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Test report

P 6090-2-E

Testing order: **Testing of the epoxy resins for steel and concrete bridge deck sealing**

- **Micopox BP**
- **Micopox BC**

according to
VVTBT – Tätskikt på broar
chapter 5 Tätskikt med epoxy
chapter 8 Förseglingar

Customer: **ELMICO A/S**
Dysterud Gaard
2100 Skarnes
Norway

Persons in charge: **J. Magner**
V. Redekop

Date of the test report: **2009-01-24**

This test report comprises: **21 pages, 1 enclosure**

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CONTENTS

1 SUBJECT

Polymer Institut was charged by the company Elmico A/S, N-Skarnes to carry out the performance testing of the steel bridge sealing epoxy resins

Micopox BP / Micopox BC

in accordance with the Swedish stipulations

VVTBT – Tätskikt på broar

chapter 5 Tätskikt med epoxi

chapter 8 Förseglingar

The technical description VVTBT Tätskikt has been published in February 2008.

Remark:
This report is indicating Swedish nouns and titles , all descriptions and results are documented in English language.

2 TESTS AND REQUIREMETNS

The test the material has to pass and the requirements to fulfil are listed in:

Bilaga B Krav på epoxipreparat

- **Tabell 1 Krav for epoxipreparat - Allmänt ingående komponenter**
- **Tabell 2 Krav for epoxipreparat Allmänt blandning eller härdat material**
- **Tabell 3 Krav for epoxipreparat Allmänt provkroppar**
- **Tabell 4 Krav for epoxipreparat Allmänt stalprimer**
- **Tabell 5 Krav på vidhäftning mellan försegling av epoxi och gjutaspahtl**

3 SYSTEM

The submitted material was described by the customer as follows:

Material	Description
Micopox BP	Two-component, solvent-free epoxy reactive resin
Micopox BC	Two-component, solvent-free epoxy reactive resin with filler, based on Micopox BP

Polymer Institut received the following sample materials:

Pos.	Designation of the material	Quantity
1	Micopox BP, component A	1 x 10 kg
2	Micopox BP, component B	1 x 5 kg
3	Micopox BC, component A with filler	1 x 10 kg
4	Micopox BC , component B with filler	1 x 5 kg

According to the manufacturers declaration Micopox BC consists of the binder resin Micopox BP, to both components aggegrates / filler are added.

4 TESTS

Unless otherwise stated, all tests were carried out in standard climate acc. to DIN EN 23270.

4.1 Tests of the primary materials

chapter	Provning/ nature of the test	Provning Bilaga B	TP-BEL-EP*
4.1.1	Densitet / Density	1	3.1.1
4.1.2	Viskositet / Dynamic viscosity	2	3.1.2
4.1.3	Sammansättning / Infrared spectroscopic analysis	3	3.1.3

** "Technical prescriptions and delivery conditions for reactive resins for primers, sealers and scratch screeds under asphalt" („Technische Prüfvorschrift für Reaktionsharze für Grundierungen, Versiegelungen und Kratzspachtelungen unter Asphaltbelägen auf Beton – TP-BEL-EP"), Official German Regulations for bridge sealing with epoxy*

4.1.1 Density

The density was determined in accordance with DIN 53 217, Part 2, as a result of two single determinations, using a pyknometer (100 cm^3) and at a temperature of 23 °C.

The results are to be taken from table 1.

Table 1: Density

Micopox BP	Density [g/cm^3]	
	Single values	Mean value
component A	1,136 ; 1,137	**1,137**
component B	0,962 ; 0,962	**0,962**

4.1.2 Dynamic viscosity

The dynamic viscosity was determined at a test temperature of 23 °C in accordance with DIN EN ISO 3219 using a rotational viscometer. Duplicate determinations were carried out. The test conditions are given in the following compilation:

	Measuring system	r/min
component A	M5 / PK5-1.0	0 → 82
component B	M5 / PK5-1.0	0 → 410

For recording the gradient the number of revolutions of the rotating body was increased at constant acceleration within 3 min to the numbers of revolution given in the above table. By this, a relative shear strength of ≥ 30 % resulted.

