

ANALYSIS OF CDT METHODS AND FACTORS AFFECTING CATHODIC DISBONDMENT

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ABSTRACT

A critical analysis of 22 Cathodic Disbondment Test methods used in the US, European Union, France, Germany, Saudi Arabia, China, Canada, Australia, New Zealand and Other countries is presented in this paper. Parameters and Procedures used in these Standards are qualified and problems of their application discussed.

ASTM G8, ASTM G42, ASTM G80 and four of their generic modifications, as well as CSAZ245.20, EN 10289, DIN 30 671 (1987), API RP 5L7 and AWWA C-210 (old) will generate comparable results, when run under same temperature and duration conditions. Also, CD results obtained with French Standard NF A 49-711 and its generic modifications will be comparable with CD values produced by these tests.

Australian AS3862 and related AS/NZS 4352 will give approximately 15 – 20 % smaller cathodic disbondments.

ASTM G95 and ISO 15711 (E) test will give larger disbondments owing to the tests' durations.

Keywords : Interface oxide , Delamination and Chemical Attack, CDT Standard Methods, Cathodic disbondment, Electrical stress, Delamination, Thinning, Chemical stress, Chlorate, Oxide dissolution, Oxide removal.

INTRODUCTION

Cathodic protection (CP) complimented by a barrier-coating is an uncontested method of combating corrosion of buried/immersed structures under natural conditions. It is a daunting task and a noticeable expenditure for users of these structures.

A substantial portion of CP-related expenditures is the consumption of electrical current, which directly depends on the size of conductive contact between the substrate and the surrounding corrosive media. Due to that barrier-coatings are used to separate the protected structures from the surrounding corrosive media. A well selected coating will substantially reduce the loss of protective current.

However, the relation between Cathodic Protection and the coating is complicated by undesired CP-side-effects, such as generation of high pH and incidental development of elemental Hydrogen, whenever the media find access to the substrate. These electro-chemical processes are a major cause of a gradual increase in the substrate area exposed to the contact with the surrounding environment.

Users of buried structures desire to have a coating which when installed would require the least amount of CP current. The selection criteria for the barrier-coatings are based mainly on results of laboratory CD testing.

The testing is performed in accordance to local Standards and Specifications, the list of which is shown in Appendix # 1. It has to be noted that many other national Standards, not included in the selection, as well as their modifications are being practiced.

An urgent question is how to interpret and correlate varying results obtained by different Standard Methods. Since the Standards contain different combinations of qualitatively identical group of Parameters and Procedures, it is possible to consider eight Standards in Table 1 as a complete representation of all the Standards detailed in Appendix # 1.

**TABLE 1
REPRESENTATIVE CDT STANDARD METHODS**

Standard	Voltage [V]	Temperature [°C]	Solution [aqueous]	Duration [days]
CSA Z-245	1.5 or 3.5 V	Selection	3 % NaCl	Selection
ASTM G8	1.5	RT	Triple solution	Selection
ASTM G42	1.5	Selection	Triple solution	30
ASTM G80	1.5	RT	Triple solution	60
ASTM G95	3.0	RT	3 % NaCl	90
ISO 15711	1.05	RT	Sea water	182
AS 3862	3 mA	Selection	3 % NaCl	Selection
NF A 49-711	1.5	Selection	3 % NaCl	Selection

Notes:

1. Selection means multiple choices.
2. Triple solution is 1/1/1 % solution of NaCl, Na₂SO₄ and Na₂CO₃.
3. Artificial sea water is used.

Comparison of the Standards was based on CD results obtained with two lines of samples coated by 2-component liquid epoxy paints. Some qualitative and limited quantitative correlations between the Standards were formulated.

EXPERIMENTAL

CDT Standard Methods Testing

Basic CD testing was run with separate sets of samples coated by two different 2-component liquid epoxy materials (coating α and coating β) having different resistance against cathodic disbondment - highly resistant coating α and less resistant coating β . In such an arrangement the extent of CD results better characterized both more and less aggressive Standards Methods. Coating thickness was kept between 800 and 900 μm .

