

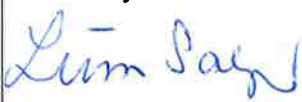


**RESEARCH REPORT**

VTT-R-01254-14/EN  
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# **SILKO test programme of liquid membrane forming curing compounds of concrete 2014**

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<b>Summary</b>	
<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-03533-11. The criteria of water retention ability have been changed.</p> <p>These test instructions are to be used in examining the suitability of liquid membrane forming curing compounds of concrete 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 liquid membrane forming curing compounds of concrete 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 two types of curing compounds for concrete, <u>evaporation reducers</u> used at an early age and <u>final curing agents</u> applied after final set. The test programme and acceptance criteria for both types of curing compounds are given here.</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. Test programme of liquid membrane forming curing compounds of concrete

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The test programmes and acceptance criteria of evaporation reducers used at an early age and final curing agents are given in Tables 1 and 2.

Table 1 shows the tests on the bases of which Finnish Transport Agency decides about the acceptance for their use. These tests are the so called obligatory tests.

Table 2 shows the so called voluntary tests on the bases of which the curing compound may be shown to have some extra properties beneficial for Finnish Transport Agency.

Concrete according to CEN/TC 14754-1 is used in all the tests where concrete is needed except in the test of the effect of curing compound on frost salt durability. In the frost salt test the concrete specimens are prepared of air-entrained concrete MC(0,40) according to the standard EN 1766.

Table 1. Test programmes and acceptance criteria of curing compounds of concrete. Obligatory tests.

Property	Evaporation reducers used at an early age		Final curing agents	
	Test	Criterion	Test	Criterion
Identification	EN 1767	-	EN 1767	-
Spreadability Spraying	SILKO test. Spraying. Test temperatures +5 °C and +30 °C. Spraying is carried out according to the instructions given by the manufacturer. If the manufacturer does not recommend any method, the size of the nozzle is $\varnothing \leq 2$ mm and spraying pressure is ca. 1 MPa.	<ul style="list-style-type: none"> <li>- Must be sprayable</li> <li>- The sprayer must not clog</li> </ul>	SILKO test. Spraying. Test temperatures +5 °C and +30 °C. Spraying is carried out according to the instructions given by the manufacturer. If the manufacturer does not recommend any method, the size of the nozzle is $\varnothing \leq 2$ mm and spraying pressure is ca. 1 MPa.	<ul style="list-style-type: none"> <li>- Must be sprayable</li> <li>- The sprayer must not clog</li> </ul>
Spreadability Applicability by roller or brush			SILKO test. Rolling or brushing. Test temperatures +5 °C and +30 °C. Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer either by roller or by brush.	Must be applicable by roller or brush
Compatibility with concrete	SILKO test Method 1 Scraping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying rate test $\geq 72$ h after casting (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03) Method 2 SILKO test. Separate specimens Test temperature +21 °C. Scraping by knife of the surface 7 d after casting.	The surface must not be softer than the water cured surface.	SILKO test Method 1 Scraping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying rate test $\geq 72$ h after casting (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03) Method 2 SILKO test. Separate specimens Test temperature +21 °C. Scraping by knife of the surface 7 d after casting.	The surface must not be softer than the water cured surface.
Content of water soluble chloride	EN 1744-1, chapter 9	$\leq 0.2$ wt-%	EN 1744-1, chapter 9	$\leq 0.2$ wt-%