Shear stress and velocity gradient (shear rate) as well as the dynamic viscosity calculated thereof are to be taken from the following table and of figure 1/ 2 of the enclosure.

Table 2: Dynamic viscosity

Micopox BP	Shear stress [Pa]	Shear rate [s^{-1}]	Dynamic viscosity [mPas]	
			Single values	Mean value
component A	500	451 ; 450	900 ; 900	**900**
component B	2500	171 ; 181	70 ; 70	**70**

4.1.3 Infrared spectroscopic analysis,

The infrared spectroscopic analysis of the primary materials was carried out on the basis of DIN 51451 without any preconditioning of resin and hardener.

Additionally an extract of the hardened material was produced and tested parallely

Parameters of the analysis:

Apparatus: FTS-40 Fourier transform spectrometer, company Bio-Rad
Direct measurement on ATR crystal (ZnSe)
Measuring range: 4000 cm^{-1} - 500 cm^{-1}

The results can be seen in figure 3/ 4 of the enclosure

4.2 Tests of the mixed and cured materials

chapter	Provning/ nature of the test	Provning Bilaga B	TP-BEL-EP*
4.2.1	Viskositet / Dynamic viscosity	4	3.2.1
4.2.2	Askhalt / Residue on ignition	7	3.2.2
4.2.3	Potlife/ Pot life	5	3.2.3
4.2.4	Hårdhet / Curing time	6	3.2.4
4.2.5	Vattentålighet/ Sensitivity to moisture	10	3.2.5
4.2.6	Icke flyktiga beståndsdelar / Non-volatile matter	8	3.2.6
4.2.7	Extraherbara beståndsdelar / Extractable matter	9	3.2.7
4.2.8	Vattenabsorption / Water absorption	11	3.2.8

** "Technical prescriptions and delivery conditions for reactive resins for primers, sealers and scratch screeds under asphalt" („Technische Prüfvorschrift für Reaktionsharze für Grundierungen, Versiegelungen und Kratzspachtelungen unter Asphaltbelägen auf Beton – TP-BEL-EP"), Official German Regulations for bridge sealing with epoxy*

4.2.1 Dynamic viscosity

The dynamic viscosity of the mixed material at 12 °C was measured in accordance with DIN EN ISO 3219, analogue to chapter 4.1.2

The results are to be taken from table 3 and of figure 5 of the enclosure.

Table 3: Dynamic viscosity at 12°C

Material	Shear stress [Pa]	Shear rate [s^{-1}]	Dynamic viscosity [mPas]	
			Single values	Mean value
Micopox BP	300	730 ; 725	2500 ; 2500	**2500**

4.2.2 Residue on ignition

The residue on ignition was determined in accordance with DIN EN ISO 3451-1.

Temperature: 550 °C
Period of ignition: 16 hours

The result is to be taken from table 4.

Table 4: Residue on ignition

Material	Residue on ignition [% by mass]	
	Single values	Mean value
Micopox BP	0,00 ; 0,00 ; 0,00	**0,00**

4.2.3 Pot life

Pot life according to this specification is the time interval between the temperature of 23°C and 40°C of 100 ml of the mixed material.

The results are to be taken from table 5 and of figure 6 of the enclosure.

Table 5: Pot life

Material	Pot life [min]	
	Single values	Mean value
Micopox BP	27 ; 27	**27**

4.2.4 Curing time

The curing time test with Buchholz indentation test in accordance with DIN EN 2815 has been carried out, using a coated glass panel (dry film thickness about 1 mm) and the following curing times and climates:

7 days at standard conditions EN 23270
18 hours at standard condition EN 23270
40 hours at 12 °C and 85 % relative humidity

The value measured after seven days standard atmosphere is taken as 100 % curing, and the values obtained at the other periods of time given above are in relation to this.

The results are to be taken from table 6.

