










RESEARCH REPORT

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Protective agents of concrete SILKO-tests 2014

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<p>Summary</p> <p>This is an unofficial translation of the Finnish research report. In case of interpretation disputes the Finnish report.</p> <p>This test programme is an updated version of the test programme VTT-R-07962-11/EN. The frost resistance of coatings, impregnating agents, hydrophobic impregnation agents and cleanable anti graffiti agents is tested according to standards EN 13678-1, EN 13678-2 and EN 13678-3 as defined in EN 1503-3 (2006).</p> <p>These test instructions are to be used in examining the suitability of protective agents for outdoor concrete structures owned by the Finnish Transport Agency (Former Finnish Road Administration). On the basis of test results Finnish Transport Agency decides on the acceptability of protective agents for their use. The approved materials are published in SILKO-directions which are available on the web pages of Finnish Transport Agency.</p> <p>There are four types of protective agents for concrete; hydrophobic impregnation agents, pore filling impregnation agents, coatings and antigraffiti agents. Antigraffiti agents are further divided in sacrificial and cleanable agents.</p> <p>Part of the SILKO-tests are obligatory and part of them are voluntary. The obligatory tests must be carried out on all protective agents for which the acceptance by Finnish Transport Agency is applied for. The voluntary tests are carried out to prove that a material has a special property suitable for a special case.</p>				
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1. Introduction

These test instructions are to be used in examining the suitability of protective agents for the protection of outdoor concrete structures owned by Finnish Transport Agency. These instructions concern hydrophobic impregnation agents, pore filling impregnation agents, coatings, sacrificial antigrffiti agents and cleanable antigrffiti agents. The test programme is prepared corresponding to typical Finnish bridge environments using as much European test standards as possible. On the basis of test results Finnish Transport Agency decides on the acceptability of protective agents for their use. The approved materials are published in SILKO-directions which are available on the web pages of Finnish Transport Agency.

The test specimens for SILKO-tests are treated with protective agent by the product agent under the surveillance of the VTT representative. Any exception to this procedure should be noted in the test report.

2. Product groups and their test programmes

Part of the SILKO-tests are obligatory and part of them are voluntary. The obligatory tests must be carried out on all the materials that are applied for acceptance by Finnish Transport Agency. The voluntary tests are carried out to prove that a material has a special property suitable for a special case. The properties of different groups of materials that are tested are given in chapter 3, along with the test methods.

Other than the European test methods of the test programme are described in chapter 4.

3. SILKO test programme and criteria of protective agents of concrete

3.1 Hydrophobic impregnation agents

Property	Test method	Criterion and grading			
		+	++	+++	++++
Obligatory tests					
Product identification	IR (EN 1767) or TGA (EN ISO 11358)	No criterion			
Water permeability	EN 13580	Absorption rate, % of reference < 15 ≤ 10 ≤ 5			
Alkali resistance	EN 13580	No grading. Absorption rate after immersion in alkali, 10 % of reference			
Water vapour permeability	EN ISO 7783-2 (film on a substrate)	s_D , m ≤ 100 ≤ 50 ≤ 5 ≤ 0.5			
Chloride permeability	SILKO-test (Chapter 4.1)	Chloride content, % of reference ≤ 100 ≤ 20 ≤ 15 ≤ 8			
Frost salt durability	EN 13581	The loss of mass of the surface of the impregnated specimen must occur at least 20 cycles later than that of the not impregnated specimen.			
Penetration depth	EN 1504-2	Penetration depth, mm ≥ 2 ≥ 5 ≥ 10 ≥ 15			
Voluntary tests					
Carbonation prevention	NT BUILD 357 CO ₂ -content 1 %, RH 60 ± 5 %, T 21 ± 2 °C	Carbonation depth, % of reference ≤ 100 ≤ 60 ≤ 20			
Cleanability	SILKO-test (Chapter 4.3)	1 Poor	3 Moderate	5 Good	
Removability	SILKO-test (Chapter 4.5)	No criterion/ grading			

