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Homologue group profiles of ΣPCAs C14-17 in environmental samples from Norwegian monitoring programmes

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ABSTRACT

Recently, chlorinated paraffins with carbon chain lengths in the range $C_{14–17}$ and chlorination levels at or exceeding 45 per cent chlorine by weight have been proposed for listing under the Stockholm Convention. To aid the process of determining the identification of sum polychlorinated alkanes ΣPCAs C_{14-17} under the regulation (i.e. number of chlorines), there is a need for data from environmental samples that specifies the homologue group profiles, not just ΣPCAs.

In this report we present data on PCAs with a focus on ΣPCAs C_{14-17} from the Norwegian Environment Agency's monitoring programmes in more detail than available in the programmes reports, focusing on homologue group patterns and chlorination degree. The programmes are i) Environmental pollutants in the terrestrial and urban environment ii) Atmospheric contaminants iii) Environmental contaminants in an urban fjord. Data presented are from the 2022 (Halvorsen et al., 2023; Heimstad et al., 2023; Ruus, 2023) and 2023 (reports in prep) programmes.

Homologgruppeprofiler av ΣPCAs C14-17 i miljøprøver fra Norske miljøovervåkningsprogram

KEYWORDS

CPs MCCPs PCAs

Nylig har klorparafiner med karbonkjedelengder i området C14–17 og kloreringsnivåer på eller over 45 vektprosent klor blitt foreslått regulert under Stockholmkonvensjonen. For å hjelpe prosessen med å fastsette identifisering av summen av polyklorerte alkaner ΣPCA C14-17 under forskriften (dvs. antall klor), er det behov for data fra miljøprøver som spesifiserer homologgruppeprofilene, ikke bare ΣPCA.

I denne rapporten presenterer vi data om PCA med fokus på ΣPCA C14-17 fra Miljødirektoratets overvåkingsprogrammer mer detaljert enn tilgjengelig i programrapportene, med fokus på homologgruppemønstre og kloreringsgrad. Programmene er i) Miljøgifter i terrestrisk bymiljø ii) Atmosfæriske forurensninger iii) Miljøgifter i en urban fjord. Data som presenteres er fra 2022 (Halvorsen et al., 2023; Heimstad et al., 2023; Ruus, 2023) og 2023 (rapporter i prep)-programmene.

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Introduction

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In this report we present data on PCAs with a focus on ΣPCAs C_{14-17} from the Norwegian Environment Agency's monitoring programmes in more detail than available in the programmes reports, focusing on homologue group patterns and chlorination degree. The programmes are i) Environmental pollutants in the terrestrial and urban environment ii) Atmospheric contaminants iii) Environmental contaminants in an urban fjord. Data presented are from the 2022 (Halvorsen et al., 2023; Heimstad et al., 2023; Ruus, 2023) and 2023 (reports in prep) programmes.
ΣPCAs C_{14-17} are often referred to as medium chain chlorinated paraffins or MCCPs, but alternative terminology for this group of compounds have been recommended by Fernandes et al. (2023), and we will apply this in this report.

Homologue group profiles of ΣPCAs C14-17 in environmental samples from Norwegian monitoring programmes

1 Methods

1.1 Samples included in the study

The sample types included in this report are from the urban terrestrial environment (earthworm (N = 1), fieldfare eggs (N = 4), rat liver (N = 2), vegetation (N = 4)), from urban aquatic environments (trout muscle (N = 3), blue mussel (N = 1), cod liver (N = 4), seal blubber (N = 10) stormwater particles (N=4), particles from an urban river (N = 3), sediment (N = 4)), indoor environments (house dust (N = 5)), and atmosphere (background air (N = 5), arctic air (N = 18), and urban air (N = 14)). Only samples where concentrations of ΣPCAs C10-17 were well above detection limits were selected for inclusion in this study in order to minimize influence of sample contamination on reported patterns. Chlorination degrees, chain length profiles and homologue group profiles presented represent the average for each sample type where N > 1, except in Table 1, where data for each individual sample is presented.

2 Analysis

Sample preparation methods are described in reports from the monitoring programmes (Halvorsen et al., 2023; Heimstad et al., 2023; Ruus, 2023).
For instrumental analysis and quantification, all samples were analysed on an Agilent GC-qToF 7200 in ECNI mode using methane as a moderating gas. All chromatograms were manually inspected using the MassHunter software. Manual integration was performed where necessary to avoid potential interferences. ΣPCAs $C_{10\text{-}17}$ were quantified using a pattern deconvolution procedure based on the method presented in Bogdal et al. (2015). The standards (Chiron AS) analysed for ΣPCAs $C_{10\text{-}13}$ were C_{10} (52.5% and 58.4% Cl), C_{11} (52.3% and 57.7% Cl), C_{12} (53,8% and 57,3% Cl) and C_{13} (45,9% and 60% Cl). For ΣPCAs $C_{14\text{-}17}$, C_{14} (49.2% and 58.7% Cl), C_{15} (47.7% and 59.3% Cl), C_{16} (51.5% and 58.4% Cl) and C_{17} (56.3% Cl) were analysed.

The resulting homologue group patterns from the standards were used to reconstruct the homologue group pattern of the sample. Sample contamination by PCAs in the indoor laboratory environment and contact with products containing PCAs is a common issue in PCA analysis (van Mourik et al., 2020). Therefore, prior to reconstruction, the signals (integrated areas relative to internal standard) in the corresponding method blank were subtracted from the signals in the samples on a homologue group basis. The pattern reconstruction was done using the nnls package in R studio, which applies the Lawson-Hanson algorithm to provide non-negative least squares estimates for the contribution of the various standards to the sample pattern. A R2 between sample pattern and reconstructed pattern of 0.5 or above was considered satisfactory (Bogdal et al., 2015).
Given the large number of standards involved in quantification, only a smaller number of standards were analysed alongside each batch of samples to confirm stability of the instrument response (typically ΣPCAs $C_{10\text{-}13}$ with 51%, 55%, and 63% Cl, and ΣPCAs $C_{14\text{-}17}$ with 42% and 52% Cl). The instrument stability and quantification procedure were also monitored by the regular analysis of known concentration in-house standard mixtures. Quantification of sample ΣPCAs $C_{10\text{-}17}$ content was achieved by constructing separate calibration curves using areas from each standard at four concentration levels, from which sample areas were compared, and concentration calculated. R2 for the calibration curves were above 0.99. Calculated sample concentration based on each standard was subsequently weighted by the non-negative least squares estimate for contribution of each standard to the sample, and contributions from all standards were summarized. All sample and standard areas

were normalized using ^{13}C labelled internal standard. In addition, areas were normalized by natural abundance of the measured m/z, enabling extension of the instruments linear range

