

## Test report

**Test report relating to a structural sealant according to ETAG 002 - Guideline for European technical approval for structural sealant glazing kits (SSGK) May 2012 - concerning the product marked as: MF899-25 Silicone structural sealant, manufactured by: Zhengzhou Zhongyuan Silande High Technology Co. Ltd**

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# 1 Introduction

## 1.1 Purpose

The tests have been performed in order to determine the properties of a sealant according to ETAG 002 standard [1].

### Revision 01

The test from paragraph 5.1.4.6.6 “UV resistance of the sealant” just failed one of the requirements. The client requested to include extra measurements with a set of H-samples which were received on 16 November 2017. These samples are made with the same sealant as the ordinal tested H-samples. The results of these test are include in appendix D of the report.

## 1.2 Description of the samples

### General

Name of the Manufacturer	Zhengzhou Zhongyuan Silande High Technology Co. Ltd
Address of the manufacturer	No. 28 Dongqing West St Zhengzhou Hi-tech Industrial Development Zone, Henan China
Production plant of the samples	Zhengzhou
Line ID where the samples are made	Turello TMD 3000 mixer
Production date	2016-11-2
Sampling date	2017-2-15
The product was marked as	MF899-25 Silicone structural sealant
Trade mark	Silande

### Specific

Samples see annex A	H samples; 50x50 substrate and 12x12x50 mm sealant joint (sample type 1)
	150 mm circular 2.2 mm thick cured film (sample type 2)
	tubes containing MF899-25
Sealant material	
Type	silicone, structural sealant
Batch No.	no batch number given
Colour	Black
Substrate Specification	
A Type of glass (coating)	Clear float glass, no coating, 6 mm thick
B Aluminium	no specifications given, 3 mm thick

### **1.3 Sampling procedure**

The samples have been submitted by the assignor. The test house, acting as notified test body, has had no influence on the selection of the samples. The samples were received on 18 April 2017.

### **1.4 Application**

The request for testing was submitted by the assignor on 25 April 2017. Assignment Form number: 16.A315.

### **1.5 Method of testing**

All applicable tests have been performed according to the standard ETAG 002-1 [1].

### **1.6 Put out to contract**

No tests were performed at third parties.

### **1.7 Privacy statement**

Due to privacy reasons, the names of involved personnel that executed the tests are not disclosed in the report. However, this information is available on internal work sheets, test forms etc. in the project file.

### **1.8 Notifications, accreditations, designations**

TÜV Rheinland Nederland B.V. has been notified by the Dutch Ministry of Infrastructure and the Environment as Notified Test Body (number 1750) and Notified Certification Body (number 0336) for the European Construction Products Directive 89/106/EEC.

TÜV Rheinland Nederland B.V. has been accredited by the Dutch Accreditation Council (RvA) as ISO 17025 Test Laboratory (nr. L 484) and ISO 17065 Certification Body (nr. C078).

TÜV Rheinland Nederland B.V. has been designated as Technical Service (Laboratory) by the Approval Authorities for the Netherlands (RDW – E4) and Germany (KBA – E1) to grant approvals as mentioned in Directive 70/156/etc. and in the 1958 Agreement of the Economic Commission for Europe of the United Nations (UN-ECE) for glass as used in the automotive sector: ECE Regulation 43, safety glazing; EC Directive 92/22, Safety glass; EC Directive 2009/144, Glazing cat. T.

TÜV Rheinland Nederland B.V. has been recognised by the German Institute for building technics (DIBt) under number NL005.

## 2 Procedure

All tests were performed according to ETAG 002-1: 2012 [1]. Before testing the samples were stored for a minimum of 28 days in an air conditioned room at (23±2) °C and (50±5) % relative humidity and tested at this condition unless otherwise specified.

The following tests were conducted.

Table 2.1 Overview tests

ETAG test §	Characteristic	Conditions	Results
5.1.4.1 Initial mechanical strength	5.1.4.1.1 Tensile strength	Tensile strength at -20 °C / +23 °C / +80 °C  Sample type 1	Stress at elongations of 5, 10, 15, 20 and 25 %; Stiffness $K_{12.5}$ ; maximum stress/strain <sup>1)</sup> ; failure mode <sup>2)</sup>
	5.1.4.1.2 Shear strength	Shear strength at -20 °C / +23 °C / +80 °C  Sample type 1	Stress at relative displacement of 5, 10, 15, 20 and 25 %; maximum stress/strain; failure mode
5.1.4.2 Residual mechanical strength after artificial ageing	5.1.4.2.1 Immersion in water and UV radiation	Tensile strength after 504±4 and 1008±4 hours exposure in water/UV at (45±1) °C. Glass side directed to UV light source. Sample type 1	Stress at elongations of 5, 10, 15, 20 and 25 %; maximum stress/strain; failure mode
	5.1.4.2.2 Salt spray exposure	Tensile strength after 480±4 hours ISO 9226 NSS atmosphere exposure. Sample type 1	
	5.1.4.2.3 SO <sub>2</sub> exposure	Tensile strength after 20 cycles ISO 3231 SO <sub>2</sub> atmosphere exposure. Sample type 1	
	5.1.4.2.4 Façade cleaning product	Tensile strength after 21 days immersion in tap water with 1% dish detergent (Dreft, Procter & Gamble) at (45±1) °C. Sample type 1	
5.1.4.6 Physical properties	5.1.4.6.1 Gas inclusions	1 Sample prepared by TUV according to figure 14 of ETAG 002-1	Occurrence of gas bubbles within 21 days
	5.1.4.6.2 Elastic recovery	EN ISO 7389 after method A conditioning 25% extension at (23±2) °C. Sample type 1	Initial and final extension; Elongation after 1 hour unloading; recovery
	5.1.4.6.3 Shrinkage	Volume change after conditioning in accordance with ISO 10563. Sample: metal rings filled with sealant prepared by TUV	Change in volume (shrinkage)
	5.1.4.6.3 Resistance to	Tensile strength at +23 °C with at two sides an incision	Stress at elongations of 5, 10, 15, 20 and 25 %;

	tearing	in the sealant. Sample type 1	maximum stress/strain; failure mode
	5.1.4.6.5 Mechanical fatigue	Tensile strength after repetitive mechanical load as per ETAG 002- 1 § 5.1.4.6.5 Sample type 1	Stress at elongations of 5, 10, 15, 20 and 25 %; maximum stress/strain; failure mode
	5.1.4.6.6 UV resistance of the sealant	Tensile strength sealant material ISO 527-3 sample type 5, of unexposed specimens and specimens exposed to (504±4) hours UV radiation (Osram Ultra Vitalux lamps) as per ETAG 002-1 § 5.1.4.6.6	Tensile strength; Elongation at max Secant modulus at 0.5-1% and between 5-25%
	5.1.4.6.7 Elastic modulus sealant	Tensile test ISO 527-3 Sample type 5	
	5.1.4.6.8 Creep under long term shear	Simultaneous specific (static) tensile and shear load for 91 days Samples type 3	Residual deformation 24 hours after unloading
5.2 Identification of product	5.2.1.1 Specific mass	Mass according to ISO 1183-1 method A, immersion method of cured sealant cut specimens from sample type 2	Specific mass
	5.2.1.2 Hardness	Shore A hardness ISO 868 of cured sealant cut specimens from sample type 2	Shore A
	5.2.1.3 Thermo gravimetric analysis	TGA plot up to 900°C from cured sealant based on ISO 7111	TGA diagram
	5.2.1.4 Colour	ISO colour coordinates of cured sealant	colour coordinates

1) The stress and strain at rupture is interpreted as the maximum force measured and corresponding elongation.

2) The failure mode is noted as percentage of C (cohesive failure in the sealant) or A (adhesive failure at the substrate).

