PFAS – PT Study

Results of the first inter-laboratory comparison based on real world samples

Michael Wilken

Why this study?

- PFAS are emerging contaminant
- The methods are still developing
- Decreasing detection limits are required, and new components are frequently added
- Currently a discrepancy in requirements between North America, Europe, Australia

- We want to understand the potential variability of data between the labs

- We want to find and identify preferred laboratories

- 13 laboratories invited, 12 participated

What does real world sample mean?

- The concept is based on providing blends of samples from various locations
- These blends have the “backbones” (matrices) from the sources which may pose analytical challenges (interferences)
- The blends are in general not spiked
- The blends are provided camouflaged and typically as a concentrated and a diluted blend. One of them may be provided as a duplicate
- It allows us to evaluate the quality at different concentration levels and to determine the RPD.
- Dow has implemented this concept for numerous parameters (VOC, SVOC, metals, dioxin).
- This concept helps to identify, qualify and re-qualify the preferred contract laboratories.

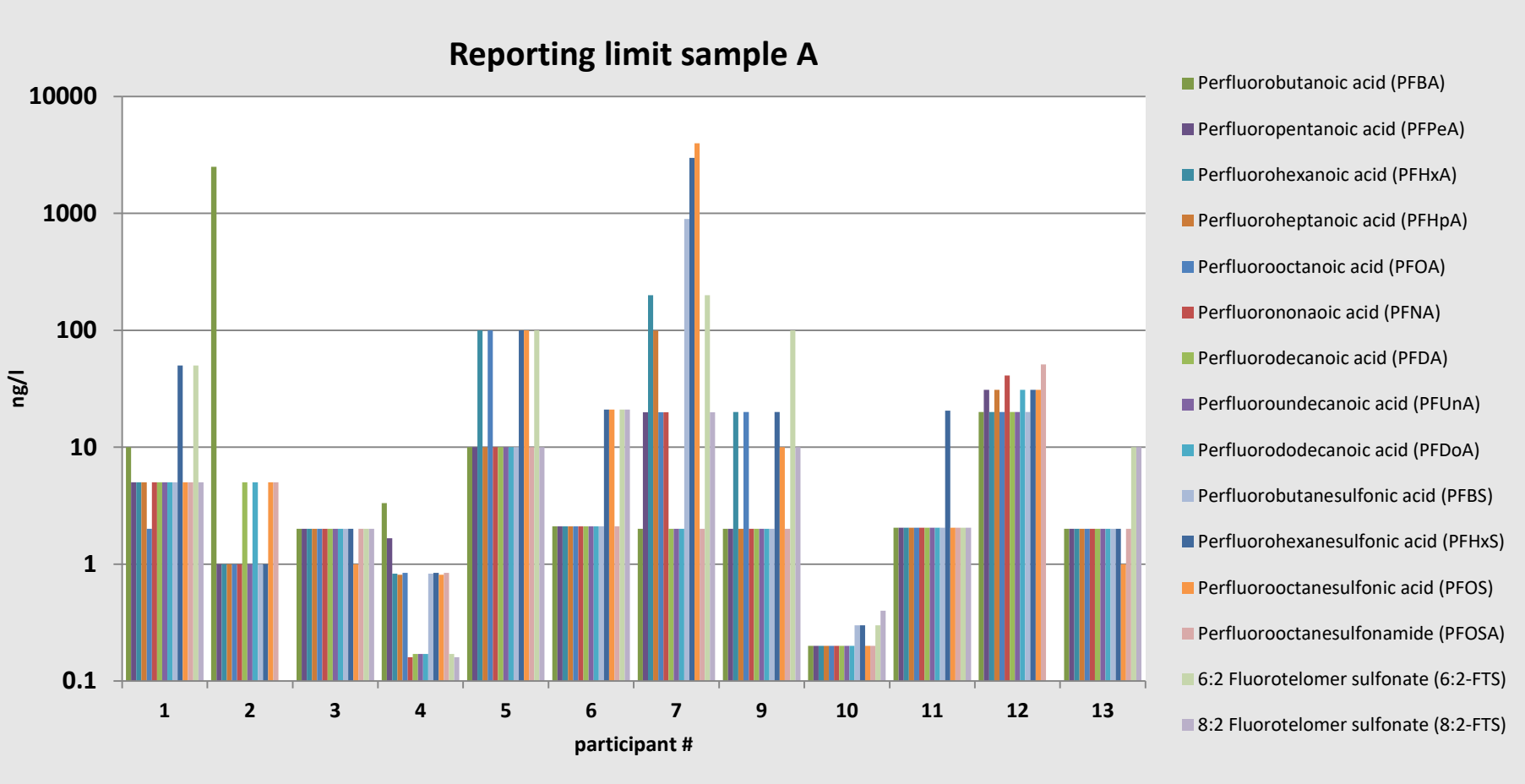
Details for this study

- Material is blend of left-overs from various non-Dow projects, mixed with additional non-PFAS containing water to add a “backbone”
- Neat sample of the blend (A) plus diluted sample as duplicate (B,C) provided to labs. For sample C extra bottles provided for lab-duplicate
- Sample D is lab water in which Teflon laboratory material was soaked for a week. Finally spiked with a mix of 12 standards to achieve 5 ng/l per component (carboxylic acids and sulfonates)
- No requirements for any specific method (US and Europe would be totally different anyway)
- Isotope dilution required
- Indication if only linear or linear + branched were analyzed
- Selection of 15 PFAS (see next table)

Requested Analytes and Reporting Limits

Analytes	Requested Reporting limit
	ng/l
Perfluorobutanoic acid (PFBA)	2
Perfluoropentanoic acid (PFPeA)	2
Perfluorohexanoic acid (PFHxA)	2
Perfluoroheptanoic acid (PFHpA)	2
Perfluorooctanoic acid (PFOA)	2
Perfluorononaoic acid (PFNA)	2
Perfluorodecanoic acid (PFDA)	2
Perfluoroundecanoic acid (PFUnA)	2
Perfluorododecanoic acid (PFDoA)	2
Perfluorobutanesulfonic acid (PFBS)	2
Perfluorohexanesulfonic acid (PFHxS)	2
Perfluorooctanesulfonic acid (PFOS)	1
Perfluorooctanesulfonamide (PFOSA)	2
6:2 Fluorotelomer sulfonate (6:2-FTS)	10
8:2 Fluorotelomer sulfonate (8:2-FTS)	10

