



ARNIKA, CHEMSEC, CHEMTRUST, CLIENTEARTH, EEB, ECOS, IPEN and HCWH submission to the REACH restriction: PFHxA, its salts and related substances

25/09/2020

The undersigning organisations would like to provide further comments to strengthen the restriction proposal on the following topics:

- 1. The proposed concentration thresholds
- 2. Some of the proposed derogations
- 3. Aspects of recycling

1 Concentration threshold

1.1 Suitability of the proposed threshold

1.1.1 Level of the threshold

In earlier submissions into this public consultation, we have provided arguments why the thresholds of 25 ppb for PFHxA and of 1000 ppb for the sum of PFHxA-related substances is not justified. In short:

• There are indications of articles intentionally treated with PFAS at concentrations below 1000 ppb.

- A bulk concentration threshold as proposed here is fundamentally an inadequate choice for materials mostly used in surface treatment in articles. A value in μg/m² or mg/m² would likely be more purposeful and simpler.
- The text does not specify whether analytical results based on *total* or *extractable* fluorine are meant. Practical enforcement analysis will likely combine both because of their respective strengths and limitations.

The dossier argues that 1000 ppb is "feasible", but does not demonstrate that lower values would not be "feasible". Moreover, it does not justify what values would be relevant for practical purposes, i.e. in order to differentiate intentional use from contamination minimised through good industrial management practises.

1.1.2 Concept of the sum threshold

The substance scope of the proposed restriction relies on a useful definition based on chemical structures, known degradation patterns and related fate in the environment. The illustrative list of substances in scope is useful, as is the fact that substances meeting the structural criteria are in scope even if they are not included in the list.

Individual applications (whether mixtures or articles) generally **do not combine a high number of PFAS** (as substances). Consequently, the sum of all substances in scope will always be strongly dominated by very few individual substances.

- Formulations for fire-fighting foam concentrates generally rely on one or two main ingredients the fact that only few fluorosurfactants are registered under REACH limits the use of non-registered fluorosurfactants to niche applications or to low-percentage ingredients in high volume formulations.
- Side-chain fluorinated polymers used to coat textiles, paper or building materials are also based on few individual fluorinated monomers in scope.¹

Concentration thresholds per molecular species (as used in restriction #73 or as initially proposed for what is now restriction #68; see also section 3.2) are therefore likely simpler and more effective ways to regulate and enforce.

1.2 Analytical sensitivity

It has been suggested that 1000 ppb is the detection limit of a so far unidentified technique. This sounds implausible, or unlikely to be the state of the art in analytical chemistry:

Restriction #68 on C8-based PFASs, initially (in 2014) proposed 2 ppb as a threshold for each single substance in scope. The RAC opinion of 2015 identified no substantiated doubts on LoD or LoQ, nor on false positives before increasing the threshold to an impressive 1000 ppb for the sum.

2

¹ Suitable registered monomers in scope are:

[•] for meth(acrylate) side-chain fluorinated polymers: EC 241-527-8, 218-407-9, 801-260-5 and 812-877-4 (the latter being able to degrade into PFHxA despite being based on 6:3 rather than 6:2 fluorotelomer)

[•] for siloxane polymers: EC 257-473-3, 288-657-1, 278-947-6, 277-551-0 (see also restriction (#73)

[•] for polyaddition polymers, e.g. based on isocyanates: EC 246-791-8, 811-523-6, 628-448-8. See <u>EEB's report</u> "PFASs: Avoiding the streetlight effect" for a more reader-friendly illustration.

- the C9-C14 PFCAs restriction², proposed in 2018 and quoted in the 2nd opinion, proposes a sum threshold of 260 ppb. The dossier clarifies (footnote 6) that "one EU producer confirmed [...] 260 ppb is achievable [...]", indicating that analytical possibilities routinely reach quantification limits well below 260 ppb for high numbers of individual species.
- Restriction #73³, decided in 2019 and covering mixtures containing some PFHxA-related substances (FTOH 6:2-based silane derivatives), applies a threshold of 2 ppb based on a limit of detection (recital (2) of Commission regulation 2019/957 and several instances of the RAC opinion).

Analytical techniques had good capabilities even a few years ago, and they have improved rather than deteriorated over the years.

We would like to remind the reader that in some sectors, such as outdoor clothing (see also our earlier submission into this public consultation), many companies have successfully transitioned away from fluorinated surface treatments. These companies have implemented tests they may use to check that purchased goods and materials meet their PFC-free specifications.

For these reasons, **we urge RAC to substantiate any claims** regarding LoDs and LoQs higher than 2 ppb for single substances or constituents in scope, and on matrix effects⁴ potentially leading to false positives or overestimated individual concentrations.

1.3 C6 is not C8

Comparing the restrictions on C8-related PFAS (restriction #68, aka "PFOA restriction")) and the present restriction on C6-related PFAS reveals a fundamental difference between the two regulatory measures to be mindful of.

