Uptake Rate Tests

Tests for a range of compounds onto four sorbent types over periods of 1 and 2 weeks

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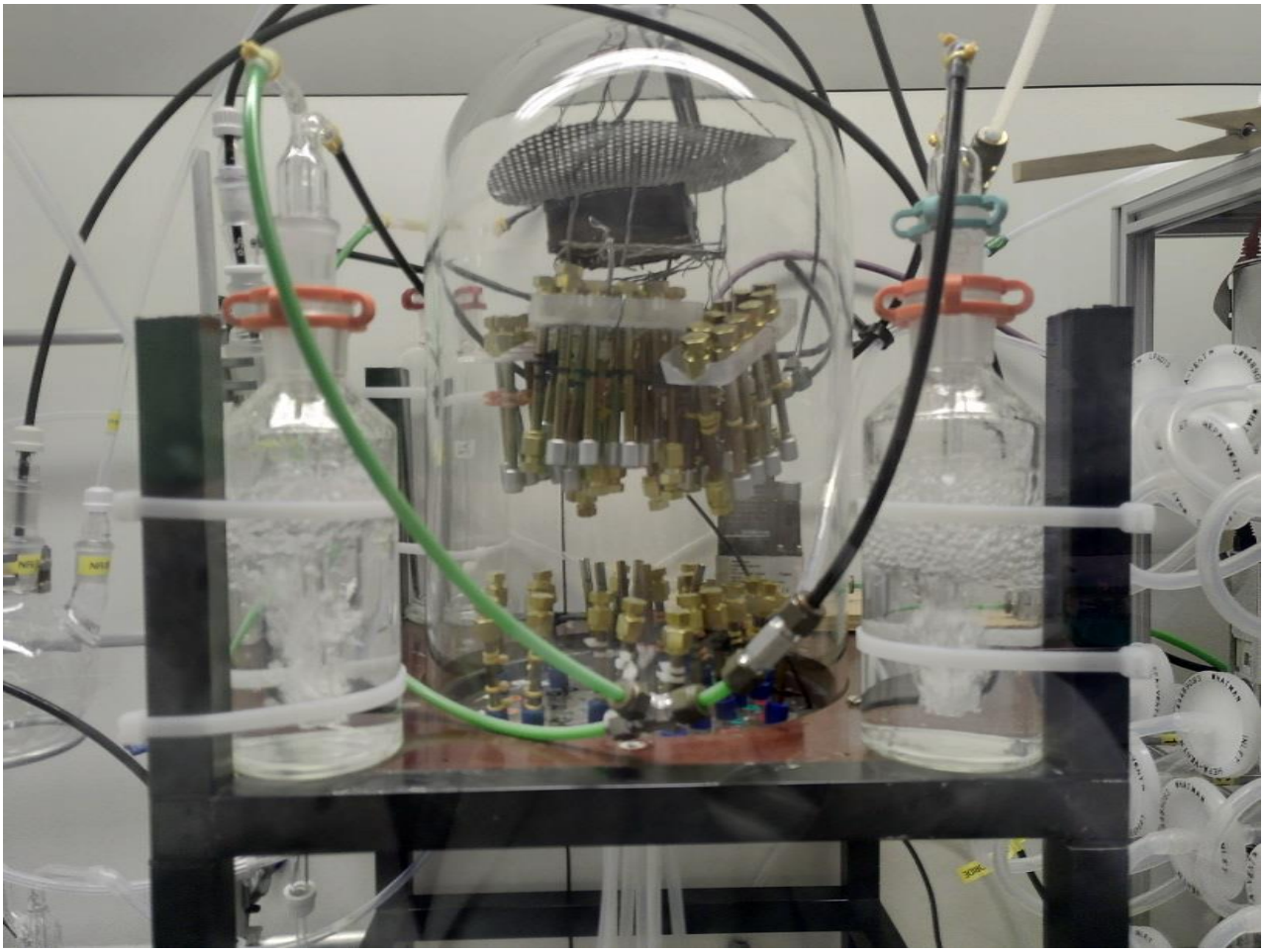

UPTAKE RATE TESTS

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Contents

Introduction

Markes International commissioned the Science Division (SD) of the Health and Safety Executive (HSE) to undertake testing of thermal desorption type passive tube samplers containing four different sorbents. This work is described in project proposal HSE-SD Uptake Rate Tests PE08609. The objective of this study was to enable the determination of diffusive uptake rates for a range of analytes on these samplers by exposing them to low levels of chemical vapours under controlled conditions.

Before commencing the uptake rate tests, HSE generated a vapour comprising a mixture of 21 volatile organic compounds (VOCs) in air using bespoke standard atmosphere generation equipment and undertook a 10 litre breakthrough test on the two sorbents to be used for active sampling. This showed that the proposed test conditions were suitable for all of the components, with the exception of dichloromethane. After consultation with the Client, it was therefore agreed to remove this compound from the test mix and replace it with n-hexane.

HSE then proceeded to expose sets of samplers to an atmosphere of the 21 compounds each at a nominal target concentration of 5 parts per billion (ppb). For these tests exposure times of 1 and 2 weeks were used. In addition, HSE collected samplers using pumped sampling for approximately 24 hour periods and accurately recorded collected sample volumes. At the end of the test the diffusive and pumped samplers were collected, sealed and returned to the Client for analysis. On supply of GC/MS data HSE calculated concentrations of the target compounds in the chamber over the exposure period and uptake rates for these compounds over the 1 and 2 week periods.

Methods

Samplers

Markes International supplied the samplers required for the diffusive sampling (Carbograph 1TD [C1TD], Carbopack B [CB], Carbograph 5TD [C5TD] and Carbopack X [CX]) and those required for the active sampling (C5TD and CX Safelok tubes). These samplers were all freshly packed and had been pre-conditioned. On receipt at HSE the tubes were conditioned at 350°C for a total time of 1 hour using a Markes TC-20 and a nitrogen flow of 80 $ml.min^{-1}$. Three of each sorbent type were analysed before the tests and no presence of any contamination was observed.

Analytes

The agreed components to be used for testing are shown in Table 1**.**

Standard Atmosphere

The HSE standard atmosphere generation facility is based upon procedures described in ISO 6145-4:2004. This ISO standard specifies a method for the continuous production of stable gas mixtures, containing two or more components, generated from the vaporisation of liquid mixes via the continuous injection of such components into a gas stream by means of a syringe. In addition, gaseous components can be introduced into the exposure chamber from pressurised cylinders or permeation tubes at a known dilution. Loading facilities of this type have been routinely used by HSE to prepare proficiency testing samples over the last 20 years and to undertake a range of studies of breakthrough volumes and uptake rate tests.

The standard atmosphere facility used for these tests consisted of the following components:

- A motorised syringe to inject the liquid test mixture into a heated (50°C) glass chamber.
- A GasTec PD-1B gas generation system to generate 1,3-butadiene from sealed permeation tubes.
- Vapour mixing chambers for air dilution. A proportion of the diluent air stream was humidified.
- A 20 litre glass exposure chamber to expose the sorbent tubes.
- A Signal HM 3000 on-line flame ionisation detector (FID), to monitor the vapour concentration.
- A Vaisala HMP 273 calibrated temperature and humidity logger.

- An Eluteng fan, to control the air speed through the exposure chamber
- Mass flow controllers (MFCs), to control the flow of air used to vaporise the solvent mixture, the diluent air, the air flow to the FID and to collect active samples
- In-house proprietary software, written in LabVIEW.

Table 1: Components to be used for Testing

Compound	Phase	CAS No.	Boiling point (°C)
1,3-Butadiene	Gas	106-99-0	-4
1,1-Dichloroethene	Liquid	75-35-4	32
n-Pentane	Liquid	109-66-0	36
Dichloromethane [1]	Liquid	75-09-2	40
Chloroform	Liquid	67-66-3	61
n-Hexane [2]	Liquid	110-54-3	69
1,1,1-Trichloroethane	Liquid	71-55-6	74
Acrylonitrile	Liquid	107-13-1	77
Ethyl acetate	Liquid	141-78-6	77
Carbon tetrachloride	Liquid	56-23-5	77
Benzene	Liquid	71-43-2	80
Propan-2-ol	Liquid	67-63-0	82
1,2-Dichloroethane	Liquid	107-06-2	83
Trichloroethene	Liquid	79-01-6	87
n-Heptane	Liquid	142-82-5	98
Methyl methacrylate	Liquid	80-62-6	101
Toluene	Liquid	108-88-3	111
Tetrachloroethene	Liquid	127-18-4	121
2-Methoxyethanol	Liquid	109-86-4	124
n-Octane	Liquid	111-65-9	125
Ethylbenzene	Liquid	100-41-4	136
p-Xylene	Liquid	106-42-3	138

[1] Removed from list following breakthrough test; [2] Added to list following breakthrough test

Breakthrough Test

A mixture of the components of interest was prepared to give airborne concentrations in the atmosphere of between 25 and 120 ppb depending on FID response. A higher concentration was selected than was used for the uptake rate test to ensure that breakthrough levels of ≥ 1% onto the secondary tube would be detectable. Pairs of tubes containing either C5TD or CX sorbent were set up in series connected with a brass Swagelock union. Six replicate sets of each sorbent were used. The tubes were placed into the exposure chamber and each connected to an MFC set to give a flow of 19 $ml.min^{-1}$ ± 0.4%. Active sampling was undertaken for 7 hours to give a sampling volume of 8 litres. Before analysis the breakthrough tubes were purged with dry nitrogen, in the sampling direction, for 27 minutes at a flow of 75 $ml.min^{-1}$ using a Markes International TC20 (sampling volume of 2 litres to give a total volume of 10 litres).

Uptake Rate Test

Chamber set up

The test mixture of the liquid analytes was prepared as shown in Table 2. The VOC atmosphere was generated by injection of the mixture of liquid analytes, at a flow rate of 0.3 $\mu l.min^{-1}$, into a flow of clean air. The air flow was diluted as it passed through the mixing chambers and directed into the exposure chamber. The 1,3-butadiene was introduced into the chamber at a flow rate of 500 $ml.min^{-1}$ from the Gastec gas generator. Test samples were taken to check that concentrations of components in the exposure chamber were in the range requested by the Client (nominally 5 ppb). The atmosphere was allowed to equilibrate overnight before commencing the test.