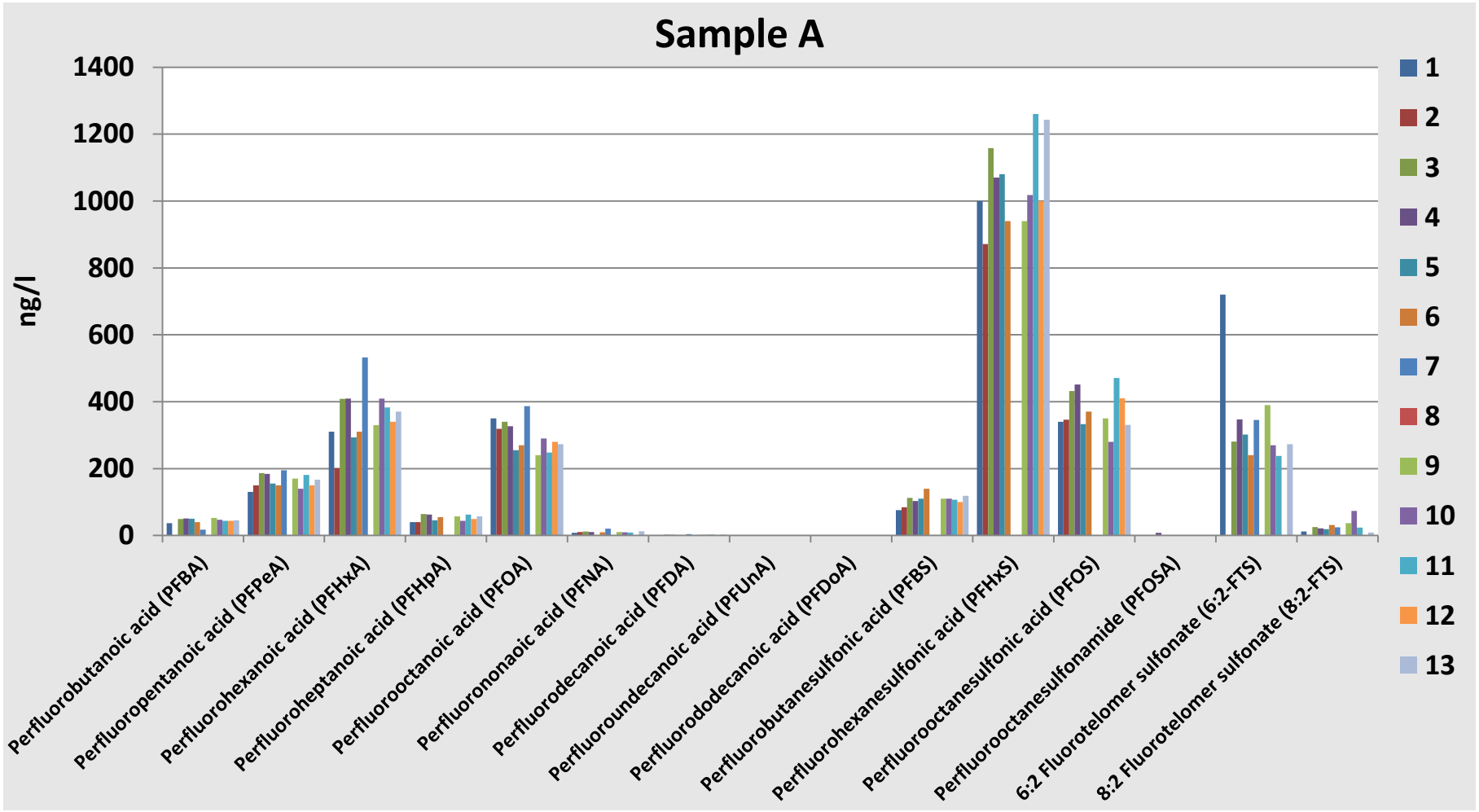
Achieved Reporting Limits

