

## Emission measurements according to M1

(3 appendices)

### Assignment

Emission measurement according to “M1 Emission Classification of Building Materials: Protocol for Chemical and Sensory Testing of Building Materials”, version 15.11.2017, after 28 days of conditioning regarding volatile organic compounds, carcinogenic compounds (EU Regulation No 1272/2008 Annex VI, cat 1A and 1B), formaldehyde, ammonia and sensory acceptability.

### Product/test specimen

Table 1.

Product type:	Self-levelling compound
Product name:	<b>Sikafloor -40 HyCem One</b>
Manufacturer:	Sika Limited, UK
Manufacturing date:	2021-04-30
Sampling date:	2021-06-15
Batch No:	3005386879
Size of sample, packaging:	25 kg, in a retail bag wrapped in plastic
Arrived at RISE:	2021-06-15
Test specimen preparation:	Floor scenario is used for the testing.  The self-levelling compound was mixed in a Hobart blender according to the manufacturer’s instructions: Add 1 kg powder to every 220 g water and mix for 4 minutes, leave for 3 – 5 min.  Chemical testing: The compound was poured into a stainless steel mould (250 x 250 mm) to a thickness of 3 mm. Ammonia test: The compound was poured into a glass mould (200 x 200 mm) to a thickness of 3 mm.  Sensory testing: The compound was poured into glass moulds with a total surface area of 0.43 m <sup>2</sup> to a thickness of 3 mm.
Test period started, date:	2021-09-02 (Chemical excluding ammonia) 2021-09-23 Ammonia and Sensory testing
Conditions during ageing:	23 ± 2 °C, 50 ± 5 % RH
Emission samplings, date:	Chemical (excluding ammonia): 2021-09-30 Ammonia and Sensory testing: 2021-10-20

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## Methods

The specimens were conditioned outside the testing chambers in a room with controlled climate conditions of  $23 \pm 2$  °C and  $50 \pm 5$  % RH. The specimens were placed in the chambers three days before the measurements of the chemical emission and the sensory evaluation.

**Table 2.**

Chamber conditions of the test of chemical emissions

Test chamber volume:	0.031 m <sup>3</sup> , stainless steel
Temperature:	23 ± 1 °C
Relative Humidity:	50 ± 3 % RH
Air exchange rate:	0.68 h <sup>-1</sup>
Air velocity at specimen surface:	0.1 – 0.3 m/s
Area of sample, Chemical:	0.063 m <sup>2</sup>
Area of sample, Ammonia:	0.04 m <sup>2</sup>
Area specific air flow rate, Chemical:	0.34 m <sup>3</sup> /m <sup>2</sup> h
Area specific air flow rate, Ammonia:	0.53 m <sup>3</sup> /m <sup>2</sup> h

**Table 3.**

Chamber conditions of the test of sensory acceptability

Test chamber volume:	1.0 m <sup>3</sup> , stainless steel
Temperature:	23 ± 1 °C
Relative Humidity:	50 ± 3 % RH
Supply air flow rate:	0.6 l/s = 2.2 m <sup>3</sup> /h
Area of sample:	0.43 m <sup>2</sup>

**Table 4.**

Emission sampling and analytical methods

Test	Sampling method	Adsorbent	Sampling volume (litre)	Analysis method / Quantification	Detection limit
VOC	ISO 16000-9:2006 <sup>1</sup>	Tenax TA	2.4 – 6.9	ISO 16000-6 <sup>2</sup> / FID quantification	1 µg/m <sup>3</sup>
Formaldehyde	ISO 16000-9:2006 <sup>1</sup>	DNPH	20	SP 2303 <sup>3</sup> /HPLC-UV	0.03 µg/sampler
Ammonia	ISO 16000-9:2006 <sup>1</sup>	Treated silica gel	129 – 172	Liquid chromatograph with conductivity detector <sup>4</sup>	1.0 µg/sampler
Sensory evaluation	ISO 16000-28:2012 <sup>5</sup>	--	--	Acceptability, Untrained panel of min 15 persons	--

<sup>1</sup>) In accordance with ISO 16000-9:2006 and M1 protocol.