Table 1. continues

Property	Evaporation reducers used at an early age		Final curing agents	
	Test	Criterion	Test	Criterion
Drying time	<p>SILKO test            Concrete specimens are cast. Curing compound is spread according to the instructions given by manufacturer.            Testing conditions +21 °C, RH 50%, air velocity parallel to the surface ca. 3 m/s            (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03)            The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm.</p>	<p>Drying time must be ≤ 4 h</p>	<p>SILKO test            Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer.            Testing conditions +21 °C, RH 50%, air velocity parallel to the surface ca. 3 m/s            (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03)            The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm.</p>	<p>Drying time must be ≤ 4 h</p>
Water retention ability	<p>CEN/TC 14754-1</p>	<p>Water evaporation from the curing agent treated specimens must be at least</p> <ul style="list-style-type: none"> <li>- 50% less than from the water cured specimens 3 h after the application of the curing agent</li> <li>- 70% less than from the water cured specimens 24 h after the application of the curing agent</li> </ul>	<p>CEN/TC 14754-1</p>	<p>Water evaporation from the curing agent treated specimens must be</p> <ul style="list-style-type: none"> <li>- 75% less than from the water cured specimens 24 h after the application of the curing agent</li> <li>- 70% less than from the water cured specimens 72 h after the application of the curing agent</li> </ul>

Table 1. continues

<p>Removability of wax based curing compound</p>			<p>VTT TEST 375-93          Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer.          At the earliest 7 d after casting the curing compound is removed by the method recommended by the manufacturer. If the manufacturer does not give a recommendation the curing agent is removed by grit blasting. Eater colour is brushed on the surface and immediately wiped by cloth. Visual inspection using magnifying glass.</p>	<p>There must not be non-coloured areas.</p>
<p>Applicability on vertical surfaces</p>			<p>SILKO test          Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets At the age of 1 d curing compound is applied according to the instructions of the manufacturer.          Testing conditions +21 °C, RH 50%.          The surface is inspected visually at the earliest 1 d after the application of curing compound.</p>	<p>There must not be traces of running of the curing compound.</p>

Table 2. Test programmes and acceptance criteria of curing compounds of concrete. *Voluntary tests.*

Property	Evaporation reducers used at an early age		Final curing agents	
	Test	Criterion	Test	Criterion
Effect on soiling			<p>SILKO test</p> <p>Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. The colour is measured</p> <p>A mixture of water and soot is sprayed on the surface. The surface is flushed with water. The colour is measured</p>	<p>The result is compared with water cured surface.</p> <p>No criterion</p>
Cleanability			<p>SILKO test</p> <p>Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. Graffiti is made on the surface and the surface is cleaned.</p> <p>Visual inspection.</p>	<p>The result is compared with water cured surface.</p> <p>No criterion</p>
Performance on surface exposed to direct sunlight			<p>SILKO test</p> <p>Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. EN ISO 11507: 170 h UV 8 d after casting: Visual inspection and scratching of the surface by knife</p>	<ul style="list-style-type: none"> <li>- The visual appearance must not change because of UV exposure</li> <li>- The surface must not be softer than the water cured surface.</li> </ul>
Effect on frost salt durability			<p>SILKO test</p> <p>Concrete specimens are cast. The surface is trowelled. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. CEN/TC 12390-9 slab test, 3% NaCl solution.</p>	<p>Scaling must not be higher than that of the water cured specimens.</p>



## 2. Evaporation reducers used at an early age

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The properties required of the evaporation reducers used at an early age on bridges do not depend on their used. The required properties are:

- water retention ability
- spreadability,
- compatibility with concrete,
- content of water soluble chloride and
- drying time.

### 2.1 Test method and criteria

#### 2.1.1 Water retention ability

##### *Test method*

Water retention ability is testing according to CEN/TC 14754-1.

##### *Criterion*

Curing agent must reduce water evaporation 50% by the time of 3 h and 70% by the time of 24 h after application of curing agent.

#### 2.1.2 Spreadability

##### *Test method*

The test temperatures are  $+5 \pm 2$  °C and  $+30 \pm 2$  °C. The temperature of the compound must be  $+5 \pm 2$  °C and  $+30 \pm 2$  °C respectively. The compound is sprayed according to the manufacturer's instructions 1 000 ml in 60 min. The spraying does not have to be continuous.

If the manufacturer does not recommend any other the pressure of ca. 1 MPa and a nozzle with a diameter of not greater than 2 mm are used.

##### *Criterion*

The compound must be sprayable and the sprayer must not clog.