2.1 Influence of instrumental method on detected homologue group patterns

It is important to note that the choice of instrumental method can influence the detected homologue group profiles. This potentially affects comparability of homologue group profiles obtained via labs using different methods. In general, there are two main differences in the analytical approach, GC-MS and LC-MS. Because of the different ionization modes used in GC-MS and LC-MS, the relative responses of the different homologue groups are also different. It is most common to run GC-MS in electron capture negative ion mode (ECNI). In this case the responses will in general increase with increasing number of chlorines. The ability of analysing PCAs with 3 Cl and less (for some instruments even as low as 4 Cl is not possible to measure) is absent. In the LC-MS the responses between the different homologue groups are more or less the same and it is possible to detect PCAs with 1Cl. Comparisons between these two techniques have shown that the calculated sum of ΣPCAs $C_{10\text{-}13}$ and ΣPCAs $C_{14\text{-}17}$ are not significantly different between GC-MS and LC-MS given that the same analytical approach is used for both standards and samples, but the homologue group pattern might be different between the methods. However, the relative amounts of ΣPCAs $C_{10\text{-}13}$ and ΣPCAs $C_{14\text{-}17}$ with less than 4 Cl are connected to the production volume which is, statistically speaking, quite low (Yuan et al., 2017).

3 Chain length and chlorination patterns of ΣPCAs C14-17

The Chain length and chlorination patterns of ΣPCAs $C_{14\text{-}17}$ in the studied environmental samples are summarized in Figure 1, Figure 3, Figure 4, and Table 1. Figure 1 show the distribution of chain lengths in the sample types included in this study. There was a clear similarity of chain length patterns between the different sample types, with C_{14} dominating in all sample types except rat liver where C_{15} and C_{16} dominated (Figure 1). Trout and blue mussel had a strong dominance of C_{14} homologue groups and no detected levels of C_{16} and C_{17}, however the concentrations of ΣPCAs $C_{14\text{-}17}$ in these samples were low (see Figure 2 and Table 1), which can lead to distortions in the detected pattern compared to the "actual" pattern. Air samples also showed a stronger dominance of C_{14} compared to other sample types, which fits well with the physicochemical properties of the PCAs. On average, C_{14} homologue groups are more volatile compared to average C_{15}, C_{16}, and C_{17} homologue groups, although chlorination degree has an even larger influence on volatility than chain length (Glüge, Schinkel, Hungerbühler, Cariou, & Bogdal, 2018).

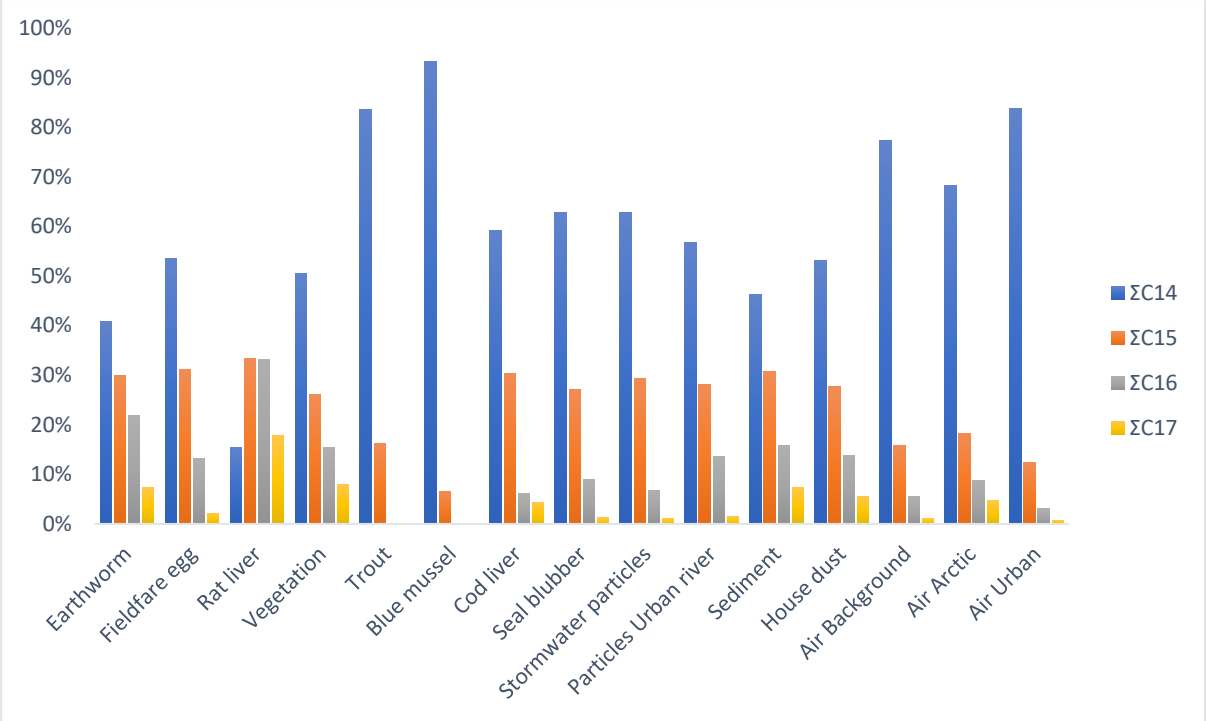

Figure 1: Distribution of chain lengths (ΣPCAs C_{14}, ΣPCAs C_{15}, ΣPCAs C_{16}, ΣPCAs C_{17}) in the sample types included in the study.

Figure 2 shows the average concentration of ΣPCAs $C_{14\text{-}17}$ in the studied environmental sample types to aid in the interpretation of chain length and chlorination degree patterns. Please note that the samples included in this study represents a selection, where samples with concentrations under or near detection limits have been excluded. The concentrations shown in Figure 2 are therefore not necessarily representative for the respective environmental matrices.

