

2021 NORMAN network PFAS Analytical Exchange

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In collaboration with Aarhus University (DK), Finnish Environment Institute SYKE (FI), IWW Water Centre (DE), Norwegian Environment Agency (NO), Örebro University (SE), University of the Basque Country (ES), VITO NV (BE), Wageningen Food Safety Research (NL)

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Executive Summary

Per-and polyfluoroalkyl substances (PFAS) present a growing analytical challenge in the field of emerging contaminants, with scientists continuing to expand analytical methods to accommodate a growing number of substances. As part of the NORMAN networks' Joint Programme of Activities 2021, the Environment Agency (UK) has led a PFAS Analytical Exchange with laboratories both within the NORMAN networks' membership and beyond.

An exchange was conducted utilising a questionnaire-based approach over the Summer of 2021, which set out to understand topics such as which PFAS the laboratories are currently focusing on, current limits of detection for individual PFAS in different matrices, the analytical techniques currently being adopted, and the future direction which laboratories are planning on taking to better understand environmental exposure of PFAS going forwards.

This technical report draws together the current understanding on PFAS analytical capabilities and progress from the responses received from 57 respondents from 20 Countries. Responding laboratories provided insights as to whether they were research laboratories, or commercial laboratories.

The report collates current capabilities on a wide variety of approaches utilised including Liquid Chromatography Mass Spectrometry (LC-MS), Sum of PFAS, PFAS-Total, non-target screening (NTS) approaches, Total Oxidizable Precursor (TOP) assays, Total Fluorine (TF) and Extractable organofluorine (EOF). The findings should provide a useful baseline at this point in time, for future collaborations on PFAS being conducted by the NORMAN network.

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1. Introduction

Analysis of PFAS presents a considerable challenge for many reasons, which includes but is not limited to the number of substances encompassed in the definition (~6,330 PFAS (US EPA, 2020)), their intrinsic properties, unknown intermediates, concentrations in environmental matrices, and the availability of certified reference standards. Knowledge exchange between all stakeholders is required to identify the Best Available Technologies (BAT), practices and techniques in analytical quantification and identification. This would ultimately improve confidence that data sets can be compared and benchmarked on a global scale and would aid subsequent risk assessment.

In June 2020, the Organisation for Economic Co-operation and Development (OECD) hosted a webinar on the latest developments in analytical and monitoring methods for PFAS in the global environment, biota and products to enable actions to reduce environmental and human exposure. In 2021, the OECD reconciled the definition of PFAS as substances with at least a single fully fluorinated methyl or methylene carbon atom, without any H/Cl/Br/I atom attached to it (OECD, 2021). In September 2021, the German Federal Institute for Materials Research and Testing (Bundesanstalt für Materialforschung und -prüfung, BAM) hosted a workshop on Advancements of Analytical Techniques for PFAS. Polls at the start of this workshop focused on 'Which PFAS analytics method were attendees using?' and 'What are the challenges in PFAS analytics?' The responses gave some early insights into the level of use of different PFAS analytical activities by attendees.

Development of validated methods for specific matrices are underway e.g., US EPA. On a global scale there is a lot of focus on developing robust, precise, and accurate methodology. Interest in advances spans national and local regulatory, academic, NGOs and industrial organisations. There is value in the knowledge exchange of practice and experience for this shared challenge to limit duplication, maximise best practice and increase our understanding of PFAS exposure via the environment.

The aim of this activity was to foster knowledge exchange on the analytical and monitoring approaches other countries are taking to detect and measure PFAS in the environment, utilising a questionnairebased approach. This report sets out the findings from this activity.

2. Objectives

The PFAS analytical exchange set out to begin to explore the below information:

- Which PFAS are organisations focussing on? Why? How have these been prioritised?
- What analytical techniques are currently being used or developed? Which environmental media are they suitable for? Any limitations and advantages of different techniques and methodology? Detection limits?
- Where are the gaps in capabilities? Are there other stakeholders outside of the NORMAN network who could inform this exchange and process?

Expected outcomes to inform organisations' own PFAS method development:

- Understanding the work of others and their interest or focus in this area
- Better informed analytical development and identification of best practice
- Identification of potential opportunities for collaboration

3. Methodological Approach

On 10th May 2021, the PFAS Analytical Exchange Activity was launched seeking volunteers to establish membership of a Steering Group to undertake this activity. Following a positive response from the NORMAN network membership, the Steering Group comprised:

- Anna Kärrman, Örebro University (SE)
- Audun Heggelund, Norwegian Environment Agency (NO)
- Belén González-Gaya, Marine Station of Plentzia / University of the Basque Country (ES)
- Griet Jacobs, VITO (BE)
- Kerry Sims, Environment Agency (UK)
- Leendert Vergeynst, Aarhus University (DK)
- Noora Perkola, Finnish Environment Institute (FI)
- Robert Carter, Environment Agency (UK)
- Stefan van Leeuwen, Wageningen Food Safety Research (NL)
- Ulrich Borchers, IWW Water Centre (DE)

The Steering Group met for the first time on 9th June 2021. In a series of virtual meetings, the Steering Group collaborated to adapt and modify a questionnaire utilised previously in 2021 by the Environment Agency with UK laboratories to fulfil the NORMAN network needs for this activity.

An initial version of the questionnaire was trialled by a Sounding Board from the NORMAN network. Feedback to improve the questionnaire was kindly received from:

- Sara Valsecchi, Water Research Institute (IT)
- Yann Aminot, IFREMER (FR)
- Jan Koschorreck, UBA (DE)

The questionnaire was finalised incorporating feedback where possible and circulated to the NORMAN network membership for participation in the Analytical Exchange activity on 28th July 2021. The final circulated PFAS analytical questionnaire can be seen in Appendix 1.

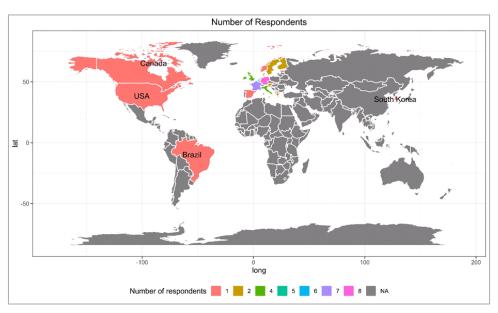
The questionnaire was additionally shared with the membership of the European Committee for Standardization (CEN) in Europe, and was also shared with the ~100 registered delegates for the PFAS workshop organised by BAM, Germany held on 1st September 2021 (Workshop Advancements of Analytical Techniques for Per- and Polyfluoroalkyl Substances (PFAS)).

A dedicated email account was set up to receive responses. A reminder of the opportunity to participate was sent to the NORMAN network membership on 6th September 2021. The initial deadline for questionnaire responses was set for 20th September 2021. The deadline for responses was extended on 22nd September until 30th September 2021.

The information and data resulting from the questionnaire responses were compiled and reviewed in October and November 2021, and the findings are summarised in the results section below. A workshop sharing the interim results of the activity was held virtually on 30th November 2021 with attendance possible for the 57 questionnaire participants. Additional feedback received from attendees led to some further information being presented in this report. The workshop was immediately prior to the NORMAN network General Assembly 2021, where an update on the activity was also given to attendees. This report will be published on the NORMAN network website and circulated to all questionnaire respondents.

4. Level of Response

By 20th September 2021, 36 responses had been received. The final response was received on 11th October 2021. Overall, 57 responses to the PFAS Analytical Exchange questionnaire were received. Responses were received from 20 countries, spanning four continents as shown in Figure 1. The responses reflect current laboratory PFAS analytical capabilities between 28th July 2021 and 11th October 2021.



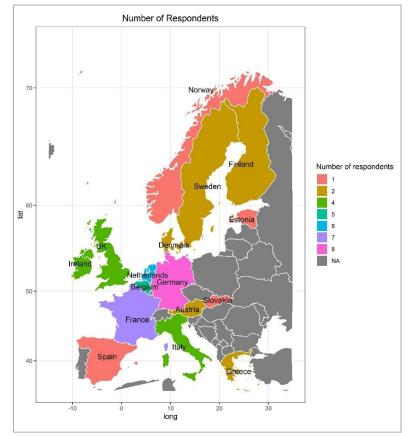


Figure 1: Global and European map showing the Countries from which questionnaire responses were received. Note that one respondent had multiple European analytical laboratories so the above reflects n=56.

5. Results

The below sections set out the interpretation of the results from the questionnaire. All results are anonymised so as not to identify any specific organisation. It should be noted, that on some related topics differing numbers of respondents provided information in different parts of the questionnaire, resulting in different percentages being presented depending on the section of the questionnaire from which the information was retrieved. A summary list of acronyms referred to for different PFAS, with CAS numbers are included in Appendix 2 as a reference.

5.1 Matrices currently studied for PFAS

Of the 57 responses received to the survey, 36 were from research laboratories and six from commercial laboratories. Of the respondents, 14 represented laboratories with both commercial and research-based activities (while one respondent did not answer this question). Of the 57 respondents, 50 laboratories reported to currently analyse PFAS, while five will do so in the future. Out of the 36 research laboratories, nine (25%) are accredited for PFAS analysis, while four out of six (67%) commercial laboratories hold such accreditation. Collectively the respondents analyse ca. 33,000 samples for PFAS per year, on average 350 samples per year for commercial laboratories and 380 samples per year for research laboratories.

Figure 2 indicates the number of laboratories performing any kind of PFAS analyses of different matrices. Water samples are predominant, while analysis of PFAS in solid samples like sediment, soil and biosolids/sewage sludge are also frequently performed. With regards to biota, there are almost four times as many laboratories that measure animal samples compared to plant samples. Analyses of matrices such as air, flue gas and indoor dust are only done in a few cases. The group 'other' in most cases referred to blood serum.

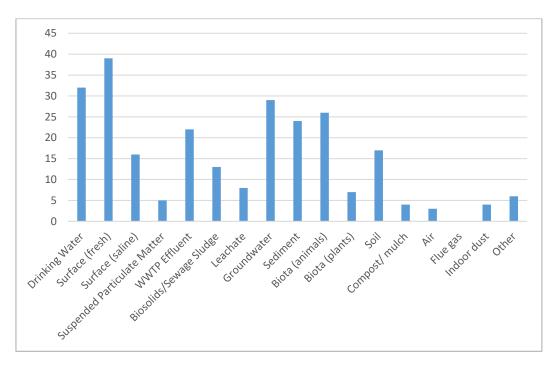


Figure 2: Number of responding laboratories performing PFAS analyses in different environmental matrices, in response to question 8 of the questionnaire.

5.2 Biota specific analysis

The species considered for PFAS analysis are of specific relevance in understanding bioaccumulation and biomagnification through the food chain to apex predators. This questionnaire gave an opportunity to explore the coverage of relevant species being studied for PFAS exposure by laboratories currently.

Some information on the number of laboratories analysing for biota was available from the information provided on LODs in respective matrices. This demonstrated 50% (26 out of 52) laboratories were analysing animals, and 13% (7 out of 52) laboratories were monitoring for plants, illustrating that there are almost four times as many laboratories that perform analyses of animal samples compared to plant samples.

On the specific survey questions focusing on biota (questions 14 and 15), there were 27 respondents to at least one of the questions on biota (50% of responding laboratories). Within this information the responses indicated a greater divide between the focus on animals and plants, with just 4% of respondents stating they analysed plants comparative to animals.

Table 1 shows a high-level breakdown of the different biota with the potential to be analysed for PFAS. The table highlights some potential key parts of the animal kingdom for which there was a complete absence of PFAS analysis by respondents for the classes: amphibians, jawless fish and reptiles. However, it is possible that respondents who stated their analytical methods for PFAS were suitable for various or multiple species, may be analysing species within these groups. Many respondents stated 'fish' in their responses without any further detail provided, as a result these responses are attributed to bony fish and for the purpose of analysis in this section have not been attributed to either freshwater or marine to avoid risking misrepresentation of the respondents' analytical focus.

Table 1: Number of respondents analysing different biota for PFAS (total n=27). This excludes four respondents who included some information on biota, but not species-specific information, and three respondents whose responses indicated 'various/multiple species'.

Kingdom	Classification	Class	Number of respondents	Percentage of respondents who undertake biota analyses
Animal	Vertebrates	Amphibian	0	0
		Bird	2	7%
		Bony fish	15	56%
		Cartilaginous fish	2	7%
		Jawless fish	0	0
		Mammal	6	22%
		Reptile	0	0
	Invertebrates		9	33%
Plant			1	4%

This led to further consideration as to whether the biota currently being analysed could be perceived as covering sufficiently the different environmental compartments, with analysis shown in Table 2. Examination of Table 2 indicates that overall animals analysed for PFAS in different environmental compartments comprises: in Freshwater: birds=0, bony fish=10, mammals=1, invertebrates=2; in Marine: birds=2, bony fish=13, cartilaginous fish=2, mammals=9, invertebrates=6; and, in Terrestrial-birds=2, mammals=6, invertebrates=2. This analysis indicated a general gap in birds with connections to freshwater environments, a fair representation of both benthic and pelagic species of fish in both freshwater and marine habitats, and showed that overall, the marine environment appeared to be the habitat for which there was the greatest diversity of species being analysed for PFAS by respondents. Freshwater invertebrates may currently be under-represented with no consideration for freshwater crustaceans such as crayfish. Alternatively, it is also possible that for any of the respondents who confirmed analyses of amphipods, plankton and mussels, where not explicitly stated whether freshwater or marine these may have comprised species associated with freshwater environments.

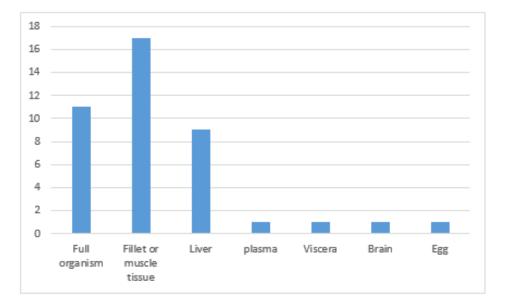
Animal group	Freshwater	Marine	Terrestrial
Birds	(0)*	Gull (1), Eagle	Eagle (1), Common buzzard (1)
Bony fish	Lake trout (1), forage fish (freshwater) (1), <i>fario trutta (1)</i> , <i>Squalius squalus (1)</i> , chub (2), loche (1), <i>Salmo trutta (1)</i> , roach (1), bream (2), sculpin (1)	Arctic cod (1), capelin (1), <i>Gobius niger (1)</i> , gobiiforme (1), sculpin (1) , <i>Liza</i> Sp. (1), <i>Mullus barbatus</i> (1), dab (1), plaice (1), flounder (1), whiting (1), eelpout (1), herring (1)	
Cartilaginous Fish		Shark (1), Spurdog (1)	
Mammals	Otter (2)	Ringed seal (1), seals (other species) (2), walrus (1), whale (1), beluga whale (1), porpoise (1), dolphin (2), otter (2)	Boar (1), fox (1), wolf (1), caribou (1), Arctic fox (1), polar bear (1)
Invertebrates	Amphipod (1), plankton (2) mussel (4)	Mussel (4), clam (1), benthos (1), plankton (2), amphipod (1), crustacean (1)	Earthworm (1), insect (1)

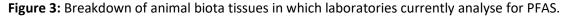
Table 2: Breakdown of animals analysed for PFAS in different environmental compartments.

*In brackets after each animal is the number of respondents which included this in their response. For those species which could feasibly be present in multiple environmental compartments they are included in all relevant compartments and highlighted in bold to indicate those organisms for which there is an overlap.

A comparison was undertaken to look at the different tissues which laboratories analysed for PFAS, the results of which are shown in Figure 3. Overall, 24 laboratories responded to this question giving collectively 41 different insights into the tissues analysed. For the single laboratory that provided feedback on plant biota tissues, it was reported that leaves, stems, roots and vegetables were considered.

From the responses it was clear that there were some major species differences in the tissues considered as would be expected based on size of the specimen. In mammals in general liver and muscle were the tissues analysed, with the exception of polar bear and Arctic fox in which plasma was instead or additionally analysed. Fish and invertebrates were more varied between full organism, fillet/muscle tissue and liver.





The separation of PFAS from interfering substances when undertaking tissue analysis is important to prevent over- or underreporting. Suitable sample preparation methods and/or chromatography/mass spectrometry separation techniques can be applied to mitigate effects of interferences on the analytical result. Taurodeoxycholic acid (TDCA) is an endogenous compound which is formed in liver cells and normally found in matrices of animal origin (mainly eggs and offal). Under typical C18 LC-MS column separation conditions it may eluate at the same retention time and shares the same mass transition as PFOS. This may lead to false positive identification or over-reporting of PFOS concentrations in liver samples. Therefore, TDCA should be removed by suitable sample preparation methods or suitable LC-MS separation methods Benskin *et al.* (2007). A further recently reported issue deals with PFBA, where an interference was reported on the predominant transition (m/z 213>169). The interferent was putatively identified as the saturated oxo-fatty acid (SOFA) 3-oxo-dodecanoic acid (Bangma *et al.* 2021). The level of potential overreporting due to this specific interferent remains to be determined in future research.

5.3 Differences in analytical capabilities of research and commercial laboratories with regards to PFAS analytical methods

The analytical capabilities of research and commercial laboratories with regards to non-target screening (NTS), total oxidizable precursor assay (TOPA), total fluorine (TF), extractable organofluorine (EOF) and adsorbable organofluorine (AOF) are indicated in Figure 4 below.

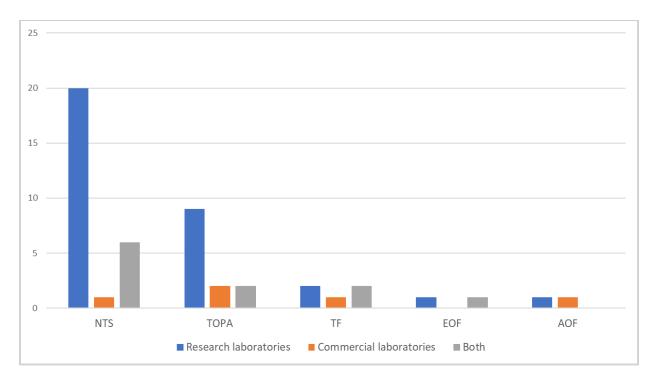


Figure 4: Analytical capabilities of research laboratories (n=33), commercial laboratories (n=6) and laboratories with both research and commercial activities (n=14).

Non-target (and suspect) screening is performed to a larger extent by laboratories with research activities, in total 55% (26 out of 47) research laboratories or laboratories which undertake both commercial and research activities. On the other hand, only 17% (one out of six) commercial laboratories are performing NTS. TOPA is implemented in approximately one third of the laboratories, with about the same share for research based and commercial laboratories. The different PFAS-Total methods are implemented in only very few laboratories currently as shown in Table 3.

	Total number of responses	TF	EOF	AOF
Research laboratories	33	2	1	1
Commercial laboratories	6	1	0	1
Laboratories which undertake both research & commercial activities	14	2	1	0

5.4 Coverage of PFAS being analysed for by laboratories

In the survey the laboratories were asked to indicate which PFAS they undertake analyses for. In relevant cases the PFAS were grouped in subclasses. A comparison of the relative amount of analyses of different subclasses of PFAS may be found in Figure 5. Unsurprisingly analyses are dominated by perfluoroalkyl carboxylic acids (PFCAs) and perfluoroalkane sulfonic acids (PFSAs). Fluorotelomer substances like fluorotelomer sulfonates (FTS), Fluorotelomer alcohols (FTOH) and FTO-

acrylates/acetates are also covered reasonably well. Quite a number of laboratories perform analyses for Perfluorooctanesulfonic acid (PFOS) precursors such as perfluorooctane sulfonamide and its different derivatives (e.g. FOSA; MeFOSA, EtFOSA, etc.).

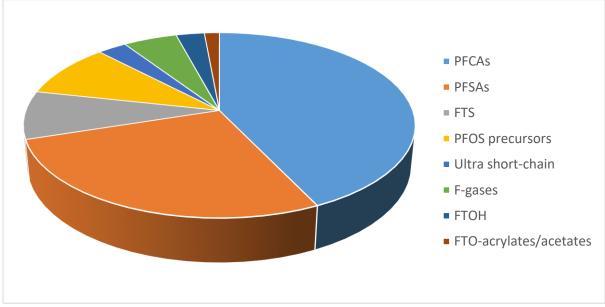


Figure 5: Analyses performed by laboratories for different sub-classes of PFAS.

Among the PFCAs, the highest numbers of analyses are performed for the most common chain lengths, i.e. Perfluorohexanoic acid (PFHxA) and Perfluorooctanoic acid (PFOA), see Figure 6. In the figure, the fluoroalkyl chain-length is gradually increasing from perfluorobutanoic acid (PFBA) (C-4) on the left, to perfluorooctadecanoic acid (PFODA) (C-18) to the right. PFCAs with chain lengths in the interval C-4 to C-12 are analysed by most laboratories, while PFCAs with longer chain lengths are analysed less frequently.

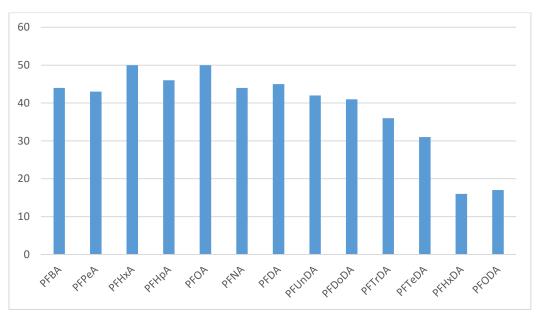


Figure 6: Number of laboratories that perform analyses of different PFCAs, out of a total of 52 laboratories. PFBA (C-4) to the left and PFODA (C-18) to the right.

Similarly, for the PFSAs the most familiar substances like PFOS, PFHxS and PFBS are analysed most often, while the longer chain length substances are analysed by a lower number of laboratories, see Figure 7. However, for the PFSAs, there is a difference between even and odd numbered C-atoms in the carbon chain, with more laboratories analysing for the even numbered substances. This is in line with the even-numbered PFSAs being more available due to production via electrophilic fluorination processes.

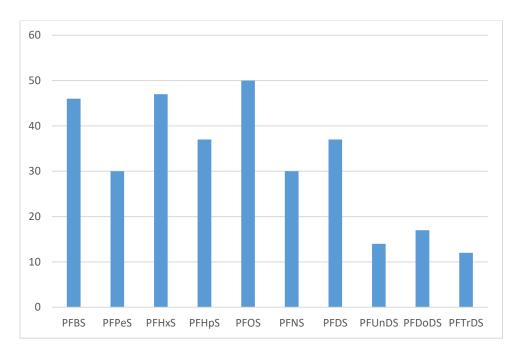


Figure 7: Number of laboratories that perform analyses of different perfluoroalkane sulfonic acids (PFSAs), out of a total of 52 laboratories, PFBS (C-4) to the left and PFTrDS (C-13) to the right.

The relative ratio of laboratories that are performing analyses for some selected PFAS of specific interest are shown in Figure 8. A high number of laboratories are analysing for HFPO-DA (30 out of 52), while also ADONA and the "cyclic PFOS" substance PFECHS are also analysed relatively frequently. Trifluoroacetic acid (TFA) and trifluoromethane sulfonic acid (triflic acid, TfOH, or TFMS), both containing only one perfluorinated C-atom, are analysed by nine and six laboratories, respectively.

