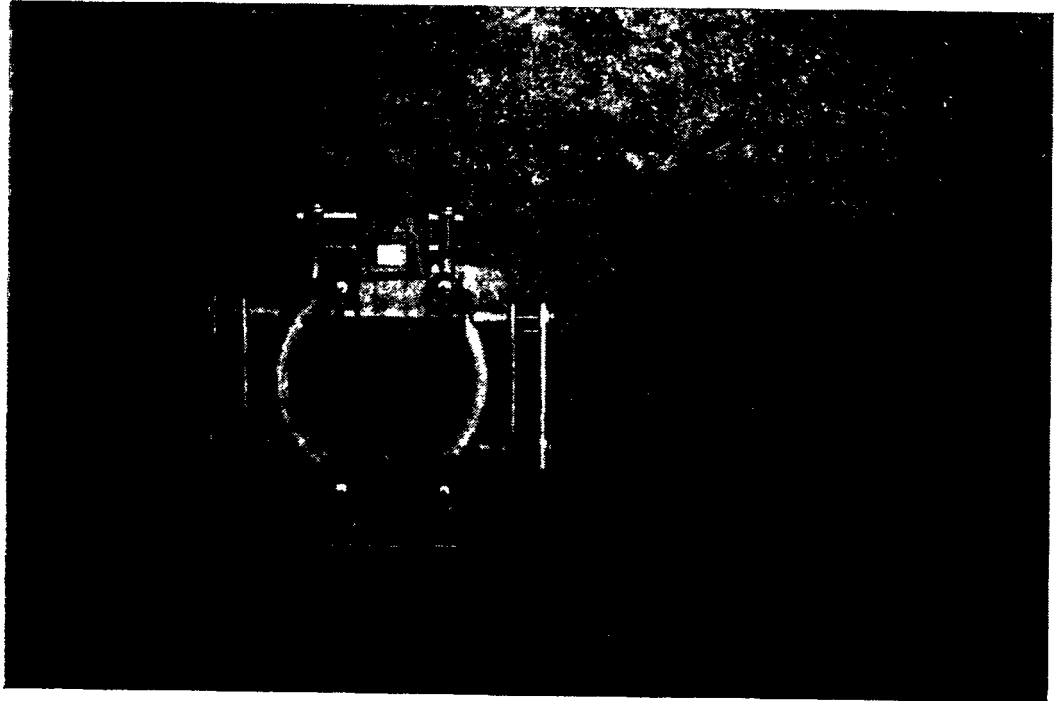
Note, this conductivity measurement is non-specific, so it is advisable to also run specific chemistry tests for chloride and sulphate later, with the solution that is removed from the cell.

The sensitivity of the limpet cells to detect contamination is good. A properly designed cell can detect chloride contamination below $\mu\text{g}/\text{cm}^2$. However, when doing readings this low, it is better to have a high sensitivity type cell. The cell's sensitivity, is determined by the ratio between the surface area covered by the cell (in cm^2) and the volume (in milliliter) of test solution used. Cells with area ratios up to 8 times their water volume can be built, but a 4 times ratio cell should be sensitive enough for most inspection work.

The cell must be cleaned carefully before use, to avoid false readings. A good grade of distilled water must be used. Once an inspector is practiced in using these meters, he can take about 10 measurements per hour, to back up his visual inspections.

At the present time these limpet meters are just beginning to be used in the field. There are still no industry standards for what is or isn't a passable contamination level. It was the goal of this research project to help determine the contamination numbers. They are discussed in the experimental part of the report. The present research has developed some preliminary numbers. These numbers can be use as a guide until more in field contamination test data can be recorded. These field generated numbers, should be compared with the actual performance of the coatings, over various levels of contamination. This field performance testing will take many years, to complete. Therefore at the present time, the numbers generated by the current study and similar studies can be used as guides.

It should be noted, that the current study differs from many of the earlier studies, as it addresses the effects of underfilm corrosion, on the development of osmotic blistering. The levels of contamination needed to produce serious underfilm corrosion, are much lower than those needed to cause rapid film blistering. The present study therefore recommends tighter contamination control standards, than some earlier studies that considered only osmotic blistering as the sign of failure.



