

Test Report
Mulcol International
Product Emissions Test
(AgBB/DIBt Test Protocol)

Mulcol® Multisealant A

March 2012

Client: Mulcol International
Arnesteinweg 18
4338 PD Middelburg
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Testing Laboratory: Eurofins Product Testing A/S
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Introduction

On 12 January 2012 Eurofins Product Testing A/S received a sample of a sealant named

Mulcol® Multisealant A

for emissions testing in accordance with the AgBB/DIBt method. The sample was clearly labelled, properly packaged and not damaged. Testing was carried out in the laboratories of Eurofins Product Testing A/S. Before starting the testing procedure on 6 February 2012 the sample had been stored unopened at room temperature.

1 Description of the Applied Testing Method

The applied testing method complies with the test method as defined by AgBB (version May 2010), in its interpretation by DIBt in its version by October 2010. Any necessary deviations are pointed out in this report. The test method is based on the published methods: ISO 16000-3, ISO 16000-6, 16000-9, 16000-11. The internal method numbers are: 9810; 9811, 9812, 2808, 8400.

1.1 Test Specimen

The sample was applied onto a glass plate and drawn off over a model giving a 3 mm thick and uniform layer with a broadness of 10 mm. The test specimen was transferred into a test chamber immediately (internal method no.: 9810).

1.2 Test Chamber

The test chamber was made of stainless steel and had a volume of 119 litres. The air clean-up was realised in multiple steps. Before loading the chamber a blank check of the empty chamber was performed. The operation parameters were 23 °C, 50 % relative air humidity (in the supply air) with an air exchange rate of ½ per hour. The loading of the test chamber was 0.007 m² test specimen per m³ air volume (internal method 9811).

1.3 Sampling, Desorption, Analyses

1.3.1 Testing for Carcinogens after 3 and 28 Days

The presence of carcinogens (EU Categories C1 and C2, as read from the latest publication on the homepage of German BGIA Institute) was tested by drawing air samples from the chamber outlet through Tenax TA tubes (main tube and backup tube). Analyses were done by thermal desorption (Perkin Elmer) and gas chromatography / mass spectroscopy (30 m column, 0.25 mm ID, 0.25 µm HP-1 film, Agilent) (internal methods no.: 9812 / 2808). The absence of a listed carcinogen was stated if the specific combination of fragment ions was lacking at the specific retention time in the chromatogram. Otherwise it was checked whether the required detection limit (1 µg/m³) was exceeded. In this case the identity was finally checked by comparing full scan sample mass spectra with full scan standard mass spectra.

This test covered only substances that can be adsorbed on Tenax TA and that can be thermally desorbed. If other emissions occurred, then these could not be monitored (or with limited reliability only).

1.3.2 VOC Emissions Testing after 3 and 28 Days

The emissions of organic compounds were tested by drawing air samples from the chamber outlet through Tenax TA tubes (main tube and backup tube). Analyses were done by thermal desorption (Perkin Elmer) and gas chromatography / mass spectroscopy (30 m column, 0.25 mm ID, 0.25 µm HP-1 film, Agilent) (internal methods no.: 9812 / 2808).

All single substances that are listed with a NIK value in the latest AgBB publication were identified if the toluene equivalent in the Total Ion Chromatogram (TIC) exceeded 5 µg/m³. Quantification was done with the respective response factor and the TIC signal, or in case of overlapping peaks by calculating with fragment ions. All other single substances, as well as all non-identified substances, were quantified as toluene equivalent if giving more than 5 µg/m³.

The results are only valid for the tested sample(s).

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The results of the individual substances were calculated in three groups depending on their appearance in a gas chromatogram when analysing with a non-polar column (HP-1):

- Volatile organic compounds VOC: All substances appearing between these limits.
- Semi-volatile organic compounds SVOC: All substances appearing after n-hexadecane (n-C₁₆).
- Very volatile organic compounds VVOC: All substances appearing before n-hexane (n-C₆).

Calculation of the TVOC (Total Volatile Organic Compounds) was done as defined by AgBB/DIBt test method by addition of the results of all individual substances in the retention time interval C₆-C₁₆. Furthermore the TVOC was calculated as the toluene equivalent of all substances between C₆ and C₁₆, as defined in ISO 16000-6.

Calculation of the TSVOC (Total Semi-Volatile Organic Compounds) was done by addition of the results of all substances between C₁₆ and C₂₂ as toluene equivalent, as defined in ISO 16000-6.

Calculation of the TVVOC (Total Very Volatile Organic Compounds) was done by addition of the results of all substances appearing before C₆ as toluene equivalent, as defined in ISO 16000-6.