#### 2.1.3 Compatibility with concrete

There are two alternate methods to test the compatibility with concrete.

##### *Test method 1*

The compatibility is tested by scraping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying time test described below at the earliest  $\geq 72$  h after casting.

##### *Test method 2*

The testing temperature is  $+ 21 \pm 2$  °C.

Four concrete specimens are cast. The minimum dimensions of the top surface of the specimens are  $150 \cdot 150$  mm<sup>2</sup> and their thickness is 50 mm. Two specimens are cured using curing compound according to the instructions given by the manufacturer. Two reference specimens are covered with plastic sheets after casting. 1 d after casting a water layer is put

on the surface of the reference specimens. The water layer is removed 7 d after casting. 1 d after removal of the water the surface of all specimens is scraped with knife.

#### *Criterion*

The surface cured with curing compound must not be softer than the water cured surface.

#### 2.1.4 Content of water soluble chloride

##### *Test method*

The content of water soluble chloride is measured according to EN 1744-1 chapter 9.

#### *Criterion*

The content of water soluble chloride must not be higher than 0.2 wt-%.

#### 2.1.5 Drying time

##### *Test method*

The testing conditions are  $+21 \pm 2$  °C, RH  $50 \pm 10\%$  and air velocity parallel to the surface ca. 3 m/s.

Two concrete specimens are cast the minimum dimensions of the upper surface of which are  $150 \cdot 150$  mm<sup>2</sup> and the thickness of which are 50 mm. The curing compound is applied according to the instructions of the manufacturer. The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm. Drying time is the time elapsed from curing compound application to the dry to touch condition.

#### *Criterion*

Drying time must not exceed 4 h.

### **3. Final curing agents**

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The properties required of the final curing agents depend on their use. the properties required in different uses on bridges are:

- water retention ability
- spreadability: applicability by spray, brush or roller
- compatibility with concrete
- content of water soluble chloride
- drying time
- applicability on vertical surfaces, when the compound is used on vertical surfaces
- performance on surface exposed to direct sunlight, when the compound is used on a surface exposed to direct sunlight during the hardening of concrete
- effect on frost salt durability, when the compound is used on a surface exposed to frost and salt and it is not removed
- effect on soiling, when the compound is used on a visible surface and it is not removed
- cleanability, when the compound is used on a visible surface and it is not removed.

### 3.1 Test methods and criteria

#### 3.1.1 Water retention ability

##### *Test method*

Water retention ability is testing according to CEN/TC 14754-1.

##### *Criterion*

Curing agent must reduce water evaporation 75% by the time of 24 h and 70% by the time of 72 h after application of curing agent.

#### 3.1.2 Spreadability

##### 3.1.2.1 Sprayability

##### *Test method*

The test temperatures are  $+5 \pm 2$  °C and  $+30 \pm 2$  °C. The temperature of the compound must be  $+5 \pm 2$  °C and  $+30 \pm 2$  °C respectively. The compound is sprayed according to the manufacturer's instructions 1 000 ml in 60 min. The spraying does not have to be continuous.

If the manufacturer does not recommend any other the pressure of ca. 1 MPa and a nozzle with a diameter of not greater than 2 mm are used.

##### *Criterion*

The compound must be sprayable and the sprayer must not clog.

##### 3.1.2.2 Applicability by brush/ roller

##### *Test method*

The test temperatures are  $+5 \pm 2$  °C and  $+30 \pm 2$  °C. The temperature of the compound must be  $+5 \pm 2$  °C and  $+30 \pm 2$  °C respectively.

Four concrete specimens are prepared/application method. The dimensions of the specimens are  $150 \cdot 150 \cdot 50$  mm<sup>3</sup>. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d, 2 specimens/ application method are removed to the temperature of  $+5 \pm 2$  °C and two to the temperature of  $+30 \pm 2$  °C. After 2 h curing the plastic sheets are removed. When the surface is mat dry, the curing agent is applied on all four specimens according to the instructions given by the manufacturer either by brush or by roller.

