Soluble salts: their incidende on the protection of metallic structures by paint coatings(·)

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Abstract

The presence of soluble salts at the metal/paint interface is known to have a detrimental effect on the integrity of most paint systems. Though this is a long-standing problem, it has recently come to receive greater attention from the protective coatings industry. In the paper the following points are reviewed: degradation mechanisms of the metal/paint system, the role of the metallic substrate, the nature, origin and detection of soluble salts, expected levels of soluble salts in practice, critical thresholds of soluble salts and risk levels for premature failures, role of the type and thickness of paint systems and exposure conditions, and prevention measures. The author presents an overview of the subject, making reference to the related research that has been carried out by him and his co-workers over the last 16 years.

Keywords

Soluble salts. Anticorrosive paints. Degradation mechanisms. Metallic corrosion. Osmotic blistering.

Sales solubles: su incidencia en alta protección de estructuras metálicas mediante recubrimiento de pintura

Resumen

Es un hecho conocido que la presencia de sales solubles en la intercara metal/pintura tiene un efecto negativo sobre la mayoría de los sistemas de pintura. Aunque se trata de un problema conocido desde hace tiempo, ha sido recientemente cuando ha recibido una gran atención por parte de la industria de recubrimientos protectores. En el presente trabajo se revisan los siguientes aspectos: mecanismos de degradación del sistema metal/pintura, el papel que juega el substrato metálico, la naturaleza, origen y detección de las sales solubles, niveles esperados de sales solubles en la práctica, niveles críticos de sales solubles y niveles de riesgo de fallo prematuro del sistema de pintura, papel que juega el tipo y espesor del sistema de pintura, el ambiente de exposición y las medidas de prevención. El autor presenta una revisión del tema, haciendo referencia a los trabajos de investigación que ha llevado a cabo, junto con su grupo de investigación, durante los últimos 16 años.

Palabras clave

Sales solubles. Pinturas anticorrosivas. Mecanismos de degradación. Corrosión metálica. Ampollamiento osmótico.

1. INTRODUCTION

It was Mayne[1] who brought this topic to light when he published his interesting article on painting rusty steel. He observed premature degradation of paint systems applied over rusty steel when this was withdrawn from the atmosphere in the winter months for laboratory painting.

Mayne demonstrated that this effect was due to the presence of ferrous sulphate in the rust, and that the failure of the paint system occurred only when the ferrous sulphate content reached a certain level (938 mg/m^2). During the winter months (at which time the atmosphere reached its highest levels of sulphur dioxide content) the ferrous sulphate content in the rust was higher, while its level was lower when the specimens were taken out in the summer months.

Though the topic lay dormant for some time, interest in studying this interesting phenomenon has grown over the last 20 years[2 and 3] to the point that much of the protective coatings

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industry currently requires testing for soluble salts, and trends show that these requirements are increasing.

This paper presents an overview of the subject, reviewing the different factors which influence the premature deterioration of paint systems due to the existence of soluble salts at the metal/paint interface and making reference to the related research that has been carried out by the author and his co-workers over the last fifteen years[4].

2. DEGRADATION MECHANISMS

The presence of water-soluble contaminants at the metal/paint interface is an accelerating factor of the metallic corrosion process. They also have a negative effect on the behaviour of organic coatings applied over contaminated substrates.

The presence of hydrosoluble species, e.g. chlorides and sulphates, at the metal/paint interface promotes osmotic blistering of the coating and underfilm metallic corrosion. Both processes can lead to the deterioration of the paint system in a very short period of time. Painting of rusty steel is prone to suffer this problem[5]. Loss of adhesion, cathodic disbondment, scribe creep and a decrease in the adhesion/cohesion strength of the coating can be also consequences of the presence of soluble salts[6].