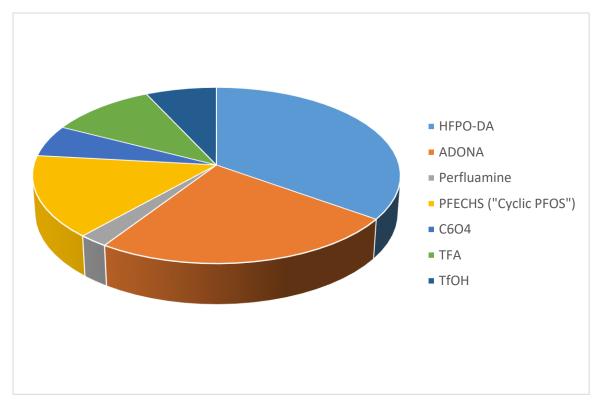


Figure 8: Analyses performed by laboratories for selected single PFAS.

Analysis of F-gases was primarily reported by two to three laboratories in the survey, depending on the individual F-gases considered. However, most F-gas analyses were reported for water samples, while there were only very few cases where laboratories reported the analysis of F-gases in air samples. The PFAS analysed in air samples in the survey were more often than not different carboxylic and sulfonic acids which are usually expected to partition to the water compartment.

5.5 Analysis of branched and linear isomers of PFAS

Of the laboratories that responded to the questionnaire with insights to this topic, 64% reported that they consider branched and linear isomers in their quality assurance at least 'sometimes'. Based on the responses shown in Figure 9, research laboratories and those which are both commercial and research laboratories consider the isomers more often than commercial laboratories. However, it is important to note that the way in which laboratories have answered this question may be strongly impacted by the PFAS analysed by any individual laboratory.

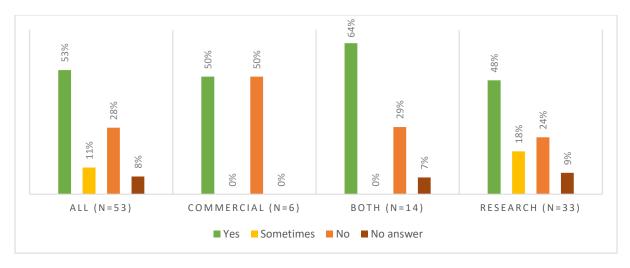


Figure 9: Percentage of laboratories which consider isomers in quality assurance.

5.6 Limits of detection for PFAS

The data provided by the participants was homogenised in terms of units in order to obtain comparable ranges for each matrix. Not every participant explicitly indicated if the figures provided were normalized or wet/dry weights in the case of solid samples, therefore, these ranges should be taken with precaution. The data received have been interpreted as representing values in the original matrix, not in the final extract, though this aspect may not have been made explicit in participants' responses. For the limits of detection (LOD) information gathered via the questionnaire, additional information to provide insight into validity, repeatability or calculation method, was not requested which may also influence suitability for making direct comparisons between the various laboratories' LODs.

The number of LOD reports for individual compounds is quite variable as shown in Figure 10. For the number of respondents which reported as analysing any of the individual PFAS considered by this analytical exchange in the various matrices see Table A3.1 in Appendix 3. The LODs ranged from the most commonly measured for PFOS and PFOA, whose LODs were reported by 35 and 33 laboratories respectively in surface water, to the less frequently analysed substances, whose LODs were not provided by any respondent. The LODs provided were also quite variable in terms of the matrices analysed. A higher number of laboratories provided LOD values in water samples or in sediments and soils, whilst zero or very few LODs were provided for other matrices such as air, dust or human samples.

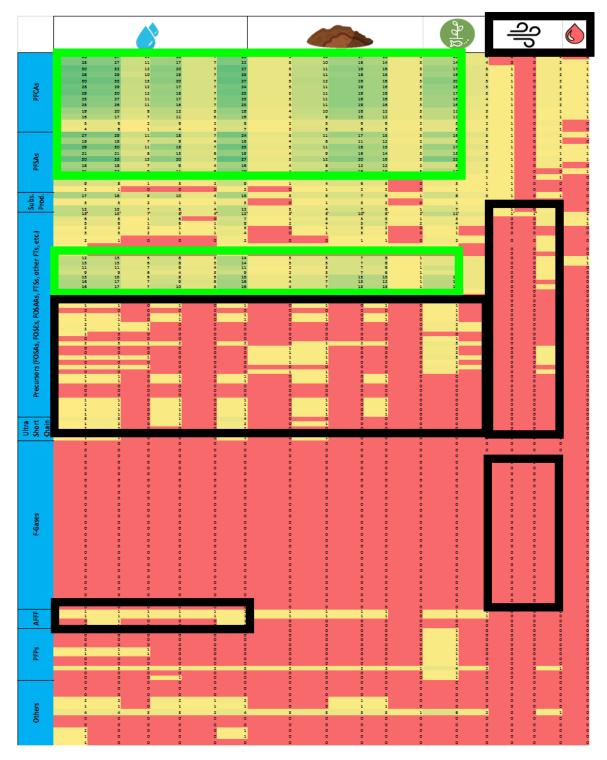


Figure 10: Number of laboratories that provided LODs for individual PFAS in different matrices. Columns correspond to different matrices reported grouped into water, solid, biological, air and blood samples. Each line corresponds to an individual PFAS joined here in functioning groups. Red squared areas show that zero laboratories are currently analysing for this PFAS in a given matrix. For information as to the PFAS considered under each functional group see Appendix 2, substances 1-94.

Bright green squared areas in Figure 10 indicate well represented individual PFAS, with LODs calculated and with analysis undertaken in more than ten to over 30 laboratories, although the questionnaire does not provide insight into the Quality Assurance/ Quality Control procedures which underly each stated LOD. There is good representation for PFCAs and PFSAs in most of water, solid and plant matrices, as well as for FTS. This is probably due to the fact that those are compounds with easily available commercial standards as well as being included in many monitoring programmes. On the contrary, it is noticeable that there are some 'black boxes' in the data provided. A few relevant compounds are not sufficiently evaluated in terms of proper sensitivity, such as precursors in commonly analysed matrices like water, solid or biological samples. Furthermore, some compounds that are particularly prone to be found in certain matrices, are not being analysed with the needed QA/QC requirements, as they do not have well established LODs, such as the most volatile precursors and F-gases in air samples or Aqueous Film Forming Foams (AFFF) in water. These 'black boxes' should be researched further by the scientific community in the near future in order to enhance analytical capabilities and to increase understanding of environmental exposure of these PFAS in these matrices. LOD ranges per matrix and per PFAS can be found in Appendix 3 in Tables A3.2, A3.3 and A3.4, for aqueous, solid/abiotic, biological and air samples, respectively. For those compounds frequently reported, such as carboxylic and sulfonic acids, LOD ranges were generally larger. This was due mainly to the few laboratories which reported very high LODs for some compounds (over 100 ng/L in drinking waters for instance). Those compounds reported by only a few participants, showed narrower LOD ranges representing more specialised laboratories performing more sensitive analysis.

Carboxylic and sulfonic acids reported LODs (Appendix 3, Figure A3.1, A3.2 and A3.3) ranged over six orders of magnitude in water samples (few thousands to 0.001 ng/L) due to the required lower limits of detection for drinking and surface water as shown in Figure 11a, whilst wastewater effluent or leachate LODs were not achieved lower than 0.05 ng/L. In solid samples, less than half of the participants provided LODs. Of those reported, they ranged from 10-0.001 ng/g and 10-0.01 ng/g in sediments and soils respectively and from 100-0.01 ng/g and 1-0.01 ng/g in biota (animals and plants respectively, as shown in Figure 11c). Air LODs were only reported by two laboratories and were provided in terms of ng/filter and/or in the order of μ g/m³ for the most relevant and regulated compounds and its precursors. LODs in relevant matrices for human exposure, such as indoor dust and blood serum were also reported, ranging between 0.1-0.1 and 0.5-0.04 ng/g respectively.

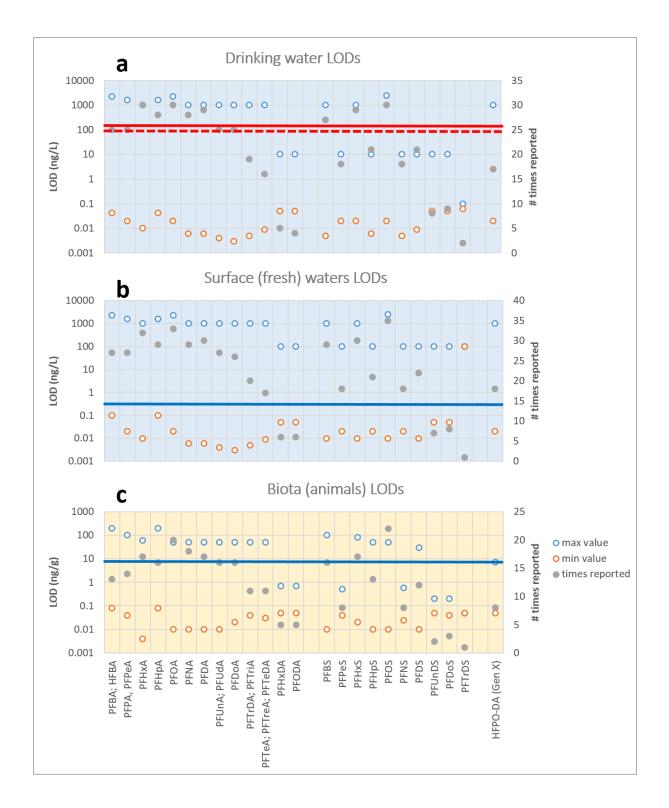


Figure 11: Reported ranges for PFCAs, PFSAs and HFPO-DA LODs in drinking water (a) fresh surface water (b) and in animals (c) as examples. Red full and dotted lines represent the regulatory limits from the revised Drinking Water Directive of 0.5 μ g/L for PFAS-Total and 0.1 μ g/L for a sum of 20 individual PFAS respectively. Blue lines represent the Water Framework Directive Environmental Quality Standards for PFOS in surface water and in fish. Further graphs showing additional PFAS and further matrices can be found in Appendix 3.

5.7 Suitability of current analytical approaches to meeting new and future PFAS regulations The supply and development of analytical services is partly driven by current regulations and the need for compliance. In January 2021, the European Commission put into force the revised Drinking Water Directive (DWD), to be implemented by Member States within two years and including limit value for the sum of 20 individual PFAS. The number of laboratories responding they already include the 20 individual PFAS is given in Table 4. The lowest analytical capacity is seen for C11-C13 sulfonic acids, which are rarely reported and for which high quality analytical standards of the odd number acids became available relatively recently as a response to the draft EU DWD in 2020.

Table 4: List of 20 individual PFAS included in the revised Drinking Water Directive, and number of laboratories analysing for them. The list is sorted from the highest to lowest of the number of laboratories currently providing the analysis. Based on 50 laboratories currently analysing for PFAS in drinking water.

PFAS	Abbreviation	Number of laboratories analysing respective PFAS
Perfluorohexanoic acid	PFHxA	50
Perfluorooctanoic acid	PFOA	50
Perfluorooctanesulfonic acid	PFOS	50
Perfluorohexanesulfonic acid	PFHxS	47
Perfluoroheptanoic acid	PFHpA	46
Perfluorobutanesulfonic acid	PFBS	46
Perfluorodecanoic acid	PFDA	45
Perfluorobutanoic acid	PFBA	44
Perfluorononanoic acid	PFNA	44
Perfluoropentanoic acid	PFPeA	43
Perfluoroundecanoic acid	PFUnDA	42
Perfluorododecanoic acid	PFDoDA	41
Perfluoroheptanesulfonic acid	PFHpS	37
Perfluorodecanesulfonic acid	PFDS	37
Perfluorotridecanoic acid	PFTrDA	36
Perfluoropentanesulfonic acid	PFPeS	30
Perfluorononanesulfonic acid	PFNS	30
Perfluorododecane sulfonic acid	PFDoDS	17
Perfluoroundecane sulfonic acid	PFUnDS	14
Perfluorotridecane sulfonic acid	PFTrDS	12

Considering the challenge of covering analytical readiness and capacity for the growing class of PFAS of concern, several strategies for non-targeted analysis or total analysis is being used. The revised EU DWD also includes a limit value for the sum parameter intended to cover all possible PFAS, PFAS-Total. It will be applied only when the methods and technical rules for monitoring have been developed but the Member States can decide to use either one of the parameters PFAS-Total or sum of 20 PFAS, or both. However, there are already National requests for different sum-parameter tests as the exposure and contamination situation becomes more complex. The responding laboratories were asked the question whether they intend to use sum of 20 PFAS, or include something broader aiming at covering PFAS-Total. The lack of standardized methods for the sum-parameter test might be the reason why 50% of the respondents did not reply to this question and 36% responded that they intend to use sum of 20 PFAS. The laboratories (8%) aiming for a broader approach mentioned using methods like non-target screening analysis, extending the list of target PFAS, TOP Assay, combinations of EOF and TOP Assay, or sum of all thyroid disruptors and/or PPAR inhibitors and/or steroidogenesis activators.

Comparing the reported LODs in drinking water (see Section 5.6, Figure 11a) with the limits set by the revised DWD of 0.1 μ g/L for a sum of 20 individual PFAS, it can be seen that both the limits and maximum LODs are relatively similar particularly for the PFSAs. This demonstrates that many laboratories are currently achieving the analytical sensitivity requirements in terms of LODs for many of the individual compounds set out in this legislation. The most restrictive proposals to date in Europe, such as the health-based limits of 0.002 μ g/L for the sum of four PFAS (PFOA, PFOS, PFNA and PFHxS) in drinking water in Denmark (Danish Environmental Protection Agency, 2021) and the Swedish action levels of 0.09 μ g/L of sum of 11 PFAS in drinking water (Swedish National Food Agency, 2021), are further from LODs reported, demonstrating a likely need for more sensitive analytical methods with lower LODs when future regulations are being implemented.

Regarding other regulations, PFOS is a priority hazardous substance under the Water Framework Directive (EU, 2013). An annual average (AA) environmental quality standard (EQS) in water is derived by back calculation from the biota EQS. The AA EQS for PFOS is $6.5 \times 10^{-4} \mu g/L$ for freshwaters and there is a maximum allowable concentration of 36 $\mu g/L$ of PFOS in freshwater (Environment Agency, 2021). In the case of PFOS, the annual average EQS for PFOS in freshwater is within the range of most of the LODs reported for PFOS in freshwater in the questionnaire responses, but the environmental quality standards are closer to the lower LODs reported in comparison to drinking water standards, highlighting an increase of sensitivity needed as well for environmental aqueous matrices and not only for assessing human exposure through drinking water. The biota EQS for PFOS in fish, has a maximum allowable concentration of 9.1 $\mu g/kg$. The limit of detection for fish based on the example of PFOS is demonstrably sufficient with the LODs reported by many of the participants (Figure 11c).

In 2020, a Scientific Opinion of the European Food Safety Authority (EFSA) was published (EFSA, 2020), where EFSA has set a group tolerable weekly intake (TWI) of 4.4 ng/kg of body weight per week for the sum dietary exposures of PFHxS, PFOS, PFOA and PFNA. As a result, the EC is now discussing setting maximum limits (MLs) for these PFAS, and the sum of the four, in a variety of foods, including fish and (in the future) plant materials (agricultural crops) as well. Although MLs have not been set yet, and discussions are ongoing, it is likely that method sensitivity needs substantial improvement, e.g. for plant materials, as those are consumed in large quantities on a daily basis, and already low levels (low pg/g range) may lead to substantial dietary exposures. Method sensitivities need to be improved to meet such demands in the future. Within Europe, there are networks of reference laboratories (EURL)) established for accurate determination of a wide range of food and feed contaminants. The EURL network on Persistent Organic Pollutants has established a Working Group on PFAS that currently is discussing guidelines that enable laboratories to perform

reliable measurements in food and feed, in a wide variety of animal and plant based materials, and a concentration span of 3-4 orders of magnitude (low pg/g to ng/g range). The guidelines are anticipated to be published during the first half of 2022.

5.8 Minimising contamination during PFAS analysis

In order to assess PFAS contamination nearly all laboratories report using method blanks (98%) and instrumental blanks (96%) to monitor contamination during analyses. The approaches taken to reducing laboratory contamination from PFAS are shown in Figure 12. Other common precautions are the use of old clothing in the laboratory (88%) to reduce shedding of clothing fragments which may contain PFAS and checks for laboratory materials and chemicals (88%). Only 73% of the laboratories replied that they check the monitoring processes do not add further PFAS, e.g. through the use of field blanks. A possible reason for this might be that many of the analysing laboratories are not often responsible for the planning and implementation of the sampling. Only 31% of the laboratories avoid using cosmetics in the laboratory to avoid PFAS contamination.

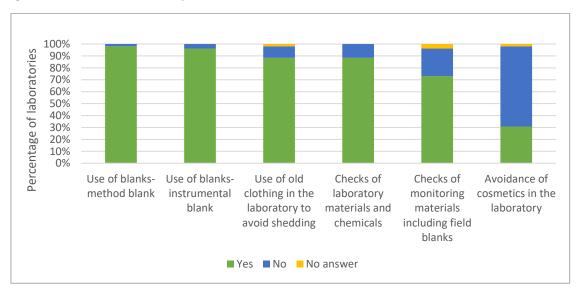


Figure 12: Precautions currently undertaken by laboratories to avoid contamination in the PFAS analysis.

There was no clear difference between the different types of laboratories in the number of the abovementioned precautions taken as shown in Figure 13. Two research laboratories, one categorized as both research and commercial laboratory, and no commercial laboratories reported that they apply all of the six precautions included in the questionnaire to avoid contamination. The minimum number of precautions was two, as reported by three laboratories (two research laboratories and one commercial laboratory). All these three laboratories apply method blanks, two of them also instrumental blanks and one of them checks laboratory materials and consumables.

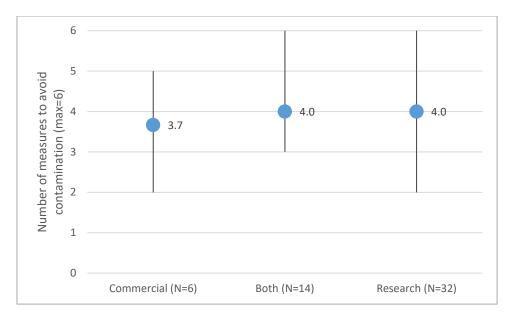


Figure 13: Number of precautions per laboratory type. The lines represent the min*imum*-max*imum* and the number (blue dot) is the average.

Other reported precautions can be divided into five categories: precautions related to 1) laboratory rooms, 2) materials, 3) chemicals, 4) analytical systems and 5) other precautions.

Precautions in category 1 include controlling the contamination caused by laboratory materials by treating PFAS samples in a designated room (two laboratories), treating the highly contaminated samples in separate rooms/fume hoods than the medium or low concentration samples (three laboratories), preparing and storing standards in separate rooms (one laboratory) and working in clean laboratories under a low-dust atmosphere and positive pressure (ISO 8 class according to NF EN ISO 14644) (one laboratory).

In category 2, avoiding fluoropolymer containing materials is a very common way to avoid contamination in PFAS analysis. Some laboratories specifically stated avoiding using PTFE, aluminium foil and parafilm. The cleanliness of the labware is often ensured by rinsing with methanol and baking the glassware before use or substitution of polypropylene/HDPE flasks instead of glassware. For category 3, laboratories reported using pre-tested chemicals and other materials in PFAS analysis, controlling their cleanliness with blank tests for new batches and ensuring continuity when purchasing materials from the same suppliers.

For analytical systems (category 4), a very common practise is to replace the fluoropolymer LC tubing with e.g. PEEK tubing. Two laboratories indicated the use of an LC system with chromatographic column between the solvents and the injectors in order to shorten retention time and reduce contamination from the solvents used. Other reported precautions (category 5) include practices that are related to cross-contamination (SPE dividers to avoid splashing carryover), quality control (use of certified reference material and participation in intercalibration exercises) and general good laboratory practices (weekly routine cleaning of the laboratory).

5.9 Spectrometry methods currently utilised to analyse for PFAS by respondents

The 52 responders listed a total of 80 analytical methods, of which 73, six and one were based on liquid-, gas- and supercritical fluid chromatography (LC, GC, SFC), respectively. All methods used MS for detection. This showed quite a different perspective to that which had been gained from the BAM PFAS workshop in September 2021 where the poll on 'Which PFAS analytics methods are you using?',

gave insights that 62% of workshop attendees were using LC-MS-MS, 20% were using combustion ion chromatography (CIC), and 0% were using supercritical fluid chromatography.

Of the 73 (91%) LC-based methods, three methods used a mixed-mode reversed phase - anion exchange column using methanol and water as mobile phase, modified with 20-50mM ammonium acetate at a pH of 9. All other LC-based methods made use of reversed phase chromatography using C18 columns. As mobile phase, combinations of methanol, acetonitrile and water were used modified with 2-10mM ammonium acetate or ammonium formate in most cases. A total of nine methods used a combination of ammonium acetate and 0.01-0.05% formic or acetic acid as organic modifier. One of those methods mentioned that the pH was set to 3.5. One other method used 1mM ammonium fluoride as modifier. Four other methods used only 0.05-0.1% formic or acetic acid as organic modifier. At least one of these methods used this modifier to obtain better separation of short-chain PFAS at low pH, as most PFAS will have neutral charge at low pH potentially resulting in better retention. Nearly all LC-based methods used negative electrospray ionisation. Three methods mentioned the use of both positive and negative ionisation, and one method mentioned only positive ionisation mode. Most PFAS included in monitoring are anionic substances and negative ionization is therefore most commonly used, however to include a broader range of PFAS, including some AFFFs, both negative and positive ionization is needed. MS/MS based on triple quadrupole or Q-trap was used by most LCbased methods, but HRMS is clearly gaining attention as 14 methods specified the use of Orbitrap of TOF-based technologies.

The six GC-based methods all used H_2 as carrier gas. Electron ionisation (EI) and positive chemical ionisation (PCI) were indicated four and three times, respectively, as ionisation method. MS (ion trap/quadrupole) and HRMS (orbitrap) were mentioned four and two times, respectively, as the detector.

The SFC-based method used CO_2 and MeOH with 0.1% ammonia as mobile phase and ESI-MS/MS as detector.

For future questionnaires, it is recommended to ask responders to list matrices, analytes and detection limits (Question 8) per analytical method, as in many instances it was not possible to derive what kind of matrices or analytes were analysed by the different methods. For this reason, it was not possible to draw detailed conclusions about which methods would be more suitable for specific matrices or analyte groups.

Relevant questions that could not be unambiguously answered based on this dataset related to (1) the use of GC with EI or PCI, or SFC as opposed to LC-based-methods, (2) the use of mixed-mode reversed phase – anion exchange column as opposed to reversed phase LC columns, and (3) the optimal use of organic modifier in LC (ammonium acetate/formate, formic/acetic acid, ammonium fluoride) and the corresponding pH of the mobile phase.

5.10 Suspect and Non-target screening

Of the 50 laboratories which responded to the question regarding utilisation of suspect and non-target screening as part of their suite of analytical tools to determine presence of further PFAS, 54% of laboratories were currently using these techniques. One laboratory provided additional information that this was specific to PFUnDa, PFDoA, PFTrDA, PFTeDA.

5.11 Methods used for group-based PFAS analysis

The number of laboratories utilising different methods for group-based PFAS analysis are shown in Figure 14. Several laboratories reported using the TOP Assay method, 13 laboratories for water

analysis and 12 laboratories also for analysis of other matrices. There was not a complete overlap between laboratories, some laboratories only used TOP Assay for other matrices than water and vice versa. Five laboratories confirmed that they use total fluorine measurement, while three and two laboratories used EOF and AOF, respectively. While AOF is a standardized method (DIN 38409-59:2020-11 (draft)), EOF results depends mainly on which extraction method is being used and can be widely different depending on matrix. The three laboratories using EOF also stated different extraction methods, including methanol extraction with sonication, SPE, and ion-pair extraction. Four laboratories confirmed the use of bioassays as group methods. The reported tests were *in-vitro* thyroid transport protein transthyretin (TTR)- Thyroid Receptor (TR), Fluorescein isothiocyanate (FITC)-T4, Peroxisome Proliferator Activated Receptor (PPAR) alpha and Yeast Estrogenic Screening and *in-vivo* Fish Embryo Test.