Compatibility of the disbondment was enhanced by selecting a temperature of 65°C and a duration of 28 days whenever Standard procedures included the parameters. Correct temperatures and durations in use are listed in Table 2.

All the testing was done with an impressed current system correcting the Voltage and measuring the current draw daily during the testing period.

Electrolyte was refreshed and make-up water added as required by the Standards.

The results of the testing are displayed in Table 2.

**TABLE 2
CD RESULTS ON COATING A AND COATING B**

Standard	Voltage [mA]	Current [mA]	Temp. [°C]	Duration [day]	Disbondment [mm]	
					Coating α	Coating β
CSAZ245.20	1.5	3 - 4	23	28	1	4
	1.5	5 - 7	65	28	2 - 4	11 - 14
	3.0	40 - 55	65	28	6	12 - 16
ASTM G 8	1.5	20 - 25	23	30	2	6
ASTM G 42	1.53	40 - 45	65	30	4 - 5	8 - 14
ASTM G 80	1.5	20 - 25	23	30	2	6
ASTM G95	3	25 - 30	23	90	5	15 - 25
ISO 15711 (E)	1.05	0.9 - 1.1	23	182	7.5	13.5
AS3862	1.3-1.4	3	65	28	2 - 3	10 - 13.5
NF A 49-711	1.5	3 - 5	95/30	28	2.5 - 3	5 - 6

- Note:
1. The current draw corresponds to one sample being tested.
 2. The coatings were 800 to 900 μm thick

Investigation of Parameters and Procedures

Qualitative comparison of the Standard CDT Methods was based on further analysis of Voltage-born complex stress, resulting in coating disbondment. A special testing system illustrates the forces participating in the process of cathodic disbondment.

Current. The most obvious effect of Voltage is current flow. Typical values of the first day current passing through the CD cells having 3.2 mm (1/8") or 6.35 mm (1/4") diameter holiday are shown in Figures 1 & 2. The current draw after 28 days of 65°C testing increased approximately twice with 900 -1000 µm thick coatings while a three-fold increase was observed with 500 µm coatings.

Interface oxide removal. An 1400 µm coating disbonded to a distance of 1 mm when tested under 1.5 V stress but a 6 mm distance was found after a 3 V test. The difference in the disbondment might be attributed to increased removal of the interface oxides and transport of water and ions along the interface.

