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Emission measurements

(3 appendices)

Object

One sample of sealant was delivered to RISE by the customer.

Product name:	HILTI CFS ACR
Production date:	May 2017
Batch:	2118582
Size of sample:	310 mL cartridge
Date of arrival to RISE:	2018-04-12
Date of analysis:	week 16 - 22

Assignment

Emission measurement according to GEV - Testing Method, 04.10.2017. The measurements are made after 3 and 28 days regarding volatile organic compounds, carcinogenic compounds (EU Regulation No 1272/2008 Annex VI, cat 1A and 1B) and after 3 days also regarding formaldehyde and acetaldehyde.

For evaluation of test results the principle of shared risk is applied, i.e. for a max limit (\leq) a result \leq the limit complies and a result $>$ the limit does not comply (ILAC G8 section 2.7).

Method

The sealant was applied 3 mm thick on a glass plate with a diameter of 150 mm. Applied amount was 97 g. The date of the application was 2018-04-16.

Open surface area was 0.018 m². The specimen was placed directly into the chamber for the 3 days air samplings. Air samplings after 3 days of conditioning were carried out on 2018-04-19.

After 3 days the specimen was placed in a separate conditioning container (with air velocity of ca 0.2 m/s) in a room with controlled climate conditions of 23 \pm 2 °C and 50 \pm 5 % RH. The test specimen was put into the chamber three days prior to 28 days air samplings. Air samplings after 28 days of conditioning were carried out on 2018-05-14.

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Test conditions in the chamber:

Chamber volume:	0.25 m ³
Temperature:	23 ± 0.5 °C
Relative humidity:	50 ± 5 % RH
Surface area of test specimen:	0.018 m ²
Air exchange rate:	0.5 h ⁻¹
Area specific air flow rate:	7.0 m ³ /m ² h.
Air velocity at specimen surface:	0.1 – 0.3 m/s

Tenax TA was used as adsorption medium for VOC. The tubes were thermally desorbed and analysed in accordance to RISE method 0601, similar to ISO 16000-6:2011 (Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS/FID). This means an analysis in a gas chromatograph and detection with a flame ionisation detector (FID) and mass selective detector (MS). The capillary column used is coated with 5% phenyl/ 95 % methylpolysiloxane. The FID signals are used for compound quantification. The total volatile organic compounds (TVOC) means compounds eluting between and including n-hexane to hexadecane, having boiling points in the range of about 70-260 °C. Minimum duplicate air samples were taken and the results are mean values. Sampled volumes are 3 to 9 L.

Tenax TA was also used as adsorption medium for testing of volatile carcinogenic compounds according to EU Regulation No 1272/2008 Annex VI, cat 1A and 1B), (exclusive formaldehyde), 1 µg/m³ and above.

The samplings of aldehydes were carried out with DNPH samplers. The samplers were analysed according to RISE method 2302, similar to ISO 16000-3:2011 (Indoor air - Part 3: Determination of formaldehyde and other carbonyl compounds – Active sampling method). This means analysis on a liquid chromatograph with absorbance detector. Duplicate air samples were taken and the results are mean values. Sampled volumes were 30 to 50L.

Results

The results in Table 1 and 2 are expressed as area specific emission rates and as concentrations in a reference room (according to EN 16516:2017). The reference room has a base area of 3 m x 4 m and a height of 2.5 m, with an air exchange rate of 0.5 h⁻¹. The wall area is 31.4 m², floor area is 12 m², small area, like a door, is 1.6 m² and very small area, like sealant, is 0.2 m². Very small area is used for the calculation of the concentrations.