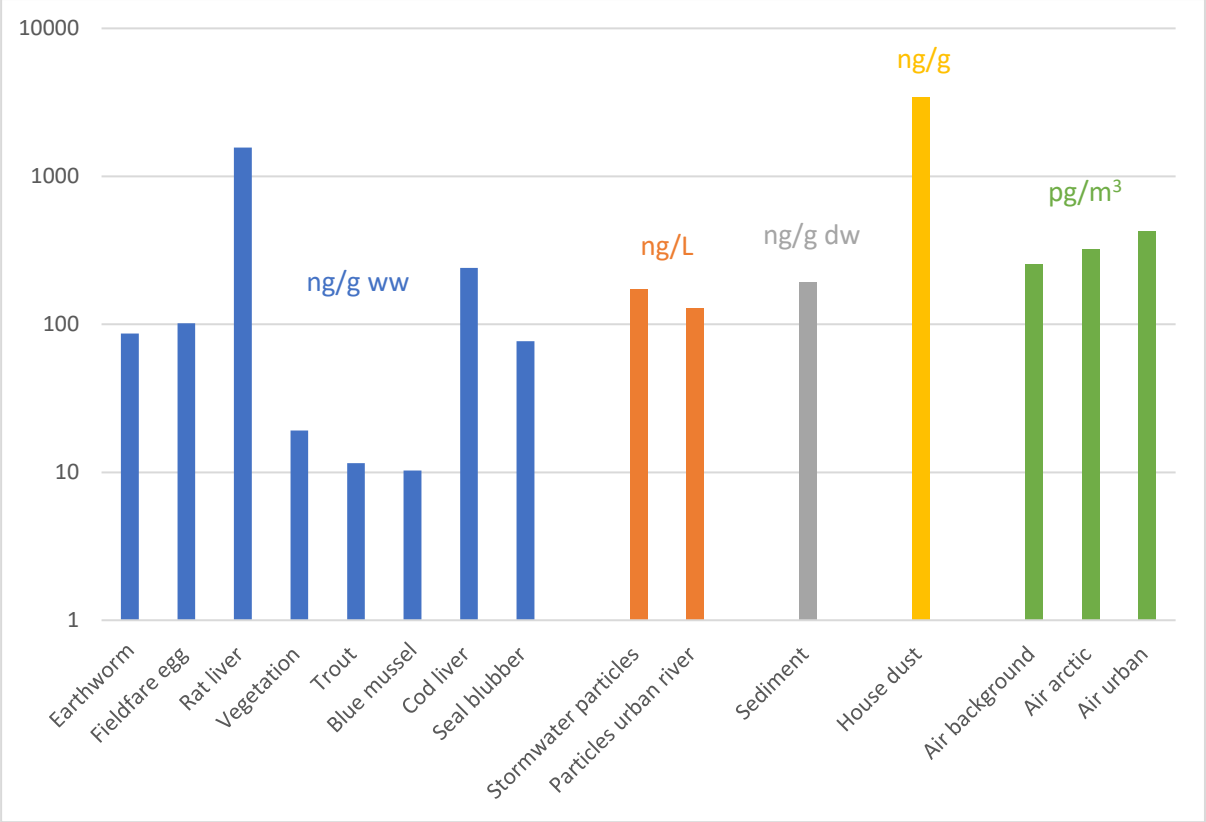

Figure 2: Average concentration of ΣPCAs $C_{14\text{-}17}$ in the studied environmental sample types. Please note logarithmic scale. Unit for each sample type shown above each category (blue: biota, orange: particles in water, grey: sediments, yellow: house dust, green: atmospheric samples).

Figure 3 show the distribution of chlorine content in the homologue groups detected in the sample types included in this study. Chlorine content in Figure 3 is represented by the sum of all homologue groups with 4 Cl, sum of all homologue groups with 5 Cl etc. There was a larger degree of variation in the chlorine content (Figure 3) for the different sample types compared to the variation in chain lengths (Figure 1). Overall, there was a dominance of homologue groups with 6 and 7 Cl. Notable exceptions are rat liver, where higher chlorinated homologue groups dominated (7, 8, and 9 Cl), and urban air, where lower chlorinated homologue groups dominated (5 and 6 Cl). Interestingly, arctic air and background air did not show the same dominance as urban air of homologue groups with 5 Cl. This is further discussed below.

Homologue groups with 4 Cl were detected at notable levels in air (arctic, background, and urban) and in cod liver.

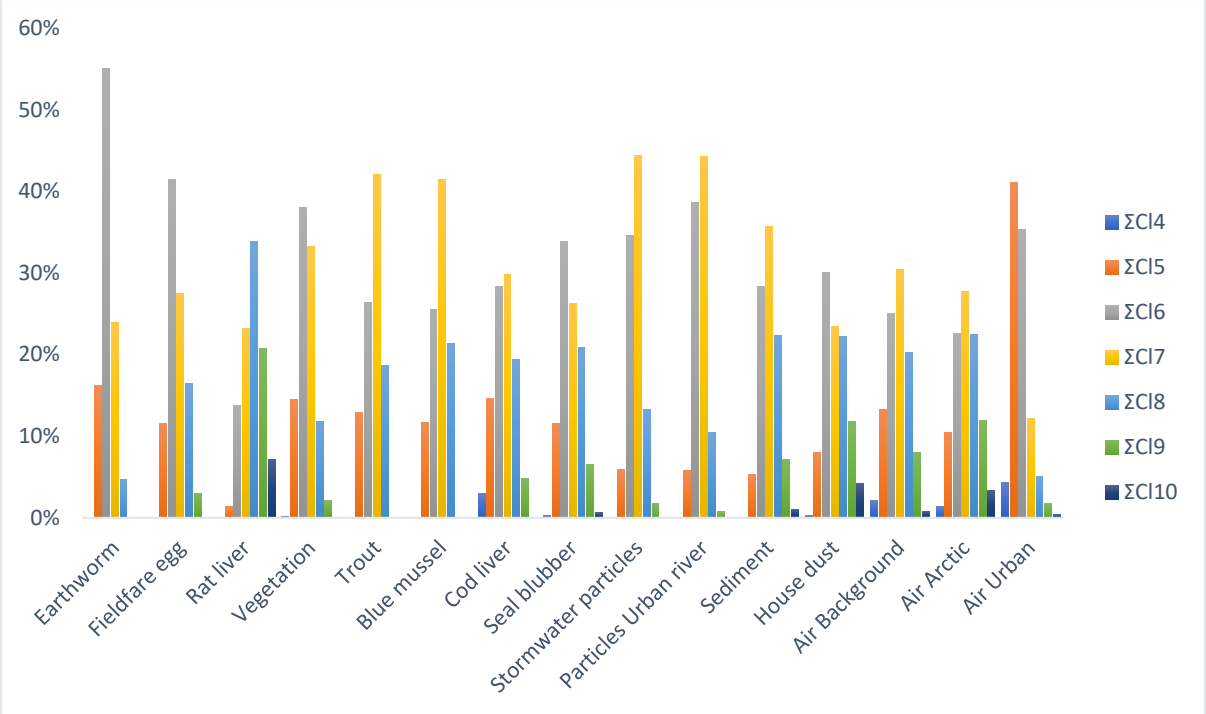

Figure 3: Distribution of chlorine content in the homologue groups detected in the environmental sample types included in the study.

