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ANALYSIS REPORT

Client	TWO Teknik ApS Korngården 6 4660 St. Heddinge Att: Tim Warner
Case number/ref.	TWO, tests of trace elements
Journal number	M0009 og M0010
Receiving date	2016-05-04
Report version	Version 1.0

This report describes to assignments performed by Dansk MiljøAnalyse for TWO Teknik ApS. The Report consists of two parts.

Part 1: DMA report journal number M0009. This report describes three tests of the two-component silicate coating "SPS Primær" and a synthetic tracer compound: 4,4´-2,2,2- trifluoroethoxy biphenyl (in the following referred to as "SPS Trace").
The first test is an evaporation test. The test shows that a coating of SPS Primær is able to contain PCB and Biphenyl. The test also shows that SPS Trace first evaporates at an elevated temperature.
The next two tests are surface wipe tests. These tests again demonstrate that a coating of SPS Primær is able to contain Biphenyl, PCB, Bisphenol-A and SPS Trace.

Part 2: DMA report journal number M0010. This report is an expanded test of SPS Primær and SPS Trace.
The test is an evaporation test. Like the first test in M0009. The difference between the two tests is the addition of an Aroclor standard as the PCB source instead of "PCB capacitor oil", and the use of a higher SPS Trace concentration. The test demonstrates a similar evaporative behaviour of PCB and SPS Trace when the SPS Primær sealing is broken.

Part 1

Assignment

Dansk MiljøAnalyse has been given the assignment to test different chemical compounds for use as tracers in connection with sealing of PCB with the two-component silicate coating "SPS Primær". The assignment is divided into three parts.

1. Test of evaporation from concrete tiles which have been partially coated with 1) a PCB standard mix, 2) the tracer compound 4,4´-2,2,2- trifluoroethoxy biphenyl (in the following referred to as "SPS Trace"), Biphenyl and perdeuterated biphenyl (Biphenyl-d_6) and how this is affected by sealing with "SPS Primær", a two-component silicate coating. The test also includes an analysis of the evaporative behaviour of the components when the sealing is broken.
2. Test of concrete tiles sealed with SPS Primær and coated with; PCB, SPS Trace, Biphenyl and Biphenyl-d
3. Test of concrete tiles sealed with SPS Primær and coated with; PCB, SPS Trace, Bisphenol-A and Bisphenol-A-d

Chemicals

PCB oil from a capacitor (the oil is diluted 100 times in a mixture of cyclohexane/acetone 50/50), SPS Trace diluted in ethanol (250 ppm), Bisphenol A, Bisphenol A-d_{16} (Sigma Aldich), Biphenyl, Biphenyl-d_{10} (Sigma Aldrich), Dutch Seven PCB Mixture (NEN 5734/VPR C85-16, LGC Standards), PCB mix (certified reference material bcr – 365, LGC Standards), DBOB 4,4´-Dibromoocta-fluorobiphenyl (Sigma Aldrich), PCB202 2,2',3,3',5,5',6,6'-Octachlorobiphenyl (13C12,99%, LGC Standards) SPS Primær + curing agent (delivered by TWO Teknik ApS), cyclohexane, and acetone.

Sources of error

A main source of error during the test is the use of "free" PCB (PCB in solution not bounded). When adding a PCB in a solution to the concrete and afterward sealing the concrete, some of the PCB may be dissolved in the sealing layer which might explain the background level of PCB in the air sample. Another consequence hereby is the possible pollution of sealing on the brush which can contaminate the sealing in the sealing bucket.

Method

Air

The method is a modification of the DMA accredited analysis for PCB in air DMA103, Acc. No. 549

The sample is transferred to a glass vial and 5 ml cyclohexane/acetone (1/1) containing extraction standard DBOB is added. The PCB content is extracted by sonication for 2 hours. 900 µL of the extract is mixed with 100 µL injection standard PCB 202 C_{13} and analyzed on a GC-MS in SIM mode

The concentrations of 7 different PCB congeners are calculated from their peak areas. The concentrations are added together and multiplied by a factor of 5 to find the total PCB concentration. The concentration of SPS Trace is determined by its peak area.

