Establishing Bresle Patch Equivalence (ISO8502-9 Method) for the new Elcometer 130 SSP

1. Foreword

The purpose of this test method is to establish equivalence to the Bresle Patch Method for the new Elcometer 130 SSP, as defined by ISO 8502-9.

Equivalency is to be determined by testing and comparing results of the Elcometer 130 SSP to the method specified in ISO 8502-9 across a number of salt contaminated steel panels of various blasted profiles.

2. Introduction

Soluble salts, which are often invisible to the eye, can result in premature corrosion, leading to coating failures – resulting in high re-coating, maintenance costs, asset downtime or availability.

In order to show equivalency of measurement between any two test methods it is important to ensure that no other parameters change except the gauges under test. For equivalency to be established, both gauges should read a similar value, taking into account the accuracy and resolution of each gauge.

Whilst both the Bresle Patch Test Method and the Elcometer 130 SSP use different techniques, they both measure the concentration of soluble salts on a substrate using the conductivity method. This requires that the salts have to first be removed from the substrate using deionised water, and the resulting solution tested using a conductivity meter. The higher the level of conductivity of the resulting solution, the higher the level of soluble salt concentration.

As both test methods (Bresle and Elcometer 130 SSP) require the removal of the soluble salts from the substrate, each instrument must measure on different test areas (as the salt would have been removed from the first test area).

This requires the development of a repeatable, reproducible and uniform method for doping a clean substrate.

Research undertaken by Elcometer has established that the NACE SP0508-2010 method for determining a gauge's equivalency to the Bresle Patch Method is not valid. The recommended doping method within NACE SP0508-2010 produces wide variations of salt concentration across a test panel – making comparisons between instruments impossible. Additionally, some of the requirements set down within NACE SP0508-2010 require measurements on surfaces that would be wholly unacceptable for a coating to be applied – namely corroded steel.

With this in mind, Elcometer, with the help of the School of Materials at The University of Manchester (UK), has developed an automated, repeatable and reproducible doping method which can accurately apply a wide range of concentrations to a variety of substrates. For more information on the Elcometer Doping Method see Appendix 1.

In addition to developing an automated, more consistent doping method, new parameters for establishing equivalency have been developed which are more appropriate to the industrial application in question, incorporating salt concentration and surface profile.

In order to ensure impartiality of the test results, Mr C Molloy, an undergraduate in BSc Physics at the University of Edinburgh, was brought in to independently undertake the tests and generate the report.

3. Method

The following test considerations have been used to establish equivalency to the Bresle Test Patch method.

3.1.Surface Salt Concentration

The level of 'acceptable' threshold concentration levels of soluble salts on a ferrous substrate, within the Protective Coatings Industry, are as follows:

NAVSEA	009-32 FY-12	30 – 50 mg/m²
NORSOK	M-501	20 – 50 mg/m²
DNV	RP-F102	20 mg/m²
IMO	MSC.215(82) & MSC.244(83)	50 mg/m ²

To determine equivalency, this test method will dope panels at five ranges of surface salt concentration levels:

- 1) 15 mg/m^2 to 25 mg/m^2
- 2) 25 mg/m^2 to 35 mg/m^2
- 3) 35 mg/m^2 to 45 mg/m^2
- 4) 45 mg/m^2 to 55 mg/m^2
- 5) >55 mg/m²

3.2.Salt Ratio

Each panel will be immersed in deionised water to remove all residual soluble salts and impurities prior to the doping process. The salt solution used will be industry recognised reference for salts, as defined by NACE SP0508-2010.

The following mass ratio of salts shall be used:

- 24.3% Na₂SO₄
- 22.1% NaNO₃
- 53.6% NaCl

Deionised water with a maximum conductivity of $3.00 \,\mu$ S/cm shall be used for both the Bresle Patch Test Method and the Elcometer 130 SSP. Any background conductivity will be measured, recorded and deducted from the measurements.

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3.3.Surface Profile

Common practice within the United States of America dictates that coatings are only applied to blast cleaned surfaces, free of rust or corrosion. Blasted profiles within the Protective Coatings Industry typically have a nominal peak-to-valley height of either 50 μ m, 75 μ m or 100 μ m (2.0, 3.0 and 4.0 mils). European practice may also apply a protective coating up to a nominal 150 μ m (6 mils). This practice is being considered in the US as well.

