Technical Know-How Update

Salt Contamination

Introduction

A salt can be defined as an ionic chemical compound that dissolves in water to form a solution of positively charged cations and negatively charged anions.

The industrial environment has many sources of salt contaminants, both natural and man-made. Chlorides (Cl^-) are the most common, being present in marine environments, water treatment and de-icing products. Sulphates (SO_4^{2-}) are present in many natural sources and are generated from gas and diesel emissions. Nitrates (NO_3^-) are present in fertilisers and auto emissions. There can also be salt contaminants present in the blast media used to prepare metal surfaces, and salts can be found on the surface of new metal which has been exposed to chlorides or sulphates from coastal environments during fabrication or transportation.

The presence of soluble salts as contaminants on metallic substrates can result in premature coating failure when present in sufficient concentrations, particularly in hot immersion applications. Typically, salts are not completely removed by blast cleaning and, in fact, can sometimes be driven into the blast profile of the metal by blast cleaning.

When coatings are applied over soluble salt contaminated steel, subsequent moisture migration (normal for coatings in humid or immersed environments) in and out of the coating film can result in moisture solubilising the salts and condensing on the steel surface under the coating film (Figure 1). Eventually this creates small osmotic cells pulling more moisture toward the concentrated solution and causes osmotic pressure to develop (Figure 2). This pressure pushes the coating off the surface, creating blistering of the coating with subsequent delamination (Figure 3).

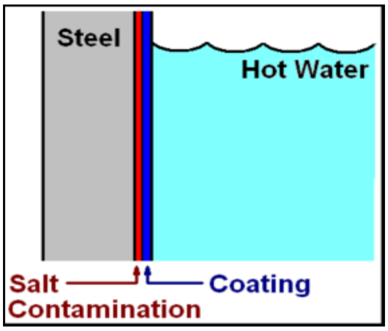

Figure 1

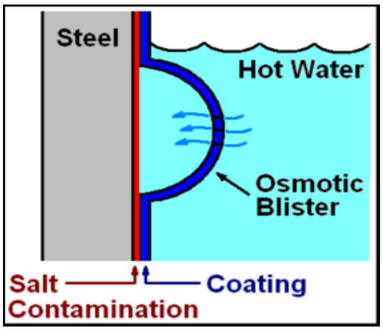

Figure 2


Figure 3

The presence of soluble salts on the metallic substrate will also promote under-film metallic corrosion and further blistering/disbondment of the coating when the concentration of soluble salts exceeds a critical level.

To ensure the success of elevated temperature immersion coatings on metallic substrates it is therefore necessary to measure surface salt levels, and then to reduce those salt levels if they are found to be unacceptable.

Methods for the Determination of Salt Levels

Measuring the degree of salt contamination present on a substrate prior to coating is critical to the long-term performance of the application. It is therefore very important to be able to quantify the level of soluble salt present and there are a number of recognised test methods available for this.

1. Elcometer 138 Bresle Salt Contamination Test Kit

The Elcometer 138 Bresle kit includes all the materials and equipment required to determine the surface salt contamination level on a metal substrate, and complies with ISO 8502-6 and US Navy PPI 63101-000 (Rev 10).

Soluble salts are extracted from the substrate surface using an adhesive Bresle patch and the total dissolved salt (TDS) content of the resultant solution is measured using a conductivity meter provided in the kit. The higher the conductivity reading, the higher the salt concentration. The surface density of salts is then calculated by multiplying the conductivity value by a factor in order to obtain a value for surface density of salts in milligrams per square metre (mg/m^2)[1].

Each kit contains 25 test patches, though more can be purchased separately. Refills for the different solutions are also available. The test duration for the Elcometer 138 is in the region of 40 minutes when conducted according to the ISO 8502-6 standard, and the test can be performed on vertical, horizontal or overhead surfaces.

This test is recommended by Belzona as it is easy to use, cost-effective and provides accurate results.

2. Elcometer 134S Chloride Detection Kit

Like the Elcometer 138 Bresle test, the Elcometer 134S kit is designed to be used on blast-cleaned metal surfaces and complies with ISO 8502-6. However, this kit measures chloride ions only.

The chloride salts are extracted using a rubber tube and Chlor*Extract solution. The level of chloride on the surface is identified using a simple glass tube which shows the concentration level in the range 1 – 60 parts per million (equivalent to $\mu g/cm^2$).

3. Elcometer 134 CSN Test Kit for Chlorides, Sulphates and Nitrates

This test is designed to accurately measure surface levels of chloride, sulphate and nitrate ions from a single test sample. Results are reported in parts per million ($\mu g/cm^2$) and the test complies with ISO 8502-5, 8502-11, SSPC SP TU4 and NACE 6G186.