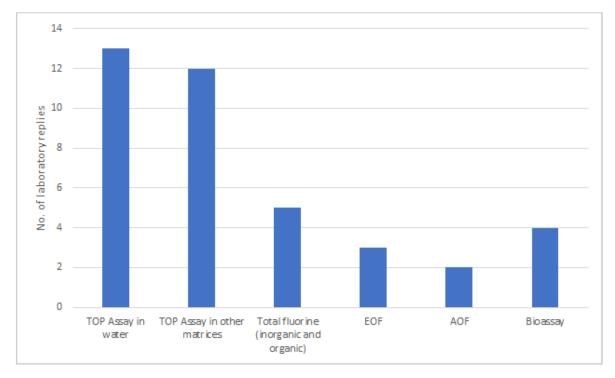


Figure 14: Number of laboratories using different methods for group-based PFAS analysis.

5.12 Sample preparation and treatment approaches

Of the 57 respondents, seven were not currently undertaking any analyses for PFAS compounds; of these, five indicated that they would be in future and of these two provided sufficient information to be included in this review. From the 52 respondents included it is possible to see that the matrices studied by the most respondents are: surface (fresh water) (79%), drinking water (65%), groundwater (58%), biota – animals (50%), waste water treatment effluent (44%), and sediment (42%). Least studied matrices included: air (8%), food – dairy (8%), compost/mulch (6%), and flue gas (<2%). As noted previously, differing numbers of respondents provided information on related topics in different parts of the questionnaire. This resulted in variation in the percentages being presented depending on the part of the questionnaire that the data were retrieved from. Figure 15 indicates the proportion of respondents that currently or will in future analyse for a particular matrix.

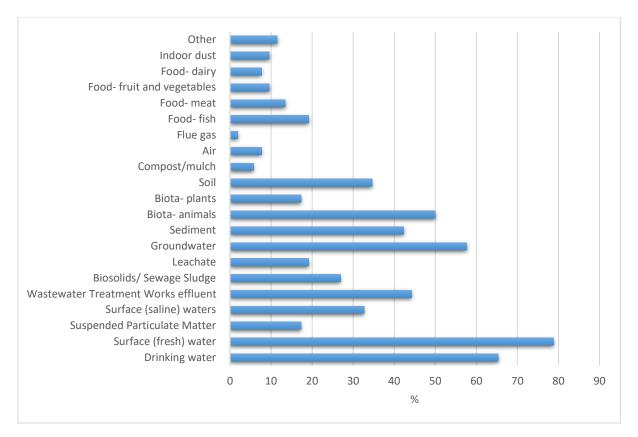


Figure 15: Chart showing the proportion of respondents analysing for each matrix based on responses given to the question 12 of the questionnaire focusing on specific analytical methods in each individual matrix.

Of the respondents that indicated that they analyse for a particular compartment/matrix, it is apparent that not every laboratory undertook every preparation procedure included within the survey prior to analysis (see Appendix 4 for full information of the number of respondents currently considering each preparation procedure).

All respondents that analysed samples from the following matrices confirmed that for biota- plants, flue gas, food – dairy, food - fruit and vegetables and other (Serum), they undertook sample storage or preservation. Similarly, for the flue gas, food – dairy, food - fruit and vegetables matrices, all respondents confirmed that they undertook extract storage or preservation.

All respondents that analysed samples from the following matrices confirmed that for air, biosolids/ sewage sludge, biota - plants, soil, flue gas, food – dairy, food - fruit and vegetables, food- meat, indoor dust, leachate, sediment and soil, they undertook an extraction conditions step.

All respondents that stated they analysed samples from the following matrices confirmed that for biota- plants, flue gas, food – dairy, food – fish, food - fruit and vegetables and food-meat, they incorporated a sample clean-up stage.

Of the 52 respondents who confirmed that their laboratory currently undertook analysis for PFAS (including two that indicated that they would be in future and provided sufficient information to be included in this topic), 38 (73%) provided additional information of specific details for at least one of the preparation steps for analysis of samples of a particular matrix.

Responses from the laboratories that provided additional information are summarised in Appendix 5. From the additional information provided regarding the preparation steps it is evident that there is

wide variation between laboratories when analysing samples from the same matrix and even between similar techniques.

For this review, the various matrices have been classified into: 'Liquid' (i.e. drinking water, leachate, groundwater, surface (fresh) water, surface (saline) waters, wastewater treatment works effluent); and, 'Solid' (i.e. biosolids/ sewage sludge, biota – animals, biota – plants, compost/mulch, food – dairy, food – fish, food - fruit and vegetables, food – meat, indoor dust, sediment, soil, suspended particulate matter). Whilst there are also gaseous matrices (i.e. air and flue gas) and four respondents have confirmed that they analyse for these, no additional supporting information are supplied for preparation techniques (apart from one respondent that indicated Air samples were stored at 4° C for a maximum of 30 days) and they are not discussed further.

Sample/extract storage

Most common 'Sample Storage' and 'Extract Storage' techniques involve reduced temperature (i.e. refrigeration or freezing) with storage temperatures listed as: frozen (unspecified temperature), -20°C, <0°C; and, 1 to 5°C. Samples are also stored at 8°C and even room temperature. Storage retention times vary from: as soon as possible, or frozen; frozen until use, four days, one week, 14 days; or, up to 30 days/1 month. Other preservation techniques include 50% methanol, or sodium thiosulfate. The majority of laboratories exercised some form of sample and extract storage regardless of the sample matrix. However, five laboratories indicated that for at least one matrix they did not, but did not provide further supporting information for the possible reason (e.g. processed the same day).

Filtration

For the Liquid matrices, 20 respondents confirmed that they undertook a filtration process. The various filtration techniques described in the supporting information included: Buchner filtration with filter paper, regenerated cellulose (0.22 μ m), sand filters and glass fibre filters (0.45 μ m to 0.7 μ m). Sample filtration through glass fibre filters of various diameters was the most common technique, with four of twelve respondents who provided supporting information choosing this technique. Additionally, four respondents confirmed that they undertook alternative procedures including decanting, centrifugation and Solid Phase Extraction (SPE) including Weak Anion eXchange (WAX).

For the Solid matrices, four respondents confirmed that they undertook filtration steps. Of these only one respondent provided supporting information: filtration through regenerated cellulose filters, for biota –animals, sediment and soil matrices.

Pre-treatment techniques

Depending on the compartment/matrix, typical 'Pre-treatment' techniques included centrifugation, pH adjustment, sieving, drying, freeze drying, grinding, homogenisation or alkaline digestion.

For the Liquid matrices, eight respondents confirmed that they undertook a Pre-treatment step. Of these six provided supporting information on pre-treatment techniques as follows: centrifugation, three respondents – for drinking water, surface (fresh) water, surface (saline) waters, wastewater treatment works effluent and groundwater matrices; pH adjustment (including adjustment to pH 3, adjustment to pH 6.5, or treatment with formic acid (98%)), two respondents – for drinking water, surface (fresh) water, surface (saline) waters, wastewater treatment works effluent matrices; and, shaking, one respondent – for the wastewater treatment works effluent matrix.

For the Solid matrices, 34 respondents confirmed that they undertook a Pre-treatment step. Of these, 23 provided supporting information on pre-treatment techniques (either as a single process or in combination) as follows: sieving, six respondents – for biota-animals, compost/mulch, indoor dust, sediment, soil and suspended solids particulate matter matrices; drying (including drying with silica,

or \leq 40°C), six respondents – for biosolids/sewage sludge, biota-animals, sediment and soil matrices; freeze-drying, 13 respondents - for biosolids/sewage sludge, biota-animals, biota-plants, sediment, soil and suspended solids particulate matter matrices; chopping – one respondent – for the biotaanimals matrix; grinding, 12 respondents – for biosolids/sewage sludge, biota-animals, biota-plants, sediment, soil, suspended solids and particulate matter matrices; homogenisation, four respondents – for biota-animals, sediment and soil matrices; and, alkaline digestion (sodium hydroxide), one respondent – for biota-animals and food – fish matrices.

Additionally, one respondent stated that for the pre-treatment step for analysis of AFFF they utilised dilution (between 5000 and 10 million-fold) in ultra-high performance liquid chromatography mass spectrometry (ULC-MS) grade water.

Extraction

Again, depending on the compartment/matrix, 'Extraction' techniques typically included ion-pair extraction, SPE, centrifugation with solvent extraction, ultrasonication with solvent extraction, Accelerated Solvent Extraction (ASE), or alkaline digestion plus solvent extraction. Typical extraction solvents included methanol, acetonitrile, methyl tert-butyl ether, acetic acid, ethyl acetate, dichloromethane/2-propanol, or sodium hydroxide.

For the Liquid matrices, 29 respondents confirmed that they undertook an extraction process. Of these 21 provided supporting information on the extraction processes utilised. One respondent confirmed that depending on the required LOD, they utilised ion-pair extraction (pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether), or SPE for all liquid matrices. The majority of respondents confirmed that they utilised some method of SPE, including SPE-DVB, on-line SPE, Chromabond; WAX, including OASIS – WAX and Phenomenex Strata-X AW; and, SPE-WAX, for all liquid matrices.

For the solid matrices, 35 respondents confirmed that they either undertook an extraction process or utilised an extraction solvent without specifying the extraction process. Of these 25 provided supporting information on the extraction processes utilised: ion-pair extraction (including addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether), two respondents – for biosolids/ sewage sludge, biota – animals, biota – plants, compost/mulch, food – dairy, food – fish, food - fruit and vegetables, food – meat, sediment, soil and suspended particulate matter matrices; ultrasonication (including alkaline digestion, methanol and acetic acid-based extraction, orbital shaking or centrifugation, focused ultrasonic solid-liquid extraction (FUSLE); and, methanol or acetonitrile as solvents), 14 respondents – for biosolids/ sewage sludge, biota – animals, biota – plants, food – dairy, food – fish, food - fruit and vegetables, sediment, soil, suspended particulate matter matrices; solvent (including shaking, vortexing and centrifugation; and, acetonitrile, ethanol, methanol and methanol/water as solvents), four respondents – for biota – animals, food – fish, sediment matrices; SPE (including QuEChERS (acetonitrile solvent) and WAX), four respondents - for the biota – animals, food – fish, sediment and soil matrices; ASE (including methanol, or hexane/dichloromethane solvents, evaporation and filtration (0.22 μ m filter)), two respondents – for biota – animals, biota – plants, food – fish, food – meat, sediment, soil matrices; and, alkaline digestion plus solvent extraction (including potassium hydroxide or sodium hydroxide, with agitation and with acetonitrile or methanol as solvents), three respondents – for biosolids/ sewage sludge, biota – animals, food – dairy, food – fish, food - fruit and vegetables, food – meat, sediment and soil matrices.

Additionally, one respondent utilised methanol and ultrasonication in the extraction step for unspecified 'Production facility products'.

Clean-up

The 'Clean-up' techniques typically include: SPE (including WAX), washing, filtration, solvent matching, activated charcoal and freezing.

For the liquid matrices, 18 respondents confirmed that they undertook a clean-up step and of these eight provided further supporting information. Techniques comprised: addition of ammonium acetate, filtration, solvent matching, washing; and, with four respondents utilising SPE (including SPE-WAX and Oasis WAX), for the drinking water, groundwater, surface (fresh) water, surface (saline) waters matrices.

For the solid matrices, 30 respondents confirmed that they undertook a clean-up step and of these 22 provided further supporting information. Eighteen respondents utilised some method of SPE (including filtration through polypropylene (PP) filters, HybridSPE, Envi-carb, SPE-DVB, SPE-WAX, WAX,OASIS – WAX and Phenomenex Strata-X AW), for the biosolids/ sewage sludge, biota – animals, biota – plants, food – dairy, food – fish, food - fruit and vegetables, food – meat, indoor dust, sediment, soil, suspended particulate matter matrices.

Five respondents utilised alternative techniques including: activated charcoal, graphitised carbon and acetic acid, dispersive carbon, freezing to remove fatty compounds and proteins, and turbulence flow chromatography, for the biosolids/ sewage sludge, biota – animals, biota – plants, food – dairy, food – fish, food - fruit and vegetables, food – meat and sediment matrices.

Pre-concentration

The 'Pre-concentration' techniques for both Solid and Liquid matrices included: SPE and on-line SPE, evaporation under nitrogen gas, extraction, reconstitution of solid samples into various volumes, or dilution.

5.13 Approaches to method development

Responses were received to Q27 '*Did you use any standard publications to support your method development for PFAS analysis? Please specify and include hyperlinks if available. Giving details of methodologies used*' from 62% (31 out of 50) of laboratories currently analysing for PFAS. Of the 31 respondents, 9.6% of those stated that they did not use any standard publications in their method development (including statements clarifying that methods did not exist when they began work in this area more than 10 years ago), 3% stated 'yes' and 3% stated 'sometimes' but did not provide any further details.

The methodologies cited by survey respondents are included in Appendix 6 in the order for which they were most frequently cited and are divided into methods considering PFAS analysis by mass spectrometry methods or bioassay methods, a further four methods were not currently available in English.

The most consistently referred to methods were the US EPA methods (29%), and the ISO water quality methods (19%), and the DIN method (10%), though this also likely reflects the focus of PFAS monitoring in water matrices over other matrices. As well as the standard methods respondents also referred to MS manufacturers application notes and the Agilent method.

5.14 Future Developments in PFAS capabilities

Figure 16 shows the collection of responses considering future development aspirations. Developing robust methods for detecting TF associated with PFAS compounds is very desirable and can provide

valuable information on the total PFAS burden within a sample. This can then be supported with targeted analysis of known PFAS compounds to get a more complete understanding of PFAS contamination. The development of TOP can also provide continuity between environmental laboratories and allow better comparison of data, however understanding the deviation between this analysis with regards to method and recovery rate is an important factor. In relation to TOP assays, 13 laboratories confirmed that they already have the ability to determine TOP in water with nine of these able to determine TOP in other matrices. Three laboratories indicated that they have the capability to determine TOP in other matrices but not water. Responses focusing on future analytical development suggests that a further eight laboratories are prioritising developing future TOP capabilities. Only two laboratories currently have AOF capabilities with only one laboratory looking to develop EOF and one laboratory developing AOF capabilities in the near future.

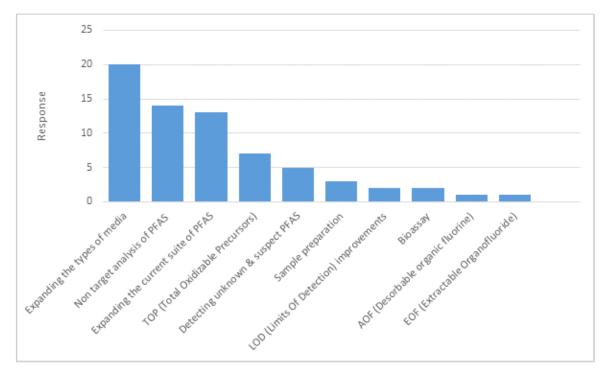


Figure 16: Development priorities for future PFAS analysis by number of respondents.

The extent of unidentified PFAS found in environmental samples has led to development in nontargeted screening methods, this commonly involves the use of full scan data and the selection of signals of interest from this output. Responses focusing on existing capabilities suggests that 27 laboratories currently have the ability to perform non-target and suspect screening of PFAS. Responses focusing on future analytical development suggests that a further 14 laboratories are prioritising the development of non-target screening methods. This shows a significant capacity for suspect screening across the cohort and highlights potential for collaboration between laboratories to compare screening data with known PFAS signals.

Responses to questions focusing on current PFAS analysed in different matrices indicate a widespread ability for fully quantitative analysis of water matrices (drinking, surface, ground and saline waters), waste (biosolids, sludge) and animal biota and soil. A smaller number of laboratories have the capability to consider suspended particulate matter, plant biota, compost air and dusts. The development area with the most interest was the ability to analyse PFAS in different media and matrices, with 20 responses suggesting development in this area was a priority. It is clear there is an appetite for laboratories to expand their capability, this presents an opportunity for the NORMAN network to provide a steer on areas where capabilities are lacking and to introduce some continuity

when new methods are adopted. Interest was shown in developing capabilities for sludge and biosolids (3) waters (2) food and feed (2) biota (1) and air (2).

The greatest PFAS analytical capabilities remain in water and animal biota, however there is a clear appetite to diversify and include analysis of more complex sample matrices. It is also important to note that not all development goals would have been captured by this questionnaire, and due to the rapid development of PFAS analysis, continued developments are expected. Typically, this will include the adoption of standard methods, LOD improvements, expanding the suite of PFAS and non-target analysis.

6. Conclusions

This questionnaire-based activity set out to collaborate to understand the insights from laboratories as to their current approaches for PFAS analysis, including improved understanding into the PFAS analysed, the matrices studied, and the methodologies undertaken both for analysis and sample preparation.

Collation of the results from this work give a useful baseline of the approaches that laboratories are currently undertaking for PFAS analysis.

The questionnaire results indicated that for the majority of the carboxylic and sulfonic acids, there is analytical capability down to the levels needed for compliance to regulatory limits set out in both environmental quality standards and in the revised Drinking Water Directive. However, many other PFAS, particularly those most recently being sought to be analysed and precursors, still need further development for reaching sufficient sensitivity in many sample types.

The results demonstrate potential needs for laboratories to increasingly consider short chain PFAS (an activity has been proposed in the NORMAN network JPA 2022 for specific inter-laboratory collaboration on this area of PFAS analysis, as well as interlaboratory comparisons for a number of PFAS). There were some notable areas where there was limited focus by laboratories on specific matrices, with the vast majority of the focus remaining on water matrices. Additionally greater clarity is required on the approaches needed for compliance with the Revised Drinking Water Directive aspects relating to PFAS-Total and Sum of PFAS- with options being explored. There is further potential for utilisation of bioassay approaches than is currently being utilised and increasing potential for non-target screening in the years ahead.

This activity has given the unique opportunity to gain awareness into the areas of PFAS analysis in which laboratories will seek to develop their capabilities over coming years. A proposal for a further activity in JPA 2022 for the NORMAN network was put forward (stemming from the insights from this PFAS Analytical Exchange) focusing on TOP assay capabilities through a further questionnaire to be able to build an understanding of current approaches. This was one of several key focal areas in which laboratories indicated a wish to expand along with goals to improve sensitivity, exploring further matrices to better understand exposure, and greater utilisation of suspect and non-target screening tools- developments on all of these areas are potential opportunities for future collaboration.

7. Acknowledgements

The Steering Group would like to thank the Sounding Board, and the 57 respondents to the questionnaire for their time and contribution to this activity, without which we would have been unable to gain such significant insights into current analytical approaches to PFAS analysis. Thank you all for your collaboration. The Environment Agency would like to thank all of the members of the Steering Group for providing their knowledge and insights on PFAS analysis into both shaping this activity, and interpreting the information received from the various laboratories. Thank you to Christian Vogel at BAM for provision of the poll results from the Workshop held on 1st September 2021.

8. Abbreviations

AA - Annual Average

- AFFF Aqueous film forming foams
- AOF Adsorbable organofluorine
- ASE Accelerated Solvent Extraction
- CEN Comité Européen de Normalisation / European Committee for Standardization
- DVB Divinylbenzene
- EFSA European Food Safety Authority
- EOF Extractable organofluorine
- EQS Environmental Quality Standard
- **EURL European Reference Laboratories**
- FTOH- Fluorotelomer alcohol
- FTS Fluorotelomer sulfonate
- FUSLE focused ultrasonic solid-liquid extraction
- JPA Joint Programme of Activities
- LC-MS Liquid Chromatography Mass Spectrometry
- LOD Limit of detection
- LOQ Limit of quantification
- ML Maximum Limit
- NTS Non-Target Screening
- PFAS Per- and polyfluoroalkyl substances
- PFCAs perfluoroalkylcarboxylic acids
- PFHxA Perfluorohexanoic acid
- PFOA perfluorooctanoic acid
- PFOS perfluorooctanesulfonic acid
- PFSAs perfluoroalkane sulfonic acids
- PP polypropylene
- QuECHERS Quick, Easy, Cheap, Effective, Rugged and Safe
- SPE Solid Phase Extraction
- TF Total Fluorine
- **TOP Total Oxidizable Precursors**

- TOPA Total Oxidizable Precursor Assay
- TWI Tolerable weekly intake
- ULC-MS Ultra high performance liquid chromatography mass spectrometry
- WAX Weak Anion eXchange

9. References

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Appendices

Appendix 1: The circulated PFAS Analytical Exchange questionnaire

The Excel file of the questionnaire which was circulated can be found on the Interlaboratory Studies webpage of the NORMAN network website at https://www.norman-network.net/?q=node/27

The below shows the information relating to PFAS that was sought from each questionnaire respondent.

Completion of this questionnaire is voluntary.

Why am I being asked to complete this questionnaire?

There is a growing interest in analytical capabilities for perfluoroalkyl and polyfluoroalkyl substances (PFAS). The NORMAN network in 2021 as part of their Joint Programme of Activities has opted to undertake a PFAS Analytical Exchange Activity which was put forward by the Environment Agency, England. The primary purpose of this activity is to gain an understanding of the current analytical capability for this large group of substances in international laboratories. The questionnaire focuses on PFAS analysis from an environmental perspective and areas of research of specific interest to the NORMAN network. It is recognised that responses to this questionnaire represent a snapshot in time, and that analytical capability in this area is subject to change over time.

We would welcome you taking the time to complete the questionnaire on behalf of your organisation/ laboratory in this rapidly evolving area of research- thank you in advance.

The sections which you are asked to complete are highlighted in yellow, many have drop down boxes, with space also provided for you to give more detail.

1. What is the name of your laboratory/organisation?

2. Name of individual competing the questionnaire?

3. Contact email address:

4. Is your laboratory primarily a commercial laboratory, a research laboratory or both?

5. Does your laboratory currently analyse for PFAS substances?

Please indicate: Yes, No or Will be in Future.

6. Is your laboratory an accredited laboratory for PFAS analysis?

Please indicate: Yes, No or Will be in Future.

7. How many samples does your organisation analyse per year for PFAS analysis (approximately)?

8. Which of the following PFAS substances does your laboratory currently have a validated analytical method to analyse for in each of the sample types?

What are the Limits of Detection for each of those substances?

We have included the names and CAS numbers for what we anticipate to be some of the most frequently analysed for PFAS substances in the worksheet below

Please include the Limit of Detection (LOD) you are able to achieve for these substances.

Those PFAS substances at the top of the table are those for which we anticipate there may be wider analytical capability currently.

Please add the same information (Name, CAS Number and the Limit of detection) for any further additional per-fluorinated substances for which your laboratory currently has validated analytical

capability- there is space available in rows 112-117.

Please include the LOD in the spreadsheet below if you have analytical capability

of any of these sample types for any of these PFAS substances (or an 'X' if the LOD is unavailable).

Please include the Unit in which you have stated your LOD i.e. ng/L or mg/kg

Unit that your LOD is stated in for each sample type:

Question 8 was asked for each of the following matrices for both semi-quantitative and fully quantitative analysis for 94 PFAS with space provided for laboratories to add information on any further PFAS analysed:

Drinking Water, Surface (fresh) waters, Surface (saline) waters, Suspended particulate matter, Wastewater treatment work effluent, Biosolids / sewage sludge, Leachate, Groundwater, Sediment, Biota (animals), Biota (plants), Soil, Compost/mulch, Air, Flue gas, Indoor dust and Other (please specify).