The air speed in the exposure chamber was checked before the start of the test using a calibrated air velocity meter (TSI TA430) and was found to be approximately 0.2 $m.s^{-1}$. Variations in concentration level were recorded at intervals of 10 seconds during the test using on-line FID. Relative humidity and temperature were also monitored every 10 seconds.

Diffusive Samplers

Sets of sampling tubes of sorbent types C1TD, CB, C5TD and CX were exposed for one nominal 168 hour and one 336 hour period, with the exact exposure periods, in minutes, being recorded. Samplers were hung vertically with the diffusion heads at the lowest position. Each set of samplers comprised 8 samplers fitted with standard diffusion heads plus 2 blanks placed in the exposure chamber next to the samples. A further 3 blanks of each sorbent type were located in the base plate of the exposure chamber. Samplers were placed in the exposure chamber as indicated in Table 3. Full details of samplers exposed are given in Tables A1 to A8.

Table 2: Details of Liquid Mixture used in Syringe to Generate VOC atmosphere

Compound	Molecular weight	Density (g/ml)	Volume added (ml)	Expected composition (%w/w)
1,1-Dichloroethene	96.9	1.21	0.56	4.72
n-Pentane	72.1	0.63	0.81	3.49
Chloroform	119.4	1.50	0.56	5.78
n-Hexane	86.2	0.65	0.93	4.25
1,1,1-Trichloroethane	133.4	1.32	0.71	6.64
Acrylonitrile	53.1	0.81	0.46	2.62
Ethyl acetate	88.1	0.90	0.69	4.29
Carbon tetrachloride	153.8	1.59	0.68	7.60
Benzene	78.1	0.88	0.63	3.84
Propan-2-ol	60.1	0.79	0.54	2.95
1,2-Dichloroethane	98.9	1.25	0.56	4.90
Trichloroethene	131.4	1.46	0.64	6.50
n-Heptane	100.2	0.68	1.04	4.94
Methyl methacrylate	100.1	0.94	0.75	4.95
Toluene	92.1	0.87	0.75	4.52
Tetrachloroethene	165.8	1.62	0.72	8.16
2-Methoxyethanol	76.1	0.97	0.56	3.77
n-Octane	114.2	0.70	1.15	5.62
Ethylbenzene	106.2	0.87	0.87	5.28
p-Xylene	106.2	0.86	0.87	5.19

Table 3: Introduction of Samplers to the Chamber

Days 1-7	Days 8-14
8 x 336 hr Set – C1TD (plus 5 blanks) 8 x 336 hr Set – CB (plus 5 blanks) 8 x 336 hr Set – C5TD (plus 5 blanks) 8 x 336 hr Set – CX (plus 5 blanks)	
8 x 168 hr Set – C1TD (plus 5 blanks) 8 x 168 hr Set – CB (plus 5 blanks)	
	8 x 168 hr Set – C5TD (plus 5 blanks) 8 x 168 hr Set – CX (plus 5 blanks)

Active Samplers

Fourteen sets of active samples were taken over the 14 day sampling period, using Safelock sample tubes and collecting one active sample on sorbent 5TD and one on sorbent CX per set. Samples were collected for periods of approximately 24 hours, with the time being recorded to the nearest minute. The MFCs used for active sampling were calibrated prior to the test using a calibrated flowmeter (Gilian Gilbrator 3) to give a flow rate of 5 $ml.min^{-1}$ ± 1%. The flows were checked with the flowmeter before and after collection of each active sample. Exposed tubes were stored at room temperature until returned to Markes for analysis at the end of the 2-week diffusive exposure period. Sampling details are given in Table A9.

Preliminary Analysis at HSE

Analysis of breakthrough tubes and test samplers for checking atmosphere concentration was undertaken at HSE using thermal desorption with gas chromatography and flame ionisation (GC/FID). Thermal desorption and GC parameters used are given below:

TD: Graphitised carbons: 330°C for 8 min, Tenax/GR: 280°C for 8 min, no inlet split, desorb flow 100 $ml.min^{-1}$, outlet split 15 $ml.min^{-1}$, purge 1 min, trap hold 2 min, cold trap Tenax/Unicarb at -30°C to 280°C.

GC: Column flow: 0.8 $ml.min^{-1}$, column: 50 m BP1 x 0.22 mm ID x 1 µm film thickness, temperature program: 35°C for 13 min, 5°C/min to 80°C, 10°C/min to 130°C, 20°C/min to 250°C, hold for 5 min.

Calibration of the analytical system for the 20 liquid components was undertaken using methanolic solutions. A multi-point calibration covering the range of expected analyte masses for the breakthrough tubes and test samples was obtained by analysis of tubes containing conditioned Tenax GR sorbent spiked with 1 µl or 3 µl of the relevant standard solutions. Standard tubes were purged with clean nitrogen for 6 minutes at a flow rate of 25 ml.min^{-1}. Calibration of 1,3-butadiene was undertaken by spiking controlled volumes of a reference gas containing 5 ppm of 1,3-butadiene in nitrogen onto tubes containing conditioned CX sorbent using gas-tight syringes. Tubes were spiked in a flow of nitrogen at a flow rate of approximately 100 ml.min^{-1} and purged for 1 minute. Further sets of standard tubes were prepared to supply to Markes for analysis of the samplers for the uptake rate test. Loadings for the standard tubes are given in Tables A11 to A12.

Main Test Analysis at Markes

This section is based on information provided by Markes. The analysis carried out at Markes used a Markes TD100-xr automated thermal desorption system coupled to a GC-MS. An internal standard (D8-toluene) was automatically added to the sampling end of the sorbent tube or focusing trap (for the system blank) from a gas standard as part of each analytical run. Background checks were carried out first (including system and sorbent tube blanks). Example blank chromatograms are shown in Figure A1.

Samples were automatically and quantitatively re-collected during analysis to allow test runs to be repeated if required. In order to accommodate the different sorbent used, it was necessary to vary the tube desorption temperature during the sequence so this parameter of the thermal desorption method was ‘unlocked’. ‘Unlocking’ is a convenient facility for method development as it allows parameters to be modified in the automation sequence table rather than creating two or more analytical methods.

TD: Flow path temperature: 150°C. Pre-desorption: Pre-purge time 1 minute; Trap in line with 50 ml.min^{-1} flow. Internal standard: D8-toluene (10ppm); injection volume 1 ml. Tube desorption: Desorb time 8 minutes; desorb temperature 280°C (Tenax GR) or 330°C (Graphitised carbon); Trap in line 100 ml.min^{-1} flow. Trap desorption: Tenax TA and Sulficarb trap; Purge time 1 minute (50 ml.min^{-1} flow); Trap temperature -30°C to 280°C, trap desorb time 7 minutes; Trap split flow 19 ml.min^{-1}.

GC: 60 m x 0.25 mm x 1 µm DB1 column; Column flow 1 ml.min^{-1}; constant flow. Temperature program: Initial temperature 35° for 13 minutes; 3°C/min to 80°C, 10°C/min to 110°C, 5°C/min to 130°C, 20°C/min to 200°C. MS Acquisition mode: Scan; Mass Range 30 - 300; MSD transfer line temperature 290°C; MS source temperature 230°C; MS quad temperature 150°C.

Example chromatograms for diffusive samples collected over one and two weeks are shown in Figures A2 and A3 respectively.

Results

Breakthrough Test

The mean percentage carryover found for the 21 analytes on the two sorbents is given in Table 4. Under the test conditions, dichloromethane was found to show significant breakthrough on both sorbents. After consultation with the Client it was therefore agreed not to include this compound in the uptake rate study and to replace in with n-hexane. The observation that n-pentane does not show any breakthrough under the test conditions provided sufficient confidence that this would also be the case for n-hexane. Acrylonitrile was found to break through using a sampling volume of 10 litres on C5TD, but not significantly on CX, suggesting that use of values from the CX active samples may be preferable for this compound in the uptake rate test. No other compounds were found to break through significantly (>5%) on either sorbent. The recoveries and repeatability obtained for each compound on the two sets of primary samplers can also be used to indicate which might be the preferred sorbent from which to take concentration values from during the uptake rate test. The results from the C5TD and CX active samples are given in Table 5.

Table 4: Mean % Breakthrough found using 10 litre Sampling Volume

Compound	% Carryover using C5TD	% Carryover using CX
1,3-Butadiene	<1.0%	**1.8%**
1,1-Dichloroethene	<1.0%	<1.0%
n-Pentane	<1.0%	<1.0%
Dichloromethane	**71.5%**	**48.5%**
Chloroform	<1.0%	**1.0%**
1,1,1-Trichloroethane	<1.0%	<1.0%
Acrylonitrile	**34.5%**	**1.5%**
Ethyl acetate	<1.0%	<1.0%
Carbon tetrachloride	<1.0%	<1.0%
Benzene	<1.0%	<1.0%
Propan-2-ol	<1.0%	<1.0%
1,2-Dichloroethane	<1.0%	<1.0%
Trichloroethene	<1.0%	<1.0%
n-Heptane	<1.0%	<1.0%
Methyl methacrylate	<1.0%	<1.0%
Toluene	<1.0%	<1.0%
Tetrachloroethene	<1.0%	<1.0%
2-Methoxyethanol	<1.0%	<1.0%
n-Octane	<1.0%	<1.0%
Ethylbenzene	<1.0%	<1.0%
p-Xylene	<1.0%	<1.0%

Table 5: Comparison of Chamber Concentrations (ppb) Determined during breakthrough test using C5TD and CX