This is a relatively expensive method of testing however, and each kit only contains enough equipment for five tests.

[1] Note that some test methods report in mg/m^2 whereas others report in $\mu g/cm^2$. To convert from $\mu g/cm^2$ to mg/m^2 multiply by 10.

4. Elcometer 130 SCM400 Salt Contamination Meter

This method uses the SCM400 Salt Contamination Meter, and provides an extremely accurate analysis of salt contamination on a surface, though it is comparatively expensive. Soluble salts on a surface are absorbed into a special filter paper soaked with distilled water. The conductivity of the wet filter paper is then measured and the salt level subsequently calculated and displayed in units of µg/cm² (range 0.1 – 20 µg/cm²).

Salt Removal Solution

Salt-Away is a water-based, non-hazardous, biodegradable salt removal solution which can be used to dissolve and remove salt crystals from surfaces if levels of contamination are unacceptable. Salt-Away has a dense foaming action when applied, which acts as an indicator where it has been applied and helps the solution cling to surfaces to slowly soak through layers of salt build-up. Included in the Salt-Away formula are rinse aids and liquid/vapour corrosion inhibitors which help to readily dissolve the Salt-Away residue off the surface and resist the formation of flash-rusting after treatment.

When Salt-Away is rinsed off the substrate salt crystals are lifted off and washed away. It must be noted however, that Salt-Away is only designed to remove chloride salts and will not have an appreciable effect on sulphates or nitrates[2].

Salt-Away has been found to be the most effective salt-removal treatment on the market for use with Belzona coatings[3]. It does not cause flash-rusting and does not have any detrimental effect on the performance of Belzona coatings if used correctly.

Figure 4

Figure 5

Application of Salt-Away

Figures 6-10 below illustrate the steps involved in the recommended use of Salt-Away on a salt contaminated steel panel.

- Figure 6 shows the salt contaminated panel[4] before treatment. Note the heavy rusting caused by salt-water exposure.

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[2] The most effective treatment for removal of sulphates or nitrates is ultra high pressure (UHP) water jetting.
[3] Although Salt-Away is Belzona's preferred salt removal product based on performance testing in the lab, Chlor*rid International offer similar treatments which have also been found to be acceptable.
[4] The panel had been subjected to 1000 hours continuous salt spray in 5% sodium chloride solution at 35°C.

- Figure 7 shows the Salt-Away being applied to the contaminated panel using a trigger spray dispenser.
- Figure 8 shows the use of a brush to work Salt-Away into surface of the panel (note the foaming action) before leaving for 10-30 minutes depending on the severity of contamination and substrate orientation[5].
- Figure 9 shows rinsing of the panel with deionised water[6] for a period of one minute to remove the Salt-Away residue.
- Figure 10 shows the finished surface after grit-blasting (note no flash-rusting).

Figure 6

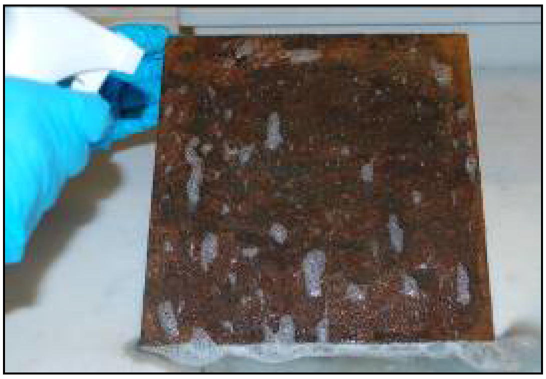

Figure 7

Figure 8

Figure 9

Figure 10

For most effective salt-removal it is recommended that Salt-Away be used at a concentration of no less than 18% (the “ready to use” form of the product). Weaker solutions of Salt-Away have been found to be less effective.

[5] A 10 minute Salt-Away treatment is typically sufficient for horizontal surfaces, but a 20-30 minute treatment is recommended on vertical or overhead surfaces.
[6] Note that it is very important to carry out rinsing with distilled or deionised water, i.e. use water that does not contain high levels of salts. Test kits are available for checking that wash water is not high in chlorides, e.g. the Elcometer 134W.

As a guide, one litre of diluted Salt-Away should be sufficient to treat an area of 5-6 m^2.

Note: Residual salt levels must always be checked after treatment (e.g. using an Elcometer 138 Bresle kit) to confirm that levels are suitably low for the intended application/service temperature.

Lab Testing

The effectiveness of Salt-Away as a salt removal system has been extensively tested on mild steel. An illustration of the performance of Salt-Away can be found below.