## 2.1 Test equipment and measurement uncertainty

All tensile tests were performed on a Zwick Z100 universal test machine with a test speed of 5 mm/min and 20 kN load cell. The displacement was measured with the crosshead movement.

Uncertainty on measured dimensions: ±0.05 mm

Tensile test

Stress  $\sigma$ : ±0.01 MPa

Stress  $\varepsilon$ : ±0.5% ( absolute)

Mass: ±0.001 g

Hardness: ±1

### 3 Test results

#### 3.1 Initial mechanical strength

Tensile and shear strength at -20, +23 and +80 °C of H-sample (sample type 1) after performing all applicable conditioning steps according to the relevant ETAG 002-1 [1] paragraphs.

Table 3.1.1 Results tensile strength initial at -20 °C

	K12,5	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	N/mm <sup>2</sup>	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	3.14	0.21	0.34	0.44	0.52	0.58	1.35	202	100% C
2	3.53	0.27	0.39	0.49	0.56	0.63	1.29	211	100% C
3	3.36	0.24	0.37	0.47	0.54	0.61	1.29	225	100% C
4	3.48	0.26	0.38	0.48	0.56	0.63	1.31	199	100% C
5	3.36	0.25	0.37	0.46	0.54	0.61	1.30	203	100% C
X <sub>mean</sub>	3.37	0.25	0.37	0.47	0.55	0.61	<b>1.31</b>	208	
s	0.15	0.02	0.02	0.02	0.02	0.02	0.02	11	
R <sub>u,5</sub>							<b>1.26</b>		

Table 3.1.2 Results tensile strength initial at 23 °C

	K12,5	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
No.	N/mm <sup>2</sup>	MPa	MPa	MPa	MPa	MPa	MPa	%	mode
1	3.26	0.18	0.34	0.46	0.55	0.62	0.96	94	100% C
2	3.27	0.19	0.35	0.46	0.55	0.62	0.95	101	100% C
3	3.22	0.19	0.34	0.45	0.54	0.61	1.02	132	100% C
4	3.08	0.16	0.32	0.44	0.53	0.60	0.96	111	100% C
5	3.37	0.20	0.36	0.47	0.56	0.63	1.09	147	100% C
6	3.35	0.20	0.36	0.47	0.56	0.63	1.15	169	100% C
7	3.51	0.23	0.38	0.49	0.57	0.64	0.95	83	100% C
8	3.33	0.21	0.36	0.47	0.55	0.61	1.00	121	100% C
9	3.37	0.22	0.36	0.47	0.56	0.62	1.06	134	100% C
10	3.35	0.22	0.36	0.47	0.55	0.62	1.11	161	100% C
X <sub>mean</sub>	3.31	0.20	0.35	0.47	0.55	0.62	<b>1.02</b>	125	
s	0.11	0.02	0.02	0.01	0.01	0.01	0.07	29	
R <sub>u,5</sub>							<b>0.87</b>		

Table 3.1.3 Results tensile strength initial at 80 °C

	K12,5	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	N/mm <sup>2</sup>	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	3.29	0.19	0.35	0.47	0.57	0.66	0.92	55	100% C
2	3.32	0.19	0.35	0.47	0.58	0.67	0.99	54	100% C
3	3.21	0.17	0.33	0.46	0.57	0.66	0.93	50	100% C
4	3.20	0.17	0.33	0.46	0.57	0.66	1.01	55	100% C
5	3.29	0.19	0.35	0.47	0.57	0.66	0.96	57	100% C
X <sub>mean</sub>	3.26	0.18	0.34	0.47	0.57	0.66	<b>0.96</b>	54	
s	0.05	0.01	0.01	0.01	0.00	0.00	0.04	3	
R <sub>u,5</sub>							<b>0.86</b>		

Table 3.1.4 Results shear strength initial at -20 °C

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.03	0.06	0.09	0.12	0.15	1.08	224	100% C
3	0.05	0.08	0.12	0.15	0.18	1.10	218	100% C
4	0.02	0.04	0.07	0.10	0.13	1.13	232	100% C
5	0.02	0.05	0.09	0.12	0.14	1.00	219	100% C
6	0.04	0.07	0.11	0.14	0.16	1.13	222	100% C
X <sub>mean</sub>	0.03	0.06	0.09	0.13	0.15	<b>1.09</b>	223	
s	0.01	0.02	0.02	0.02	0.02	0.05	5	
R <sub>u,5</sub>						<b>0.97</b>		

Table 3.1.5 Results shear strength initial at 23 °C

	s-5%	s-10%	s-15%	s-20%	s-25%	-max	-max	failure
No.	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.04	0.08	0.11	0.14	0.17	0.88	183	100% C
2	0.03	0.06	0.10	0.13	0.15	0.85	173	100% C
3	0.04	0.07	0.11	0.14	0.17	0.87	178	100% C
4	0.04	0.08	0.12	0.15	0.18	0.91	190	100% C
5	0.04	0.07	0.11	0.14	0.17	0.81	166	100% C
6	0.04	0.07	0.10	0.14	0.17	0.90	189	100% C
7	0.04	0.08	0.11	0.15	0.18	0.88	188	100% C
8	0.04	0.08	0.12	0.15	0.18	0.91	187	100% C
9	0.04	0.08	0.12	0.15	0.18	0.93	196	100% C
10	0.04	0.08	0.11	0.14	0.17	0.88	185	100% C
X <sub>mean</sub>	0.04	0.08	0.11	0.14	0.17	<b>0.88</b>	175	
s	0.00	0.01	0.01	0.01	0.01	0.04	9	
R <sub>u,5</sub>						<b>0.80</b>		

Table 3.1.6 Results shear strength at 80 °C

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.03	0.07	0.11	0.14	0.17	0.66	118	100% C
2	0.04	0.08	0.12	0.16	0.19	0.79	123	100% C
3	0.03	0.07	0.10	0.14	0.17	0.82	130	100% C
4	0.05	0.09	0.13	0.17	0.20	0.78	138	100% C
5	0.04	0.08	0.12	0.16	0.20	0.86	133	100% C
X <sub>mean</sub>	0.04	0.08	0.12	0.15	0.19	<b>0.78</b>	128	
s	0.01	0.01	0.01	0.01	0.01	0.07	8	
R <sub>u,5</sub>						<b>0.61</b>		



### 3.2 Residual mechanical strength after artificial ageing

Tensile strength of H-samples (1) after performing all applicable conditioning and or exposure steps according to the relevant ETAG 002-1 [1] paragraphs.

#### § 5.1.4.2.1 Immersion in water and UV radiation

Pre-treatment: 10 test pieces are immersed in demineralised water with a temperature of  $(45 \pm 1)^\circ\text{C}$  with the glass substrate flushed with the water level in combination with solar radiation. The U.V. source are new Osram UltraVitalux 300 W lamps, placed 25 cm from the glass surface of the samples. After  $(504 \pm 4)$  hours 5 test pieces were conditioned for 24 hours at standard laboratory conditions and subjected to a tensile test. After another  $(504 \pm 4)$  hours exposure the remaining 5 test pieces were tested after 24 hours at standard laboratory conditions.