To determine equivalency, this test method will dope panels at the following nominal steel grit blast profiles:

- 25 to 50 µm (1.0 to 2.0 mils)
- 50 to 75 μm (2.0 to 3.0 mils)
- 75 to 150 μm (3.0 to 6.0 mils)

In addition to blasted profiles, a clean coated panel will also be tested to establish equivalency for inter-coat assessment.

The blasted profile of each uncoated steel panel will be measured using a certified Elcometer 224C Digital Surface Profile Gauge (using the average of 40 measurements on each panel), to determine the average peak-to-valley height, in accordance with ASTM D 4417 Method B.

3.4.Preparation of Salt Contaminated Test Panels

The test panels shall be prepared to the specified surface salt density levels using the following parameters:

- Test panel size: 300 x 400 mm (11.81 x 15.75 inches)
- Clean the test panels via immersion in deionised water
- Dry the panel rapidly by blowing off the water with a heat gun (to reduce flash rusting).
- Each test panel will be nominally contaminated as follows:
 - \circ 3 µl of salt solution spaced on a 4 mm grid across the test panel.

3.5.Method of Doping of Test Panels

This section lists the doping method of the test panel.

- Place the test panels onto a heating element
- Set the heating element's temperature controller to 70 °C and leave the temperature to stabilise for two minutes after the temperature controller has met the target value.
- Initiate the robot software with the gridded programme which takes just short of 3 hours to complete.
- Upon completion remove the panel from the heating element (protective gloves maybe required!)
- The Panels will be carried to a nearby test area in an enclosed plastic box.
- The Panels will be kept in the enclosed plastic box until they are tested.
- The measurements will be taken within 72 hours of the completion of the salt contamination process.
- All measurements will be completed in the same test area.

3.6.Arrangement of Bresle Patches & Elcometer 130 SSP Filter Papers

Each Test Panel will be split into 4 test areas of 150 x 200 mm (5.91 x 7.87 inches). 4 x Bresle patches (B1, B2, B3, B4) and 1 x Elcometer 130 SSP filter paper (FP1) will be applied to each test area in the following arrangement:



Figure 1 : Bresle Patch and Elcometer 130 SSP Layout

As Bresle patches can leak under test, in order to preserve the area under test, all four Bresle patches will be adhered to the doped test panel. The correctly prepared Elcometer 130 SSP filter paper will then be applied and measured in accordance with the manufacturer's instructions. In this way, the foam seal around each Bresle patch will preserve the doped surface during the Elcometer 130 SSP filter paper test. Each Bresle patch will then be tested in turn.

Whilst each Bresle Test Patch provides a single value, the Elcometer 130 SSP measures and records four Bresle equivalent areas and the average of all four (4). All measurements will be recorded.

Ambient temperature and relative humidity will be recorded prior to testing. All testing will be carried out in laboratory conditions.

3.7.Test Measurements

Whilst all efforts have been taken to ensure each test panel is consistently doped, and each test is carried out under laboratory conditions, in the eventuality that a single reading (either Bresle Patch or a Bresle Equivalent area) is significantly different to the surrounding measurements on the test area, the test panel will be treated as invalid and discarded. The measurement results will be recorded separately for information purposes only.

In order to establish equivalency between the Bresle Patch Test Method and the Elcometer 130 SSP, four (4) valid test areas will be undertaken for each concentration and for each surface profile.

3.8.Background contamination

Whilst every effort will be made to clean each test panel prior to doping (immersion in deionised water), there are other background contaminants to take into consideration:

3.8.1. Bresle patch contamination

Bresle patches can suffer from different levels of background contamination. The following method will be used to determine the background contamination of the Bresle patch batch.

- Bresle patches from boxes of 25 will be used. As every doped test panel will require 12 Bresle patches, a spare Bresle patch will be available each time that the testing of two test panels is completed. This spare Bresle patch will be used to measure the Bresle patch background contamination.
- Remove the foam centre from the patch, and fold it in half so that the adhesive edges are stuck together. Squeeze the centre section of the patch as it is folded to remove the majority of the air from the centre. Press the edges tightly together to ensure it is sealed.
- Using 3 ml of deionised water, inject into the cavity of the folded Bresle patch.