1.3.1 Technology vs. constituents

To illustrate this difference, let us distinguish between C6 and C8 *technology* and C6 and C8 *individual constituents* (see schematic in Figure 3). This is a distinction that is especially relevant for PFAS synthesised by telomerisation, i.e. essentially all substances based on 6:2 or 8:2 fluorotelomers. This distinction is important because telomer-based synthesis does not only produce the intended PFAS, but the end product contains substantial fractions of longer and shorter PFAS oligomers.

- *Technology*: the industry switched from the C8 technology to C6 technology around fifteen years ago. This simply means that they changed from basing their products on C8 PFAS to C6 PFAS. C6 or C8 is reflected in the name used for the substance, e.g. EC 248-580-6, registered as "3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctanesulphonic acid"⁵ belongs to the C6 technology.
- Constituents: due to the telomerisation reaction mechanism, a distribution of chain lengths, spaced by 2 carbon atoms, will always be obtained. A substance from the C8 technology will

² Currently at the level of a proposal only, <u>link</u> to the background document to the Annex XV dossier. This proposal from 2018 covers the longer-chain perfluoro acids and "related substances" in a way analogous to this restriction proposal.

³ <u>ECHA's website</u>, Commission regulation 2019-957

⁴ A potential source of confusion is the use of the word "matrix effect". It can refer to effects from switching ionisation matrices in mass spectrometry techniques, but it can also refer to effects from the material matrix on or in which PFASs (or other analytes) are present (such as a polymer, a fabric, live tissues etc.).

⁵ The reader should not be misled by the presence of the string "octane" in the name. Of the eight carbon atoms ("octane"), six are perfluorinated, making it belong to the C6 technology.

therefore always contain mostly the C8 constituent, with variable amounts⁶ of C6 and C10 constituents, and likely traces of C4 and C12. Similarly, a substance produced using C6 technology will contain C4 and C8 (as well as traces of C2 and C10) substances in variable amounts.

Figure 1: schematic of the distinction between technology and constituents



1.3.2 Intention of the restrictions

3M famously phased out their C8 technology in 2003 and replaced it by C4 technology. In the following years, the shift from C8 to C6 technology took place among most manufacturers. As opposed to a common misconception, C6 fluorotelomer technology was largely available and accepted by 2010 and the bulk of the conversion from C8 fluorotelomer technology did not wait till the proposal of the C8 restriction in 2014, but had been completed earlier.⁷

- When the restriction on C8 (now restriction #68) was proposed in 2014, the dossier submitter proposed 2 ppb as the threshold per substance in scope (see also section 3.2). This was later adopted as 1000 ppb for the sum of PFOA-related substances, with the rationale in the RAC opinion (p. 26) being to " allow [...] <u>C-6</u> mixtures used as alternative substances" [emphasis added] and acknowledges C8 *constituents* being present as " unintentional impurities [in alternatives]" (p. 23).
- The restriction proposal on C9-C14 PFAS (Background Document, p. 25) also correctly describes the substances in scope as "unavoidable by-products" of manufacturing C8 and C6 *technologies*.
- The C8 restriction⁸ reads: "The aim of the proposed restriction is to stop all intentional use of PFOA and PFOA-related substances", i.e. the C8 technology.

⁶ The sum of the "other" chain length oligomers (i.e. all except the name of the *technology*) will not exceed 20% by weight, as fluorotelomer-based substances are generally registered as *mono-constituent substances*, in line with <u>REACH guidance</u>. The minor constituents are termed *impurities* under REACH, although they are expected and unavoidable reaction products. Oligomers present in a *mono-constituent substance* are always spaced by 2 units (C4-C6-C8) as the telomerisation acts by adding blocks of two perfluorinated carbon atoms (the monomer tetrafluoroethylene) to the CF₃-CF₂- "starter", referred to as a *telogen*. See also text in the Annex (section 3).

⁷ This is corroborated by several independent publications from 2005 onwards:

[•] the <u>Danish EPA</u> in 2005 "the alternative telomer alcohols mainly are based on a chain length of C6", related to firefighting foams, photographic applications, textiles and paper;

^{• &}lt;u>German UBA</u> in 2009 " The chemical industry has recently begun to make increased use of short-chain PFCs", in a report entitled "Do without perfluorinated chemicals and prevent their discharge into the environment";

^{• &}lt;u>EU Commission</u> in 2010, "the majority (75%) of fluorosurfactants in telomer based AFFF are based on six-carbon (C6) technology" (p. 57);

^{• &}lt;u>Buck *et al.*</u> in 2011 " the major global fluorotelomer manufacturers are making available [...] products [...] based on 6 (rather than 8) perfluoroalkyl atoms" (p. 519).

In addition to this no C8-based chemical has ever been registered under REACH. At the 2013 deadline (100-1000 t/y), two 6:2 FTOH-based (meth)acrylates (EC 241-527-8, 218-407-9) and the by far most important fluorosurfactant (EC 252-046-8) were registered (as non-phase-in substances).