2.1. Osmotic blistering

A coating behaves as an impermeable membrane; thus, allowing moisture but not salts to penetrate [7 and 8]. When a paint coating is applied on a metallic surface or paint layer contaminated with soluble salts, an osmotic blistering process takes place (Fig. 1). Once the painted surface has been wetted and water has permeated through the paint coating, the osmotic force generated by the difference in the concentration of the solutions on either side of the film will promote the diffusion of water through the semi-permeable paint layer from the more dilute solution (paint surface) to the more concentrated solution (salt dissolved at the metal/paint interface or between the paint layers). The process will continue for as long as there is a difference between the concentration of the two solutions. As the soluble substance dissolves under the paint layer, the pressure caused by the increase in volume can exert a greater force than the paint adhesion and cohesion forces, giving rise to the formation of a blister.
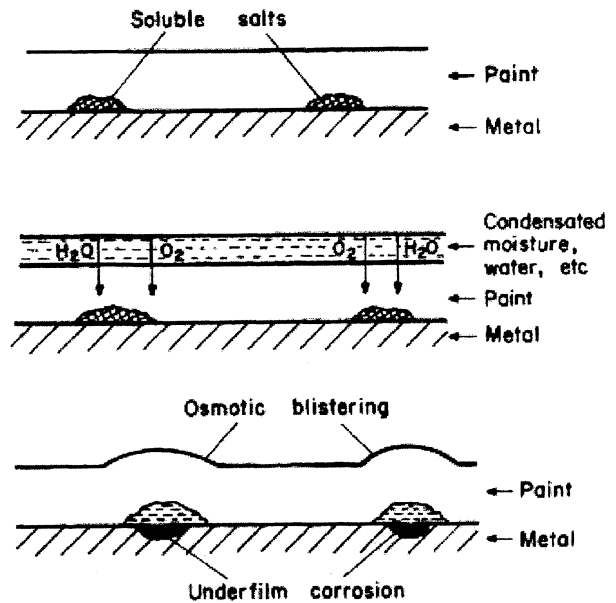

Figure 1. Osmotic blistering of the paint coating and underfilm corrosion of the metal substrate induced by the presence of soluble salts at the metal/paint interface.

Figura 1. Ampollamiento osmótico de un recubrimiento de pintura y corrosión del substrato metálico bajo dicho recubrimiento inducido por la presencia de sales aolubles en la interfase metal/pintura.

2.2. Underfilm corrosion

In the case of rusty steel, the presence of $FeSO_4$, as water soluble contaminant in the corrosion products layer, promotes an accelerated rust formation, which very quickly leads to blistering and the destruction of the coating.

Figure 2 left shows the cyclic process of rusting as caused by ferrous sulphate. Similar processes could operate in the case of other ions[10 and 11].
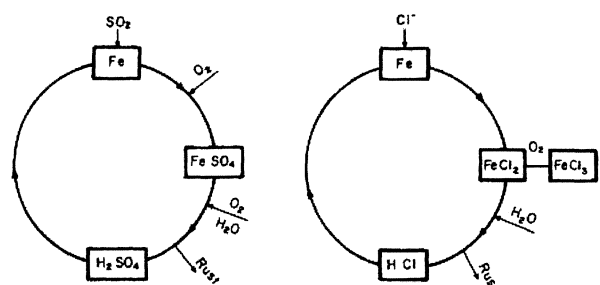

Figure 2. Cyclic mechanism of rust formation caused by the presence of ferrous sulphate[9] (left) and chlorides[3] (right) at the steel/paint interface.

Figura 2. Mecanismo de formación cíclica de herrumbre causada por la presencia de sulfato ferroso[9] (izquierda) y cloruros[3] (derecha) en la interfase acero/pintura.

220

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In the case of chlorides (Fig. 2 right), the cyclic process is similar[3].

In a search for details about the underfilm corrosion mechanism, different concentrations of contaminants (FeSO$_4$ and NaCl) and combinations thereof were applied and subsequently a wire-wound rod was used to apply a peelable (strippable) transparent varnish film, to a dry film thickness of 8 μm. The specimens were then placed in a chamber where they were exposed to an aerosol of distilled water[12]. Free films of the varnish used showed permeation rates for water vapour and oxygen (under aqueous film) of 1.2 × 10^{-2} and 0.85 × 10^{-3} g.cm^{-2}.d^{-1} respectively.

The absence of corrosion (at least not measurable with the gravimetric technique employed) for uncontaminated interfaces suggested that the controlling factor of the underfilm corrosion process on contaminated surfaces is the ionic conduction resulting from the saline deposit at the interface; low contaminant concentrations are enough to promote significant underfilm corrosion of steel after very few hours of testing. In the case of highly contaminated surfaces, ionic conduction is no longer the controlling factor of underfilm corrosion, which is now governed by the diffusion of oxygen through the varnish film.

