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Evaluation of corrosion resistance and measurement of coating thickness

(1 appendix)

Accreditation

RISE is an accredited (by SWEDAC) laboratory for cyclic corrosion testing according to ISO 11997-1:2018 Cycle B, for visual examination of base metal corrosion according to SS-EN ISO 10289:2001, for determination of corrosivity with reference panels according to ISO 9226:2012, and for measurement of coating thickness according to SS-EN ISO 1463:2004.

Commission

Cyclic corrosion test according to the standard ISO 11997-1 Cycle B:2018 and determination of corrosivity class through exposure of reference test panels according to ISO 9226:2012. The test is performed according to the NORDTEST-method NT MAT 003 (2002).

Measurement of coating thickness according to SS-EN ISO 1463:2004.

Test objects

1 set of coated screws (at minimum 40 pieces) were received at RISE Research Institutes of Sweden on February 4, 2020. The product information can be seen in table 1.

Table 1: The table gives information of the product tested.

Sample identification	Dimension(mm)	Marking	Coating
3	M5,0x50/30	Wood Screw	Gray Electrocoat/PPG Electroseal V

Coating thickness

20 screws of sample 3 was controlled according to SS-EN ISO 1463:2004. A piece of the head was cut out from each screw and embedded in cold-setting epoxy. After grinding and polishing of the test objects, the coating thickness was measured using a microscope and a calibrated scale. The appearance of the topcoat was vague whereas no measurement was possible. The magnification was 625 times. The results are shown in Table 2 and 3.

The results are arithmetic mean values of measurements at 5 different points on each screw. The measuring accuracy is ± 0.8 µm.

The reported uncertainty of measurement is an expanded uncertainty (U), based on a standard uncertainty multiplied by a coverage factor, k=2, which provides a level of confidence of approximately 95 %. The uncertainty of measurement applies for a single measured value. The spread in results due to variations in sample characteristics is not accounted for in the reported uncertainty of measurement.

The measurements were performed in June, 2020 by operator Martina Rosqvist . The equipment used was microscope no. 401155 with calibration scale no. 402400.

Table 2: Results from coating thickness measurements according to SS-EN ISO 1463:2004 for sample 3.

Test object	Coating thickness, Mean value [µm]		Coating thickness, Max value [µm]		Coating thickness, Min value [µm]		Standard deviation	
	Layer 1 (Zinc)	Layer 2 (Top coat)	Layer 1 (Zinc)	Layer 2 (Top coat)	Layer 1 (Zinc)	Layer 2 (Top coat)	Layer 1 (Zinc)	Layer 2 (Top coat)
1 (1)	13,6	22,3	17,3	30,0	7,6	18,5	3,3	3,5
1 (2)	15,6	19,2	17,2	20,2	13,8	18,0	1,4	0,8
1 (3)	14,4	17,7	15,9	18,8	12,5	16,5	1,2	0,8
1 (4)	15,5	17,2	16,7	18,8	14,4	15,7	0,8	1,2
1 (5)	16,2	18,7	16,7	25,5	14,9	11,4	0,6	5,0
2 (1)	13,4	21,0	17,0	23,0	11,7	18,3	1,9	1,5
2 (2)	10,7	16,9	24,5	20,7	3,1	6,2	5,4	4,2
2 (3)	13,5	20,2	14,9	22,8	12,9	18,7	0,7	1,5
2 (4)	15,9	19,6	18,3	20,9	14,4	17,9	1,3	1,2
2 (5)	13,7	19,0	16,3	21,0	8,1	17,4	2,4	1,1
3 (1)	13,8	22,7	15,8	24,1	12,9	21,6	0,9	0,8
3 (2)	12,8	22,0	16,0	24,1	0,1	20,4	4,6	1,3
3 (3)	15,1	21,5	16,5	23,5	13,9	18,9	0,9	1,5
3 (4)	14,6	16,7	15,8	22,0	13,1	3,8	1,1	6,9
3 (5)	15,0	22,4	17,2	23,7	13,6	20,5	1,0	1,1
4 (1)	13,3	18,9	16,0	25,0	11,4	1,9	1,4	6,5
4 (2)	13,6	19,6	17,5	22,6	9,8	16,0	2,5	2,1
4 (3)	11,3	18,8	14,3	20,1	9,6	17,6	1,6	0,7
4 (4)	15,4	19,1	17,9	21,7	13,6	15,6	1,5	2,2
4 (5)	13,2	21,7	18,4	27,0	10,3	18,2	2,8	3,1

Table 3. Mean values and standard deviations of the measured coating thicknesses for each layer for sample 3.

Layer	Mean value of measured coating thickness [µm]	Standard deviation (n = 20)
1 (Zinc)	14,0	1,4
2 (Top coat)	19,8	1,8

Evaluation of corrosion resistance

20 screws of each sample were mounted on a piece of polystyrene foam with a 15 - 30° angle from the vertical plane. The test specimens were exposed according to ISO 11997-1 Cycle B:2018 for 13 weeks.