A VIEW OF A LIMPET CELL TESTING
BLASTED STEEL THAT IS STILL CONTAMINATED

The limpet type cells attach themselves to the steel surface by magnets. These units seal against the steel with a soft rubber O-ring to form a cell. The limpet cell shown is a Soltz Cell, which has overall dimensions of 6 inches (15cm) X 6 inches. The area tested for contamination with this cell is about 9.5 square inches (60 square centimeters). Testing is done with 25 milliliters of distilled water for 3 minutes. The dark area to the right of the cell was just tested for its contamination level. The conductivity of the retrieved solution was 117 micro-Siemens and it contained 20 ppm chloride, 12 ppm sulphate and 25ppm iron. Tests on clean steel areas produce solutions with conductivity well below 10 micro-Siemens. These lower conductivity solutions had chloride levels below 1 pp.m and no sulphate or iron. The cell *must be kept very clean* when taking the lower level readings, to avoid getting falsely high readings. This limpet type cell can detect chloride contamination to below $1 \text{ Ug Cl}^- \text{ L}^{-1}$. The limpet cells have built in conductivity meters to detect the presence and approximate level of any type ionic contamination, but they do not tell which type ions are *present*. Other tests must be used to determine the specific ion types and their solution concentration. Note: The solutions final ion pickup concentration, is affected by the total area tested and the volume of test solution used. Smaller amounts of solution per unit area of surface produce a higher ion concentration compared to using larger volumes. This factor must be accounted for, when determining the level of contamination present per unit area. Also the total amount of contaminant present is not fully retrieved and a correction factor to account for this loss should be included.

III-1.3 SWAB TESTING FOR SURFACE CONTAMINATION -

Swab testing has been used for a number of years by a relatively small number of coating inspectors, to test surfaces for soluble salt contamination. At the present time only one major paint manufacturer requires that surfaces be tested for their chloride level, and that contamination must be lower than a specified maximum level before application can proceed.

While the swab test is relatively simple to do, it is very prone to operator error. The more a person does swab testing, the better skilled they become. The swabs are usually made of high quality cotton balls (these must be checked that they are free from chloride) or small clean sponge cubes. Using carefully cleaned sponge cubes instead of cotton is recommended by SIGMA Coatings. Sponges are easier to use than using cotton swabs, since they don't tear apart on the rough steel. Also obtaining very low chloride cotton swabs, can be difficult.

Attention must be taken not to introduce extraneous chloride contamination from the person doing the test. Clean throw away surgical gloves must be used when handling the swabs or sponges. Every thing must be handled very carefully, to prevent chloride pickup from the hands. There is enough chloride on a person's hand to cause high false readings.

The contamination must be swabbed from a specified surface area (square centimeters) with a known amount of distilled water (in milliliters). The area divided by the volume, determines the test sensitivity. The sensitivity of this test, can be increased by increasing the area swabbed, without increasing the amount of water used. This increase in test sensitivity, must be corrected for, when you calculate the level of contamination. This level is expressed in micrograms of contamination, per square centimeter. The lower the level of surface contamination, the higher the swab test sensitivity needed. The retrieved solution can be tested immediately in the field with a pocket conductivity meter. The solution is then saved for later testing by the same analytical methods used for the limpet cell solutions. There is a kit (SCAT), for swabbing surfaces, put out by KTA-Tator Inc., 115 Technology Drive Pittsburgh, PA. 15275, Tel, .# 412-788-1300.

GIII-1.4 OTHER DETECTION METHODS

GIII-1.4.1 ADHESIVE STICK ON CELL (BRESSEL CELL)

There is a single use, Band-Aid type patch called a Bressel cell, that was recently developed in Sweden. This type cell, is stuck on to the surface being checked, by it's self contained adhesive edges. Distilled water is then injected into it's rubber center by a hypodermic needle. The patch fills up like a large paint blister. The liquid is then retrieved from the patch and tested the same as from the other detection methods. This cell is similar in principle to the limpet cells, but it can only be used once. There are no reports yet on the effectiveness of this type cell. This cell is being developed by:

EXPERTUS KEMITEKNIK AB - Kungsvagen 1 S, 182 75 Stocksund,
Sweden Telephone# 01-46-08-85 68 55

GIII-1.4.2 SALT CONTAMINATION METER

This is a new type of meter, called the SCM 400 (Salt Contamination Meter), that uses absorbent paper to pick up salt from a surface. The paper is then inserted into a special meter that gives a reading related to the level of contamination. This unit is also very new and there are no reports available on its accuracy. This unit is made by: NNC Limited -Warrington Road, Risley, Warrington, Cheshire WA36BZ, ENGLAND TEL#01-44-925-51291

GIII-1.4.3 TREATED PAPERS

There have been a number of attempts to develop a chemically treated paper that can be put on a surface and immediately detect the absence or presence of contamination. Potassium ferricyanide paper has been used to detect the presence of iron salts on the surface, but it too sensitive and hard to interpret what is being indicated.