3.2 Pore filling impregnants

Property	Test method	Criterion and grading			
		+	++	+++	++++
Obligatory tests					
Product identification	IR (EN 1767) or TGA (EN ISO 11358)	No criterion			
Water permeability	EN 1062-3	No grading, only criterion: $w, \text{kg/m}^2 \cdot \text{h}^{1/2}$			
Water vapour permeability	EN ISO 7783-1 (free film) ¹⁾ EN ISO 7783-2 (film on a substrate) ¹⁾	s_D, m			
Chloride permeability	SILKO-test (Chapter 4.1)	Chloride content, % of reference			
Adhesion ²⁾	EN 1542	Adhesion strength, MPa ^{*)} ≥ 1.5 (1.0)			
Frost salt durability	EN 13687-1 EN 13687-2	a) no bubbles, cracks and delamination b) Pull-off-test Application/Load Average [N/mm ²] vertical ≥ 0.8 (0.5) ^b horizontal without mechanical load ≥ 1.0 (0.7) ^b horizontal with mechanical load ≥ 1.5 (1.0) ^b ^b The value in brackets is the lowest accepted value of any reading.			
Penetration depth	EN 1504-2	Penetration depth, mm			
Voluntary tests					
Carbonation prevention	NT BUILD 357	Carbonation depth, % of reference			
Cleanability	SILKO-test (Chapter 4.3)	1 Poor	3 Moderate	5 Good	
Removability	SILKO-test (Chapter 4.5)	No criterion/ grading			

1) Alternative tests

2) Systems that form a continuous film.

3.3 Coatings

Property	Test method	Criterion and grading			
		+	++	+++	++++
Obligatory tests					
Product identification	IR (EN 1767) or TGA (EN ISO 11358)	No criterion			
Water permeability	EN 1062-3	No grading, only criterion: $w, \text{kg/m}^2 \cdot \text{h}^{1/2}$ < 0.1 - - -			
Water vapour permeability	EN ISO 7783-1 (free film) ¹⁾ EN ISO 7783-2 (film on a substrate) ¹⁾	s_D, m ≤ 100 ≤ 50 ≤ 5			
Chloride permeability	SILKO-test (Chapter 4.1)	Chloride content, % of reference ≤ 50 ≤ 20 ≤ 15 ≤ 8			
Carbonation prevention ²⁾	NT BUILD 357 CO ₂ content 1 %, RH 60 ± 5 %, T 21 ± 2 °C	Carbonation depth, % of reference ≤ 20 ≤ 15 ≤ 8			
Carbon dioxide permeability ²⁾	SFS-EN 1062-6	No grading, only criterion: s_D, m > 50 - - -			
Adhesion	SFS-EN 1542	Adhesion strength, MPa Crack bridging coatings: ≥ 0.8 Others: ≥ 1.5			
Cleanability	SILKO-test (Chapter 4.3)	1 Poor	3 Moderate	5 Good	
Freeze thaw durability	EN 13687-3	a) no bubbles, cracks and delamination b) Pull-off-test Average [N/mm ²] Crack-bridging or Rigid systems c flexible systems without trafficking: ≥ 0.8 (0.5) ^b ≥ 1.0 (0.7) ^b with trafficking: ≥ 1.5 (1.0) ^b ≥ 2.0 (1.5) ^b ^b The value in brackets is the lowest accepted value of any reading. ^c Rigid coatings are coatings with shore D ≥ 60 according to EN ISO 868.			
Dry film thickness	SILKO-test (Chapter 4.6)	Polymer coatings: ≥ 300 μm Cement based coatings: ≥ 2000 μm			
Voluntary tests					
Crack bridging ability	EN 1062-7, Method A, -30 °C	Crack width at failure, mm ≥ 0.3 ≥ 0.6 ≥ 1.0			
Removability	SILKO-test (Chapter 4.5)	Easily removable			
Declared by manufacturer					
UV-resistance	EN 11507, 500 h	No sensory changes after the test			
Water resistance	SILKO-test (Chapter 4.7)	No sensory irreversible changes after the test			
Alkali resistance	SILKO-test (Chapter 4.8)	No sensory irreversible changes after the test			

1) Alternative tests

2) Alternative tests.

