

Test report no.: 69298/05-I

Customer: Sika Deutschland GmbH
Kornwestheimer Straße 107
70439 Stuttgart
GERMANY

Order: Testing of chemical resistance towards liquid manure
of joint sealant Sikaflex®-TS plus

Letter of: 2005-09-02 **Ref:** Mr Ralf Heinzmann

Receipt of samples: 2005-08-18 **Sampling:** ---

Test period: 2005-09-22 to 2006-11-16

This test report comprises 9 pages.

Würzburg, 2006-12-06
Mü/ste/we *sw*

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1. Order

In its letter of 2 September 2005 the company Sika Deutschland GmbH, Kornwestheimer Straße 107, 70439 Stuttgart, GERMANY, instructed SKZ - TeConA GmbH to test the chemical resistance towards liquid manure of joint sealant Sikaflex®-TS plus.

2. Test material

On 18 August 2005 SKZ - TeConA GmbH received following samples:

6 foil bags à 600 ml of one-component joint sealant

Designation: Sikaflex®-TS plus
Base material: Polyurethane
Color: black
Charge: 0010993748

1 can of cleaner

Designation: Sika-Haftreiniger-1
Charge: 0010990445

1 can of primer

Designation: Sika-Primer 3
Charge: 3M-045

3. Test procedure

Testing of chemical resistance towards liquid manure of joint sealant **Sikaflex®-TS plus** was determined on one hand by amending adhesion and cohesion behavior, specified by shear strength, stress-strain value $\sigma_{\varepsilon 100\%}$, as well as elongation at break ε_B , on the other by compatibility, specified by amending tensile strength σ_{\max} and its elongation ε_M and change in weight, after storage in test liquid.

DIBt-test program proposed to use a test liquid as aqueous solution for inner layers of containers made of steel concrete for storing liquid manure and silage liquids to meet the requirements according to DIN 11622-2, issue October 1999. This test liquid is composed as follows:

Test liquid to simulate liquid manure

A solution of 7 % $(\text{NH}_4)_2\text{HPO}_4$, adjusted by means of NH_4OH to reach pH-value 8.5 to 9.0, if necessary.

All samples including foil (see sample production) have been pre-stored for 6 weeks at standard conditioning atmosphere 23/50-2 according to DIN EN ISO 291. Thereafter samples as well as films have been stored in specific liquid manure for 13, 26 and 52 weeks. The proportion of sample surface to test liquid volume was at least 1 cm² sample surface to 4 cm³ test liquid. During storage test liquid was changed once.

Usually we carry out tests according to standards for which we have an accreditation. The list of all standards for which we are accredited is shown on the homepage at www.skz.de.

Preparation of samples:

For testing the amending shear strength samples with an overlapping joint of a width of 50 mm, a length of 30 mm and a thickness of 2 mm were made. Glass samples as well as steel samples made of special steel 1.4301 (V2A) of the dimensions 100 mm x 50 mm were used as substrate.

The glass for the glass samples have been cleaned by means of water of a reduced surface tension (dishwashing detergent) and then by acetone and thereafter by desalinated water again. The glass samples have been joined without any primer. Steel samples have been cleaned first by means of Sika-Haftreiniger 1 (cleaner), then Sika Primer 3 was applied on the steel substrate.

In adoption to DIN EN ISO 8340: 2005-09 samples were made of concrete for testing amended stress-strain value $\sigma_{e100\%}$ including elongation at break ϵ_B . These samples were made of concrete according to ISO 13640: 1999-12. Their adhesive surfaces were pre-treated by means of Sika Primer 3.

After application the primer was evaporated for about 100 minutes.

For testing compatibility foils of a thickness of 2 mm were pressed out of joint sealant. According to DIN 53504, dumbbell samples S 2 were punched out of abovementioned foils to determine the tensile strength σ_{\max} as well as coordinated elongation ϵ_M . Disk-shaped samples (test disks, diameter of 30 mm and thickness of 2 mm) were made out of these foils to determine amendment in weight.

3.1 Testing of amended adhesion and cohesion behavior after storage in test liquid

3.1.1 Testing of amended shear strength of glass samples and steel samples

Shear strength was determined on samples with overlapping joints of substrates glass and V2A-steel. After having taken samples out of test liquid they have been cleaned with distilled water and stored for 24 hours in a standard atmosphere 23/50-2.

Testing speed was 5 mm/min. The clamping jaws of the tensile testing device were adjusted so that the force was applied at joint level.

It was determined maximal shear strength in N/mm^2 on samples of overlapping joints as well as elongation in %. Mean \bar{x} was created out of 5 single values. Kind of fracture (cohesion / adhesion) was evaluated.

3.1.2 Testing of amended stress-strain value as well as elongation at break for samples with substrate concrete

According to ISO 8340: 2005-09 stress-strain value $\sigma_{\epsilon 100\%}$ as well as elongation at break ϵ_B were determined on concrete samples after storage in test liquid. After having taken samples out of test liquid they were cleaned with distilled water and stored for 24 hours in a standard atmosphere 23/50-2.

Test speed was 5 mm/min.

Mean \bar{x} was created out of 5 single values. Kind of fracture (cohesion / adhesion was) evaluated.

3.2 Testing of compatibility after storage in test liquid

3.2.1 Testing of amended shear strength σ_{\max} including elongation ϵ_M

According to DIN 53504, tensile property of dumbbell samples S 2 were tested to determine the amended tensile strength σ_{\max} including elongation ϵ_M prior to and after storage in test liquid. After having taken samples out of test liquid they have been cleaned with distilled water and stored for 24 hours in a standard atmosphere 23/50-2.