This test covered only substances that can be adsorbed on Tenax TA and that can be thermally desorbed. If other emissions occurred then these could not be monitored (or with limited reliability only).

1.3.3 Calculation of R Values after 3 and 28 Days with the German NIK List

The concentrations of all substances in the interval between n-C₆ and n-C₁₆ with more than 5 µg/m³ were divided by their respective NIK value (if given). The sum of the quotients gives the R value:

$$R = \sum_i^n \left(\frac{c_i}{\text{NIK}_i} + \dots + \frac{c_n}{\text{NIK}_n} \right)$$

In parallel, all results were summed up for the substances without published NIK value, but in the interval between n-C₆ and n-C₁₆, when giving more than 5 µg/m³.

1.3.4 Testing of Aldehydes after 3 and 28 Days

The presence of aldehydes was tested by drawing air samples from the chamber outlet through DNPH-coated silicagel tubes. Analysis was done by solvent desorption, HPLC and UV-/diode array detection (ISO 16000-3, internal methods no.: 9812 / 8400).

The absence of formaldehyde was stated if the specific wavelength UV detector response was lacking at the specific retention time in the chromatogram. Otherwise it was checked whether the detection limit was exceeded. In this case the identity was finally checked by comparing full scan sample UV spectra with full scan standard UV spectra.

1.3.5 Testing of Ammonia after 3 and 28 days

The presence of ammonia was tested by drawing air samples from the chamber outlet through silicagel tubes coated with sulphuric acid. Analysis was done by solvent desorption and UV/VIS spectroscopy (internal methods: 9812 / 4430).

1.3.6 Deviations from the Test Method

No calibrations standards have been available for the measured individual substances with NIK value but with identification categories 2 or 3. Consequently these substances were not quantified with their relative response factor, but instead as toluene equivalent.

No other deviations.

1.3.7 Quality assurance

Before loading the chamber a blank check of the empty chamber was performed and compliance with background concentrations in accordance with ISO 16000-9 was determined. Sampling at the chamber outlet and subsequent analysis was realised as double determination. For monitoring any breakthrough or overloading of the tubes, two Tenax TA tubes were used in series (or two adsorption areas for silicagel tubes).

In each sequence stability of GC system was checked by a general function test of device and column, and by use of control charts for monitoring mean values and standard deviations for individual VOC. Reproducibility of the method was monitored for two selected VOC per sequence.

1.3.8 Accreditation

The testing methods described above have been accredited (EN ISO/IEC 17025:2005) by DANAK (no. 522). But some parameters are not yet covered by that accreditation. At present the accreditation does not cover the parameters marked with a note *. But the analysis was done for these parameters at the same level of quality as for the accredited parameters.

1.4 Uncertainty of the test method

The relative standard deviation of the test method is amounted to 22% (RSD). The expanded uncertainty U_m is 45% and equals $2 \times \text{RSD}\%$, see also www.eurofins.dk, search: Uncertainty.

2 Results

2.1 Emissions Test after 3 Days

| Mulcol® Multisealant A | CAS No. | Retention time min | ID-Cat. | After 3 days $\mu\text{g}/\text{m}^3$ | Criteria e.g. NIK $\mu\text{g}/\text{m}^3$ | R 3 days (c / NIK) | Emission rate $\mu\text{g}/(\text{m}^2 \cdot \text{h})$ | Toluene equivalent $\mu\text{g}/\text{m}^3$ |
|---|-----------|--------------------|---------|---------------------------------------|--|--------------------|---|---|
| TVOC (AgBB/DIBt) (C₆-C₁₆) | | | | 83 | 10 000 | - | 5900 | 58 |
| Substances with NIK value | | | | | | | | |
| 1-Butanol | 71-36-3 | 1.98 | 1 | 39 | 3100 | 0.013 | 2800 | 15 |
| Butyl acetate | 123-86-4 | 3.71 | 2 | 8.6 | 4800 | 0.0018 | 610 | 8.6 |
| Total R for VOC with NIK | | | | - | - | 0.014 | - | - |
| Substances without NIK value | | | | | | | | |
| n-Butyl ether * | 142-96-1 | 5.81 | 2 | 23 | - | - | 1600 | 23 |
| Butylpropionat * | 590-01-2 | 6.20 | 2 | 6.5 | - | - | 460 | 6.5 |
| Not identified * | - | 10.75 | 4 | 5.4 | - | - | 390 | 5.4 |
| Total VOC without NIK value | | | | 35 | - | - | < 400 | < 5 |
| Total VVOC (< n-C₆) | | | | < 5 | - | - | < 400 | < 5 |
| Single VVOC Substances: | | | | | | | | |
| n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total SVOC (> n-C₁₆) | | | | < 5 | - | - | < 400 | < 5 |
| Single SVOC Substances: | | | | | | | | |
| n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total Carcinogens | | | | < 1 | 10 | - | < 80 | < 1 |
| n.d. | - | - | - | < 1 | - | - | < 80 | < 1 |
| Volatile Aldehydes C₁-C₆ measured with DNPH-Method and Ammonia (see 1.3.4 & 1.3.5) | | | | | | | | |
| Formaldehyde | 50-00-0 | - | - | < 3 | 120 | - | < 300 | - |
| Acetaldehyde | 75-07-0 | - | - | < 3 | - | - | < 300 | - |
| C ₃ – C ₆ Aldehydes | - | - | - | < 3 | - | - | < 300 | - |
| Ammonia | 7664-41-7 | - | - | 41 | - | - | 2900 | - |