⁸ RAC Opinion to the C8 restriction, p. 23.

Although not specified in the Annex XV dossier, we understand that the aim of the present restriction is to prohibit intentional use of PFHxA, its salts and related substances, i.e. the C6 technology.

In the light of the aim of the present C6 restriction, the thresholds must not be copied from the C8 restriction – the aim here is not to allow an impurity, but to restrict the main constituent.

Some may object that if 1000 ppb is the level of an impurity in the C8 restriction, then certainly the concentration of the main constituent must be higher – and that consequently, 1000 ppb is an appropriate threshold to ban intentional uses of the C6 technology. This argument is fallacious for at least two reasons:

- It was and has not been demonstrated since (at least not transparently) that 1000 ppb is the lowest possible level on C8 *constituents* allowing the continued use of the C6 *technology*. In fact it could well be that 500 ppb, 100 ppb or even 50 ppb would also work.
- The levels of PFAS used in treatments (or concentrations, for mixtures) vary strongly from one application to another; the variation is exacerbated by the dossier submitter's choice for a bulk concentration limit (in ppb) instead of a surface concentration limit (in mg/m² or μg/m²).

As the situation is complicated by the distinction (C6-C8) restriction/technology/constituent, we attempt to represent the situation schematically below.



Figure 2: schematic of concentration thresholds applied and practical consequences

This graph deserves a few words of explanation:

C10

- The schematic distinguishes between the restriction on C8 (#68) in its original and final definition, and the present C6 restriction on PFHxA and related substances;
- For each technology, typical ratios of C4-C6-C8-C10 constituents (as the main constituent or impurities) are represented schematically (see section 3 for further explanation and justification);
- The levels are not to be taken as exactly at scale; however, the reader may assume a logarithmic vertical axis (indicated by the yellow horizontal guide lines and numbers at the right);
- The level indicated as "useful level" and as advocated for by the co-signing NGOs is 2 ppb to ensure prevention of intentional use. The wording "useful level" is chosen on the reasoning that the restriction is useful when it prevents all intentional uses. If any other level is more

appropriate, then this must be determined transparently and based on scientific evidence, and that it allows to distinguish between intentional use and concentrations due to contamination, where all available measures have been taken to avoid contamination;

 The graph illustrates whether the consequences of applying a certain threshold (blue (C6) and red (C8) dashed horizontal lines) leads to allowing or banning certain mass concentration levels in certain technologies, and whether those practical consequences are in line or not with the aim of the restriction.

It appears from this representation that the **C8 restriction is more effective at allowing the use of C6 technology** (3rd from the left)⁹ than at restricting the use of the (anyway mostly phased out) C8 technology (4th from the left). Furthermore, the current restriction proposal appears to restrict only some, but far from all intentional uses in scope (2nd from the right).

We therefore reiterate our demand that the dossier submitter **should use an evidence-based approach**, and to **transparently document** what threshold (1st from the right; whether as a bulk concentration or a surface concentration) allows to **distinguish between intentional uses and concentrations linked to unintentional contamination** in the presence of best available management techniques to avoid such contamination. In the absence of such an evidence-based justification, we urge RAC to base the **thresholds on analytical sensitivity for each substance**, as in the original proposal of 2 ppb for the C8 restriction.

2 Derogations

2.1 Effectiveness in reducing the identified risks

The dossier submitter has provided estimates (annex to the Annex XV report p. 121-124) of how emissions between 2020 and 2040 evolve with respect to the existing pollution stock in 2020, in the scenarios of the proposed restriction and without a restriction. The proposed restriction will result in a substantial decrease of future emissions.

It is also clear that an (hypothetical) immediate and total ban of the substances in scope would not eliminate all future emissions, as emissions from articles already on the market would continue throughout their useful life. Equally, existing and future landfilling will contribute to emissions, as will future incineration. The proposed unlimited and time-limited derogations will also add to future emissions.

It is therefore important to assess what part of the future emissions in the restriction scenario can be attributed to the overall transition period (18 months) and to the various derogations.

2.2 PPE

The Montreal Protocol¹⁰ defines essential uses as those that are necessary for health, safety or the functioning of society; and for which there are no available technically and economically feasible alternatives. The annex to the Annex XV dossier (p. 181, p. 193), lists the legally correct types of PPE for which the essentiality criterion is fulfilled, based on the definitions in Regulation 2016/425. Derogations

⁹ This is confirmed in the RAC opinion to the C8 restriction, on p. 26.

¹⁰ In Decision IV/25.

should clearly be limited to these types of PPE. The summary on p. 4 and 5 of the Annex XV dossier, right column, 9.(b) also states the categories with the correct legal reference.

We urge the dossier submitter, RAC and SEAC to ensure this limitation is systematically added when a "derogation for PPE" is mentioned.