<sup>2</sup>) In accordance with ISO 16000-6:2011 and M1 protocol.

<sup>3</sup>) In accordance with ISO 16000-3:2001.

<sup>4</sup>) Not accredited method.

<sup>5</sup>) In accordance with M1 protocol, not accredited method.

Tenax TA was used as adsorption medium for VOC. The tubes were thermally desorbed and analysed in accordance 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 FID signals are used for compound quantification. The TVOC is quantified as toluene equivalents.

The mass selective detector is used for identification of compounds. The capillary column used is coated with 5% phenyl/ 95 % methylpolysiloxane. 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), 0.001 mg/m<sup>3</sup> and above.

The sampling of formaldehyde was carried out with DNPH samplers. The samplers were analysed according to SP method 2302, similar to ISO 16000-3:2011(Indoor air--Part 3: Determination of formaldehyde and other carbonyl compounds – Active sampling method), which means analysis on a liquid chromatograph with absorbance detector.

The sampling of ammonia was carried out with silicagel treated adsorbent tubes and analysis on a liquid chromatograph with conductivity detector.

Minimum two subsequent samples were taken for the determination of VOC, formaldehyde and ammonia respectively.

## Results

The results relate only to the items tested.

Decision rule: When comparing the measured results and requirement level, the average value of the measured results has been compared with the requirement level. No account is taken to the measurement uncertainty.

The results of the chemical testing are expressed as area specific emission rates and as concentrations in a model room. The model 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<sup>-1</sup>. The wall area is 31.4 m<sup>2</sup>, floor area is 12 m<sup>2</sup>, small area, like a door, is 1.6 m<sup>2</sup> and very small area, like sealant, is 0.2 m<sup>2</sup>. Floor area is used for the calculation of the concentrations.

Calculation of the concentration from the emission rate:

$$Conc = \frac{SER_A \times A}{n \times V}$$

Conc = concentration of a VOC in the model room, in µg/m<sup>3</sup>  
SER<sub>a</sub> = area specific emission rate, in µg/m<sup>2</sup>h  
A = area of sample, in m<sup>2</sup>  
n = air exchange rate, in changes per hour  
V = volume of the model room, in m<sup>3</sup>

**Table 5.**

Results of the chemical testing of the sample **Sikafloor -40 HyCem One**, after 28 days

Compound	Concentration in model room mg/m <sup>3</sup>	Emission rate mg/m <sup>2</sup> h	Criteria M1 mg/m <sup>2</sup> h
TVOC <sup>6</sup>	< 0.005	< <b>0.010</b>	< 0.2
Carcinogens	< 0.001	< <b>0.001</b>	< 0.001
Single VOC (µg/m <sup>3</sup> )	< EU-LCI	--	≤ EU-LCI
Formaldehyde	0.020	<b>0.025</b>	< 0.05
Ammonia <sup>7</sup>	< 0.003	< <b>0.004</b>	< 0.03

<sup>6</sup>) The TVOC is the sum of the individual concentration ≥ 5 µg/m<sup>3</sup> in model room.

<sup>7</sup>) Not accredited method.

**Table 6.**

Results of the sensory acceptability evaluation of the sample **Sikafloor -40 HyCem One**, after 28 days

Evaluator	Sensory evaluation	Criteria M1
1	0.86	
2	0.92	
3	0.94	
4	0.59	
5	0.96	
6	0.95	
7	0.94	
8	1.00	
9	0.98	
10	1.00	
11	0.93	
12	0.92	
13	0.98	
14	0.98	
15	0.96	
Arithmetic mean of acceptability <sup>8</sup>	<b>0.93</b>	$\geq + 0.0$
Standard deviation	0.10	
90 % confidence interval of arithmetic mean	0.04	$\leq 0.2$

<sup>8)</sup> Not accredited method.