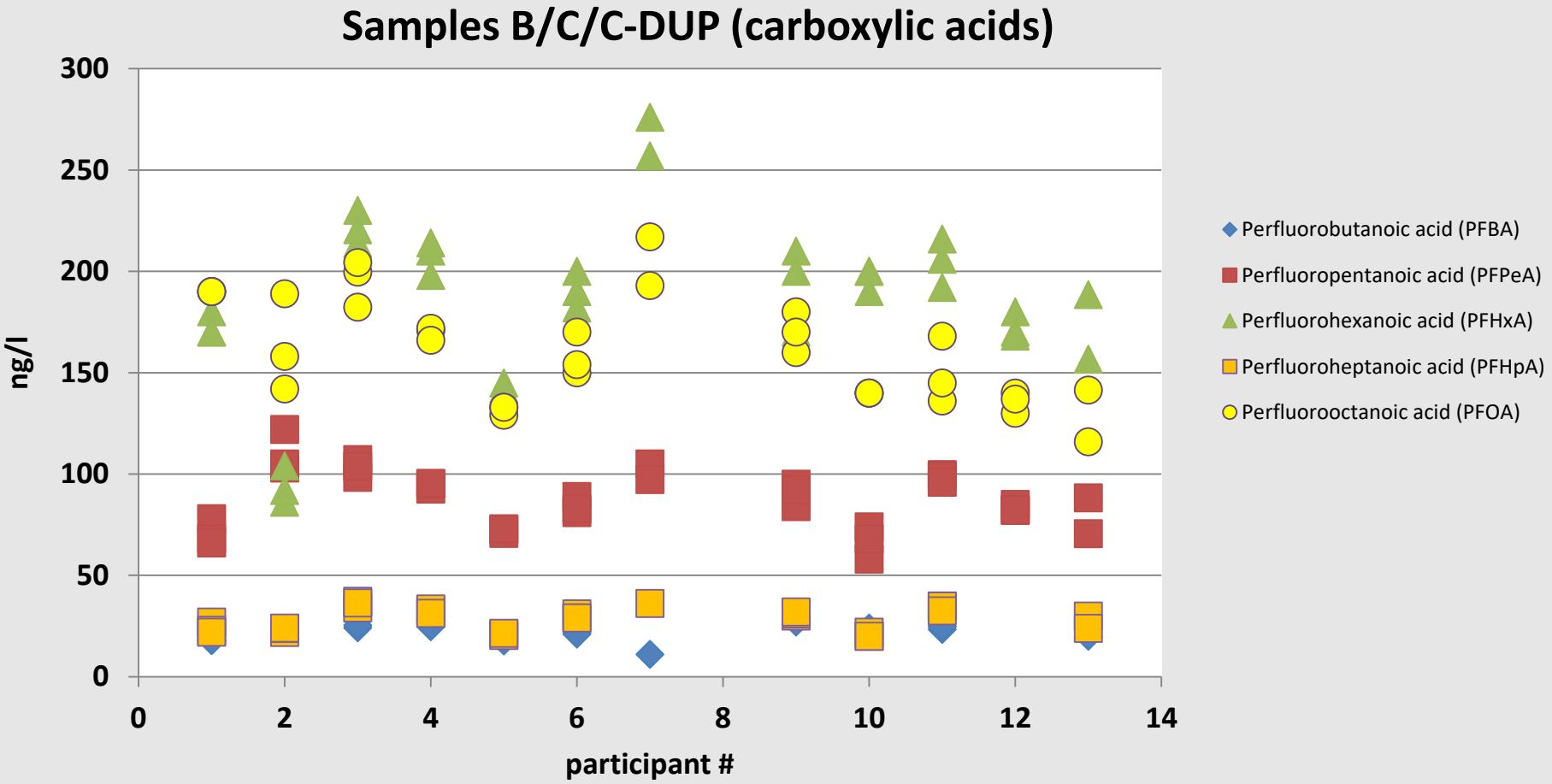