2.3 Fire-fighting foam

In June 2020, DG Environment and ECHA released a report on <u>The use of PFAS and fluorine-free alternatives</u> <u>in fire-fighting foam</u>, a state of the art report developed using information from a wide range of experts in the field. It is important to note that some of the derogations and timelines proposed in the PFHxA restriction are not in line with this report, clear justifications for some of the proposed derogations such as the defence applications are lacking, and there are several provisions that are impossible or very difficult to monitor and enforce.

2.3.1 Derogations for defence applications

Derogations are proposed for the defence applications that include seagoing units, air traffic facilities and storage of fuel, and for training purposes provided that emissions occur in enclosed areas and wastewater is collected and disposed of safely. However, why these defence uses should be considered separately and as having different requirements than similar non-derogated uses is not explained or justified. Indeed, the ECHA report in its analysis of technical feasibilities of alternatives states on page 129 that "the military applications are similar to those seen in airports and municipal fire brigades and the [fluorine-free] foam used are, after the necessary testing and adjustment of equipment, considered to be useful for military equipment as well".

This is supported by section 7.5. of the report that provides details on PFAS-free foams and their compliance with international performance standards. Looking at the individual defence uses, certified, PFAS-free foams seem to comply with the performance requirements for these uses. The only standard not met with PFAS-free foams is the *U.S. military specification for firefighting foams* (MIL-F-24385F), that was developed before effective fluorine-free alternatives were widely available on the market, which explicitly mandates PFAS. Tests according to this standard for airports conducted already in 2009 at Copenhagen Airport showed that fluorine-free foams performed just as well as PFAS foams also for this standard (see ECHA report page 123).

The ECHA report also describes in the same section that "a wide range of PFAS-free foams are marketed for marine applications and it has not been indicated by any stakeholder that there might be particular challenges in changing to PFAS-free foams apart from the general need for adjustment and testing of equipment". It should be presumed that the seagoing units included in the proposed derogation should therefore have PFAS-free alternatives unless information and justification for this derogation is provided. It should also be noted that Germany reported defence application use of PFAS foams for training purposes regarding fire-fighting on ships. While the derogation clearly specifies that all releases should be contained, it is difficult to see how that is possible on a ship given the large amount of liquid typically used and how monitoring and enforcement would be conducted.

Further reason to not approve these derogations is provided by the evidence that military bases are one of the most common sources of high PFAS contamination around the world. In the US, more than 600

military sites and surrounding communities was identified by the Department of Defence as potentially contaminated with perfluorinated chemicals¹¹ and examples in the EU include Germany, Sweden and the United Kingdom¹²

2.3.2 Transition times

The transition times proposed are not supported by the conclusion of the ECHA report, see page 194:

- No transition time may be needed for training and testing purposes
- Except for oil, gas and petrochemical industry, all other applications are expected to being able to shift in 3-6 years
- It should especially be noted that the time-unlimited derogation for defence applications proposed is considered a 3-6 year transition in the ECHA report, further supporting the conclusion that this derogation is not justified. In fact, the derogation caveat that "as long as no successful transition to military operable fluorine free foams can be achieved" can be considered already achieved, noting that several countries have already phased out PFAS-foams for this purpose. This would also save time and resources by making point 12 of the restriction proposal redundant since no further review is needed.
- The ECHA report suggests 10 years as a feasible time for the oil, gas and petrochemical industry, as compared to the twelve years proposed in the Dossier. The report noted that the Norwegian oil and gas company Equinor phased out PFAS foams as the first company in the world in eight years and it could be feasible to presume that other companies can learn from the process already undertaken by Equinor.
- Finally, the proposed five year derogation for concentrated fire-fighting foam mixtures that are
 used or are to be used in the production of other fire-fighting foam mixtures does not include
 any restriction on use, which means that it is likely to be released directly into the environment as
 well as risking continued exposure for the fire-fighters using that foam. Noting the emission
 calculations provided in this submission, it is even more important that these foams are instead
 recalled and disposed safely in line with the Extended Producer Responsibility Principle.

2.4 Recycling

While not proposed as a derogation, there are indications that the issue of excluding recycling from the restriction has been considered. We strongly support the Dossier Submitter in their view that recycling should be included in the restriction to avoid further releases to the environment.