3. UNSOLVED QUESTIONS

In the premature failure of paint coatings induced by the presence of soluble salts at the metal/paint interface, there are many questions that it would be interesting to clear up, and parallel studies aimed at clearing up these questions are lacking.

Considering the metallic surface/soluble salt/paint coating/environment system, we shall now enumerate some of these questions in relation with each of the elements of the system and related research undertaken by the author.

4. THE METALLIC SUBSTRATE

Problems reported in the literature have referred mainly to carbon steel. However, the question is whether the phenomenon can also take place on other metallic substrates. Gomez et al.[13], found osmotic blistering in paint films which had been induced by soluble zinc salts among the atmospheric corrosion products of galvanized steel, coinciding with studies carried out by M. Morcillo et al.[14] and Morcillo and coworkers[15 and 16] with galvanized steel aged in salt fog and a humid

atmosphere with SO$_2$. In this research the following conclusions were obtained:

– In general, in the case of uncontaminated weathered galvanized steel, better corrosion behaviour is seen with the paint systems applied on surfaces prepared by brushing. In contrast, blasting seems to be a better solution for surface preparation prior to painting when the galvanized steel is contaminated with soluble salts.

– Chlorinated rubber systems pigmented with micaceous iron oxide and the vinyl/polyurethane system offer the best behaviour when applied, with an adequate thickness, on preoxidized galvanized steel surfaces. These systems seem to perfectly tolerate preoxidation prior to painting when preoxidation has taken place in the absence of saline contaminants. In the case of preoxidation in the presence of chloride ions, they also present good behaviour when the surface is prepared by blasting. In the case of preoxidation in the presence of SO$_2$, no system seems to perfectly tolerate the presence of soluble salts at the interface, though in general better behaviour is observed on the specimens prepared by blasting.

– The worst behaviour is shown by the acrylic system and the alkyd system, especially the latter, which presents a high degree of paint delamination in almost all the tested conditions.

In recent research by the author on the effect on the anticorrosion behaviour of paint systems of soluble salts deposited on zinc plates, the following conclusions were obtained[17 and 18]:

– The paint systems tested show different degrees of sensitivity to the presence of soluble salts. The least sensitive are the chlorinated rubber systems pigmented with micaceous iron oxide for which no type of blistering is perceptible to the naked eye. The most sensitive are the Vinyl/Acrylic, Vinyl/Vinyl and Acrylic/Acrylic systems, while the high solids epoxy system shows intermediate behaviour.

– No effects have been detected with concentrations of less than 100 mg/m^2 of chlorides or 200 mg/m^2 of sulphates.

– A greater coating thickness can delay the appearance of blistering and slightly lessen its

Rev. Metal. Madrid Vol. Extr. (2003) 219-227

221

severity, though in general, when a certain concentration causes the blistering of a particular system, this will occur irrespective of the coating thickness.

- The humidity condensation cabinet test is a suitable accelerated test for anticipating the sensitivity of the different systems to the presence of soluble salts.

5. THE SOLUBLE SALT

There are numerous the sources that cause the existance of soluble salts over metallic surfaces. Let us cite some of them:

- Atmospheric pollution causes the presence of soluble salts within the corrosion products layer.
- Chlorides are deposited on bridges and vehicles as a result of de-icing salts applied on roads in the winter[19].
- Contamination by cleaning waters in surface treatment processes: chlorides, soluble inhibitors (e.g. chromates), etc.
- Contamination in sandblasting operations through the use of contaminated abrasives: chlorides, sulphates, fluorides, etc., either in first use or recycled.
- Contamination by paint residues, e.g. primers containing soluble chromates, pigments contaminated with soluble salts[20], etc.

5.1. Nature of contaminant

Chlorides and sulphates are not the only salts that affect corrosion. Most salts have the effect of increasing the rate of absorption of moisture from the atmosphere by lowering the critical relative humidity for condensation[21 and 22].

The author has also carried out laboratory research to try to discern the effect of different anions (Cl$^-$, SO$_4^{2-}$, NO$_3^-$) and cations (Na$^+$ NH$_4^+$, Ca^{2+}) integrating soluble salts on subfilm contamination of model coatings (transparent low thickness paint films) applied over steel plates. The conclusions reached were as follows[23 and 24].