III-5 DISCUSSION ON THE DIFFERENT DETECTION METHODS

It should be realized that neither limpet cells nor the swabbing tests, can retrieve all the contamination present on a surface. Testing done by the Steel Structures Painting Council, found that there is only about a 50% retrieval rate when using a limpet cell and even less with cotton swabbing. Limited field tests found that the sponge swabbing retrieval rate, was similar to the limpet cell. Therefore when measuring surface contamination the inspector must realize the readings obtained, are on the low side. In spite of this limit, the present detection methods are still much better than what was being done before, i.e. just ignoring the problem. In fact, if used properly the present detection methods can give the inspector very good information, on the extent and the level of surface contamination.

When checking surfaces for contamination problems, it is important to differentiate the contamination levels on smooth surfaces and rough surfaces. The minimum level of contamination is usually obtained on very smooth areas, that are well blasted and look clean. In fact, properly cleaned steel, will remain white metal for weeks, even under very high humidity conditions. These smooth areas are representative of, the lowest practical levels of contamination that can be achieved, with the quality of grit being used. The cleaner the grit, the lower the reading that can be obtained.

The minimum contamination level will be slightly higher on rough, or pitted steel, even if they are properly blasted. This is due to the fact, that it's harder to remove contamination from these areas. Therefore, rough and pitted areas need much more attention during blasting, compared to smoother or intact painted areas. Also, the rough and pitted surfaces should be checked more closely for contamination, after blasting (but before application of coating), than smoother areas, as this is where the contamination is more likely to be. This is also the reason that it is very important to stress to the sandblasters, that they must take extra effort, and properly blast rough and pitted areas more than smooth areas.

Another factor it is important to get the blasters to understand, is that areas which are turning fast, are still contaminated. These specific areas are not satisfactory, in spite of the fact that the rest of the steel is satisfactory for coating. These still contaminated rough or pitted areas, need hard blasting and not the quick sand sweeping that is typically done. The common practice of re-sweeping the entire area, rather than properly cleaning just the turned areas, is another good example of people not understanding the contamination problem. In fact, the natural turning of contaminated blasted steel, is probably the most practical detector we have for locating the still contaminated areas.

STEEL TURNING VISUALLY SHOWS US WHERE THE CONTAMINATED AREAS ARE. STEEL TURNING SHOULD BE USED AS AN INSPECTION TOOL (LIKE AN AUTOMATIC CHALK MARKER) TO TELL US WHERE TO BEST DIRECT OUR EFFORTS TO REMOVE THE CONTAMINATION PROBLEM.

If this positive use of steel turning, can be brought home to paint inspectors and the blasters, much time and effort can be saved and they can start cleaning up the badly contaminated areas properly and more quickly. This targeting of the problem areas might be called 'SMART BLASTING'.

The testing of surfaces for contamination with swabbing or the use of limpet cells, is a very important part of a good coating application procedure. Careful testing, changes the process from an art to engineering. This change from art to an engineering approach is long over due. Coating work represents a very large part of the total cost of ship construction or repair. The largest single cost in coating work is surface preparation.

The limpet type cell is a quicker and more controllable method to check for surface contamination, particularly under poor working conditions, compared to swabbing. Swabbing on the other hand needs very little equipment. Regardless of which method is used, it is much better to test for contamination, than doing nothing at all.

The limpet cell can be used in tanks that are in the process of being blasted, to better determine where more blasting work is needed. The ideal time to test for remaining contamination is when the tanks are free of heavy grit build up and are roughly blown down. It is usually better to wait until the end of the rough grit removal, as contaminated surfaces will have had time to re-rust and be easier to find. It is also better not to use too much dehumidification, in the early stages of blasting as this inhibits re-rusting and makes it harder to visually inspect for contamination. Low humidity conditions will not affect testing for contamination by swabbing or limpet cell, it only makes it harder to find the contaminated areas. Higher humidity conditions (70-80% Rh.) will not cause properly cleaned steel to re-rust, only the contaminated areas will show up.

NOTE: The temperature of the steel surface, not the dry air temperature, should be considered the dry bulb temperature when measuring for % relative humidity.

proper inspections for contamination, should always be done before final grit clean up work is started and not at the final dust free inspection. The spot areas tested by swabbing or limpet cell should be re-blasted at the same time the final blasting touch up work is done. If a careful pre-inspection is made and the blasters properly reblast all the areas marked up, the tank should be ready for final dust removal. If there are a lot of areas marked up at this pre-inspection, it would pay to do a second pre-inspection, before final dust pickup and final inspection. Turned surfaces at the time of final inspection, indicate that the blasters did not follow the final blasting instructions and properly reblast the marked up contaminated areas, or that more detailed pre-inspections are needed to mark up the contaminated areas, before final blasting and cleanup work is started.