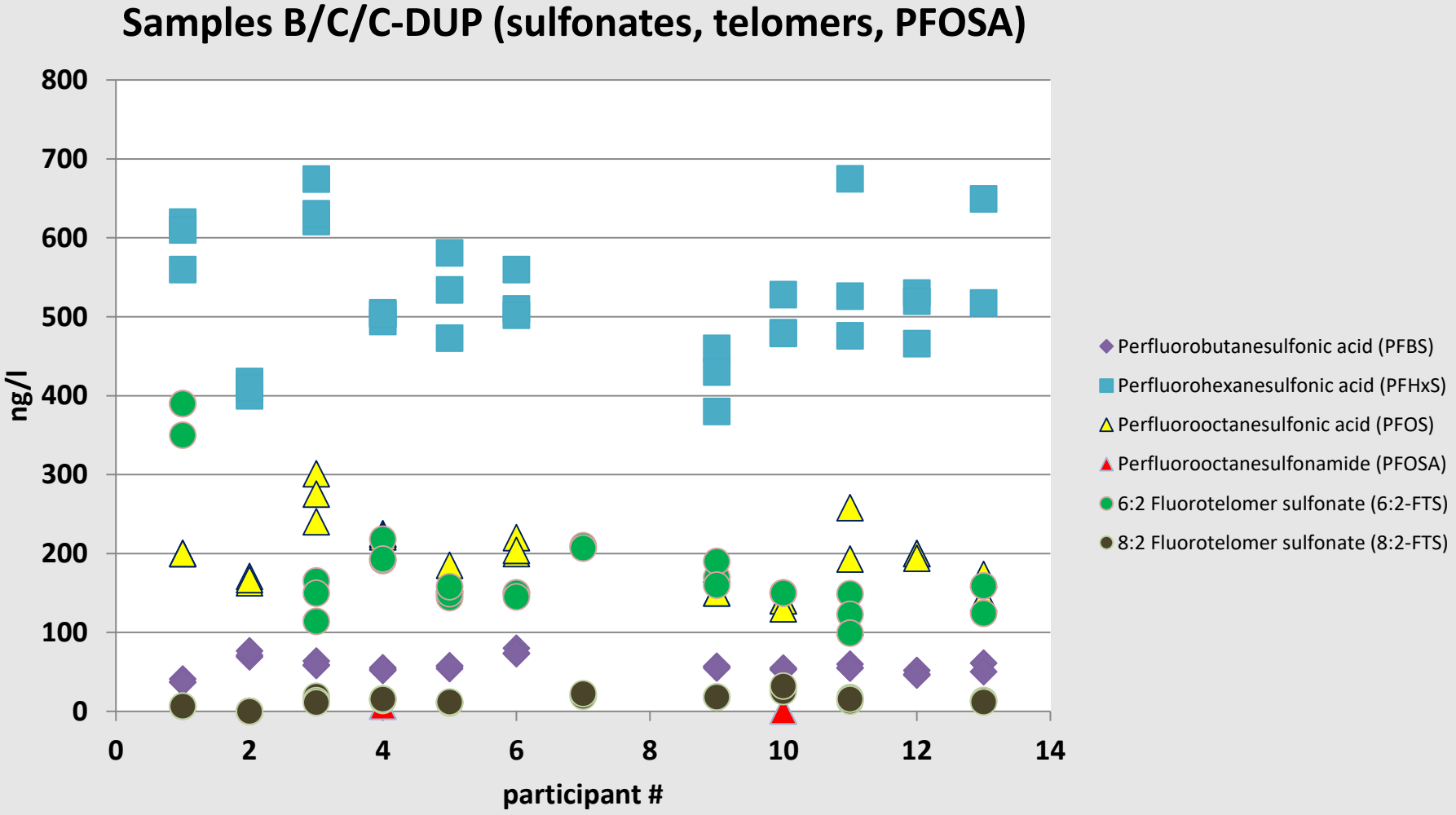
Y-axis is LOGARITHMIC scale!!!