Compound	C5TD (n = 6)		CX (n = 6)		Preferred sorbent for active samples
	Mean Concentration (ppb)	%RSD	Mean Concentration (ppb)	%RSD	
1,3-Butadiene	56.4	7.9%	52.2	7.5%	Either
1,1-Dichloroethene	68.2	1.9%	71.7	3.4%	Either
n-Pentane	38.8	1.7%	38.2	3.2%	Either
Dichloromethane	21.8	3.7%	46.6	8.7%	Neither
Chloroform	102	2.3%	26.8	66.4%	C5TD
1,1,1-Trichloroethane	113	1.8%	104	7.7%	C5TD
Acrylonitrile	20.3	2.8%	27.5	3.8%	CX
Ethyl acetate	68.1	1.9%	43.5	22.3%	C5TD
Carbon tetrachloride	112	2.1%	74.1	44.9%	C5TD
Benzene	35.2	1.7%	34.1	3.8%	Either
Propan-2-ol	73.7	1.8%	60.6	5.0%	C5TD
1,2-Dichloroethane	73.6	1.9%	67.2	6.4%	C5TD
Trichloroethene	108	1.8%	83.8	23.8%	C5TD
n-Heptane	36.3	1.9%	34.9	4.2%	Either
Methyl methacrylate	34.7	1.9%	24.3	29.2%	C5TD
Toluene	36.9	2.6%	35.5	3.9%	Either
Tetrachloroethene	108	2.2%	107	4.4%	Either
2-Methoxyethanol	122	2.0%	72.4	39.7%	C5TD
n-Octane	37.0	2.0%	35.0	5.4%	Either
Ethylbenzene	37.0	1.8%	35.3	4.6%	Either
p-Xylene	36.9	1.9%	34.7	5.5%	Either

Real Time Concentration Data

Graphical plots of on-line FID data from the standard atmosphere rig are given in Figures 1 to 3 and show the sum concentration of VOC as methane equivalent (the calibration gas). The resultant mean concentrations and relative standard deviations (%RSD) are given in

Table 6. This shows that, while the level fluctuates on a short term basis, the average concentration remained fairly stable across the two week period. Note that for environmental concentrations the FID samples from a point prior to the final dilution of the air flow.

Table 6: Mean FID response for each Sample Exposure Period

Time Period (hrs)	Real Time FID		
	Mean Concentration (ppm)	%RSD (all data)	%RSD (daily means)
0-168	46.4	17.1	0.7
168-336	45.9	15.1	1.7
0-336	46.1	16.2	1.4

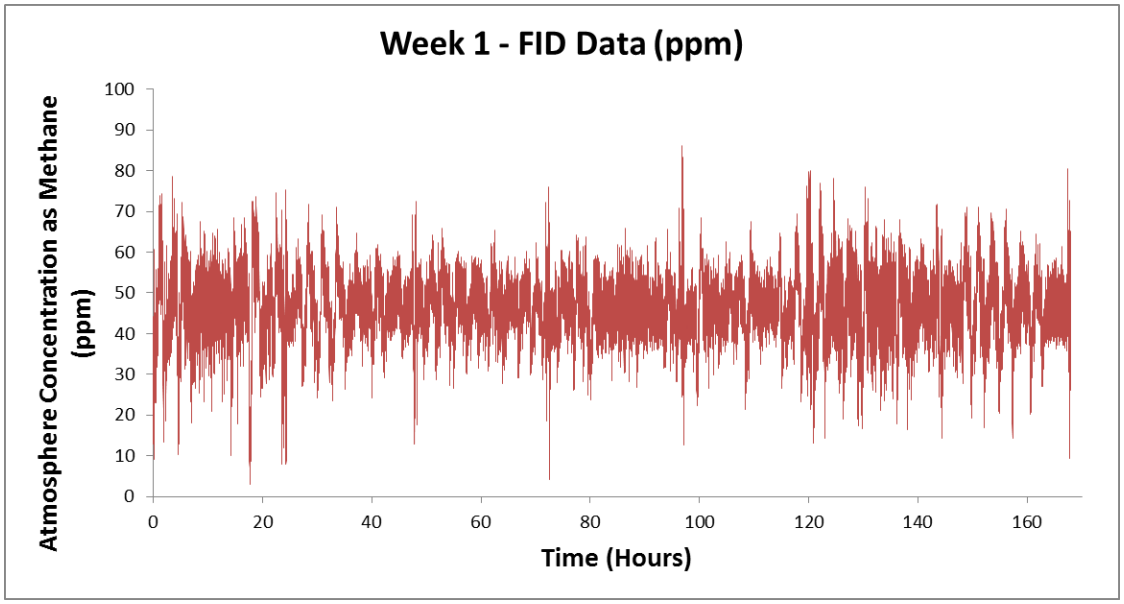

Figure 1: FID Data – 0 to 168 hours of the 14 day test

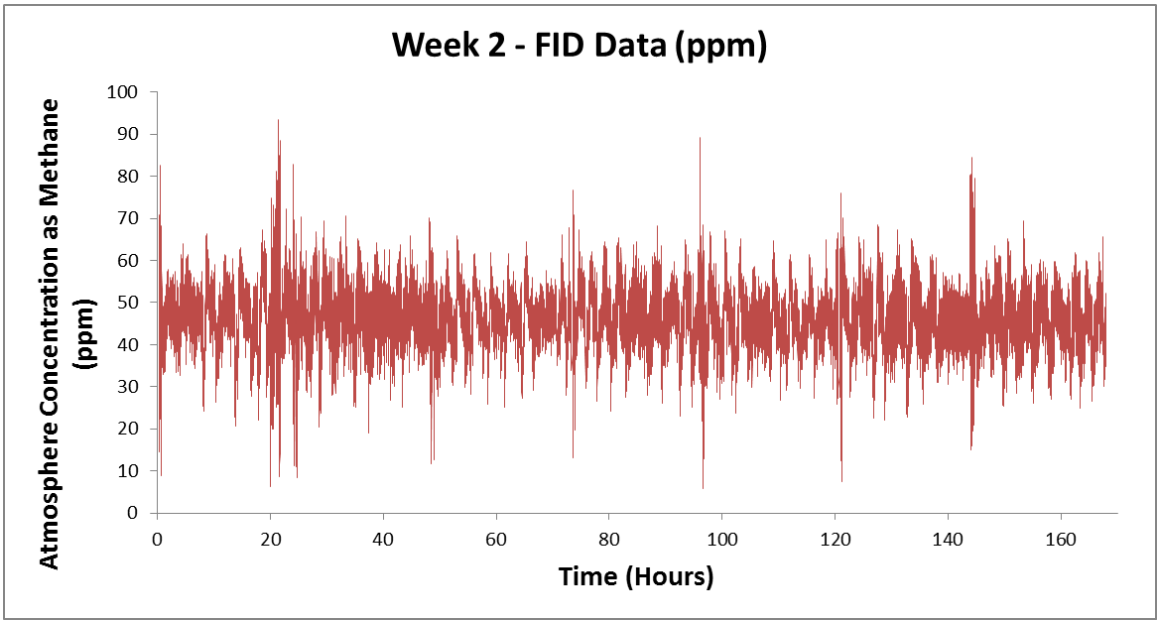

Figure 2: FID Data – 168 to 336 hours of the 14 day test

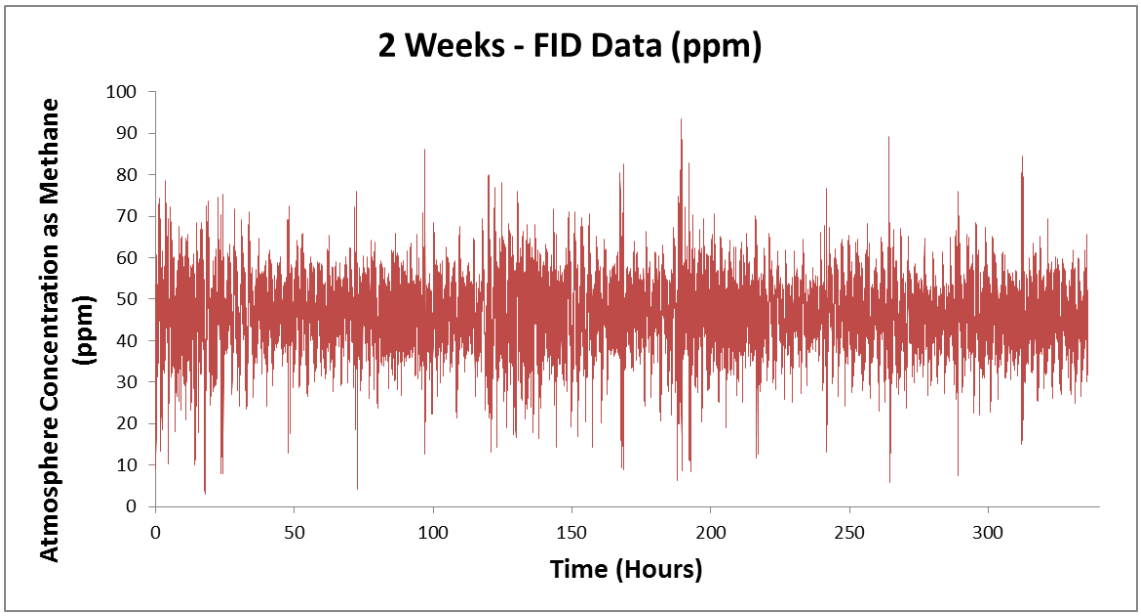

Figure 3: FID Data – 0 to 336 hours of the 14 day test

Real Time Temperature and Humidity

The mean temperature during the test was 22.0°C and the relative humidity was 51.1%. Plots of continuous real time data are given in Figures 4 to 6 and the mean, %RSD, minimum and maximum data are given in Table 7.