The PFAS which the questionnaire focused on was substances 1-94 in Appendix 2.

Chromatographic system pecify type, manufacturer and model)	Chromatographic column (pre- and analytical, specify type, manufacturer and brand)	Mobile phase (describe)	Source of ionization (specify type, manufacturer and model)	MS System (specify type, manufacturer and model)	Method that you have published (if available please include a hyperlink to your publication

10. Which measures do you take as p	recautions to avoid cross-contamination of samples by PFAS unintentionally?
Measure	Yes or No
Checks of monitoring materials	
including field blanks	
Use of blanks- instrumental blank	
Use of blanks- method blank	
Checks of laboratory materials and	
chemicals	
Use of old clothing in the laboratory	
to avoid shedding	
Avoidance of cosmetics in the	
laboratory	
Any others? Please provide details	

11. How do you store your samples Please specify for each sample type			sample preparation on the s	amples such as filtration	on, extraction, pre-t	reatment, pre-conc	entration or s	ample clean-up?
Sample Type	Sample storage e.g. freezing or preservative	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction solvent or sorbent	Extraction conditions/ assistion	Extract storage e.g. freezing or preservative	Clean-up	Pre-concentration
Drinking water (Yes or No)								
Space to provide further details:								
Surface (fresh) water (Yes or No)								
Space to provide further details:								
Suspended Particulate Matter (Yes or No)								
Space to provide further details:								
Surface (saline) waters (Yes or No)								
Space to provide further details:								
Wastewater Treatment Works effluent (Yes or No)								
Space to provide further details:								
Biosolids/ Sewage Sludge (Yes or No)								
Space to provide further details:								
Leachate (Yes or No)								
Space to provide further details:								
Groundwater (Yes or No)								
Space to provide further details:								
Sediment (Yes or No)								
Space to provide further details:								
Biota- animals (Yes or No)								
Space to provide further details:								
Biota- plants (Yes or No)								
Space to provide further details:								
Soil (Yes or No)								
Space to provide further details:								

Space to provide further details:				
Compost/mulch (Yes or No)				
Space to provide further details:				
Air (Yes or No)				
Space to provide further details:				
Flue gas (Yes or No)				
Space to provide further details:				
Food- fish (Yes or No)				
Space to provide further details:				
Food- meat (Yes or No)				
Space to provide further details:				
Food- fruit and vegetables (Yes or No)				
Space to provide further details:				
Food- dairy (Yes or No)				
Space to provide further details:				
Indoor dust (Yes or No)				
Space to provide further details:				
Other (Please specify)				

12. What level of validation Please provide any addition					
Sample type	Not validated	In-house validation	Accredited (please specify your accreditation)	Have you participated in any external comparisons of reference standards? E.g. Ring Tests, Proficiency Testing and certified standards. (Please specify)	Do you use labelled or other internal standards? (Please specify)
Drinking water					
Surface (fresh) water					
Surface (saline) waters					
Wastewater Treatment Works effluent					
Biosolids/ sewage sludge					
Leachate					
Groundwater					
Sediment					
Biota- animals					
Biota- plants Soil					
Compost/mulch					
Air					
Flue gas					
Food- fish					
Food- meat					
Food- fruit and vegetables					
Food- dairy					
Indoor dust					
Other (Please specify)					

13. Do you consider different PFAS isomers (e.g. branched and linear) in your quality assurance for PFAS analysis? E.g. for PFOS

Please indicate: Yes, No or Sometimes.

14. If you monitor in biota- which species do you monitor for PFAS in?

15. If you monitor in biota- do you undertake full organism analysis or specific tissues for PFAS?- please specify which tissues i.e. full organism, fillet/muscle tissue, liver

Other:

16. Are vou utilisin	g non-target	(and suspect) screening ap	proaches to determine	presence of further PFAS substances?

Please indicate: Yes or No.

17. Do you have any capability to perform a Total Oxidisable Precursor (TOP) Assay on water samples?

Please indicate: Yes or No.

Space for further detail on the type of TOP assay you use e.g. original method, direct TOP:

18. Do you have capability to perform a TOP assay on any other sample type?

Please indicate: Yes or No.

19. Do you have any capability to undertake total fluorine methodology, extractable organic fluorine (EOF) or adsorbable organic fluorine (AOF)?

Total fluorine methodology (inorganic and organic)

Please indicate: Yes or No.

Extractable organic fluorine methodology

Please indicate: Yes or No.

Adsorbable organic fluorine methodology

Please indicate: Yes or No.

20. If you use extractable organic fluorine method- which type of extraction method are you using?

21. If you use adsorbable organic fluorine method- which type of extraction method are you using?

22. Are you working towards analysing for PFAS Total connected to the EU Drinking Water Directive?

Please indicate: Yes or No.

23. In relation to Drinking Water Analysis do you intend to sum the 20 PFAS values or include something broader to cover total PFAS?

Please indicate Sum or Other.

Other (please give more details):

24. Are you using bioassays to assess PFAS currently?

Please indicate: Yes or No.

25. If you are using bioassays, which bioassay(s) are you using for this purpose?

26. Which areas of PFAS analysis is your laboratory currently working on developing?

27. Did you use any standard publications to support your method development for PFAS analysis? Please specify and include hyperlinks if available.

28. Is there any other information that you would like to provide about your laboratories current PFAS analytical capabilities which you have not covered in your questionnaire response? Please provide any additional information in the blank section below...

Appendix 2: List of PFAS, acronyms and CAS numbers referred to in this report

#	Name	Abbreviation	CAS No	OECD*	US DSSTOX ^{\$}	US Master
		Carboxylic acids			•	
1	Perfluorobutanoic acid	PFBA; HFBA	375-22-4	Y	Y	Y
2	Perfluoropentanoic acid	PFPA, PFPeA	2706-90-3	Y	Y	Y
3	Perfluorohexanoic acid	PFHxA	307-24-4	Y	Y	Y
4	Perfluoroheptanoic acid	PFHpA	375-85-9	Y	Y	Y
5	Perfluorooctanoic acid	PFOA	335-67-1	Y	Y	Y
6	Perfluorononanoic acid	PFNA	375-95-1	Y	Y	Y
7	Perfluorodecanoic acid	PFDA	335-76-2	Y	Y	Y
8	Perfluoroundecanoic acid	PFUnA; PFUdA	2058-94-8	Y	Y	Y
9	Perfluorododecanoic acid	PFDoA	307-55-1	Y	Y	Y
10	Perfluorotridecanoic acid	PFTrDA; PFTriA	72629-94-8	Y	Y	Y
11	Perfluorotetradecanoic acid	PFTeA; PFTreA; PFTeDA	376-06-7	Y	Y	Y
12	Perfluorohexadecanoic acid	PFHxDA	67905-19-5	Y	Y	Y
13	Perfluorooctadecanoic acid	PFODA	16517-11-6	Y	Y	Y
		Sulfonic acids				
14	Perfluorobutanesulfonic acid	PFBS	375-73-5	Y	Y	Y
15	Perfluoropentanesulfonic acid	PFPeS	2706-91-4	Y	Y	Y
16	Perfluorohexanesulfonic acid	PFHxS	355-46-4	Y	Y	Y
17	Perfluoroheptanesulfonic acid	PFHpS	375-92-8	Y	Y	Y
18	Perfluorooctanesulfonic acid	PFOS	1763-23-1	Y	Y	Y
19	Perfluorononanesulfonic acid	PFNS	68259-12-1	Y	Y	Y
20	Perfluorodecanesulfonic acid	PFDS	335-77-3	Y	Y	Y
21	Perfluoroundecane sulfonic acid	PFUnDS		N	N	Ν
22	Perfluorododecane sulfonic acid	PFDoS	79780-39-5	Y	Y	Y
23	Perfluorotridecane sulfonic acid			N	N	N
		Substitution products				
24	Hexafluoropropylene oxide dimer acid (Gen X)	HFPO-DA (Gen X)	13252-13-6	Y	Y	Y
25	Hexafluoropropylene oxide trimer acid	HFPO-TA	13252-14-7	Y	Y	Y
26	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	DONA; ADONA	919005-14-4	Y	Y	Y

	Perfluorooctanesul	fonamides (and other sulfonam	nides)			
28	Perfluorooctanesulfonamide	FOSA; PFOSA	754-91-6	Y	Y	Y
38	N-methylperfluorooctanesulfonamide	MeFOSA; N-MeFOSA	31506-32-8	Y	Y	Y
39	N-Ethylperfluorooctanesulfonamide	EtFOSA; N-EtFOSA	4151-50-2	Y	Y	Y
35	Perfluorobutylsulfonamide (perfluorobutane sulfonamide)	FBSA	30334-69-1	Y	Y	Y
36	Perfluorohexanesulfonamide	FHxSA	41997-13-1	Y	Y	Y
	Perfluoroo	ctanesulfonamidoethanols				
42	N-Methyl-N-(2- hydroxyethyl)perfluorooctanesulfonamide	MeFOSE	24448-09-7	Y	Y	Y
43	N-Ethyl-N-(2- hydroxyethyl)perfluorooctanesulfonamide	EtFOSE	1691-99-2	Y	Y	Y
	Perfluorooc	tanesulfonamidoacetic acids				
102	Perfluoro-1-octanesulfonamidoacetic acid	FOSAA	2806-24-8	N	Y	Y
40	2-(N-Methylperfluorooctanesulfonamido)acetic acid	NMeFOSAA; MeFOSAA	2355-31-9	Y	Y	Y
41	2-(N-Ethylperfluorooctanesulfonamido)acetic acid	NEtFOSAA; EtFOSAA	2991-50-6	Y	Y	Y
	Fluorinate	d Telomer sulfonates (TFS)				
29	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (F-53B Major)	6:2 CI-PFESA; 9CI-PF3ONS	756426-58-1	Y	Y	Y
30	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor)	8:2 CI-PFESA; 11CI-PF3OUdS	763051-92-9	N	Y	Y
31	4:2 Fluorotelomer sulfonic acid	4:2 FTSA; 4:2 FTS	757124-72-4	Y	Y	Y
32	6:2 Fluorotelomer sulfonic acid	6:2 FTSA; 6:2 FTS	27619-97-2	Y	Y	Y
33	8:2 Fluorotelomer sulfonic acid	8:2 FTSA; 8:2 FTS	39108-34-4	Y	Y	Y
34	10:2 Fluorotelomer sulfonic acid	10:2 FTSA	120226-60-0	Y	Y	Y
		ra Short Chain PFAS				
52	Trifluoroacetic acid	TFA, TFAA	76-05-1	N	N	N
53	Perfluoropropanoic acid	PFPrA	422-64-0	N	Ν	Y
54	Trifluoromethane sulfonic acid	TFMS	1493-13-6	Ν	Ν	Ν
55	Perfluoroethane sulfonic acid	PFEtS	354-88-1	N	N	Y
56	Perfluoropropate sulfonic acid	PFPrS	423-41-6	Ν	Y	Y
		F-Gases				
57	Fluoroform	HFC-23	75-46-7	Ν	Ν	Ν
58	Difluoromethane	HFC-32	75-10-5	N	N	Ν

59	1,1,1,2,2,3,4,5,5,5- Decafluoropentane	HFC-43-10mee	138495-42-8	N	N	Y
60	Pentafluoroethane	HFC-125	354-33-6	N	Ν	Y
61	1,1,1,2-Tetrafluoroethane	HFC-134a	811-97-2	N	Ν	Y
62	1,1,1-Trifluoroethane	HFC-143a	420-46-2	Ν	Ν	N
63	1,1-Difluoroethane	HFC-152a	75-37-6	Ν	Ν	N
64	1,1,1,2,3,3,3-Heptafluoropropane	HFC-227ea	431-89-0	Ν	Ν	Y
65	1,1,1,3,3,3-Hexafluoropropane	HFC-236fa	690-39-1	Ν	Ν	Y
66	1,1,1,3,3-Pentafluoropropane	HFC-245fa	460-73-1	Ν	Ν	N
67	1,1,1,3,3-Pentafluorobutane	HFC-365mfc	406-58-6	Ν	Ν	Ν
68	1-Chloro-1,2,2,2-tetrafluoroethane	HCFC-124	2837-89-0	Ν	Ν	Y
69	1,1-Dichloro-1-fluoroethane	HCFC-141b	1717-00-6	Ν	Ν	N
70	3,3-Dichloro-1,1,1,2,2-pentafluoropropane	HCFC-225ca/cb	422-56-0	Ν	Ν	Y
71	1-Chloro-2,3,3,3-tetrafluoropropene	HFO-1224yd	111512-60-8	Ν	Ν	Y
72	1-Chloro-3,3,3-trifluoro-1-propene	HFO-1233zd(E)	102687-65-0; 2730- 43-0	Ν	Ν	Ν
73	2,3,3,3-Tetrafluoropropene	HFO-1234yf	754-12-1	Ν	Ν	Y
74	1,3,3,3-Tetrafluoropropene	HFO-1234ze(E)	1645-83-6	Ν	Ν	N
75	Trans-1,1,1,4,4,4-hexafluorobut-2-ene	HFO-1336mzz(E)	692-49-9	Ν	Ν	N
76	Cis-1,1,1,4,4,4-Hexafluoro-2-butene	HFO–1336mzz(Z)	692-49-9	Ν	Ν	N
77	2-Bromo-3,3,3-trifluoroprop-1-ene	Halotron BrX, 2-BTP	1514-82-5	Ν	Ν	N
78	Methoxytridecafluoro-heptene isomers	MPHE, Sion	No data	Ν	Ν	N
79	Dodecafluoro-2-methyl-3-pentanone	NOVEC 612	756-13-8	Y	Ν	Y
80	2,3,3,3-tetrafluoro-2- (trifluoromethyl)propanenitrile	NOVEC 4710	42532-60-5	Y	Ν	Y
81	1,1,2,2-Tetrafluoro-1-(2,2,2-trifluoroethoxy) ethane	HFE-347pc-f2	406-78-0	Ν	Ν	Y
82	Methyl nonafluorobutyl ether + Methyl nonafluoroisobutyl ether	HFE-449s1 (7100)	163702-08-7; 163702-07-6	Ν	Ν	Ν
83	1-Ethoxy-nonafluorobutane	HFE-569sf2 (7200)	163702-05-4	Y	Y	Y
84	3-Ethoxyperfluoro(2-methylhexane)	HFE-7500	297730-93-9	Y	Y	Y
		orinated telomer alcohols				
85	2-perfluorobutyl ethanol (4:2)	FBET	2043-47-2	Y	Y	Y
86	1-perfluoropentyl ethanol (5:2 secondary)	5:2sFTOH	914637-05-1	Y	Y	Y
87	2-pefluorohexyl ethanol (6:2)	FHET	647-42-7	Y	Y	Y
88	1-pefluoroheptyl ethanol (7:2 secondary)	7:2sFTOH	24015-83-6	Ν	Y	Y

90 103 104	2-pefluorodecyl ethanol (10:2) Fluo 2-perfluorohexyl ethanoic acid (6:2) 2-perfluorooctyl ethanoic acid (8:2)	FDET rinated Telomer acids	865-86-1	Y	Y	Y
	2-perfluorohexyl ethanoic acid (6:2)					
104	2-perfluorooctyl ethanoic acid (8·2)	6:2 FTA, FHEA	53826-12-3	Y	Y	Y
		8:2 FTA, FOEA	27854-31-5	Y	Y	Y
105	2-Perfluorodecyl ethanoic acid (10:2)	10:2 FTA /FDEA	53826-13-4	Y	Y	Y
106	3-Perfluoropropyl propanoic acid (3:3)	FPrPA	356-27-4	Y	Y	Y
27	2H,2H,3H,3H-Perfluorooctanoic acid	5:3 FTCA	914637-49-3	Y	Y	Y
107	3-Perfluoropentyl propanoic acid (5:3)	5:3 FTCA / FPePA	914637-49-3	Y	Y	Y
108	3-Perfluoroheptyl propanoic acid (7:3)	7:3 FTCA / FHpPA	812-70-4	Y	Y	Y
	Fluorinated	Unsaturated Telomer acids				
109	2H-Perfluoro-2-octenoic acid (6:2)	6:2 FTUCA / FHUEA	70887-88-6	Y	Y	Y
110	2H-Perfluoro-2-decenoic acid (8:2)	8:2 FTUCA / FOUEA	70887-84-2	Y	Y	Y
97	2H-perfluoro-2-decenoic acid (8:2)	8:2 FTUA, FOUEA	70887-84-2	Y	Y	Y
111	2H-Perfluoro-2-dodecenoic acid (10:2)	10:2 FTUCA / FDUEA	70887-94-4	Y	Y	Y
	Fluorinated telor	ner acrylates, acetates and iod	ides			
125	6:2 Fluorotelomer acrylate	6:2FTAcr	17527-29-6	Y	Y	Y
91	1H,1H,2H,2H-perfluorodecyl acrylate	8:2FTAcr	27905-45-9	Ν	Ν	N
92	1H,1H,2H,2H-perfluorododecyl acrylate	10:2FTAcr	17741-60-5	Y	Y	Y
93	1H,1H,2H,2H-perfluorodecyl acetate	8:2FTOAc	37858-04-1	Y	Y	Y
94	1H,1H,2H,2H-perfluorododecyl acetate	10:2FTOAc	37858-05-2	Ν	Y	Y
123	6:2 Fluorotelomer methacrylate		2144-53-8	Y	Y	Y
124	8:2 Fluorotelomer methacrylate		1996-88-9	Y	Y	Y
126	4:2 Fluorotelomer iodide	4:2 FTI	2043-55-2	Y	Y	Y
127	6:2 Fluorotelomer iodide	6:2 FTI	2043-57-4	Y	Y	Y
	Aqueous	Film-Forming Foam PFAS				
95	2-[(4,4,5,5,6,6,7,7,8,8,8- Undecafluorooctyl)dimethyl-ammonio]acetate	5:3FTB		Ν	Ν	N
96	2-[(3,4,4,5,5,6,6,7,7,8,8,8- Dodecafluorooctyl)dimethyl- ammonio]acetate	5:1:2FTB		N	Ν	N
98	DPOSA	Capstone 1183 A	80475-32-7	Y	Y	Y
99	CDPOS	Capstone 1157 B	34455-29-3	Ŷ	Y	Ý
		proalkylphophonic acids		· ·	•	· ·

112	Perfluorohexylphosphonic acid	PFHxPA	40143-76-8	Y	Y	Y
113	6-Chloroperfluorohexylphosphonic acid	CI-PFHxPA	40143-76-10	Ν	Ν	N
114	Perfluorooctylphosphonic acid	PFOPA	40143-78-0	Y	Y	Y
115	Perfluorodecylphosphonic acid	PFDPA	52299-26-0	Y	Y	Y
	Perflu	oroalkyl phophinates				
116	bis(perfluorohexyl)phosphinic acid	6:6 PFPiA	40143-77-9	Y	Y	Y
117	perfluorohexylperfluorooctyl phosphinic acid	6:8 PFPiA	610800-34-5	Y	Y	Y
118	Sodium bis(perfluorooctyl)phosphinate	8:8PFPi	40143-79-1	Y	Y	Y
	Mono and di-substitut	ed Polyfluorinated Phosphate	esthers			
119	Sodium 1H,1H,2H,2H-perfluorooctylphosphate	6:2PAP	57678-01-0	Y	Y	Y
37	6:2 Fluorotelomer phosphate diester	6:2 diPAP	57677-95-9	Y	Y	Y
120	Sodium 1H,1H,2H,2H-perfluorodecylphosphate	8:2PAP	57678-03-2	Y	Y	Y
121	Sodium bis(1H,1H,2H,2H-perfluorodecyl)phosphate	8:2diPAP	678-41-1	Y	Y	Y
		Other				
44	Perfluoro[(2-ethyloxy-ethoxy)acetic acid], ammonium salt	EEA-NH4	908020-52-0	Ν	Ν	Ν
45	Perfluorobutylethlyene	PFBE	19430-93-4	Ν	Ν	Ν
46	3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctyl methacrylate		2144-53-8	Y	Y	Y
47	1-Propanamine, 1,1,2,2,3,3,3-heptafluoro-N,N- bis(1,1,2,2,3,3,3-heptafluoropropyl)- [Perfluamine]		338-83-0	Y	Y	Y
48	Carboxymethyldimethyl-3- [[(3,3,4,4,5,5,6,6,7,7,8,8,8- tridecafluorooctyl)sulfonyl]amino]propylammoniu m hydroxide	6:2 FTAB	34455-29-3	Y	Y	Y
49	N-[3-(Dimethylamino)propyl]- 3,3,4,4,5,5,6,6,7,7,8,8,8- tridecafluorooctanesulfonamide N-oxide	FTSAAm	80475-32-7	Y	Y	Y
50	Perfluoro-4-ethylcyclohexane sulfonic acid	PFECHS	646-83-3	Ν	N	Y
51	Perfluoro([5-methoxy-1,3-dioxolan-4-yl]oxy) acetic acid	C6O4	1190931-27-1	Y	Ν	Ν
122	Ammonium difluoro[1,1,2,2-tetrafluoro-2- (pentafluoroethoxy) ethoxy]acetate		908020-52-0	Y	Ν	Y
128	Perfluoro-3-methoxypropanoic acid / Perfluoro-4- oxapentanoic acid (PFMPA)	PFMPA / PF4OPeA	377-73-1	Y	Ν	Y

129	Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	Y	Ν	Y
130	Perfluoro-3,6-dioxaheptanoic acid (NFDHA)	3,6-OPFHpA	151772-58-6	Y	Ν	Y

Original list updated from the original questionnaire to include additional compounds and abbreviations submitted by laboratories.

*OECD (Information on individual structure categories used in the spreadsheets and supplementary information)

https://www.oecd.org/officialdocuments/publicdisplaydocumentpdf/?cote=ENV/CBC/MONO%282021%2925&docLanguage=en

[&]US DSSTox (DTXSID records – List definition as of March 2018). <u>https://www.epa.gov/chemical-research/distributed-structure-searchable-toxicity-dsstox-database</u>

[^]PFASMASTER (Last Updated: August 10th 2021) <u>https://comptox.epa.gov/dashboard/chemical-lists/pfasmaster</u>

Appendix 3: Limits of Detection ranges per matrix and PFAS

Table A3.1: Number of reported LODs of individual compounds in different matrices. Colours indicate the number of laboratories from none (red) to a relatively high number (green).