3.4 Anti graffiti agents - Sacrificial

Property	Test method	Criterion and grading			
		+	++	+++	++++
Obligatory tests					
Product identification	IR (EN 1767) or TGA (EN ISO 11358)	No criterion			
Water permeability	EN 13580 ¹⁾	Absorption rate, % of reference			
	EN 1062-3 ²⁾	No grading, only criterion: $w, \text{kg/m}^2 \cdot \text{h}^{1/2}$			
Water vapour permeability	EN ISO 7783-1 (free film) ³⁾	s_D, m			
	EN ISO 7783-2 (film on a substrate) ³⁾	≤ 100	≤ 50	≤ 5	
Cleanability	SILKO-test (Chapter 4.1)	1 Poor	3 Moderate	5 Good	
Frost-salt durability using non-carbonated specimens ¹⁾	CEN/TS 12390-9	Scaling, % of reference			
Freeze thaw durability ²⁾	CEN/TS 12390-9	No visible defects			
Removability	SILKO-test (Chapter 4.5)	Pressure cleaning			
Dry film thickness ²⁾	SILKO-test (Chapter 4.6)	≥ 30 μm	≥ 50 μm	≥ 80 μm	
Voluntary tests					
Chloride permeability	SILKO-test (Chapter 4.1)	Chloride content, % of reference			
Carbonation prevention	NT BUILD 357 CO ₂ -pitoisuus 1 %, RH 60 ± 5 %, T 21 ± 2 °C	Carbonation depth, % of reference			
Declared by manufacturer					
UV-resistance	EN 11507, 500 h	No sensory changes after the test			

1) Systems not forming a continuous film

2) Systems that form a continuous film

3) Alternative tests.

3.5 Anti graffiti agents - Cleanable

Property	Test method	Criterion and grading			
		+	++	+++	++++
Obligatory tests					
Product identification	IR (EN 1767) or TGA (EN ISO 11358)	No criterion			
Water permeability	EN 13580 ¹⁾	Absorption rate, % of reference			
	EN 1062-3 ²⁾	No grading, only criterion: $w, \text{kg/m}^2 \cdot \text{h}^{1/2}$			
Water vapour permeability	EN ISO 7783-1 (free film) ³⁾	s_D, m			
	EN ISO 7783-2 (film on a substrate) ³⁾	≤ 100	≤ 50	≤ 5	
Adhesion ²⁾	EN 1542	Adhesion strength ≥ 1.5 (1.0) MPa The value in brackets is the lowest accepted value of any reading.			
Cleanability	SILKO-test	1 Poor	3 Moderate	5 Good	
Frost-salt durab. using carbonated specimens ¹⁾	CEN/TS 12390-9	Scaling, % of reference			
Freeze thaw durability ²⁾	CEN/TS 12390-9	After the test: Only slightly defected. Adhesion: ≥ 1.5 MPa.			
Frost-salt durability ¹⁾	EN 13581	The loss of mass of the surface of the treated specimen must occur at least 20 cycles later than that of the reference specimen.			
Freeze thaw durability ²⁾	EN 13687-3	a) no bubbles, cracks and delamination b) Pull-off-test Average [N/mm^2] Crack-bridging or flexible systems Rigid systems ^c without trafficking: ≥ 0.8 (0.5) ^b ≥ 1.0 (0.7) ^b with trafficking: ≥ 1.5 (1.0) ^b ≥ 2.0 (1.5) ^b ^b The value in brackets is the lowest accepted value of any reading. ^c Rigid systems are systems with shore D ≥ 60 according to EN ISO 868.			
Dry film thickn. ²⁾	SILKO-test (Chapter 4.6)	$\geq 30 \mu\text{m}$	$\geq 50 \mu\text{m}$	$\geq 80 \mu\text{m}$	
Voluntary tests					
Chloride permeability	SILKO-test (Chapter 4.1)	Chloride content, % of reference			
Carbonation prevention	NT BUILD 357 CO ₂ -pitoisuus 1 %, RH 60 ± 5 %, T 21 ± 2 °C	Carbonation depth, % of reference			
Crack bridging ability ²⁾	EN 1062-7 Method A, -30 °C	Crack width at failure, mm			
Declared by manufacturer					
Removability	SILKO-test (Chapter 4.5)	No criterion/ grading			
UV-resistance	EN 11507, 500 h	No criterion/ grading			

- 1) Systems not forming a continuous film
2) Systems that form a continuous film
3) Alternative tests.

4. SILKO-TEST METHODS

4.1 Chloride permeability

4.1.1 Concrete specimens to be treated with the protective agent and their curing

The test specimens consist of concrete cubes measuring 100 x 100 x 100 mm³, with 3 cubes per each protective agent and 3 additional reference cubes per each series of test.

The concrete from which the cubes are made is the same as used for the water permeability test, SFS-EN 13580.

For 28 days after casting, the cubes are stored at a temperature (t) of 20 ± 2 °C and a relative humidity (RH) of 95 ± 5%. For the following 14 days up to the age of 42 days, the cubes are stored at t = 20 ± 2 °C and RH = 65 ± 5%.