Solids

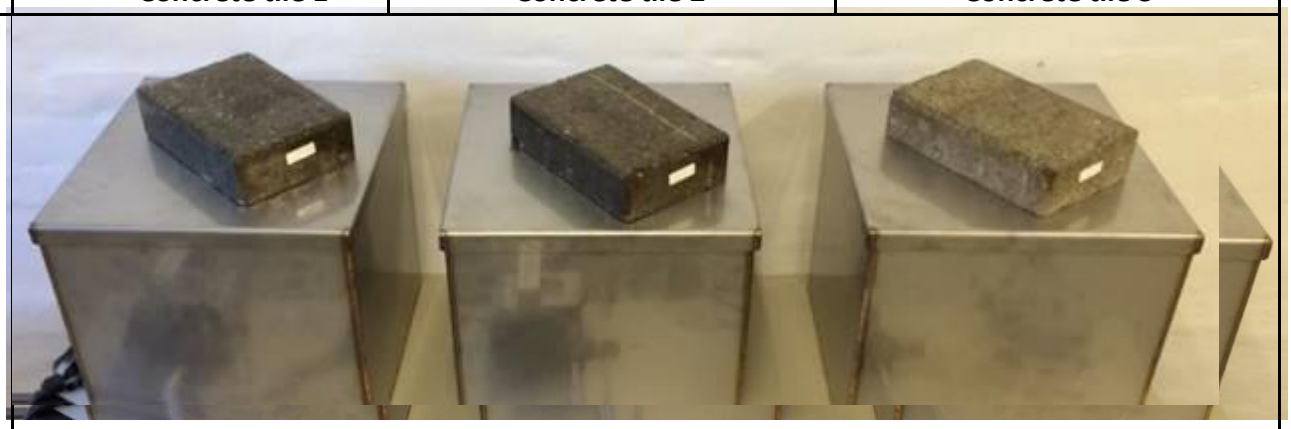
The method is a modification of the DMA accredited analysis for PCB in solids DMA102, Acc. No. 549

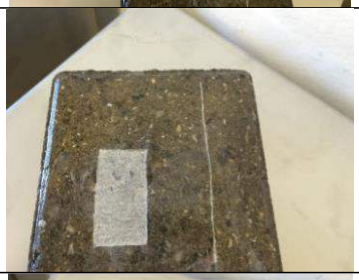
The sample is wiped by an alcohol serviette and the serviette is transferred to a glass vial and 40 ml cyclohexane/acetone (1/1) with extraction standard DBOB is added. The PCB is extracted by sonication for 2 hours. 900 µL of the extract is added 100 µL injection standard PCB 202 C^{13} and is analyzed on a GC-MS in SIM mode

The concentrations of 7 different PCB congeners are calculated from their peak area. The concentrations are added together and multiplied by a factor of 5 to find the total PCB concentration. The concentration of SPS Trace is determined by its peak area.

Experimental setup

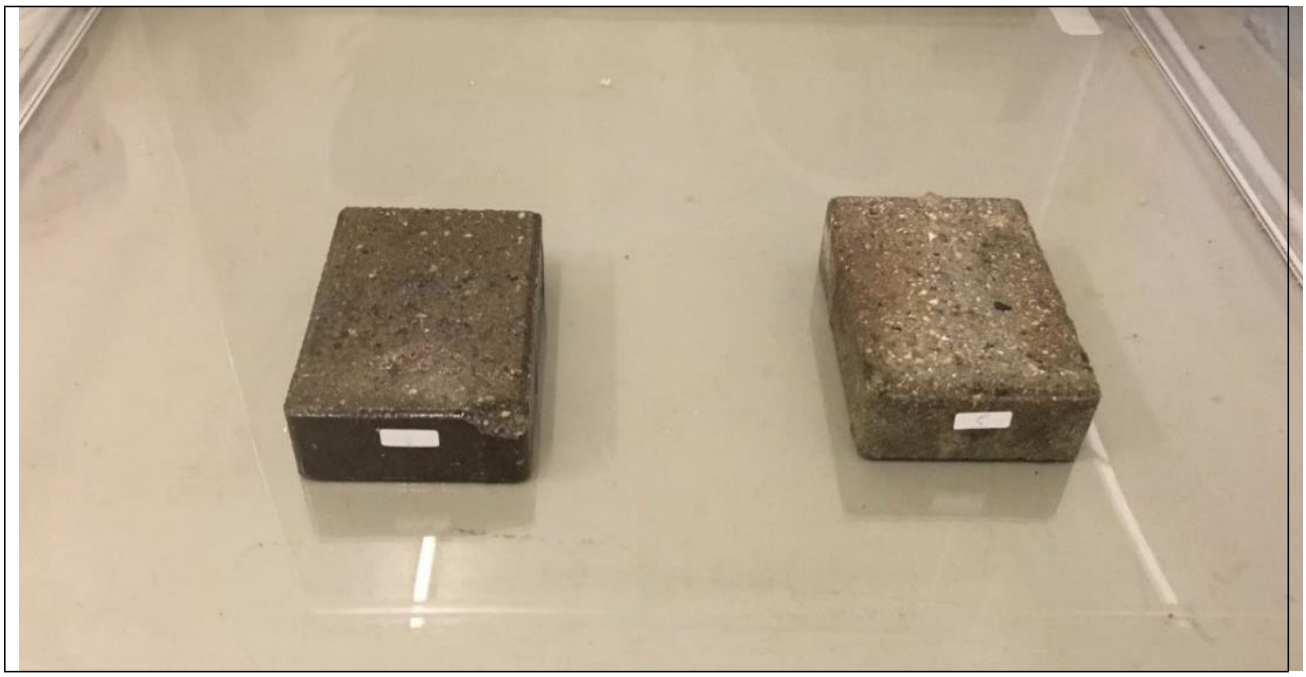
1.
The setup consists of three concrete tiles. Each concrete tile is treated with the same amount of PCB and trace elements. Two of the concrete tiles are then sealed with "SPS Primær" and the last concrete tile is used as a control. One of the sealed concrete tiles is used as a reference, and the sealant on the other sealed concrete tile is gradually removed. See the table below for an overview of the experiment