The empty sensory test chamber acceptability was determined 2021-10-18. The mean acceptability vote of the empty chamber was  $> 0.8$ .

### Interpretation of the results

The tested product **Sikafloor -40 HyCem One** complies with all the requirements of M1 for the tested parameters.

### Detailed results

**Table 7.**

Detailed results (emission rates) of the chemical testing after 28 days

Sample	TVOC (mg/m <sup>2</sup> h) as toluene equivalents between C <sub>6</sub> -C <sub>16</sub>	Formaldehyde (mg/m <sup>2</sup> h)	Ammonia (mg/m <sup>2</sup> h)	Carcinogens (mg/m <sup>2</sup> h) between C <sub>6</sub> -C <sub>16</sub>
1	< 0.010	0.025	< 0.003	< 0.001
2	< 0.010	0.025	< 0.004	< 0.001

**Table 8.**Single VOCs above 5 µg/m<sup>3</sup> in the model room (floor area scenario)

Single VOCs	CAS number	Retention time (min)	ID <sup>9</sup>	Emission rate (µg/m <sup>2</sup> h)	Concentration (µg/m <sup>3</sup> )
<b>Single VOCs C<sub>6</sub>-C<sub>16</sub>:</b>	--	6.2 – 38			
No substances detected	--	--	B	< 2	< 5
<b>TVOC</b>	--	6.2 – 38	B	< 10	< 5
<b>Volatile Carcinogens <sup>10</sup></b>		6.2 – 38			
No substances detected	--	--	B	< 1	< 1
<b>Single VOC outside C<sub>6</sub> – C<sub>16</sub>:</b>					
VVOC (< C <sub>6</sub> ) <sup>11</sup>		4.5 – 6.2			
No substances detected	--	--	B	< 2	< 5
SVOC (C <sub>16</sub> – C <sub>22</sub> ) <sup>12</sup>		38 – 51			
No substances detected	--	--	B	< 2	< 5

<sup>9)</sup> ID: A = quantified compound specific, B = quantified as toluene-equivalent

<sup>10)</sup> Volatile carcinogens = VOCs according to EU Regulation No 1272/2008 Annex VI, cat 1A and 1B

<sup>11)</sup> VVOC = very volatile organic compounds, as defined in ISO 16000-6 (not accredited)

<sup>12)</sup> SVOC = semi-volatile organic compounds, as defined in ISO 16000-6 (not accredited)

TVOC is the sum of all individual substances with concentrations  $\geq 5 \mu\text{g}/\text{m}^3$  in the model room (in toluene equivalents). Level of identification of compounds is 100 % for all compounds  $\geq 5 \mu\text{g}/\text{m}^3$

There were no compounds quantified and no EU-LCI-values were evaluated.

### Measurements uncertainty

The expanded measurement uncertainty of VOC result is 15 % (rel) and formaldehyde is 30 % (rel). For ammonia the measurement uncertainty is estimated to 20 % (rel).

See Appendix 1 for a gas chromatogram from the VOC determination and Appendix 2 for a photo of a test specimen. Appendix 3 is the Sampling report received from the customer.