4.1.2 Application of the protective agent and its curing

The cubes are sandblasted and the protective agent is applied to all sides of the concrete cubes in accordance with the manufacturer's instructions.

The test specimens are cured in accordance with the manufacturer's instruction. After this the specimens are kept at t = 20 ± 2 °C and R = 65 ± 5% for 7 days.

4.1.3 Test procedure

The following curing times and conditions are required for the tests:

- 56 days in a 15% NaCl solution.

The concrete cubes are weighed in the beginning of the test and when they are removed in the NaCl-solution. The accuracy of the weighing machine must be 0.1 g.

Water-soluble chloride contents are determined from powder samples using the Mohr method (EN 1744-1:1998 Chapter 9) at depths of 0 to 20 mm and 20 to 50 mm. The samples are detached from vertical sides, with the casting face on the top. One sample is taken from both depths of each cube. Chloride content is determined from the powder obtained by combining the samples from the three cubes.

4.1.4 Test results

Test results are presented as percentage of chloride by weight of the concrete at depth ranges of 0 to 20 mm and 20 to 50 mm. Also the ratios of the chloride contents compared to the chloride contents of the reference cubes are presented.

4.2 Effect of the protective agent on carbonation rate

4.2.1 Concrete specimens to be treated with the protective agent and their curing

The test is carried out using concrete prisms measuring 40 x 40 x 160 mm³, with two prisms per each protective agent as well as 2 reference prisms per each series of tests.

Composition of the concrete is identical to that used in the water permeability test, SFS-EN 13580. After casting, the prisms are cured at t = 20 ± 2 °C and RH = 95 ± 5% for a period of 28 days. Following this, the prisms are sandblasted and they are dried for 24 hours in a vacuum oven with a temperature of 30 ± 2 °C. Vacuum suction must be in operation for 8 hours from the start of the drying process.

The prisms are kept in plastic bags until they are treated with the protective agent at the age of about 42 days.

4.2.2 Application of the protective agent and its curing

The protective agent is applied to all sides of the prisms and cured in accordance with the manufacturer's instructions.

4.2.3 Test procedure

The test is carried out according to NT BUILD 357.

The test consists of storing the treated prisms and non-treated reference prisms in air with a carbon dioxide content of 3...5% for a period of 3 months. The storage temperature is 20 ± 2 °C and relative humidity $65 \pm 5\%$.

Carbonation is monitored during the test by cutting one treated prism and one reference prism at one third of its length following a carbon dioxide treatment of 0, 0.5, 1, 2 and 3 months, respectively, and by applying a phenolphthalein indicator to the exposed surface. The carbonation depth of the exposed surfaces is measured. The exposed surfaces can also be photographed.

Accelerated carbonation of the prisms through the exposed surfaces is prevented by applying a suitable resin to these surfaces.

4.2.4 Test results

The ratio of the carbonation depth of the treated prisms to that of the non-treated reference prisms following a carbon dioxide treatment of 0, 0.5, 1, 2 and 3 months are presented as the test results. If so desired, the pictures may be accompanied by photographs showing the carbonated areas.

4.3 Cleanability

4.3.1 Concrete specimens to be treated with the protective agent

The test is carried out using ready-made sandblasted concrete paving slabs measuring $40 \times 300 \times 300$ mm³, with 2 slabs per each protective agent and 2 non-treated reference slabs per each series of tests. The slabs are stored at least for 14 days before using at 20 ± 2 °C and relative humidity $65 \pm 5\%$.

4.3.2 Application of the protective agent and its curing

The protective agent is applied to the sandblasted surface of the concrete slabs and cured in accordance with the manufacturer's instructions.

4.3.3 Test procedure

The sandblasted surface of the treated slabs and non-treated reference slabs is divided into three sections. Graffiti are drawn on one section using paint from a spray can, on one section using alkyd paint and on one section using a water-proof felt pen.

After the application of the graffiti, the slabs are stored for 7 days at a relative humidity of $65 \pm 5\%$ and a temperature of 20 ± 2 °C, after which they are photographed and one slab per each protective agent and one reference slab is cleaned using (high-pressure) water cleaning (using hot water) and one using a cleaning agent and method recommended by the manufacturer of the protective agent.

After cleaning, a visual inspection of the slabs is carried out to evaluate how effectively the applied paint has been removed. The slabs are also photographed.

