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Date
13 August 2015

Test Report- VOC emission regulations in Europe

1 Sample Information

Sample identification	Protox Hysan
Batch no.	202041
Production date	29-05-15
Product type	Coating
Date when sample was received	02.06.2015
Testing (start - end)	10.06.2015 - 08.07.2015

2 Evaluation of the Results

Germany

The tested product complies with the requirements of DIBt (October 2010) and AgBB (June 2012).

France

CMR-Substances

The tested product fulfills the requirements of the French regulation DEVP0908633A of 30 April 2009 and DEVP0910046A of 28 May 2009.

VOC-emission classification

The tested product is classified to emission class A+.This recommendation is based on the French regulations of March 23 2011 (décret DEVL1101903D) and of April 19 2011 (arrêté DEVL1104875A).

The product was assigned a VOC emission class without taking into account the measurement uncertainty associated with the result. As specified in French Decree no. 2011-321 of March 23 2011, correct assignment of the VOC emission class is the sole responsibility of the party responsible for distribution of the product in the French market.

Belgium

The tested product complies with the requirements of the Royal Decree for establishing threshold levels for the emissions to the indoor environment from construction products for certain intended uses (May 2014).

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3 Test Method

Method	**Principle**	**Parameter**	**Quantification limit**	**Uncertainty**
AgBB Method (version June 2012), DIBt (version October 2010) ISO 16000 parts -3, -6, -9, -11				
Internal method numbers: 9810, 9811, 9812, 2808, 8400	GC/MS GC/MS HPLC	VVOC, VOC, SVOC TVVOC, TVOC, TSVOC Volatile Aldehydes	5 µg/m³ 5 µg/m³ 3 µg/m³	22% (RSD) U_m = 2 x RSD= 45 %
Test chamber parameter				
Chamber volume, l 119	Temperature, °C 23±1		Relative humidity, % 50±3	
Air exchange rate, 1/h 0.5	Loading ratio, m²/m³ 1			
Sample preparation				
Application amount, g/m² 250	Sample was mixed in a ratio Sample:Water = 1:1. The sample was homogenised and applied onto petri discs with planar bottom.			
Deviations from the test method	None			

For detailed method description see below

4 Results

4.1 Emissions Test after 3 Days

	CAS No.	Retention time min	ID-Cat	Concen-tration µg/m³	NIK-value µg/m³	R- value	Emission rate µg/(m²*h)	Toluene equivalent µg/m³
TVOC (C_6-C_{16})				< 5	-	-	< 3	< 5
VOC with NIK n.d.	-	-		< 5	-	-	< 3	< 5
R-value = Σ Conc$_i$/NIK$_i$						0		
VOC without NIK n.d.	-	-	-	< 5	-	-	< 3	< 5
Total VOC without NIK				< 5	-	-	< 3	< 5
Total VVOC (< C_6)				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total SVOC (> C_{16})				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total Carcinogens				< 1	-	-	< 1	< 1
n.d.	-	-	-	< 1	-	-	< 1	< 1

n.d. Not detected
< Means less than
* Not a part of our accreditation.

4.2 Emissions Test after 7 Days

	CAS No.	Retention time min	ID-Cat	Concen-tration µg/m³	NIK-value µg/m³	R- value	Emission rate µg/(m²*h)	Toluene equivalent µg/m³
TVOC (C_6-C_{16})				< 5	-	-	< 3	< 5
VOC with NIK n.d.	-	-		< 5	-	-	< 3	< 5
R-value = Σ $Conc_i/NIK_i$						0		
VOC without NIK n.d.	-	-	-	< 5	-	-	< 3	< 5
Total VOC without NIK				< 5	-	-	< 3	< 5
Total VVOC (< C_6)				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total SVOC (> C_{16})				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total Carcinogens				< 1	-	-	< 1	< 1
n.d.	-	-	-	< 1	-	-	< 1	< 1

n.d. Not detected
< Means less than
* Not a part of our accreditation.

4.3 Emissions Test after 14 Days

	CAS No.	Retention time min	ID-Cat	Concen-tration µg/m³	NIK-value µg/m³	R- value	Emission rate µg/(m²*h)	Toluene equivalent µg/m³
TVOC (C_6-C_{16})				< 5	-	-	< 3	< 5
VOC with NIK n.d.	-	-		< 5	-	-	< 3	< 5
R-value = Σ Conc$_i$/NIK$_i$						0		
VOC without NIK n.d.	-	-	-	< 5	-	-	< 3	< 5
Total VOC without NIK				< 5	-	-	< 3	< 5
Total VVOC (< C_6)				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total SVOC (> C_{16})				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total Carcinogens				< 1	-	-	< 1	< 1
n.d.	-	-	-	< 1	-	-	< 1	< 1

n.d. Not detected
< Means less than
* Not a part of our accreditation.