Having recycled materials excluded from the restriction cannot be justified. Greaseproof paper, clothing, carpets and furnishing are likely sources of PFHxA in the municipal waste. Following the recycling targets without having PFHxA-emitting materials excluded from the recycling process will lead to exposure risks mainly for workers and consumers, but also for communities living in the surrounding of the recycling plants. Taking into account the fact that carpets and paper are shredded and grinded before being provided to manufacturers for recycling, workers may be inhaling and ingesting PFAS since the initial moment of the recycling process. The same mechanism is responsible for the elevated levels of flame retardants in workers in carpet pad or e-waste recycling facilities¹³. Moreover, the recycling of material

¹¹ <u>https://www.ewg.org/release/pfas-could-contaminate-more-600-military-installations-dod-says</u>

¹² <u>https://www.eea.europa.eu/themes/human/chemicals/emerging-chemical-risks-in-europe</u>

¹³ W.A. Stubbings et al. (2014), Environment International, 71, 164-175.

emitting PFHxA and related substances will lead to contamination of new products and losing control over PFHxA/PFAS circulation in the product-waste cycle. If the life-span of carpeting is about 15 years, PFHxA will hold the presence in consumer products for an unjustifiable long time (15+ years) after having the restriction enforced. The mentioned effects are not hypothetical, this scenario became true when recycling exemptions and double standards for virgin and recycled items were applied for SVHC brominated flame retardants¹⁴. The authors came to the conclusion that e-waste plastic recycling led to contamination of new products designed for sensitive parts of society (i.e., children toys, hair accessories and kitchen utensils). Having highly toxic chemicals including PFHxA circulated in recycled products decreases credibility of recycling and circular economy and may lead to avoidance of recycled products by responsible consumers.

Several studies demonstrated that the recycling sector belongs to significant sources of PFHxA emissions into the environment. Kim *et al.*¹⁵ made the comparison of PFAS chemical profiles of surface waters and came to the conclusion that PFHxA was found in significantly higher concentrations in surface water flooding in the vicinity of e-waste recycling site than in the vicinity of municipal dump site or reference rural areas. The highest concentrations of PFHxA were found in the leachates from the municipal dump site. Taking into account the fact that the study was located in Vietnam, where we can expect a lower rate of separation of textile and paper from landfilled waste than in Europe, we might expect a higher proportion of PFHxA emission from recycled materials in the EU than in Vietnam. Zhang *et al.*¹⁶ tested human serum of elderly people living near recycling and reference areas in South China. The serum concentrations of PFHxA were significantly higher in elderly people living near e-waste areas than those in reference areas. Association between PFASs exposure and personal health history showed that body mass index (BMI) was significantly positively associated with serum PFHxA, suggesting that PFHxA exposure is associated with adverse health outcomes in elderly population living around e-waste recycling sites. Despite both studies being conducted in Asia, the findings have direct connection to the European chemical policy as the countries of investigation are the importers of European waste.

3 Annexes

3.1 Origin of the impurities and purification

Impurities (e.g. C4 and C8 species in a C6 substance) can range from levels far below 1% to almost 50%. For all uses subject to REACH registration of the substance used, currently impurities may not exceed 20%, as all currently registered PFASs are registered as monoconstituent substances.

Information from companies producing, selling or using PFAS on the impurity levels is scarce. However, the independent literature provides useful analytical information, and basic theory polymer chemistry allows some further deductions. Let us consider the simplified synthesis route of a side-chain fluorinated acrylic copolymer (Figure 5).

Figure 5: simplified scheme of the synthesis of a side-chain fluorinated acrylic copolymer¹⁷

¹⁴ <u>Arnika (2018)</u>, Toxic Loophole: Recycling Hazardous Waste into New Products

¹⁵ J.-W. Kim et al. (2013), Environ. Monit. Assess., 185, 2909-2919.

¹⁶ <u>T. Zhang et al.</u> (2019), Environ. Sci. Technol. Lett., 6(3), 133-140.

¹⁷ Throughout this document, perfluorinated carbon atoms are indicated by a light green halo.



The five steps can be summarised as follows:

- R1 represents the telomerisation reaction, where a perfluoroalkyl iodide (PFAI) mixture with different even-numbered chain lengths is produced from perfluoroethyl iodide (CF₃-CF₂-I) and tetrafluoroethylene (C₂F₄). ¹⁸
- The PFAI mixture can be purified by distillation (P2 a physical separation, not a chemical reaction), (partially or essentially fully) removing undesired chain lengths (such as the 4:2 and 8:2 PFAIs in C6 technology). After this purification step, the oligomer ratios remain essentially fixed. Analytical results (based on FTOH mixtures) show that this distillation step is carried out with variable separation quality or potentially not at all.
- R3 is a two-step sequence to insert ethylene (C₂H₄) (i.e. the "2" in 6:2 FTOH) and to substitute the iodide with a hydroxyl group to give the fluorotelomer alcohol (FTOH) mixture.
- R4 is the subsequent functionalisation of the fluorotelomer alcohol to an acrylic monomer (in this example; in reality, many other chemical transformations can be carried out on the FTOH).
- R5 is the copolymerisation reaction in which the FTOH-based acrylic monomer is reacted with one or more co-monomers to yield a side-chain fluorinated polymer.

The distribution of the different chain lengths can be impacted in two ways:

- The stoichiometry of reactants and the reaction conditions in R1 will determine whether e.g. the C6 or the C8 species will be the dominant species, and which will be relative proportions of lower and higher oligomers.
- Unwanted oligomers can be removed by distillation at reduced pressure (P2).¹⁹ It should be noted that the technically useful oligomers (such as the C8 and higher oligomers) are possibly sold to regions where this is not restricted or enforced.