Figure 2 : Determining the Bresle Patch Contamination

- Agitate for 90 seconds, remove the contaminated water and measure its conductivity.
- The patch contamination can be calculated as follows:

 γ (patch contamination) = γ (extracted solution) - γ (injected solution)

Where:

 γ (*patch contamination*) = conductivity attributable to patch contamination in μ S/cm γ (*injected solution*) = conductivity of the injected solution in μ S/cm γ (*extracted solution*) = conductivity of the extracted solution in μ S/cm

Then convert the patch contamination conductivity value to an equivalent salt density using the equation:

$$\rho_A = c * \frac{v}{A} * y$$
 = 5 * 3/12.5 * y = 1.2 * y

Where:

 ρ_A is the equivalent surface salt density of the salts in mg/m² c = 5.0 kg*m⁻²*S A = 12.5 cm² (surface area of a A-1250 Bresle patch) V = Volume of injected solution (3 ml) y = patch contamination conductivity in μS/cm

• Record the equivalent surface salt density of this contamination and use it to calculate an average background salt density value for each batch of Bresle patches used.

3.8.2. Deionised Water concentration

Deionised water with a maximum conductivity of $3.00 \,\mu$ S/cm shall be used for both the Bresle Patch Test Method and the Elcometer 130 SSP tests. Any background conductivity will be measured, recorded and deducted from the Bresle Patch Test Method measurements. The Elcometer 130 SSP will automatically adjust its measurement to take account of this background conductivity once the "offset procedure" has been followed. See section 3.8.3

3.8.3. Elcometer 130 SSP filter paper and water, offset procedure:

As per the manufacturer's operating instructions, when a new box of filter papers is opened or a new container of deionised water is used, the filter paper and deionised water offset needs to be set on the Elcometer 130 SSP.

- Extract, with tweezers, a filter paper from the box.
- Place the dry filter paper onto the Elcometer 130 magnetic disc which has been cleaned using deionised water and sensor wipes.
- Fill the syringe completely with deionised water and discard it. Perform this rinsing three times.
- Fill the syringe with 1.6 ml of deionised water. Spread the water, from the syringe, evenly across the whole of the Elcometer 130 SSP filter paper. Best achieving by starting in the middle of the filter paper and working out two the edge using multiple drops. Then tilt the magnetic disc as necessary until the water is evenly spread across the filter paper.
- Place the magnetic disc with filter paper onto the Elcometer 130 SSP measuring electrodes (which has been cleaned using deionised water and sensor wipes).
- On the gauge select *Menu\Setup\Calibration\Calibration Offset* and follow the screen instructions. When complete the gauge reports "Calibrated", press Ok and return to the Normal Reading Screen.
- With the magnetic disc and filter paper on the measuring electrodes take a reading. Check that the reading is no more than $1 2 \text{ mg/m}^2 (0.1 0.2 \mu \text{g/cm}^2)$

4. Data Analysis

When the results are taken they will conform to the following criteria:

4.1.Elcometer130SSP to Bresle Patch Data analysis

To be deemed equivalent to the Bresle Patch Method the Elcometer 130SSP results shall meet the following requirements:

The average of the four Bresle readings in each test area will be within ± 9 mg/m² of the average filter paper reading for that area (which ever is the greatest).

4.2.Conversion Factor

An internal conversion factor will be used to bring the Elcometer 130 SSP results in line with the measured Bresle Patch Method results across all surfaces and all salt densities. The conversion factor will not be reported.

5. Results Summary

The Results displayed in Appendix two represent the average values of 5 test areas – a test area is defined as 4 Bresle Patches and 1 Elcometer 130 SSP filter paper (which is the average of 4 Bresle equivalent areas).

Each column in the graphs in Appendix 2 represents the average of 5 individual tests.

6. Conclusion

The Elcometer 130 SSP has undergone extensive side by side comparison testing against the Bresle Test Patch Method.

- Background (inherent) contamination within the Bresle Test Patch has shown that the Bresle Test Patch has a background contamination range of 0.88µg/cm² - see Appendix A2.2
- The variation in readings between the Elcometer 130SSP and the Bresle Test method are significantly within the background contamination range of the Bresle Patches (0.88µg/cm²); being less than 0.41µg/cm² for concentrations below 8.0µg/cm², and less than 0.46µg/cm² across concentrations below 16.5µg/cm²
- No consideration has been taken into account with regards to the measurement accuracy of the Horiba Conductivity meter, which can further affect the Bresle Test Patch Method variations.