Table 7 Temperature and Humidity Range

Time (hrs)	Temperature (°C)				Humidity (%RH)			
	Mean	%RSD	Min	Max	Mean	%RSD	Min	Max
0 - 168	22.1	1.61%	21.1	23.0	51.0	1.22%	48.1	52.9
168 - 336	22.0	1.48%	21.0	22.7	51.3	1.31%	46.6	53.3
0 - 336	22.0	1.58%	21.0	23.0	51.1	1.29%	46.6	53.3

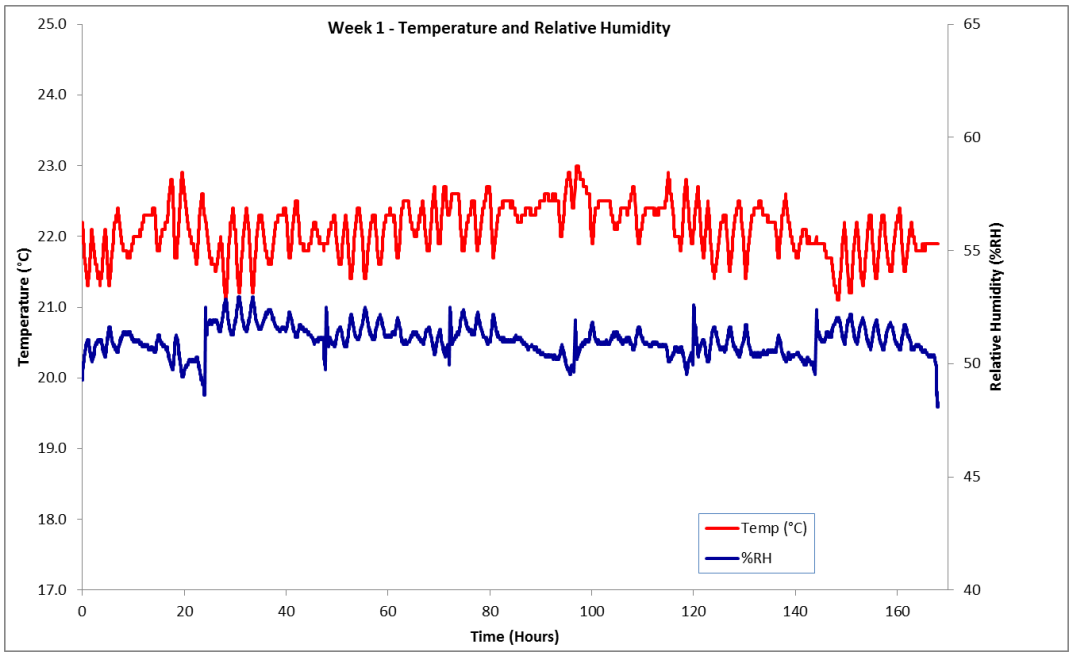

Figure 4: Temperature and Humidity Data – 0 to 168 hours of the 14 day test

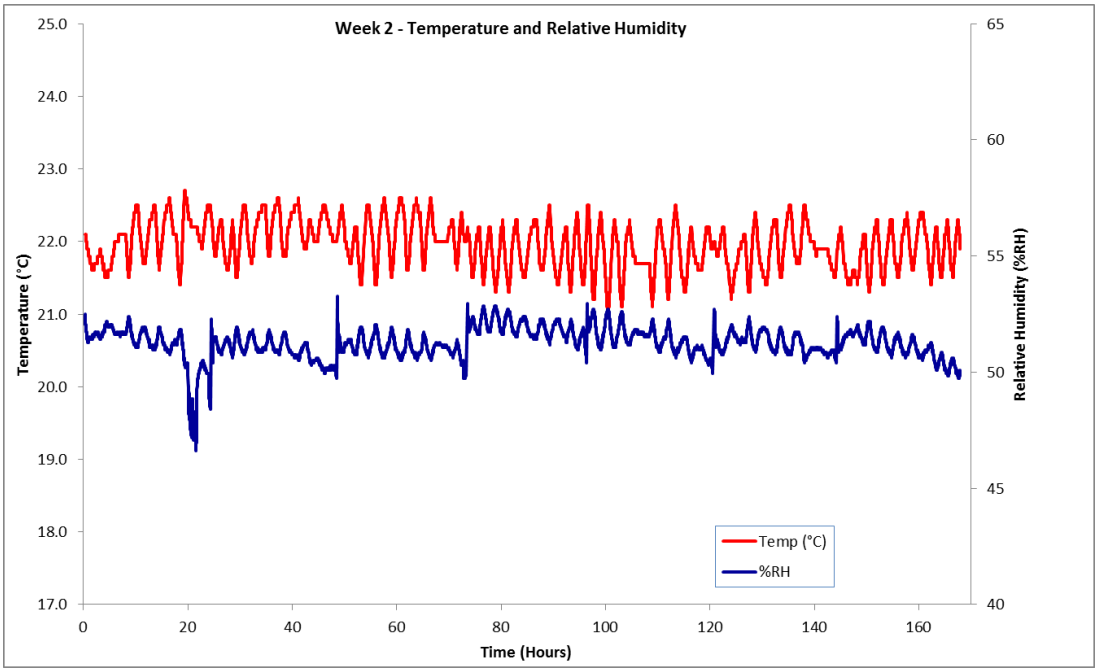

Figure 5: Temperature and Humidity Data – 168 to 336 hours of the 14 day test

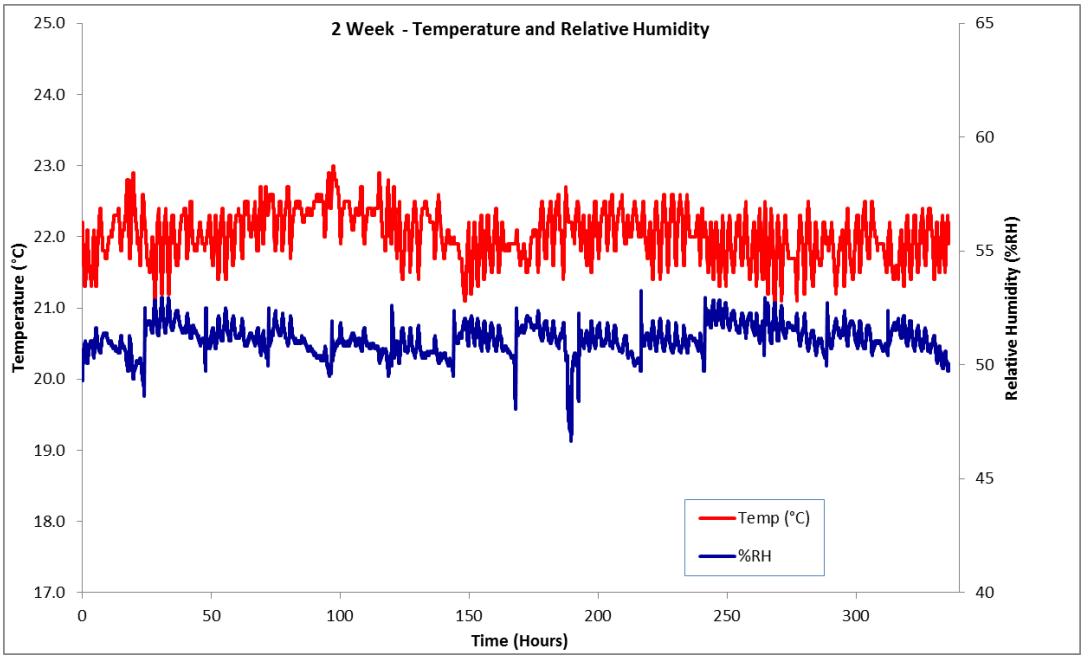

Figure 6: Temperature and Humidity Data – 0 to 336 hours of the 14 day test

Chamber Concentrations

On receipt of the GC/MS data from Markes, the concentrations of the analytes in the chamber were calculated by quantifying the data from the active samples using response factors given by the calibration tubes. Small amounts of benzene and toluene (≤ 4 ng)

were found on blank tubes. Mean benzene and toluene peak areas on the blanks were used to correct the sample peak areas. Table 8 shows the mean concentrations for each compound using the 12 C5TD tubes for which results were obtained and for the 14 CX tubes.

As in the breakthrough test, lower recovery and higher variability was found for some compounds using the CX tubes than using the C5TD tubes (with no ethyl acetate or methyl methacrylate being detected in the CX tubes). No results were obtained for 2-methoxyethanol from the active samplers, with no peak being detected with the CX tubes and only very small peaks with C5TD. The reasons for this are unclear, but could be due to the polarity of this compound. Consequently, it is not possible to determine uptake rates for this compound from this test.

Results obtained using CX tubes were not used in the determination of uptake rates for propan-2-ol, acrylonitrile, ethyl acetate, chloroform, 1,2-dichloroethane, 1,1,1-trichloroethene, carbon tetrachloride, trichloroethene and methyl methacrylate. The mean concentrations of all compounds over the 2 weeks obtained using the remaining tubes are given in Table 9 and the variation in concentration of selected compounds is shown in Figures 7 and 8. The concentrations are seen to be between 4 and 9 ppb and to be adequately stable over time.

Table 8: Mean Concentrations (ppb) obtained from Analysis of CX and C5TD Active Samples

Compound	C5TD (n = 12)		CX (n = 14)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	5.6	2.6%	6.5	2.3%
Propan-2-ol	5.7	8.4%	0.7	23.6%
Acrylonitrile	4.6	4.6%	4.0	20.2%
n-Pentane	8.4	4.5%	9.1	3.3%
1,1-Dichloroethene	6.9	5.1%	7.9	6.2%
Ethyl acetate	4.2	11.0%	NR [1]	NR
n-Hexane	7.1	3.5%	7.5	2.0%
Chloroform	5.6	4.7%	0.1	71.5%
2-Methoxyethanol	NR	NR	NR	NR
1,2-Dichloroethane	5.8	4.4%	1.6	71.0%
1,1,1-Trichloroethane	5.8	4.3%	3.9	21.9%
Benzene	6.3	3.8%	6.4	1.7%
Carbon tetrachloride	5.2	4.1%	1.4	60.0%
Trichloroethene	4.6	4.4%	0.7	110%
Methyl methacrylate	4.1	10.9%	NR	NR
n-Heptane	6.8	3.1%	7.1	1.4%
Toluene	6.3	3.3%	6.3	1.6%
n-Octane	6.7	2.8%	6.8	2.8%
Tetrachloroethene	4.8	3.6%	4.5	2.9%
Ethylbenzene	6.3	3.0%	6.2	2.7%
p-Xylene	6.1	3.1%	5.9	4.5%

[1] NR = no result

Table 9: Mean Concentrations (ppb) for the 1 and 2 week Exposure Periods using the Preferred Sorbent(s)