Table 6: Curing time

Micopox BP	**Curing time**			
Curing conditions	**Buchholz indentation resistance**	**Mean value**	**Time**	**%**
23 °C , 50 % r. h.	118; 109; 118; 118; 109	**114**	7 d	100
	80; 77; 83; 80; 87	**81**	18 h	71
12 °C , 85 % r. h.	58; 59; 58; 56; 60	**58**	40 h	51

4.2.5 Sensitivity to moisture

The sensitivity to moisture is tested at the same test specimen as was used in 4.2.4 at 12 °C and 85 % r. h. and after 40 h.

The surface of the coated glass panel is examined visually for visible inclusion or emulsification of atmospheric constituents, i.e. blushing.

The results are to be taken from table 7.

Table 7: Sensitivity to moisture

curing conditions	**Sensitivity to moisture Impact of atmospheric constituents**
40 h at 12 °C and 85 % r. h.	no inclusion; no blushing, no tack

4.2.6 Non-volatile matter of the mixture

The non-volatile matter content is determined following DIN ISO 3251 after 24 h storage in standard climate at DIN EN 23270, followed by 3 h drying at 100 °C.
The results are to be taken from table 8.

Table 8: Non-volatile matter content

Material	Non-volatile matter [% by mass]	
	Single values	Mean value
Micopox BP	99,1 ; 99,3 ; 99,1	**99,2**

4.2.7 Extractable matter

The extractable matter was determined following the Soxhlet hot extraction method in accordance with DIN 53738, using the film of *Micopox BP* with a film thickness of about 1 mm, cured at standard climate DIN EN 23270 for seven days, ethanol as the extraction solvent and an extraction time of 16 hours, as a duplicate determination, extraction vessel: glass filter frit.
The extracts were examined qualitatively with regard to their consistency and examined by infrared spectroscopy with regard to their composition (sub-clause 4.1.2).
The results are to be taken from table 9.

Table 9: Extractable matter

Micopox BP	Extractable matter [% by mass]	Composition of the extract
Single values	7,3 ; 7,2	no plasticizers able to migrate and no saponifiable plasticizers
Mean value	**7,3**	

4.2.8 Water absorption

The water absorption was tested in accordance with DIN 53495, method 3 L-23-14d-W, using films cured at standard climate DIN EN 23270 for seven days and of dimensions of approximately 50 mm x 50 mm x 1 mm. The specimens were stored in demineralised water for 14 days and, after removal of surface moisture, immediately weighed to the nearest 0,1 mg

The results are to be taken from table 10.

Table 10: Water absorption

Material	Water absorption [% by mass]	
	Single values	Mean value
Micopox BP	1,5 ; 1,5 ; 1,5	**1,5**

4.3 Tests of the composite specimens

4.3	Nature of the test	Provning Bilaga B	TP-BEL-EP*
4.3.1	Tillverkning av provkroppar / Preparation of the test specimen	12	
4.3.2	Temperaturbeständighet / Hot silicone oil of 250°C	14 A	3.3.3.1
4.3.3	Temperaturbeständighet / method using open flame (welding-on)	14 B	3.3.3.2
4.3.4	Täthet / Freedom from incompletely coated areas	13	3.3.2
4.3.5	Beständighet vid frost och töcykler / -	15	-
4.3.6	Beständighet på ny betong	16	3.4

** "Technical prescriptions and delivery conditions for reactive resins for primers, sealers and scratch screeds under asphalt" („Technische Prüfvorschrift für Reaktionsharze für Grundierungen, Versiegelungen und Kratzspachtelungen unter Asphaltbelägen auf Beton – TP-BEL-EP"), Official German Regulations for bridge sealing with epoxy*

4.3.1 Preparation of the test specimen

Table 11: preparation of the mixtures of the resin/ mortar

Material	Mixing proportion in parts by mass comp. A : comp. B
Micopox BP	100 : 30
Micopox BC	100 : 50

Both components of Micopox BP and Micopox BC were dosed in the above mixing proportion and mixed for about 2 min by stirring, using a spatula.

preparation of the composite specimens - concrete

climate: standard conditions acc. to DIN EN 23270.

substrate: concrete paving flags in accordance with DIN EN 1339, dimension of 400 x 400 x 50 mm, sandblasted, roughness ~0,5 mm

Type A without bituminous membrane on concrete

- Primer Micopox BP, consumption 500 g/m²
- Scattering of silica sand; size 07 – 1,2 mm, consumption 1 kg/m²
- Sealer Micopox BP, consumption 600 g/m²

The waiting period between 1st and 2nd layer of *Micopox BP* was 16 hours.