##### *Criterion*

The curing compound must be applicable by roller or brush so that a layer thickness according to the instructions given by the manufacturer is obtained.

#### 3.1.3 Compatibility with concrete

There are two alternate methods to test the compatibility with concrete.

##### *Test method 1*

The compatibility is tested by scraping by knife the surface of the specimens of water retention test CEN/TC 14754-1 or drying time test described below at the earliest  $\geq 72$  h after casting.

### *Test method 2*

The testing temperature is  $+ 21 \pm 2$  °C.

Four concrete specimens are cast. The minimum dimensions of the top surface of the specimens are  $150 \cdot 150$  mm<sup>2</sup> and their thickness is 50 mm. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer. At the age of 7 d the water layer is removed. At the age of 8 d the surface of the specimens is scraped with knife.

### *Criterion*

The surface cured with curing compound must not be softer than the water cured surface.

#### 3.1.4 Content of water soluble chloride

### *Test method*

The content of water soluble chloride is measured according to EN 1744-1 chapter 9.

### *Criterion*

The content of water soluble chloride must not be higher than 0.2 wt-%.

#### 3.1.5 Drying time

### *Test method*

The testing conditions are  $+21 \pm 2$  °C, RH  $50 \pm 10\%$  and air velocity parallel to the surface ca. 3 m/s.

Two concrete specimens are cast the minimum dimensions of the upper surface of which are  $150 \cdot 150$  mm<sup>2</sup> and the thickness of which are 50 mm. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d the plastic sheets are removed. Curing compound is applied on mat dry surface specimens according to the instructions given by the manufacturer. The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm. Drying time is the time elapsed from curing compound application to the dry to touch condition.

### *Criterion*

Drying time must not exceed 4 h.

#### 3.1.6 Applicability on vertical surfaces

### *Test method*

The testing temperature is  $+ 21 \pm 2$  °C.

A concrete specimen is cast. The minimum dimensions of the vertical surface to be treated with curing compound are  $150 \cdot 150$  mm<sup>2</sup>. For the first day the specimen is cured in its mould covered with a plastic sheet. At the age of 1 d the plastic sheet is removed. The curing compound is applied according to the instructions given by the manufacturer on the vertical concrete surface. The surface is inspected visually not earlier than 24 h after curing compound application.

*Criterion*

There must not be traces of running of the curing compound.

### 3.1.7 Removability of wax based curing compound

*Test method*

The testing conditions are  $+21 \pm 2$  °C, RH  $50 \pm 10\%$ .

A concrete specimen is cast minimum dimensions of the upper surface of which is  $150 \cdot 150$  mm<sup>2</sup> and the thickness of which is 150 mm. The upper surface is finished with steel float. For the first day the specimen is cured in its mould covered with a plastic sheet. At the age of 1 d the plastic sheet is removed. The curing compound is applied on mat dry surface of the specimen according to the instructions given by the manufacturer. If the manufacturer does not give instructions the curing compound is removed by grit blasting.

The remnants of the curing compound are inspected according to VTT TEST 375-93. Eater colour is applied by brush on the surface. The brush must not be dripping wet. Immediately while the surface is still wet the surface is wiped with a dry cloth.

The surface is inspected visually using a magnifying glass. There are no remnants of curing compound where the surface is coloured by water colour. Whereas the not coloured surfaces indicate curing compound remnants.

*Criterion*

There must not be uncoloured areas on the surface treated with curing compound.

### 3.1.8 Performance on surface exposed to direct sunlight (Voluntary)

*Test method*

Two concrete specimens are cast the minimum dimensions of the upper surface of which are  $150 \cdot 150$  mm<sup>2</sup> and the thickness of which are 50 mm. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d the plastic sheets are removed. A water layer is applied on one specimen and curing compound is applied on mat dry surface of the other specimen according to the instructions given by the manufacturer. The water layer is maintained for 6 d. At the age of 7 d the specimen is removed to RH  $50 \pm 10\%$ . Immediately after the application of the curing compound the surface is exposed to UV for 170 h according to EN ISO 11507 after which the specimen is removed to RH  $50 \pm 10\%$ .