Compound	First week		Second week		2 weeks		
	Mean	%RSD	Mean	%RSD	Mean	%RSD	Sorbent
1,3-Butadiene	6.1	2.6%	6.1	1.3%	6.1	2.0%	Both
Propan-2-ol	6.0	10.0%	5.5	4.7%	5.7	8.4%	C5TD
Acrylonitrile	4.7	4.9%	4.5	3.2%	4.6	4.6%	C5TD
n-Pentane	9.1	3.4%	8.5	1.6%	8.8	4.4%	Both
1,1-Dichloroethene	7.8	4.6%	7.1	2.6%	7.5	6.0%	Both
Ethyl acetate	4.3	8.8%	4.1	12.9%	4.2	11.0%	C5TD
n-Hexane	7.5	2.7%	7.2	1.4%	7.3	2.9%	Both
Chloroform	5.8	6.0%	5.5	1.5%	5.6	4.7%	C5TD
1,2-Dichloroethane	5.9	6.1%	5.7	1.4%	5.8	4.4%	C5TD
1,1,1-Trichloroethane	5.9	5.4%	5.7	1.8%	5.8	4.3%	C5TD
Benzene	6.5	2.1%	6.3	1.2%	6.4	2.4%	Both
Carbon tetrachloride	5.4	4.6%	5.1	1.8%	5.2	4.1%	C5TD
Trichloroethene	4.8	5.5%	4.5	1.4%	4.6	4.4%	C5TD
Methyl methacrylate	4.2	9.2%	4.1	12.6%	4.1	10.9%	C5TD
n-Heptane	7.1	2.4%	6.9	1.3%	7.0	2.2%	Both
Toluene	6.4	2.0%	6.3	1.6%	6.3	2.1%	Both
n-Octane	6.8	2.6%	6.8	1.9%	6.8	2.2%	Both
Tetrachloroethene	4.7	2.0%	4.6	2.2%	4.6	2.2%	Both
Ethylbenzene	6.3	2.0%	6.2	2.2%	6.3	2.0%	Both
p-Xylene	6.0	2.5%	6.0	3.1%	6.0	2.7%	Both

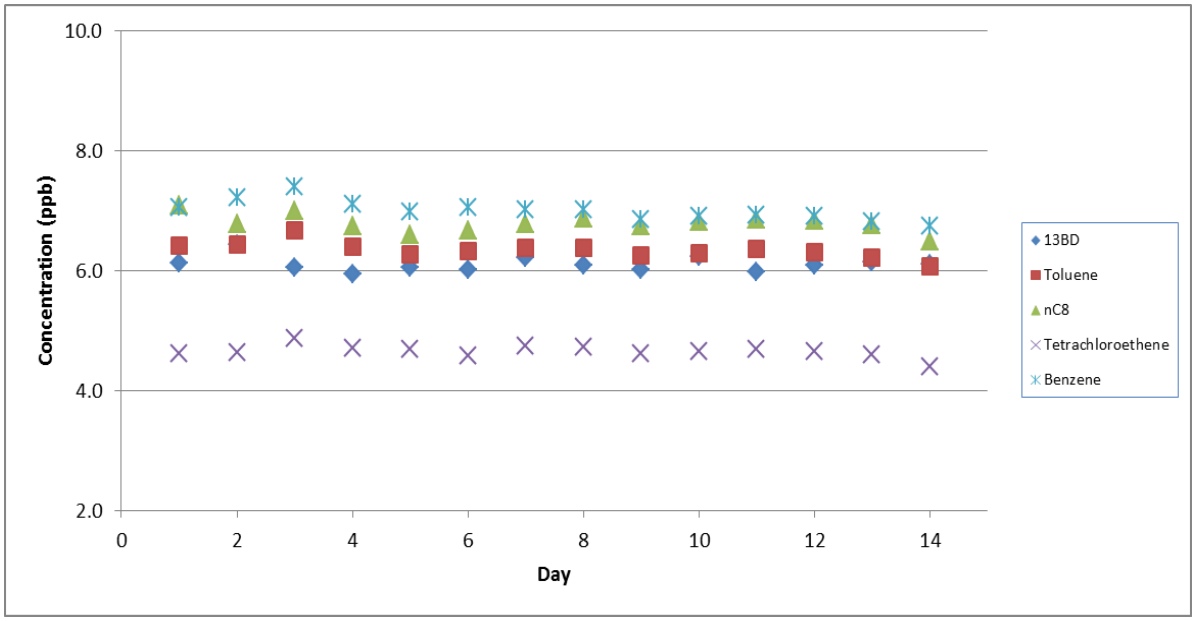

Figure 7: Variation in Concentration over Time for Selected Analytes - 1

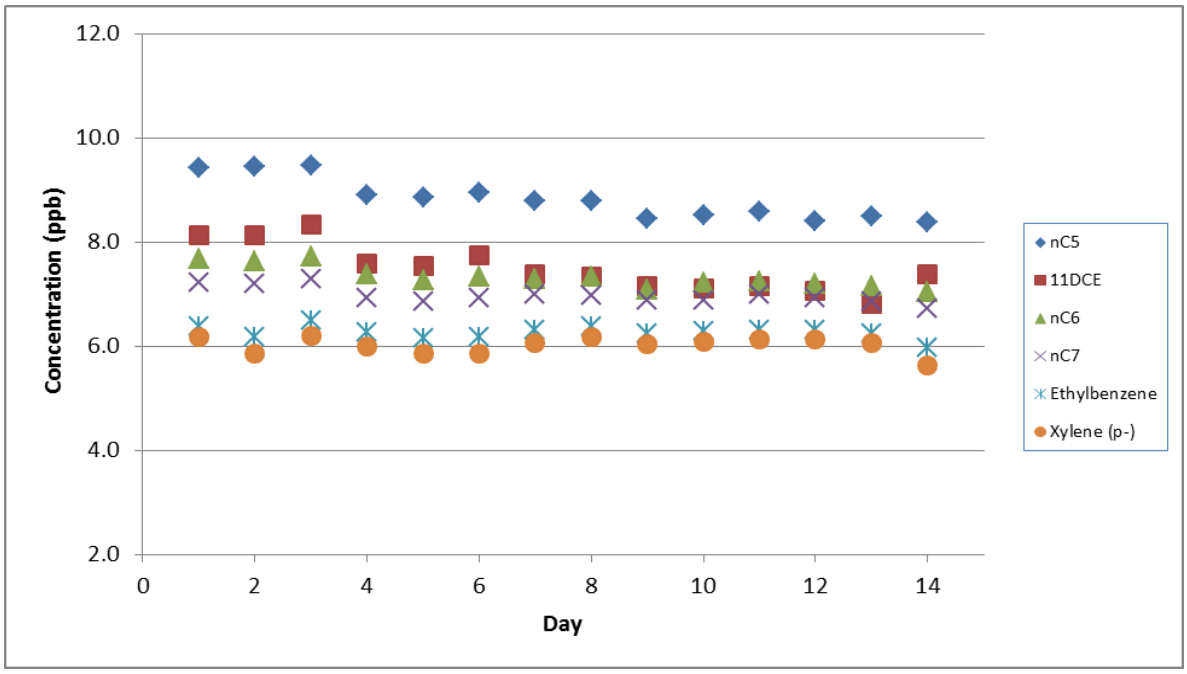

Figure 8: Variation in Concentration over Time for Selected Analytes - 2

Uptake Rates for 1 and 2 Weeks

GC/MS data produced by Markes were used to calculate uptake rates for the target analytes on each of the four sorbents for exposure periods of 1 and 2 weeks. Small amounts of benzene and toluene (≤ 4 ng) were found on unexposed blank tubes. No other compounds were observed on the blank tubes which had been placed in the chamber capped. Mean benzene and toluene peak areas on the blanks were used to correct the sample peak areas prior to calculation of uptake rates.

Uptake rates were determined by dividing the mean normalised peak areas for each compound from a set of 8 diffusive samplers with the mean peak areas per ml from the active samplers for the appropriate sampling period. The value obtained is then divided by the sampling time for the diffusive samplers to give a sampling rate as ml/min. Uptake rates (as ng/ppm/min) are then obtained by multiplying the sampling rate by the molecular weight of the compound and dividing by the molar gas volume (taken as 24.2 for a temperature of ~22˚C). Sampling rates on the four sorbents are given in Tables 10 to 13 and uptake rates in Tables 14 to 17, together with the % relative standard deviations obtained for the 8 replicates.

Excellent repeatability was obtained within the 8 replicate analyses for the majority of compounds on each of the different sorbents, providing confidence in the performance of these compounds on the sorbents tested. However, as with the active samples, some compounds showed greater variability and lower recovery in some of the data sets. Such variations in performance may be due to variety of potential factors including:

Back diffusion, resulting in lower than expected uptake rates (highlighted in blue in the Tables), can occur for more volatile compounds on weaker sorbents. Compounds that may be affected in this way are butadiene, propan-2-ol and acrylonitrile on C1TD and CB and 1,1-dichloroethene on C1TD.

Some instances of high variability could be due to activity on the sorbent (highlighted in pink in the Tables). Compounds where this may be a factor are ethyl acetate, chloroform, 1,2-dichloroethane and methyl methacrylate on CB and CX; 1,1-dichloroethene and 1,1,1-trichloroethane on CB and propan-2-ol, acrylonitrile, carbon tetrachloride and trichloroethene on CX.

Ethyl acetate and methyl methacrylate (highlighted in green in the Tables) showed some variable results on C1TD and repeatable but unfeasibly high results on C5TD. It is unclear if this is due to storage or analytical issues. The observation of good repeatability on C5TD suggests that these compounds are performing effectively on this sorbent for diffusive sampling, but that the uptake rates obtained for these compounds may be being influenced by poor results from the active samplers.

2-Methoxyethanol was observed on C5TD but not on the other sorbents. It was not possible to derive an uptake rate for this compound due to variability in the peaks areas obtained and lack of reliable active sampling data.