all data points reported as < RL or ND are set to 0

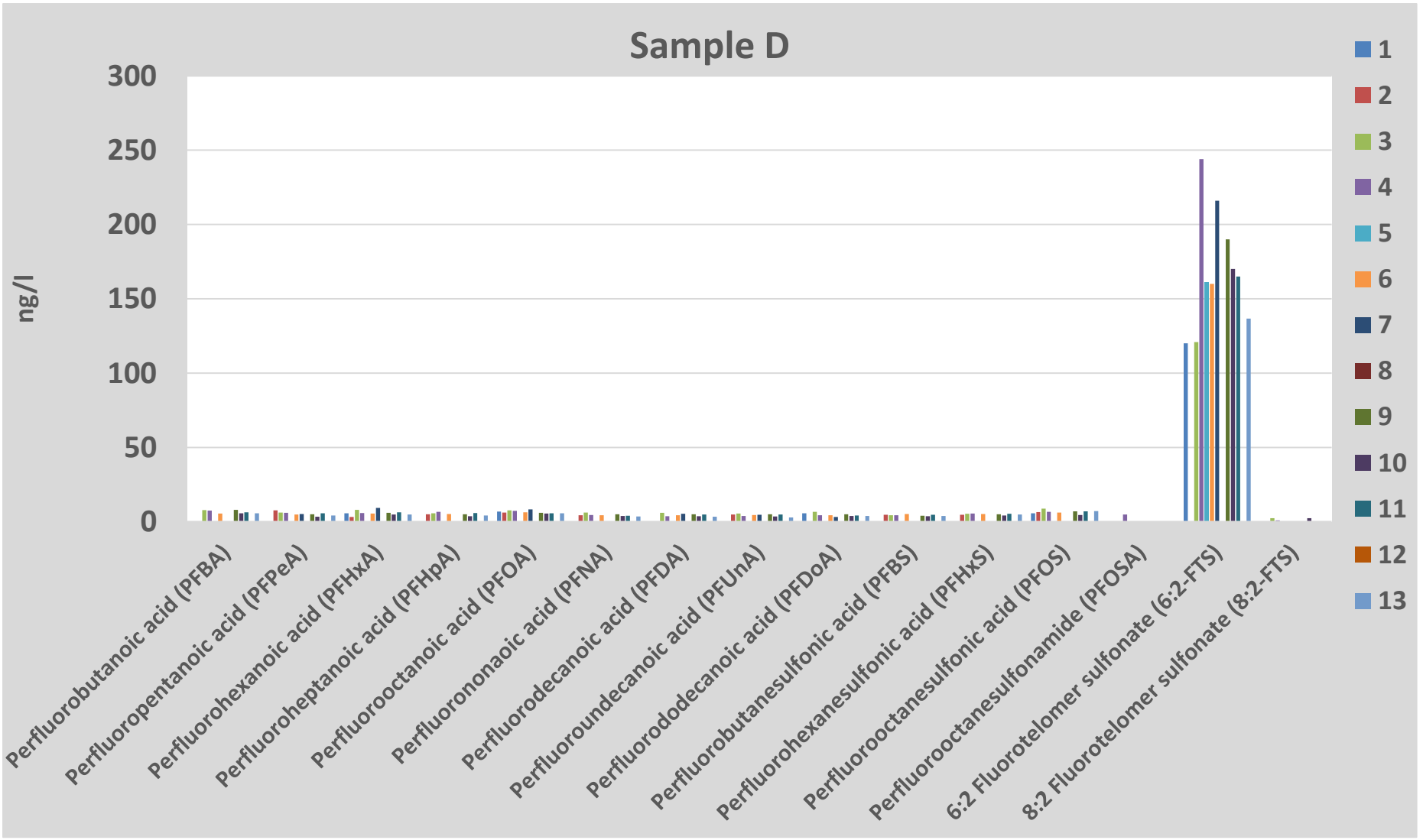
Results

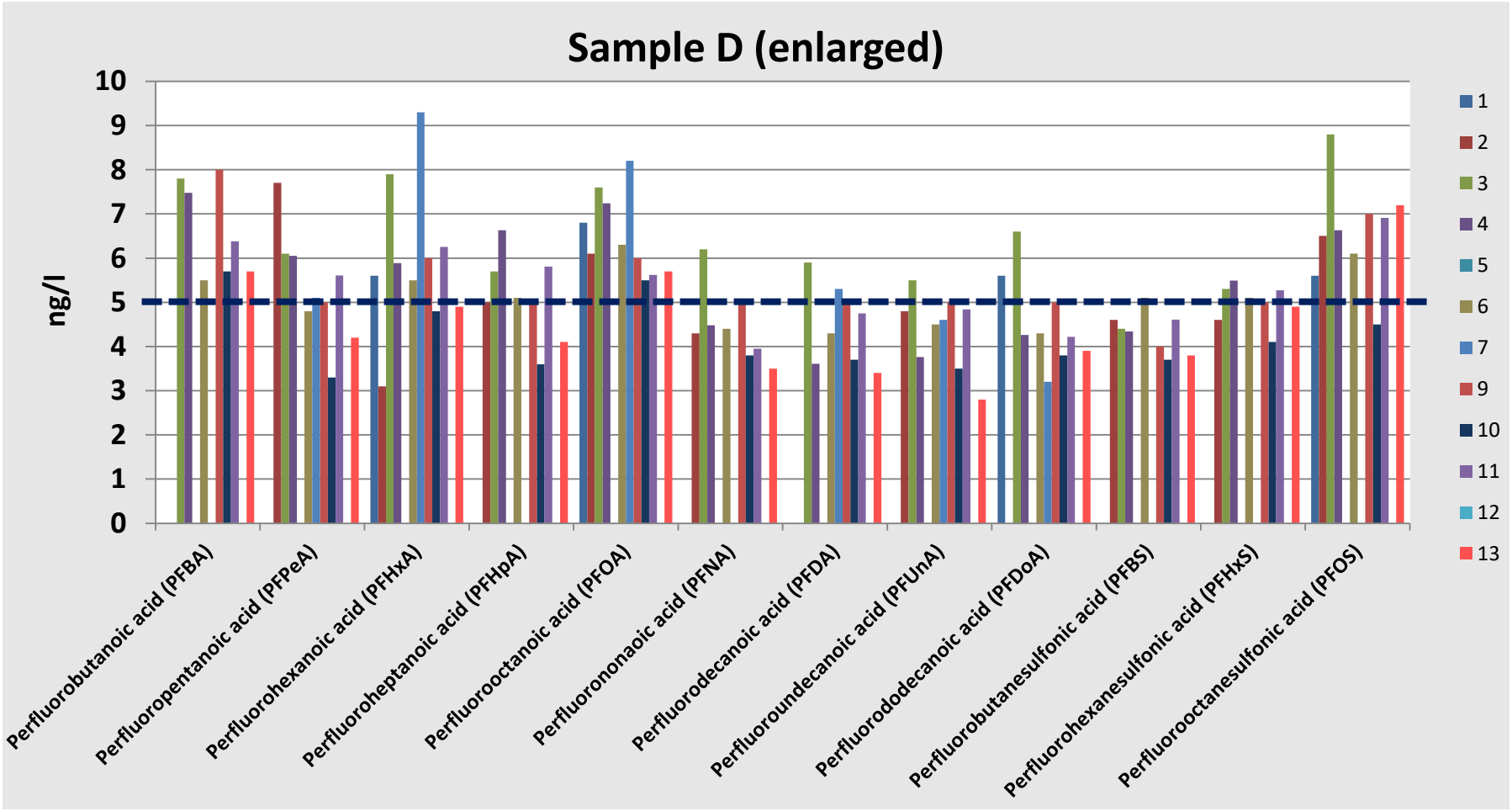

Sample A
ng/l
1400
1200
1000
800
600
400
200
0
1
2
3
4
5
6
7
8
9
10
11
12
13
Perfluorobutanoic acid (PFBA)
Perfluoropentanoic acid (PFPeA)
Perfluorohexanoic acid (PFHxA)
Perfluoroheptanoic acid (PFHpA)
Perfluorooctanoic acid (PFOA)
Perfluorononaoic acid (PFNA)
Perfluorodecanoic acid (PFDA)
Perfluoroundecanoic acid (PFUnA)
Perfluorododecanoic acid (PFDoA)
Perfluorobutanesulfonic acid (PFBS)
Perfluorohexanesulfonic acid (PFHxS)
Perfluorooctanesulfonic acid (PFOS)