Good dehumidification is highly recommended just prior to and during coating application. This insures the cleaned metal surfaces are absolutely dry at the time of application and that the coating cures, under dry and well ventilated conditions.

III-2 TESTING METHODS FOR MEASURING CHLORIDE CONCENTRATION

III-2.1 CONDUCTIVITY METER

This method is very simple and very sensitive. It can easily detect chloride levels below 1 PPM. However, this meter is NON SPECIFIC in its measurements, i.e it does not determine which contaminants are present. This factor is not a problem, as the meter should be used to alert field inspectors of potential contamination problems. If the meter shows unusual high levels of conductivity, then more specific tests for chloride and sulphate should be made. If the meter shows low conductivity, then chloride, sulphate or other ions are not present.

With the advent of low cost (\$45-\$90), pocket size & rugged conductivity meters, field testing of conductivity can now become as common as measuring film thickness.

An inspector using one of these meters and distilled water, can regularly check the conductivity of the grit going into the hoppers. This grit testing should be done through out the job, to insure no contaminated or off specification grit is being used. The meter can also be used in the field, to immediately check the conductivity of the water retrieved from surfaces by swabbing or from a limpet cell. The conductivity test does not contaminate the solution for further chloride or sulphate testing that may be required, if the conductivity is found to be high.

NOTE : The conductivity meter should be rinsed in distilled water before it is introduced into the next sample to prevent transfer from one sample to another.

NOTE : SAMPLES SHOULD NOT BE TESTED WITH A pH METER before testing for chloride, SINCE THIS METER WILL INDUCE CHLORIDE INTO THE SAMPLE.

NOTE : By checking the swabbing or limpet cell solution's conductivity at the test site, the inspector can immediately compare the reading with the appearance of the surface it came from. This gives the inspector better feedback on the contamination problem. Also it is very useful to be able to show the blast foreman and the workers that there is a contamination problem, even though they can't see it clearly. Contamination can usually be seen once you learn what signs to look for. The conductivity meters and limpet cells are very useful for helping confirm what you see.

Pocket conductivity meters are now available from several sources from about \$50 and up including:

Cole Parmer tel. #1-800-323-4340

Markson tel. #1-800-528-5114

Omega tel. #1-800-826-6342

The pocket conductivity meters come in several types. They can measure in micro-Siemens (same as micro-Ohms) or dissolved solids. The micro-Siemens (uS) range is preferred, and is the standard term used to indicate conductivity. A meter with a range of 0-100 uS is recommended for testing samples from surfaces by swabbing or limpet cells. The Pure H₂O meter made by HANNA INSTRUMENTS and available from Cole Parmer model number N-01491-80 has this low range. This meter has been found to be very useful for both the laboratory and field work. This digital meter can read up to 200 uS (on over range) but it is not as accurate above the manufacturer recommended 100 uS level as it is below 100 uS. However, the 100-200 uS range is usually accurate enough for most grit contamination measurements in the field if high grade, low conductivity grit is being used. If higher range meter 0-2000 uS (Cole Palmer #N-01491-62) is better for checking grit, and it is definitely required if a medium or higher conductivity grit is being used. When ordering conductivity meters also order conductivity calibration solutions (Cole Parmer #N-01491-85) for the low micro-Siemens meter and (Cole Parmer #L-01482-70) for the 2000 micro-Siemens meter. These solutions will allow you to check and adjust these meters.

NOTE: If the meters stop reading properly, or won't go to zero in air, check that the batteries are not weak and that the meter is properly rinsed in distilled water.

NOTE: The meters from Cole Parmer are temperature compensated, therefore when adjusting them in the calibration solution let them sit for a minute and then set the reading to the solution's conductivity value at 25°C.

NOTE: Distilled water with 1 ppm of chloride ions in it, has a conductivity of about 4 uS. This is a useful number to use in the field as a rough estimate of the chloride in the solution up to about 400 uS i.e. divide the conductivity number read on the meter by 4 to get chloride in PPM. Note this is only a rough estimate of chloride level, as other salts may also be present.



A TYPICAL POCKET CONDUCTIVITY METER
& CHLORIDE TESTING STRIPS

The modern pocket digital conductivity has only been available a few years. It is very compact (about 6 inches long) rugged and easy to use. However, its use is not yet wide spread in the coating industry. It is a very useful tool to quickly spot if there is a contamination problem with the grit or on a blasted surface. Depending on the instrument model, conductivity measurements as low as 0.1 micro-Siemens(uS) (0.1 micro-Ohm) to over 20,000uS can be made. The lower reading meters measure in increments of 0.1uS, while others read in increments of 1, 10, or 100 uS. There are also some pocket meters with dual ranges. A range of 0-100 or 200 US is very useful for checking distilled water, surface contamination and better quality grit. A 0-2000 US meter is very useful for testing wash water and grit.