Test speed was 200 mm/min.

After storage in test liquid samples have been additionally stored for 7 days at standard atmosphere 23/50-2. Abovementioned values were measured again and referred to initial values (preconditioning). Mean \bar{x} was created out of 5 single values.

3.2.2 Testing of amendment in weight

Prior to and after storage in test liquid mass of test disks were compared to determine amended weight.

After having taken samples out of test liquid they were dried on an absorbent paper, dried and weighed immediately.

After storage in test liquid samples have been additionally stored for 7 days at standard atmosphere 23/50-2. Weight was measured again and referred to initial values (preconditioning).

Mean \bar{x} was created out of 3 samples.

4. Test results

4.1 Amended adhesion and cohesion behavior after storage in test liquid

4.1.1 Testing of shear strength of glass samples and steel samples

Shear strength				
Storage	Substrate	σ -max in N/mm ²	Elongation at σ -max in %	Fracture
6 weeks at atmosphere 23/50-2	glass	0.79 (s = ---)	54 (s = ---)	100 % cohesion
13 weeks	glass	0.81 (s = 0.06)	48 (s = 12.8)	100 % cohesion
26 weeks	glass	0.85 (s = 0.09)	51 (s = 5.6)	100 % cohesion
52 weeks	glass	0.92 (s = 0.07)	42 (s = 11.3)	100 % cohesion
6 weeks at atmosphere 23/50	special steel	0.52 (s = 0.05)	55 (s = 2.9)	100 % cohesion
13 weeks	special steel	0.48 (s = 0.08)	57 (s = 11.7)	50 % cohesion / 50 % adhesion
26 weeks	special steel	0.58 (s = 0.07)	59 (s = 8.2)	50 % cohesion / 50 % adhesion
52 weeks	special steel	0.61 (s = 0.04)	63 (s = 6.1)	100 % cohesion

s = standard deviation

4.1.2 Amended stress-strain value as well as elongation at break for samples with substrate concrete

Stress-strain value / Elongation at break				
Storage	Substrate	σ at $\varepsilon = 100\%$ in N/mm^2	Elongation at break ε_B in %	Fracture
6 weeks at atmosphere 23/50-2	concrete	0.47 (s = 0.01)	340 (s = 51)	100 % adhesion
13 weeks	concrete	0.48 (s = 0.01)	390 (s = 32)	100 % adhesion
26 weeks	concrete	0.51 (s = 0.01)	362 (s = 64)	mainly cohesion
52 weeks	concrete	0.52 (s = 0.02)	293 (s = 46)	mainly cohesion

s = standard deviation

4.2 Compatibility after storage in test liquid

4.2.1 Amended shear strength σ_{\max} including elongation ε_M

Storage	Tensile strength σ_{\max} in N/mm^2	Elongation ε_M in %
6 weeks at atmosphere 23/50-2	1.02 (s = 0.01)	557 (s = 25)
13 weeks at atmosphere 23/50	1.16 (s = 0.02)	464 (s = 48)
26 weeks at atmosphere 23/50	1.25 (s = 0.05)	433 (s = 67)
52 weeks at atmosphere 23/50-2	1.32 (s = 0.02)	403 (s = 29)
13 weeks in test liquid	1.13 (s = 0.02)	542 (s = 31)
+7 days at atmosphere 23/50-2	1.18 (s = 0.02)	497 (s = 36)
26 weeks in test liquid	1.14 (s = 0.03)	491 (s = 60)
+7 days at atmosphere 23/50-2	1.17 (s = 0.05)	469 (s = 71)
52 weeks in test liquid	1.13 (s = 0.02)	456 (s = 24)
+7 days at atmosphere 23/50-2	1.14 (s = 0.03)	460 (s = 31)

s = standard deviation

4.2.2 Amendment in weight

Storage	Amendment in weight %
13 weeks	+1.1
+7 days at atmosphere 23/50-2	0.0
26 weeks	+1.7
+7 days at atmosphere 23/50-2	+0.6
52 weeks	+2.5
+7 days at atmosphere 23/50-2	+1.1

5. Assessment of test results

5.1 Adhesion and cohesion behavior after storage in test liquid

After a storage of 52 weeks in test liquid mentioned in item 3 shear strength of glass and steel samples with overlapping joints increased to 16 % and 17 %. Adhesion to corresponding samples has always been very good (100 % cohesion fracture).

While adhesion has always been very good, too, stress-strain value $\sigma_{\varepsilon 100\%}$ was slightly increased on concrete samples. Even after storage in test liquid determined elongation values have always reached a constant high level. Joint sealant did not get brittle at all.

Due to storage in test liquid adhesion and cohesion behavior was only slightly reduced.

5.2 Compatibility after storage in test liquid

After a storage of 52 weeks in test liquid tensile tests resulted in a slight increase of tensile strength σ_{\max} and decrease of elongation ε_M referred to initial values.

Due to storage in test liquid this amended or reduced elastic behavior of joint sealant of samples was even lower than during a storage in standard atmosphere.

After a conditioning of 7 days in standard atmosphere there wasn't any significant differences.

After a storage of 52 weeks in test liquid first weight was slightly increased by 2.5 %, but after a storage of 7 days in standard atmosphere increased weight was reduced to 1.1 % only.

As a result test liquid did not influence joint sealant negatively.