Oxygen diffusing through the organic film and dissolving in the water layer formed at the interface is the controlling factor which determines underfilm corrosion. The structure and thickness of the coating are the main parameters in the corrosion of the steel substrate. Once the oxygen has reached the interface, the corrosion stimulant and its concentration define the underfilm

corrosion process. The kinetics of steel corrosion is governed by the osmotic pressure, ionic conductivity and oxygen solubility of the aqueous electrolyte solution and by the solubility in water of the corrosion products.

The contaminant concentration was shown to be a secondary factor since the osmotic pressure soon causes a dilution of the contaminant. The effect of the anion was shown to be preponderant over that of the cation. Thus, the order from low to high corrosion for anions was SO$_4^{2-}$<NO$_3^-$<Cl$^-$ and from high to low for cations was NH$_4^+$>Ca^{2+}>Na$^+$. In this same line of research, another study[25] indicated the following facts:

- The importance of the transfer of oxygen (controlling factor) through the organic film for the underfilm corrosion process. Alkyd films showed greater degrees of oxygen transfer than chlorinated rubber films of the same thickness (30 μm), and thus permitted greater corrosion of the base steel.
- The presence of rust together with soluble salts at the steel/paint interface slightly accelerates the underfilm corrosion process (average corrosion values are 30 %-100 % higher for the chlorinated rubber and alkyd coatings respectively).
- The lowest corrosion takes place with the NH$_4$NO$_3$ salt, while the greatest is found with sodium and ferrous chlorides.
- Underfilm corrosion increases linearly at first with the saline concentration (100-500 mg/m^2) and levels off at the highest concentrations (1000 mg/m^2).

5.2. Its distribution on the metallic surface

Salt contaminants tend to be located at the metal/corrosion products interface and at the bottoms of pits, making it difficult to eliminate them by the techniques normally used for the preparation of steel surfaces.

The identification and quantification of saline contaminants among the atmospheric corrosion products of steel has been the subject of some research. On a literature survey on this matter, Flores and Morcillo[26] draw two conclusions: (i) the concentration of saline contaminants in rust is seemingly also related to the season in which the steel is withdrawn from the aggressive atmosphere, i.e. it is subject to a seasonal effect[1, 27 and 28]. This is specially the case with sulphates, which move to

the outer rust layer in periods where sulphate nests are scarcely active[29] and can thus be readily leached by rainwater, with their concentration decreasing considerably as a result. On the other hand, the washing action of rainwater on chlorides, which tend to accumulate at the steel/rust interface, is less important, and (ii) the presence of saline contaminants in rust is closely related to the type of atmosphere to which the steel is previously exposed[27, 28, 30 and 31].

In the research that is carried out to study the effect of soluble salts on the behaviour of anticorrosive coatings it is usual to dose precise amounts of soluble salts on a certain surface. This is far from the situation that occurs in practice, where the contaminant is not distributed uniformly on the metallic surface but is distributed heterogeneously between the rust layer and frequently accumulated in the inner corrosion product layers, shallow pits under the rust layer[32 and 33], trapped between the superficial folds of the steel[34], etc.

Another study that is being carried out in our laboratories[35] aims to develop a methodology for preparing testing specimens of rusted steel with certain predetermined soluble salt levels. To this end use is made of exposure in salt fog and sulphur dioxide cabinets. The rusted and contaminated specimens subsequently undergo mechanical cleaning by brushing, simulating the manual preparation of surfaces in site painting operations.

5.3. Expected levels of soluble salts in weathered metallic surfaces

According to Alblas and van Londen[3], there is a lack of sufficient reliable data about contaminant concentrations in practical situations.

In the painting of weathered contaminated metals, and once it is known what are the critical levels of interfacial salts which lead to the premature failure of organic coatings, knowledge of the expected levels of saline contaminants in a certain situation would make it possible to predict the potential risks involved in applying paint systems under these conditions.

This information has been obtained in the MICAT Iberoamerican project[36], in which specimens of low-carbon steel, zinc, copper and aluminium were exposed outdoors for 1 year in 75 different atmospheres of the Iberoamerican region, covering a broad spectrum of atmospheric conditions (rural, urban, industrial and marine environments).

5.4. Measuring soluble salts

Much of the industry now requires testing for soluble salts, and trends show that these requirements are increasing. Laboratory and field methods for sampling and analyzing soluble salts have been developed[37-42].