Two chloride test strips are also shown on the photograph. These strips can give you a good idea of the chloride level in a solution if it is between 30 and 180ppm. The strips are not sensitive enough for testing limpet cell solutions but are ok for some swab testing solutions and are good for grit testing. Note the 1 inch scale (white part is one inch) just below the two strips to see how compact the strips and conductivity meters are.

III-2.2 CHLORIDE TESTING STRIPS

These are chemical sensitive test strips, that can roughly measure chloride ion levels between 30-180 parts per million. While this chloride level would seem low it is not sensitive enough for limpet cells. It is sensitive enough for some swab tests provided the volume of fluid is kept small and the area swabbed is relatively large. This is a good range for testing chloride in grit.' Also, as this test strip uses only a drop of solution, it can test the chloride level within a blister. A convenient size bottle of these strips can do about 50 tests at about \$.50 per test. Note these strips are light sensitive and should be kept in their dark container. They are manufactured by Environmental Test Systems, Inc. ,Elkhart, IN. Tel. #219-262 2060

III-2.3 CHEMICAL VACUUM AMPOULE-

This is a quick and simple test method to check chloride levels. This method is sensitive down to 2 PPM chloride. The test requires about 15 ml of test solution. This test method is suitable for all the retrieval methods. Note: The measuring scale on the ampoule is logarithmic, this causes very crude readings above 10PPM, therefore it may be necessary to dilute solutions above 10PPM down to a better to read range of 2-8PPM. Note also that these high sensitivity ampoules have a limited shelf life. Similar ampoules are also available for testing 20-200PPM chloride levels. This is the recommended range for testing chloride in grit. These higher range ampoules also have a logarithmic reading scale, and a limited shelf life. There are 30 glass ampoules in a box costing about \$20 from: CHEMetricts Inc. Rt. 28 Calverton, VA 22016 TEL. #703-788-9026

III-2.4 NEW WET CHEMISTRY TESTS FOR CHLORIDE-

While this method is not as easy for the occasional user as the strips or vacuum ampoules described earlier, it is more practical and economical if a routine testing program is being set up. This method uses a Hanna Instruments chloride kit that has a Diphenylcarbazone-Bromophenol" Blue indicator and a Mercury Nitrate titrant. The kit costs about \$25 and has sufficient chemicals for hundreds of tests.

This kit is used in conjunction with a Hach digital titrator (costs \$90). With this combination, 5 milliliters of solution can be measured for chloride levels between 1 PPM and 200 PPM with an accuracy of 1 PPM. This test method is also linear. The test method was developed for the DEPT. OF FEDERAL HIGHWAYS, as there was no single source chloride test available for checking the small volumes involved in the surface contamination tests.

EQUIPMENT NEEDED:

- A.) Hanna CHLORIDE TEST KIT available from:
Cole Palmer Instrument Company Chicago, Illinois 60648
(tel. #1-800-323-4340) Catalog No. N-02652-10
- B.) Hach digital Titrator #16900-01 (titrator only) or
Hach digital Titrator kit #22709-00 (note this kit comes with a much better size storage case that hold other equipment needed for contamination testing)
- c.) 6 Empty Titration Cartridges #14495-01,
- D.) Spare Delivery Tubes 90° #17342-00
10 glass tubes marked for 5 ML #1926-00 from: (items B,C,D]
Hach Co., PO Box 389, Loveland, CO 80539, TEL#1-800-227-4224

PROCEDURE:

The empty titration tubes are filled with the mercury nitrate solution (HANDLE CAREFULLY POISON) supplied in the Hanna kit, after it has been diluted to 80% and 8% of original by volume with distilled water. This odd dilution is required to match the Hanna solution to the Hach digital titrator which reads 800 digits per milliliter discharged.

The 80% Hanna mercury nitrate is for chloride testing above 25 PPM, at this strength it requires 1 digits per PPM Cl⁻ in a 5 ML sample.

The 8% Hanna mercury nitrate solution is for chloride testing below 25 PPM. It requires 10 digits per PPM Cl⁻ in a 5ML sample.

A 5 ML sample is prepared for titrating, by first putting in 2 drops of Hanna solution A and then 2 drops of solution B. "

The digital titrator is then used to test the sample for chloride. Mercury nitrate is added, until the solution changes color from pale brown to pale blue.

Divide the number on the titrator by 10 to read solution Cl⁻ in PPM, when using the 8% Hanna mercury nitrate solution. Read ppm directly, when using the 80% solution.