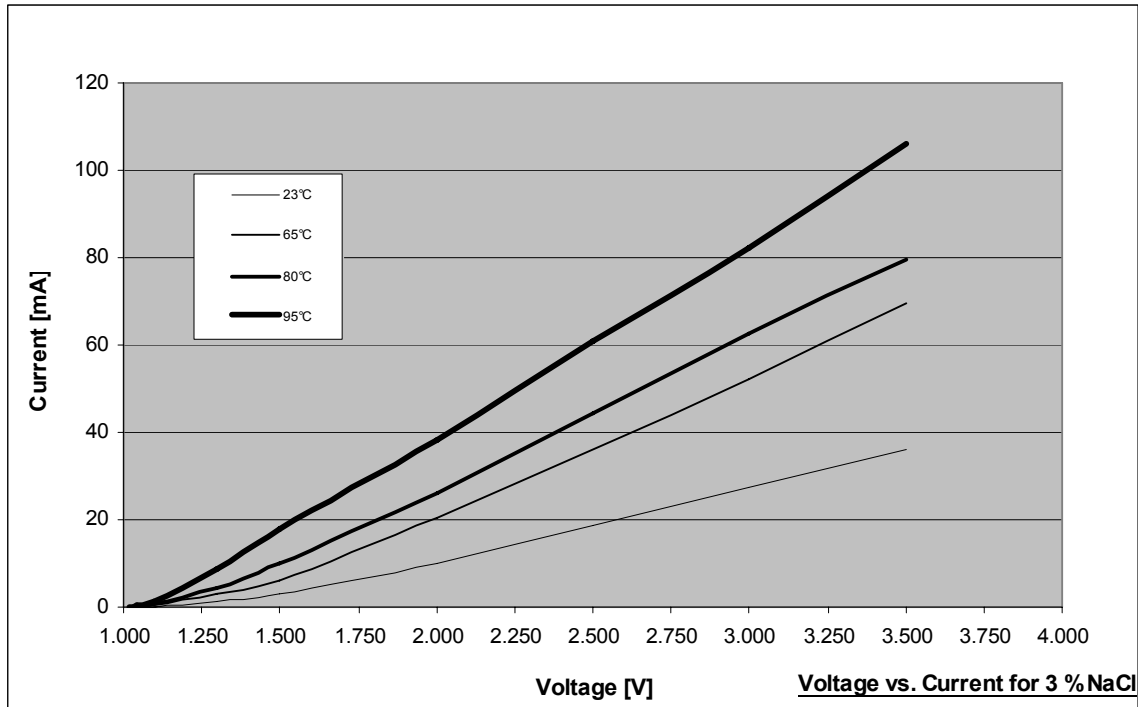


FIGURE 1 - 3 % NaCl solution - 3.2 mm diameter holiday

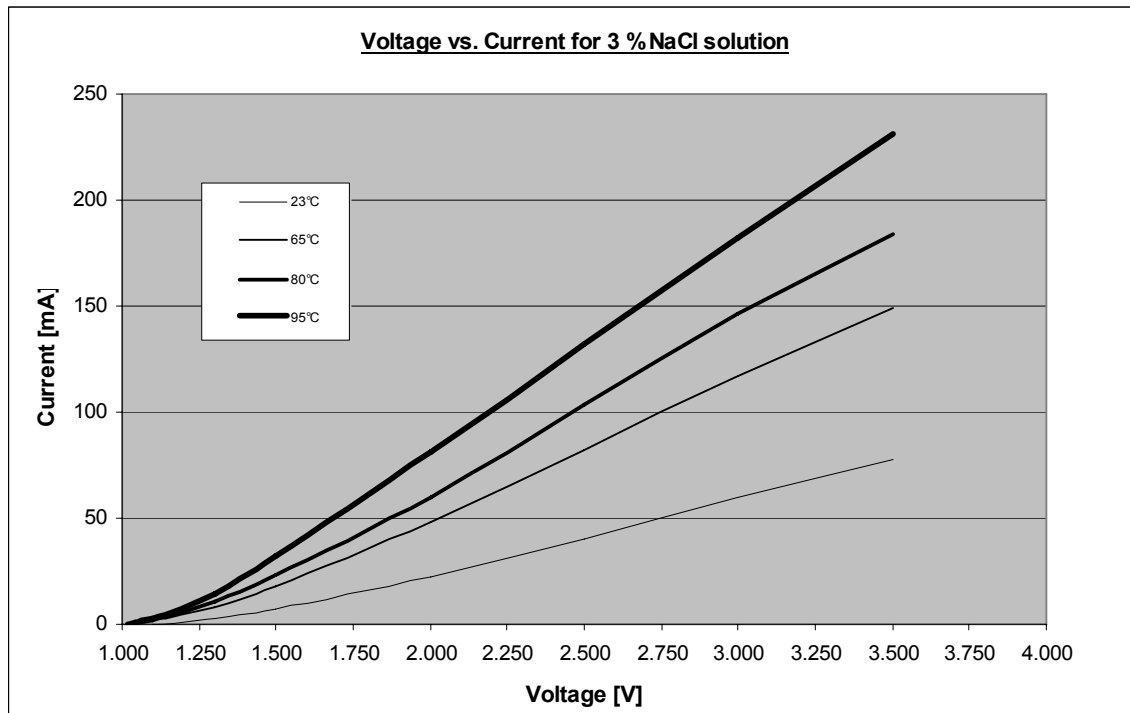


FIGURE 2 - 3 % NaCl solution – 6.35 mm diameter holiday

Anode Isolation Influence. Tests run with an isolated (separated) vs. a non-isolated anode investigated the role of chlorate in the disbonding process. No change in disbondment was observed with two 900 μm thick coatings when both tests were run using 1.5 V at 65°C for 28 days. Coating α disbonded 3 mm and coating β disbonded 16 mm in both non-isolated and isolated anode arrangements. It has to be noted that the AS-3862 Standard Method specifies both anode isolation and a current of 3 mA as opposed to 6 mA used for comparable testing.