Table 3.2.1 Results after  $(504 \pm 4)$  hours immersion in water and U.V. radiation

	K <sub>12,5</sub>	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	MPa	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	2.84	0.17	0.30	0.40	0.47	0.53	0.85	158	100% C
2	2.80	0.16	0.30	0.39	0.47	0.53	0.86	171	100% C
3	2.92	0.19	0.32	0.41	0.48	0.53	0.89	170	100% C
4	2.74	0.16	0.29	0.39	0.46	0.51	0.86	178	100% C
5	2.73	0.14	0.29	0.39	0.47	0.52	0.87	195	100% C
X <sub>mean</sub>	2.80	0.16	0.30	0.40	0.47	0.53	<b>0.87</b>	139	
s	0.08	0.02	0.01	0.01	0.01	0.01	0.02	16	
R <sub>u,5</sub>							<b>0.82</b>		

Table 3.2.2 Results after  $(1000 \pm 8)$  hours immersion in water and U.V. radiation

	K <sub>12,5</sub>	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	MPa	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	2.84	0.18	0.31	0.40	0.47	0.52	0.87	144	100% C
2	2.62	0.15	0.28	0.37	0.44	0.49	0.87	153	100% C
3	2.74	0.17	0.29	0.38	0.45	0.51	0.84	191	100% C
4	2.73	0.16	0.29	0.38	0.45	0.51	0.86	174	100% C
5	2.70	0.16	0.29	0.38	0.45	0.50	0.88	184	100% C
X <sub>mean</sub>	2.73	0.17	0.29	0.38	0.45	0.51	<b>0.86</b>	169	
s	0.08	0.01	0.01	0.01	0.01	0.01	0.01	20	
R <sub>u,5</sub>							<b>0.84</b>		

§ 5.1.4.2.2 Salt spray exposure

Table 3.2.3 Results after salt spray exposure

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
No.	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.16	0.29	0.38	0.46	0.52	0.93	174	100% C
2	0.16	0.30	0.40	0.47	0.54	0.93	136	100% C
3	0.16	0.29	0.38	0.45	0.51	0.95	174	100% C
4	0.16	0.28	0.37	0.44	0.50	0.93	163	100% C
5	0.16	0.29	0.38	0.45	0.52	0.93	175	100% C
6	0.17	0.30	0.39	0.47	0.53	0.93	154	100% C
7	0.16	0.29	0.39	0.46	0.52	1.00	157	100% C
8	0.17	0.31	0.40	0.48	0.54	0.92	167	100% C
9	0.18	0.31	0.40	0.47	0.53	0.94	184	100% C
10	0.16	0.29	0.38	0.46	0.52	0.98	177	100% C
X <sub>mean</sub>	0.16	0.29	0.39	0.46	0.52	<b>0.94</b>	166	
s	0.01	0.01	0.01	0.01	0.01	0.02	14	
R <sub>u,5</sub>						<b>0.90</b>		

§ 5.1.4.2.3 SO<sub>2</sub> exposure

Table 3.2.4 Results after SO<sub>2</sub> exposure

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
No.	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.19	0.32	0.41	0.48	0.54	0.90	108	100% C
2	0.21	0.33	0.42	0.49	0.55	0.93	136	100% C
3	0.20	0.33	0.42	0.49	0.55	0.91	117	100% C
4	0.16	0.29	0.39	0.46	0.52	0.94	125	100% C
5	0.21	0.33	0.42	0.50	0.56	0.91	123	100% C
6	0.20	0.33	0.42	0.49	0.55	0.91	126	100% C
7	0.15	0.29	0.39	0.47	0.53	0.94	121	100% C
8	0.21	0.33	0.42	0.49	0.55	0.96	139	100% C
9	0.21	0.33	0.42	0.49	0.55	0.89	97	100% C
10	0.22	0.34	0.42	0.49	0.55	0.96	162	100% C
X <sub>mean</sub>	0.20	0.32	0.41	0.48	0.54	<b>0.92</b>	126	
s	0.02	0.02	0.01	0.01	0.01	0.02	18	
R <sub>u,5</sub>						<b>0.87</b>		

§ 5.1.4.2.4 Façade cleaning product

Table 3.2.5 Results after 21 days immersion in cleaning product at (45±2)°C (1 % “Dreft” solution)

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
Nr	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.16	0.27	0.35	0.41	0.46	0.86	196	100% C
2	0.15	0.27	0.35	0.41	0.46	0.88	225	100% C
3	0.15	0.26	0.34	0.40	0.46	0.87	205	100% C
4	0.16	0.27	0.35	0.41	0.46	0.85	161	100% C
5	0.16	0.27	0.35	0.41	0.46	0.83	157	100% C
6	0.17	0.28	0.37	0.43	0.48	0.88	222	100% C
7	0.17	0.28	0.36	0.43	0.48	0.84	155	100% C
8	0.16	0.27	0.35	0.41	0.47	0.93	229	100% C
9	0.13	0.25	0.33	0.40	0.45	0.91	191	100% C
10	0.18	0.28	0.36	0.42	0.47	0.87	178	100% C
X <sub>mean</sub>	0.16	0.27	0.35	0.41	0.46	<b>0.87</b>	297	
s	0.01	0.01	0.01	0.01	0.01	0.03	21	
R <sub>u,5</sub>						<b>0.81</b>		

### 3.3 Physical properties

#### § 5.1.4.6.1 Gas inclusions

With the sealant MF899-25 received from the client in tubes (see appendix A) a test specimen as in figure 1 was filled with the sealant. The test specimen was stored at a standard atmosphere of  $(23 \pm 2) \text{ }^\circ\text{C}$  and  $(50 \pm 5) \text{ } \%$  RH for 21 days. Every each 7 days the test specimen was observed visually.

*Results* : No generation of gas bubbles was observed.

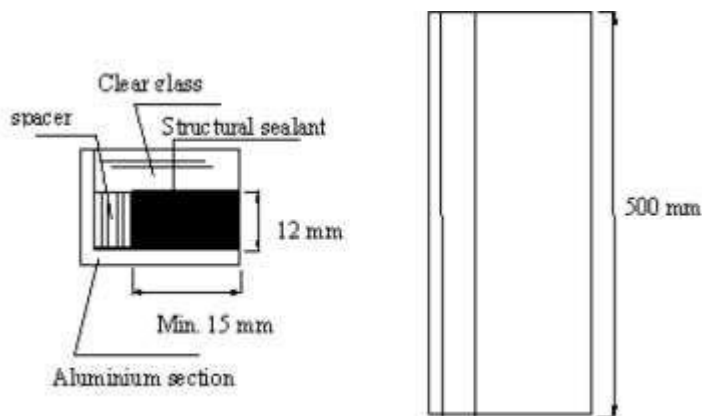


Figure 1: Tests specimen for gas inclusion test

#### § 5.1.4.6.2 Elastic recovery

Three test specimens, H-samples (1), were extended by 25% of the thickness ( $\approx 3 \text{ mm}$ ) and maintained for 24 hours (see figure 2). After one hour after removing the extension, the elastic recovery of the sealant was determined (ISO 7389 [3]). After 24 hours unloading the elongation in respect to the initial elongation was determined.

Table 3.3.1 Elastic recovery at  $23 \text{ }^\circ\text{C}$

Specimen	Extension	Elastic recovery	Elongation 24 after unloading
No.	%	%	%
1	24.9	96.3	2.7
2	24.8	96.0	2.7
3	25.0	95.9	2.7