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
PFBA; HFBA	25	27	11	16	7	23	5	10	17	15	3	13	4	0	0	2	0
PFPA, PFPeA	25	27	11	17	7	22	5	10	16	14	3	14	4	0	0	2	1
PFHxA	30	32	12	20	7	27	5	11	19	16	3	17	5	1	0	2	1
PFHpA	28	29	10	19	7	25	5	11	18	15	3	16	5	1	0	2	1
PFOA	30	33	13	20	7	27	5	12	19	16	3	20	5	1	0	2	1
PFNA	28	29	12	17	7	24	5	11	19	16	3	18	5	1	0	2	1
PFDA	29	30	12	18	7	25	5	11	19	16	3	17	5	1	0	2	1
PFUnA; PFUdA	25	27	11	17	7	23	5	11	19	16	3	16	4	1	0	2	1
PFDoA	25	26	11	16	7	23	5	11	19	16	3	16	4	1	0	2	1
PFTrDA; PFTriA	19	20	8	12	5	18	4	10	16	12	3	11	3	1	0	1	1
PFTeA; PFTreA; PFTeDA	16	17	7	11	5	16	4	9	15	12	3	11	3	1	0	1	1
PFHxDA	5	6	2	6	2	8	2	5	6	6	2	5	2	1	0	2	0
PFODA	4	6	1	4	2	7	2	6	6	5	2	5	2	1	0	1	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
PFBS	27	29	11	18	7	24	5	11	17	15	3	16	5	1	0	2	1
PFPeS	18	18	7	9	4	16	4	8	11	12	2	8	3	1	0	1	0
PFHxS	29	30	11	18	7	25	5	11	18	15	3	17	5	1	0	2	1
PFHpS	21	21	8	13	6	19	4	9	16	14	3	13	3	1	0	1	1
PFOS	30	35	13	20	7	27	5	12	20	16	3	22	5	1	0	2	1
PFNS	18	18	7	9	4	16	4	8	12	12	2	8	3	1	0	2	0
PFDS	21	22	9	11	6	19	4	9	16	13	3	12	3	1	0	0	1
PFUnDS	8	7	0	2	1	8	0	2	4	4	0	2	1	1	0	1	0
PFDoS	9	8	1	3	2	9	1	4	6	5	0	3	1	1	0	0	0
PFTrDS	2	1	0	0	0	2	0	1	2	2	0	1	1	1	0	0	0
HFPO-DA (Gen X)	17	18	9	10	4	15	3	5	7	6	1	8	3	1	0	1	0
HFPO-TA	3	3	2	1	1	3	0	1	3	2	0	1	1	1	0	0	0
DONA; ADONA	12	13	7	6	4	12	3	4	7	6	1	7	2	1	0	1	0
FOSA; PFOSA	13	13	7	8	4	12	3	5	10	8	2	11	5	1	1	2	2
MeFOSA; N-MeFOSA	6	5	1	6	0	7	1	3	3	5	1	3	2	0	0	2	1
EtFOSA; N-EtFOSA	5	6	1	6	1	8	2	4	5	7	2	4	2	0	0	2	1

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
FBSA	2	2	2	1	1	3	0	1	3	2	0	1	0	0	0	0	0
FHxSA	3	3	2	1	1	4	0	1	3	2	0	1	0	0	0	0	0
MeFOSE	2	1	0	0	0	2	0	0	1	1	0	2	1	0	0	1	0
EtFOSE	1	0	0	0	0	1	1	1	1	1	1	1	0	0	0	1	0
FOSAA	1	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0
NMeFOSAA; MeFOSAA	12	13	6	8	3	14	3	5	7	8	1	5	3	0	0	1	1
NEtFOSAA; EtFOSAA	13	13	6	8	3	14	3	5	7	8	1	5	3	0	0	1	1
6:2 CI-PFESA; 9CI-PF3ONS	11	11	7	6	4	11	2	3	7	6	1	8	3	0	0	1	0
8:2 CI-PFESA; 11CI-																	
PF3OUdS	9	9	6	4	3	9	2	3	7	6	1	4	3	0	0	1	0
4:2 FTSA; 4:2 FTS	15	15	6	8	5	15	4	7	13	13	1	11	3	0	0	1	0
6:2 FTSA; 6:2 FTS	16	17	7	9	6	16	4	7	13	12	1	12	3	0	0	0	0
8:2 FTSA; 8:2 FTS	16	17	7	10	5	16	4	7	13	13	1	12	3	0	0	1	0
10:2 FTSA	2	1	0	1	0	2	0	0	1	1	0	2	1	0	0	0	0
FBET	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
5:2sFTOH	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
FHET	1	1	0	1	0	1	0	1	0	1	0	1	0	0	0	0	0
7:2sFTOH	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
FOET	1	1	0	1	0	1	0	1	0	1	0	1	0	0	0	0	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
FDET	1	1	0	1	0	1	0	1	0	1	0	1	0	0	0	0	0
6:2 FTA, FHEA	2	1	1	0	0	0	0	0	0	0	0	2	1	0	0	0	0
8:2 FTA, FOEA	2	1	1	0	0	0	0	0	0	0	0	2	1	0	0	0	0
10:2 FTA /FDEA	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
FPrPA	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
5:3 FTCA	2	3	1	2	0	2	0	1	2	2	0	3	2	0	0	0	0
5:3 FTCA / FPePA	0	0	0	0	0	0	1	1	0	0	0	2	0	0	0	1	0
7:3 FTCA / FHpPA	0	0	0	0	0	0	1	1	0	0	0	2	0	0	0	1	0
6:2 FTUCA / FHUEA	1	1	1	0	0	0	1	1	0	0	0	3	1	0	0	1	0
8:2 FTUCA / FOUEA	0	0	0	0	0	0	1	1	0	0	0	1	0	0	0	1	0
8:2 FTUA, FOUEA	1	2	2	0	0	0	0	0	0	0	0	2	1	0	0	0	0
10:2 FTUCA / FDUEA	0	0	0	0	0	0	1	1	0	0	0	2	0	0	0	1	0
6:2FTAcr	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0
8:2FTAcr	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0
10:2FTAcr	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8:2FTOAc	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10:2FTOAc	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
6:2 FTM	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0
8:2 FTM	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0
4:2 FTI	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
6:2 FTI	1	1	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0
TFA, TFAA	3	3	0	1	0	4	0	0	0	0	0	0	0	0	0	0	0
PFPrA	1	2	0	1	0	2	0	1	0	0	0	0	0	0	0	0	0
TFMS	2	2	0	0	0	2	0	0	0	0	0	0	0	0	0	0	0
PFEtS	0	1	0	0	0	0	0	1	0	0	0	0	0	0	0	0	0
PFPrS	1	1	0	0	0	1	0	1	0	0	0	0	0	0	0	0	0
HFC-23	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-32	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-43-10mee	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-125	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-134a	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-143a	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-152a	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-227ea	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-236fa	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-245fa	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFC-365mfc	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
HCFC-124	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-141b	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-225ca/cb	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1224yd	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1233zd(E)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1234yf	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1234ze(E)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1336mzz(E)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFO–1336mzz(Z)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Halotron BrX, 2-BTP	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
MPHE, Sion	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 612	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 4710	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFE-347pc-f2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFE-449s1 (7100)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HFE-569sf2 (7200)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
HFE-7500	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
5:3FTB	1	1	1	1	1	1	1	1	1	1	0	1	1	0	0	0	0
5:1:2FTB	1	1	1	1	1	1	1	1	1	1	0	1	1	0	0	0	0
Capstone 1183 A	0	1	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0
Capstone 1157 B	0	1	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0
PFHxPA	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
CI-PFHxPA	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
PFOPA	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
PFDPA	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
6:6 PFPiA	1	1	1	0	0	0	0	0	0	0	0	1	0	0	0	0	0
6:8 PFPiA	1	1	1	0	0	0	0	0	0	0	0	1	0	0	0	0	0
8:8PFPi	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
6:2PAP	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
6:2 diPAP	4	3	3	2	2	3	2	3	2	2	1	4	2	0	0	1	0
8:2PAP	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
8:2diPAP	0	0	0	1	0	0	0	0	0	0	0	1	0	0	0	0	0
EEA-NH4	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
PFBE	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Perfluamine	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

	Drinking Water (ng/L)	Surface (fresh) waters (ng/L)	Surface (saline) waters (ng/L)	WWT Effluent (ng/L)	Leachate (ng/L)	Groundwater (ng/L)	Suspended Particulate Matter (ng/g)	Biosolids/Sewage Sludge (ng/g)	Sediment (ng/g)	Soil (ng/g)	Compost/ mulch (ng/g)	Biota (animals) (ng/g)	Biota (plants) (ng/g)	Air (ng/L)	Flue gas	Indoor dust (ng/g)	Blood serum (ng/g)
6:2 FTAB	2	2	0	2	1	2	0	0	1	1	0	0	0	0	0	0	0
FTSAAm	1	1	0	1	1	1	0	0	1	1	0	0	0	0	0	0	0
PFECHS	4	4	3	3	2	4	3	3	3	3	1	6	2	0	0	1	0
C6O4	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
PFE	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
PFMPA / PF4OPeA	2	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0
PFEESA	1	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0
3,6-OPFHpA	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

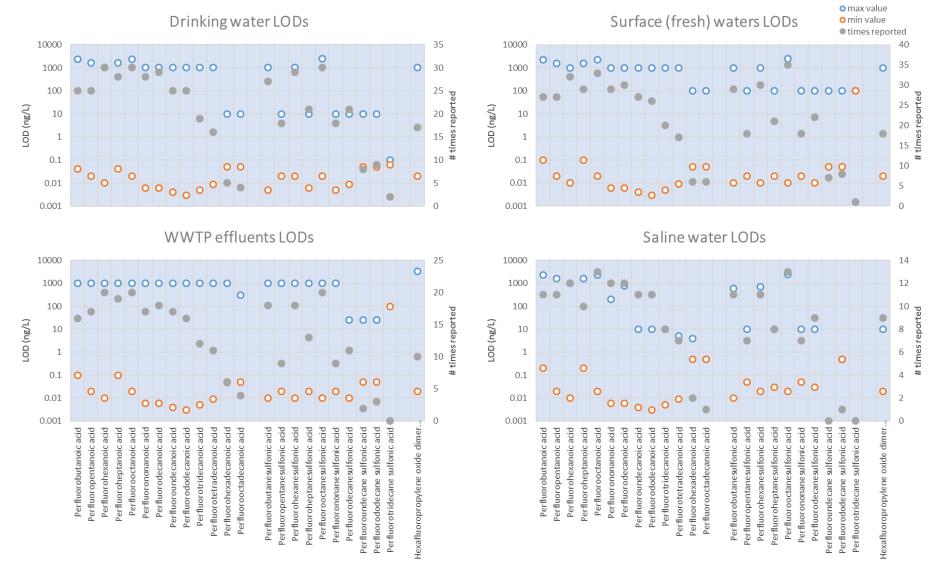


Figure A3a: LODs in drinking water, surface water, WWTP and saline waters

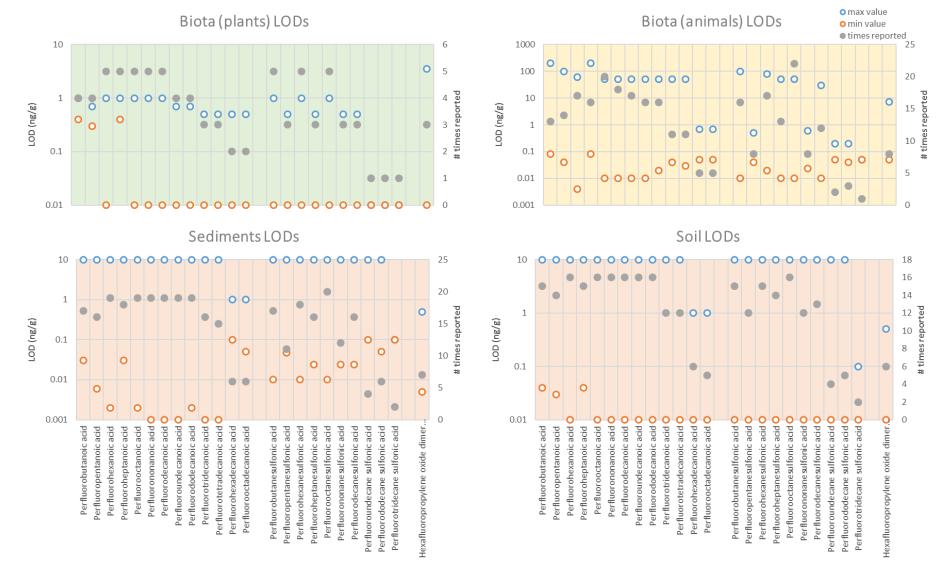


Figure A3b: LODs in biota (plants), biota (animals), sediments and soil

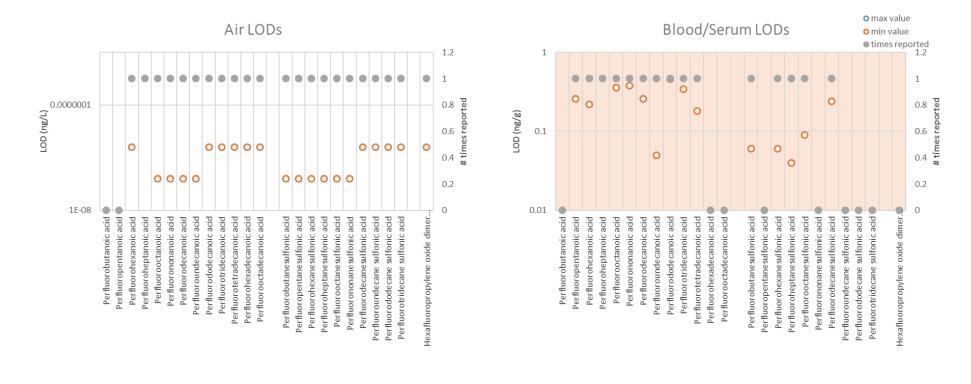


Figure A3c: LODs in air and blood/serum

Table A3.2: Maximum (max) and minimum LOD reported for PFAS individual compounds in aqueous matrices.

	Drinking	y Water	Surface	(fresh)	Surface	(saline)	WWTW	Effluent	Leac	hate	Groun	dwater
	(ng		waters		waters		(ng			;/L)		;/L)
	max /alue	min value	max value	min value								
PFBA; HFBA	2300	0.042	2300	0.1	2300	0.2	1000	0.5	50	0.5	100	0.17
PFPA, PFPeA	1600	0.02	1600	0.02	1600	0.02	1000	0.4	20	0.4	100	0.1
PFHxA	1000	0.01	1000	0.01	1000	0.01	1000	0.1	50	0.1	1000	0.02
PFHpA	1600	0.042	1600	0.1	1600	0.2	1000	0.5	50	0.5	100	0.17
PFOA	2300	0.02	2300	0.02	2300	0.02	1000	0.05	20	0.05	1000	0.02
PFNA PFDA	1000 1000	0.006	1000 1000	0.006	200 800	0.006	1000 1000	0.05 0.05	10 20	0.05	1000 1000	0.02
PFUnA; PFUdA	1000	0.008	1000	0.008	10	0.008	1000	0.05	20	0.05	1000	0.02
PFDoA	1000	0.003	1000	0.003	10	0.003	1000	0.05	10	0.05	1000	0.02
PFTrDA; PFTriA	1000	0.005	1000	0.005	10	0.005	1000	0.05	5	0.05	1000	0.05
PFTeA; PFTreA;												
PFTeDA	1000	0.009	1000	0.009	5	0.009	1000	0.1	5	0.1	1000	0.05
PFHxDA PFODA	10	0.05	100	0.05	4	0.5	1000	0.5	2	0.5	100	0.05
PFODA	10 1000	0.05	100 1000	0.05	0.5 600	0.5 0.01	300 1000	0.5 0.05	2 10	0.5 0.05	100 1000	0.05
PFPeS	1000	0.003	1000	0.01	10	0.01	1000	0.05	5	0.05	1000	0.02
PFHxS	1000	0.02	1000	0.01	700	0.02	1000	0.05	10	0.05	1000	0.02
PFHpS	10	0.006	100	0.02	10	0.03	1000	0.05	10	0.05	100	0.02
PFOS	2500	0.02	2500	0.01	2500	0.02	1000	0.05	20	0.05	1000	0.02
PFNS	10	0.005	100	0.02	10	0.05	1000	0.05	5	0.05	100	0.02
PFDS	10	0.009	100	0.01	10	0.03	25	0.05	10	0.05	100	0.02
PFUnDS	10	0.05	100	0.05	0	0	25	1	1	1	100	0.05
PFDoS PFTrDS	10	0.05	100	0.05	0.5	0.5	25	0.5	1	0.5	100	0.05
HFPO-DA (Gen X)	0.1	0.062	100 1000	100 0.02	0 10	0.02	0 3300	0 0.5	0 5	0.5	100 1000	0.1
HFPO-TA	0.5	0.02	0.5	0.02	0.5	0.02	0.5	0.5	0.5	0.5	5	0.05
DONA; ADONA	10	0.004	10	0.008	5	0.004	1000	0.5	5	0.5	10	0.037
FOSA; PFOSA	12.6	0.008	18.9	0.008	22.7	0.008	1000	0.05	2	0.05	10	0.044
MeFOSA; N-												
MeFOSA	10	0.05	100	0.05	0.2	0.2	1000	0.29	0	0	100	0.05
EtFOSA; N-EtFOSA	10	0.05	100	0.05	0.8	0.8	1000	0.5	5	5	100	0.05
FBSA FHxSA	2	0.5	2	0.5 0.072	2	0.5 0.5	0.5 0.5	0.5 0.5	0.5 0.5	0.5 0.5	2	0.5 0.072
MeFOSE	0.25	0.019	0.05	0.072	0	0.5	0.3	0.3	0.3	0.3	0.25	0.072
EtFOSE	0.25	0.05	0.05	0.05	0	0	0	0	0	0	0.25	0.25
FOSAA	0.1	0.1	0	0	0	0	0	0	0	0	0.1	0.1
NMeFOSAA;												
MeFOSAA	1000	0.05	1000	0.05	5	0.05	1000	0.05	5	0.05	1000	0.05
NEtFOSAA; EtFOSAA	1000	0.023	1000	0.05	5	0.05	1000	0.05	5	0.05	1000	0.05
6:2 CI-PFESA; 9CI- PF3ONS	10	0.007	10	0.007	5	0.007	1000	0.18	5	0.5	10	0.038
8:2 CI-PFESA; 11CI-	10	0.007	10	0.007	5	0.007	1000	0.10	5	0.5	10	0.050
PF3OUdS	10	0.007	10	0.007	5	0.007	1000	0.5	5	0.5	10	0.05
4:2 FTSA; 4:2 FTS	10	0.001	100	0.03	5	0.03	1000	0.5	10	0.5	100	0.05
6:2 FTSA; 6:2 FTS	19	0.044	100	0.025	5	0.07	300	0.5	10	0.5	100	0.05
8:2 FTSA; 8:2 FTS	10	0.006	100	0.04	5	0.04	1000	0.1916	10	0.5	100	0.04
10:2 FTSA	0.1	0.05	0.05	0.05	0	0	300	300	0	0	0.1	0.05
FBET	0	0	0	0	0	0	0	0	0	0	0	0
5:2sFTOH FHET	0 50	0 50	0 50	0 50	0	0	0 50	0 50	0	0	0 50	0 50
7:2sFTOH	50 0	<u>50</u> 0	<u>50</u> 0	50	0	0	0	50 0	0	0	50	50
FOET	50	50	50	50	0	0	50	50	0	0	50	50
FDET	25	25	25	25	0	0	25	25	0	0	25	25
6:2 FTA, FHEA	17000	0.1	17000	17000	17000	17000	0	0	0	0	0	0
8:2 FTA, FOEA	11000	0.1	11000	11000	11000	11000	0	0	0	0	0	0
10:2 FTA /FDEA	0.1	0.1	0	0	0	0	0	0	0	0	0	0
FPrPA	0	0	0	0	0	0	0	0	0	0	0	0
5:3 FTCA	500	5	500	0.5	500	500	5	0.5	0	0	5	1
5:3 FTCA / FPePA 7:3 FTCA / FHpPA	0	0	0	0	0	0	0	0	0	0	0	0
6:2 FTUCA / FHUEA	200	200	200	200	200	200	0	0	0	0	0	0
8:2 FTUCA / FOUEA	0	0	0	0	0	0	0	0	0	0	0	0
	-	-			-							
8:2 FTUA, FOUEA	700	700	700	0.1	700	0.1	0	0	0	0	0	0

	(ng max	<mark>:/L)</mark> min	waters	$(n\sigma/I)$	wetere	1 1- 2						
6:2FTAcr		main			waters	(ng/L)	(ng		(ng	g/L)	(ng	
6:2FTAcr	Value	value	max value	min value	max value	min	max value	min	max	min value	max	min value
	value 50	value 50	value 50	value 50	value 0	value 0	50	value 50	value 0	value 0	value 50	50
	50	50	50	50	0	0	50	50	0	0	50	50
10:2FTAcr	0	0	0	0	0	0	0	0	0	0	0	0
8:2FTOAc	0	0	0	0	0	0	0	0	0	0	0	0
10:2FTOAc	0	0	0	0	0	0	0	0	0	0	0	0
6:2 FTM	25	25	25	25	0	0	25	25	0	0	25	25
8:2 FTM	50	50	50	50	0	0	50	50	0	0	50	50
4:2 FTI	10	10	10	10	0	0	10	10	0	0	10	10
6:2 FTI	20	20	20	20	0	0	20	20	0	0	10	10
TFA, TFAA	50	20	100	25	0	0	200	200	0	0	100	20
PFPrA	0.25	0.25	100	1	0	0	1	1	0	0	100	0.25
TFMS	5	2	5	0.002	0	0	0	0	0	0	5	2
PFEtS	0	0	0.001	0.001	0	0	0	0	0	0	0	0
PFPrS	0.25	0.25	0.0003	0.0003	0	0	0	0	0	0	0.25	0.25
HFC-23	0	0	0	0	0	0	0	0	0	0	0	0
HFC-32	0	0	0	0	0	0	0	0	0	0	0	0
HFC-43-10mee	0	0	0	0	0	0	0	0	0	0	0	0
HFC-125	0	0	0	0	0	0	0	0	0	0	0	0
HFC-134a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-143a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-152a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-227ea	0	0	0	0	0	0	0	0	0	0	0	0
HFC-236fa	0	0	0	0	0	0	0	0	0	0	0	0
HFC-245fa	0	0	0	0	0	0	0	0	0	0	0	0
HFC-365mfc	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-124	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-141b	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-225ca/cb	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1224yd HFO-1233zd(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-123320(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1234ze(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1336mzz(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1336mzz(Z)	0	0	0	0	0	0	0	0	0	0	0	0
Halotron BrX, 2-BTP	0	0	0	0	0	0	0	0	0	0	0	0
MPHE, Sion	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 612	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 812 NOVEC 4710	0	0	0	0	0	0	0	0	0	0	0	0
HFE-347pc-f2	0	0	0	0	0	0	0	0	0	0	0	0
	0	0	0	0	0			0	0	0	0	0
HFE-449s1 (7100) HFE-569sf2 (7200)	0	0	0	0	0	0	0	0	0	0	0	0
				0						_		0
HFE-7500	0	0	0		0	0	0	0	0	0	0	0
5:3FTB	1	1	1	1	1	1	1	1	1	1	1	1
5:1:2FTB	1	1	1	1	1	1	1	1	1	1	1	
Capstone 1183 A	0	0	80	80	0	0	0	0	0	0	80	80
Capstone 1157 B PFHxPA	0	0	80	80	0	0	0	0	0	0	80	80
	0	0	0	0	0	0	0	0	0	0	0	0
CI-PFHxPA PFOPA	0	0	0	0	0	0	0	0	0	0	0	0
PFOPA	0	0	0	0	0	0	0	0	0	0	0	0
6:6 PFPiA	0.005	0.005	0.005	0.005	0.005	0.005	0	0	0	0	0	0
6:8 PFPIA	0.005	0.005	0.005	0.005	0.005	0.005	0	0	0	0	0	0
8:8PFPi												0
	0	0	0	0	0	0	0	0	0	0	0	0
6:2PAP	0	0	0	0	0	0	0	0	0	0	0	0
6:2 diPAP	300	0.25	300	0.5	300	0.5	0.5	0.5	5	0.5	5	0.25
8:2PAP	0	0	0	0	0	0	0	0	0	0	0	0
8:2diPAP	0	0	0	0	0	0	0.3	0.3	0	0	0	0
EEA-NH4	0	0	0	0	0	0	0	0	0	0	0	0
PFBE	0	0	0	0	0	0	0	0	0	0	0	0
Perfluamine 6:2 FTAB	0 200	0 25	0	0 25	0	0	0	0 25	0	0	0	0 25
6:2 FTAB FTSAAm			200		0	0	200		200	200	200	
PFECHS	200	200	200	200	0	0	200	200	200	200	200	200
Freuns	10 0	0.04	10 0	0.04	5 0	0.04	1000	0.5 0	5 0	1	10 0	0.5

	Drinkin (ng	g Water ;/L)	Surface waters	(fresh) (ng/L)		(saline) (ng/L)	WWTW (ng	Effluent ;/L)	Leac (ng	hate /L)	Ground (ng	
	max	min	max	min	max	min	max	min	max	min	max	min
	value	value	value	value	value	value	value	value	value	value	value	value
PFE*	0	0	0	0	0	0	0	0	0	0	0	0
PFMPA / PF4OPeA	0.1	0.05	0	0	0	0	0	0	0	0	0.1	0.1
PFEESA	0.1	0.1	0	0	0	0	0	0	0	0	0.1	0.1
3,6-OPFHpA	0.05	0.05	0	0	0	0	0	0	0	0	0	0

Table A3.3: Maximum (max) and minimum LOD reported for PFAS individual compounds in solid abiotic matrices.