Figure 4 shows chlorine content in the environmental sample types included in the study given as percentage. Highest average chlorination degree was seen for rat liver (56.3% Cl), while the lowest average chlorination degree was seen for urban air (50.9% Cl).

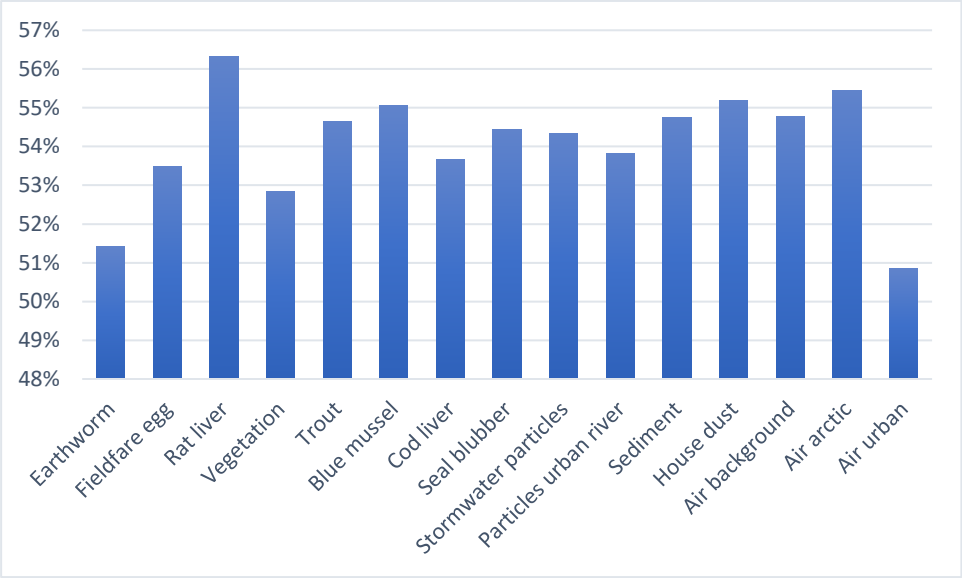

Figure 4: Chlorination degree of ΣPCAs $C_{14\text{-}17}$ in the environmental sample types included in the study given as percentage.

Table 1: Distribution of chain lengths (ΣPCAs C_{14}, ΣPCAs C_{15}, ΣPCAs C_{16}, ΣPCAs C_{17}), ΣPCAs C_{14-17} and chlorination degree in percentage for all environmental samples included in the study.

	ΣPCAs C_{14}	ΣPCAs C_{15}	ΣPCAs C_{16}	ΣPCAs C_{17}	ΣPCAs C_{14-17}	Cl degree
Terrestrial biota (ng/g ww)						**(%)**
Earthworm	35.3	26.0	19.0	6.3	86.6	51.4
Fieldfare egg 1	101.0	61.3	33.0	8.5	203.8	51.9
Fieldfare egg 2	28.7	17.2	8.7	2.6	57.2	52.6
Fieldfare egg 3	46.3	24.0	8.1	0.0	78.4	54.2
Fieldfare egg 4	37.3	22.8	7.3	0.0	67.4	55.2
Rat liver 1	186.3	276.1	327.9	150.9	941.1	56.3
Rat liver 2	246.5	823.1	690.7	432.8	2193.1	56.3
Vegetation 1	6.1	4.2	3.2	1.9	15.3	52.1
Vegetation 2	12.1	5.5	3.0	1.4	22.1	52.8
Vegetation 3	9.3	4.4	2.2	0.8	16.7	53.0
Vegetation 4	11.8	5.9	3.2	1.7	22.6	53.5
Aquatic biota (ng/g ww)						**(%)**
Trout 1	7.1	1.5	0.0	0.0	8.5	54.8
Trout 2	13.7	3.6	0.0	0.0	17.3	54.5
Trout 3	7.8	1.0	0.0	0.0	8.8	54.7
Blue mussel	9.6	0.7	0.0	0.0	10.3	55.1
Cod liver 1	128.0	69.7	24.1	11.9	233.7	53.8
Cod liver 2	285.5	214.8	32.6	43.9	576.8	52.0
Cod liver 3	66.2	42.4	10.8	5.5	124.9	53.9
Cod liver 4	23.0	6.0	0.0	0.0	29.0	55.0
Seal blubber 1	44.3	21.1	7.6	2.1	75.2	54.0
Seal blubber 2	37.6	18.0	4.5	1.0	61.1	53.7
Seal blubber 3	42.4	18.8	6.3	0.9	68.4	54.4
Seal blubber 4	39.7	17.6	5.7	0.0	63.0	54.1
Seal blubber 5	34.5	15.1	4.9	0.0	54.5	53.7
Seal blubber 6	31.8	14.4	5.3	0.0	51.5	54.8
Seal blubber 7	54.7	22.6	6.8	0.0	84.0	54.8
Seal blubber 8	72.0	22.5	2.6	0.0	97.2	55.8
Seal blubber 9	70.9	33.7	12.2	4.5	121.4	55.2
Seal blubber 10	57.4	22.5	12.1	2.5	94.5	54.0
Aquatic abiotic samples (particles: ng/L, sediment ng/g dw)						**(%)**
Stormwater particles 1	122.6	60.8	29.8	4.0	217.2	54.3
Stormwater particles 2	191.2	84.5	43.1	8.4	327.3	54.2
Stormwater particles 3	62.2	25.6	0.0	0.0	87.9	54.6
Stormwater particles 4	34.6	18.3	0.0	0.0	52.9	54.2
Particles urban river 1	90.5	50.0	27.4	8.0	175.9	53.9
Particles urban river 2	68.5	34.5	15.7	0.0	118.7	53.8
Particles urban river 3	53.7	23.7	10.5	0.0	87.9	53.7
Sediment 1	33.2	9.0	0.0	0.0	42.2	56.0
Sediment 2	85.8	76.9	47.3	27.3	237.2	53.0
Sediment 3	38.0	43.1	24.2	5.1	110.4	54.5
Sediment 4	**134.3**	**113.3**	**80.,9**	**49.3**	**377.8**	**55.5**