Type B with bituminous membrane on concrete

- Primer Micopox BP, consumption 500 g/m²
- Scattering of silica sand ; size 07 – 1,2 mm, consumption 1 kg/m²
- Sealer Micopox BP, consumption 600 g/m²
- Waterproofing layer: Deckproof C 5

The bituminous membrane was welded on test composite specimens with a two-flame laying apparatus fed with propane gas.

The waiting period between 1st and 2nd layer of *Micopox BP* was 16 hours. The bituminous membrane was welded after another 2 days.

preparation of the composite specimens – steel

The trial plates were cleaned by high pressure washing with min 20 MPa. High pressure air and grit blasting material fulfilled the demand acc. to corrosion institutes "guideline for control of rustcoating" chapter 2.3 and 2.4.2. The steel surfaces was sandblasted to SA 2 ½ acc to SS-EN ISO 8501-1. with sharp edged sand like grit.

Type C asphalt on steel

- Primer Micopox BP, consumption 150 g/m², ~ 100 µm

Type D with polymer modified asphalt on steel

- Primer Micopox BP, consumption 150 g/m²; ~ 100 µm
- Scratch mortar Micopox BC thickness 0,6 mm
- polymer modified asphalt thickness 30 mm

The waiting period between 1st and 2nd layer was 24 hours. the asphalt was laid down after a period of 7 days.

The asphalt fulfilled the same demands as asphaltmastix acc table 3.1.1,

material	content [%]	requierment [%]
SBS mod bitumen, type Olexobit 45	15	14,0-17,0
calcium filler	32	25,0-38,0
sand 0-2	55	50,0-60,0

but with following changes.
The ballast material fulfilled the demand acc VVTBT bituminous layers, chapt 5. Longtime heating was performed with asphaltmastix with the one used for pouring asphalt. Stamp heating test was performed acc test SS-EN 12697-20. Obtained sinking was between the interval 1-6 mm. The test of form stability acc SS-EN 12970, annex B, was performed at 80 °C.
The temperature of the asphalt was 215 ±5 °C.

All materials fulfilled the requirements for pouring asphalt acc. to VVTBT chapter 4. polymerbitumen contained direct distilled bitumen and min 4,0 weight % SBS polymer. Bitumen was not oxidized. Min 95% of the sand passed 2mm sieve and 100% 4 mm (see figure below).

4.3.2 Resistance to heat, method with hot silicone

The test was carried out with a bath using silicone oil of 250 °C with specimens type A. The temperature and the cooling process was temperature controlled The coated specimens were submitted to the hot silicone oil after 3 days of curing. The examination took place visually after 24 h of cooling down.

The results are to be taken from table 12.

Table 12: Resistance to heat, method with hot silicone oil

Micopox BP	**Resistance to heat**
criterion for examination	**condition after heat stress**
- blisters	no blisters
- cracks	no cracks
- detachments	no detachments
- break out of the sprinkled layer	no break out of the sprinkled layer

4.3.3 Resistance to heat, method using open flame

Specimen of type B were used for this test.
The examination took place visually after 24 h of cooling down and after removal of the bituminous membrane. The tensile bond strength was tested in accordance with EN 1542.

Apparatus used: adhesion tester Type Easy, calibrated
Test speed: 100 N/s
Adhesive: solvent-free two-component-EP adhesive

The results are to be taken from table 13.