Note, also refer to instructions given by Hanna and Hach.

It is very useful to practice testing for chloride on distilled water samples and the 84 micro-Seimen conductivity meter *solution*. By testing these solutions a number of times, one gets use to detecting the color changes that occur. Also, after practicing, you get a better feel as to the limits of this method. You will see that when testing distilled water the color change is not instant, and about 10 digits of 8% Hanna mercury nitrate solution is added before a strong change occurs. This slack will always occur and a correction factor is needed, to account for the missing 1 PPM. Therefore when reading unknown solutions, it is proper to subtract a few digits from the final reading to account for this color change error. The color change error varies from person to person, depending on how they perceive the color change. This reading of the color change is also a problem with the CHEMetrics ampoules.

The advantages of this method is that it is low cost to setup, low cost per test, rapid per test (less than a minute), relatively easy to learn, reasonably accurate and compact.

The disadvantages are diluting the mercury nitrate solutions accurately and putting the mercury nitrate (POISON) into the Hach cartridges.

III-2.5 METHODS FOR MEASURING SULPHATE CONTAMINATION

Sulphate is another recognized contaminant. It was found that a considerable higher amount of sulphate than chloride contamination, is required to cause blistering problems. However, some of the tests with clear epoxy also found that there could be a lot of substrate discoloration from sulphate, without blister formation. Sulphate can come from several sources; seawater, grit and atmosphere.

At this time there are no suggested maximum levels for sulphate contamination in grit. But since simple methods are available it should be checked for, particularly if the chloride ion measurements are found to be much smaller, than is indicated by the conductivity measurement.

Sulphate can be tested for with one of several test methods. The easiest method of quickly checking for the presence of sulphate is by turbidity. A small amount of barium chloride (POISON) is added to a sample and if it turns cloudy, sulphate is present. The degree of cloudiness indicates the amount of sulphate present. The simplest way to measure the degree of cloudiness is by optical comparator.

LaMotte Chemical Products Co. PO Box 329, Chestertown, Maryland, 21620. Tel. #1-800-344-3100 makes a device called the Octet Comparator. It is included in their Sulfate Test Kit, Model PSAT, Code 7778. This kit costs about \$50. The comparator reads sulphate only to the nearest 15ppm. , between 0 and 200 ppm. This is good enough at the present time.

If a more accurate sulphate reading are desired, then \$200 calorimeters are available from either LaMotte Chemical or Hach Company PO Box389, Loveland, Colorado 80539, tel. #1-800-227-4224

III-3 METHODS FOR TESTING AND MEASURING GRIT CONTAMINATION

III-3.1 MINERAL GRIT TESTING

The testing of mineral grit in the field, is a very simple procedure that should be done regularly, while blasting is being done. The test is non disruptive and simple to do.

The only equipment needed is distilled water, some small clean containers and a low cost conductivity meter. The range of the conductivity meter should be from 0 to 2000 *US* in most cases. The normal ASTM grit test calls for using one volume of grit and one volume of distilled water. These are mixed together and shaken. The solution conductivity can then be measured after about a minute, with reasonable accuracy. Note that the high sensitivity Hanna H_2O meter, can read only to a maximum of 200 *uS*. To use this meter on a higher conductivity grit, you must use two or even more volumes of distilled water, per volume of grit. Each doubling in solution volume, halves the conductivity reading. Grit with very high conductivity, must be checked with a conductivity meter able to read 0-2000 *uS*.

The grit can also be tested specifically for chloride by strip, ampoule, or wet chemistry. If the majority of the grit conductivity is caused by chloride then you can make a rough judgement as to the chloride level in PPM, by dividing the conductivity reading in *US* by 4. Therefore a 200*uS* reading, indicates about 50 PPM of chloride in the solution.

Note the above *lKiTM* method is used to check grit conductivity in a standard manner, but it does not actually measure grit contamination in terms of PPM chloride per unit weight of grit.

The quality of the grit can affect, the average level of contamination on the blasted steel surface. The effects of grit contamination can be measured by swabbing or by limpet cell. These readings should be taken on smooth, none corroded surfaces, rather than corroded areas. By taking readings in smooth areas, you can better judge what amount of contamination the grit itself is putting on the surface. These readings should be compared with the level of contamination in the grit and recorded for future reference.

III-3.2 STEEL SHOT OR GRIT TESTING

The testing of steel grit or shot for contamination has not been a common practice. However this testing should be done on a routine basis, to make sure the reusable grit has not pickup - contamination unknowingly. The chloride can be picked up by seemingly unimportant factors, such as vehicles bringing subassemblies into the blast shed. The wheels of the vehicle can pick up chloride, particularly in the winter time, and deposit it on the floor of the shed. From here it can be picked up by the grit, and then transferred to the new steel. At first it may appear that very little chloride could be picked up this way, but it would only take a few ounces of road salt to contaminate many tons of grit. The same methods used to check for chloride in mineral grit can be used for steel grit. Steel grit should also be checked for oil pick up.