The other reaction steps keep the relative distribution of oligomers unchanged; due to its reactive nature the acrylic monomer is less amenable to purification by distillation, or by any other classical large-scale purification technique.

3.2 Typical impurity levels

In Figure 4, we have graphically represented concentrations of the next-highest oligomer (i.e. a C10 constituent in C8 technology, or a C8 constituent in C6 technology)) of around 10% of the total mass. Lower oligomer levels (esp. the C4 oligomer in C6 technology) is likely lower as 4:2 fluorotelomers have been reported to have substantially poorer technological properties than 6:2 species. The 4:2 oligomer

¹⁸ The real reaction passes through a perfluoroalkyl iodide, in which the oligomer distribution is already defined. For more details, the reader is referred to <u>Buck *et al.* (2011)</u>, p. 519.

¹⁹ The lower oligomers are the most volatile; according to analytical chemicals supplier Alfa Aesar, for perfluorohexyl iodide bp = 117 °C and perfluorooctyl iodide bp = 160-161 °C.

can be minimised by optimising the reaction conditions and stoichiometry in R1 (Figure 5) and is conveniently removed by distillation in R2b, being more volatile than the 6:2 FTOH.

To get a realistic picture of the oligomer distribution in C6 technology, we have analysed results from the recent peer-reviewed literature.²⁰ We have found that on average (22 samples, see screenshot from excel file below – the file can be obtained from EEB), the C8 oligomer represents 11% of the total of C6 and C8 oligomers. However, there is also a broad spread in the impurity levels, ranging from unquantified concentrations to an impressive 47%.