Table 13: Resistance to heat, method with open flame

Micopox BP - Resistance to heat	
Criterion for examination	**Condition after heat stress**
• detachment > 0,01 cm² • dense blisters • cracks ≥ 1 mm length • other damage	• no detachments • no blisters • no cracks • no other damage
Tensile bond strength single values [MPa] mean value [MPa]	 3,86 ; 3,11 ; 3,42 ; 3,95, 3,62 3,59
area of failure	100 % concrete

4.3.4 Detection of pores

The test for freedom of pores in the coating was carried out using the exposed test specimens prior to and 24 h after applying the heat stresses (with hot silicone oil of 250°C).
The electrical resistance of the sealing is measured, using a resistance measuring instrument ISOWID of company Siemens and a voltage of 500 V.

The results are to be taken from table 14.

Table 14: Freedom of pores

	Electrical resistance [MΩ]	
	Single values	**Mean value**
Micopox BP	> 2000 ; > 2000 ; > 2000;	> 2000

4.3.5 Beständighet vid frost och töcykler / -

The test of the resistance to freeze and dew cycling was carried out with the specimens of type B.

Remark:
In addition to the regulation of the stipulations of chapter 15 also type A specimen were used, means <u>without</u> bituminous membrane. So the test condition had been intensified acc. to the official test regime to prove the performance of the material under more severe condition.

The specimens were isolated with polystyrene on the back and the four sides of the concrete slabs. On the coated upper surface of the specimens a solution of 3% sodium chloride remains throughout the test series. The tests were carried out with 3 samples each. The freeze and dew cycling was carried out in accordance with SS 13 72 44 with 56 cycles.

Parameters of one cycle:

time [h]	**temperature [°C]**	**time [h]**	**temperature [°C]**
	upper limit		**lower limit**
0	24,0	0	16,0
5	-3,0	3	-5,0
12	-15,0	12	-22,0
16	-18,0	16	-22
18	-1,0	20	-1,0
22	24,0	24	16

After completion of the freeze and dew cycling the bituminous membrane of type B specimens was removed and all specimens (type A and B) were inspected about detachments, cracks and blisters in the coating.

Result:
After the alternating temperature stress no cracks, blisters or detachments could be detected by visual assessment in any specimen.

Tensile bond strength

The tensile bond strength was determined following EN 1542.

Apparatus used:	adhesion tester Type Easy, calibrated
Test speed:	200 N/s
Adhesive:	solvent-free two-component-PU-adhesive

The results are to be taken from the following table 15.

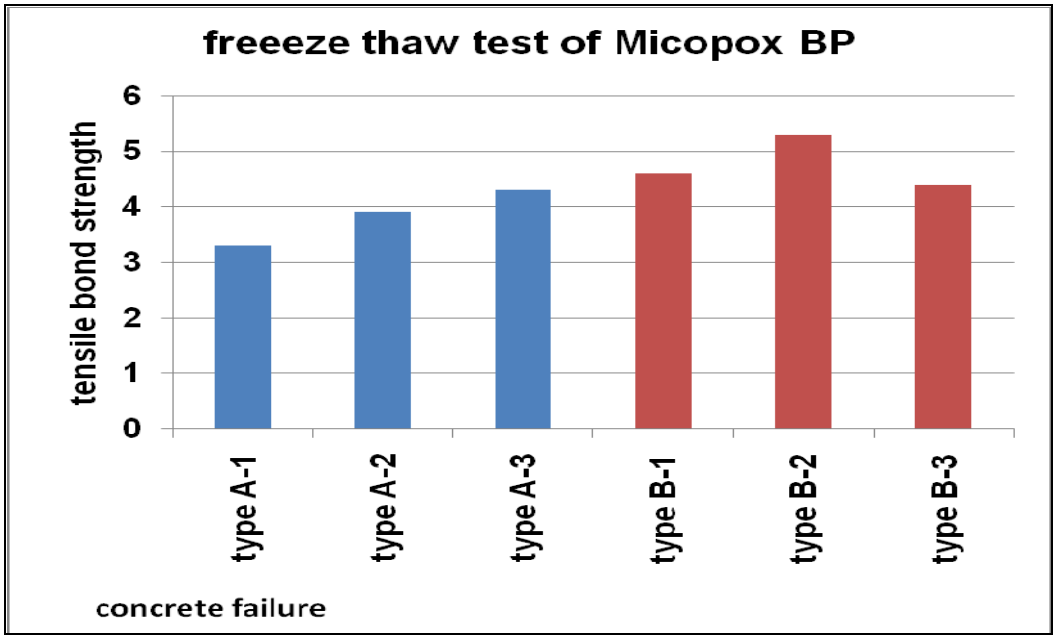
Table 15: Tensile bond strength after freeze and dew cycling

type	**No.**	**Tensile bond strength [N/mm²] Single values**
Type A	1	3,3
	2	3,9
	3	4,3
Type B	1	4,6
	2	5,3
	3	4,4
Mean value		**4,3 (3,0)**