At the earliest 3 d after the end of the exposure to UV the surfaces are inspected visually and they are scraped with knife.

*Criterion*

The surface cured with curing compound must not be softer than the water cured surface and the visual appearance must not change because of UV exposure.

### 3.1.9 Effect on frost salt durability (Voluntary)

*Test method*

Four concrete specimens are prepared of air-entrained concrete MS(0,40) according to the standard EN 1766. The dimensions of the specimens are  $150 \cdot 150 \cdot 50$  mm<sup>3</sup>. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds

covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer. At the age of 7 d the water layer is removed and all the specimens are removed to  $+21 \pm 2$  °C and RH  $65 \pm 10$  %. At the age of  $25 \pm 1$  d a rubber mat is glued on all other but test surfaces according to CEN/TS 12390-9. A frost salt test according to CEN/TS 12390-9 slab test using 3-% NaCl solution is carried out.

#### *Criterion*

Scaling must not be higher than that of the water cured specimens.

#### 3.1.10 Effect on soiling (Voluntary)

##### *Test method*

The testing conditions are  $+21 \pm 2$  °C, RH  $50 \pm 10$ %.

Four concrete specimens are cast the minimum dimensions of the surface to be treated with curing agent are  $150 \cdot 150$  mm<sup>2</sup>. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer. At the age of 7 d the water layer is removed and the specimens are removed to RH  $50 \pm 10$ %.

At the age of 28 d the surfaces of the specimens are inspected visually and the colour of the surfaces is measured using spectrophotometer. At least three points of every surface is measured. The test surfaces are placed vertically and they are soiled by lightly spraying Degussa Printex 300 furnace black powder mixed in water on them. When the surfaces have dried they are inspected visually and their colour is measured. The colour change of the soiled surfaces compared to the colour of original clean surfaces of the with curing compound treated specimens is  $\Delta E_{\text{JHA-1}}$  and that of the water cured reference specimens  $\Delta E_{\text{VERT-1}}$ . The absolute value of the remainder,  $|\Delta E_{\text{CUR-1}} - \Delta E_{\text{REF-1}}|$ , must not exceed 1.0. The test surfaces are carefully flushed with water. After drying they are visually inspected and their colour is measured.

The means of the change of colour at the end of the test compared to the colour of the original clean surfaces is calculated,  $\Delta E_{\text{CUR-2}}$  and  $\Delta E_{\text{REF-2}}$ .

#### *Criterion*

The result is compared with water cured surface. There is no criterion.

#### 3.1.11 Cleanability (Voluntary)

##### *Test method*

The testing conditions are  $+21 \pm 2$  °C, RH  $50 \pm 10$ %.

Six concrete specimens are prepared. The minimum dimensions of the surface to be cured with curing agent are  $150 \cdot 150$  mm<sup>2</sup>. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d the plastic sheets are removed. A water layer is applied on three specimens and the curing agent is applied on three specimens according to the instructions given by the manufacturer when the surface is mat dry. At the age of 7 d the water layer is remove and he specimens are removed to RH  $50 \pm 10$ %.

At the earliest 3 d after the removal of water layer the specimens are inspected visually. The surface of two specimens cured with curing compound and two water cured specimens are divided in 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. At the age of 35 d one specimen cured with curing compound and one water cured specimen are cleaned using (high-pressure) water cleaning (using hot water) and one using a cleaning agent. When the surfaces have dried they are inspected visually to evaluate how effectively the applied graffiti have been removed by comparing their appearance to that of the specimens with no graffiti.

The result evaluated numerically using scale from 0 to 5.0 means that the graffiti does not remove at all and 5 means that the graffiti is totally removed. The cleanability is compared to that of the water cured reference specimens.

#### Criterion

The result is compared with water cured surface. There is no criterion.

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