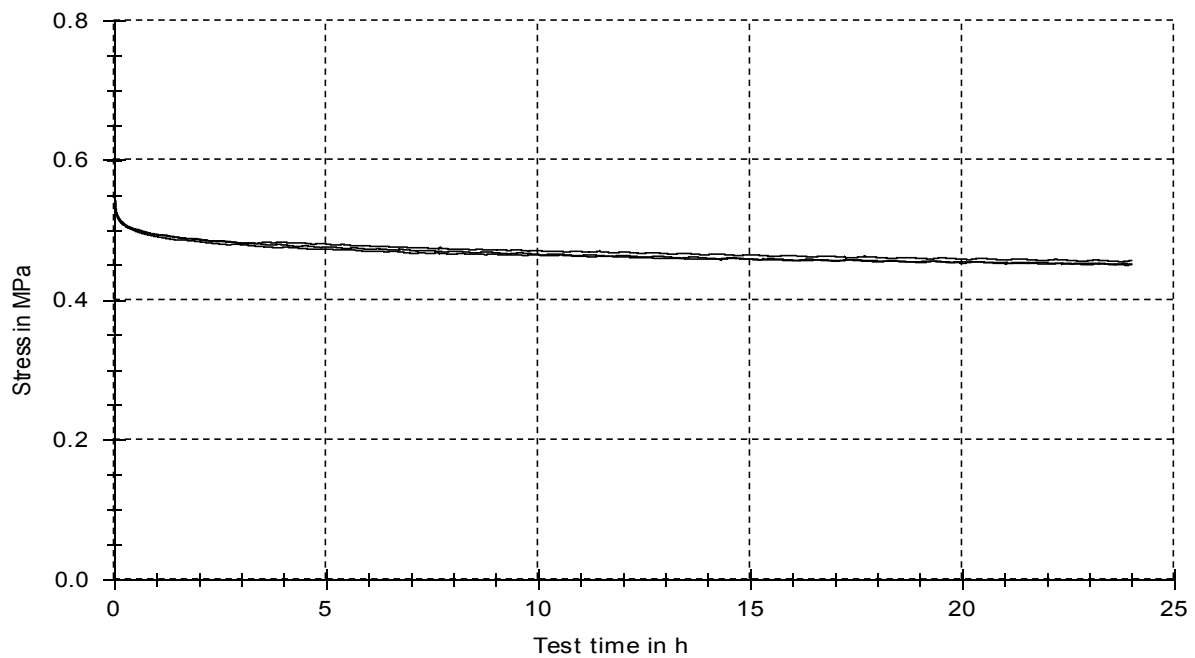


Figure 2: Elastic relaxation curve MF899-25 at 25 % extension.

#### § 5.1.4.6.3 Shrinkage

With the sealant MF8991-25 received from the client in tubes (see appendix A) three open cylindrical rings with an internal diameter of 30 mm and 10 mm height were filled with the sealant. The mass in air ( $m_3$ ) and in water ( $m_4$ ) of the filled rings was determined 60 minutes after filling.

Hereafter the test specimen were stored at a standard atmosphere of  $(23 \pm 2)^\circ\text{C}$  and  $(50 \pm 5)\%$  RH for 28 days and then stored in an oven at  $(70 \pm 2)^\circ\text{C}$  for 7 days. After 1 day conditioning at standard atmosphere the mass in air ( $m_5$ ) and in water ( $m_6$ ) was determined.

The change in volume was calculated according to ISO 10536 [4].

Table 3.3.2 Result change in mass and volume (shrinkage)

Specimen no.	change in mass [%]	change in volume [%]
1	2.2	3.5
2	2.1	3.4
3	1.3	2.6
Average	1.9	3.2

§ 5.1.4.6.3 Resistance to tearing

Pre-treatment: In the middle of both ends of the sealant of a sample type 1 (H- sample) a horizontal incision is made of 5 mm depth. This is done with a razor blade.

Table 3.3.3 Results tear test at 23 °C

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure mode
No.	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.20	0.33	0.45	0.54	0.62	0.87	70	100% C
2	0.20	0.35	0.46	0.54	0.61	0.85	60	100% C
3	0.17	0.33	0.45	0.54	0.62	0.86	63	100% C
4	0.20	0.35	0.46	0.55	0.62	0.85	67	100% C
5	0.19	0.35	0.46	0.55	0.63	0.87	72	100% C
$X_{mean}$	0.19	0.34	0.45	0.54	0.62	<b>0.86</b>	67	
s	0.01	0.01	0.01	0.00	0.01	0.01	5	
$R_{u,5}$						<b>0.84</b>		

§ 5.1.4.6.5 Mechanical fatigue

Pre-treatment: The 10 specimens (1) were subjected to cyclic mechanical loads before determination of the tensile strength.

The cyclic load was applied as follows:

1. 100x from 0.1  $\bar{\sigma}_{des}$  to 1x  $\bar{\sigma}_{des}$
2. 250x from 0.1  $\bar{\sigma}_{des}$  to 0.8x  $\bar{\sigma}_{des}$
3. 5000x from 0.1  $\bar{\sigma}_{des}$  to 0.6x  $\bar{\sigma}_{des}$

The  $\bar{\sigma}_{des}$  is determined according to ETAG 002 annex A2.1

$\bar{\sigma}_{des}$  = tension design stress  $\bar{\sigma}_{des} = R_{u,5} / \gamma_{tot}$ , with  $\gamma_{tot}$  total safety factor = 6

$\bar{\sigma}_{des} = 0.98 / 6 = 0.163$  MPa

Table 3.3.4 Results tensile strength after mechanical fatigue test at 23°C

	-5%	-10%	-15%	-20%	-25%	-max	-max	failure
No.	MPa	MPa	MPa	MPa	MPa	MPa	%	
1	0.20	0.34	0.45	0.53	0.60	1.03	131	100% C
2	0.21	0.34	0.44	0.52	0.59	1.05	134	100% C
3	0.19	0.32	0.42	0.50	0.56	1.02	140	100% C
4	0.19	0.33	0.43	0.52	0.58	0.96	121	100% C
5	0.18	0.32	0.43	0.51	0.58	1.05	149	100% C
6	0.21	0.34	0.44	0.52	0.58	1.09	180	100% C
7	0.17	0.33	0.43	0.52	0.59	1.06	126	100% C
8	0.20	0.34	0.43	0.52	0.58	1.06	123	100% C
9	0.18	0.30	0.39	0.46	0.52	1.02	138	100% C
10	0.18	0.32	0.42	0.50	0.57	1.02	154	100% C
$X_{mean}$	0.19	0.33	0.43	0.51	0.58	<b>1.04</b>	140	
s	0.01	0.01	0.02	0.02	0.02	0.04	18	
$R_{u,5}$						<b>0.96</b>		

#### § 5.1.4.6.6 UV resistance of the sealant

Ten test pieces as per ISO 527-3 type 5, were punched out (2.2 ±0.2) mm thick cured sealant film as received from the client (see appendix A). Five test pieces were tested as such according to ISO 527-3 at a test speed of 5 mm/min (ISO 527-3/5/5) with 0.2 N pre-stress. The other 5 test pieces were submitted to UV aging for 500 hours in a Weather O meter Ci 4000 according to EN ISO 4892-2 [14] exposure cycle no. 1 (Xenon lamp).

The tensile strength (maximum stress) and corresponding elongation are given in table 3.2.6. The (secant) modulus between 0.25 and 1% (near origin) and between 5 and 25 % elongation are given in table 3.2.7 before and after UV exposure.

The stress-elongation curves are represented in appendix C.

Table 3.3.5 Result tensile strength sealant before (initial) and after U.V. exposure at 23 °C

Specimen no.	Initial		After UV exposure	
	Tensile strength [MPa]	Elongation [%]	Tensile strength [MPa]	Elongation [%]
1	1.80	364	1.61	288
2	1.76	331	1.61	277
3	1.75	327	1.65	276
4	1.72	300	1.59	272
5	1.67	301	1.61	296
<b>X<sub>mean</sub></b>	<b>1.74</b>	<b>325</b>	<b>1.61</b>	<b>282</b>
S	0.05	26	0.02	10

#### § 5.1.4.6.7 Elastic modulus sealant

Table 3.3.6 Result elastic (secant) modulus sealant before and after UV exposure

Specimen no.	Elastic modulus as such [MPa]		Elastic modulus after UV exposure [MPa]	
	0.25-1 %	5-25 %	0.25-1 %	5-25 %
1	4.21	1.78	5.00	1.69
2	4.93	1.85	5.37	1.76
3	5.31	1.82	5.39	1.79
4	4.55	1.83	5.59	1.73
5	4.78	1.88	4.72	1.69
<b>X<sub>mean</sub></b>	<b>4.76</b>	<b>1.83</b>	<b>5.22</b>	<b>1.73</b>
s	0.41	0.04	0.35	0.04

#### § 5.1.4.6.8 Creep under long term shear

Before testing 3 specimens were stored for a minimum of 28 days in an air conditioned room at (23±2) °C and (50±5) % relative humidity and tested at this condition.