Coating Thickness Influence. The increase in cathodic disbondment of progressively thinner coating is shown in the Figure 3. The tests were run in 500 ml attached cell using 1.5 V, 65°C, 28 days parameters.

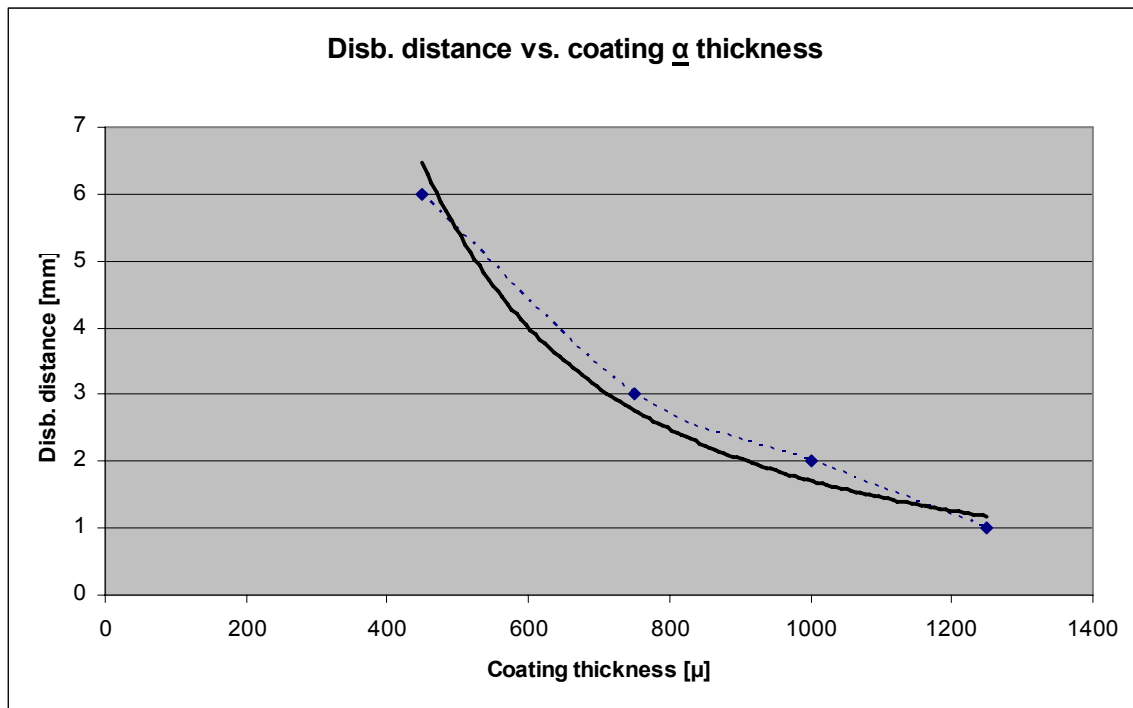


FIGURE 3 - Effect of coating α thickness on cathodic disbondment

Delamination and Chemical Attack. Thinning of the coating in CD cell is caused by chemical attack resulting from electro-chemical processes driven by the electrical current. The current converts NaCl into a highly aggressive chlorate (Appendix # 3), which gradually removes the top layer of the coating. The resulting Rate of Delamination (thinning) is directly proportional to chlorate concentrations in the electrolyte (Figure 4). Therefore anode isolation or frequent electrolyte refreshment is necessary in order to avoid coating delamination.