PFRA 19 05 25 0.01 100 0.03 11 0.03 10 0.04 11 0.03 10 0.04 11 0.03 10 0.03 11 0.03 10 0.03 11 0.03 10 0.03 10 0.04 11 0.03 10 0.04 11 0.03 10 0.04 11 0.05 0.05 0.05 0.05 0.05 0.05 0.05 10 0.001 10 0.01 0.05 0.05 0.05 0.05 10 0.001 10 0.01		Suspe		Biosolids	/ Sewage	Sedi	ment	Sc	bil	Compos	t/ mulch
IPPEA 13 0 0.1 10 0.03 1 0 0.04 1 0 0 PFPA.PPEA 1 0.5 25 0.05 10 0.002 10 0.01 0.03 10 0.04 1 0.05 PFPA 0.3 0.05 25 0.05 10 0.01 10 0.01 0.5 0.05 PFPA 0.3 0.05 25 0.05 10 0.01 10 0.01 0.5 0.05 PFTOA 0.3 0.05 25 0.05 10 0.001 10 0.01 0.5 0.05 PFTOA 1 0.05 25 0.05 10 0.001 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 10 0.01 0.01 0.01 <th></th> <th></th> <th></th> <th>Sludge</th> <th>(ng/g)</th> <th>(ng</th> <th>g/g)</th> <th>(ng</th> <th>;/g)</th> <th>(ոք</th> <th>;/g)</th>				Sludge	(ng/g)	(ng	g/g)	(ng	;/g)	(ոք	;/g)
PFPA, PFPA 1 0.5 25 0.05 10 0.005 10 0.03 10 0.01 0.5 0.00 PFHA 1.9 0.5 25 0.01 10 0.03 10 0.04 1.5 0.05 PFNA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 10.0 PFNA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 10.0 PFNA 0.5 0.05 0.5 10 0.001 10 0.01 0.5 10.0 PFTCA 1 0.1 1 0.01 10 0.01 1.0 0.01 1.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0				max value	min value						
PFHAA 0.7 0.05 12 0.05 10 0.002 10 0.01 0.01 0.05 0.05 0.05 10 0.002 10 0.04 10 0.07 PFDA 0.5 0.05 10 0.001 10 0.01 0.5 0.05 PFDA 0.5 0.05 125 0.05 10 0.001 10 0.01 0.5 0.05 PFDA 0.5 0.05 125 0.05 10 0.001 10 0.01 0.5 0.05 PFTDA, FFTIA, 1 0.05 0.05 10 0.001 10 0.01 1 0.01 1 0.05 0.05 PFTDA, FFTIA, FFTIA, 0.5 0.5 0.5 0.05 11 0.061 10 0.01 10 0.01 10 0.01 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05											0.1
PFHA 1.9 0.5 1.5 0.1 10 0.03 10 0.04 1 0.05 PFOA 0.5 0.05 12 0.05 10 0.001 10 0.01 0.5 0.00 PFNA 0.5 0.05 10 0.001 10 0.01 0.5 0.00 PFNA 0.5 0.05 10 0.001 10 0.01 0.5 0.00 PFNA 0.5 0.05 10 0.001 10 0.01 0.5 0.0 PFTEAP 1 0.05 2.5 0.05 10 0.001 10 0.01 1 1.01 1 1.01 1 0.01 1 0.01 1 0.01 1 0.01 0.0 0.01 10 0.01 0.0 0.01 10 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0 0.01 0.0											0.05
PF0A 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 0.05 PFDA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 0.05 PFDA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 0.0 PFT0A 1 0.05 25 0.05 10 0.001 10 0.01 1 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01											0.03
PFYA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 0.00 PFDAA 0.5 0.05 25 0.05 10 0.001 10 0.01 0.5 0.0 PFDAA 0.5 0.05 25 0.05 10 0.002 10 0.01 0.5 0.0 PFTAA 1 0.05 25 0.05 10 0.001 10 0.01 1 0.01 10 0.01 10 0.01 10 0.01 1 0.01 10 0.01 10 0.01 10 0.01 10 0.01 0.01 0.0 10 0.01 0.0 10 0.01 0.0 10 0.01 0.0 10 0.01 0.0 0.05 0.05 10 0.01 0.01 0.0 0.0 0.0 10 0.01 0.05 0.0 0.05 0.05 0.05 10 0.00 10 0.0.0	· · · ·										0.05
PFU0A 0.5 0.06 25 0.05 10 0.001 10 0.01 0.5 0.0 PTGA 1 0.05 25 0.05 10 0.001 10 0.01 0.0 0.0 PTRA 1 0.1 10 0.01 10 0.01 1 0.01 0.0 0.0 0.0 PTRA 0.5 0.1 1 0.1 1 0.01 10 0.01 0.01 0.01 0.01 0.05 0.0 0.05 0.0 0.01 10 0.01 0.05 0.0 0.05 0.0 0.01 10 0.01 0.01 0.05 0.0 0.00 0.01 0.01 0.05 0.0 0.00 0.05 0.00 0.01 0.01 0.01 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0											0.05
PFD0A 0.5 0.05 25 0.05 10 0.002 10 0.01 0.0 1 0.00 PFT0A, PFT0A, PTT0A, PTT0A, PTT0A, PTT0A, PTT0A, D 0.1 10 0.1 10 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.05 0.01 0.01 0.01 0.05 0.01 0.01 0.01 0.01 0.05 0.01 0.01 0.01 0.05 0.00 0.01 0.01 0.05 0.01 0.05	PFDA	0.5	0.05	25	0.05	10	0.001	10	0.01	0.5	0.05
PFTCAD, PTICA 1 0.05 25 0.05 10 0.001 10 0.01 1 0.01 PTTEAD, FTEAD, 1 0.1 10 0.01 1 0.1 0.0 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.0 0.1 0.1 0.1 0.1 0.0 0.1 0.1 0.1 0.1 0.0											0.05
PFTEAL Interface I											0.1
PFRDA 1 01 10 0.01 1 0.01 1 0.01 1 0.01 1.001 0.05 0.01 PF0DA 05 0.05 50 0.01 1 0.001 0.01 0.01 0.01 0.05 0.05 PFPS 0.5 0.05 25 0.01 10 0.044 10 0.01 0.5 0.0 PFPs5 0.5 0.05 25 0.05 10 0.044 10 0.01 0.5 0.0 PFNS 0.5 0.05 25 0.05 10 0.01 0.0 <td< td=""><td></td><td>1</td><td>0.05</td><td>25</td><td>0.05</td><td>10</td><td>0.001</td><td>10</td><td>0.01</td><td>1</td><td>0.05</td></td<>		1	0.05	25	0.05	10	0.001	10	0.01	1	0.05
PF00A 50 05 50 01 1 005 1 001 05 0 PFPs 0.5 0.05 25 0.05 10 0.01 0.01 0.5 0.00 PFPuS 0.5 0.05 25 0.1 10 0.04 10 0.01 0.5 0.0 PFPuS 0.5 0.05 25 0.05 10 0.01 10 0.01 0.5 0.0 PFVAS 0.5 0.05 25 0.05 10 0.01 0.0 0.01 0.5 0.0 PFVOS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.5 0.0		1	0.1	10	0.1	10	0.001	10	0.01	1	0.15
PPES 0.5 0.05 25 0.1 10 0.04 10 0.01 0.5 0.0 PPHS 0.5 0.05 25 0.05 10 0.04 10 0.01 0.5 0.0 PPHS 0.5 0.05 25 0.05 10 0.024 10 0.01 0.5 0.0 PPTOS 0.5 0.05 25 0.05 10 0.024 10 0.01 0.5 0.0 PPTOS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PPTOS 0.3 0.0 0.25 0.1 10 0.01 0.0 0	PFHxDA	0.5	0.1		0.1	1	0.1		0.01	0.5	0.5
PFPES 0.5 0.05 25 0.1 10 0.048 10 0.01 0.5 0.0 PFHs5 0.5 0.05 0.1 25 0.05 10 0.01 10 0.01 0.5 0.0 PFN5 0.5 0.05 25 0.05 10 0.01 10 0.01 0.5 0.0 PFD5 0.5 0.05 25 0.1 10 0.01 0.0<					0.1			1	0.01		0.5
PFHs 0.5 0.05 25 0.05 10 0.01 10 0.01 0.5 0.0 PFHpS 0.5 0.05 25 0.05 10 0.01 10 0.01 0.5 0.0 PFNS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFNS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFDS 0.5 0.5 0.5 10 0.01 0.0 </td <td></td> <td>0.05</td>											0.05
PFHpS 0.5 0.1 25 0.05 10 0.024 10 0.01 0.5 0.0 PFOS 0.5 0.05 25 0.1 10 0.01 10 0.01 0.5 0.0 PFVS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFUDS 0 0 0 25 0.1 10 0.01 0.0 0											0.5
PPOS 0.5 0.05 25 0.05 10 0.01 10 0.01 0.5 0.05 PFNS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFDS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFDDS 0.1 0.1 10 0.05 10 0.01 0 0 PFDOS 0.1 0.1 0.5 0.05 0.05 0.01 0.5 0.05 0.01 0 0 PFDOS 0.1 0.5 0.5 0.5 0.01 0.5 0.00 0											
PPNS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0.0 PFDS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0. PFDOS 0.1 0.1 25 0.1 10 0.05 10 0.01 0 PFDOS 0.1 0.0	· · · · · · · · · · · · · · · · · · ·										0.05
PFDS 0.5 0.05 25 0.1 10 0.024 10 0.01 0.5 0. PFUnDS 0.1 0.1 25 0.1 10 0.01 0.0 <td></td> <td>0.05</td>											0.05
PFDOS 0.1 0.1 25 0.1 10 0.05 10 0.01 0.1 PFTrDS 0 0 0.1 0.1 0.1 0.1 0.1 0.01 0.1 PFPO-TA 0 0 0.5 0.5 0.55 0.01 0.5 0.01 0.5 DONA, ADDNA 0.5 0.5 0.5 0.55 0.5 0.01 0.5 0.01 DONA, ADDNA 0.5 0.5 0.5 0.05 0.5 0.01 0.5 0.01 MeGSA, N-	PFDS	0.5	0.05	25	0.1	10	0.024	10	0.01	0.5	0.1
PFrDS 0 0.1 <td>PFUnDS</td> <td>0</td> <td>0</td> <td>25</td> <td>0.1</td> <td>10</td> <td>0.1</td> <td>10</td> <td>0.01</td> <td>0</td> <td>0</td>	PFUnDS	0	0	25	0.1	10	0.1	10	0.01	0	0
HFPO-DA (Gen X) 1 0.5 1 0.5 0.5 0.005 0.5 0.01 0.5 0.0 DONA, ADDNA 0.5 0.5 0.5 0.5 0.005 0.5 0.01 0 DONA, ADDNA 0.5 0.05 0.5 0.005 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.5 0.02 0 0.01 0.05 0.5 0.02 0 0 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.05 0.02 0.01 0.01 0.01 0.05 0.01 0.01 0.01											0
HFPO-TA 0 0.5 0.5 0.5 0.1 0.5 0.01 0 DONA, ADONA 0.5 0.05 0.5 0.05 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.5 0.01 0.01 0.05 0.05 0.05 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 0.05 0.05 0.02 0.0 0 0 0 0 0 0 0 0.0 0											0
DONA: ADONA 0.5 0.5 0.5 0.00 0.5 0.01 0.5 0.0 FOSA; POSA 0.1 0.05 10 0.1 1 0.003 1 0.01 0.5 0.0 MeFOSA; N 0.5 0.5 10 0.1 0.5 0.1 1 0.01 0.5 0.05 EEFOSA; NEFFOSA 1 0.5 10 0.1 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 0.1 0.1 0.1 0.01 0.01 0											0.5
FOSA; PFOSA 0.1 0.05 10 0.1 1 0.003 1 0.01 0.5 0. MEFOSA; N- MEFOSA 0.5 0.5 10 0.1 0.5 0.1 1 0.01 0.5 0.0 FBSA 0 0.5 0.5 0.5 0.05 0.5 0.2 0 FHSA 0 0 0.5 0.5 0.025 0.2 0 MEFOSE 1											0.5
MeFOSA 0.5 0.5 10 0.1 0.5 0.1 1 0.01 0.5 0.0 EtCOSA, N-EtFOSA 1 0.5 10 0.1 1 0.1 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 0											0.5
EtFOSA 1 0.5 10 0.1 1 0.1 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 1 0.01 <	MeFOSA; N-										
FBSA 0 0 0.5 0.5 0.05 0.05 0.2 0 FHXSA 0 0 0.5 0.5 0.025 0.5 0.2 0 MEFOSE 0 0 0 0.1 0.1 0.01 0.01 0 EFOSE 1 <						0.5					0.5
FHXSA 0 0 0.5 0.5 0.25 0.5 0.2 0 MEFOSE 0 0 0 0.1 0.1 0.01 0.01 0 ELFOSE 1						1					0.5
MeFOSE 0 0 0 0.1 0.1 0.01 0.01 0 EFFOSE 1											0
EtFOSE 1 <td></td> <td>0</td>											0
NMEFOSAA; MeFOSAA 1 0.05 10 0.1 1 0.05 1 0.01 0.5 0. NEtFOSAA 1 0.05 10 0.1 1 0.05 1 0.01 0.5 0. PETOSAS, SETOSA 1 0.05 1 0.05 1 0.01 0.5 0. PF3ONS 1 0.05 1 0.5 1 0.002 1 0.01 0.5 0. 8:2 CLPFESA; 9CL- PF3OUdS 1 0.5 1 0.5 1 0.002 1 0.01 0.5 0. 8:2 CLPFESA; 11CL- PF3OUdS 1 0.5 1 0.01 0.5 0.01 0.5 0. 6:2 FTSA; 6:2 FTS 1 0.05 10 0.1 2.5 0.01 0.5 0. 10:2 FTSA 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 <td></td> <td>1</td>											1
MeFOSAA 1 0.05 10 0.1 1 0.05 1 0.01 0.5 0. NEtFOSAA; EtFOSAA 1 0.05 10 0.1 1 0.05 1 0.01 0.5 0. 6:2 CLPFESA; 9CI- PF3ONS 1 0.05 1 0.5 1 0.002 1 0.01 0.5 0. 8:2 CLPFESA; 11CI- PF3OUdS 1 0.5 1 0.05 1 0.008 1 0.01 0.5 0. 6:2 FTSA; 4:2 FTS 1 0.05 10 0.1 2.5 0.046 2.5 0.01 0.5 0. 6:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0. 0. 0. 0. 0. 0. 0. 0. 0. 0. 0. 0. 0.	FOSAA	0	0	0	0	0	0	0	0	0	0
NEtFOSAA; EtFOSAA 1 0.05 10 0.1 1 0.05 1 0.01 0.5 0. PF30NS 1 0.05 1 0.5 1 0.002 1 0.01 0.5 0. 8:2 CL-PFESA; 11CL- PF30UdS 1 0.5 1 0.55 1 0.008 1 0.01 0.5 0. 4:2 FTSA; 4:2 FTS 1 0.05 10 0.1 2.5 0.046 2.5 0.01 0.5 0. 6:2 FTSA; 6:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA; 8:2 FTS 1 0.05 0 <td< td=""><td></td><td>_</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>		_									
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PF3OUdS 1 0.5 1 0.008 1 0.01 0.5 0. 4:2 FTSA; 4:2 FTS 1 0.05 10 0.1 2.5 0.046 2.5 0.01 0.5 0. 6:2 FTSA; 6:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 8:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA; 8:2 FTS 0 0 0 0 0 0 0.		1	0.05	1	0.5	1	0.002	1	0.01	0.5	0.5
4:2 FTSA; 4:2 FTS 1 0.05 10 0.1 2.5 0.046 2.5 0.01 0.5 0.0 6:2 FTSA; 6:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0.0 8:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0.0 10:2 FTSA 0 0 0 0 0.1 0.1 0.1 0.01 0.01 0.5 0.0 10:2 FTSA 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0.0 0 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>											
6:2 FTSA; 6:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 8:2 FTSA; 8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA 0 0 0 0 0.1 0.1 0.01											0.5
8:2 FTS 1 0.05 10 0.1 2.5 0.05 2.5 0.01 0.5 0. 10:2 FTSA 0 0 0 0 0.1 0.1 0.01 0.01 0 FBET 0											0.5
10:2 FTSA 0 0 0 0.1 0.1 0.01 0.01 0 FBET 0											0.5
FBET 0											0.5
5:2sFTOH 0<											0
7:2sFTOH 0<					-						0
FOET 0 0 50 50 0 0 1 1 0 FDET 0 0 25 25 0 0 1 1 0 6:2 FTA, FHEA 0 0 0 0 0 0 0 0 0 8:2 FTA, FOEA 0	FHET	0	0	50	50	0	0	1	1	0	0
FDET 0 0 25 25 0 0 1 1 0 6:2 FTA, FHEA 0	7:2sFTOH	0	0	0	0	0	0	0	0	0	0
6:2 FTA, FHEA 0 <											0
8:2 FTA, FOEA 0 <											0
10:2 FTA /FDEA 0											0
FPrPA 0 <td></td> <td>0</td>											0
5:3 FTCA 0 0 10 10 1 0.1 1 0.01 0 5:3 FTCA / FPePA 0.5 0.5 0.5 0											0
5:3 FTCA / FPePA 0.5 0.5 0.5 0					-						0
6:2 FTUCA / FHUEA 0.1 0.1 0.1 0.1 0 <td>5:3 FTCA / FPePA</td> <td>0.5</td> <td>0.5</td> <td>0.5</td> <td>0.5</td> <td>0</td> <td></td> <td>0</td> <td>0</td> <td>0</td> <td>0</td>	5:3 FTCA / FPePA	0.5	0.5	0.5	0.5	0		0	0	0	0
8:2 FTUCA / FOUEA 0.1 0.1 0.1 0											0
8:2 FTUA, FOUEA 0 0 0 0 0 0 0 0 0 0 0 0 0											0
	-										0
10:2 FTUCA / FDUEA 0.1 0.1 0.1 0.1 0 0 0 0 0 0											0

	Suspended Particulate Matter (ng/g)		Biosolids Sludge	/ Sewage (ng/g)		ment ;/g)	So (ng			t/ mulch ;/g)
	max value	min value	max value	min value	max value	min value	max value	min value	max value	min value
6:2FTAcr	0	0	50	50	0	0	1	1	0	0
8:2FTAcr	0	0	50	50	0	0	1	1	0	0
10:2FTAcr	0	0	0	0	0	0	0	0	0	0
8:2FTOAc	0	0	0	0	0	0	0	0	0	0
10:2FTOAc	0	0	0	0	0	0	0	0	0	0
6:2 FTM	0	0	50	50	0	0	2	2	0	0
8:2 FTM	0	0	50	50	0	0	2	2	0	0
4:2 FTI	0	0	10	10	0	0	1	1	0	0
6:2 FTI	0	0	10	10	0	0	1	1	0	0
TFA, TFAA PFPrA	0	0	0	0	0	0	0	0	0	0
TFMS	0	0	0	0	0	0	0	0	0	0
PFEtS	0	0	0.1	0.1	0	0	0	0	0	0
PFEtS	0	0	0.1	0.1	0	0	0		0	0
HFC-23	0	0	0.1	0.1	0	0	0	0	0	0
HFC-23	0	0	0	0	0	0	0	0	0	0
HFC-32 HFC-43-10mee	0	0	0	0	0	0	0	0	0	0
HFC-43-10mee HFC-125	0	0	0	0	0	0	0	0	0	0
HFC-125	0	0	0	0	0	0	0	0	0	0
HFC-134a HFC-143a	0	0	0	0	0	0	0	0	0	0
HFC-143a	0	0	0	0	0	0	0	0	0	0
HFC-152a	0	0	0	0	0	0	0	0	0	0
HFC-227ea	0	0	0	0	0	0	0	0	0	0
HFC-245fa	0	0	0	0	0	0	0	0	0	0
HFC-365mfc	0	0	0	0	0	0	0	0	0	0
HCFC-124	0	0	0	0	0	0	0	0	0	0
HCFC-141b	0	0	0	0	0	0	0	0	0	0
HCFC-225ca/cb	0	0	0	0	0	0	0	0	0	0
HFO-1224yd	0	0	0	0	0	0	0	0	0	0
HFO-1233zd(E)	0	0	0	0	0	0	0	0	0	0
HFO-1234yf	0	0	0	0	0	0	0	0	0	0
HFO-1234ze(E)	0	0	0	0	0	0	0	0	0	0
HFO-1336mzz(E)	0	0	0	0	0	0	0	0	0	0
HFO–1336mzz(Z)	0	0	0	0	0	0	0	0	0	0
Halotron BrX, 2-BTP	0	0	0	0	0	0	0	0	0	0
MPHE, Sion	0	0	0	0	0	0	0	0	0	0
NOVEC 612	0	0	0	0	0	0	0	0	0	0
NOVEC 4710	0	0	0	0	0	0	0	0	0	0
HFE-347pc-f2	0	0	0	0	0	0	0	0	0	0
HFE-449s1 (7100)	0	0	0	0	0	0	0	0	0	0
HFE-569sf2 (7200)	0	0	0	0		0	0		-	0
HFE-7500	0	0	0	0	0	0	0	0	0	0
5:3FTB									0	0
	1	1	1	1	1	1	1	1		0
5:1:2FTB	1	1	1	1	1	1	1	1	0	0
Capstone 1183 A	0	0	0	0	0	0	0	0	0	0
Capstone 1157 B	0	0	0	0	0	0	0	0	0	0
PFHxPA	0	0	0	0	0	0	0	0	0	0
CI-PFHxPA	0	0	0	0	0	0	0	0	0	0
PFOPA	0	0	0	0	0	0	0	0	0	0
PFDPA	0	0	0	0	0	0	0	0	0	0
6:6 PFPiA	0	0	0	0	0	0	0	0	0	0
6:8 PFPiA	0	0	0	0	0	0	0	0	0	0
8:8PFPi	0	0	0	0	0	0	0	0	0	0
6:2PAP	0	0	0	0	0	0	0	0	0	0
6:2 diPAP	0.5	0.1	0.5	0.1	0.5	0.5	0.5	0.5	0.5	0.5
8:2PAP	0	0	0	0	0	0	0	0	0	0
8:2diPAP	0	0	0	0	0	0	0	0	0	0
EEA-NH4	0	0	0	0	0	0	0	0	0	0
PFBE	0	0	0	0	0	0	0	0	0	0
Perfluamine	0	0	0	0	0	0	0	0	0	0
6:2 FTAB	0	0	0	0	2.5	2.5	2.5	2.5	0	0
FTSAAm	0	0	0	0	2.5	2.5	2.5	2.5	0	0
PFECHS	1	0.1	1	0.1	1	0.048	1	0.15	0.5	0.5
C6O4	0	0	0	0	0	0	0	0	0	0

