Report
Determination of possible surface contamination caused by siloxanes from NOFIRNO sealant.

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1 Introduction

By order of BEELE Engineering BV (Beele), TÜV Rheinland Nederland BV (TRN) performed an investigation into the possibility of a spontaneous siloxane contamination on a metal surface, caused by the application of a silicone based sealant NOFIRNO on adjacent areas. The investigation was performed in accordance with the conditions described in TRN quotation 46066, dated July 31st, 2014.

2 Experimental

2.1 Samples received

Sample material was made available to TRN by Beele on July 29th, 2014 and registered under a TRN sample number. In table 1 the TRN sample number, description and photographs are given.

Table 1: Sample materials, descriptions and photographs.

TRN sample number	Sample description	Photograph
14.0089	NOFIRNO Silicone sealant NFN J 07 0714 AM 3	

2.2 Test setup

2.2.1 Preparation

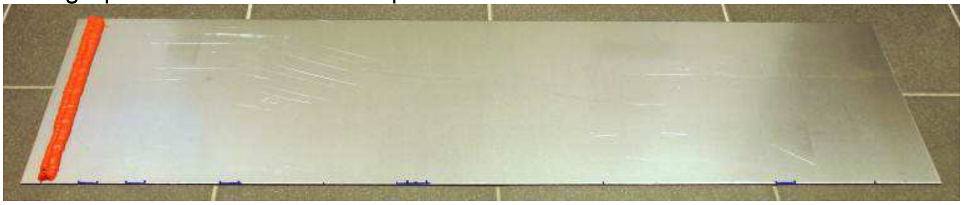
The surface of a metal (steel) sheet (250 mm x 1000 mm x 2 mm) was cleaned thoroughly with water and detergent, dried and subsequently cleaned with pure hexane. Cotton balls were extracted with hexane to remove possible contaminants in the cotton.

2.2.2 Sampling of blanks

After 24 hours, four locations on the cleaned metal surface were sampled using cotton wetted with hexane (quality: Pro Analysis). 5 ml of internal standard solution of toluene-d8 in hexane was added to the cotton and placed in an ultrasonic bath to extract possible contaminants. The extracts were analyzed by means of gas chromatography mass spectrometry.

2.2.3 Testing of the sealant.

35 grams of sealant was applied at a distance of 20 mm from the edge over the full length parallel to the short (250 mm) side of the metal sheet. The amount of applied sealant was determined by weighing the cartridge before and after application.
After 1 and 24 hours areas of 20 cm^2 on the metal sheet were sampled (in duplicate) by means of cotton balls wetted with hexane. The metal surface was sampled at a 50mm, 100mm, 200 mm, 400 mm and 800 mm distance from the applied sealant. 5 ml of internal standard solution of toluene-D8 in hexane was added to the cotton balls, which were placed in an ultrasonic bath to extract possible absorbed contaminants. The extracts were analyzed by means of gas chromatography mass spectrometry.
Photograph 1 shows the test setup.



Photograph 1: Test setup.

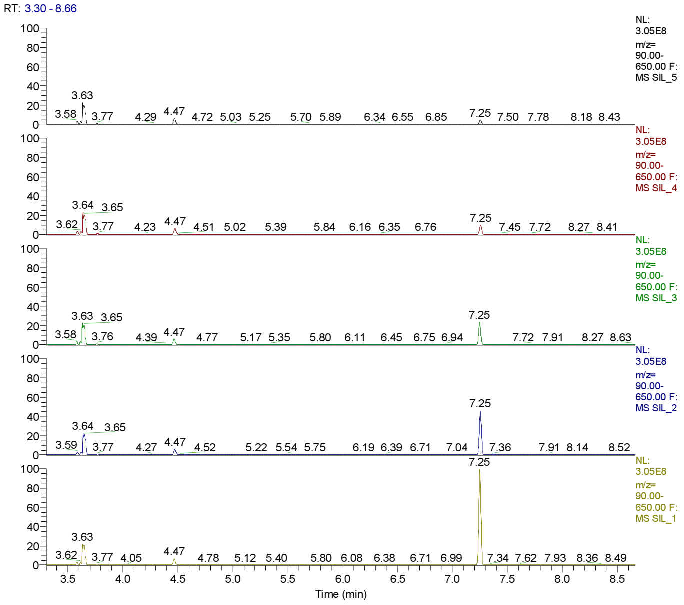
2.3 Gas Chromatography Mass Spectrometry (GCMS)

Using gas chromatography, the different volatile organic compounds are separated from each other on the basis of differences in boiling point and affinity with the column material. Mass spectrometry is used to detect and identify the separated components. This analysis results in a chromatogram in which the various volatile organic compounds are visible as peaks. The surface areas under the peaks are a measure for the amounts of the components identified in a sample. The contaminants were quantified as octamethylcyclotetrasiloxane (D4) equivalents by means of an internal standard calibration.

3 Results

3.1.1 Calibration

The GCMS chromatograms of the standard solutions are given in figure 1. The internal standard calibration curve is given in figure 2. The detected components are listed in table 2.