One test cycle corresponds to 7 days and consists of:

1. 24 h salt spray according to the standard ISO 9227 NSS
2. 96 h condensation (Four periods consisting of: 8 h at 100 % RH and 40 ºC followed by 16 h at 50 % RH and 23 ºC)
3. 48 h conditioning at 23 ºC and 50 % RH

8 reference test panels of carbon steel and 8 reference test panels of zinc were exposed with the samples. The reference test panels were ground and polished with diamond spray down to a grain size of at least 9 µm, cleaned in ethanol and weighed before the test. Prior to exposure, the unpolished face of the reference panels was protected with an adhesive plastic film.

Reference panels were removed regularly from the chamber during the exposure. The adhesive plastic film was removed and the corrosion products were removed by repetitive pickling according to the standard ISO 8407:2014. The reference panels of carbon steel were pickled in a solution of concentrated hydrochloric acid in water (1:1), containing inhibitors. The reference panels of zinc were pickled in a saturated solution of glycine. The panels were then weighed and the metal loss, expressed in micrometres, was calculated as a function of exposure time.

Conditions of testing, salt spray

The salt used for the preparation of salt solution was Sanal P salt containing at least 99,9% sodium chloride from Nouryon. The impurities were less than 0.001 % copper, less than 0.001 % nickel, less than 0,001% lead and less than 0.1% sodium iodide. The sodium chloride was dissolved in deionized water with conductivity lower than 10µS/cm.

The salt concentration of the collected solution is measured as conductivity instead of direct concentration. A salt concentration of 50 g/l ± 5g/l NaCl results in a conductivity between 70,2-84,0 mS/cm.

Test conditions:

Temperature	35 ± 2°C
pH in collected solution:	6,5 – 6,9
Volume of collected solution:	1,1 – 2,0 ml / 80 cm^2, hour
Conductivity of collected solution:	77,1 – 87,0 mS/cm*
Test equipment:	Ascott 6, inventory number: BX722439
Latest calibration:	2019-10-21
Date of test:	March 3, 2020 to May 26, 2020
Test engineer:	Martina Rosqvist, Jennifer Jacobsson

**Deviations: In exposure week 3 the salinity was higher than 84,00* mS/cm. *The salt reservoir was adjusted to the coming week. As reference material was exposed under the same conditions this was judged not to influence the result.*

Relationship between corrosivity class and exposure time

Using the results of the reference panels of zinc and carbon steel, the relationship between corrosivity class and exposure according to ISO 11997-1:2018 Cycle B was calculated. The results are shown in Table 4 and 5 below.

Table 4: Metal loss of zinc and carbon steel depending on the exposure according to ISO 11997-1:2018 cycle B and corresponding corrosivity class based on a technical life-time of 15 years.

Testing time (cycles)	Zinc		Steel		Corrosivity class, mean value
	Metal loss (µm)	C-class	Metal loss (µm)	C-class	
0	0	-	0	-	-
1	8,8	2,1	23,7	1,2	1,7
2	17,5	2,8	47,3	1,4	2,1
4	35,0	3,8	94,7	1,9	2,9
8	70,0	4,8	189,4	2,7	3,8
12	105,0	5,2	284,1	3,5	4,3

Table 5: Requirements for different corrosivity classes according to ISO 11997-1:2018 Cycle B based on a technical life-time of 15 years.

Corrosivity class	Testing time according to ISO 11997-1 Cycle B (test cycles of one week)
C1	$0,6 \leq t < 1,8$
C2	$1,8 \leq t < 4,5$
C3	$4,5 \leq t < 9,1$
C4	$t \geq 9,1$

Result, assessment of corrosivity class

During the corrosion test, the test areas of the samples were visually examined every week with respect to base metal corrosion. The degree of base metal corrosion was assessed according to the standard SS-EN ISO 10289:2001 by giving a grade between 0 and 10 to each sample. The testing time when more than 10 % of the samples exhibited base metal corrosion (grade 9, i.e. between 0 and 0.1 % of the surface area was corroded) was assessed by interpolation. The pictures from the inspections can be seen in appendix 1.

Conclusion

The corrosivity class are summarize in table 6. The corrosivity class is for 15 years technical life-time according to the requirements of the method.

Table 6: Corrosivity class

Sample identification	Weeks until >10 % base metal corrosion	Corrosivity class
3	>12	C4

Comments and restrictions

The results in this report concern only the tested products. The test method does not include damage to the products that may arise during installation in the field (in actual use). Accelerated corrosion testing is not an exact model of long-term exposure in the field. However, cyclic corrosion tests (like ISO 11997-1:2018 Cycle B) correlate much better with real exposure than tests with continuous salt spray.

Table A.1 and A.2 in NORDTEST-method NT MAT 003 (2002) are revised according to new values for metal loss in the latest version of ISO 9224:2012.

RISE Research Institutes of Sweden AB
Corrosion – Product Durability

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Appendix

1. USB Flash drive

Appendix 1

USB flash drive with photographs from the inspections