The value in parenthesis symbolizes the requirement level.

In any case a failure of concrete occurred

figure 1: freeze dew test of Micopox BP



4.3.6 Beständighet på ny betong / -

The usability on fresh concrete is assessed by testing the pull-off strength of composite specimens.

Concrete specimens at the age of 7 days acc. To DIN EN 1766 were prepared in accordance with sub-clause 3.4.1 of TP-BEL-EP:

- 1 day in formwork
- 5days in water at 20°C
- sand blasting opf the surface to seal at day no. 6 (surface roughness 0,45 mm)
- wrapping of 5 sides with PE-foil and storage at 8°C / 85 % of rel. Hum.
- 7 days after specimen production: sealing of the top surface

After another 7 days storage at the above climate a bituminous membrane is welded on one composite specimen.

After removing the sheeting, the pull-off strength is measured in accordance with EN 1542 both on the specimen stressed by welding as well as on the unstressed reference specimen, at 5 measuring areas of each specimen.

Apparatus used:	adhesion tester Type Easy, calibrated
Test speed:	100 N/s
Adhesive:	solvent-free two-component-EP adhesive
Test temperature:	23 °C ± 2 K

Results:

No detachments, blisters, cracks or other destruction appeared in the sealing after stress by welding on a bituminous membrane.

The results of testing the pull-off strength are to be taken from table 16.

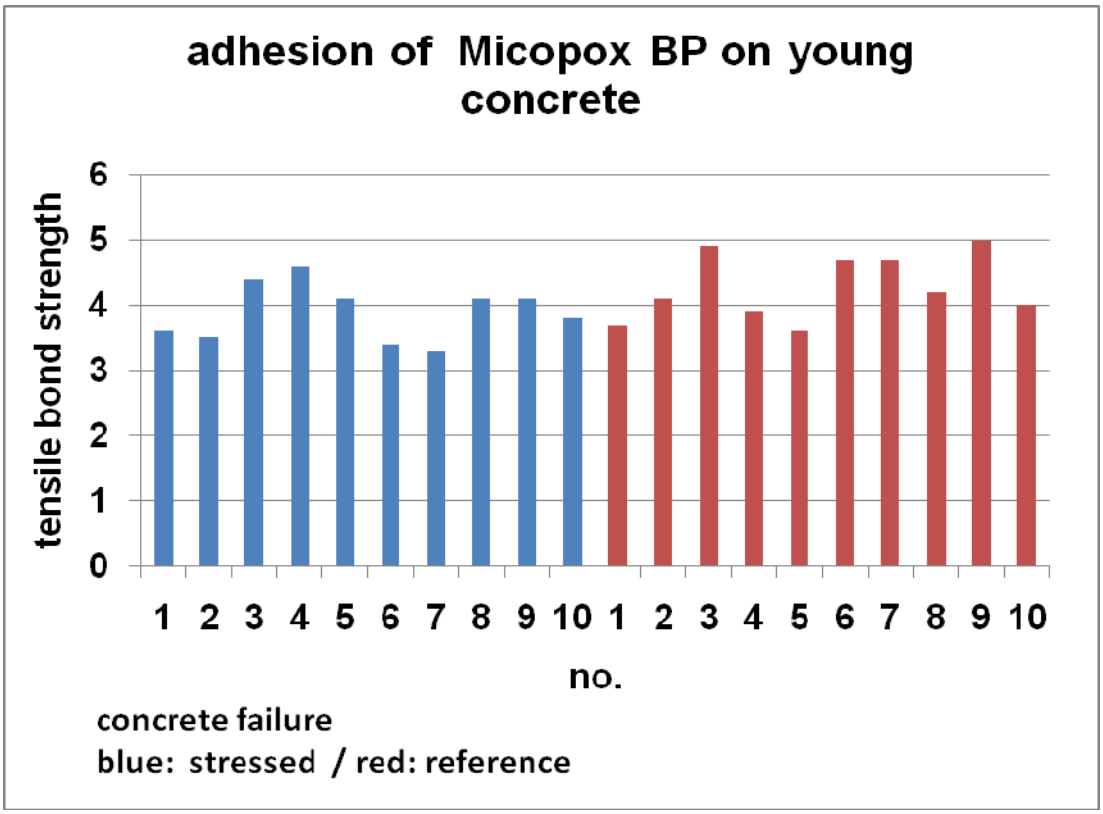
Table 16: Pull off strength on stressed and unstressed specimens