The use of steel grit to blast old structures has been propose as a method to reduce grit disposal problems. Using steel grit for old structures must be done with great caution as the grit may become contaminated during use. The grit should be monitored to make sure it stays clean enough. The surfaces would also have to be checked to see how much contamination remained and if the grit was transferring contamination to the steel while it was removing scale. Surface testing should be a key part of any job using steel grit on contaminated old surfaces.

GUIDE IV- METHODS TO REMOVE CONTAMINATION AND PROPERLY PREPARE STEEL SURFACES FOR COATING APPLICATION

IV-1 DRY ABRASIVE BLASTING

This is the most commonly used method for preparing surfaces for painting. This method is very productive and relatively effective in removing the majority of surface contamination normally present. However,

THIS METHOD IS NOT ALWAYS EFFECTIVE FOR REMOVING
DEEPLY IMBEDDED CONTAMINATION, OIL or GREASE
FROM HEAVILY PITTED AND ROUGH STEEL SURFACES

Soluble corrosion products still remaining at the bottom of pits after blasting, are a prime cause of early coating breakdown in these harder to clean areas. In fact, it is this still trapped contamination that causes the very common problem of rapid steel turning (rust back) in way of pitted areas, very soon after they have been blasted. Since the pits are natural points of coating system weakness, they tend to get deeper and deeper and therefore harder to clean. A different method of surface preparation must be employed to effectively remove the more deeply imbedded contamination from pits and stop the pit/coating failure cycle.

Fortunately the answer to the problem is relatively simple. The mechanically difficult to remove contamination is water soluble and high pressure fresh water can be used to remove it from surfaces. However, the use of fresh water alone, even at pressures high enough to remove hull fouling (2,500-4000 psi/150-250 bar), will not remove the soluble contamination, if it is hidden under hard scale. Therefore, the hard scale corrosion products must be removed first by normal dry or wet blasting procedures, to allow effective water washing of the corrosion products at the bottom of pits.

IV-2 WET BLASTING

Wet blasting was developed as a modified sand blasting method, to help reduce the dust problems associated with dry blasting. This method is also better than dry blasting alone, for removing the deeply imbedded soluble chloride contamination found in way of pits. The use of wet blasting inside tanks however, is not practical because of the difficulty of removing the wet grit from tanks.

Wet blasting can be used effectively for the initial surface preparation in way of badly pitted exterior hull steel.

Note: unless an inhibitor is used in the water, the wet blasted steel will turn brown very quickly. It is recommended practice to dry sweep, or dry blast these re-rusted surfaces back to near white, before painting them. Because of this extra sweep blast step, wet blasting is usually considered less productive than dry blasting. However, using simple productivity analysis alone is incorrect, since it does not take into account, that the wet blasted surface is free of both the visible and invisible soluble contamination. In fact if only dry blasting was used, much more time and grit would be needed to achieve the same degree of surface cleaning particularly in way of pitting. However, as extra equipment and steps are needed for wet blasting, its use should be limited to pitted steel. It is not being recommended for new steel surface preparation, or for *removal of* paint systems in fair to good condition. Wet blasting should also be considered for removal of marine paint systems that are failing by under film corrosion.

When underfilm corrosion is extensive, it is not unusual for the steel surface under the paint, to be rough and highly contaminated with corrosion products high in chloride. These may be difficult to remove by dry blasting alone.

On a typical large scale hull recoating jobs, there are usually deeply pitted areas where active corrosion is occurring. This localized corrosion will continue in these pitted areas, unless special corrective action is taken. The problem is caused by the deeply imbedded soluble corrosion products that migrate to the rough corroding metal surfaces during the normal corrosion process. Dry blasting alone does not reach all of these contaminants, unless the area being blasted per hour, is low enough to insure very complete blasting in way of pitted areas. Even if considerably more time is spent dry blasting the heavily" pitted areas, it may not fully correct the problem since grit still does not always reach to the very bottom of some pits. Note: it is advisable to dry blasting with a grit mixture that contains mainly medium and fine mesh, when active deep pitting is present. The medium grit is needed to remove the heavy corrosion products and the finer mesh grit helps to remove the contamination from the bottoms of pits on rough surfaces. On the other hand, very heavy grit tends to upset the metal's surface and its use helps to entrap some surface contamination and grit particles. Therefore, very heavy mesh grit should only be used when # very thick hard scale is present. Wet blasting should be considered more often as a preliminary step in older hull surface preparation. It should be done prior to dry blasting of the hull, particularly in way of active pitting areas. When possible, this limited wet blasting should be done before high pressure water washing of the entire hull, to further insure more complete removal of the soluble contaminants.