Indoor environment (ng/g)						(%)
House dust 1	787.8	408.0	365.6	96.9	1658.,3	54.6
House dust 2	2260.1	2130.3	611.8	269.0	5271.3	55.7
House dust 3	1328.5	575.4	325.5	167.0	2396.,4	54.4
House dust 4	2301.1	905.9	439.4	193.3	3839.8	56.0
House dust 5	2379.4	1013.7	391.0	170.1	3954.2	55.2
Atmosphere (pg/m^3)						**(%)**
Air background 1	54.0	26.0	11.1	2.0	93.1	53.8
Air background 2	200.6	36.2	13.8	5.6	256.2	55.9
Air background 3	86.8	18.0	4.0	0.0	108.7	54.7
Air background 4	97.4	21.2	6.9	1.7	127.2	53.4
Air background 5	655.6	28.8	6.2	2.3	692.8	56.1
Air arctic 1	512.8	60.7	17.3	8.0	598.8	56.4
Air arctic 2	411.1	50.6	18.8	8.6	489.1	55.5
Air arctic 3	217.8	68.2	28.8	13.2	328.0	57.6
Air arctic 4	267.7	216.7	133.2	67.9	685.6	55.5
Air arctic 5	140.0	104.,4	67.7	37.4	349.4	56.0
Air arctic 6	243.8	44.9	13.6	6.0	308.4	56.4
Air arctic 7	38.5	3.4	0.6	0.6	43.1	52.4
Air arctic 8	50.3	4.0	0.1	0.0	54.4	55.7
Air arctic 9	3.6	9.4	7.6	5.0	25.5	53.8
Air arctic 10	49.6	13.3	5.9	3.8	72.6	53.0
Air arctic 11	52.6	28.0	15.2	8.2	104.0	56.4
Air arctic 12	163.4	32.4	16.0	7.7	219.5	56.8
Air arctic 13	47.3	27.1	11.0	4.6	90.0	56.5
Air arctic 14	173.7	30.7	10.5	4.4	219.2	55.8
Air arctic 15	241.2	26.8	10.1	4.4	282.5	56.2
Air arctic 16	1551.0	153.7	55.6	23.0	1783.3	56.6
Air arctic 17	46.2	16.8	6.2	2.3	71.5	54.3
Air arctic 18	49.9	9.7	3.8	2.1	65.5	53.1
Air urban 1	365.9	65.,4	17.3	4.7	453.3	50.2
Air urban 2	151.4	23.9	6.2	1.6	183.1	49.3
Air urban 3	191.5	25.6	6.4	1.9	225.4	51.2
Air urban 4	256.7	42.3	14.8	6.1	320.0	52.6
Air urban 5	176.8	31.1	10.6	4.4	222.9	53.8
Air urban 6	323.9	45.9	9.4	1.6	380.8	50.7
Air urban 7	232.5	38.0	8.3	1.1	279.8	50.2
Air urban 8	729.3	75.4	11.9	0.4	817.0	49.8
Air urban 9	348.1	44.7	6.6	0.0	399.4	49.9
Air urban 10	768.6	94.3	15.0	0.9	878.8	50.6
Air urban 11	301.9	49.2	9.9	0.0	361.0	50.4
Air urban 12	394.3	55.4	12.2	1.2	463.1	51.6
Air urban 13	513.2	52.3	6.0	0.0	571.5	49.4
Air urban 14	280.5	58.0	23.4	9.9	371.7	52.4

4 Homologue group profiles of ΣPCAs C14-17

In this section, homologue group profiles in the sample types included in the study are presented in more detail. In general, the detected profiles of PCA homologue groups in environmental samples are influenced by i) analytical limitations (see further discussion on this in method section), ii) the homologue group profiles in sources, iii) fractionation of homologue groups via environmental processes, given varying rates for evaporation, condensation, sedimentation, degradation, etc., and additionally for biota, iv) varying rates of uptake for the different homologue groups from the environment, and v) varying rates of metabolism for the different homologue groups.
The calculated chlorination degrees of the environmental samples included the study are shown in Figure 4 and Table 1. Although there is variation between the sample types, all samples have average chlorination degrees well above the suggested limit for regulation, 45% Cl on average in technical mixtures. The following individual homologue groups included in this study has chlorination degree below 45%: $C_{14}H_{26}Cl_4$, $C_{15}H_{28}Cl_4$, $C_{16}H_{30}Cl_4$, $C_{16}H_{29}Cl_5$, $C_{17}H_{32}Cl_4$, and $C_{17}H_{31}Cl_5$. As can be seen from the figures below (Figure 5, Figure 6, Figure 7, Figure 8, and Figure 9), several samples, including biological samples, contain detectable levels of these homologue groups with chlorination degree below 45%.

4.1 Terrestrial biota

Figure 5 shows the homologue group profiles in samples from terrestrial biota from an urban area (Heimstad et al., 2023)(Heimstad et al., in prep). As indicated by the chain length and chlorination degree profiles presented above, the homologue group profile detected in rat liver differed substantially from other sample types by higher chlorination degree and longer chain lengths.