Table 10: Sampling rates (ml/min) obtained for Carbograph 1TD

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.09	7.8%	0.05	4.0%
Propan-2-ol	0.01	9.6%	0.01	12.8%
Acrylonitrile	0.10	5.9%	0.06	3.4%
n-Pentane	0.35	2.4%	0.35	2.4%
1,1-Dichloroethene	0.14	4.8%	0.08	4.9%
Ethyl acetate	0.27	15.0%	0.35	5.6%
n-Hexane	0.43	1.4%	0.47	2.6%
Chloroform	0.22	1.7%	0.16	2.1%
1,2-Dichloroethane	0.26	1.5%	0.21	2.1%
1,1,1-Trichloroethane	0.29	3.0%	0.25	1.6%
Benzene	0.47	2.4%	0.45	1.8%
Carbon tetrachloride	0.33	1.9%	0.26	2.6%
Trichloroethene	0.39	2.4%	0.34	1.8%
Methyl methacrylate	0.48	7.4%	0.61	1.6
n-Heptane	0.43	1.9%	0.49	3.9%
Toluene	0.51	1.5%	0.52	2.4%
n-Octane	0.41	2.3%	0.46	3.0%
Tetrachloroethene	0.41	2.6%	0.36	2.8%
Ethylbenzene	0.48	1.7%	0.49	2.6%
p-Xylene	0.50	1.8%	0.50	2.5%

Table 11: Sampling Rates (ml/min) obtained for Carbopack B

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.18	5.9%	0.11	7.4%
Propan-2-ol	NR [1]	NR	NR	NR
Acrylonitrile	0.10	37.0%	0.06	4.9%
n-Pentane	0.40	1.4%	0.40	2.0%
1,1-Dichloroethene	0.20	9.5%	0.15	10.9%
Ethyl acetate	NR	NR	NR	NR
n-Hexane	0.44	3.3%	0.47	2.1%
Chloroform	0.20	14.5%	0.19	3.0%
1,2-Dichloroethane	0.25	8.9%	0.25	12.0%
1,1,1-Trichloroethane	0.28	13.1%	0.29	5.1%
Benzene	0.51	2.4%	0.50	1.3%
Carbon tetrachloride	0.33	5.3%	0.32	2.2%
Trichloroethene	0.46	1.9%	0.43	1.4%
Methyl methacrylate	NR	NR	0.01	60.8%
n-Heptane	0.43	2.4%	0.46	1.8%
Toluene	0.51	1.4%	0.52	1.6%
n-Octane	0.40	1.9%	0.43	1.7%
Tetrachloroethene	0.41	2.0%	0.39	2.1%
Ethylbenzene	0.48	1.6%	0.49	1.8%
p-Xylene	0.49	1.6%	0.49	1.9%

[1] NR = no result obtained

Table 12: Sampling Rates (ml/min) obtained for Carbograph 5TD

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.49	2.2%	0.48	4.0%
Propan-2-ol	0.63	1.2%	0.54	2.7%
Acrylonitrile	0.54	2.0%	0.39	3.3%
n-Pentane	0.64	2.0%	0.67	2.6%
1,1-Dichloroethene	0.49	1.7%	0.43	3.2%
Ethyl acetate	1.03	2.5%	1.06	3.2%
n-Hexane	0.56	1.6%	0.58	2.7%
Chloroform	0.55	1.3%	0.51	2.7%
1,2-Dichloroethane	0.61	1.6%	0.58	2.6%
1,1,1-Trichloroethane	0.54	1.7%	0.53	2.5%
Benzene	0.64	1.1%	0.63	2.8%
Carbon tetrachloride	0.56	1.5%	0.51	2.6%
Trichloroethene	0.56	1.3%	0.52	3.1%
Methyl methacrylate	0.89	1.3%	0.91	2.5%
n-Heptane	0.52	1.5%	0.54	2.7%
Toluene	0.57	1.3%	0.56	2.7%
n-Octane	0.47	1.7%	0.49	2.9%
Tetrachloroethene	0.52	1.4%	0.47	3.2%
Ethylbenzene	0.52	1.3%	0.51	2.7%
p-Xylene	0.52	1.3%	0.51	2.7%

Table 13: Sampling Rates (ml/min) obtained for Carbopack X

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.54	1.6%	0.50	2.8%
Propan-2-ol	NR [1]	NR	NR	NR
Acrylonitrile	0.32	5.9%	0.32	8.9%
n-Pentane	0.55	1.7%	0.55	5.0%
1,1-Dichloroethene	0.52	7.5%	0.46	5.1%
Ethyl acetate	0.04	29.7%	0.09	19.6%
n-Hexane	0.50	2.6%	0.52	4.0%
Chloroform	0.01	19.5%	0.01	13.1%
1,2-Dichloroethane	0.10	6.3%	0.14	13.5%
1,1,1-Trichloroethane	0.41	2.1%	0.44	4.7%
Benzene	0.60	1.5%	0.60	3.5%
Carbon tetrachloride	0.22	4.7%	0.24	9.1%
Trichloroethene	0.07	15.2%	0.11	15.1%
Methyl methacrylate	0.09	43.9%	0.25	15.5%
n-Heptane	0.46	1.8%	0.47	4.0%
Toluene	0.54	1.4%	0.54	3.0%
n-Octane	0.42	1.4%	0.43	3.7%
Tetrachloroethene	0.47	1.3%	0.45	2.9%
Ethylbenzene	0.49	1.5%	0.49	3.1%
p-Xylene	0.49	1.4%	0.49	3.1%

[1] NR = no result obtained

Table 14: Uptake Rates (ng/ppm/min) obtained for Carbograph 1TD

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.19	7.8%	0.11	4.0%
Propan-2-ol	0.04	9.6%	0.03	12.8%
Acrylonitrile	0.21	5.9%	0.13	3.4%
n-Pentane	1.03	2.4%	1.04	2.4%
1,1-Dichloroethene	0.56	4.8%	0.33	4.9%
Ethyl acetate	0.95	15.0%	1.22	5.6%
n-Hexane	1.57	1.4%	1.73	2.6%
Chloroform	1.08	1.7%	0.80	2.1%
1,2-Dichloroethane	1.07	1.5%	0.85	2.1%
1,1,1-Trichloroethane	1.57	3.0%	1.35	1.6%
Benzene	1.51	2.4%	1.45	1.8%
Carbon tetrachloride	2.12	1.9%	1.67	2.6%
Trichloroethene	2.12	2.4%	1.87	1.8%
Methyl methacrylate	1.99	7.4%	2.54	1.6%
n-Heptane	1.80	1.9%	2.02	3.9%
Toluene	1.95	1.5%	1.97	2.4%
n-Octane	1.95	2.3%	2.15	3.0%
Tetrachloroethene	2.80	2.6%	2.49	2.8%
Ethylbenzene	2.12	1.7%	2.14	2.6%
p-Xylene	2.19	1.8%	2.18	2.5%

Table 15: Uptake Rates (ng/ppm/min) obtained for Carbopack B

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	0.40	5.9%	0.24	7.4%
Propan-2-ol	NR [1]	NR	NR	NR
Acrylonitrile	0.21	37.0%	0.14	4.9%
n-Pentane	1.20	1.4%	1.18	2.0%
1,1-Dichloroethene	0.89	9.5%	0.59	10.9%
Ethyl acetate	NR	NR	NR	NR
n-Hexane	1.60	3.3%	1.72	2.1%
Chloroform	0.97	14.5%	0.95	3.0%
1,2-Dichloroethane	1.01	8.9%	1.03	12.0%
1,1,1-Trichloroethane	1.56	13.1%	1.62	5.1%
Benzene	1.65	2.4%	1.63	1.3%
Carbon tetrachloride	2.09	5.3%	2.01	2.2%
Trichloroethene	2.49	1.9%	2.36	1.4%
Methyl methacrylate	NR	NR	0.03	60.8%
n-Heptane	1.79	2.4%	1.90	1.8%
Toluene	1.95	1.4%	1.99	1.6%
n-Octane	1.89	1.9%	2.03	1.7%
Tetrachloroethene	2.82	2.0%	2.69	2.1%
Ethylbenzene	2.13	1.6%	2.15	1.8%
p-Xylene	2.15	1.6%	2.16	1.9%

[1] NR = no result obtained

Table 16: Uptake Rates (ng/ppm/min) obtained for Carbograph 5TD

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	1.09	2.2%	1.06	4.0%
Propan-2-ol	1.56	1.2%	1.33	2.7%
Acrylonitrile	1.18	2.0%	0.86	3.3%
n-Pentane	1.92	2.0%	2.01	2.6%
1,1-Dichloroethene	1.96	1.7%	1.74	3.2%
Ethyl acetate	3.60	2.5%	3.72	3.2%
n-Hexane	2.05	1.6%	2.12	2.7%
Chloroform	2.70	1.3%	2.50	2.7%
1,2-Dichloroethane	2.48	1.6%	2.38	2.6%
1,1,1-Trichloroethane	2.99	1.7%	2.90	2.5%
Benzene	2.05	1.1%	2.02	2.8%
Carbon tetrachloride	3.54	1.5%	3.26	2.6%
Trichloroethene	3.04	1.3%	2.82	3.1%
Methyl methacrylate	3.68	1.3%	3.75	2.5%
n-Heptane	2.15	1.5%	2.23	2.7%
Toluene	2.18	1.3%	2.13	2.7%
n-Octane	2.22	1.7%	2.32	2.9%
Tetrachloroethene	3.53	1.4%	3.22	3.2%
Ethylbenzene	2.28	1.3%	2.26	2.7%
p-Xylene	2.27	1.3%	2.25	2.7%