	Concrete tile 1	**Concrete tile 2**	**Concrete tile 3**
			
	During the experiment the follow steps are made		
Steps	**Concrete tile 1**	**Concrete tile 2**	**Concrete tile 3**
1	Applied SPS Trace, 2 mL PCB (approx. 10.000 ppm), 2mL biphenyl (approx. 1500ppm) and 2mL d-biphenyl (approx. 1300 ppm)	Applied SPS Trace, 2 mL PCB (approx. 10.000 ppm), 2mL biphenyl (approx. 1500ppm) and 2mL d-biphenyl (approx. 1300 ppm)	Applied SPS Trace, 2 mL PCB (approx. 10.000 ppm), 2mL biphenyl (approx. 1500ppm) and 2mL d-biphenyl (approx. 1300 ppm)
2	Sealed with SPS Primær 2 times with a 30min. interval	Sealed with SPS Primær 2 times with a 30min. interval	Not sealed
3	First air sample 3 days after sealing	First air sample 3 days after sealing	First air sample 3 days after sealing of concrete tile 1 and 2
4	Second air sample, 1 hour after first sample	One large scratch in the sealing. Second air sample, 1 hour after first sample	Second air sample, 1 hour after first sample
5	Third air sample, 1 hour after second sample	Square of sealing removed. Third air sample, 1 hour after second sample	Third air sample, 1 hour after second sample
6	Fourth air sample, 1 hour after third sample	Rectangle of sealing removed. Fourth air sample, 1 hour after third sample	Fourth air sample, 1 hour after third sample
7	Fifth air sample, 1 hour after fourth sample	Larger square of sealing removed. Fifth air sample, 1 hour after fourth sample	Fifth air sample, 1 hour after fourth sample
8	Sixth air sample, 18 hours after fifth sample	Sixth air sample, 18 hours after fifth sample	Sixth air sample, 18 hours after fifth sample
9			Seventh air sample, concrete tile heated to 55°C at the beginning and 71°C at the end

	concrete tile 2 (Scrape off)
1. Start	
2. Scratch	
3. Square of sealing removed	
4. Rectangle of sealing removed	
5. Larger square of sealing removed	

2.
The setup for the second test. After the last air samples have been completed, the surfaces of the three concrete tiles are wiped with a piece of cloth which is subsequently extracted and analyzed. Concrete tile 1: Reference fully sealed, Concrete tile 2: Concrete tile with a large square of seal removed, Concrete tile 3: Control concrete tile with applied trace elements and PCB, but without sealing.

3.
The setup for the third test. Two concrete tiles are treated with SPS Trace, 2mL Bisphenol-A (approx. 1500ppm) and 2mL d-Bisphenol-A (approx. 1000 ppm). One of the concrete tiles is then sealed with SPS Primær. After three days the surfaces of the concrete tiles are wiped with a piece of cloth which is subsequently extracted and analyzed.