IV-3 HIGH PRESSURE FRESH WATER WASHING

High pressure water washing has been used for many years to remove surface contamination such as marine fouling and other dirt prior to dry blasting. Ultra-high pressure (10-35,000 psi) washing can even be used to whiten metal surfaces, but productivity is very low and no surface profile is created. The *metal* whitening productivity can be improved by injecting small amounts of sand into the water stream.

Medium high pressure washing (2500-4000 psi) is probably the best method available to properly prepare a fouled hull paint system, for recoating. This pressure is also very good for removing the deeply imbedded soluble contaminants found at the bottom of active pits. However, in order to remove this contamination quickly, it is necessary to first clean steel surfaces free of all hard scale, by hard grit blasting or *commercial* blasting. It is necessary to remove all the solid corrosion products, to get the water to the underlying soluble contamination.

Therefore wet (outside hull only), dry spot blasting in way of limited badly corroded areas or if the problem is extensive, heavy commercial blasting of *all* surfaces, are necessary first steps, for removing a deeply embedded soluble salt contamination problem from surfaces to be coated.

High pressure water washing, in way of the blasted steel surfaces, can remove all of the contamination if done correctly. The high pressure nozzle must be held closely to the surface, particularly if it is rough and pitted. A hand held high pressure nozzle is not effective on pitting, if it is held more than 6 inches from the pits being washed out. The workers washing the surface, must understand that it is important to wash all the surfaces very well. It can be a problem visually checking that the washing is done right, as all surfaces turn brown or black after getting wet. However you can quickly spot check the steel with a limpet cell. The limpet cell can test if an area has been properly washed in a few seconds. The dry clean surfaces are then re-blasted.

IV-4 PROCEDURE FOR CLEANING BADLY CONTAMINATED TANK STEEL

If an old tank that is scheduled for recoated, is known to be or is suspected of being badly contaminated with soluble salt, it may be much quicker to do the following steps and not try to use dry blasting alone:

a. Plan to blast off all old coating and corrosion to a commercial blast level or higher. The higher grade blast is needed in rough and pitted areas. Use a grit mix that has fine, medium and heavy grit in it. The fine grit is needed to help clean out the bottoms of pits. Do not use too coarse a grit, unless the scale conditions are 'very bad, as' it tends to embed corrosion products deep into the surface making them difficult to wash out. Concentrate on those areas that have active surface/pitting corrosion or extensive under film corrosion. At this time it is not necessary to remove deeply embedded sound coating. Also during this rough blasting work it is not necessary to use any dehumidification. The pitted areas should be checked to see that they have been blasted enough. If scale is still seen more blasting is recommended. It is important to do this step right, so the following steps can be done easier.

b. The tank is then cleaned of all grit and paint debris. It is not necessary to vacuum the surfaces for dust, only the grit particles.

c. The tank's surfaces are then high pressure (2-3000 psi) washed with good grade of fresh water. Distilled water is not required for this washing, but industrial grade water should not be used. Fresh water with a conductivity below 400uS and chloride below 100 ppm, should be all right. If the water quality is poor, extra effort should be made to prevent the puddles that form on the tank's bottom and other flat surfaces from drying up and leaving salt deposits.

If necessary the flat bottom can be quickly rinsed with distilled or demineralized water or better grade fresh water after all other washing is completed to remove any puddles or salt deposits. The steel should be checked by limpet cell particularly in pitted areas for proper cleaning.

d. The tank is then dried with fresh air or dehumidification.

e. The tank is finally blasted with a medium/fine grit mixture.

This blast work can be done relatively quickly if all the prior steps were done properly. However, if some of the first steps were not done properly, extra effort is needed at this stage to remove any pockets of heavy contamination. This must be done by hard blasting not sweeping.

Note: The conductivity of the blast grit used during the final blast must be relatively low or it can raise the level of soluble surface contamination. The conductivity of the blast grit used during rough blasting can be higher but it is more desirable to use low conductivity grit through out all phases of blasting. Poor quality grit should not be used for the first blast as contamination may become embedded under it and this could be hard to wash out.

Note: The above rough blasting/debris removal/high pressure washing/drying/final blast procedure has been used very successfully on several chemical tankers and other vessels with very contaminated steel surfaces. This procedure was used when even repeated dry blasting could not lower the contamination level enough to permit safe coating application. .

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