A salt-contaminated test panel was prepared by immersing a mild steel plate in 5% salt solution (sodium chloride) for a period of 9 months at 20°C. The surface salt contamination level of the panel was measured using an Elcometer 138 Bresle kit in order to establish a baseline value, then a variety of surface preparation/cleaning regimes were employed to remove the salt. After each cleaning phase the surface salt levels were retested. For results see Table 1 below.

Cleaning Regime	Salt Contamination Level, mg/m²
No treatment (control – baseline salt level)	1404
One grit-blasting cycle	240
Two grit-blasting cycles	167
Three grit-blasting cycles	121
Wash with deionised water + one grit-blasting cycle	56
Two deionised water washes + two grit-blasting cycles	13
Single treatment with Salt-Away[7] + one grit-blasting cycle	12

Table 1

This test work illustrates that blast-cleaning alone is typically insufficient to reduce salt contamination down to satisfactorily low levels. A combination of blast-cleaning and water washing can reduce contamination to low levels, but multiple blast/wash cycles are normally required which are time consuming and often unacceptable. Salt-Away, however, is capable of significantly reducing the salt contamination level with just one treatment.

Testing of Belzona coatings applied onto Salt-Away treated surfaces has shown that there is no detrimental effect on coating performance. Lab testing has showed that there is no noticeable drop in adhesion (ASTM D4541/ISO 4624 dolly pull-off test) or hot immersion resistance (NACE TM 0174 Atlas cell test) when tested with a variety of Belzona coatings.

Acceptable Salt Limits for Belzona Coatings

<u>Chlorides</u>

Belzona has carried out research to determine the acceptable chloride salt contamination levels for Belzona immersion coatings. Testing was based on artificially-contaminated steel panels (using sodium chloride solutions) and the Elcometer 138 Bresle salt test. A range of salt contamination levels between 20-150 mg/m^2 of chloride were prepared and overcoated with a number of Belzona coatings and tested for Atlas cell immersion resistance (NACE TM 0174) at a range of temperatures.

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[7] Treatment using an 18% concentration of Salt-Away for 10 minutes, followed by a 1 minute deionised water wash.

As expected, it was found that salt contamination becomes more critical as immersion temperatures increase. Based on this work it was possible to generate recommended maximum levels of surface chloride contamination for coatings operating in immersed conditions[8]. See Table 2 below.

Immersion Temperature, °C	**Maximum Acceptable Level of Surface Chloride Contamination, mg/m²**
20	60
50	30
90	20

Table 2

It must be noted that the above recommendations for acceptable contamination levels have been based solely on chloride testing. This is because chlorides are the most commonly encountered type of salt contaminant, and are also the most damaging.

They are more damaging than sulphate or nitrate salts because of the lower molecular weight of the chloride ion. In other words, for the same mass of contamination, there will be comparatively more chloride ions than sulphate or nitrate ions, with the actual number being proportional to the molecular weights of those ions[9]. This effectively means that chloride ions – and therefore the limits stated in the above table – represent a worst-case scenario.

So, if the level of total dissolved salt (e.g. as measured by an Elcometer 138 Bresle test) is less than or equal to the figure in the above table (for chlorides), it will always be safe to proceed with that application.

However, it may also be safe to proceed even if measured contamination levels are higher than those listed in the above table, provided that some or all of the dissolved salts are sulphates or nitrates. To determine if levels of mixed salts are acceptable the calculation described below should be used.

<u>Sulphates and Nitrates</u>

The level of sulphate/nitrate contamination can be determined directly using an Elcometer 134 CSN Kit. However, this detection equipment is comparatively expensive and may not always be available. Alternatively, levels of sulphate/nitrate contamination can be determined using an Elcometer 134 kit (to measure total dissolved salts) combined with an Elcometer 134S detection kit (for chlorides). The level of sulphate/nitrate salt contamination may then be determined by difference, i.e. total dissolved salt in mg/m^2 minus chloride salts in mg/m^2.

To determine if the level of mixed salt contamination is acceptable, in this case for 90ºC immersion, the following calculation should be used:

$$\frac{[Cl^-]}{20} + \frac{[\text{Other salts}]}{35} = \leq 1$$

Where; $[Cl^-]$ is the level of chloride contamination present in mg/m², and
[Other salts] is the level of combined sulphate/nitrate present in mg/m²

[8] Results first communicated in Belzona Technical Information No.180/06 – 7th July, 2006.
[9] The comparative ion molecular weights are 35 g/mol for chloride (Cl^-), 62 g/mol for nitrate (NO_3^-) and 96 g/mol for sulphate (SO_4^{2-}).

If the answer to the equation is one or less, then the combined salt contamination level is acceptable.