A very small proportion of the current is consumed dissolving the interface oxides as showed comparisons of current draw obtained with specific tests.

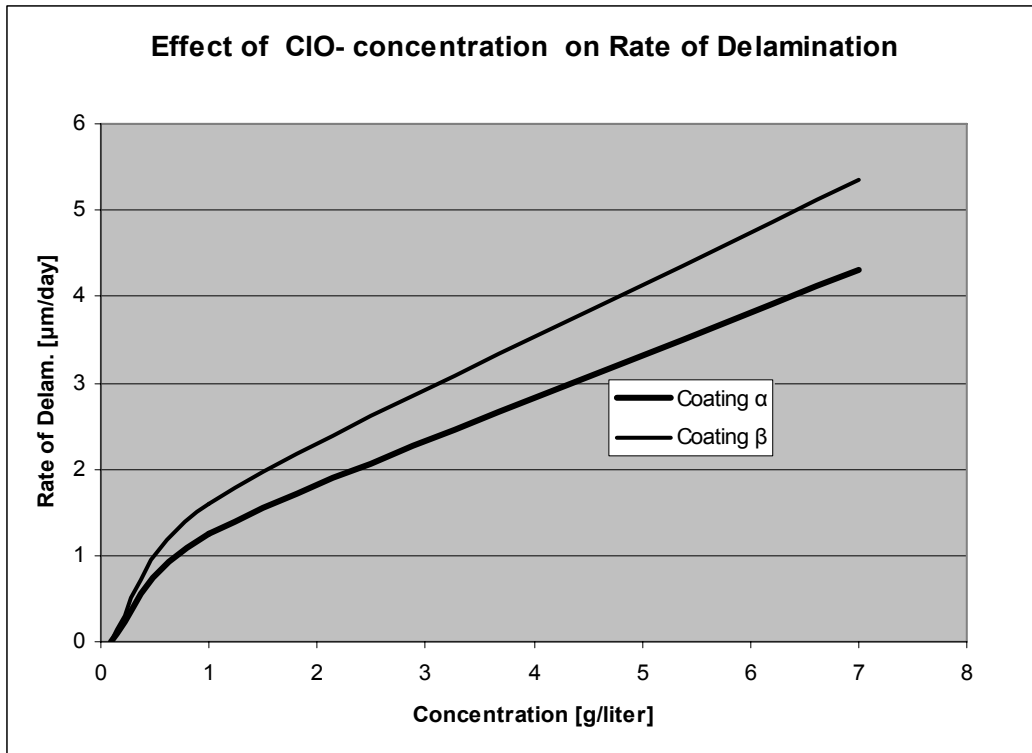


FIGURE 4 - Rate of delamination (μm per day) for coating α & β

Temperature influence. The Rate of Disbondment [mm per day] is directly proportional to temperature. A higher temperature will cause a greater expansion of disbonded area following an increase of the current.

A typical first-day value of current measured at 65°C is a double of the current at 23°C temperature. The cathodic disbondment is more than doubled.

Even bigger is the difference in the Rate of Delamination. No delamination can be observed with an average chlorate concentration of 1 gram per liter at 23°C , while the same concentration will remove approximately $40\ \mu$ when the electrolyte is heated at 65°C .

The effect of the temperature on the transport of water and ions was not measured by specific experiments. However, higher water vapor pressure increases water vapor permeation through the coating and porosity of the coating grows as a result of chemical attack and thermal expansion.

Also dissolution of interface oxides will be accelerated at elevated temperatures.