	Suspended Particulate Matter (ng/g)			/ Sewage (ng/g)		ment ;/g)	So (ng			t/ mulch g/g)			
	max value	min value	max value	min value	max value	min value	max value	min value	max value	min value			
PFE*	0	0	0	0	0	0	0	0	0	0			
PFMPA / PF4OPeA	0	0	0	0	0	0	0	0	0	0			
PFEESA	0	0	0	0	0	0	0	0	0	0			
3,6-OPFHpA	0	0	0	0	0	0	0	0	0	0			

Table A3.4: Maximum (max) and minimum LOD reported for PFAS individual compounds in biotic and air matrices.

m	Biota (aı (ng/ nax alue		Biota ((ng		(ทรู	\ir 7/1)	Flue	e gas		r dust	Blood	
va PFBA; HFBA		min	100		118	5/ -/				g/g)		:/g)
		value	max value	min value	max value	min value	max value	min value	max value	min value	max value	min value
ΡΕΡΔ ΡΕΡοΔ	200	0.08	1	0.4	0	0	0	0	1.97	0.1	0	0
	100	0.04	0.7	0.3	0	0	0	0	0.36	0.1	0.26	0.26
PFHxA	60	0.004	1	0.01	0.0000004	0.0000004	0	0	3.21	0.1	0.22	0.22
PFHpA PFOA	200	0.08	1	0.4	0	0	0	0	1.97 7	0.1	0	0 26
PFOA	50 50	0.01	1	0.01	0.0000002	0.0000002	0	0	0.96	0.1	0.36 0.38	0.36
PFDA	50	0.01	1	0.01	0.00000002	0.00000002	0	0	1.85	0.1	0.38	0.38
PFUnA; PFUdA	50	0.01	0.7	0.01	0.00000002	0.00000002	0	0	0.42	0.1	0.05	0.20
PFDoA	50	0.02	0.7	0.01	0.00000004	0.00000004	0	0	0.27	0.1	0.46	0.46
PFTrDA; PFTriA	50	0.04	0.5	0.01	0.00000004	0.0000002	0	0	0.02	0.02	0.34	0.34
PFTeA; PFTreA;												
PFTeDA	50	0.03	0.5	0.01	0.0000004	0.00000004	0	0	0.02	0.02	0.18	0.18
PFHxDA	0.7	0.05	0.5	0.01	0.00000004	0.0000004	0	0	0.1	0.03	0	0
PFODA PFBS	0.7	0.05	0.5	0.01	0.00000004	0.00000004	0	0	0.05	0.05	0	0
PFBS	100 0.5	0.01	1 0.5	0.01	0.0000002	0.0000002	0	0	0.39 0.1	0.1	0.06	0.06
PFHxS	80	0.04	1	0.01	0.00000002	0.00000002	0	0	0.1	0.09	0.06	0.06
PFHpS	50	0.02	0.5	0.01	0.00000002	0.00000002	0	0	0.1	0.05	0.00	0.04
PFOS	50	0.01	1	0.01	0.00000002	0.00000002	0	0	0.68	0.1	0.09	0.09
PFNS	0.59	0.024	0.5	0.01	0.00000002	0.00000002	0	0	0.96	0.1	0	0
PFDS	30	0.01	0.5	0.01	0.00000004	0.0000004	0	0	0	0	0.24	0.24
PFUnDS	0.2	0.05	0.01	0.01	0.00000004	0.0000004	0	0	0.01	0.01	0	0
PFDoS	0.2	0.04	0.01	0.01	0.00000004	0.0000004	0	0	0	0	0	0
PFTrDS	0.05	0.05	0.01	0.01	0.00000004	0.0000004	0	0	0	0	0	0
HFPO-DA (Gen X)	7.1	0.05	3.5	0.01	0.00000004	0.00000004	0	0	0.25	0.25	0	0
HFPO-TA	0.05	0.05	0.01	0.01		0.00000004	0	0	0	0	0	0
DONA; ADONA	0.7	0.048	0.5	0.01	0.0000004	0.0000004	0	0	0.1	0.1	0	0
FOSA; PFOSA MeFOSA; N-	100	0.025	1.9	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0.08	0.08
MeFOSA	1.25	0.05	0.5	0.01	0.00000004	0.00000004	0	0	1.5	0.1	0.14	0.14
EtFOSA; N-EtFOSA	1.25	0.05	0.5	0.01	0.00000004	0.00000004	0	0	0.3	0.1	0.26	0.26
FBSA	0.05	0.05	0	0	0	0	0	0	0	0	0	0
FHxSA	0.025	0.025	0	0	0	0	0	0	0	0	0	0
MeFOSE	1.25	0.05	0.01	0.01	0.00000004	0.0000004	0	0	3.4	3.4	0	0
EtFOSE	1.25	1.25	0	0	0	0	0	0	1	1	0	0
FOSAA	0	0	0	0	0	0	0	0	0	0	0	0
NMeFOSAA; MeFOSAA	1	0.04	1	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0.3	0.3
NEtFOSAA;		0.04	I	0.01	0.00000004	0.0000004	0	0	0.1	0.1	0.5	0.5
EtFOSAA	1	0.04	1	0.01	0.00000004	0.0000004	0	0	0.1	0.1	0.3	0.3
6:2 CI-PFESA; 9CI-												
PF3ONS	20	0.04	1	0.01	0.0000004	0.0000004	0	0	0.1	0.1	0	0
8:2 CI-PFESA;		0.040		0.04	0.00000004	0.00000004	0	0	0.4	0.4	0	0
11Cl-PF3OUdS	1	0.048	1	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0	0
4:2 FTSA; 4:2 FTS 6:2 FTSA; 6:2 FTS	1 200	0.02	1	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0	0
8:2 FTSA; 8:2 FTS 8:2 FTSA; 8:2 FTS	50	0.02	1	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0	0
10:2 FTSA	200	0.02	0.01	0.01	0.00000004	0.00000004	0	0	0.1	0.1	0	0
FBET	1.25	1.25	0.01	0.01	0.00000004	0.00000004	0	0	0	0	0	0
5:2sFTOH	0	0	0	0	0	0	0	0	0	0	0	0
FHET	1.25	1.25	0	0	0	0	0	0	0	0	0	0
7:2sFTOH	1.25	1.25	0	0	0	0	0	0	0	0	0	0
FOET	1.25	1.25	0	0	0	0	0	0	0	0	0	0
FDET	1.25	1.25	0	0	0	0	0	0	0	0	0	0
6:2 FTA, FHEA	0.17	0.1	2.4	2.4	0	0	0	0	0	0	0	0
8:2 FTA, FOEA	0.5	0.17	0.5	0.5	0	0	0	0	0	0	0	0
10:2 FTA /FDEA FPrPA	0 26	0.26	0	0	0	0	0	0	0	0	0	0
5:3 FTCA	0.26	0.26	0 1	0.01	0.00000004	0.00000004	0	0	0	0	0 0	0
5:3 FTCA / FPePA	0.26	0.05	0	0.01	0.00000004	0.00000004	0	0	0.08	0.08	0	0
7:3 FTCA / FHpPA	0.26	0.04	0	0	0	0	0	0	0.08	0.08	0	0
6:2 FTUCA / FHUEA	0.20	0.04	0.5	0.5	0	0	0	0	0.03	0.03	0	0
8:2 FTUCA / FOUEA	0.04	0.04	0.5	0.5	0	0	0	0	2.2	2.2	0	0

	Biota (a	nimals) z/g)	Biota (plants) ;/g)		.ir g/L)	Flue	e gas		r dust g/g)		serum g/g)
	max	min	max	min			max	min	max	min	max	min
	value	value	value	value	max value	min value	value	value	value	value	value	value
8:2 FTUA, FOUEA	0.6	0.26	0.6	0.6	0	0	0	0	0	0	0	0
10:2 FTUCA /					_			_				
FDUEA	0.26	0.04	0	0	0	0	0	0	0.1	0.1	0	0
6:2FTAcr	0	0	0	0	0	0	0	0	0	0	0	0
8:2FTAcr	0	0	0	0	0	0	0	0	0	0	0	0
10:2FTAcr 8:2FTOAc	0	0	0	0	0	0	0	0	0	0	0	0
10:2FTOAC	0	0	0	0	0	0	0	0	0	0	0	0
6:2 FTM	0	0	0	0	0	0	0	0	0	0	0	0
8:2 FTM	0	0	0	0	0	0	0	0	0	0	0	0
4:2 FTI	0	0	0	0	0	0	0	0	0	0	0	0
6:2 FTI	0	0	0	0	0	0	0	0	0	0	0	0
TFA, TFAA	0	0	0	0	0	0	0	0	0	0	0	0
PFPrA	0	0	0	0	0	0	0	0	0	0	0	0
TFMS	0	0	0	0	0	0	0	0	0	0	0	0
PFEtS	0	0	0	0	0	0	0	0	0	0	0	0
PFPrS	0	0	0	0	0	0	0	0	0	0	0	0
HFC-23	0	0	0	0	0	0	0	0	0	0	0	0
HFC-32	0	0	0	0	0	0	0	0	0	0	0	0
HFC-43-10mee	0	0	0	0	0	0	0	0	0	0	0	0
HFC-125	0	0	0	0	0	0	0	0	0	0	0	0
HFC-134a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-143a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-152a	0	0	0	0	0	0	0	0	0	0	0	0
HFC-227ea	0	0	0	0	0	0	0	0	0	0	0	0
HFC-236fa	0	0	0	0	0	0	0	0	0	0	0	0
HFC-245fa	0	0	0	0	0	0	0	0	0	0	0	0
HFC-365mfc	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-124	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-141b	0	0	0	0	0	0	0	0	0	0	0	0
HCFC-225ca/cb	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1224yd HFO-1233zd(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-123320(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1234ze(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO-1336mzz(E)	0	0	0	0	0	0	0	0	0	0	0	0
HFO–1336mzz(Z)	0	0	0	0	0	0	0	0	0	0	0	0
Halotron BrX, 2-					•							
BTP	0	0	0	0	0	0	0	0	0	0	0	0
MPHE, Sion	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 612	0	0	0	0	0	0	0	0	0	0	0	0
NOVEC 4710	0	0	0	0	0	0	0	0	0	0	0	0
HFE-347pc-f2	0	0	0	0	0	0	0	0	0	0	0	0
HFE-449s1 (7100)	0	0	0	0	0	0	0	0	0	0	0	0
HFE-569sf2												
(7200)	0	0	0	0	0	0	0	0	0	0	0	0
HFE-7500	0	0	0	0	0	0	0	0	0	0	0	0
5:3FTB	1	1	1	1	0	0	0	0	0	0	0	0
5:1:2FTB	1	1	1	1	0	0	0	0	0	0	0	0
Capstone 1183 A	0	0	0	0	0	0	0	0	0	0	0	0
Capstone 1157 B	0	0	0	0	0	0	0	0	0	0	0	0
PFHxPA	0.26	0.26	0	0	0	0	0	0	0	0	0	0
CI-PFHxPA	1.17	1.17	0	0	0	0	0	0	0	0	0	0
PFOPA	0.26	0.26	0	0	0	0	0	0	0	0	0	0
PFDPA	0.26	0.26	0	0	0	0	0	0	0	0	0	0
6:6 PFPiA	0.13	0.13	0	0	0	0	0	0	0	0	0	0
6:8 PFPiA	0.1	0.1	0	0	0	0	0	0	0	0	0	0
8:8PFPi	0.1	0.1	0	0	0	0	0	0	0	0	0	0
6:2PAP	0.17	0.17	0	0	0	0	0	0	0	0	0	0
6:2 diPAP	0.5	0.04	1	0.5	0	0	0	0	0.1	0.1	0	0
8:2PAP	0.17	0.17	0	0	0	0	0	0	0	0	0	0
8:2diPAP	0.17	0.17	0	0	0	0	0	0	0	0	0	0
EEA-NH4	0	0	0	0	0	0	0	0	0	0	0	0
PFBE	0	0	0	0	0	0	0	0	0	0	0	0
Perfluamine	0	0	0	0	0	0	0	0	0	0	0	0

		Biota (animals) (ng/g)		plants) ;/g)		vir g/L)	Flue	e gas	Indoo (nទួ	r dust g/g)	Blood (ng	serum :/g)
	max value	min value	max value	min value	max value	min value	max value	min value	max value	min value	max value	min value
6:2 FTAB	0	0	0	0	0	0	0	0	0	0	0	0
FTSAAm	0	0	0	0	0	0	0	0	0	0	0	0
PFECHS	20	0.04	1	0.5	0	0	0	0	0.1	0.1	0	0
C6O4	0	0	0	0	0	0	0	0	0	0	0	0
PFE*	0	0	0	0	0	0	0	0	0	0	0	0
PFMPA /												
PF4OPeA	0	0	0	0	0	0	0	0	0	0	0	0
PFEESA	0	0	0	0	0	0	0	0	0	0	0	0
3,6-OPFHpA	0	0	0	0	0	0	0	0	0	0	0	0

Appendix 4: Table of the number of respondents undertaking sample preparation steps for each matrix

Sample Type	stor free	imple age e.g. ezing or ervative	(inc	ation lude ter erial)	treat (e siev fre dry	re- ment .g. ring, eze ing, ding)	solve	action ent or pent		action ditions	stora _{ freez	ract ge e.g. ing or vative	Cle	an-up	con	Pre- centration
	Yes	No	Yes	No	Yes	No	Yes	No	Yes	No	Yes	No	Yes	No	Yes	No
Air	4	0	1	2	1	2	3	0	3	0	3	1	3	1	3	0
Biosolids/ Sewage Sludge	12	2	2	9	10	2	12	0	10	0	9	3	9	2	8	4
Biota- animals	25	1	2	15	24	1	25	0	21	1	17	8	20	5	14	8
Biota- plants	9	0	1	5	8	1	9	0	8	0	6	2	9	0	6	2
Compost/mul ch	1	2	0	3	1	2	2	1	1	1	1	2	1	2	0	3
Drinking water	27	6	9	24	4	26	20	13	18	11	15	16	12	18	19	12
Flue gas	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0
Food- dairy	4	0	1	2	2	1	4	0	3	0	4	0	4	0	3	1
Food- fish	9	1	1	6	8	1	10	0	7	1	6	4	10	0	7	3
Food- fruit and vegetables	5	0	1	3	3	1	4	0	4	0	4	0	4	0	3	1
Food- meat	6	1	2	3	5	1	7	0	4	0	5	2	7	0	4	2
Groundwater	25	4	9	19	3	20	16	11	16	7	14	14	8	15	19	8
Indoor dust	4	1	2	2	4	1	5	0	3	0	4	1	3	1	4	0
Leachate	8	1	6	3	2	6	9	0	5	0	6	3	4	4	7	2
Other (Please specify)	1	1	0	3	3	0	3	1	3	1	0	3	2	1	0	3
Other (SERUM)	1	0	1	0	0	1	1	0	0	1	0	1	0	1	1	0
Sediment	20	2	2	16	21	1	22	0	19	0	16	6	19	3	13	6
Soil	16	2	2	13	17	1	18	0	16	0	12	6	14	3	10	6
Surface (fresh) water	32	8	18	22	6	27	26	12	24	9	21	16	14	21	29	10
Surface (saline) waters	14	2	5	11	4	10	12	4	11	4	9	6	7	8	11	5
Suspended Particulate Matter	5	3	1	4	4	3	6	2	5	2	5	3	3	4	3	4
Wastewater Treatment Works effluent	20	2	11	11	6	13	16	5	15	3	9	11	8	10	14	7

Appendix 5: Full detail of sample preparations utilised per matrix

LIQUID MATRICES

Drinking Water

Sample storage	Filtration (include filter material)	Pre- treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Cooler Sodium thiosulfate 50% methanol 8°C maximum, 14 days maximum If possible processed the same day, sometimes stored at 4°C Samples stored in the dark at 4°C until analysis 4°C - up to four days 4°C for up to a week Fridge at 4°C, processed within four weeks after sampling Fridge at 5°C Freezing -20°C storage 	 SPE serves as a filtration step WAX SPE Sandfilter Glass fibre, 0.7 µm glass filters, Whatman Waters Buchner filtration with filtering paper Decant 	 Centrifuge Formic acid (98%); 50 μL in 5 mL pH adjustment to 3 	 SPE clean up or ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) on-line SPE Oasis WAX + 5 mL 0.25% ammonium hydroxide in methanol SPE Phenomenex Strata-X AW/ Macherey-Nagel Chromabond LV-HR-XAW + pH 6-8 SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1 L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6 mL methanol and 6 mL 0.1% ammonium hydroxide in methanol Oasis WAX. Conditioning: 0.1% ammonium hydroxide in methanol 80:20 (v/v); elution: methanol, 0.1% ammonium hydroxide in methanol SPE: Conditioning: 4ml 0.1% ammonia in methanol, 4ml methanol, 4ml water Washing: 4ml 25mmol/L acetate buffer, 4ml water, Elution: 4ml methanol, 4ml 0.1% ammonia in methanol. SPE with WAX Oasis 150mg cartridges SPE –DVB + methanol elute Strata X-AW® 200 mg/6 mL; Phenomenex, France; SPE – WAX: 1 mL of methanol, 4 mL of methanol with 0.1% vol ammonium hydroxide 	 Fridge 4°C Fridge at 5°C Freezing of concentrated sample extract (-20°C) 	 SPE (XAW) SmL 25mM ammonium acetate Filtration Solvent match Oasis WAX, 500 mg SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6mL methanol and 6mL 0.1% ammonium hydroxide in methanol 	 SPE Exti on- Eva Fina Dry The nitr in 100 into 500

Pre-concentration

PE

- xtraction volume: 5 mL
- n-line SPE
- vaporation to 200 µL
- inal extract nitrogen blowdown to 150 µL
- Drying: nitrogen gas, 35°C, Final volume: 500μL
- he extract was evaporated to dryness under nitrogen at 45°C and the residue reconstituted n
- $.00\mu L$ of 50:50 water/methanol; 0.5L water nto 0.5 mL
- 00 times

Groundwater

Sample storage	Filtration (include filter material)	Pre- treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Analysed as soon as possible or frozen Sodium thiosulfate 8°C maximum, 14 day maximum Samples stored in the dark at 4°C until analysis; 4°C - up to 4 days 4°C for up to 30 days Fridge at 5°C Freezing -20°C storage 	 SPE serves as a filtration step We settle and centrifuge to remove suspended but we do not filter Sandfilter Decant Glass fibre (0.7 µm) 0.45 µm 	• Centrifuge	 SPE clean-up or ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) on-line SPE WAX with neutral fraction and basic fraction. Usually we do not pre-treat the sample but if the sample has pH > 7, we will acidify with formic acid OASIS Wax or Chromabond. Washing cartridges with water, acetonitrile: acetone: formic acid 1:1:0.02, methanol; extraction with methanol + 0.1%ammonia OASIS Wax + 5 mL 0.25% ammonium hydroxide in methanol SPE WAX + pH 4 Phenomenex Strata-X AW/ Macherey-Nagel Chromabond LV-HR-XAW + pH 6 8 SPE - DVB + methanol elute Strata X-AW® 200 mg/ 6mL; Phenomenex, France: 1 mL of methanol, 4 mL of methanol with 0.1% vol ammonium hydroxide and then 2 mL of 70:30 dichloromethane/2-propanol with 0.1% vol ammonium hydroxide SPE WAX + ISO method 	 Fridge Freezing -20°C storage 	 Wash step SPE SPE (XAW) 5 mL 25 mM ammonium acetate Solvent match 	 SPE Ext Eva on- The nitrin 1 0.5

Pre-concentration

PE

xtraction volume: 5 mL

vaporation with nitrogen

n-line

he extract was evaporated to dryness under hitrogen at 45°C and the residue reconstituted n 100μL of 50:50 water/ methanol

.5L water into 0.5 mL

Leachate

Sample storage	Filtration (include filter material)	Pre- treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 4°C 4°C for up to a week Freezing 	 SPE serves as a filtration step We settle and centrifuge to remove suspended but we do not filter Buchner filtration with filtering paper, Dilution of leachate at a ratio of 4:100ml water Decant 	• pH adjustment to 6.5	 SPE clean-up or ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) WAX with neutral fraction and basic fraction. Usually we do not pre-treat the sample but if the sample has pH > 7, we will acidify with formic acid SPE Conditioning: 4 mL 0.1% ammonia in methanol, 4 mL methanol, 4 mL water, washing; 4 mL water, Elution; basic solution (6 mL ethyl acetate/methanol (50/50 v/v) 2% ammonia(v/v)), acidic solution (4 mL ethyl acetate/methanol (50/50, v/v) 1.7 % FA (v/v). SPE with WAX Oasis 150mg and HLB Oasis 200mg cartridges SPE Oasis WAX 	 Fridge 4°C 4°c, away from light Fridge at 5°C Freezing -20°C storage 	• Wash step • Solvent match	• SPE • Dryi

Pre-concentration

PE

Drying: nitrogen gas, 35°C, Final volume: 500μL

Surface (fresh) water

Sample storage	Filtration (include filter material)	Pre- treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 If possible processed the same day, sometimes stored at 4°C 8°C maximum, 14 days maximum. Samples stored in the dark at 4°C until analysis 4°C for up to four days 4°C for up to a week Fridge at 4°C, processed within four weeks after sampling Fridge at 5°C Freezing -20°C storage 	y we centrifuge to remove suspended but we do not filter • Sandfilter	 Sieving and freeze drying Formic acid (98%); 50µL in 5 mL Centrifugat ion pH adjustment to 3 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) on-line SPE SPE pH4 + 0.1% ammonium hydroxide in methanol WAX with neutral fraction and basic fraction. Usually we do not pre-treat the sample but if the sample has pH > 7, we will acidify with formic acid OASIS Wax or Chromabond. Washing cartridges with water, acetonitrile:acetone:formic acid 1:1:0.02, methanol; extraction with methanol + 0.1%ammonia Oasis WAX.5 mL 0.25% ammonium hydroxide in methanol Phenomenex Strata-X AW/ Macherey-Nagel Chromabond LV-HR-XAW + pH 6-8 SPE – WAX + pH 4 SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1 L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6 mL methanol and 6mL 0.1% ammonium hydroxide in methanol, 0.1 % ammonium hydroxide in methanol Oasis WAX. S00 mg SPE – DVB. Methanol elute SPE with WAX Oasis 150mg cartridges. SPE: Conditioning: 4ml 0.1% ammonia in methanol, 4ml methanol, 4ml water 	 Fridge 4 °C 4°C, away from light Fridge at 5°C Freezing of concentrated sample extract (-20°C) 	 Wash step SPE (XAW) 5 mL 25 mM ammonium acetate solvent match Oasis WAX, 500 mg SPE SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6mL methanol and 6mL 0.1% ammonium hydroxide in methanol 	• TI ni 1(0.