Both laboratory and field testing involves a two-step procedure: (a) extraction and (b) measuring.

In research carried out by Flores et al.[43], a comparative study was made of the sensitivity and reliability of different methods for extracting and analyzing soluble contaminants on steel surfaces.

Experiments were carried out on clean unalloyed steel specimens contaminated with known amounts of chlorides and sulphates and rusted unalloyed steel specimens weathered for 1 year in 7 atmospheres of different aggressiveness.

The following conclusions were reached:

– Field sampling techniques for soluble salt concentrations (swabbing and plastic patch methods) are appropriate for use on non-rusted steel surfaces.
– Nevertheless, extractions by the plastic patch method lead to overestimated chloride concentrations, and the swabbing method results in underestimated sulphate concentrations.
– Extraction efficiency on rusted steel surfaces is considerably decreased by the difficulty of removing contaminants that usually accumulate at the steel/rust interface.
– The extraction procedures that use a chloride indicator test strip and the ferrous sulphate indicator test strip for determining soluble contaminants are fairly accurate with non-rusted steel but provide highly underestimated values for rusted steel.

The literature contains reviews[44 and 45] and assessments of the different detection and analysis methods[3, 6, 37, 41, 42 and 46]. The results of detection methods are affected by temperature, mechanical forces, and the chemicals and type of analytical methods used[3].

On the other hand, extraction efficiency depends on extraction time, steel surface conditions (roughness, degree of rusting, etc.), salt concentration on the steel surface,[6] etc.

Rev. Metal. Madrid Vol. Extr. (2003) 219-227

223

The current problems with regard to the extraction and quantification of soluble salts contents on surfaces that are subsequently to be painted can be summarized in the following points:

– Improvement of the extraction coefficients and of the measuring devices in existing commercial procedures.
– Field quantification of the partial soluble salt contents (sulphates, chlorides and nitrates), rather than conductivity measurements of the aqueous extract.

Recent research in our laboratories[47] has focussed on attaining these objectives.

5.5. Guidance levels for soluble salt concentrations

A systematic field work in relation with this issue was carried out by the author more than 15 years ago[5, 48 and 50].

No blistering was apparent on the control panels where paint systems were applied over uncontaminated blasted steel. However, deterioration occurred whenever the level of NaCl at the interface was 500 mg/m^2. In the case of $FeSO_4$ the critical level was 2500 mg/m^2.

In an interesting review by Alblas and Van Londen[3] of studies carried out with chlorides as interfacial contamination it is commented that despite the lack of consensus on precise contamination levels, all researchers agree that soluble salts have a dramatic effect on coating performance. At present, a maximum allowable chloride level of 10 to 50 mg/m^2 can be taken as a safe critical value or a rough guideline depending on usage and exposure conditions. A great

dispersion of the maximum allowable chloride concentrations from experimental studies was found[3]. However, many of these studies have been conducted in the laboratory and there is no clear correlation between the results of the traditional accelerated tests and the results of outdoor corrosion tests.

Much time and many conversations have passed in trying to determine acceptable concentration levels. It is difficult to set acceptable levels since each type of coating and thickness varies in susceptibility to soluble salt degradation. The types of services encountered also vary. ISO/TC35/SC12/WG5 [51] has for some time been trying to develop a standard (ISO-15235-1[52]) about guidance levels for water-soluble salt contamination before the application of paints and related products. Table I shows the critical levels of saline concentration, by chlorides and sulphates, found in atmospheric tests by different researcher, above which there is a significant deterioration of the paint coating.

5.6. Removal of soluble salts

Researchers have also failed to achieve the complete elimination of these corrosion promoters through abrasive blasting. The residual ferrous sulphate remaining on the steel surface decreased as the blasting time increased and the grit size decreased.

According to McKelvie[9] it could be expected that wet blast cleaning techniques (with and without extrained abrasive) are more effective than dry blast techniques for the removal of soluble contaminants. Because of capillary action in deep and narrow pits, a slow diffusion action may be more appropriate than a high pressure water blast.