4.4 Emissions Test after 28 Days

	CAS No.	Retention time min	ID-Cat	Concentration µg/m³	NIK-value µg/m³	R- value	Emission rate µg/(m²*h)	Toluene equivalent µg/m³
TVOC (C_6-C_{16})				< 5	-	-	< 3	< 5
VOC with NIK								
n.d.	-	-		< 5	-	-	< 3	< 5
R-value = Σ Conc$_i$/NIK$_i$						0		
VOC without NIK								
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total VOC without NIK				< 5	-	-	< 3	< 5
Total VVOC (< C_6)				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total SVOC (> C_{16})				< 5	-	-	< 3	< 5
n.d.	-	-	-	< 5	-	-	< 3	< 5
Total Carcinogens				< 1	-	-	< 1	< 1
Benzene	71-43-2	-	-	< 1	-	-	< 1	< 1
Trichlorethylene	79-01-6	-	-	< 1	-	-	< 1	< 1
Dibutylphthalate	84-74-2	-	-	< 1	-	-	< 1	< 1
Diethylhexylphthalate	117-81-7	-	-	< 1	-	-	< 1	< 1
Volatile Aldehydes C_1-C_6 measured with DNPH-Method								
Formaldehyde	50-00-0	-	-	< 3	-	-	< 2	-
Acetaldehyde	75-07-0	-	-	< 3	-	-	< 2	-
Propionaldehyde	123-38-6	-	-	< 3	-	-	< 2	-
Butyraldehyde	123-72-8	-	-	< 3	-	-	< 2	-
Single VOC for French VOC Label								
Toluene	108-88-3	-	-	< 2	-	-	< 2	< 2
Tetrachloroethylene	127-18-4	-	-	< 2	-	-	< 2	< 2
Ethylbenzene	100-41-4	-	-	< 2	-	-	< 2	< 2
Xylene	1330-20-7	-	-	< 2	-	-	< 2	< 2
Styrene	100-42-5	-	-	< 2	-	-	< 2	< 2
2-Butoxyethanol	111-76-2	-	-	< 2	-	-	< 2	< 2
Trimethylbenzene	95-63-3	-	-	< 2	-	-	< 2	< 2
1,4-Dichlorobenzene	106-46-7	-	-	< 2	-	-	< 2	< 2

n.d. Not detected
< Means less than
* Not a part of our accreditation.

Categories of Identity:

1: Identified and specifically calibrated

2: Identified by comparison with a mass spectrum obtained from library and supported by other information. Calibrated as toluene equivalent

3: Identified by comparison with a mass spectrum obtained from a library. Calibrated as toluene equivalent

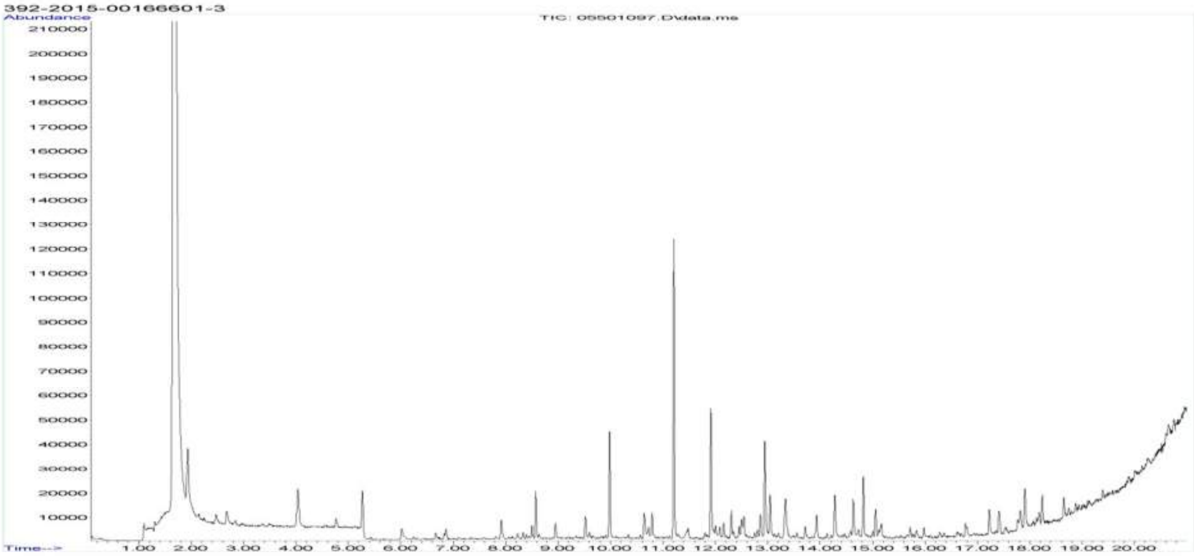
4: Not identified, calibrated as toluene equivalent