Table 17: Uptake Rates (ng/ppm/min) obtained for Carbopack X

Compound	1 week (n = 8)		2 weeks (n = 8)	
	Mean	%RSD	Mean	%RSD
1,3-Butadiene	1.21	1.6%	1.12	2.8%
Propan-2-ol	NR [1]	NR	NR	NR
Acrylonitrile	0.71	5.9%	0.71	8.9%
n-Pentane	1.63	1.7%	1.65	5.0%
1,1-Dichloroethene	2.07	7.5%	1.84	5.1%
Ethyl acetate	0.13	29.7%	0.32	19.6%
n-Hexane	1.82	2.6%	1.88	4.0%
Chloroform	0.05	19.5%	0.06	13.1%
1,2-Dichloroethane	0.41	6.3%	0.59	13.5%
1,1,1-Trichloroethane	2.25	2.1%	2.40	4.7%
Benzene	1.93	1.5%	1.93	3.5%
Carbon tetrachloride	1.37	4.7%	1.54	9.1%
Trichloroethene	0.40	15.2%	0.58	15.1%
Methyl methacrylate	0.35	43.9%	1.04	15.5%
n-Heptane	1.90	1.8%	1.95	4.0%
Toluene	2.05	1.4%	2.05	3.0%
n-Octane	1.98	1.4%	2.04	3.7%
Tetrachloroethene	3.20	1.3%	3.09	2.9%
Ethylbenzene	2.14	1.5%	2.15	3.1%
p-Xylene	2.13	1.4%	2.16	3.1%

[1] NR = no result obtained

Conclusions

This project has determined uptake rates for a range of VOCs on four sorbents over exposure periods of 1 and 2 weeks. The majority of values obtained showed low variability and consistency over the different exposure periods. The more volatile compounds showed more variable results on the weaker sorbents, probably as a result of back diffusion over the extended exposure periods. An issue of low recovery/high variability was also identified for some of the compounds during both active and passive sampling from CX sorbent, which is thought to arise from activity on the sorbent. The same issue was found to affect some passive results on CB. Results obtained for ethyl acetate and methyl methacrylate on C1TD and C5TD remain unexplained. Further tests are planned at HSE to investigate storage and uptake rates at occupational level for these compounds on this set of sorbents.

Appendices

Table A1: Diffusive Sampling Details – Carbograph 1TD, 1 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
803311	**16/03/22**	**14:20 GMT**	**23/03/22**	**14:20 GMT**	**10,080**	**Sample, fitted with diffusive gauze**
803112						
803113						
803114						
803115						
803116						
803117						
803118						
803119						**Chamber blank**
803120						
803131						
803132						
803133						
803137						**Travel blank**
803138						

Table A2: Diffusive Sampling Details – Carbograph 1TD, 2 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
803121	**16/03/22**	**14:20 GMT**	**30/03/22**	**15:20 BST**	**20,160**	**Sample, fitted with diffusive gauze**
803122						
803123						
803124						
803125						
803126						
803127						
803128						
803129						**Chamber blank**
803130						
803136						
803135						
803134						
803139						**Travel blank**
803140						

Table A3: Diffusive Sampling Details – Carbopack B, 1 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
809925	**16/03/22**	**14:20 GMT**	**23/03/22**	**14:20 GMT**	**10,080**	**Sample, fitted with diffusive gauze**
809930						
809928						
809926						
809921						
809924						
809913						
809906						
809923						**Chamber blank**
809902						
809922						
809903						
809904						
809907						**Travel blank**
809908						

Table A4: Diffusive Sampling Details – Carbopack B, 2 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
809920	**16/03/22**	**14:20 GMT**	**30/03/22**	**15:20 BST**	**20,160**	**Sample, fitted with diffusive gauze**
809918						
809914						
809915						
809911						
809910						
809912						
809927						
809916						**Chamber blank**
809919						
809917						
809901						
809905						
809909						**Travel blank**
809929						

Table A5: Diffusive Sampling Details – Carbograph 5TD, 1 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
802951	**23/03/22**	**14:22 GMT**	**30/03/22**	**15:20 BST**	**10,078**	**Sample, fitted with diffusive gauze**
802952						
802953						
802954						
802955						
802956						
802957						
802958						
802959						**Chamber blank**
802960						
808391						
808392						
808393						
808397						**Travel blank**
808398						

Table A6: Diffusive Sampling Details – Carbograph 5TD, 2 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
802961	**16/03/22**	**14:20 GMT**	**30/03/22**	**15:20 BST**	**20,160**	**Sample, fitted with diffusive gauze**
802962						
802963						
802964						
802965						
802966						
802967						
802968						
802969						**Chamber blank**
802970						
808394						
808395						
808396						
808399						**Travel blank**
808400						

Table A7: Diffusive Sampling Details – Carbopack X, 1 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
808371	**23/03/22**	**14:22 GMT**	**30/03/22**	**15:20 BST**	**10,078**	**Sample, fitted with diffusive gauze**
808372						
808373						
808374						
808375						
808376						
808377						
808378						
808379						**Chamber blank**
808380						
752391						
752392						
752393						
808626						**Travel blank**
808632						

Table A8: Diffusive Sampling Details – Carbopack X, 2 week

Tube Number	Date Start	Time Start	Date End	Time End	Exposure period (min)	Notes
808381	**16/03/22**	**14:20 GMT**	**30/03/22**	**15:20 BST**	**20,160**	**Sample, fitted with diffusive gauze**
808382						
808383						
808384						
808385						
808386						
808387						
808388						
808389						**Chamber blank**
808390						
752394						
752395						
752397						
808638						**Travel blank**

Table A9: Active Sampling Volumes

Day	Date	Sorbent	Tube No.	Time On	Flow before (ml/min)	Time Off	Flow after (ml/min)	Volume (litres)
1	16-17/03/22	5TD	808616	14:20	5.04	14:20	5.00	**7.23**
1	16-17/03/22	CX	808623	14:20	5.02	14:20	4.95	**7.18**
2	17-18/03/22	5TD	808601	14:29	5.02	14:05	4.93	**7.04**
2	17-18/03/22	CX	808633	14:29	5.00	14:05	4.91	**7.02**
3	18-19/03/22	5TD	808612	14:14	4.93	14:26	4.96	**7.18**
3	18-19/03/22	CX	808640	14:14	4.90	14:26	4.89	**7.11**
4	19-20/03/22	5TD	808613	14:35	4.97	14:54	5.00	**7.27**
4	19-20/03/22	CX	808635	14:35	4.92	14:54	4.95	**7.20**
5	20-21/03/22	5TD	808606	15:08	5.05	14:17	5.01	**6.99**
5	20-21/03/22	CX	808639	15:08	4.99	14:17	4.96	**6.91**
6	21-22/03/22	5TD	808617	14:26	5.00	14:10	5.00	**7.12**
6	21-22/03/22	CX	808636	14:26	4.96	14:10	4.95	**7.06**
7	22-23/03/22	5TD	808608	14:28	5.00	14:20	4.99	**7.15**
7	22-23/03/22	CX	808631	14:28	4.97	14:20	4.97	**7.12**
8	23-24/03/22	5TD	808610	14:40	4.98	14:41	5.04	**7.22**
8	23-24/03/22	CX	808627	14:40	4.96	14:41	4.98	**7.16**
9	24-25/03/22	5TD	808620	14:52	5.04	14:52	5.00	**7.23**
9	24-25/03/22	CX	808625	14:52	5.01	14:52	4.98	**7.19**
10	25-26/03/22	5TD	808603	15:01	5.00	15:35	4.99	**7.36**
10	25-26/03/22	CX	808621	15:01	4.98	15:35	4.94	**7.31**
11	26-27/03/22	5TD	808602	15:54	5.07	15:45 *	4.99	**6.95**
11	26-27/03/22	CX	808624	15:54	5.02	15:45 *	5.01	**6.88**
12	27-28/03/22	5TD	808611	15:53 *	5.08	15:57 *	5.05	**7.31**
12	27-28/03/22	CX	808628	15:53 *	4.98	15:57 *	5.05	**7.24**
13	28-29/03/22	5TD	808605	16:15 *	5.08	15:36 *	5.06	**7.10**
13	28-29/03/22	CX	808629	16:15 *	5.01	15:36 *	5.08	**7.07**
14	29-30/03/22	5TD	808604	15:44 *	5.10	15:20 *	5.17	**7.27**
14	29-30/03/22	CX	808637	15:44 *	5.06	15:20 *	5.14	**7.22**

***** BST

Table A10: Blanks for Active Samples

Sorbent	Tube Number
5TD	808615
5TD	808618
5TD	808609
CX	808630
CX	808634
CX	808622

Table A11: Loadings for 1,3-Butadiene Standard Tubes

Standard Identity	Loading (ng)
Std 1G	11.9
Std 2G	47.6
Std 3G	95.2
Std 4G	154.6
Std 5G	297.4

Table A12: Loadings (in ng) for Liquid Component Standard Tubes

Component	Std 1L	Std 2L	Std 3L	Std 4L	Std 5L
1,1-Dichloroethene	6.9	20.9	76.0	229.6	833.5
n-Pentane	5.0	15.0	54.5	164.6	597.5
Chloroform	8.3	25.0	90.9	274.6	997.0
n-Hexane	5.5	16.6	60.3	182.2	661.4
1,1,1-Trichloroethane	8.6	26.1	94.8	286.5	1039.9
Acrylonitrile	4.6	13.9	50.5	152.5	553.5
Ethyl acetate	6.5	19.8	71.7	216.6	786.2
Carbon tetrachloride	11.6	35.1	127.3	384.6	1396.0
Benzene	5.8	17.5	63.4	191.4	694.9
Propan-2-ol	5.1	15.5	56.1	169.5	615.4
1,2-Dichloroethane	7.0	21.3	77.2	233.2	846.4
Trichloroethene	9.5	28.8	104.6	316.1	1147.6
n-Heptane	6.5	19.8	71.8	216.9	787.4
Methyl methacrylate	7.4	22.5	81.6	246.5	894.8
Toluene	6.3	19.0	69.1	208.7	757.5
Tetrachloroethene	11.8	35.8	129.9	392.4	1424.4
2-Methoxyethanol	6.3	19.0	68.9	208.1	755.4
n-Octane	7.9	23.7	86.2	260.4	945.3
Ethylbenzene	6.3	19.0	68.9	208.1	755.5
p-Xylene	6.3	18.9	68.6	207.1	751.8