Results

1.

Results – PCB in air				
Lab no.	**Sample name**	**PCB concentration in ng/m³**	**Air volume, m³**	**Average temp. °C**
1.1	Concrete tile 1, air sample 1	**625**	0,039	20,8
2.1	Concrete tile 1, air sample 2	**264**	0,039	20,8
3.1	Concrete tile 1, air sample 3	**297**	0,039	20,8
4.1	Concrete tile 1, air sample 4	**201**	0,039	20,8
5.1	Concrete tile 1, air sample 5	**280**	0,039	20,8
6.1	Concrete tile 1, air sample 6	**545**	0,039	20,8
1.2	Concrete tile 2, air sample 1	**370**	0,039	20,8
2.2	Concrete tile 2, air sample 2	**404**	0,039	20,8
3.2	Concrete tile 2, air sample 3	**468**	0,039	20,8
4.2	Concrete tile 2, air sample 4	**306**	0,039	20,8
5.2	Concrete tile 2, air sample 5	**495**	0,039	20,8
6.2	Concrete tile 2, air sample 6	**985**	0,039	20,8
1.3	Concrete tile 3, air sample 1	**35500**	0,039	20,8
2.3	Concrete tile 3, air sample 2	**21200**	0,039	20,8
3.3	Sample toppled and had to be disregarded		-	-
4.3	Concrete tile 3, air sample 4	**19000**	0,039	20,8
5.3	Concrete tile 3, air sample 5	**15200**	0,039	20,8
6.3	Concrete tile 3, air sample 6	**8500**	0,039	20,8
7.3	Concrete tile 3, air sample 7, heated concrete tile	**275000**	0,039	55-71°C
Method	DMA103 (Acc. no. 549)			

Notes The concentration is calculated and the result is expressed in ng/m³ based on the air amount. The air amount and the average temperature are not a part of the accreditation.

The Danish Board of Health has the following action values for PCB in indoor air:

- <300 ng/m³ is not considered to cause increased health risk
- 300-3000 ng/m³ demands a plan to lower the level to a concentration below 300 ng/m³
- >3000 ng/m³ estimated to cause a significant health risk. A lasting solution must promptly be sought which can reduce the level.

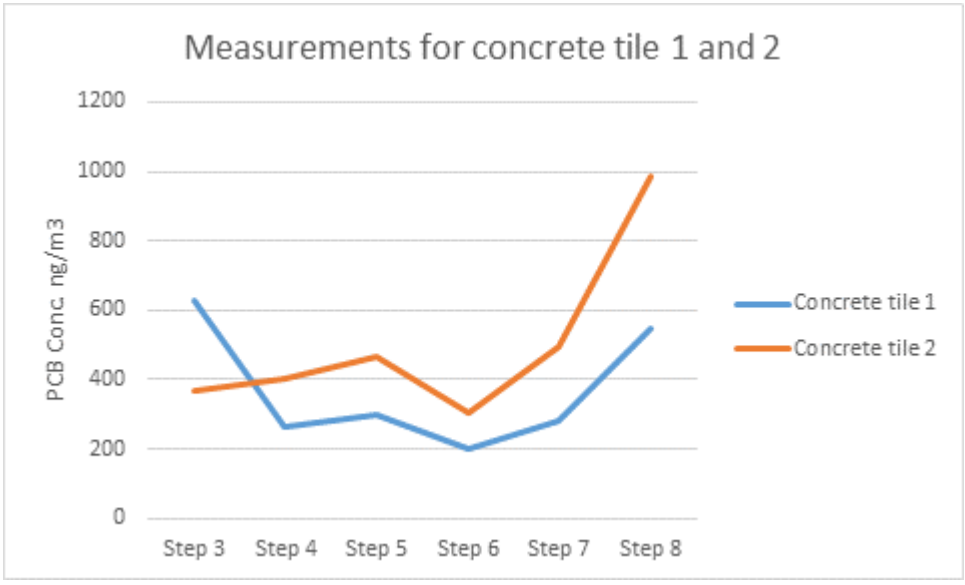
n.d.: Not detected above the detection limit for the single congener