Thomas Bjerring
Analytical Service Manager

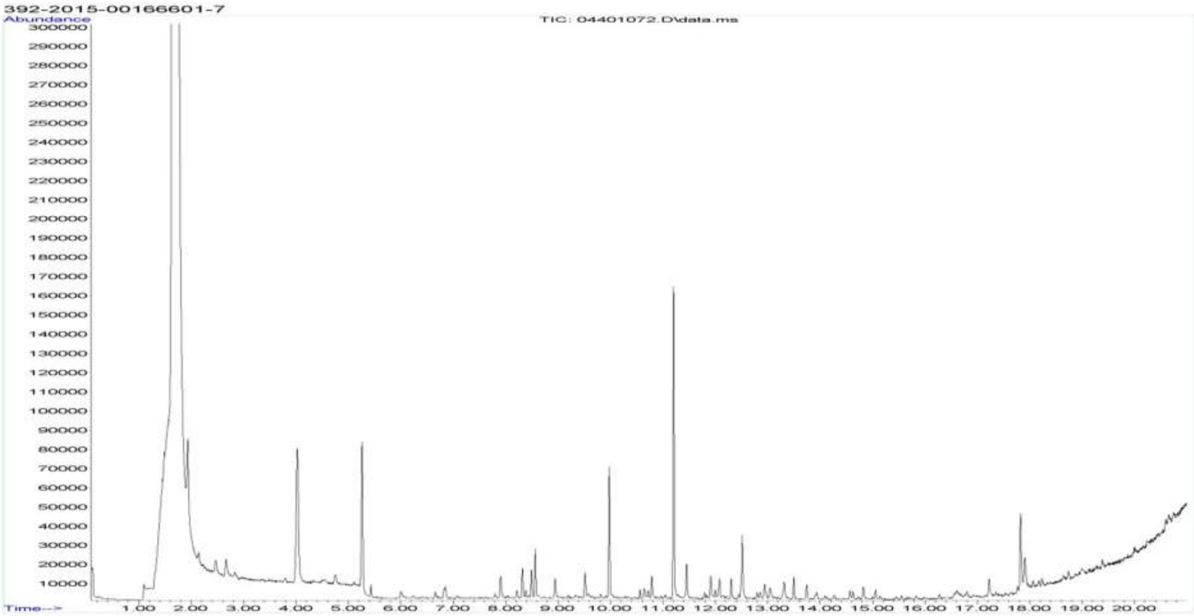
Maria Pelle
Chemist

4.5 Chromatograms

4.5.1 Chromatogram after 3 days

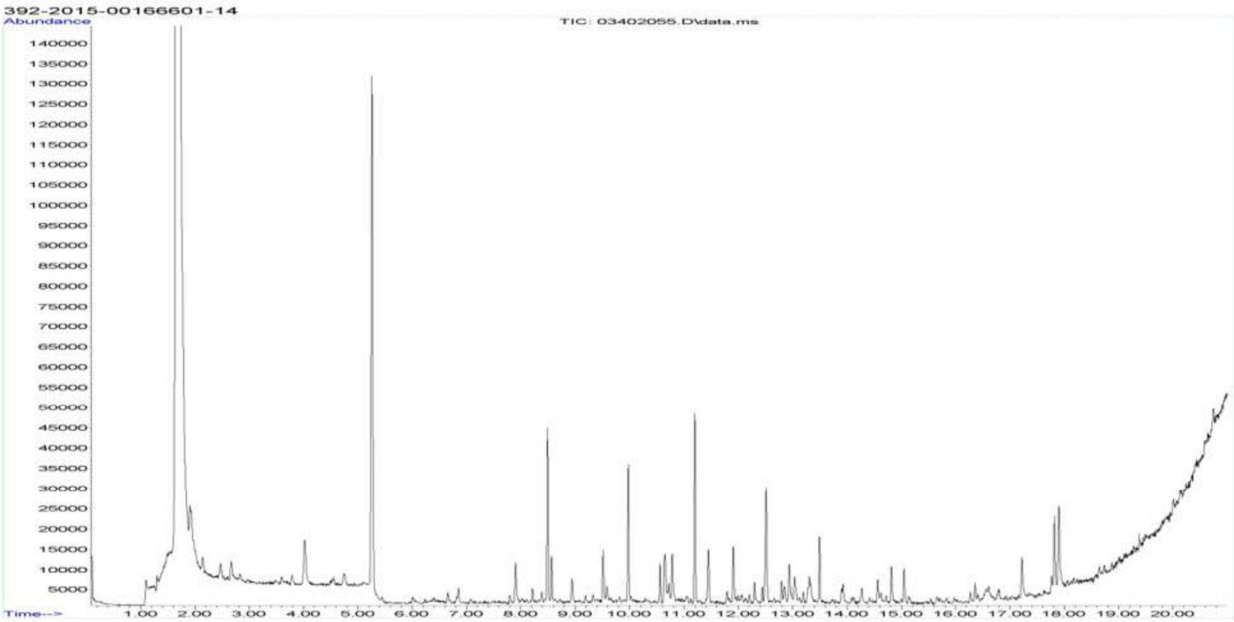


4.5.2 Chromatogram after 7 days

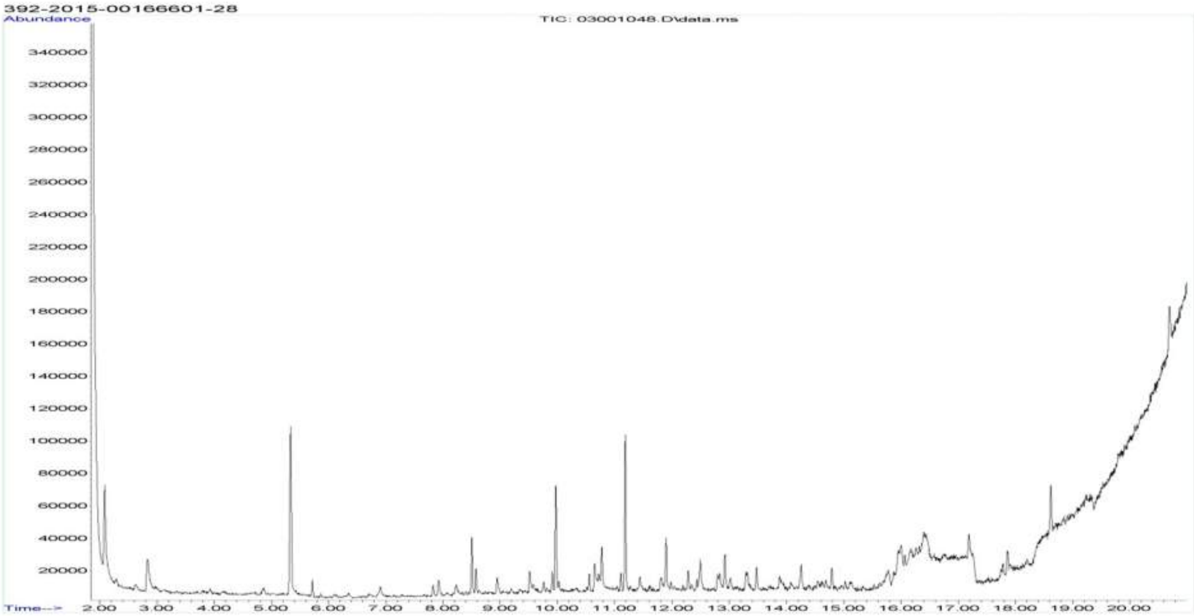


Please consider the different scales

4.5.3 Chromatogram after 14 days

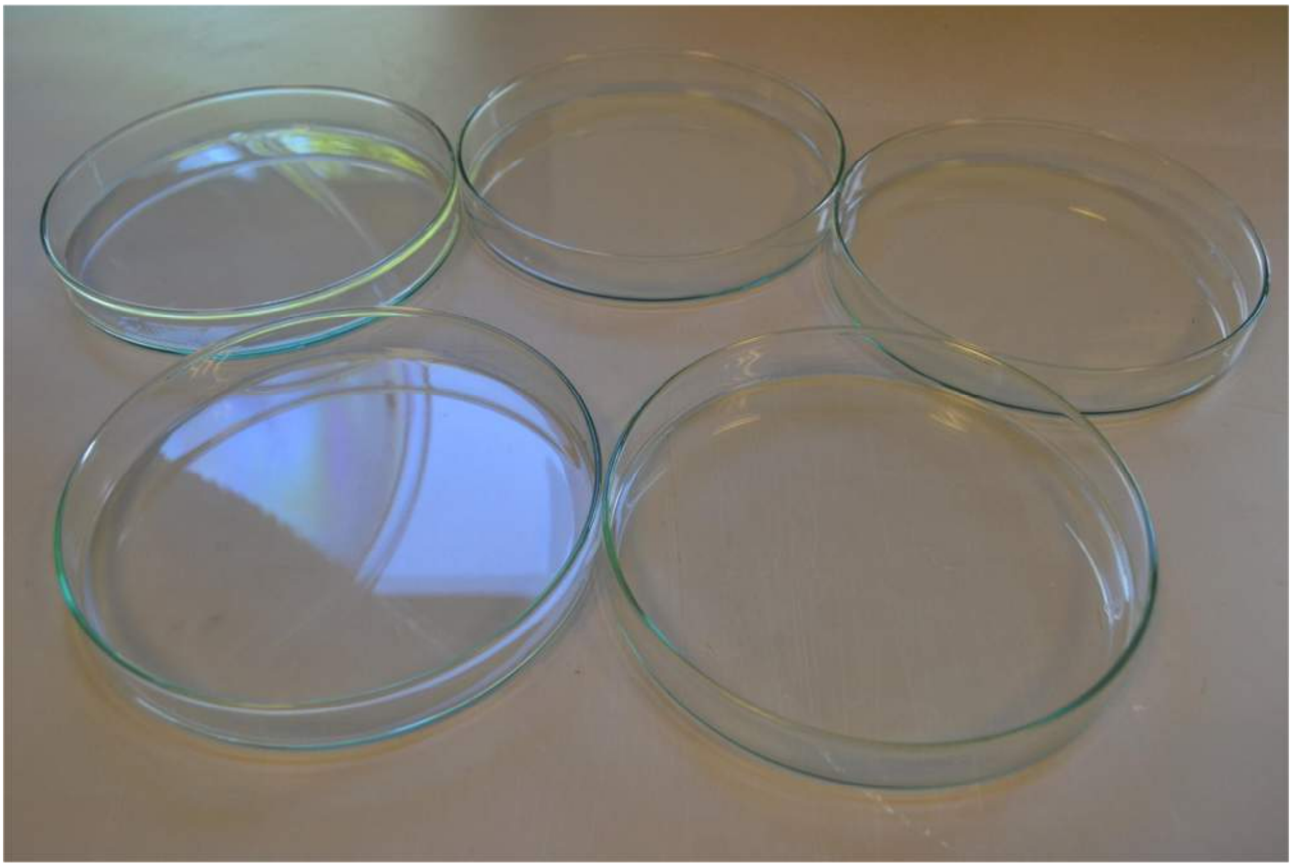


4.5.4 Chromatogram after 28 days



Please consider the different scales

4.6 Image of the sample



5 Appendices

5.1 Description of the applied test method

5.1.1 Test Chamber

The test chamber is made of stainless steel. A multi-step air clean-up is performed before loading the chamber, and a blank check of the empty chamber is performed. The operation parameters are 23 °C, 50 % relative air humidity in the supply air.

5.1.2 Sampling, Desorption, Analysis

Testing for Carcinogens
The presence of carcinogens (EU Categories C1A and C1B, as per the latest publication on the homepage of German BGIA Institute) was tested by drawing sample air from the chamber outlet through 2 Tenax TA tubes (main tube and backup tube). Analysis was performed by thermal desorption and gas chromatography / mass spectroscopy (30 m column, 0.25 mm ID, 0.25 µm HP-5 film, Agilent) (internal methods no.: 9812 / 2808). The absence of a listed carcinogen was stated if the specific combination of fragment ions was absent at the specific retention time in the chromatogram. If no listed carcinogens were found but the required detection limit was exceeded, the identity was checked by comparing full scan sample mass spectra with full scan standard mass spectra.