Table I. Critical levels causing an anticipated deterioration of the paint systems in atmospheric exposures

Tabla I. Niveles críticos que causan un deterioro prematuro de los sistemas de pintura en exposiciones atmosféricas

Reference	Atmosphere	Time years	N° of paint sytems	Thicknesses µm	Critical levels mg Cl^-/m^2	Critical levels mg SO_4^{2-}/m^2
Mayne 1959 [1]	Urban	5-7	1	100-150	–	588
Morcillo 1987 [5]	Rural, urban and industrial	14	10	60-190	60-300	–
Morcillo 1997 [49]	Marine	4	4	150-180	–	630-1580
Boocock, 1991 [37]	Marine	3	7	150-380	300-500	–

224

Rev. Metal. Madrid Vol. Extr. (2003) 219-227

6. THE PAINT SYSTEM

Coating degradation induced by the presence of soluble salts at the metal/paint interface depends upon osmotic water migration through the coatings, that can lead in many cases to blistering and delamination of the coating. Thus, it is dependent on the thickness and the types of binders, fillers, pigments and other additives included in its formulation. Svoboda reported in 1977[20] the harmful effect of the existence of water-soluble materials in the pigments used in paint formulations.

The alternatives currently available for this situation are as follows:

– Use of very thick coatings (mastic) which are extraordinarily effective in preventing the entrance of water and oxygen molecules through the organic coating. In this way there is a limitation to the development of osmotic blistering and underlying corrosion processes which lead to the premature deterioration of the paint system. The disadvantage of this type of coatings is their high cost and the difficulty of their application.

– Use of "inertisating" pigments. The inertisation of aggressive rust species (Cl^-, SO_4^{2-}) can be achieved by means of different mechanisms: (i) the formation of insoluble compounds through the chemical reaction of some pigments with saline species. The literature contains some somewhat old studies[53-55] in relation with this, with promising results, and (ii) zinc rich paints (ZRP)[48-50]. Recent research in our laboratories[56], aimed at determining the inerting capacity of different phosphates included in ZRPs, confirmed the good behaviour of ZRPs in the painting of steel substrates contaminated with soluble salts, as had previously been observed in other studies[48-50], as well as the good behaviour of certain phosphates included in paint formulations.

7. THE ENVIRONMENT

What role is played by the environment in which the paint coating applied on a substrate contaminated with soluble salts is to be exposed?

Much of the information available has been obtained in atmospheric exposure tests.

In immersion conditions in different solutions (distilled water, fresh water, sea water, etc.), the most aggressive solution is distilled water, in which osmotic processes develop with greater intensity, though saline solutions can also cause rapid blistering of the paint systems, Ships' hulls, ballast tanks, pipelines, etc. are metallic constructions which are often subject to early deterioration of paint system due to the presence of soluble salts at the metal/paint interface.

The Bristish Ship Research Association (BSRA)[57] carried out a study in sea water in order to relate the degree of paint system blistering with the type of paint, film thickness, and the level of interfacial salina contamination. For the paint thicknesses which are normally applied in these situations (~ 250 µm) blistering was not generally observed at saline contamination levels, with chlorides and sulphates, below 500 mg/m^2.

Morcillo et al.[50] tested paint systems in sea water for four years, in total immersion and boottop zone conditions, however the degradation effect caused by soluble salts was not seen as clearly as in the atmosphere.

Another matter of interest would be to know the role played by the saline concentration in the environment in the osmotic blistering processes promoted in the presence of soluble salts at the metal/paint interface. To this end research has been carried out[58] in which two paint systems applied on steel contaminated with different sodium chloride levels (0 to 500 mg Cl^-/l) were exposed to sodium chloride aqueous solutions of different concentrations (0-40 mg Cl^-/l). The main conclusions reached in this study were as follows:

– The presence of saline contamination at the interface produces a notable change in the morphology of the paint coating's deterioration. The voluminous blistering (without apparent attack of the base metal) that takes place when the paint system applied on non-contaminated steel is exposed to sodium chloride aqueous solutions changes to a fine blistering with abundant rust formation when there is saline contamination at the interface. This change takes place clearly when the threshold of 50 mg Cl^-/m^2 is exceeded.

– No great influence of the exterior saline concentration is observed on the osmotic deterioration of the paint system promoted by the existence of contamination at the steel/paint interface. The only change observed is perhaps a slowing of the deterioration process

Rev. Metal. Madrid Vol. Extr. (2003) 219-227

225

as the saline concentration in the exterior environment increases.

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226

Rev. Metal. Madrid Vol. Extr. (2003) 219-227

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Rev. Metal. Madrid Vol. Extr. (2003) 219-227

227