Detailed PCB results

	PCB Congeners (ng/m³)									
Lab no.	28	52	101	118	138	153	180	∑7PCB	Factor	Total PCB content
1.1	125,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	125,0	5,0	625
2.1	52,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	52,7	5,0	264
3.1	59,3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	59,3	5,0	297
4.1	40,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	40,2	5,0	201
5.1	56,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	56,0	5,0	280
6.1	89,1	19,7	n.d.	n.d.	n.d.	n.d.	n.d.	109,0	5,0	545
1.2	74,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	74,0	5,0	370
2.2	80,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	80,7	5,0	404
3.2	70,3	23,3	n.d.	n.d.	n.d.	n.d.	n.d.	93,6	5,0	468
4.2	61,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	61,2	5,0	306
5.2	73,4	25,6	n.d.	n.d.	n.d.	n.d.	n.d.	99,0	5,0	495
6.2	162,0	34,9	n.d.	n.d.	n.d.	n.d.	n.d.	197,0	5,0	985
1.3	6130,0	964,0	n.d.	n.d.	n.d.	n.d.	n.d.	7090,0	5,0	35500
2.3	3540,0	687,0	n.d.	n.d.	n.d.	n.d.	n.d.	4230,0	5,0	21200
3.3										
4.3	3210,0	586,0	n.d.	n.d.	n.d.	n.d.	n.d.	3800,0	5,0	19000
5.3	2490,0	545,0	n.d.	n.d.	n.d.	n.d.	n.d.	3040,0	5,0	15200

6.3	1440,0	264,0	n.d.	n.d.	n.d.	n.d.	n.d.	1700,0	5,0	8500
7.3	46600,0	7860,0	280,0	138,0	n.d.	n.d.	n.d.	54900,0	5,0	275000

Notes: The sum of detected concentrations (areas under curve) of the seven congeners is multiplied by a factor of 5 in order to calculate the total PCB concentration (in accordance with the Danish guideline "Energistyrelsen 2010: vejledning for måling af PCB i indeklimaet"). Detection limits for the single congener: 10 ng/m³
The expanded uncertainty for the single congener is 20 %
For concentrations, close to the detection limit the expanded uncertainty may be up to 30 %


Measurements for concrete tile 1 and 2
1200
1000
800
600
400
200
0
PCB Conc. ng/m3
Step 3
Step 4
Step 5
Step 6
Step 7
Step 8
Concrete tile 1
Concrete tile 2

Results – Biphenyl, Biphenyl-d and SPS Trace in air						
Lab no.	**Sample name**	**Biphenyl conc. in ng/m³**	**Biphenyl-d conc. in ng/m³**	**SPS Trace conc. in ng/m³**	**Air volume, m³**	**Average temp. °C**
1.1	Concrete tile 1, air sample 1	**2310**	**1470**	**I.D.**	0,039	20,8
2.1	Concrete tile 1, air sample 2	**2590**	**746**	**I.D.**	0,039	20,8
3.1	Concrete tile 1, air sample 3	**1400**	**615**	**I.D.**	0,039	20,8
4.1	Concrete tile 1, air sample 4	**1620**	**805**	**I.D.**	0,039	20,8
5.1	Concrete tile 1, air sample 5	**1790**	**866**	**I.D.**	0,039	20,8
6.1	Concrete tile 1, air sample 6	**1140**	**309**	**I.D.**	0,039	20,8
1.2	Concrete tile 2, air sample 1	**3610**	**2520**	**I.D.**	0,039	20,8
2.2	Concrete tile 2, air sample 2	**4500**	**2370**	**I.D.**	0,039	20,8
3.2	Concrete tile 2, air sample 3	**4080**	**3520**	**I.D.**	0,039	20,8
4.2	Concrete tile 2, air sample 4	**4810**	**4580**	**I.D.**	0,039	20,8
5.2	Concrete tile 2, air sample 5	**3490**	**4870**	**I.D.**	0,039	20,8
6.2	Concrete tile 2, air sample 6	**9100**	**13900**	**I.D.**	0,039	20,8
1.3	Concrete tile 3, air sample 1	**633000**	**634000**	**I.D.**	0,039	20,8
2.3	Concrete tile 3, air sample 2	**455000**	**408000**	**I.D.**	0,039	20,8
3.3	Analytical sample toppled and had to be disregarded				0,039	20,8
4.3	Concrete tile 3, air sample 4	**275000**	**229000**	**I.D.**	0,039	20,8
5.3	Concrete tile 3, air sample 5	**195000**	**164000**	**I.D.**	0,039	20,8
6.3	Concrete tile 3, air sample 6	**36900**	**30600**	**I.D.**	0,039	20,8
7.3	Concrete tile 3, air sample 7, heated concrete tile	**56600**	**46600**	**121**	0,039	55-71°C
Method	DMA103 (modified)					

2.