The creep installation subjects simultaneously the test specimens for 91 days to:

- Tensile loading of  $M_1 = 2 * L * h * P_t$   
Where  
L: length the of the seal (mm)  
h: wide of seal (mm)  
 $P_t = 0,3 * \delta_{des}$  and  $\delta_{des} = R_{u;5} / 6$

$R_{u;5}$  is the characteristic stress value at 23°C giving 75% confidence that 95% of the test results will be higher than this value.

The  $R_{u;5}$  in tension has been determined as 0.98 MPa, see test table 3.1.2.

- Permanent shear loading of :  $M_2 = 2 * L * h * \tau_{\infty}$   
 With  $\tau_{\infty}$ , stress given by the manufacturer, taking into account a creep factor ( $\gamma_c$ ) of 10 as given by the manufacturer  
 No value for  $\tau_{\infty}$  was given by manufacturer.  
 For the test a value for  $\tau_{\infty}$  of  $\tau_{des} / \gamma_{tot} = (R_{u,5} / 6) / 10$  is used.

The  $R_{u,5}$  in shear has been determined as 0.80 MPa, see test table 3.1.5.

For the test the sealant length and average wide of the test specimens was used to calculate the tensile and shear loading. The load was applied with dead weights.

Table 3.3.7: Load arrangement per specimen

Specimen	average wide (h) x length left seal [mm]	average wide (h) x length right seal [mm]	$\bar{\sigma}_{des}$ MPa	Tensile load $M_1$ N (kg)	Shear load $M_2$ N (kg)
1	7.25x200	7.40x200	0.133	117 N (11.9 kg)	39.1 N (4.0 kg)
2	6.75x200	7.29x200	0.133	112 N (11.4 kg)	37.4 N (3.8 kg)
3	6.64x200	7.28x200	0.133	111 N (11.4 kg)	37.1 N (3.8 kg)

The creep of the specimens during the test duration is shown in figure 3.

The deformation after 91 days loading and after 24 h. unloading is given in table 3.2.8.

Table 3.2.8: Deformation creep test [mm]

Specimen	After 91 days loading	after 24 h unloading
1	0.079	0.048
2	0.079	0.042
3	0.068	0.039
average	0.075	0.043

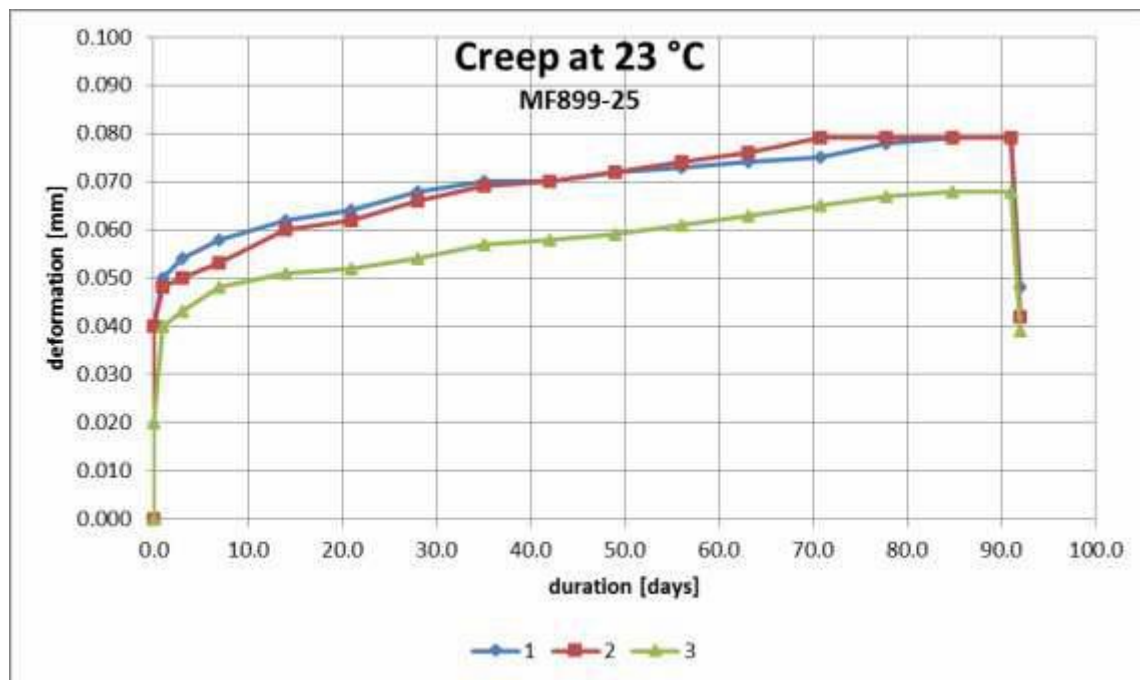


Figure 3: Creep sealant MF899-25 at 23°C



### 3.4 Identification of product

#### § 5.2.1.1 Specific mass

The specific mass was determined of three specimens from cured sealant film (2 mm thick) according to ISO 1183 method A immersion method (water) at  $(23\pm 1)$  °C.

Table 3.4.1 Specific mass cured sealant at 23°C

Test piece	Specific mass g/cm <sup>3</sup>
1	1.504
2	1.505
3	1.505
Average	1.505
S	0.001

#### § 5.2.1.2 Hardness

The Shore A hardness was determined according to ISO 868. From the cured 2 mm thick film samples three test pieces were made by stacking 3 layers of film on top of each other. Of each test piece 5 readings were taken after 3 seconds.

Table 3.4.2 Shore A Hardness at 23°C

Test piece	Shore A hardness
1	51
2	48
3	50
Average	50
S	2

### § 5.2.1.3 Thermogravimetric analysis

The weight/mass loss of the sealant as function of the temperature was determined with a TGA Q500 TA Instruments apparatus at the following conditions based on ISO 11358-1:2014 [11]:

Temperature: 30 – 900 °C, 5 minutes isotherm at 900 °C

Heating: 10 °C/min under nitrogen atmosphere

Mass: 56.2840 mg cut from 2 mm thick cured film sample from manufacturer

The relative weight loss with temperature (green curve) and first derivative (blue curve) is given in figure 2. There are two main decomposition steps. In the first step up to approx. 620 °C there is a weight loss of 43.6 %. The second double decomposition step consists of a small and a larger step. Up to approx. 685 °C there is a 2.3 % weight loss and up to approx. 685 °C another 19.5%. At 900 °C a residue of 34.6 % is left.