It can therefore be concluded that, following extensive testing across numerous salt concentrations, on smooth and a wide range of grit blast profiled test panels, that the Elcometer 130 SSP, when set up in the gauge's Bresle Equivalence Mode, provides an equivalence to the Bresle Test Method in accordance with ISO8502-9.

Appendix 1: - Coating Panels With Salt

The Bresle method of testing is a destructive test, in that the process of testing removes the salt. This means that the test can not be repeated again in exactly the same position. Therefore before Bresle equivalence can be tested, it is necessary to produce panels that are evenly covered in salt so that Bresle patches and the candidate method can reasonable be expected to give the same measurement when placed adjacent to each other on a panel.

However coating a panel evenly with salt is a difficult task to achieve. One issue is that the salt needs to adhere to the surface, so simply placing it in a dry state does not work, even if a mechanism for doing so was to be devised. This problem can be overcome by making a solution using the salt. The solution can then be applied to the surface and allowed to dry, leaving the salt behind on the surface of the panel. But applying salt evenly using a solution is not easy. The solution will tend to flow to the lowest point on a panel and form into pools as it dries. Also, if given time, the salt will start to form into crystals, creating high concentration zones.

Several methods have been investigated to overcome these problems:-

A1.1 Manual Glass Rod Method

A1.1.1 Method – This method is suggested in NACE SP0508-2010, which states "Apply an appropriate volume of doping solution in a column down the centre of the test panel using a pipette (1 mL has been found appropriate for 200 x 150 mm [8 x 6 in] test panel). Immediately spread the doping solution evenly over the entire test panel with an approximately 125 mm (5 in) in length by 3 mm (0.125 in) outside diameter (OD) glass rod. The glass rod shall be continuously wiped (but not rotated) over the test panel to keep the doping solution uniformly distributed until the liquid dries. A gentle stream of dry, compressed air may then be blown over the test panel to accelerate drying" **A1.1.2 Rationale** – Moving the solution uniformly over the panel as it dries will ensure that the same amount of solution dries in each area of the panel, and so deposits the same amount of salt in each area of the panel.

A1.1.3 Result – This method failed to produce a consistent coating of salt. It was not possible to visually determine that the solution depth was even as it dried, and as the solution volume reduced it immediately clumped into pools, after the glass rod had passed over it. Also it was a very time consuming process to perform manually.

A1.2 Automatic Glass Rod Method

A1.2.1 Method – This method is the same as the 'Manual Glass Rod Method' above, but using an automatic film applicator to continually move the glass rod over the panel surface as the solution dries. The automatic film applicator also has a heated base to heat the panel and so speed up the drying process.

A1.2.2 Rationale – The automatic film applicator will move the glass rod in a consistent and repetitive motion, thus ensuring that the glass rod is passed over all areas of the panel equally. A1.2.3 Result – This method was also unsuccessful. It still failed to maintain an even depth of solution over the surface as it dried, as the solution still clumped into pools as it dried.

A1.3 Drying Through a Mesh Method

A1.3.1 Rationale – The mesh or net would create a grid over the panel which would keep the solution evenly spread out as it dried by the influence of surface tension.

A1.3.2 Result – This method was unsuccessful. The solution still flowed to lower points on the panel due to slightly bowed panels or due to the panels being on a surface which was not perfectly level.





Figure 3a : Mesh or Net

Figure 3b : The deposition using the Mesh Method

A1.4 Single High Concentration Drop Method

A1.4.1 Method – This method is suggested in NACE SP0508-2010, which states "*Place a single drop of the doping solution in the centre of each marked area on the test panel using a pipette.* Allow the doping solution to spread and begin to evaporate. Once the majority of the doping solution has evaporated, a gentle stream of dry, compressed air may be blown over each spot to remove residual moisture. The volume of each drop shall be determined such that when spread on the test panel surface a single test is capable of measuring the entirety of the deposited salts"

A1.4.2 Rationale – As all the salt at the test location is contained within the Bresle patch and candidate test method, then it can be assumed that the candidate method should give the same result as the Bresle patch method in the same way as it would if the salt was evenly spread over the test area.