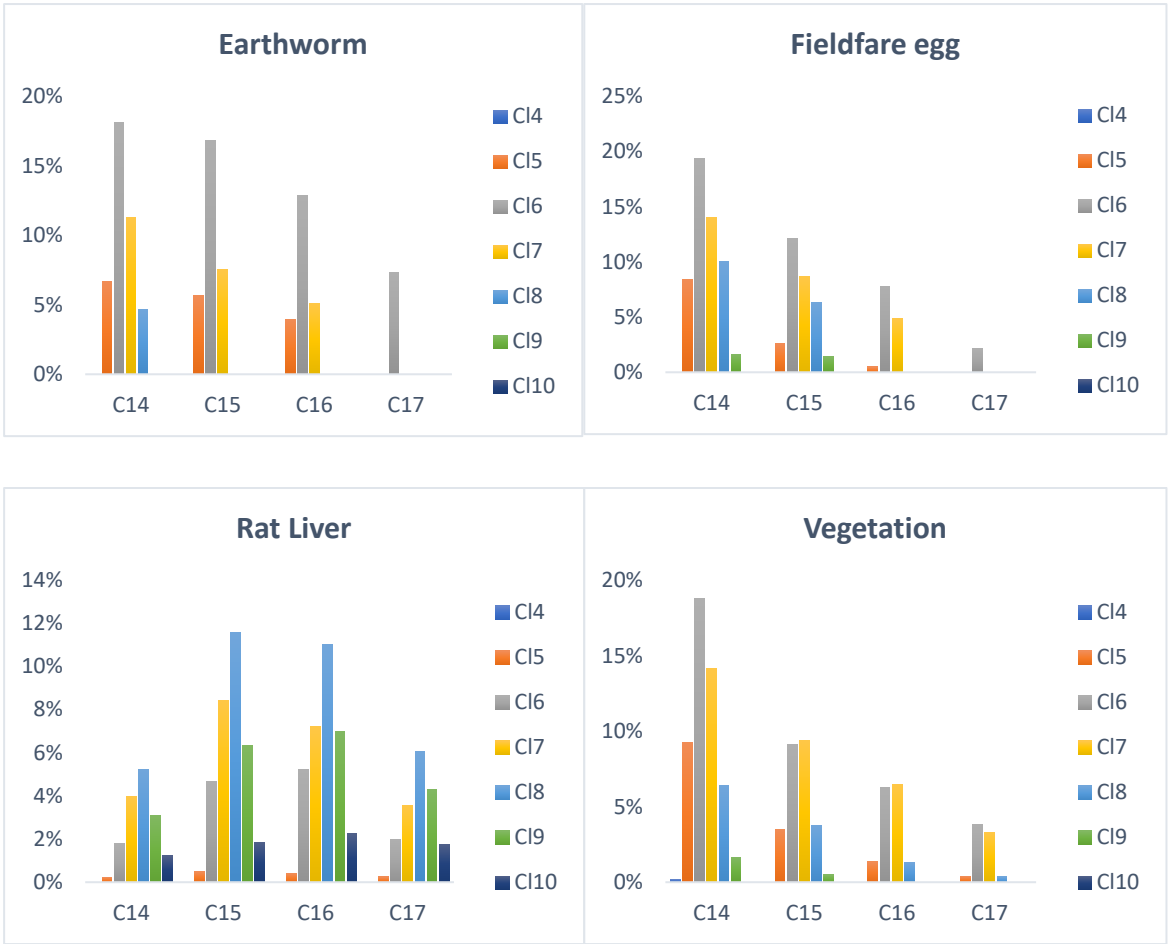

Figure 5: Homologue group pattern in terrestrial biota

4.2 Aquatic biota

Figure 6 shows the homologue group profiles in samples from aquatic biota from an urban fjord (Ruus, 2023)(Ruus et al., in prep). Cod liver and seal blubber (both sample types high in lipids) showed higher concentrations (Table 1) and more presence of longer chain lengths compared to trout muscle and blue mussel.

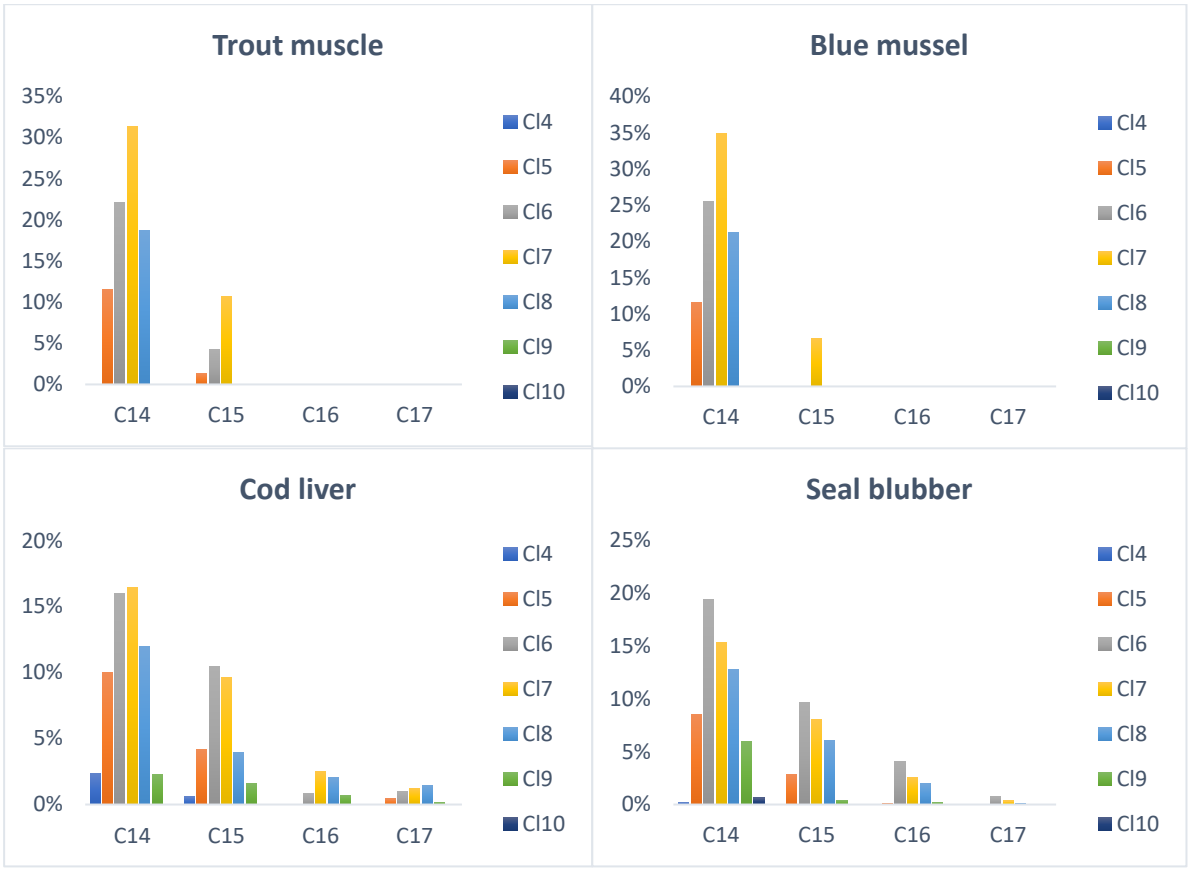

Figure 6: Homologue group pattern in aquatic biota

4.3 Aquatic abiotic samples

Figure 7 shows the homologue group profiles in samples from the aquatic environment near an urban area (Ruus, 2023)(Ruus et al., in prep). Sediments had a homologue group pattern dominated by longer chain lengths and higher chlorination compared to particles in water, although concentrations were comparable (Table 1).