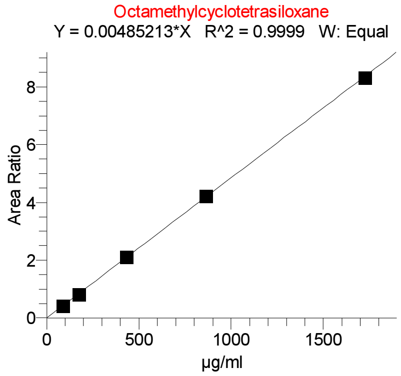


Figure 1: GCMS chromatograms of the standard solutions. Top to bottom: 86 µg/ml, 173 µg/ml, 431 µg/ml, 862 µg/ml and 1730 µg/ml.

Figure 2: Internal standard calibration curve of octamethylcyclotetrasiloxane.

Table 2: Detected components.

RT (min)	Compound
3.63	Hexane
4.47	Toluene-d8
7.25	Octamethylcyclotetrasiloxane (D4)

3.1.2 Quantification

The GCMS chromatograms of the samples taken after 1 hour are given in figure 3. The detected components are listed in table 3.

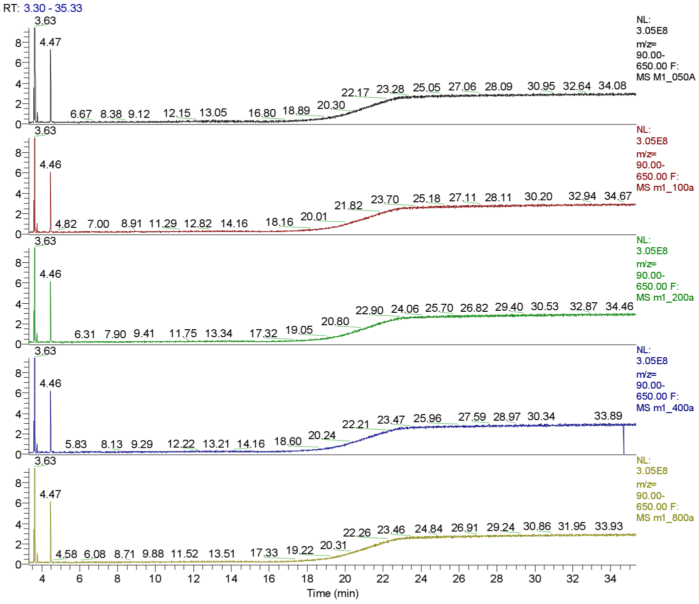


Figure 3: GCMS chromatograms of the 1 hour samples. Top to bottom: 1h-50mm, 1h-100mm, 1h-200mm, 1h-400mm, 1h-800mm.

Table 3: Detected components.

RT (min)	Compound
3.63	Hexane
4.47	Toluene-d8

Apart from hexane and the internal standard toluene-d8 no volatile organic compounds were detected. The calculated limit of detection for D4 of the surface of the metal sheet is 300 µg/m^2.

The GCMS chromatograms of the samples taken after 24 hours are given in figure 4. The detected components are listed in table 4.

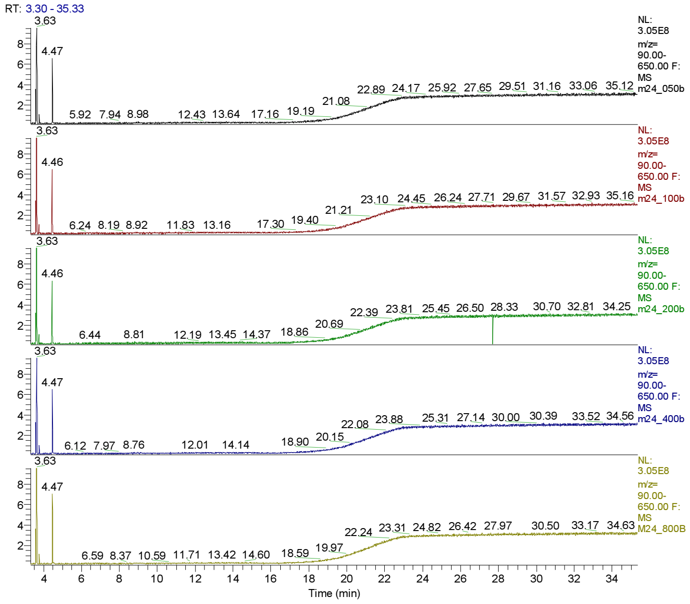


Figure 4: GCMS chromatograms of the 24 hour samples. Top to bottom: 24h-50mm, 24h-100mm, 24h-200mm, 24h-400mm, 24h-800mm.

Table 4: Detected components.

RT (min)	Compound
3.63	Hexane
4.47	Toluene-d8

Apart from hexane and the internal standard toluene-d8 no volatile organic compounds were detected. The calculated limit of detection for D4 of the surface of the metal sheet is 300 µg/m^2.

4 Conclusions

No volatile contaminants were detected on the surface of the metal sheet after 1 and 24 hours after the application of sample 14.0089. The detection limit of octamethylcyclotetrasiloxane (D4) is 300 µg/m^2.

5 Signatures

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Peer review	Signature
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(End of this report).