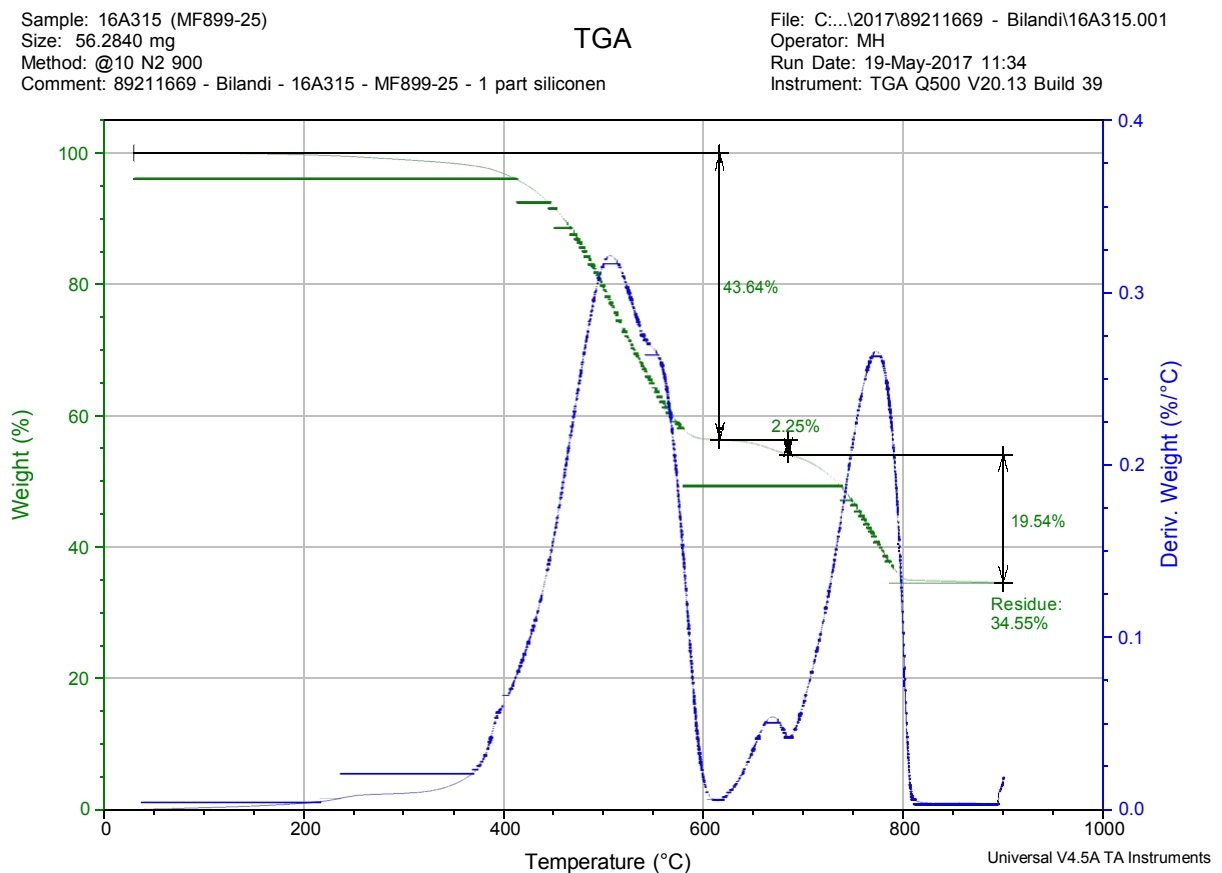


Figure 2: TGA curve, weight loss of MF889-25 Silicone structural sealant up to 900 °C.

#### § 5.2.1.4 Colour

Instead of defining the colour of the sealant according to the colour scale of ISO 4660 [12] the colour coordinates were determined according to LAB colour space. Of a piece of 2 mm thick cured film both sides (shiny and matt side) were measured with a Minolta CM 2600 D spectrophotometer D65 illumination at an angle of 10° according to ISO 7724 [13].

Table 3.4.3 Results LAB measurements

Test piece	L*	a*	b*
shiny	25.85	-0.15	-1.33
matt	26.85	-0.20	-1.40

## 4 Evaluation

The requirements for safety in use (ER4) of structural glazing sealants are set out in table 8.3 of ETAG 002 part 1:2012 [1]. The test results and requirements and the sealant identification properties are summarised in table 4.1.

Table 4.1 Summary of test results and requirements

Verification method	Requirements	Results
<b>3.1 Initial mechanical strength</b>		
Tensile strength at -20, 23 and 80°C	$K_{12.5; -20/23/80^{\circ}\text{C}}$  $R_{u,5; -20/23/80^{\circ}\text{C}}$  $\Delta X_{\text{mean}} = X_{\text{mean}, -20^{\circ}\text{C}} / X_{\text{mean}, 23^{\circ}\text{C}} \geq 0.75$ $\Delta X_{\text{mean}} = X_{\text{mean}, 80^{\circ}\text{C}} / X_{\text{mean}, 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$K_{12.5; -20^{\circ}\text{C}} = 3.37 \text{ MPa}$ $K_{12.5; 23^{\circ}\text{C}} = 3.06 \text{ MPa}$ $K_{12.5; 80^{\circ}\text{C}} = 3.26 \text{ MPa}$ $R_{u,5; -20^{\circ}\text{C}} = 1.26 \text{ MPa}$ $R_{u,5; 23^{\circ}\text{C}} = 0.98 \text{ MPa}$ $R_{u,5; 80^{\circ}\text{C}} = 0.86 \text{ MPa}$ $\Delta X_{\text{mean}} = 1.31 / 1.07 = 1.22$ $\Delta X_{\text{mean}} = 0.96 / 1.07 = 0.90$ All test specimens more than 90% cohesive rupture
Shear strength at -20, 23 and 80°C	$R_{u,5; -20/23/80^{\circ}\text{C}}$  $\Delta X_{\text{mean}} = X_{\text{mean}, -20^{\circ}\text{C}} / X_{\text{mean}, 23^{\circ}\text{C}} \geq 0.75$ $\Delta X_{\text{mean}} = X_{\text{mean}, 80^{\circ}\text{C}} / X_{\text{mean}, 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$R_{u,5; -20^{\circ}\text{C}} = 0.97 \text{ MPa}$ $R_{u,5; 23^{\circ}\text{C}} = 0.80 \text{ MPa}$ $R_{u,5; 80^{\circ}\text{C}} = 0.61 \text{ MPa}$ $\Delta X_{\text{mean}} = 1.09 / 0.88 = 1.24$ $\Delta X_{\text{mean}} = 0.78 / 0.88 = 0.87$ All test specimens more than 90% cohesive rupture
<b>3.2 Residual strength after artificial ageing</b>		
Immersion in hot water (1000 hours) and UV	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean}, \text{initial } 23^{\circ}\text{C}} \geq 0.75$ $0.5 \leq K_{12.5} / K_{12.5, \text{initial } 23^{\circ}\text{C}} \leq 1.10$ Rupture $\geq 90\%$ cohesive	$\Delta X_{\text{mean}} = 0.86 / 1.02 = 0.84$ $2.73 / 3.31 = 0.82$ All test specimens more than 90% cohesive rupture
Humidity and NaCl (salt spray test)	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean}, \text{initial } 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$\Delta X_{\text{mean}} = 0.924 / 1.07 = 0.88$ All test specimens more than 90% cohesive rupture
Humidity and SO <sub>2</sub>	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean}, \text{initial } 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$\Delta X_{\text{mean}} = 0.92 / 1.07 = 0.86$ All test specimens more than 90% cohesive rupture
Façade cleaning products (1% Dreft solution)	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean}, \text{initial } 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$\Delta X_{\text{mean}} = 0.87 / 1.07 = 0.81$ All test specimens more than 90% cohesive rupture