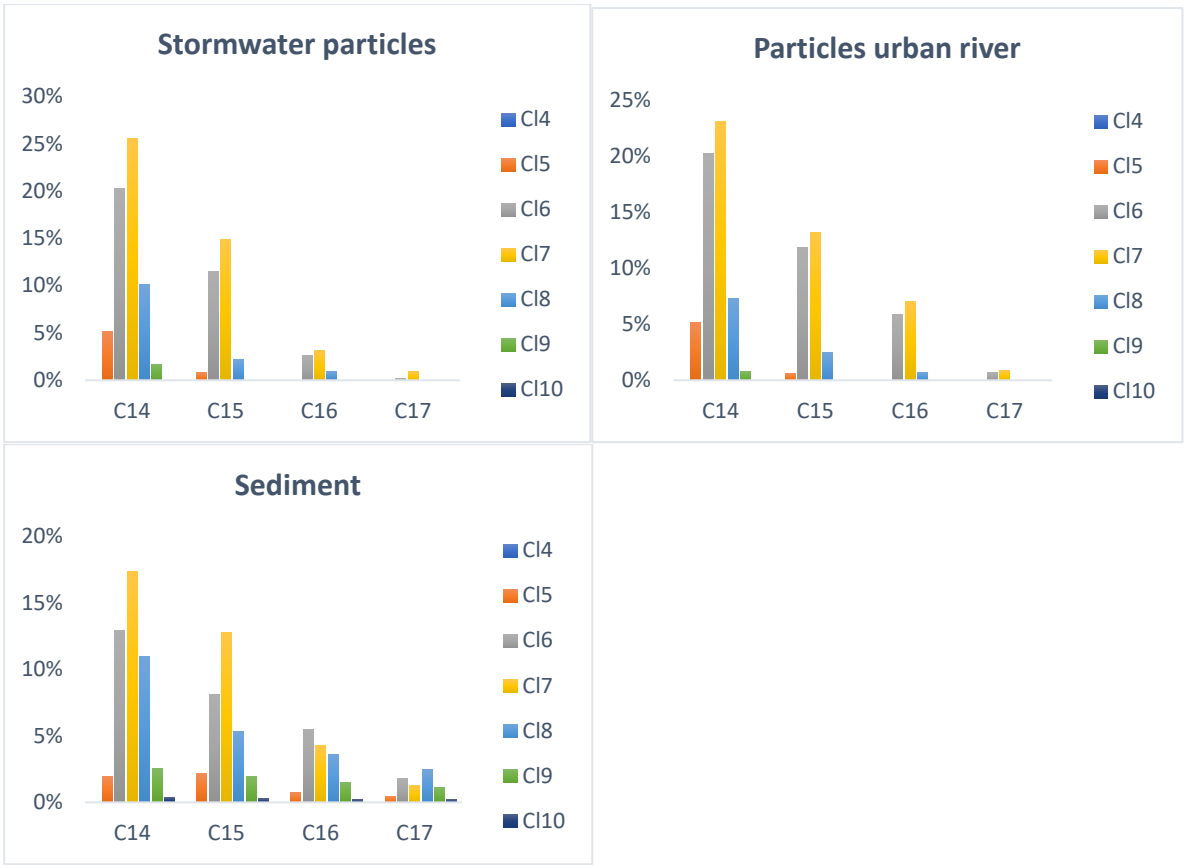

Figure 7: Homologue group pattern in aquatic abiotic samples

4.4 Indoor environment

Figure 8 shows the homologue group pattern in samples collected from the indoor environment (Heimstad et al., 2023). House dust showed very high concentrations of ΣPCAs C_{14-17} (Table 1).

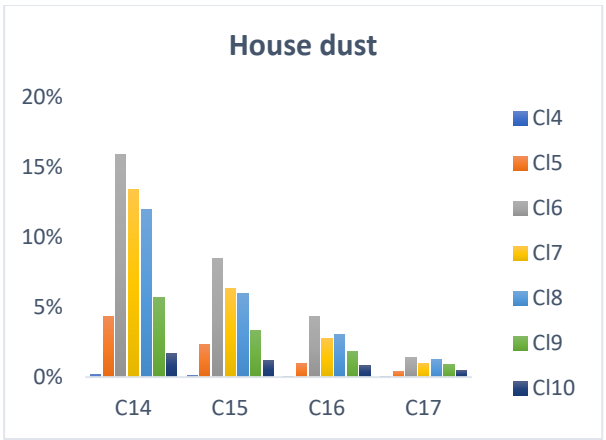

Figure 8: Homologue group pattern in house dust

4.5 Atmosphere

Figure 9 shows the homologue group pattern in atmospheric samples from an arctic area (Zeppelin observatory in Ny Ålesund, Svalbard), a background site (Birkenes observatory, Southern Norway), and an urban area (Sofienbergparken, Oslo) (Halvorsen et al., 2023)(Halvorsen et al., in prep). The air samples from the Arctic and from the background site showed presence of longer chain lengths and had a higher chlorination degree compared to the air samples from an urban area. The Arctic and background site are expected to have a larger influence of long-range transported PCAs compared to PCAs from local sources. In general, for long-range atmospheric transport, there is an expectation for a shift in pattern of organic contaminants towards more volatile contaminants with increasing distance from sources. However, for PCAs, the atmospheric half-life increases with increasing chlorination degree (Gawor & Wania, 2013). There is some overlap in volatility between the homologue groups with increasing number of chlorines (Figure 10).

It is not clear whether the observed homologue group pattern at the Arctic site and background site are a result fractionation to higher chlorinated homologue groups, or if the profiles are additionally affected by local sources and/or sample contamination.