A1.4.3 Result – This method is unsuitable for candidate methods which do not enclose and contain the salt within the sample area, such as the filter paper method. As the filter paper method does not enclose the salt, an indeterminate quantity will migrate beyond the area of the filter paper that is measured (the sensor contact area). This method also requires the salt to be placed on the panel in very high concentration spots that can not be fully absorbed by the filter paper, and may be beyond the measurement range of the sensor.



Figure 4 : The Single High Concentration Drop Method

A1.5 Manual Multi-Drop Method

A1.5.1 Method – This method is similar to the 'single high concentration drop' method above. But, the salt is placed evenly over the entire panel in a grid of low concentration drops. The drops must be placed on a fine grid for this method to approximate to a continuous even coating of salt. A grid size of 4mm was decided upon, as this was the minimum grid size at which 3 μ l drops of solution could be placed without touching and running into each other, and 3 μ l was the minimum drop size that could be repeatable produced.

A1.5.2 Rationale – A surface which is covered with a fine grid of salt drops that almost touch, will give measurements that are the same as a surface with a continuous even coating of salt when tested using the Bresle method or a candidate method.

A1.5.3 Result – This method gave reasonable results. But it was not possible to maintain a perfect grid pattern by hand, as slight variations in the position, angle and height, caused drops to miss the grid and run in to each other creating small pools of solution and gaps in the grid. Also this is a very tedious process to perform manually taking about 3 hours to place the approximately 7,500 drops required for a 30 x 40 cm panel. It was also suspected that the manual handling of the panel for several hours was resulting in areas of accidental contamination.



Figure 5 : The Manual Multi-Drop Method

A1.6 Automated Multi-Drop Method

A1.6.1 Method – This method uses a robotic system to place 3 μ l drops on a 4mm grid, the same as the 'manual multi-drop method' above.

A1.6.2 Rationale – An automated system will be able to place the drops continuously and accurately on the 4mm grid. Also there will be less opportunity for accidental contamination as there is less manual handling.

A1.6.3 Result – This method gave good results, but some issues were still apparent. One problem was that sometimes the drop dispensing value's drop size drifted during the course of the doping process. This drift in drop size caused a gradient in salt concentration to occur from the point at which the salt deposition started to the point where it finished. Another problem was that the time taken for the salt solution to dry caused variations in the way that it was deposited. If the salt was allowed to dry slowly, it would form into high concentration crystals. But the salt didn't dry in a consistent way across the panel. This resulted in areas where the salt had dried into very concentrated spots or rings, which were not easily removed by the test methods. This resulted in varying measurements across the panel.

A1.7 Improved Automated Multi-Drop Method

A1.7.1 Method – This method uses a robotic system to place 3 μ l drops on a 4mm grid, the same as the 'automated multi-drop method' above, but with two improvements. Firstly, the order in which the drops were placed was randomised. And secondly, a heater was used to heat the panels as the drops were applied.

A1.7.2 Rationale – By randomising the order in which the drops are place, any drift in the drop size will be evened out across the panel. Heating the panel as the drops are applied causes them to dry quickly before they were able to form into crystals.

A1.7.3 Result – This method works well, and is the method which will be used for the Bresle equivalence testing. Experimentation showed that heating the panels to 70°C gave good results without being dangerously hot



Figure 6 : The Automated Multi-Drop Method complete with heated table

Appendix 2 – Results

A2.1 Elcometer 130 SSP to Bresle Method Equivalence

The Elcometer 130 SSP has undergone extensive side by side comparison testing against the Bresle Test Patch Method.

- Background (inherent) contamination within the Bresle Test Patch has shown that the Bresle • Test Patch has a background contamination range of 0.88µg/cm² - see Appendix A2.2
- The variation in readings between the Elcometer 130SSP and the Bresle Test method are • significantly within the background contamination range of the Bresle Patches (0.88µg/cm²); being less than 0.41µg/cm² for concentrations below 8.0µg/cm², and less than 0.46µg/cm² across concentrations below 16.5µg/cm²
- No consideration has been taken into account with regards to the measurement accuracy of the Horiba Conductivity meter, which can further affect the Bresle Test Patch Method variations.

It can therefore be concluded that, following extensive testing across numerous salt concentrations, on smooth and a wide range of grit blast profiled test panels, that the Elcometer 130 SSP, when set up in the gauge's Bresle Equivalence Mode, provides an equivalence to the Bresle Test Method in accordance with ISO8502-9.