DISCUSSION OF THE RESULTS

In general, Voltage is the basic characteristic of CD testing. A low-voltage test better simulates the field conditions but it requires relatively long test times to offer reasonable disbondment. A higher-voltage test needs shorter times, but its result may be unduly affected by chemical conditions which do not exist in the field.

It is prudent to state that all of the Standard Methods using 1.5 V vs. SCE would generate results within common experimental error when run under the same temperature and duration conditions. A coating thickness of 1000 μ (or more) will sufficiently reduce the effect of chemical attack and compensate for a majority of the differences in the CD cell arrangement. High Voltage (3V or higher) tests generate an excessive amount of chlorate. Thus coating delamination becomes frequently the reason of the test failure. Measurable cathodic disbondment will be obtained only using anode isolation or a very frequent electrolyte refreshment procedure. Also, a thick coating whose thickness would not overly delaminate during the testing might be used.

All the CDT Standard Methods in Appendix # 1 specify the impressed current system, as well as the technical parameters, under which the Electrical stress (Voltage) is being applied. The impressed current system supplies the Voltage, which is independent of the anode size, type and depth of immersion. Also, parameters such as electrolyte volume, composition, refreshment, circulation, as well as holiday size and amount of samples in the CD cell would not affect the Voltage.

The electrical current flowing through the CD cell is directly proportional to the Voltage. It converts the salt (NaCl) into the highly aggressive chlorate (ClO_2^-). The most apparent effect of the conversion is oxidative destruction of the coating. All test parameters which increase concentration of the oxidant will increase its activity and consequently delamination-thinning of the coating. The Rate of Delamination shown in Figure 4 illustrates the extent of the phenomenon.

Transport of water and ionic species through the coating and along the interface and dissolution of the oxides are the direct reason for cathodic disbondment. The silvery portion of the disbonded area shows the extent of oxide removal. The coating thickness will change the rate of transport of water and ions through the coating as is illustrated in Figure 3. This parameter is neglected by the majority of the Standards despite the fact that a sufficiently thin coating would fail any CD test.

Test Duration is the most controversial parameter of CD testing. In an effort to adapt results of laboratory testing to mimic those in the field, some professionals extend CD test duration up to 90 days. In such an extended time chemical delamination - not cathodic disbondment - might become the reason for the coating failure. It has to be noted that delamination due to chlorate oxidation does not exist under CP conditions in the field.

Distribution of the disbondments obtained with identical tests documents effects of other significant factors, such as local coating inhomogeneity, coating porosity and substrate contamination. Effect of these factors was minimized by running multiple tests and discarding the extreme results.

CONCLUSIONS

Series of comparable CD tests performed as per selected CDT Standard Methods showed that ASTM G8, ASTM G42, ASTM G80 and four of their generic modifications, as well as CSAZ245.20, EN 10289, DIN 30 671 (1987), API RP 5L7 and AWWA C-210 (old) will generate comparable results, when run under same temperature and duration conditions. Also, CD results obtained with French Standard NF A 49-711 and its generic modifications will be comparable with CD values produced by these tests.

Reduction of disbondment due to anode separation in Australian AS3862 and related AS/NZS 4352 is limited to 15 – 20 % when compared to the first group of Standards. This is caused by a lower current draw and corresponding lower Voltage.

On the other hand, ASTM G95 seems to be more severe than the first group of tests. Effect of the test duration is the reason why approximately 25 % larger disbondments were obtained.

The size of cathodic disbondment after the ISO 15711 (E) test is markedly larger than CD after the other Standards' tests.

It has to be noted that the CDT Standard Methods leave some degree of freedom in specification of the parameters and procedures, which might become a source of controversy. The most frequent is lack of current control and thickness definition. Almost generally the definition of electrolyte refreshment is neglected.

On the other hand, some tests specify details unrelated to CD testing. A good CDT Standard should specify current measuring, recommended coating thickness and maximum allowable concentration of chlorate, alongside the voltage specification.