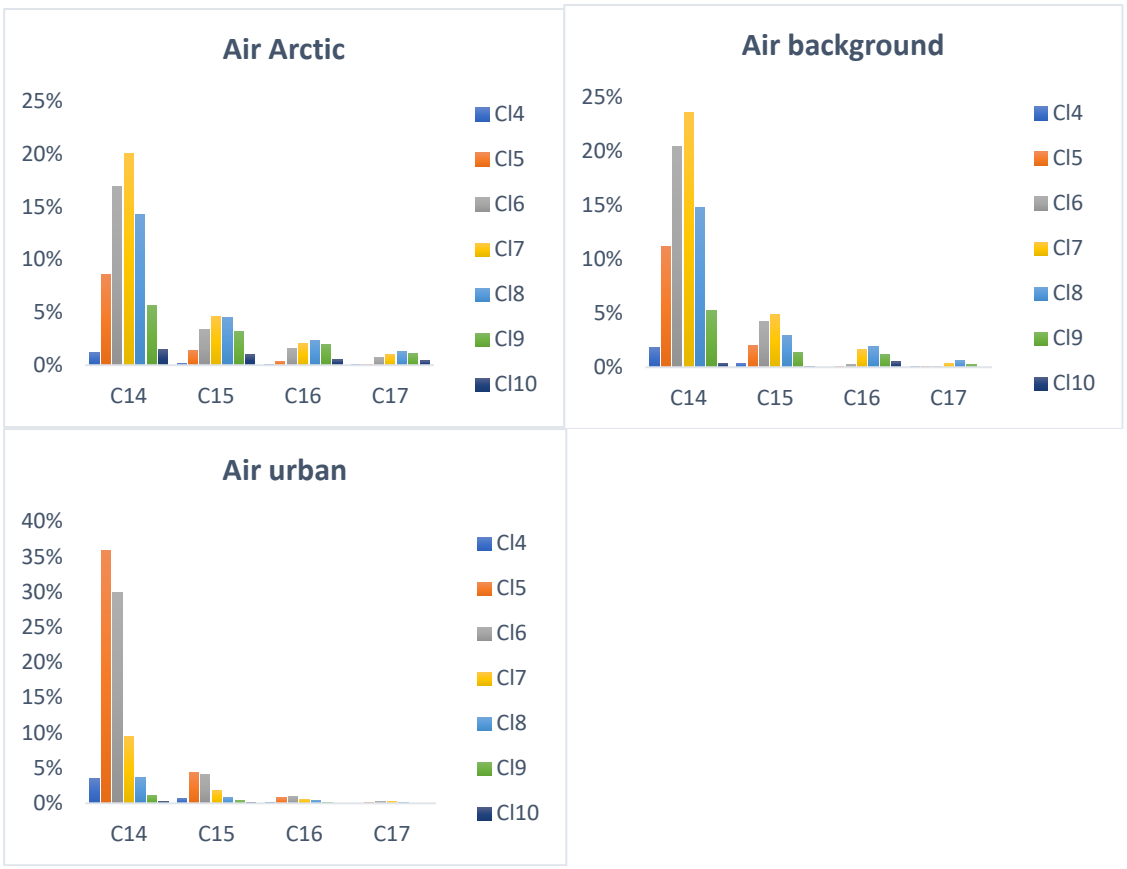

Figure 9: Homologue group pattern in atmospheric samples

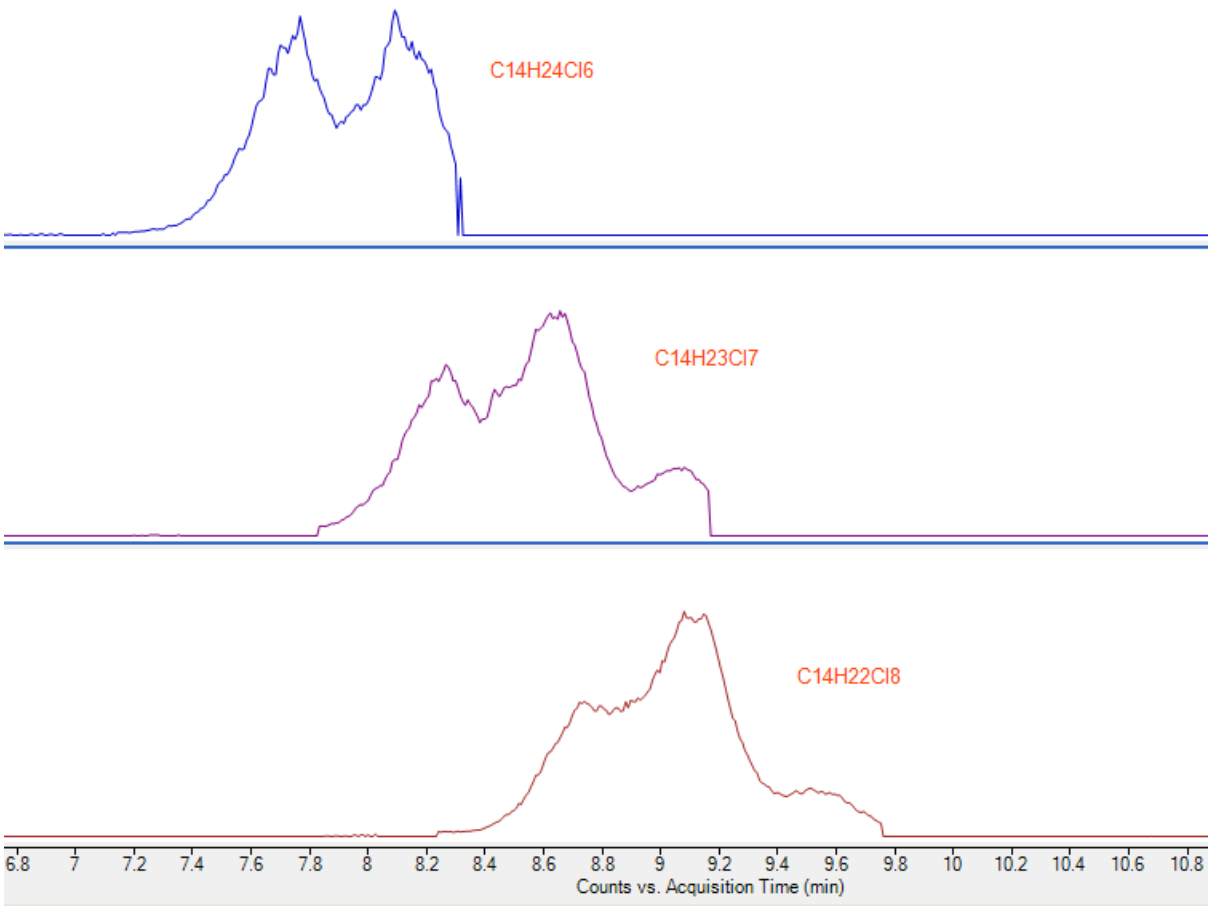

Figure 10: Extracted ion chromatogram of C14Cl6, C14Cl7, and C14Cl8, illustrating overlap in retention time (i.e. volatility) between the homologue groups.

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