Result – PCB in solids			
Lab no.	**Sample name**	**PCB concentration in ng/m³**	**Comments**
1	Concrete tile 1	**n.d.**	14X21cm
2	Concrete tile 2	**1070**	14X21cm, area of seal removed
3	Concrete tile 3	**13700**	14X21cm
Method	DMA102 (Acc. no. 549)		

Detailed PCB results

	PCB Kongener (µg/m²)									
Lab nr.	28	52	101	118	138	153	180	∑7PCB	Faktor	Total PCB indhold
1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-	5,0	n.d.
2	160,0	44,7	3,910	3,990	n.d.	n.d.	n.d.	213,0	5,0	1070
3	2010	585,0	58,9	77,20	n.d.	n.d.	n.d.	2730	5,0	13700

Notes
A factor of 5 is used to calculate the total PCB concentration according with MST-7543-00007, 5. July 2011
Detection limits for the single congener: 2,0 µg/m²
The expanded uncertainty for the single congener is 25 %
For concentrations close to the detection limit the expanded uncertainty may be up to 35 %

Results – Biphenyl, Biphenyl-d and SPS trace in solids					
Lab no.	**Sample name**	**Biphenyl concentration in ug/m²**	**Biphenyl-d concentration in ug/m²**	**SPS Trace concentration in ug/m²**	**Comments**
1	Concrete tile 1	**n.d.**	**n.d.**	**n.d.**	14X21cm
2	Concrete tile 2	**342**	**161**	**8,9**	14X21cm, area of seal removed
3	Concrete tile 3	**556**	**664**	**159**	14X21cm
Method	DMA102 (Modified)				

3.

Results – Bisphenol-A, Bisphenol-A-d and SPS trace in solids					
Lab no.	**Sample name**	**Bisphenol-A concentration in µg/m²**	**Bisphenol-A-d concentration in µg/m²**	**SPS trace concentration in µg/m²**	**Comments**
1	Sealed concrete	**n.d.**	**n.d.**	**n.d.**	14X21cm
2	Not sealed	**6000**	**5339**	**29**	14X21cm
Method	DMA102 (Modified)				

2017-01-04

Anders Jensen

Responsibility: For samples submitted for analysis DMA is only responsible for the laboratory analysis of the sample. This means that DMA is not responsible for the sampling. i.e. whether the sample represents the specific material that has been sampled or whether the number of samples is sufficient to draw any conclusions about the type of material in the sample area.

DMA is not responsible for any practical actions performed on the site by the recipient of the analytical results as a consequence of the results reported herein.

Part 2

Assignment

Dansk MiljøAnalyse has been given the assignment to test the tracer compound "SPS Trace", i.e. 4,4´-2,2,2-trifluoroethoxy biphenyl in an experimental setup where the containment of PCB by the two-component silicate coating "SPS Primær" (delivered by TWO Teknik ApS) is tested.
The test relates to evaporation of PCB and SPS Trace from concrete tiles partially coated with these compounds, and specifically to how the evaporation of the compounds is affected by a coating of "SPS Primær" sealing. Furthermore, the test analyzes the effect of partially breaking/removing the SPS Primær sealing.

Chemicals

Aroclor 1248 solution (certified reference material, 1000 µg/mL in isooctane 44807, Supelco, Sigma Aldrich), "SPS Trace", i.e. 4,4´-2,2,2-trifluoroethoxy biphenyl), Dutch Seven PCB Mixture (NEN 5734/VPR C85-16, LGC Standards), PCB mix (certified reference material bcr – 365, LGC Standards), DBOB 4,4´-Dibromooctafluorobiphenyl (Sigma Aldrich), PCB202 2,2',3,3',5,5',6,6'-Octachlorobiphenyl (13C12,99%, LGC Standards) SPS Primær + curing agent (delivered by TWO Teknik ApS), cyclohexane and acetone.
Sources of error
A main source of error during the test is the use of "free" PCB (PCB in solution not bounded). When adding a PCB in a solution to the concrete and afterward sealing the concrete, some of the PCB may be dissolved in the sealing layer which might explain the background level of PCB in the air sample. Another consequence hereby is the possible pollution of sealing on the brush which can contaminate the sealing in the sealing bucket.