specimen / Kondition	**Pull off strength [N/mm²]**
stressed 1	3,6 3,5 4,4 4,6 4,1
stressed 2	3,4 3,3 4,1 4,1 3,8
mean value	**3,9 (1,5)**
unstressed 3	3,7 4,1 4,9 3,9 3,6
unstressed 4	4,7 4,7 4,2 5,0 4,0
mean value	**4,3 (1,5)**

The value in parenthesis symbolizes the requirement level.

In any case a failure of concrete occurred.

figure 2: adhesion on young concrete of Micopox BP



5 Tests as steel primer / Krav för epoxipreparat, Stålprimer

The tests describe the materials hardening behaviour at ambient, low temperature and in contact with hot asphalt when used as a steel primer on bridges.

	Nature of the test	Provning Bilaga B	TP-BEL-EP*
5.1	Härdningstid	17	-
5.2	Vidhäftning	18	-
5.3	Gjutasphalt	19	-

5.1 Härdningstid /Curing time

A specimen of type C was used.

The adhesion towards steel was tested after 18 hours curing time at standard condition acc to DIN EN 23270.

The result and the requirement are shown in table 17 and figure 3.

5.2 Vidhäftning

A specimen of type C was used.

The adhesion towards steel was tested after 7 days curing time at standard condition acc to DIN EN 23270.

The result and the requirement are shown in table 17 and figure 3.

5.3 Gjutasfalt

A specimen of type D was used.

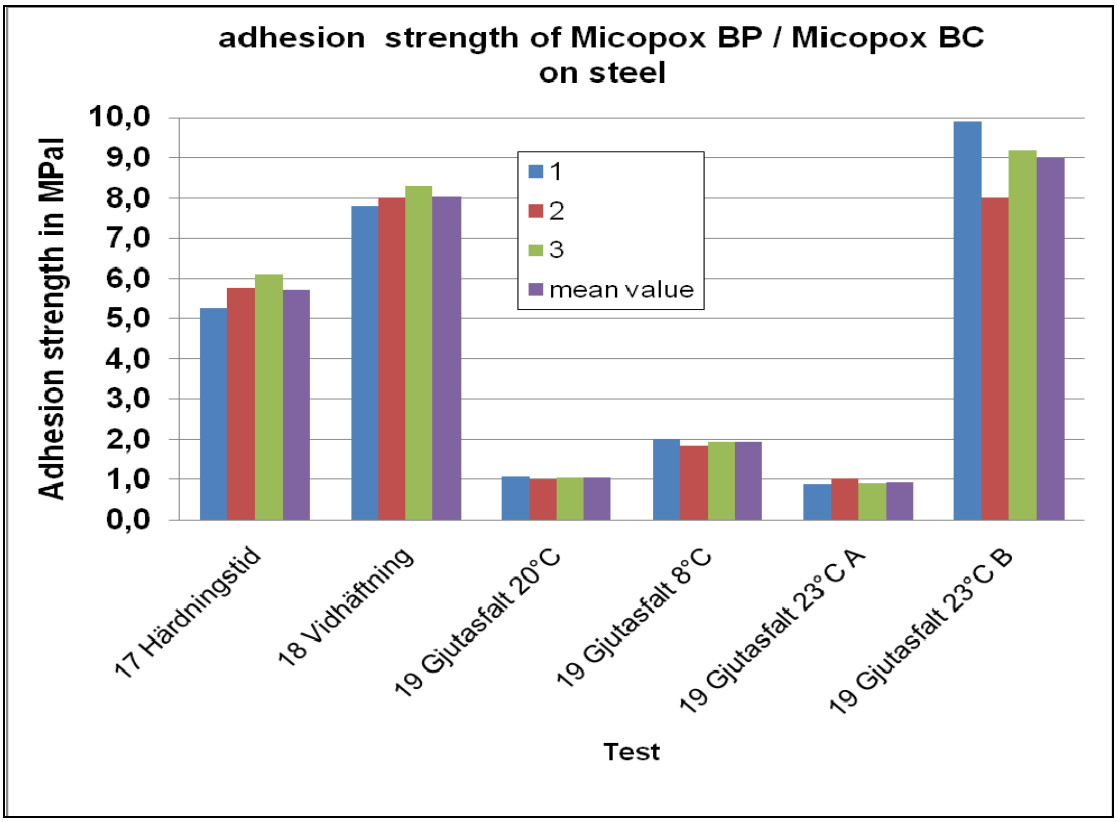
Adhesion between pouring asphalt and epoxy was tested after storage in 5 days in standard condition acc to DIN EN 23270. The tensile strength test was performed at 20 ± 1°C and additionally at 8°C and 23°C storage.