Verification method	Requirements	Results
<b>3.3 Physical properties</b>		
Gas inclusions	No visible gas bubbles	No gas bubbles observed
Elastic recovery (at initial elongation of 25%)	Elongation 24 hours after unloading shall be < 5% of the initial elongation	Elongation after 24 hours unloading 2.7%
Shrinkage	Shrinkage shall be less than 10%	Shrinkage 2.6 – 3.5%
Resistance to tearing	Category 1 $\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean,initial } 23^{\circ}\text{C}} \geq 0.75$ Category 2 $\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean,initial } 23^{\circ}\text{C}} \geq 0.50$	$\Delta X_{\text{mean}} = 0.86 / 1.07 = 0.80$
Mechanical fatigue	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean,initial } 23^{\circ}\text{C}} \geq 0.75$ Rupture $\geq 90\%$ cohesive	$\Delta X_{\text{mean}} = 1.04 / 1.07 = 0.97$ All test specimens more than 90% cohesive rupture
U.V. resistance of the sealant	$\Delta X_{\text{mean}} = X_{\text{mean}} / X_{\text{mean,initial } 23^{\circ}\text{C}} \geq 0.75$ for elongation and stress	Elongation: $\Delta X_{\text{mean}} = 282 / 325 = 0.87$ Stress $\Delta X_{\text{mean}} = 1.61 / 1.74 = 0.93$
Elastic modulus of the sealant	Declared value from test	Initial $E_{\text{secant } 0.25-1\%} = 4.76 \text{ MPa}$ $E_{\text{secant } 5-25\%} = 1.83 \text{ MPa}$ After U.V. ageing $E_{\text{secant } 0.25-1\%} = 5.22 \text{ MPa}$ $E_{\text{secant } 5-25\%} = 1.73 \text{ MPa}$
Creep under long –term shear and cyclic tensile loading	Horizontal displacement after 24 hr unloading < 0.1 mm Movement stabilised after 91 days	Displacement: 0.039 - 0.048 mm Yes
<b>3.4 Identification of product</b>		
Specific mass	$V_{\text{mean}}$ and S	$V_{\text{mean}} = 1.505 \text{ g/cm}^3$ $S = 0.001 \text{ g/cm}^3$
Hardness	$V_{\text{mean}}$ and S	$V_{\text{mean}} = 50 \text{ Shore A}$ $S = 2 \text{ Shore A}$
Thermogravimetric analysis	Thermogravimetric curve	See at page 18
Colour	Colour parameters LAB colour space	shiny / matt $L : 25.85 / 26.85$ $a : -0.15 / -0.20$ $b : -1.33 / -1.40$

## 5 References

- 1 ETAG No. 002-1  
Guideline for European Technical Approval for Structural Sealant Glazing Kits (SSKK),  
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European Organisation for Technical Approvals (EOTA), May 2012.
- 2 EN ISO 11431  
Building construction – Jointing products – Determination of adhesive/cohesion properties of  
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- 3 EN ISO 7389  
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- 4 EN ISO 10563  
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- 8 EN ISO 527-3  
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- 9 ISO 1183-1:2004  
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- 10 ISO 868:2003  
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- 11 ISO 11358-1:2014  
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- 12 ISO 4660:2011  
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- 13 ISO 7724-2:1984  
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International Organization for Standardization, 1984
- 14 EN ISO 4892-2:2006  
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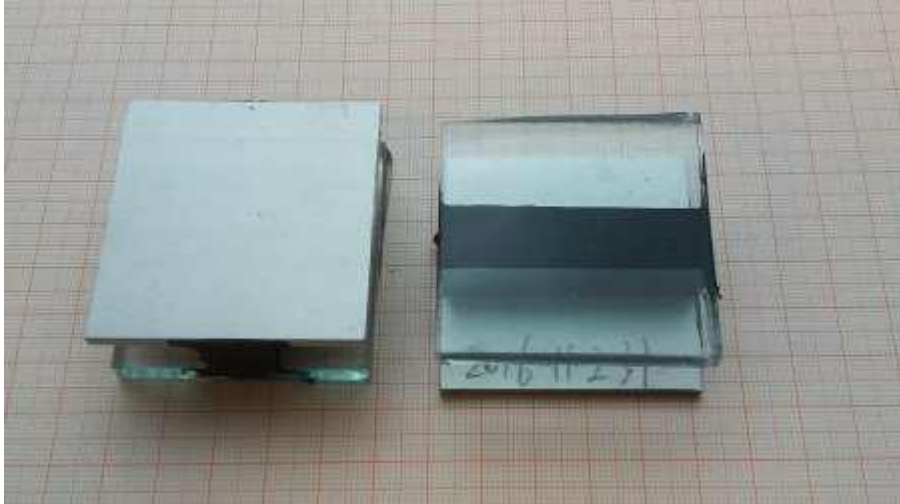
## 6 Signatures

<b>Author</b> Mr. M.A.A.M. Schets, B.Sc.	Signature 
Specialist	
<b>Peer review</b> Mr. R. Brandhorst	Signature i.a. S. El Bardai
Specialist	
<b>Approved by</b> Mrs. C.C.M. van Houten	Signature 
Manager operations	



## Appendix A Pictures of the tested object(s)

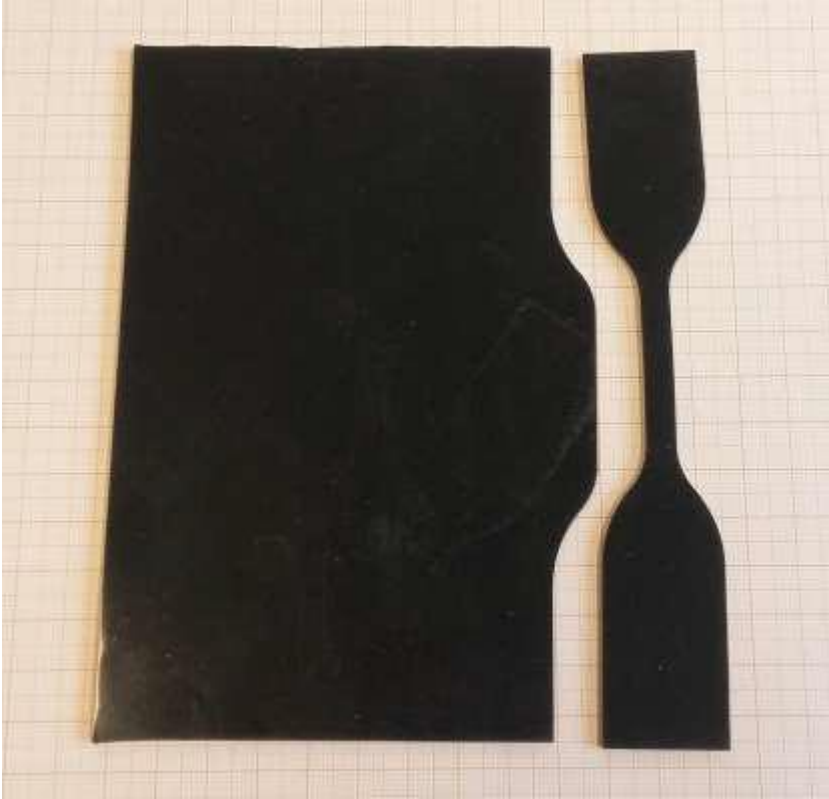
Sample type 1 (H-sample) 6 mm float glass, 3 mm anodised aluminium, seal joint 12x12x50 mm