4.3.4 Test results

Test results are presented in the form of a numeric evaluation of how effectively the applied paint was removed, accompanied by photographs taken after cleaning. The numeric value varies from 0 to 5.0 means that the graffiti does not remove at all and a value of 5 means that the graffiti removes totally.

4.4 Removability of a protective agent

4.4.1 Concrete specimen to be treated with the protective agent and its curing

A sandblasted prefabricated concrete paving slab, 40 x 300 x 300 mm³, is used as test specimen.

4.4.2 Application of the protective agent and its curing

The protective agent is applied on the sandblasted surface of the slab according to the instructions of the manufacturer.

4.4.3 Test procedure

The slabs are cured at 20 ± 2 °C at $65 \pm 5\%$ relative humidity for at least 7 days.

The protective agent is removed according to the instructions given by the manufacturer.

If the manufacture does not recommend any removal method, the protective agent is removed by sandblasting from an area of 100 x 100 mm². The sandblasting is carried out using glass balls, 6 bar pressure, \varnothing 8 mm nozzle, from 40 mm distance, perpendicular to the surface. Sandblasting is carried out along 10 mm wide stripes. The nozzle is moved back and forth at the rate of 200 mm/s.

The removability of the protective agent is estimated paying attention to the difficulty of removing and remnants of the protective agent.

4.4.4 Test results

The estimation of removability as well as the amount of protective agent remnants are given as test results.

4.5 Dry thickness of the protective agent

4.5.1 Specimens

The thickness of the protective agent on the test specimens used for any test(s) is measured.

4.5.2 Test procedure

A test specimen is broken. The thickness of the protective agent is measured on the fracture surface using a microscope with a magnification of at least 50 x. The length of the surface to be measured is at least 100 mm.

4.5.3 Test results

The minimum, maximum and average thicknesses of the protective agent are given as test results.

4.6 Water resistance

4.6.1 Test specimens

Test specimens are concrete prisms, $160 \times 40 \times 40 \text{ mm}^3$, the number of which is 2 per each protective agent.

Composition of the concrete is identical to that used in the water permeability test, SFS-EN 13580.

After casting, the prisms are first cured for a period of 28 days at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 95 \pm 5\%$ and then for at least 14 days at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 65 \pm 5\%$.

4.6.2 Application of the protective agent and its curing

Three long sides of the prisms cast against the form are pre-treated and treated according to the manufacturer's instructions.

The treated specimens are cured according to the manufacturer's instructions. In addition to the before mentioned special curing the specimens are stored at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 65 \pm 5\%$ for a period of 7 days.

4.6.3 Test procedure

The prisms are placed in water at room temperature in such a way that the long side is downwards. The volume of the solution is at least four times that of the total volume of the specimens. The solution is changed in two week intervals. The duration of the test is 6 weeks after which the specimens are removed at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 65 \pm 5\%$ for 7 days.

The condition of the protective agent is evaluated visually before and after the test.

4.6.4 Test result

Changes of the protective agents are reported as test results.

4.7 Alkali resistance

4.7.1 Concrete specimens to be treated with the protective agent and their curing

Test specimens are concrete prisms, $160 \times 40 \times 40 \text{ mm}^3$, the number of which is 2 per each protective agent.

Composition of the concrete is identical to that used in the water permeability test, SFS-EN 13580.

After casting, the prisms are first cured for a period of 28 days at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 95 \pm 5\%$ and then for at least 14 days at $t = 20 \pm 2 \text{ }^\circ\text{C}$ and $\text{RH} = 65 \pm 5\%$.

4.7.2 Application of the protective agent and its curing

Three long sides of the prisms cast against the form are pre-treated and treated according to the manufacturer's instructions.

The treated specimens are cured according to the manufacturer's instructions. In addition to the before mentioned special curing, the specimens are stored at $t = 20 \pm 2$ °C and $RH = 65 \pm 5\%$ for a period of 7 days.

4.7.3 Test procedure

The prisms are placed in saturated $\text{Ca}(\text{OH})_2$ -solution at room temperature in such a way that the long side is downwards. The volume of the solution is at least four times that of the total volume of the specimens. The solution is changed in two week intervals. The duration of the test is 6 weeks after which the specimens are removed at $t = 20 \pm 2$ °C and $RH = 65 \pm 5\%$ for 7 days.

The condition of the protective agent is evaluated visually before and after the test.

4.7.4 Test result

Changes of the protective agents are reported as test results.