1 Based on reported concentrations of fluorotelomer alcohols in articles from the following sources: Image: Concentrations of fluorotelomer alcohols in articles from the following sources: Image: Concentrations of fluorotelomer alcohols in articles from the following sources: 0 Number of sample with pure sources of 0.06.043 Image: Concentrations of fluorotelomer alcohols in articles from the following sources: Image: Concentrations of fluorotelomer alcohols in articles from the following sources: 2 Centermel 0 http://dx.doi.org/10.1016/j.chemosphere.2016.06.043 Image: Concentrations of fluorotelomer alcohols in articles from the following sources: 4 Rabid 0 http://dx.doi.org/10.1016/j.chemosphere.2016.06.043 Image: Concentration of fluorotelomer alcohols in articles from the following sources: 9 Onlycases with a dominant C6 oligomer are reported here: Image: Concentration of fluorotelomer alcohols in alcohol in alco		A	В	С	D	E	F	G	H		J	K	L	M	N	
Number of samples with pure 6:2 FTOH Sample identifiers Number 6:2 FTOH Sample identifiers 6 Gremmel Fobal 0 http://doi.org/10.1016/j.chemosphere.2016.06.043 bittp://doi.org/10.1016/j.chemosphere.2016.06.043 fobal - 6 Vestergren Van der Veen 12 TR, JL, JB, tht/, Bittp://doi.org/10.1016/j.chemosphere.2020.126100 former veen - - 9 Only cases with a dominant C6 oligomer are reported here: - - - 11 CAS 2043-47-2 647-462-7 678-39-7 865-86-1 8:2/6:2 8:2/6:2 11 CAS 2043-47-2 647-462-7 678-39-7 865-86-1 8:2/6:2 8:2/6:2 12 Author sample FTOH 4.2 FTOH 4.2 FTOH 4.2 FTOH 4.2 13 13 Gremmel 17 18:6 3:88 1:34 0.00 1.01 =- 14 var der Veen 2b 12:0 1:3 0.03 =- - - 14 var der Veen 2b 12:0 1:3 0.03 0.03 =- - - 10:0 -	1	Based on reported	concentrat	tions of fluo	rotelomer al	cohols in ar	ticles from	the followin	g sources:							
Comment O http://dx.doi.org/10.1016/i.chem.os.phere.2016.06.043 6 Note: 0 http://dx.doi.org/10.1016/i.chem.os.phere.2016.06.043 6 Murtaz 2 FA5, FA10 fttps://dx.org/10.1016/i.chem.os.phere.2020.128100	2		Number of samples with pure 6:2 FTOH	Sample id	entifiers											
Robel 0 https://doi.org/10.1021/acs.set.7b.02080 5 Vestergen 12 F8, J4, J3, http://dx.doi.org/10.1016/j.etmosp.tere.2020.128100 6 Muntaz 2 FA6, FA101185//dx.org/10.1017/s1178:30:8-1145-0 7 van der Veen 1 5b https://dx.org/10.1007/s1178:30:8-1145-0 9 Only cases with a dominant C6 oligomer are reported here: Ratio Ratio 8:2/(6:2) 9 Only cases with a dominant C6 oligomer are reported here: 0 Average Ratio 8:2/(6:2) 9 Only cases with a dominant C6 oligomer are reported here: 0 Ratio 8:2/(6:2) 8:2/(6:2) 10 CAS 2043-47-2 647-42-7 678-39-7 865-86-1 Ratio 8:2/(6:2) 8:2/(6:2) 13 Gremmel JT 18.6 3.86 0.14 9.7 0.16 0.11 14 Van der Veen 2b 1.32 0.01 0.14 -4VERAGE/(15.36) 15 3b 6.8 18 14 0.26 0.21 17 8b 68 18 <td>3</td> <td>Gremmel</td> <td>0</td> <td></td> <td>http://dx.doi</td> <td>ora/10.101</td> <td>6/i.chemos</td> <td>phere 2016</td> <td>.06.043</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	3	Gremmel	0		http://dx.doi	ora/10.101	6/i.chemos	phere 2016	.06.043							
5 Vestergren 12 T8, 11, 13, http://dx.obi.org/10.1016/ii.envpol.2014.12.034 Mumtaz 2 FA6, FA10 https://doi.org/10.1016/ii.fac.htmosphere.2020.126100 Average 7 van der Veen 1 5b https://doi.org/10.1016/ii.fac.htmosphere.2020.126100 Average 9 Only cases with a dominant C6 oligomer are reported here: Average Ratio 8:2/(6:2 Ratio 8:2/(6:2 Ratio 8:2/(6:2 each Ratio Ratio <th< td=""><td>4</td><td>Robel</td><td>0</td><td></td><td>https://doi.o</td><td>ra/10.1021/</td><td>acs.est.7b</td><td>02080</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></th<>	4	Robel	0		https://doi.o	ra/10.1021/	acs.est.7b	02080								
6 Mumiaz 2 FA5, FA10.https://doi.org/10.107/s1/183-019-1145-0 7 van der Veen 1 5b https://doi.org/10.1016/ji.chemosphere.2020.126100 9 Only cases with a dominant C6 oligomer are reported here: Ratio Ratio 11 CAS 2043-47-2 647-42-7 678-39-7 865-86-1 8:2/(6:2 8:2/(6:2 8:2/(6:2 12 Autor sample FTOH 4.2 FTOH 8.2 FTOH 10.2 0.17 0.11 13 Germmel J7 15.6 3.68 1.34 0.01 0.01 =AVERAGE(/15.136) 14 van der Veen 2b 1.20 1.3 0.01 0.01 =AVERAGE(/14.3% 15 3b 4.5 0.12 0.38 0.03 0.03 =AVERAGE(/14.3% 16 7b 858 1.4 9.7 0.16 0.14 =AVERAGE(/14.3% 17 8b 668 18 14 0.26 0.21 18 10b 9.90 2.4 2.7 0.03 0.03	5	Vestergren	12	T8, J1, J3,	http://dx.doi	.org/10.101	6/j.envpol.2	014.12.034								
Van der Veen 1.5b https://doi.org/10.1016/j.chemosphere.2020.128100 Average 9 Only cases with a dominant C6 oligomer are reported here: Ratio Ratio Ratio 11 CAS 2043-47-2 647-42-7 678-39-7 665-86-1 Ratio 8:2/(6:2 8	6	Mumtaz	2	FA5, FA10	https://doi.o	rg/10.1007/	s11783-01	9-1145-0								