Method

The method is a modification of the DMA accredited analysis for PCB in air DMA103, Acc. No. 549
The sample is transferred to a glass vial and 5 ml cyclohexane/acetone (1/1) with extraction standard DBOB is added. The PCB is extracted by sonication for 2 hours. 900 µL of the extract is added 100 µL injection standard PCB 202 C^{13} and is analyzed on a GC-MS in SIM mode.
The concentrations of 7 different PCB congeners are calculated from their peak area. The concentrations are added together and multiplied by a factor of 5 to find the total PCB concentration. The concentration of SPS Trace is determined by its peak area.

Experimental setup

The test consists of 6 concrete tiles. Each concrete tile was treated with the following chemicals: 1,1mL Aroclor 1000ppm solution and 2mL solution of SPS Trace (13,2mg diluted in 12mL 50/50 cyclohexane/acetone). Five of the concrete tiles were then sealed with SPS Primær (SPS Primær + curing agent mixed together for 10 min) over two rounds with an interval of 30 min. The sealing was allowed to cure for 24 hours. The concrete tiles were subsequently treated as shown in the table below.

Table 1, Experimental setup of concrete tiles with different amount of sealing

Concrete tile 1, sealed 100 %	Concrete tile 2, sealed with a scratch	Concrete tile 3, sealed with 25 % scrapings
		
Concrete tile 4, sealed with 50 % scrapings	**Concrete tile 5, sealed with 75 % scrapings**	**Concrete tile 6, not sealed, reference**
		

Each concrete tile is placed in a stainless steel box 30x30x30cm (wipe cleaned with acetone before use). The boxes are sealed with gaffer tape for 30 min. Subsequently an air sample is taken in each box. The samples are collected on a sampling tube containing PUF/XAD-2/PUF- Sandwich sorbent material with a flow of 2 L/min for 2 hours. A typical box can be seen in the picture below. Finally, the sorbent material is analyzed as described in the methods section.

Picture 1: Stainless steel box sealed with gaffer tape and equipped with a sorbent tube

Results

Results – PCB and SPS Trace in air					
Lab no.	**Sample name**	**PCB concentration in ng/m³**	**SPS Trace concentration in ng/m³**	**Air volume, m³**	**Average temp. °C**
1	Concrete tile 1, 100 %	**640**	**n.d.**	0,12	21,3
2	Concrete tile 2, sealed with a scratch	**710**	**n.d.**	0,12	21,3
3	Concrete tile 3, sealed with 25 % scrapings	**720**	**13**	0,12	21,3
4	Concrete tile 4, sealed with 50 % scrapings	**775**	**17**	0,12	21,3
5	Concrete tile 5, sealed with 75 % scrapings	**3910**	**118**	0,12	21,3
6	Concrete tile 6, not sealed, reference	**8950**	**219**	0,12	21,3
Method	DMA103 (Acc. no. 549)				

Notes The concentration is calculated and the result is expressed in ng/m³ based on the air amount. The air amount and the average temperature are not a part of the accreditation.

The Danish board of health has the following action values for PCB in indoor air.:

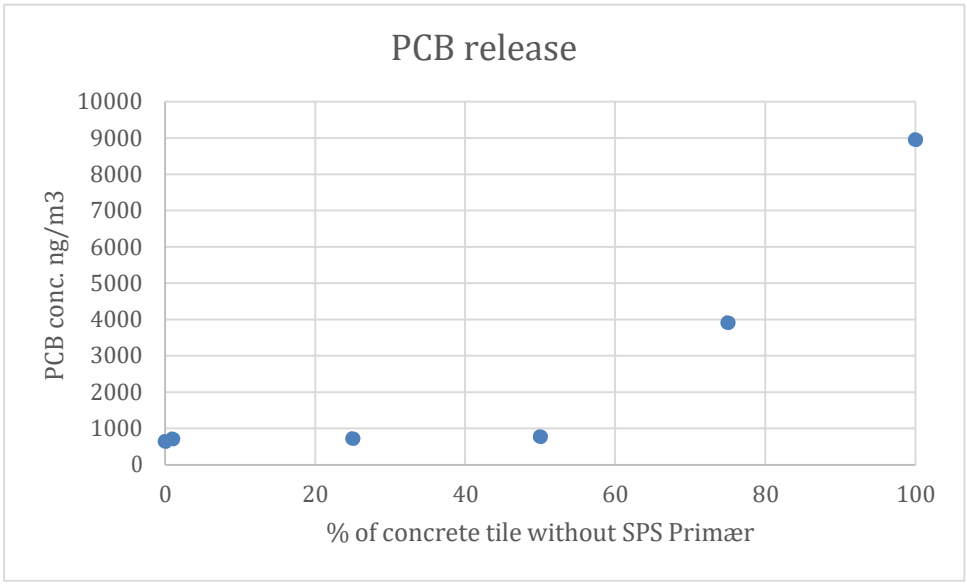
- <300 ng/m³ is not considered to cause increased health risk
- 300-3000 ng/m³ demands a plan to lower the level to a concentration below 300 ng/m³
- >3000 ng/m³ estimated to cause a significant health risk. A lasting solution must promptly be sought which can reduce the level.