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A2.1.1 Salt Density Range: 15-25mg/m² (1.5 – 2.5µg/cm²)

- Key: Bresle Test Method
 - Elcometer 130 SSP

Smooth Panels



All measurements are in mg/m²

25-50µm Blasted Panels





<u>50-75μm Blasted Panels</u>





<u>>75μm Blasted Panels</u>





A2.1.2 Salt Density Range: 25-35mg/m² (2.5 – 3.5µg/cm²)

- Key: Bresle Test Method
 - Elcometer 130 SSP

Smooth Panels



All measurements are in mg/m²

25-50μm Blasted Panels





50-75μm Blasted Panels



>75µm Blasted Panels



A2.1.3 Salt Density Range: 35-45mg/m² (3.5 – 4.5µg/cm²)

- Key: Bresle Test Method
 - Elcometer 130 SSP

Smooth Panels





All measurements are in mg/m²

25-50µm Blasted Panels



2

<u>50-75μm Blasted Panels</u>



>75μm Blasted Panels

1

2 1.5 1 0.5

0



A2.1.4 Salt Density Range: 45-55mg/m² (4.5 – 5.5µg/cm²)

- Key: Bresle Test Method
 - Elcometer 130 SSP

Smooth Panels





All measurements are in mg/m²

<u>25-50μm Blasted Panels</u>



<u>50-75μm Blasted Panels</u>



<u>>75μm Blasted Panels</u>

3.5

3 2.5

2

1

0.5

0





A2.1.4 Salt Density Range: >55mg/m² (>5.5µg/cm²)

- Key: Bresle Test Method
 - Elcometer 130 SSP

Smooth Panels



All measurements are in mg/m²

<u>25-50μm Blasted Panels</u>



50-75μm Blasted Panels



A2.2 Bresle Patch Background Contamination Results

All Bresle Test Patches used during the contamination testing were taken from Batch / Lot Number 82987 and Batch / Lot Number 84116.

Bresle Test #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
AVG	9	10	8.11	7.66	9	7	10	9	6	6	5	6	6.11	8	7	5	8	7	4	11	10
AVG-BACK	7	8	6.11	5.66	7	5	8	7	4	4	3	4	4.11	6	5	3	6	5	2	9	8
Background (mg/m2)	8.4	9.6	7.33	6.79	8.4	6	9.6	8.4	4.8	4.8	3.6	4.8	4.93	7.2	6	3.6	7.2	6	2.4	10.8	9.6

Bresle Test #	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42
AVG	7	7	8	7.22	6.11	11	6	6	7.55	8	7	7	11	7	7	9	5	9	5.5	5	9
AVG-BACK	5	5	6	6.11	4.11	9	4	4	5.55	6	5	5	9	5	5	7	3	7	3.5	3	7
Background (mg/m2)	6	6	7.2	7.33	4.93	10.8	4.8	4.8	6.66	7.2	6	6	10.8	6	6	8.4	3.6	8.4	4.2	3.6	8.4

Background Water Contamination:	2.0 mg/m ²
Salt Conversion Factor:	1.2 mg/m²
Max Bresle Background Contamination:	10.8 mg/m²
Min Bresle Background Contamination:	2.4 mg/m ²
Bresle Background Contamination Range:	8.8 mg/m ²
Average Bresle Background Contamination:	6.7 mg/m ²

Appendix 3 Calibration Checking of Elcometer 130SSP

First set the water and filter paper background contamination offset, as described in section 3.8.3.

Next, dope a filter paper with 1.6 ml of salt solution that will give a defined surface salt density reading. Surface salt density can be calculated by the following equation

$$\rho_A = c * \frac{V}{A} * \Delta y$$

Where:

 ρ_A is the surface salt density of the salts in mg/m² c = 5.0 kg*m⁻²*S A = (π *55²) mm² (surface area of filter paper = approximately 95 cm²) V = Volume of prepared solution (1.6 ml) Δy = the increase in conductivity in the test solution in μ S/cm

The surface salt density will be verified using a solution strength that equates to approximately 50mg/m^2 of salt.