Sample type 1 (H-sample) typical failure pattern

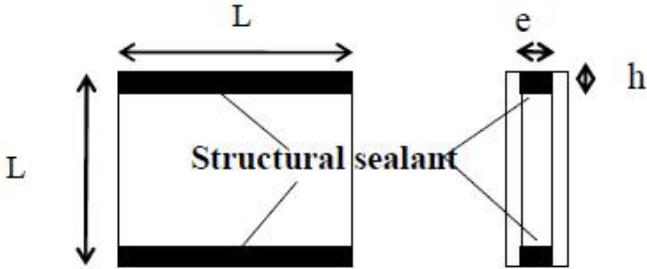


Sample type 2 and ISO 527 type 5 specimens punched from 2 mm sealant film



Sample type 3, creep behaviour





$e = 6.2-6.6 \text{ mm}$

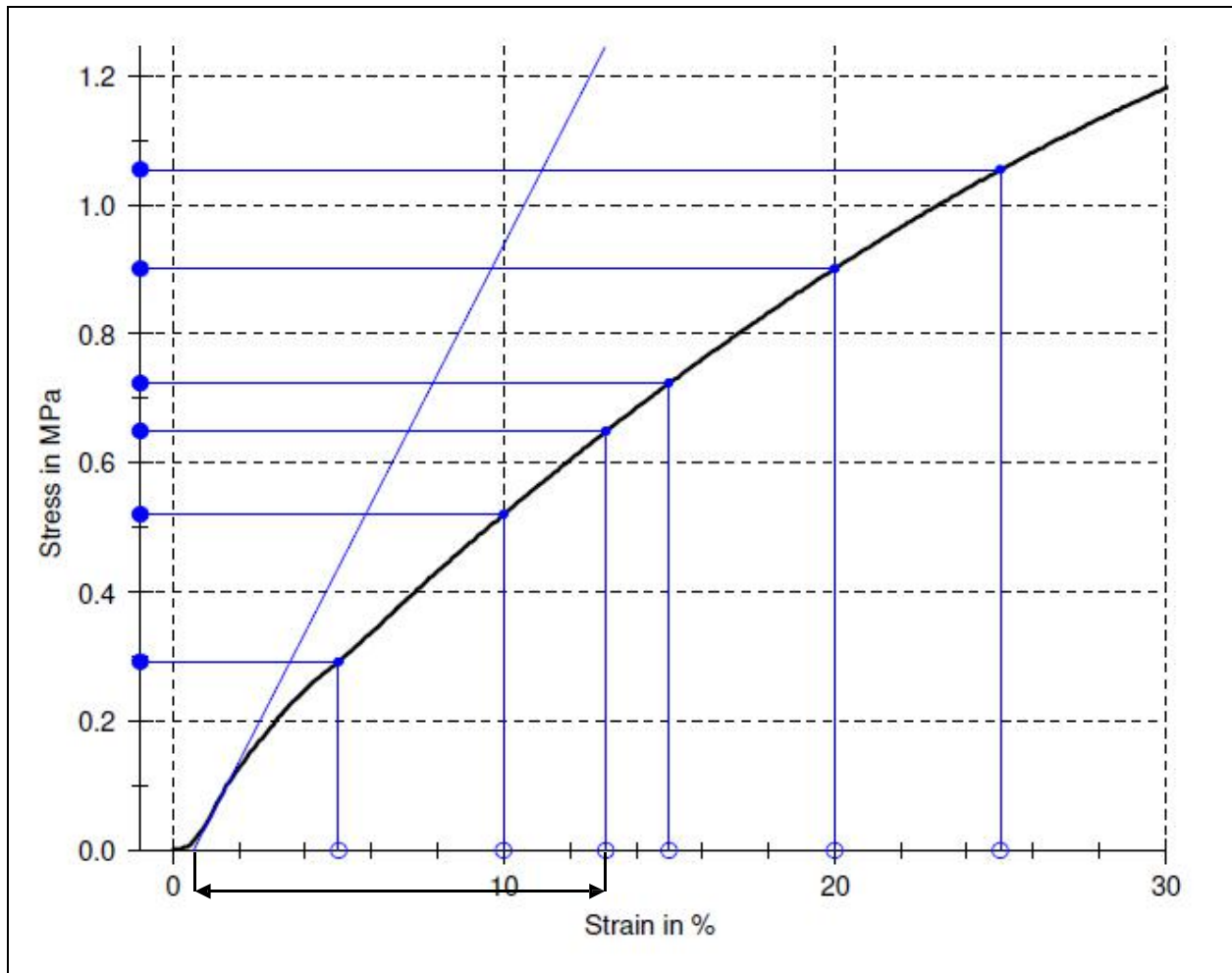


$L = 200 \text{ mm}$

Tubes with sealant Silande MF899-25

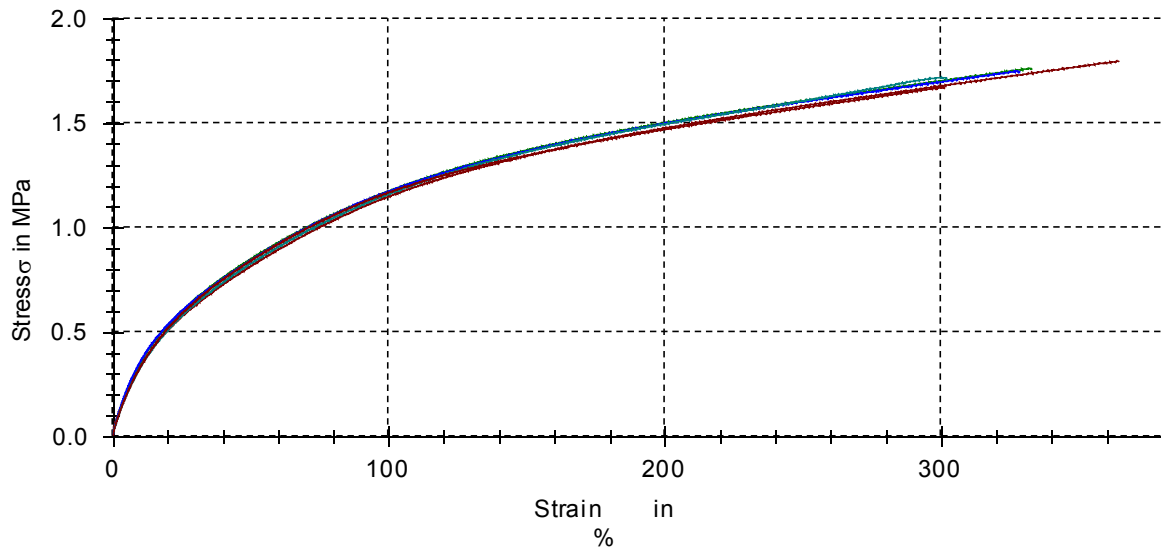


## Appendix B Determination of $K_{12,5}$

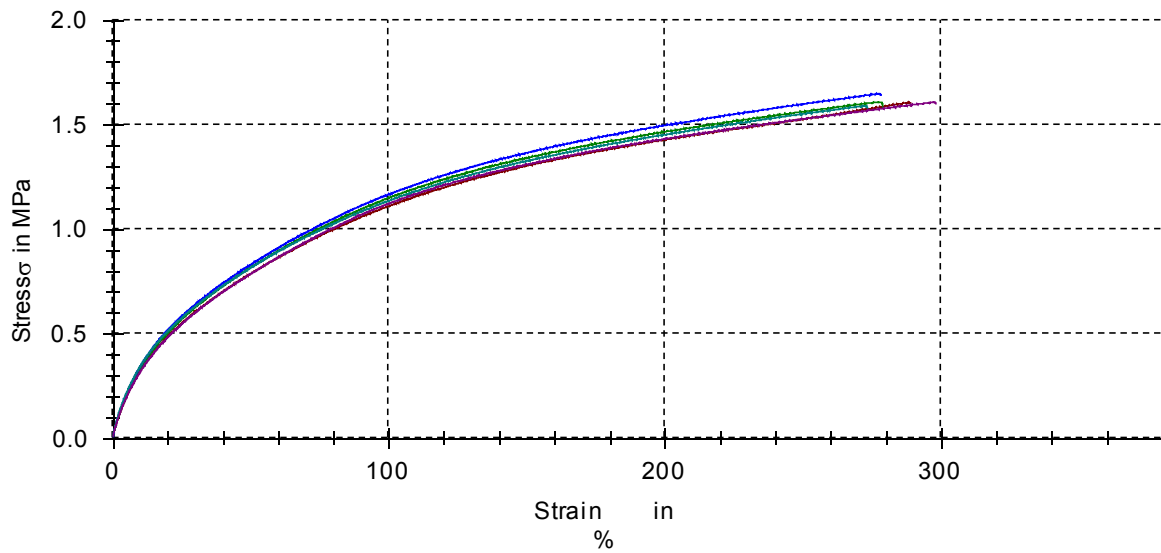


The secant stiffness at 12.5 % strain ( $K_{12,5}$ ) was calculated from a new zero point to compensate for irregularities and pre load at the start. This zero point was determined for each curve based on the slope of the curve at the start.

## Appendix C Tensile strength curves sealant before and after UV exposure



Tensile strength sealant as received



Tensile strength sealant after UV exposure.

- End of report -