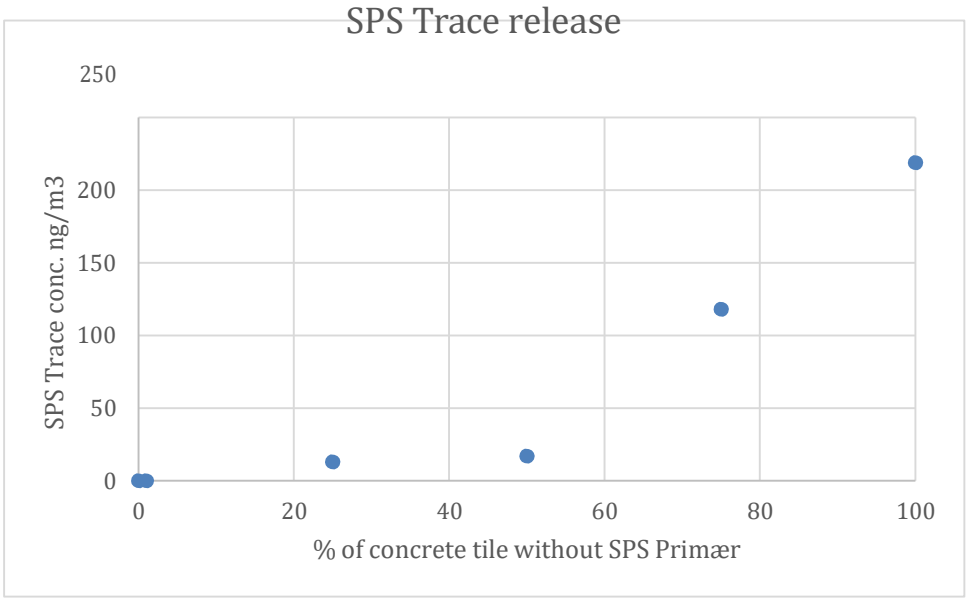
n.d.: Not detected above the detection limit for the single congener

Detailed PCB results

	PCB Congeners (ng/m³)									
Lab no.	28	52	101	118	138	153	180	∑7PCB	Faktor	Total PCB indhold
1	90,0	38,0	n.d.	n.d.	n.d.	n.d.	n.d.	128,0	5,0	640
2	101,0	41,0	n.d.	n.d.	n.d.	n.d.	n.d.	142,0	5,0	710
3	106,0	38,3	n.d.	n.d.	n.d.	n.d.	n.d.	144,0	5,0	720
4	113,0	41,7	n.d.	n.d.	n.d.	n.d.	n.d.	155,0	5,0	775
5	383,0	333,0	65,3	n.d.	n.d.	n.d.	n.d.	781,0	5,0	3910
6	775,0	638,0	138,0	178,0	33,2	26,3	n.d.	1790,0	5,0	8950

Notes
The sum of detected concentrations (areas under curve) of the seven congeners is multiplied by a factor of 5 in order to calculate the total PCB concentration (in accordance with the Danish guideline "Energistyrelsen 2010: vejledning for måling af PCB i indeklimaet").
Detection limits for the single congener: 10 ng/m³
The expanded uncertainty for the single congener is 20 %





2017-01-04

Anders Jensen

Responsibility: For samples submitted for analysis DMA is only responsible for the laboratory analysis of the sample. This means that DMA is not responsible for the sampling. i.e. whether the sample represents the specific material that has been sampled or whether the number of samples is sufficient to draw any conclusions about the type of material in the sample area.

DMA is not responsible for any practical actions performed on the site by the recipient of the analytical results as a consequence of the results reported herein.