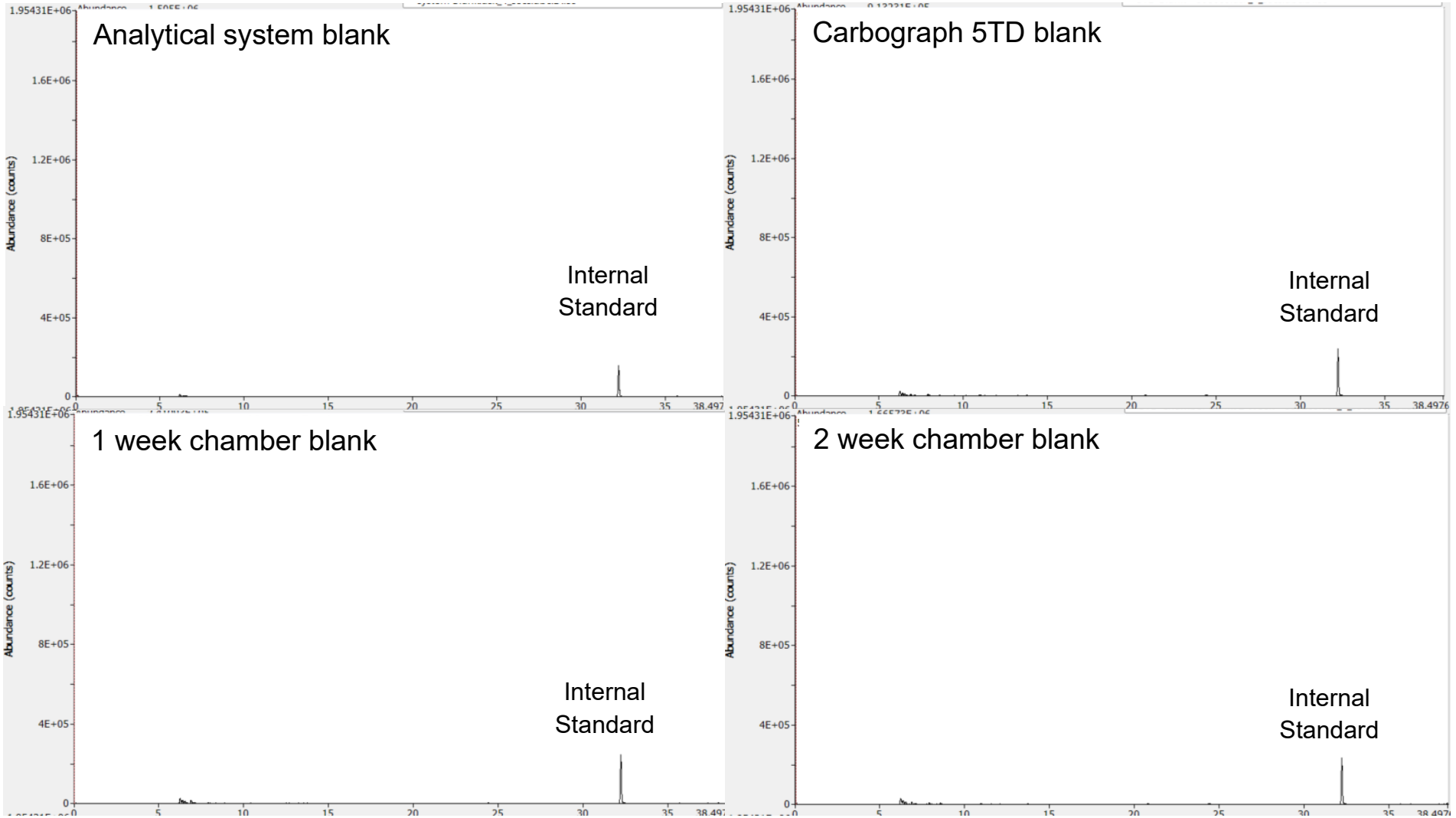

Figure A1: GC/MS Chromatograms - Blanks (data supplied by Markes)

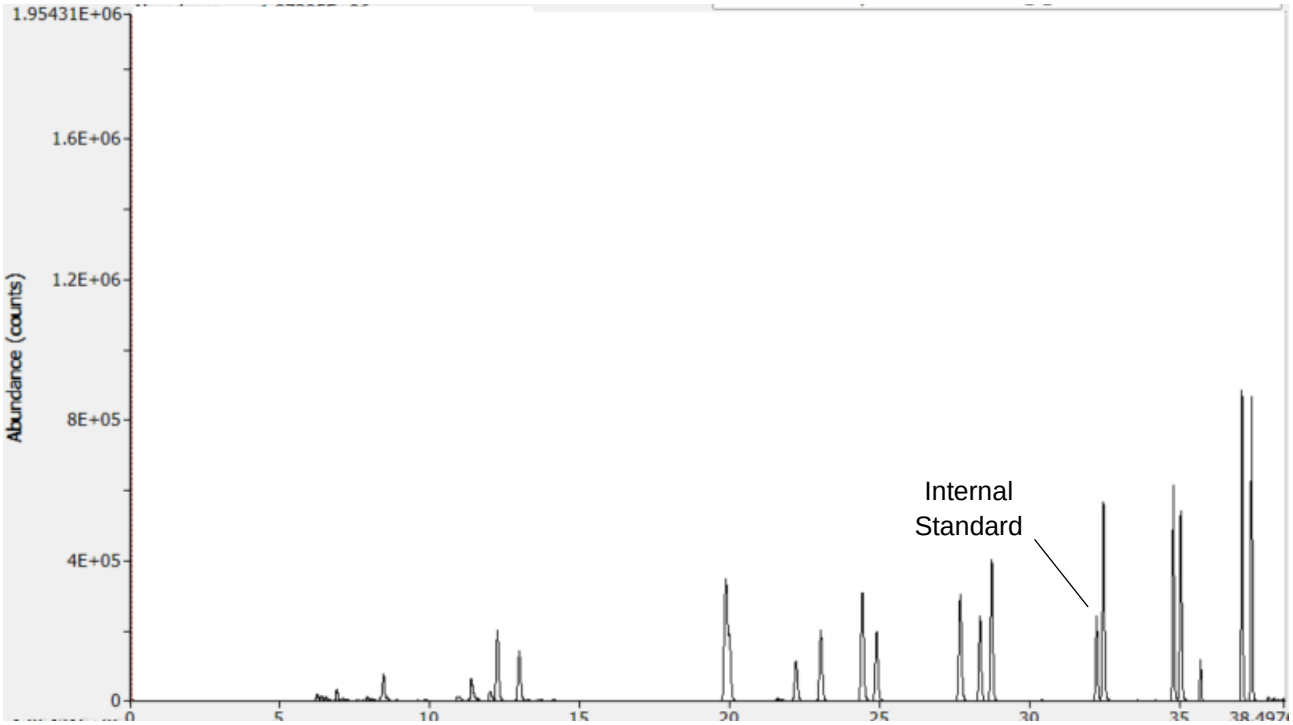

Figure A2: GC/MS Chromatogram - 1 week diffusive C5TD (data supplied by Markes)

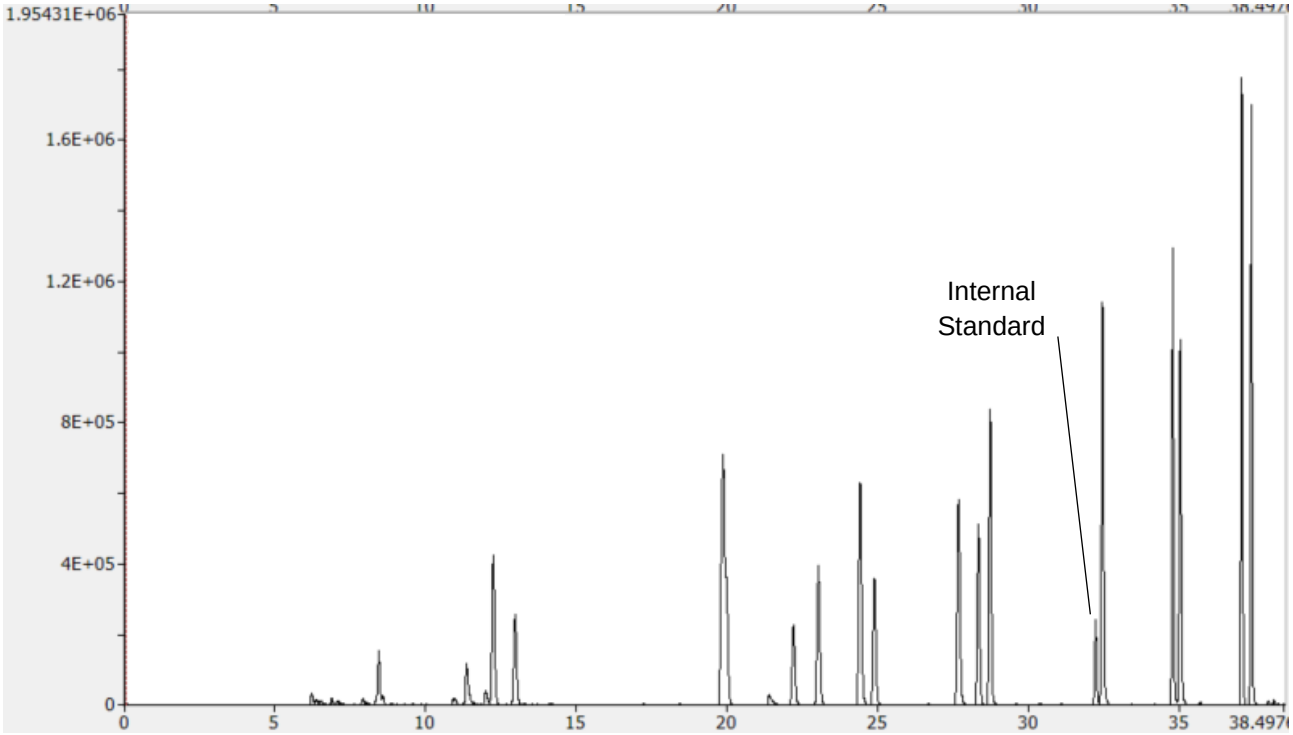

Figure A3: GC/MS Chromatogram - 2 week diffusive C5TD (data supplied by Markes)

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