Appendix 4 - Bresle Patch Method – ISO 8502-9 (from NACE SP0508)

- A4.1) Before each measurement or series of measurements and at a frequency of no less than every 4 hours, the conductivity meter will have its calibration verified. Adjust the conductivity meter as necessary to achieve accurate measurement.
- A4.2) Fill the syringe completely with deionised water and use it to rinse the conductivity cell. Perform this rinsing three times to clean both the syringe and the conductivity cell.

Note: this is a slight modification to ISO 8502-9 which has the cleaning of the syringe and the cleaning of the conductivity cell as two separate operations.

- A4.3) Measure the conductivity of this test solution and record the value.
- A4.4) Take an A-1250 Bresle patch, remove its protective backing along with its foam centre.
- A4.5) Apply the Bresle patch to the surface pressing firmly around the perimeter of the patch to ensure a complete seal.
- A4.6) Insert the needle of the syringe into the Bresle patch at an angle of 30° to the test surface near the outer edge of the patch so that it passes through the adhesive foam body into the test compartment and remove the air from the patch. Fill the syringe with 3 ml of deionised water. Replace the syringe needle into the Bresle patch and inject the 3 ml of water into the patch.
- A4.7) Remove the syringe needle to the edge of patch so that it is still plugging the hole, and preventing water from leaking. Immediately but gently, rub the surface of the patch for 90 seconds. Extract the sample solution from the patch, with the syringe, within 15 seconds of completion of the rubbing.

Note: This is a slight modification to ISO 8502-9 which states "*Remove the syringe from the Bresle patch.*"

A4.8) Inject the sample solution directly into the conductivity cell. Rinse the cell three times with the same test solution to be measured before taking nine readings. Record the conductivity value of the sample solution.

Note: Although not stated this infers we take the average of the nine readings.

A4.9) Calculate the increase in conductivity in the sample solution using the equation below.

 $\Delta \gamma = \gamma$ (extracted solution) - γ (injected solution)

Where:

 $\gamma(injected)$ = conductivity of the injected solution in μ S/cm

 γ (*extracted solution*) = conductivity of the extracted solution in μ S/cm

A5.10) Surface salt density can be calculated by the following equation

$$\rho_A = (c * \frac{v}{A} * y) - \rho_B = (5 * 3/12.5 * y) - \rho_B = (1.2 * y) - \rho_B$$

Where:

 ρ_A is the surface salt density of the salts in mg/m²

 $c = 5.0 \text{ kg}^{-2} \text{ s}^{-2}$

 $A = 12.5 \text{ cm}^2$ (surface area of a A-1250 Bresle patch)

V = Volume of injected solution (3 ml)

y = the increase in conductivity in the sample solution in μ S/cm

 ho_B is the average Bresle patch background contamination for the current batch of Bresle patches (See section 3.8.1)

Note: 7.2 mg/m² (from 6 μ S/cm solution) can be added back to account for historical salt contamination specifications that have been developed to account for the ionic contamination offset introduced by the Bresle method itself. The offset factor of 6 µS/cm used is consistent with ISO 8502-9 and confirmed by laboratory tests of Bresle patches from multiple sources. This equivalence test will not add this historical offset, but if required, data will be produced showing the results with this offset added to the Bresle results, and an equivalent offset added to the Elcometer 130 SSP results.

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Appendix 5 – Measurement Equipment Used.

5.1 Elcometer 130 SSP

Gauge Serial number PK15377

5.2 Bresle Patches

Elcometer 135B A-1250 Bresle Patches. Batch Numbers 82987 and 84116

5.3 Profile Measurement

Elcometer 224C-TI Serial Number RD10004

Certificate Number 224-RD10004-G

5.4 Environmental Measurement Elcometer G319----T Serial Number RE11596 Calibration

Certificate Number 24191 - Date 9th June 2015

5.5 Conductivity Measurement Horiba LAQUAtwin Model B-771 S/N PC44EE86 – Model S070 S/N D754V1A6

Jenway Model 4510 S/N 34200

5.6 Conductivity Calibration Solution

Horiba Standard Solution 1.41mS/cm Model Y071L Potassium chloride (nearly 0.07%)

Cole-Palmer Traceable Conductivity Standard Solution 10.27 µS/cm Cert No 4065-6859702

Cole-Palmer Traceable Conductivity Standard Solution 101.1µS/cm Cert No 4066-6906411