Remark: supplementary test at 23 °C
Additionally the test surface was cleaned after the first test series under standard condition “19 Gjutasfalt 23°C A” and the rupture of the asphalt. Subsequently the tensile strength of the remaining organic layer of Micopox BP and Micopox BC was tested at the same test area in a second trial. These values are indicating the adhesion of Micopox BP/ Micopox BC on steel after the thermal load with asphalt. The result of these supplementary tests is indicated as “19 Gjutasfalt 23°C B“ as well as all results for application on steel are laid down in the following table 17 and figure 1.

Table 17: adhesion strength of Micopox BP / Miocopox BC after stress

test	adhesion strength [MPa]					
	nr. 1	nr. 2	nr. 3	mean value	failure	require -ment
17 Härdningstid	5,27	5,75	6,11	5,71	in epoxy	>4,0
18 Vidhäftning	7,80	8,00	8,30	8,03	in epoxy	> 8,0
19 Gjutasfalt 20°C	1,06	1,00	1,03	1,03	adhesion loss of asphalt	> 1,0
19 Gjutasfalt 8°C	2,00	1,83	1,94	1,92	adhesion loss of asphalt	-
19 Gjutasfalt 23°C A	0,88	1,02	0,90	0,93	adhesion loss of asphalt	-
19 Gjutasfalt 23°C B	9,90	8,00	9,20	9,00	in epoxy	-

figure 3: adhesion strength after stress of Micopox BP / Micopox BC



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6 SUMMARY

Polymer Institut was charged by company Elmico A/S, N-Skarnes to carry out the performance testing of the concrete and steel bridge sealing epoxy resins

Micopox BP / Micopox BC

in accordance with the Swedish stipulations

VVTBT – Tätskikt på broar

chapter 5 Tätskikt med epoxi

chapter 8 Förseglingar

Bilaga B Krav på epoxipreparat

- Tabell 1 Krav for epoxipreparat - Allmänt ingående komponenter
- Tabell 2 Krav for epoxipreparat Allmänt blandning eller härdat material
- Tabell 3 Krav for epoxipreparat Allmänt provkroppar
- Tabell 4 Krav for epoxipreparat Allmänt stalprimer
- Tabell 5 Krav på vidhäftning mellan försegling av epoxi och gjutaspahtl

The tested material fulfilled all requirements listed in these technical prescriptions for sealing of concrete and steel bridges.

Flörsheim-Wicker, 2010-01-24

The head of the institute

J. Magner

Polymer Institut Flörsheim-Wicker