8 9 Orly cases with a dominant C6 oligomer are reported here: Image: CAS Ratio Ratio Ratio 8 8 CAS 2043-47-2 647-42-7 678-39-7 865-86-1 8:2/6:2 +8:2 8:2/6:2 +8:2 8:2/6:2 +8:2 8:2/6:2 +8:2 8:2/6:2 +8:2 8:2/6:2 +8:2 1 0.11 1 10 CAS 2043-47-2 647-42-7 678-39-7 865-86-1 8:2/6:2 +8:2 8:2/6:2 +8:2 8:2/6:2 +8:2 1 <td>7</td> <td>van der Veen</td> <td>1</td> <td>5b</td> <td>https://doi.o</td> <td>rg/10.1016/</td> <td>j.chemospl</td> <td>here.2020.1</td> <td>26100</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	7	van der Veen	1	5b	https://doi.o	rg/10.1016/	j.chemospl	here.2020.1	26100							
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11 CAS 2043-47-2 647-42-7 678-39-7 865-86-1 8:2/6:2 +8:2) 8:2/6:2 +8:2) 12 Author sample FTOH 4:2 FTOH 8:2 FTOH 8:2 FTOH 9:2 0.17 0.11 0.11 13 Gremmel J7 18.6 0.88 1.34 0.00 0.01 =AVERAGE(J14:J36 15 3b 4.5 0.12 0.38 0.03 =AVERAGE(J14:J36 16 7b 8b 66 14 9.7 0.16 0.14 =AVERAGE(J14:J36 17 8b 68 14 9.7 0.03 0.03 =AVERAGE(J14:J36 18 10b 90 2.4 2.7 0.03 0.03 =AVERAGE(J14:J36 19 11b 58.8 2.18 0.34 0.26 = = 28 Robel P5 18 16 15 0.99 0.47 = 28 Mumtaz FA1 1.18										Ratio	Ratio 8:2/(6:2		Ratio	Ratio 8:2/(6:2		
12 Author sample FTOH 4:2 FTOH 8:2 FTOH 10:2 FTOH 10:2 TOH 10:2 <thtoh 10:2<="" th=""> TOH</thtoh>	11			CAS	2043-47-2	647-42-7	678-39-7	865-86-1		8:2/6:2	+8:2)		8:2/6:2	+8:2)		
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25 Muntaz FA1 1.18 0.49 0.42 0.29 26 FA2 298.42 0.85 0.00 0.00 27 FA6 78.85 2.59 0.03 0.03 28 Vestergren T4 241.1 1.4 0.01 0.01 29 T5 69.99 1.3 0.87 0.02 0.02 30 T11 26.2 1 0.55 0.04 0.04 31 T12 18.2 2.4 0.98 0.13 0.12 32 T19 65.1 1.7 0.03 0.03 34 T20 0.71 25.2 0.9 0.04 0.06 35 Formula in cells above (example from the first row) =F15/E15 =F15/(E15+F15) 38 0 =F15/(E15+F15)	24		T8			14000	340	110)	0.02	0.02					
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27 FA6 73.85 2.59 0.03 0.03 28 Vestergren T4 241.1 1.4 0.01 0.01 29 T5 69.99 1.3 0.87 0.02 0.02 30 T11 26.2 1 0.55 0.04 0.04 31 T12 18.2 2.4 0.98 0.13 0.12 32 T19 65.1 1.7 0.03 0.03 33 T20 0.71 25.2 0.9 0.04 0.03 34 T21 22.3 1.4 0.06 0.06 0.06 36 Formula in cells above (example from the first row) =F15/E15 =F15/(E15+F15) 39 0 =F15/(E15+F15)	26		FA2			298.42	0.85			0.00	0.00					
28 Vestergren 14 241.1 1.4 0.01 0.01 29 T5 69.99 1.3 0.87 0.02 0.02 30 T11 26.2 1 0.55 0.04 0.04 31 T12 18.2 2.4 0.98 0.13 0.12 32 T19 65.1 1.7 0.03 0.03 33 T20 0.71 25.2 0.9 0.04 0.03 34 T21 22.3 1.4 0.06 0.06 36 Formula in cells above (example from the first row) 37 = F15/(E15+F15) 38 Set 15/(E15+F15)	27		FA6			75.85	2.59			0.03	0.03					
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112 16.2 2.4 0.36 0.13 0.12 32 T19 65.1 1.7 0.03 0.03 33 T20 0.71 25.2 0.9 0.04 0.03 34 T21 22.3 1.4 0.06 0.06 35 Image: state stat	30		111			26.2	1	0.55		0.04	0.04					
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x1.0 x1.0 1.11 0.00 0.00 35 36 Formula in cells above (example from the first row) =F15/E15 =F15/E15 38 9 =F15/E15 =F15/(E15+F15) =F15/(E15+F15)	34		T21		0.71	20.2	1.4			0.04	0.03					
S6 Formula in cells above (example from the first row) 37 =F15/E15 38 =F15/(E15+F15) 39	35		121			22.0	1.4			0.00	0.00					
37 =F15/E15 38 =F15/(E15+F15)	36									Formula ir	cells abov	e (exami	ole from the	first row)		
38 39	37									=F15/E15		- (onany				
39	38										=F15/(E15	5+F15)				
	39											,				

²⁰ The five sources used are: <u>Vestergren *et al.*</u> (2015), <u>Gremmel *et al.*</u> (2016), <u>Robel *et al.*</u> (2017), <u>Mumtaz *et al.*</u> (2019), <u>van der</u> <u>Veen *et al.*</u> (2020). In all of these studies, concentrations of PFAS in articles are reported: we used the concentrations of free 6:2 FTOH and 8:2 FTOH in articles and restricted our list to those articles in which both species are quantified, and where the C6 oligomer is the dominant one (i.e. which can be identified as C6 technology). Based on the reasoning detailed in section 5.1.1, oligomer distributions in the non-volatile and non-extractable species are expected to mirror those of the free fluorotelomer alcohols. This may lead to some overestimation of the C8 impurity, as free concentrations of FTOH 6:2 would decrease more rapidly than those of 8:2 FTOH as the former is more volatile.

Many samples contained only 6:2 FTOH, and no 8:2 FTOH was quantified (Vestergren (12 samples), Mumtaz (2), van der Veen (1)). This demonstrates that the level purification can be chosen rather freely by the manufacturer of the FTOH.