Calculation of the concentration from the emission rate:

$$C = \frac{E_a \times A}{n \times V}$$

C = concentration of VOC in the reference room, in µg/m³

E_a = area specific emission rate, in µg/m²h

A = surface area of product in reference room, in m²

n = air exchange rate, in changes per hour, here 0.5 h⁻¹

V = volume of the reference room, in m³, here 30 m³

Table 1.
Emission results of **HILTI CFS ACR** after 3 days:

Volatile organic compounds	CAS number	Retention time (min)	ID ¹	Concentration in reference room ($\mu\text{g}/\text{m}^3$)	LCI _i ($\mu\text{g}/\text{m}^3$)	R _i (c_i/LCI_i)
TVOC (C ₆ – C ₁₆)	--	6.5 – 38	B	910	--	--
Volatile Carcinogens ²		6.5 – 38				
No substances detected	--	--	B	< 1	--	--
VOC with LCI ³		6.5 – 38				
1-Butanol	71-36-3	7.7	A	28	3000	--
Propylene glycol	57-55-6	10.2	A	2500	2100	--
Σ VOC with LCI	--	--	A	2500	--	
VOC without LCI ⁴						
No substances detected	--	--	B	< 5	--	--
Σ VOC without LCI	--	--	B	< 5	--	--
SVOC (C ₁₆ – C ₂₂) ⁵		38 - 51				
No substances detected	--	--	B	< 5	--	--
Σ SVOC	--	--	B	< 5	--	--
VVOC (< C ₆) ⁶		4.8 – 6.5				
Formaldehyde ⁷	50-00-0	--	A	< 5	100	--
Acetaldehyde ⁷	75-07-0	--	A	< 5	1 200	--
Σ VVOC	--	--	A	< 5	--	--
R = $\Sigma C_i / \text{LCI}_i$ ⁸	--	--	--	--	--	--

¹⁾ ID: A = quantified compound specific, B = quantified as toluene-equivalent

²⁾ Volatile carcinogens = VOCs according to EU Regulation No 1272/2008 Annex VI, cat 1A and 1B

³⁾ VOC with LCI = identified VOC-compound with LCI-value according to EU-LCI, Dec 2016

⁴⁾ VOC without LCI = VOC-compound without LCI-value or not identified.

⁵⁾ SVOC = semi-volatile organic compounds, as defined in ISO 16000-6 (not part of accreditation)

⁶⁾ VVOC = very volatile organic compounds, as defined in ISO 16000-6 (not part of accreditation)

⁷⁾ VVOC-aldehydes measured with DNPH samplers (ISO 16000-3)

⁸⁾ All VVOC, VOC, SVOC and carcinogens with LCI

Only carcinogenic compounds $\geq 1 \mu\text{g}/\text{m}^3$ are listed in Table 1. TVOC expressed in $\mu\text{g}/\text{m}^3$ is the sum of all individual substances with concentrations $\geq 5 \mu\text{g}/\text{m}^3$ (in toluene equivalents).

Quantification limit for TVOC is $10 \mu\text{g}/\text{m}^3\text{h}$. Measurement uncertainty for VOC is 15 % (rel) and for formaldehyde 30 % (rel). Background of TVOC in the empty chamber was below $20 \mu\text{g}/\text{m}^3$ and is subtracted.

Table 2.
Emission results of **HILTI CFS ACR** after 28 days:

Volatile organic compounds	CAS number	Retention time (min)	ID ¹	Concentration in reference room ($\mu\text{g}/\text{m}^3$)	LCI _i ($\mu\text{g}/\text{m}^3$)	R _i (c_i/LCI_i)
TVOC (C₆ – C₁₆)	--	6.5 – 38	B	170	--	--
Volatile Carcinogens²		6.5 – 38				
No substances detected	--	--	B	< 1	--	--
VOC with LCI³		6.5 – 38				
Propylene glycol	57-55-6	10.2	A	480	2100	0.23
∑ VOC with LCI	--	--	A	480	--	--
VOC without LCI⁴						
No substances detected	--	--	B	< 5	--	--
∑ VOC without LCI	--	--	B	< 5	--	--
SVOC (C₁₆ – C₂₂)⁵		38 - 51				
No substances detected	--	--	B	< 5	--	--
∑ SVOC	--	--	B	< 5	--	--
VVOC (< C₆)⁶		4.8 – 6.5				
No substances detected	--	--	B	< 5	--	--
∑ VVOC	--	--	B	< 5	--	--
R = ∑ C_i / LCI_i⁷	--	--	--	--	--	0.23

¹) ID: A = quantified compound specific, B = quantified as toluene-equivalent

²) Volatile carcinogens = VOCs according to EU Regulation No 1272/2008 Annex VI, cat 1A and 1B

³) VOC with LCI = identified VOC-compound with LCI-value according to EU-LCI, Dec 2016

⁴) VOC without LCI = VOC-compound without LCI-value or not identified.