Perfluorooctanesulfonamide (PFOSA)
6:2 Fluorotelomer sulfonate (6:2-FTS)
8:2 Fluorotelomer sulfonate (8:2-FTS)


Samples B/C/C-DUP (carboxylic acids)
ng/l
300
250
200
150
100
50
0
0
2
4
6
8
10
12
14
participant #
Perfluorobutanoic acid (PFBA)
Perfluoropentanoic acid (PFPeA)
Perfluorohexanoic acid (PFHxA)
Perfluoroheptanoic acid (PFHpA)
Perfluorooctanoic acid (PFOA)


Samples B/C/C-DUP (sulfonates, telomers, PFOSA)
800
700
600
500
400
300
200
100
0
ng/l
0
2
4
6
8
10
12
14
participant #
Perfluorobutanesulfonic acid (PFBS)
Perfluorohexanesulfonic acid (PFHxS)
Perfluorooctanesulfonic acid (PFOS)
Perfluorooctanesulfonamide (PFOSA)
6:2 Fluorotelomer sulfonate (6:2-FTS)
8:2 Fluorotelomer sulfonate (8:2-FTS)

EH&S Remediation




Sample D (enlarged)
ng/l
10
9
8
7
6
5
4
3
2
1
0
1
2
3
4
5
6
7
9
10
11
12
13
Perfluorobutanoic acid (PFBA)
Perfluoropentanoic acid (PFPeA)
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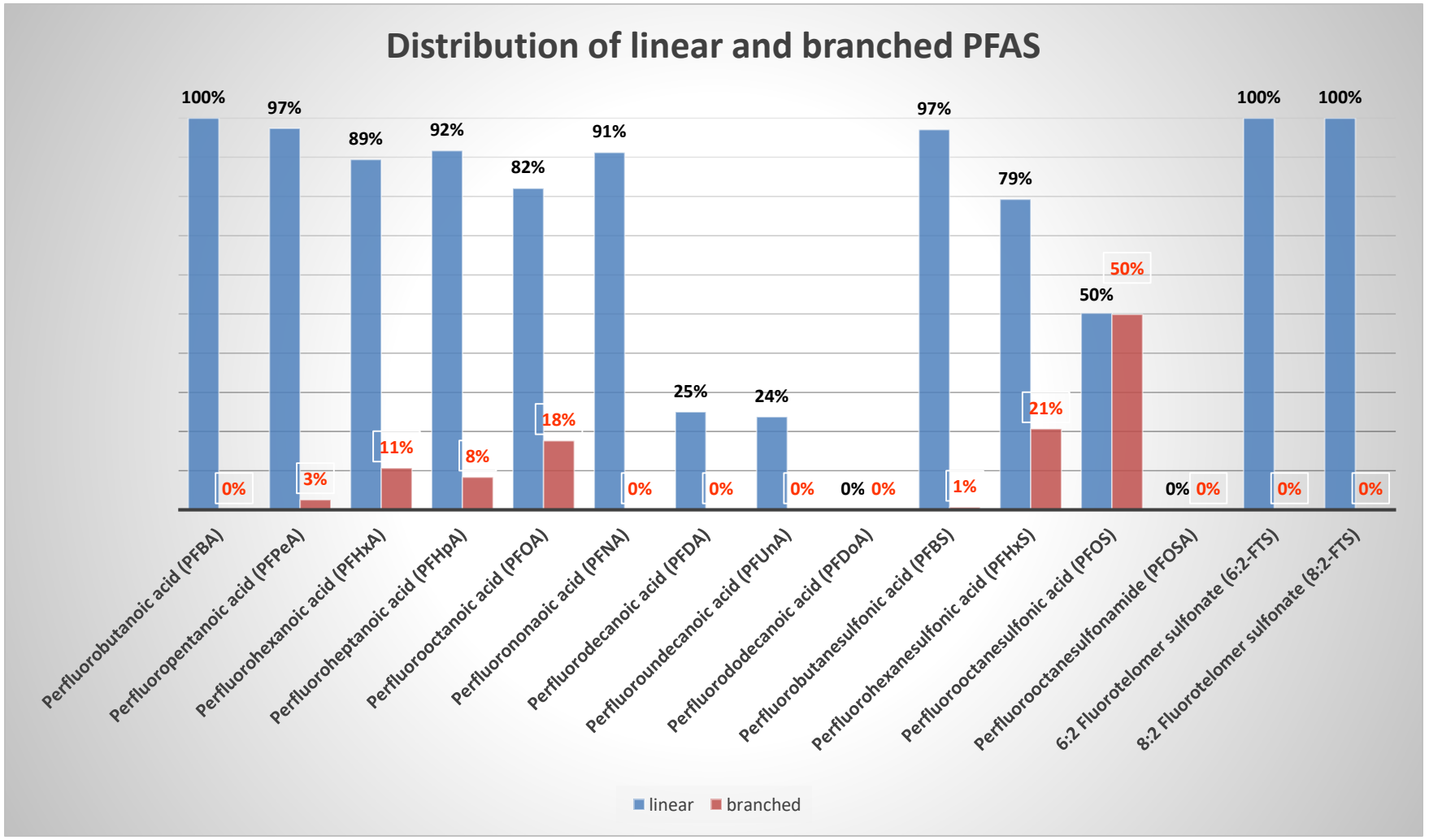
Perfluoroheptanoic acid (PFHpA)
Perfluorooctanoic acid (PFOA)
Perfluorononaoic acid (PFNA)
Perfluorodecanoic acid (PFDA)
Perfluoroundecanoic acid (PFUnA)
Perfluorododecanoic acid (PFDoA)
Perfluorobutanesulfonic acid (PFBS)
Perfluorohexanesulfonic acid (PFHxS)
Perfluorooctanesulfonic acid (PFOS)
Spike level