### RISE Research Institutes of Sweden AB Chemistry and Applied Mechanics - Chemical Product Safety

Performed by

Examined by

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### Appendices

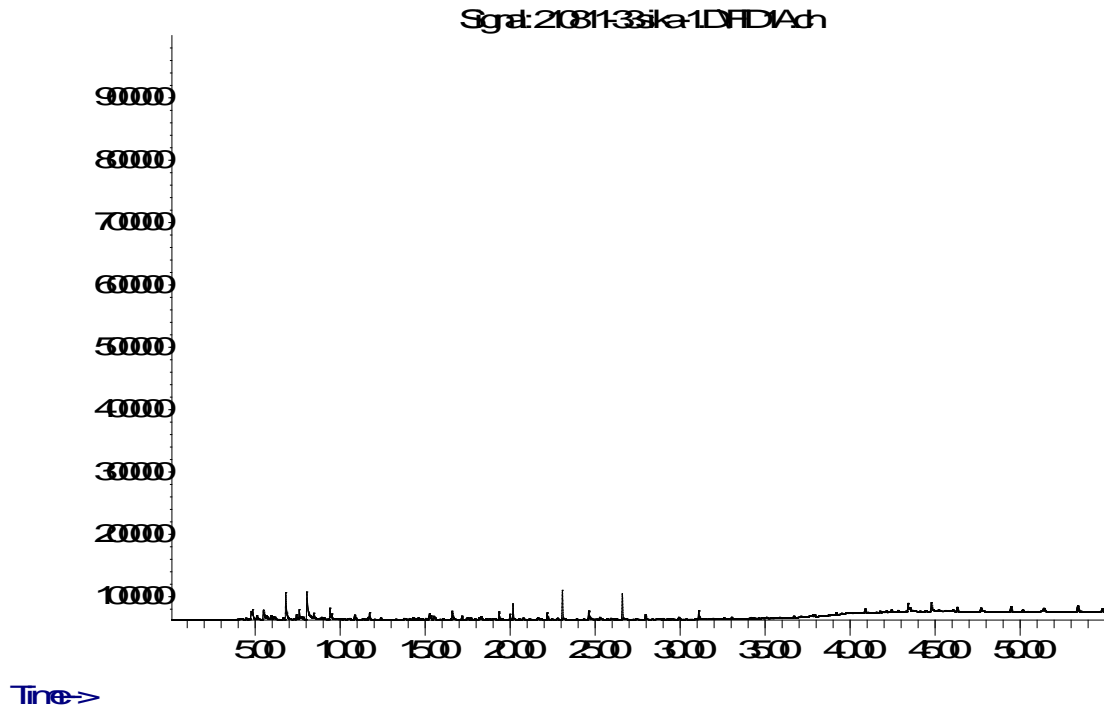
1. Gas Chromatogram
2. Photo of a test specimen
3. Sampling report

Appendix 1

**Gas chromatogram**

Sample: **Sikafloor -40 HyCem One**, after 28 days

**Aurdata**



TVOC between C<sub>6</sub> and C<sub>16</sub>, means compounds eluting between 6.2 and 38 minutes.

## Appendix 2

**Photo of a test specimen**

Specimen for the chemical emission

## Appendix 3

## Sampling Report

<b>Sampler</b> (Name, Company, contact info): SIKA Sverige AB Domnarvsgatan 15 163 55, Spånga Sweden Kaj Tuomela 070-228 95 32	<b>Manufacturer of the product</b> (Company, address): SIKA Limited (SIKA UK) Watchmead Welwyn Garden City AL7 1BQ +44 1707 39 44 44
<b>Name of product:</b> Sikafloor -40 HyCem One	<b>Type of product:</b> 1-PART POLYMER MODIFIED FLEXIBLE CEMENTITIOUS SELF-LEVELLING COMPOUND.
<b>Manufacturing Date:</b>  30/04/21	<b>Batch No:</b>  3005386879
<b>Date of sampling:</b> 2021-06-15	<b>Amount/size of material sampled:</b>  25 kg bag  <b>Packing material:</b>
<b>Sample is taken from:</b> Production line <input type="checkbox"/> Stock / Storage <input checked="" type="checkbox"/> Miscellaneous <input type="checkbox"/> -where, specify:	<b>How was the product stored before sampling?</b>  in a sack on a wrapped pallet
<b>If a sub-sample was collected from a larger material amount, describe how the sub-sample was taken:</b>	
<b>Observations and remarks:</b>	
<b>Confirmation</b> I hereby confirm that the sample was selected, taken and packed in accordance with the instructions.	
<b>Date:</b> 2021-6-15	<b>Signature:</b> 