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Appendix No. 1

Simplified list of CD Standard methods

Serial No.	Standard	Country of origin	Voltage [Volts]	Temp. [°C]	Duration [days]	Solution
1	CSA Z-245	Canada	1.5	Selection	Selection	3 % NaCl
2	ASTM G8	United States	1.5	RT	Selection	Triple sol.
3	ASTM G42	United States	1.5	Selection	30	Triple sol.
4	ASTM G80	United States	1.5	RT	60	Triple sol.
5	ASTM G95	United States	3.0	RT	90	3 % NaCl
6	EN 10289: 2000(E)	International		Selection	Selection	3 % NaCl
7	ISO15711, Meth. A	International	1.05	RT	182	Sea water
8	ISO15711, Meth. B:	International	1.05	RT	182	Sea water
9	AS-3862	Australia	3 mA	Selection	Selection	3 % NaCl
10	AS/NZS4352: 1995; Meth.A	Australia/ New Zealand	3 mA	Selection	Selection	3 % NaCl
11	AS/NZS4352: 1995; Meth. B	Australia/ New Zealand	3 mA	Selection	Selection	3 % NaCl
12	NF A 49-711	France	1.5	Selection	Selection	3 % NaCl
13	ASTM G8	SHELL modif.	1.5	RT	90	Triple sol.
14	ASTM G42	SHELL modif.	1.5	Selection	Selection	Triple sol.
15	DIN 30 671 (1987)	Germany	1.5	Selection	Selection	3 % NaCl
16	AWWA C-210 (old)	United States	1.5	RT	2	Triple sol.
17	API RP 5L7	United States	1.5	66	30	3 % NaCl
18	09-amss-089 ARAMCO	Saudi Arabia	1.5	RT	30	Triple sol.
19	ARCO China	China	1.5	RT	28	Triple sol.
20	ASTM G8	United States TOTAL modif.	1.5	Selection	Selection	Triple sol.
21	NF A49-711	France TOTAL modif.	1.5	Selection	Selection	3 % NaCl
22	NF A49-716	France TOTAL modif.	1.5	Selection	Selection	3 % NaCl

- Notes: 1. Selection means multiple choices.
2. Triple solution is 1/1/1 % solution of NaCl, Na₂SO₄ and Na₂CO₃.

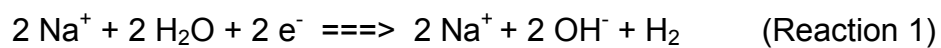
Appendix No. 2

Glossary of Terms and Abbreviations

Anode	Physical object supplying the current into the cell
CD	Cathodic Disbondment
CP	Cathodic Protection
Cell	The vessel, where the CD testing is being performed
Chlorate	highly aggressive (ClO ⁻) product of electrolysis in CD cell
Coating	Protective barrier applied onto the substrate
Coating delamination	Decomposition causing gradual thinning of the coating
Current	Electrical current flowing through the CD cell
Disbonded distance	The distance between the edge of the holiday and the edge of the disbonded area
Disbonded radius	The distance from the center of the holiday to the edge of the disbonded area
Electrolyte	The solution facilitating electrically conductive path between the anode and the cathode (substrate in the holiday)
Holiday	Circular discontinuity in the coating
Refreshment	Complete exchange of the electrolyte in the cell.
SCE	Standard Calomel Electrode
Specimen	Coated sample to be tested
Stress Electrical	Voltage applied in the CD cell
Substrate	Coated metallic material
Voltage	Electrical potential between reference electrode (SCE) and the substrate in a CD cell

Appendix No. 3

Electrolysis of NaCl in CD cell:



Chemical re-combination products of the electrolysis:



Rate of NaCl conversion into NaOCl

Current [mA]	NaCl consumed [grams/day]	NaOCl produced [grams/day]
5	0.13	0.165
10	0.26	0.33
50	1.3	1.65