Analyzed components

Analytes	1	2	3	4	5	6	7	9	10	11	12	13
Perfluorobutanoic acid (PFBA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluoropentanoic acid (PFPeA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorohexanoic acid (PFHxA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluoroheptanoic acid (PFHpA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorooctanoic acid (PFOA)	S	S	S	S	S	S	S	S	L	S	S	L
Perfluorononaoic acid (PFNA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorodecanoic acid (PFDA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluoroundecanoic acid (PFUnA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorododecanoic acid (PFDoA)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorobutanesulfonic acid (PFBS)	S	L	S	S	S	L	L	L	L	S	S	L
Perfluorohexanesulfonic acid (PFHxS)	S	L	S	S	S	S	S	S	L	S	S	L
Perfluorooctanesulfonic acid (PFOS)	S	S	S	S	S	S	S	S	L	S	S	L
Perfluorooctanesulfonamide (PFOSA)	S	L	S	S	S	L	L	L	L	S	S	L
6:2 Fluorotelomer sulfonate (6:2-FTS)	S		S	S	S	L	L	L	L	S		L
8:2 Fluorotelomer sulfonate (8:2-FTS)	S		S	S	S	L	L	L	L	S		L

L = linear components only
S = linear + branched components

EH&S Remediation



More results

- Recovery rates of surrogate standards vary strongly from component to component and from laboratory to laboratory
- Some standard recovery rates are typically in the range of 50% ($^{13}C_4$-PFBA), while others ($^{13}C_2$-6:2 FTS) are way above 100% (up to 292 %)
- This indicates the need of the isotope dilution method to correct for the apparent inconsistencies

Summary

- Variability between the laboratories is (surprisingly) small
- Biggest problem is sensitivity: varies more than factor 1000 !!!
- European (0.65 ng/l) and Australian (0.23 ng/l) surface water requirements can be achieved only by the European lab
- 6 North American labs can achieve 2 ng/l or below
- Laboratories do not analyze the same things (linear w or w/o branched)
- Sample D highlights the importance of an absolutely clean lab
- Variability of surrogate recoveries requires the use of isotope dilution

I like to thank all laboratories for their participation

and I like to thank

YOU

for your attention