Pre-concentration

Extraction volume: 5 mL

Evaporation with nitrogen

Only for PFOS

SPE

Evaporation to 200µL final extract

Nitrogen blowdown to 150 µL

100mL to 1mL

The extract was evaporated to dryness under nitrogen at 45°C and the residue reconstituted in 100 μL of 50:50 water methanol; 0.5L water into 0.5 mL

500 times

Washing: 4ml 25mmol/L acetate buffer, 4ml water, Elution: 4ml methanol, 4ml 0.1% ammonia in methanol.	
 Strata X-AW[®] 200 mg/6 mL; Phenomenex, France. 1 mL of methanol, 4 mL of methanol with 0.1% vol ammonium hydroxide and then 2 mL of 70:30 dichloromethane/2- propanol with 0.1% vol ammonium hydroxide; methanol 0.1% ammoniac 	
 Oasis WAX 6cc cartridge 150mg 30μm Waters. Vacuum induced extraction block SPE WAX - ISO method 	

Surface (saline) waters

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction solvent Extraction conditions	Extract storage	Clean-up	
 8°C maximum, 14 days maximum If possible processed the same day, sometimes stored at 4°C 4°C, processed within four weeks after sampling Freezing Analysed as soon as possible or frozen -20°C storage 	 as a filtration step Occasionally we centrifuge to remove suspended but we do not filter. Glass fibre, 0.7μm glass filters, Whatman 		addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent)	 Refrigeration Freezing of concentrated sample extract (- 20°C) 	 Wash step Solvent match Oasis WAX, 500 mg SPE (Oasis WAX, 200 mg, Waters. Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6mL methanol and 6mL 0.1% ammonium hydroxide in methanol 	• r • 8

Pre-concentration

SPE

- Extraction volume: 5mL
- Evaporation to 200µL
- Final extract nitrogen blowdown to 150µL
- nitrogen evaporation
- 800 times

Wastewater Treatment Works Effluent

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Analysed as soon as possible or frozen 8°C maximum, 14 day maximum 4°C 4°C for up to a week Samples stored in the dark at 4°C until analysis 4°C (maximum 30 days) Freezing -20°C storage 	 and centrifuge to remove suspended, but we do not filter. 0.22µm regenerated cellulose filter 	• Formic acid (98%); 50μL in 5mL	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tertbutyl ether (as extraction solvent) on-line SPE WAX with neutral fraction and basic fraction. Usually we do not pre-treat the sample but if the sample has pH > 7, we will acidify with formic acid Phenomenex Strata-X AW/ Macherey-Nagel Chromabond LV-HR-XAW + pH 6-8 SPE: Conditioning: 4ml 0.1% ammonia in methanol, 4ml methanol, 4ml water Washing: 4ml 25mmol/L acetate buffer, 4ml water, Elution: 4ml methanol, 4ml 0.1% ammonia in methanol. SPE with WAX Oasis 150mg cartridges SPE - DVB + methanol elute Oasis WAX 6cc cartridge 150mg 30µm Waters. Vacuum induced extraction block Strata X-AW® 200 mg/6 mL; Phenomenex, France. 1 mL of methanol, 4 mL of methanol with 0.1% vol ammonium hydroxide and then 2mL of 70:30 dichloromethane/2-propanol with 0.1% vol ammonium hydroxide WAX sorbent SPE oasis WAX 	 Fridge 8°C maximum, 14 day maximum Freezing -20°C storage 	• Wash step • Solvent match	

Pre-concentration

• Extraction volume: 5 mL

on-line

• Drying: nitrogen gas, 35°C, Final volume: 500µL

• 100mL to 1mL

 The extract was evaporated to dryness under nitrogen at 45°C and the residue reconstituted in 100µL of 50:50 water/ methanol

SOLID MATRICES

Biosolids/ Sewage Sludge

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Freezing, if analysis is not possible within one week 8°C maximum 4°C 4°C for up to a week Samples stored in the dark at 4°C until analysis 4°C (maximum 30 days) Freezing -20°C until freeze drying 		 Freeze drying and grinding Freeze drying or drying at 40°C, grinding (mortar) Grinding Drying < 40°C, 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tertbutyl ether (as extraction solvent) Acetonitrile + ultrasonication and orbital shaking Methanol + ultrasonic bath SPE WAX Methanol + vortex 30 min, centrifugation 20 min at 3000tr/min Methanol, Acetonitrile (1/1), with 0.05M sodium hydroxide + Solid/Liquid with agitation 	 Fridge 4°C 4°c, away from light Freezing -20°C storage 	 Dispersive carbon clean up SPE 50 mg Envicarb + 50 μL acetic acid SPE cartridge 	• E ¹

Pre-concentration

• Evaporation with nitrogen

1g of dried sample into 1 ml

Biota – animals

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Analysed as soon as possible or frozen 4°C < 0°C Samples are frozen upon receipt Freezing before grinding -20°C storage 	• Regenerated cellulose	 Samples can be analysed fresh (after careful homogenisation) as well as dry (after lyophilisation) Typically a wet extraction but we homogenise tissues Drying with water-free silica gel Freeze drying Freeze drying 24 h Grinding and freeze drying Freeze drying, grinding (ball grinder) Drying at 40°C, grinding sieving 80µm Chopping Homogenisation Whole fish samples are homogenised sodium hydroxide Digestion 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) 1.5 mL of water and acetonitrile solution (10:90 v/v) per gram of fresh sample; acidify with formic acid (98%) + sonication Acetonitrile + ultrasonication and orbital shaking Methanol + ultrasonication Hexane/dichloromethane 3/2 + ASE-extraction, evaporation, filtration (0.22 µm filter) SPE WAX Acetonitrile using AOAC QuEChERS technique extraction, samples shaken overnight 7 mL Acetonitrile:Chromatography grade water (9:1) methanol/potassium hydroxide (0.01 M of potassium hydroxide) + FUSLE assisted (pulsed mode for 2.5 min in duplicate, with a pulsed time on of 0.8 seconds and pulsed time off of 0.2 seconds and at 10% of irradiation power). Extractions were carried out at 0°C in an ice-water bath.) Methanol/potassium hydroxide (0.01 M of potassium hydroxide) and Shake/Sonicate/Centrifuge liquid solid extraction overnight ASE and 3 x 100mL methanol Methanol 66% - Water 33% + Vortex 30 min, centrifugation 20 minutes at 3000tr/min Different methods for different purposes – e.g. ion-pair extraction for fish livers 	 Immediately inject on the instrument after extraction Drying, cooling Organisation optimised to avoid storage Stored in fridge at 3±2°C Kept refrigerated fridge 4°C Fridge at 5°C Refrigerated. Long term: freezing. -20°C degrees storage 	 Freezing to remove fatty compounds and proteins 0.6 g magnesium sulfate and 0.2 g sodium chloride per gram of fresh sample + freezing one night + through HybridSPE®Phospholipid Ultra cartridge to remove phospholipids + on-line Turbulence Flow Chromatography purification Dispersive carbon clean up SPE (XAW) dSPE Extracts filtered through polypropylene (PP) filters evaporated to ~1mL. Then, diluted in 6mL water. Then SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6mL methanol and 6mL 0.1% ammonium hydroxide in methanol OASIS WAX SPE WAX + EnviCarb SPE - DVB, solvent match Graphetised carbon and acetic acid Activated charcoal, florisil, C18 and PSA sorbent 	2 v • C • E • T • 1

Pre-concentration

 Soft tissue of bivalves: 5–10g; yolk: 1-2g; fish fillet: 2-10g; fish viscera 2g; fish carcass 5g; extract volume 1mL

- on-line SPE
- Evaporation to 200µL final extract
- Evaporation with nitrogen
- Turbo Vap (nitrogen)
- 1g of dried sample into 3ml

Biota – plants

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Analysed as soon as possible or frozen Freezing 		 Samples can be analysed fresh (after carefully grinding) as well as dry (after lyophilisation or drying) Freeze drying 24hours 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) 1.5 mL of water and acetonitrile solution (10:90 v/v) per gram of fresh sample; acidify with formic acid (98%) + Sonication 5 mL of acetonitrile diluted in water (9:1 acetonitrile:Milli-Q water) and Sonication step in an ultrasonic bath for 20 minutes. Samples were centrifuged at 20 °C and 8000 rpm for 5 minutes to separate the matrix from the supernatant, which was filtered with 10mL syringes coupled with 0.22µm filters, and transferred to test tubes. This extraction cycle was repeated twice for each sample. ASE + 3 x 100mL methanol 	 Fridge Freezing 	 Freezing to remove fatty compounds and proteins 0.6g magnesium sulfate and 0.2g sodium chloride per gram of fresh sample + freezing one night + through HybridSPE®Phospholipid Ultra cartridge Extracts filtered through PP filters evaporated to ~1mL. Then, diluted in 6 mL water. Then SPE (Oasis WAX, 200 mg, Waters). Conditioning: 4 mL 0.1% ammonium hydroxide in methanol, 4 mL methanol and 4 mL chromatography water. Sample load 1L. Wash 4 mL chromatography water and dry with vacuum. When dry preserved frozen until elution. Elution conditioning: 4 mL water with ammonium acetate (25 Mm, pH = 4). Elution 6mL methanol and 6mL 0.1% ammonium hydroxide in methanol. Samples were then evaporated under a gentle flow of nitrogen at 45 °C until 1mL. A second clean-up procedure was applied to the samples in order to clarify them more and avoid a high presence of pigments. This clean-up consisted in introducing each 1mL sample inside 1.5mL Eppendorfs previously filled with 100 mg of ENVI-CarbTM carbon, by the means of Pasteur pipettes. Eppendorfs were vortex-mixed for 15 seconds and centrifuged for 10 minutes at 12000 rpm. The resulting supernatants were filtered by 1mL syringes coupled with 0.22µm filters and transferred to clean test tubes. SPE - DVB, solvent match 	

Pre-concentration

• 1-2g; extract volume 1 mL

• Evaporation to 200µL final extract

Compost/mulch

Sa	ample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
• F	Freezing		• Sieving	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) 			

Food - dairy

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up
 Freezing -20°C storage 			 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) Alkaline digestion, acetonitrile extraction 	-	 Freezing to remove fatty compounds and proteins ENVI-Carb

Food - fish

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
• < 0°C		• sodium	• Ion-pair extraction: pH buffering at pH 10, addition of	 Refrigerated. 	• Freezing to remove fatty	• r
 Freezing 		hydroxide	tetrabutylammonium hydrogensulfate and methyl	 -20°C storage 	compounds and proteins	
 -20°C storage 		Digestion	tert-butyl ether (as extraction solvent)		OASIS WAX	
			 Acetonitrile+ Shake/ Sonicate/ Centrifuge 		• SPE	
			• ASE + 3 x 100 mL methanol		 SPE - DVB, solvent match 	
			QuEChERS USFDA method		• SPE-WAX	
			 Alkaline digestion, acetonitrile extraction 		• ENVI-Carb	

Food - fruit and vegetables

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Freezing -20°C storage 			 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert- butyl ether (as extraction solvent) Alkaline digestion, acetonitrile extraction 	Refrigerated.-20°C storage	 Freezing to remove fatty compounds and proteins ENVI-Carb 	

Pre-concentration

Pre-concentration

Pre-concentration

nitrogen evaporation

Food - meat

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
• < 0°C			• Ion-pair extraction: pH buffering at pH 10, addition of	 Refrigerated. 	• Freezing to remove fatty	
• Freezing			tetrabutylammonium hydrogensulfate and methyl tert-	 -20°C storage 	compounds and proteins	
 -20°C storage 			butyl ether (as extraction solvent)		• WAX-SPE	
			• ASE + 3 x 100 mL methanol		• SPE - DVB, solvent match	
			 Alkaline digestion, acetonitrile extraction 		• ENVI-Carb	

Indoor dust

Filtration Sample storage (include filte material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up
 4°C (maximum 30 days) 	• Sieving	Alkaline digestion, methanol extraction	 -20°C storage 	 SPE Oasis WAX + HLB WAX SPE

Pre-concentration

Sediment

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Analysed as soon as possible or frozen Room temperature +8°C 8°C maximum, 14 day maximum Samples stored in the dark at 4°C until analysis 1-5°C Freezing < 0°C Freezing (-20°C) Freezing, if analysis is not possible within one week 	• Regenerated cellulose	 Sieving and freeze drying Freeze drying and grinding Freeze drying or drying at 40°C, grinding (mortar) Samples can be analysed fresh (after carefully homogenisation) as well as dry (after lyophilisation or drying) Homogenisation Sieving, Grinding, Drying Drying, sieving, grinding Grinding <250µm Drying < 40°C 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tertbutyl ether (as extraction solvent) 1.5 mL of water and acetonitrile solution (10:90 v/v) per gram of fresh sample; acidify with formic acid (98%) + sonication Acetonitrile and methanol + ultrasonication Methanol + ASE-extraction, evaporation, filtration (0.22 µm filter) Acetonitrile + ultrasonication and shaking Methanol + ultrasonication, 2x30 min, 40 °C Addition of 1% acetic acid in water, or respectively methanol/1% acetic acid in water 90:10 (v/v); vortexing, ultrasonication, centrifugation; 3x repeated; collection of supernatants ASE + 3 x 100 mL methanol 1g of sample was vortexed, then 10mL of a 1% acetic acid solution added. Each tube was then vortexed and sonicated at 60°C for 15 min. After centrifugation at 4000 rpm for 2 minutes, the supernatant was transferred to a second 50-mL PP tube. The original tube was then extracted with 2.5mL methanol: 1% acetic acid (90:10, v/v), and it was again vortexed and sonicated for 15 minutes at 60°C before being centrifuged. The supernatant was then decanted into the second tube. This process was repeated once more, and finally the original tube was washed for the last time with 10mL of a 1% acetic acid solution. All the supernatants of each sample were combined in the second tube, which was centrifuged in order to reduce SPE cartridge clogging during the clean-up step. The cartridge was rinsed with 5mL of water/ methanol (60:40, v/v) Methanol, Acetonitrile (1/1), with 0.05M sodium hydroxide + Solid/Liquid with agitation Alkaline digestion, methanol sonication 	 Immediately injection on the instrument after extraction drying, cooling 8°C max., 14 d max Fridge 4°C Fridge at 5°C Freezing Freezing of concentrated sample extract (- 20°C) 	 0.6g magnesium sulfate and 0.2g sodium chloride per gram of fresh sample and freezing one night and on-line Turbulence Flow Chromatography purification Dispersive carbon clean up SPE (XAW) SPE - DVB, solvent match Graphitized carbon and acetic acid Strata X-AW[®] 200 mg/6 mL; Phenomenex, France Activated charcoal SPE-WAX SPE anion exchange ENVI-Carb SPE cartridge 	• 0 • E

- 2-10g; extract volume 1mL
- on-line SPE
- Evaporation with nitrogen
- Nitrogen blowdown to 150 μL
- Turbo Vap (nitrogen)
- The extract was evaporated to dryness under nitrogen at 45°C and the residue reconstituted in 100 μL of 50:50 water/ methanol

Soil

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 Room temperature +8°C 8°C max., 14 day max. 4°C away from light Freezing <0°C Freezing, if analysis is not possible within one week 		 Sieving Freeze-drying-sieving-grinding Freeze drying or drying at 40°C, grinding (mortar) Homogenisation Sieving, Grinding, Drying Drying, sieving, grinding Grinding <250µm Drying < 40°C 	 Ion-pair extraction: pH buffering at pH 10, addition of tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent) Methanol + Ultrasound and shaking Methanol + 1 h, Ultrasound Methanol + Ultrasound, 2x30 min, 40 °C SPE WAX ASE + 3 x 100 mL methanol 1 g of sample was vortexed, then 10 mL of a 1% acetic acid solution added. Each tube was then vortexed and sonicated at 60°C for 15 min. After centrifugation at 4000 rpm for 2 min, the supernatant was transferred to a second 50-mL PP tube. The original tube was then extracted with 2.5 mL methanol: 1% acetic acid (90:10, v/v), and it was again vortexed and sonicated for 15 min at 60°C before being centrifuged. The supernatant was then decanted into the second tube. This process was repeated once more, and finally the original tube was washed for the last time with 10 mL of a 1% acetic acid solution. All the supernatants of each sample were combined in the second tube, which was centrifuged in order to reduce SPE cartridge clogging during the clean-up step. The cartridge was rinsed with 5 mL of water/ methanol (60:40, v/v) Methanol, Acetonitrile (1/1), with 0.05M sodium hydroxide + Solid/Liquid with agitation Alkaline digestion, methanol sonication 	concentrated sample extract (-20°C)	 ENVI-Carb SPE cartridge SPE anion exchange SPE (XAW) SPE - DVB, solvent match Strata X-AW[®] 200 mg /6 mL; Phenomenex, France 	• Ev • Th ni: 10

Suspended Particulate Matter

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
 4°C (maximum of 30 days) Freezing Freezing, if analysis is not possible within one week 		 Sieving and freeze drying Freeze drying or drying at 40°C, grinding (mortar) 	tetrabutylammonium hydrogensulfate and methyl tert-butyl ether (as extraction solvent)	 fridge 4°C -20°C storage 	• SPE	• [

Pre-concentration

Evaporation with nitrogen

The extract was evaporated to dryness under nitrogen at 45°C and the residue reconstituted in 100µL of 50:50 water/methanol

Pre-concentration

• Evaporation with nitrogen

GAS MATRICES

Air

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up
• 4°C (maximum					
30 days)					

OTHER MATRICES

Other - AFFF Concentrates

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
		• All foam concentrates				
		are prepared				
		and diluted between 5000				
		and 10 million- fold in ULC-MS				
		grade water				

Other - Production facility, products

Sample storage	Filtration (include filter material)	Pre-treatment (e.g. sieving, freeze drying, grinding)	Extraction conditions	Extract storage	Clean-up	
			 Methanol + 60 °C in an ultrasonic bath. 			

Pre-concentration

Pre-concentration

Appendix 6: Methods used to develop PFAS analyses by responding laboratories

PFAS analysis by mass spectrometry methods:

Shoemaker, J. and Tettenhorst D. (2018) Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). U.S. Environmental Protection Agency, Office of Research and Development, National Center for Environmental Assessment, Washington, DC. <u>https://cfpub.epa.gov/si/si_public_record_Report.cfm?Lab=NERL&dirEntryId=343042</u>

Shoemaker, J. A., Grimmett P., and Boutin B. (2008) Method 537: Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). U.S. Environmental Protection Agency, Washington, DC.

https://cfpub.epa.gov/si/si_public_record_report.cfm?Lab=NERL&dirEntryId=198984&simpleSearch =1&searchAll=EPA%2F600%2FR-08%2F092+

Shoemaker, J.A. (2009) Method 538: Determination of Selected Organic Contaminants in Drinking Water by Direct Aqueous Injection-Liquid Chromatography/Tandem Mass Spectrometry (DAI-LC/MS/MS), Revision 1.0. U.S. Environmental Protection Agency, Washington DC, EPA/600/R-09/149. <u>https://www.epa.gov/esam/epa-method-538-determination-selected-organic-contaminants-drinking-water-direct-aqueous</u>

Rosenblum L., Wendelken S.C. (2019) Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry. U.S. Environmental Protection Agency, Washington, DC. <u>https://www.epa.gov/sites/default/files/2019-12/documents/method-533-815b19020.pdf</u>

ISO 21675:2019 Water quality — Determination of perfluoroalkyl and polyfluoroalkyl substances (PFAS) in water — Method using solid phase extraction and liquid chromatography-tandem mass spectrometry (LC-MS/MS) <u>https://www.iso.org/obp/ui/#iso:std:iso:21675:ed-1:v1:en</u>

DIN 38407-42:2011-03 German standard methods for the examination of water, waste water and sludge - Jointly determinable substances (group F) - Part 42: Determination of selected polyfluorinated compounds (PFC) in water - Method using high performance liquid chromatography and mass spectrometric detection (HPLC/MS-MS) after solid-liquid extraction (F 42) https://www.beuth.de/en/standard/din-38407-42/137282966

ISO 25101:2009 Water quality — Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) — Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry

https://www.iso.org/standard/42742.html#:~:text=ISO%2025101%3A2009%20Water%20quality%20 %E2%80%94%20Determination%20of%20perfluorooctanesulfonate,confirmed%20in%202019.%20T herefore%20this%20version%20remains%20current.

CSN EN 15662 Foods of plant origin - Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE - Modular QuEChERS-method <u>https://www.en-standard.eu/csn-en-15662-foods-of-plant-origin-multimethod-for-the-determination-of-pesticide-residues-using-gc-and-lc-based-analysis-following-acetonitrile-extraction-partitioning-and-clean-up-by-dispersive-spe-modular-quechers-method/</u>

Genualdi S. and deJager L. (2019) Determination of 16 Per and Polyfluoroalkyl Substances (PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) https://www.fda.gov/media/131510/download

Higgins C., Field J.A., Criddle C.S., Luthy R.G (2005) Quantitative determination of perfluorochemicals in sediments and domestic sludge. ES&T 39(11), 3946-3956 DOI: <u>https://doi.org/10.1021/es048245p</u>

Hansen K.J, Clemen, L.A, Ellefson M.E, Johnson H.O (2001) Compound-Specific, Quantitative Characterization of Organic Fluorochemicals in Biological Matrices Environmental Science & Technology 35 (4), 766-770 DOI: <u>https://doi.org/10.1021/es001489z</u>

Janda, J., Nödler, K., Brauch, HJ. Zweiner, C., Lange F.T. (2019) Robust trace analysis of polar (C2-C8) perfluorinated carboxylic acids by liquid chromatography-tandem mass spectrometry: method development and application to surface water, groundwater and drinking water. Environ Sci Pollut Res 26, 7326–7336. <u>https://doi.org/10.1007/s11356-018-1731-x</u>

Paul Silcock, Anna Karrman, and Bert van Bavel (2014) Advancing Perfluorinated Compound Analysis Using Simultaneous Matrix Monitoring <u>https://www.waters.com/webassets/cms/library/docs/720003162en.pdf</u>

Holmström K.E, Berger U. (2008) Tissue Distribution of Perfluorinated Surfactants in Common Guillemot (Uria aalge) from the Baltic Sea Environmental Science & Technology 42 (16), 5879-5884 DOI: <u>https://doi.org/10.1021/es800529h</u>

Powley C.R, George S.W. Ryan T.W. Buck R.C (2005) Matrix Effect-Free Analytical Methods for Determination of Perfluorinated Carboxylic Acids in Environmental Matrixes Analytical Chemistry 77 (19), 6353-6358 DOI: <u>https://doi.org/10.1021/ac0508090</u>

Ahrens, L., Vorkamp, K., Lepom, P., Bersuder, P., Theobald, N., Ebinghaus, R., Bossi, R., Barber, J. L., McGovern, E. 2010. Determination of perfluoroalkyl compounds in water, sediment, and biota. ICES Techniques in Marine Environmental Sciences No. 48. 16 pp. DOI: <u>https://doi.org/10.17895/ices.pub.5073</u>

Some method links provided were not currently available in English:

November 2021 in Dutch: https://esites.vito.be/sites/reflabos/2022/Online%20documenten/WAC_IV_A_025.pdf

Jérôme Beaumont; Ahmad El-Masri; François Lestremau (February 2019) in French: <u>https://www.aquaref.fr/composes-perfluores-pfcs</u>

Claudine Chatellier; François Lestremau (June 2014) in French: <u>https://www.aquaref.fr/composes-perfluores-biotes</u>

Claudine Chatellier; Olivier Diago, Olivier Aguerre-Chariol (June 2014) in French: <u>https://www.aquaref.fr/composes-perfluores-boues</u>

Bioassay methods for PFAS analysis:

Behnisch PA, Besselink H, Weber R, Willand W, Huang J, Brouwer A. Developing potency factors for thyroid hormone disruption by PFASs using TTR-TRβ CALUX[®] bioassay and assessment of PFASs

mixtures in technical products. *Environ Int*. 2021 Dec;157:106791. DOI: <u>10.1016/j.envint.2021.106791</u>. Epub 2021 Aug 4. PMID: 34364217.

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Van der Burg, B. van Vugt-Lussenburg B. (2018) DB-ALM Protocol n° 197: Automated CALUX reporter gene assay procedure <u>http://cidportal.jrc.ec.europa.eu/ftp/public/JRC-OpenData/EURL-</u> <u>ECVAM/datasets/DBALM/VER3-0/online/DBALM_docs/197_P_Automated%20CALUX.pdf</u>