⁵) SVOC = semi-volatile organic compounds, as defined in ISO 16000-6 (not part of accreditation)

⁶) VVOC = very volatile organic compounds, as defined in ISO 16000-6 (not part of accreditation)

⁷) All VVOC, VOC, SVOC and carcinogens with LCI

Only carcinogenic compounds $\geq 1 \mu\text{g}/\text{m}^3$ are listed in Table 2. Only the compounds with a concentration in the reference room $> 5 \mu\text{g}/\text{m}^3$ are evaluated based on LCI (= lowest concentration of interest). TVOC expressed in $\mu\text{g}/\text{m}^3$ is the sum of all individual substances with concentrations $\geq 5 \mu\text{g}/\text{m}^3$ (in toluene equivalents).

See Appendix 1 for a gas chromatogram (FID spectra) and Appendix 2 for a photo of the test specimen. Appendix 3 is the sampling report received from the customer.

Evaluation of the test results

The test results are evaluated in Table 3.

Table 3.
Comparison of requirements and test results

Volatile organic compound	Requirement EC2 (µg/m³)	Test Results (µg/m³)	Pass / Fail
TVOC after 3 days	≤ 3000	910	PASS
TVOC after 28 days	≤ 300	170	PASS
TSVOC after 28 days	≤ 100	< 5	PASS
Formaldehyde after 3 days	≤ 50	< 5	PASS
Acetaldehyde after 3 days	≤ 50	< 5	PASS
Sum of form- and acetaldehyde after 3 days	≤ 0.05 ppm	< 0.001 ppm	PASS
Sum of CMR 1A+1B after 3 days	≤ 10	< 1	PASS
Any CMR 1A+1B after 28 days	≤ 1	< 1	PASS

The test results are in compliance with the requirements of Eimicode label EC2.