n.d. Not detected

< Means less than

* Not a part of our accreditation. See 1.3.8 Accreditation

2.2 Emissions Test after 28 Days

| Mulcol® Multisealant A | CAS No. | Retention time min | ID-Cat. | After 28 days $\mu\text{g}/\text{m}^3$ | Criteria e.g. NIK $\mu\text{g}/\text{m}^3$ | R 28 days (c / NIK) | Emission rate $\mu\text{g}/(\text{m}^2 \cdot \text{h})$ | Toluene equivalent $\mu\text{g}/\text{m}^3$ |
|---|-----------|--------------------|---------|--|--|---------------------|---|---|
| TVOC (AgBB/DIBt) (C₆-C₁₆) | | | | < 5 | 1 000 | - | < 400 | < 5 |
| Substances with NIK value n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total R for VOC with NIK | | | | - | 1 | < 1 | - | - |
| Substances without NIK value n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total VOC without NIK value | | | | < 5 | 100 | - | < 400 | < 5 |
| Total VVOC (< n-C₆) | | | | < 5 | - | - | < 400 | < 5 |
| Single VVOC Substances: n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total SVOC (> n-C₁₆) | | | | < 5 | 100 | - | < 400 | < 5 |
| Single SVOC Substances: n.d. | - | - | - | < 5 | - | - | < 400 | < 5 |
| Total Carcinogens | | | | < 1 | 1 | - | < 80 | < 1 |
| n.d. | - | - | - | < 1 | - | - | < 80 | < 1 |
| Volatile Aldehydes C₁-C₆ measured with DNPH-Method and Ammonia (see 1.3.4 & 1.3.5) | | | | | | | | |
| Formaldehyde | 50-00-0 | - | - | < 3 | 120 | - | < 300 | - |
| Acetaldehyde | 75-07-0 | - | - | < 3 | - | - | < 300 | - |
| C ₃ – C ₆ Aldehydes | - | - | - | < 3 | - | - | < 300 | - |
| Ammonia | 7664-41-7 | - | - | 11 | - | - | 790 | - |

n.d. Not detected

< Means less than

* Not a part of our accreditation. See 1.3.8 Accreditation.

Categories of identity:

- 1 = definitely identified, specifically calibrated
- 2 = identified by comparison with a mass spectrum obtained from a library, identity supported by other information, calibrated as toluene equivalent
- 3 = identified by comparison with a mass spectrum obtained from a library, calibrated as toluene equivalent
- 4 = not identified, calibrated as toluene equivalent

The results are only valid for the tested sample(s).

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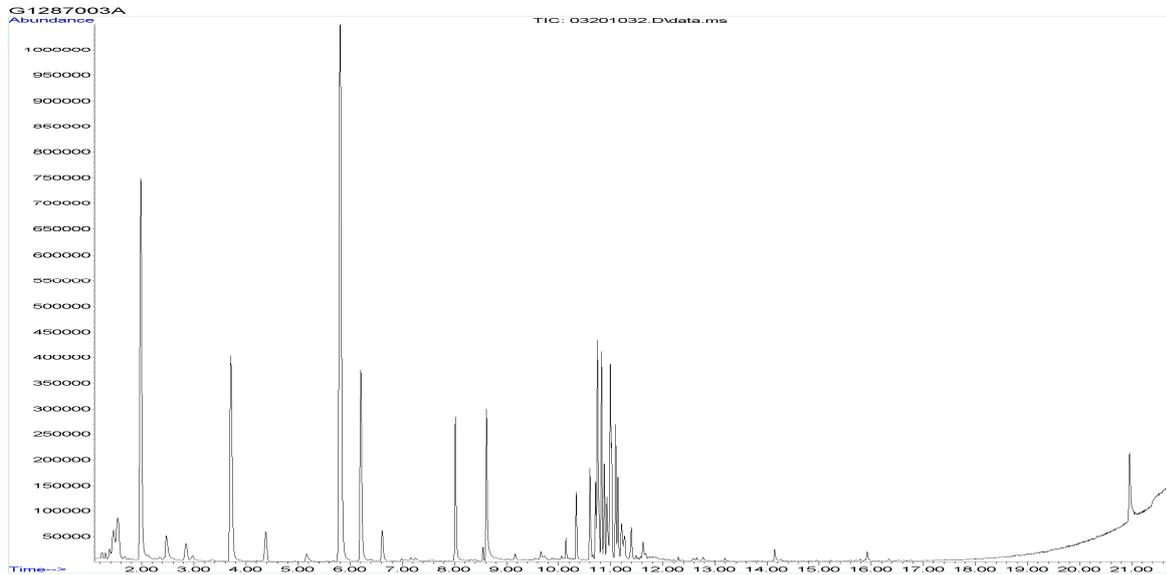
3 Interpretation of the Results