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

VOC Emissions Testing
The emissions of organic compounds were tested by drawing sample air from the chamber outlet through 2 Tenax TA tubes (main tube and backup tube). Analysis was performed by thermal desorption and gas chromatography / mass spectroscopy (30 m column, 0.25 mm ID, 0.25 µm HP-5 film, Agilent) (internal methods no.: 9812 / 2808).

All single substances that are listed with a NIK value in the latest AgBB publication were identified. 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.

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 performed according to the AgBB/DIBt test method, by addition of the results of all individual substances with concentrations ≥ 5 µg/m^3 in the retention time interval C6-C16. Furthermore the TVOC was calculated as the toluene equivalent, as defined in ISO 16000-6.

Calculation of the TSVOC (Total Semi-Volatile Organic Compounds) was performed by addition of the results of all substances with concentrations ≥ 5 µg/m^3 between C16 and C22 as toluene equivalent, as defined in ISO 16000-6.

Calculation of the TVVOC (Total Very Volatile Organic Compounds) was performed by addition of the results of all substances with concentrations ≥ 5 µg/m^3 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 substances cannot be detected (or with limited reliability only).

5.1.3 Calculation of R Values with the German NIK List

The concentrations of all substances ≥ 5 µg/m^3 in the interval between n-C6 and n-C16 were divided by their respective NIK value (if given). The sum of the quotients gives the R value:

$$R = \sum_{i}^{n} \left(c_i / NIK_I + \ldots + c_n / NIK_n \right)$$

In addition, all results were summed up for the substances without published NIK value, but in the interval be-tween n-C6 and n-C16, when concentrations were ≥ 5 µg/m^3

5.1.4 Testing of Aldehydes after 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.

5.1.5 Quality assurance

Before loading the chamber a blank check of the empty chamber was performed and compliance with back-ground concentrations in accordance with ISO 16000-9 was determined. Sampling at the chamber outlet and subsequent analysis was performed in duplicate. For monitoring any breakthrough or overloading of the tubes, two Tenax TA tubes were used in series.

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 VOCs. Reproducibility of the method was monitored for two selected VOCs per sequence.

5.1.6 Accreditation

The testing methods described above are accredited to EN ISO/IEC 17025:2005 by DANAK (no. 522). Not all parameters are covered by this accreditation. At present the accreditation does not cover the parameters marked with a note *, however analysis was performed for these parameters at the same level of quality as for the accredited parameters.

5.1.7 Uncertainty of the test method

The relative standard deviation of the test method amounts to 22% (RSD). The expanded uncertainty U_m is 45% and equals 2 x RSD%. For further information please visit www.eurofins.dk/uncertainty.

5.2 AgBB/DIBt Limit values 2012

Parameter	**Test after 3 days**	**Test after 28 days**
	Limit value, mg/m^3	**Limit value,** mg/m^3
TVOC	≤ 10	≤ 1.0
TSVOC	-	≤ 0.1
R-value (dimensionless)	-	≤ 1
Total VOC without NIK	-	≤ 0.1
Total Carcinogens	≤ 0.01	≤ 0.001
Formaldehyde	-	≤ 0.12

5.3 Classification according to French VOC-regulation

	C	**B**	**A**	**A+**
TVOC	>2000	<2000	<1500	<1000
Formaldehyde	>120	<120	<60	<10
Acetaldehyde	>400	<400	<300	<200
Toluene	>600	<600	<450	<300
Tetrachloroethylene	>500	<500	<350	<250
Ethylbenzene	>1500	<1500	<1000	<750
Xylene	>400	<400	<300	<200
Styrene	>500	<500	<350	<250
2-Butoxyethanol	>2000	<2000	<1500	<1000
Trimethylbenzene	>2000	<2000	<1500	<1000
1,4-Dichlorobenzene	>120	<120	<90	<60

5.4 Limit-values according to the Royal Belgian Decree for establishing threshold levels for the emissions to the indoor environment from construction products for certain intended uses (May 2014)

Parameter	Test after 28 days
	Limit value, µg/m^3
TVOC	≤ 1000
TSVOC	≤ 100
R-value **(dimensionless)**	≤ 1
Total Carcinogens	≤ 1
Toluene	≤ 300
Formaldehyde	≤ 100
Acetaldehyde	≤ 200