RISE Research Institutes of Sweden AB **Chemistry and Materials - Chemistry**

Performed by

Examined by

Thomas Vaessen

Tove Malin

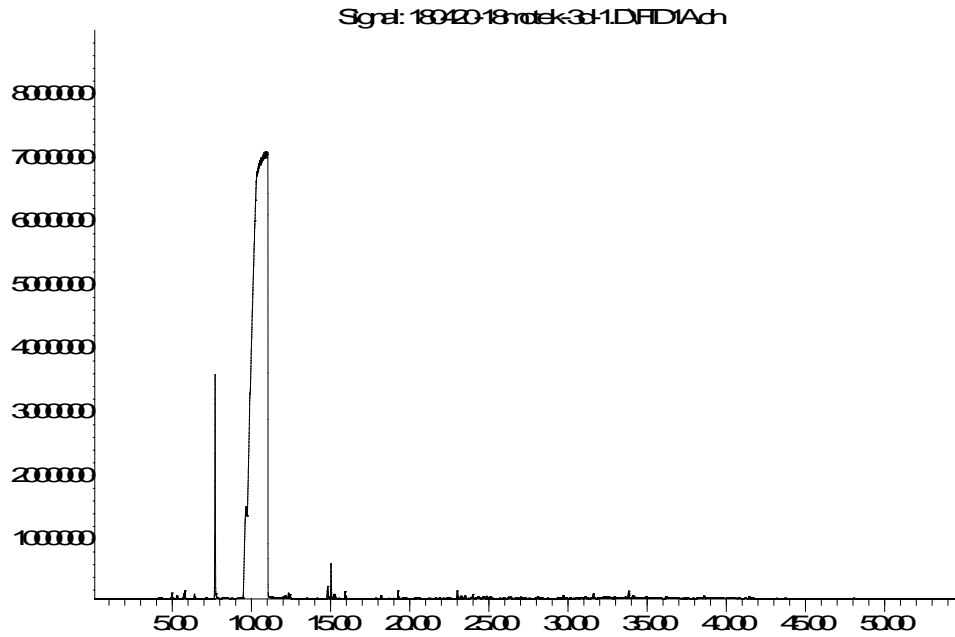
Appendices

1. Gas Chromatogram
2. Photo of the test specimen
3. Sampling report

Appendix 1

Gas chromatogram**HILTI CFS ACR, after 3 days:**

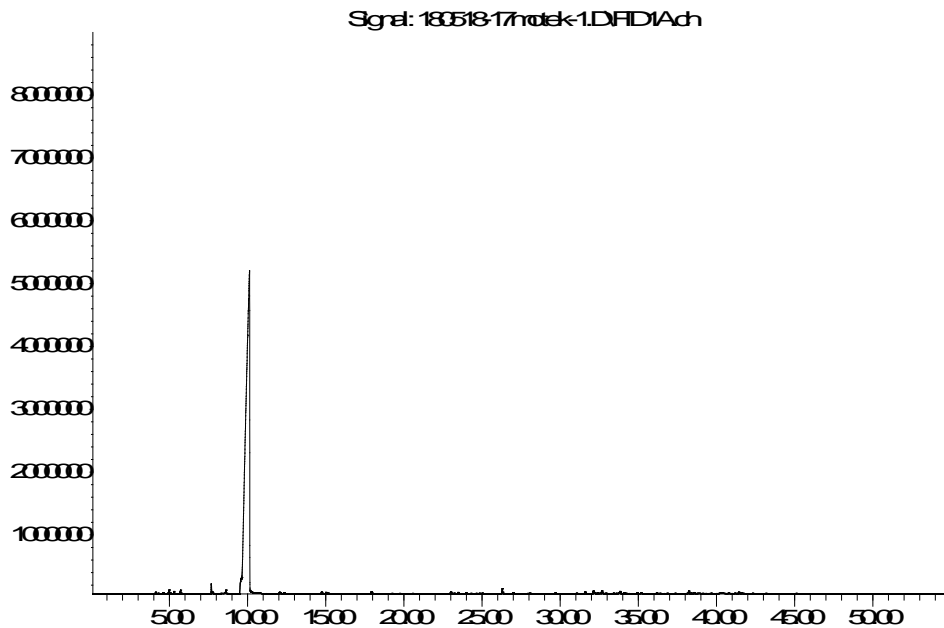
Abundance



Time->

HILTI CFS ACR, after 28 days:

Abundance



Time->

TVOC between C₆ and C₁₆, means compounds eluting between 6.2 and 37.9 minutes.

Appendix 2

Photo of test specimen**HILTI CFS ACR**

Appendix 3

Sampling Report

<p>Sampler (Name, Company, contact info):</p> <p>Dr Philippe Brachet Produktsjef / Product Manager Mobil: 474 53 930 Motek AS Alf Bjerckesvei 22 B Box 81 Økern, N-0508 Oslo www.motek.no</p>	<p>Manufacturer of the product (Company, address):</p> <p>Hilti Feldkircherstrasse 100. Postfach 333 9494 Schaan Liechtenstein</p>
<p>Name of product:</p> <p>HILTI CFS ACR</p>	<p>Type of product:</p> <p>Firestop acrylic sealant https://www.hilti.se/brandskyddssystem/brandfogmassor/r4883</p>
<p>Manufacturing Date:</p> <p>05-2017</p>	<p>Batch No:</p> <p>2118582</p>
<p>Date of sampling:</p>	<p>Amount/size of material sampled:</p> <p>310 ml</p> <p>Packing material: cartridge</p>
<p>Sample is taken from:</p> <p>Production line <input type="checkbox"/></p> <p>Stock / Storage <input checked="" type="checkbox"/></p> <p>Miscellaneous <input type="checkbox"/></p> <p>-where, specify:</p>	<p>How was the product stored before sampling?</p> <p>Storage facility</p>
<p>If a sub-sample was collected from a larger material amount, describe how the sub-sample was taken:</p>	
<p>Observations and remarks:</p> <p>Brand new cartridge</p>	

Appendix 3

Confirmation I hereby confirm that the sample was selected, taken and packed in accordance with the instructions.	
Date: 20-03-2018	Signature: 