The results of Mulcol® Multisealant A can be summarised as follows:

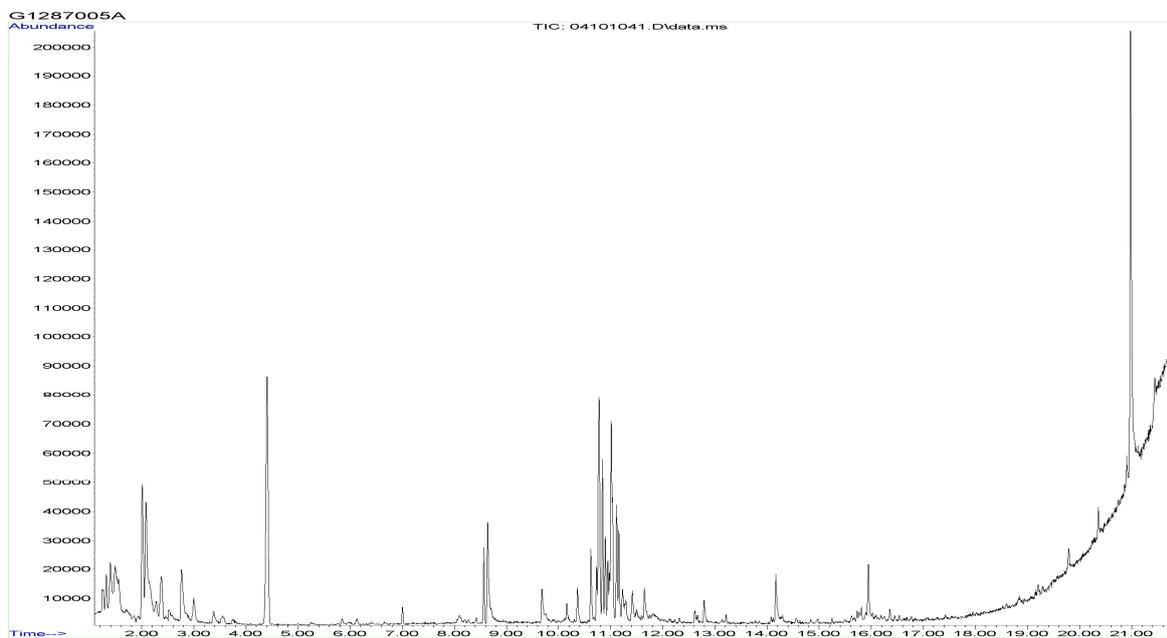
- No carcinogens could be detected after 3 and after 28 days.
- The Total VOC ("TVOC") after 3 days was **below** the classification threshold of 10 mg/m³.
- The Total VOC ("TVOC") after 28 days was **below** the classification threshold of 1.0 mg/m³.
- The Total SVOC after 28 days was **below** the classification threshold of 0.1 mg/m³.
- The classification value R for the VOC substances with more than 5 µg/m³ after 28 days was **below** the classification threshold of 1.
- The Total VOC without assigned NIK value after 28 days was **below** the classification threshold of 0.1 mg/m³.
- The formaldehyde emission after 28 days was **below** the classification threshold of 120 µg/m³.

The tested product Mulcol® Multisealant A complies with the requirements of DIBt (October 2010) and AgBB (May 2010) for use in the indoor environment.

Appendix 1: Chromatogram VOC after 3 days



Appendix 2: Chromatogram VOC after 28 days



Please consider the different scales.

The results are only valid for the tested sample(s).

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