Note that if the immersion temperature is lower than 90ºC then increased levels of salt contamination are acceptable and the calculation will be slightly different:

- For immersion temperatures of ~50ºC, replace the 20 and 35 figures with **30** and **53** respectively.
- For immersion temperatures of ~20ºC, replace the 20 and 35 figures with **60** and **106** respectively.

These adjusted figures are based on the measured limits for chlorides from lab testing and the molecular weight of the nitrate ion (the next worst-case scenario after chlorides).

<u>Example Equations</u>

Example #1: Using the Elcometer 138 Bresle test kit, the total dissolved salt value for a surface to be coated was found to be 22 mg/m². However, after testing with an Elcometer 134S test kit the chloride contamination level was found to be only 17 mg/m². The level of nitrate/sulphate contamination was therefore 5 mg/m² (22 – 17 = 5). Using the equation to test if this salt level is acceptable for an application at 90ºC produces the following result:

$\frac{17}{20} + \frac{5}{35} = 0.99$ Result is <1, therefore level of salt contamination is acceptable

Example #2: Again using the Bresle test kit, the total dissolved salt value for a surface to be coated was found to be 35 mg/m². Subsequent Elcometer 134S testing found the chloride level to be 18 mg/m². Therefore the level of other salts present was 17 mg/m² (35 – 18 = 17). Using the equation to test if this salt level is acceptable for an application at 90ºC produces the following result:

$\frac{18}{20} + \frac{17}{35} = 1.38$ Result is >1, therefore the level of salt contamination is not acceptable

Summary of Application Procedure

Belzona’s recommended procedure for measuring and removing salt contamination on metallic surfaces can be summarised as follows:

- Test the substrate for soluble salt contamination using an Elcometer 138 Bresle test kit. This will report total salt level, i.e. combined chlorides, sulphates and nitrates.
- If this figure is below the acceptable threshold for chlorides at the anticipated service temperature (e.g. ≤20 mg/m² for 90ºC service – see Table 2) then it is fine to proceed with the application.
- It may also be ok to proceed if salts such as nitrates/sulphates are present rather than chlorides, as these salts are less aggressive than chlorides. If nitrates or sulphates are detected (using an Elcometer 134 CSN kit, or by difference using an Elcometer 134S chloride test kit) then the equation described above can be used to determine whether the total salt level is acceptable.
- If tested salt levels are found to be high (or if levels are simply anticipated to be high based on the substrate, e.g. a brine tank), then they must be reduced before coating.

This can be done using a combination of washing with deionised/distilled water and grit-blasting, though multiple cycles may be required to reduce salt to an acceptable level. Alternatively an effective salt-removal treatment such as Salt-Away can be used.

- Apply the Salt-Away (as a minimum 18% concentration) onto the surface to be cleaned and scrub into the surface to create a foaming action. Leave for 10-30 minutes before rinsing the residue away using deionised/distilled water.
- Then grit-blast the surface to prepare for coating (ensuring that the blast media used does not contain salt contamination).[10]
- Carry out a final Elcometer 138 Bresle test to confirm that salt levels are acceptable and the surface is ready for coating.

Product Availability

Salt-Away is manufactured in the US but is available from a network of international distributors. See http://www.saltawayproducts.com/InternationalDistrib.htm for details.

Although Salt-Away is Belzona's preferred salt removal product, treatments from Chlor*rid International have also been found to be acceptable.[11]

Key Points

- The presence of soluble chloride, sulphate and nitrate salts on metallic substrates can result in premature coating failure, particularly in hot immersion conditions.
- The degree of salt contamination present on a substrate can be measured in a number of ways, though the test recommended by Belzona is the Elcometer 138 Bresle test. This is used to measure the total dissolved salt level (i.e. combined chlorides, nitrates and sulphates) on a substrate.
- Salt-Away is a water-based chloride removal solution which dissolves, releases and removes salt crystals from contaminated surfaces. This product has been found to be very effective and has no detrimental effect on the immersion performance and adhesion of Belzona coatings.
- Acceptable salt limits for Belzona coatings have been defined based on lab testing. These recommendations are based on testing of chlorides, since these salts represent a worst-case scenario for osmotic blistering. The acceptable thresholds for salt contamination have been found to decrease with increasing service temperature, down to 20 mg/m^2 for coatings operating at ~90ºC.
- For mixtures of salt contamination types (chlorides, nitrates and/or sulphates) the equation described above in this document should be used to determine if measured salt levels are acceptable.

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[10] An Elcometer 134A chloride test kit for abrasives can be used to check if blast media is contaminated. Note that certain media, e.g. garnet, can be relatively high in salt.

[11] A list of distributors offering alternative Chlor*rid International products can be found at http://www.chlor-rid.com/distributors/index.php