

# BEPALING VAN PER- EN POLYFLUORALKYLVERBINDINGEN (PFAS) IN WATER

DO Werkgroep Water – partim PFAS - 02/09/2020

## STUDIE UITGEVOERD iov DO (2019) - DOELSTELLINGEN

- Uitbreiding parameterlijst van WAC/IV/A/025:
  - PFOS en derivaten (B.2.3.1 indelingscriterium)
  - PFOA en gerelateerde verbindingen (Stockholm conventie aangaande POPs)
  - REACH SVHC stoffen
  - Precursoren, oa. fluortelomeerverbindingen
  - Vervangmiddelen voor PFOA en PFOS: GenX (HFPO-DA), ADONA, PFECBS, CIPFECA ...
  - Afstemming op ISO 21675:2019
- Bepaling LC-MS/MS condities
- Bepaling terugvinding na extractie
- Validatie adhv. geaddeerde DW/GW/OW en AW stalen
- Evaluatie TOPA (total oxidisable precursor assay): detectie precursoren

## REGELGEVING

- Risicogrenswaarden afgeleid voor het watercompartiment door RIVM (bron: PFAS expertisecentrum - Een handelingskader voor PFAS, 2019)

	PFOS	PFOA
<b>Grondwater</b>		
Bovengrens (interventiewaarde)	4.7 µg/l	0.39 µg/l
Ondergrens (streefwaarde)	0.00023 µg/l	-
Humane risicogrens (wonen met moestuin)	-	12 µg/l
<b>Oppervlaktewater</b>		
Bovengrens jaargemiddelde	0.00065 µg/l	0.048 µg/l
<b>Drinkwater</b>		
Toetsingswaarde	0.53 µg/l	0.0875 µg/l

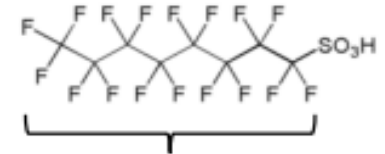
## REGELGEVING (vervolg)

- Milieukwaliteitsnorm OW (Vlarem II bijlage 2.3.1): PFOS en afgeleiden: 0.00065 µg/l
- Nieuwe EU Drinkwaterrichtlijn :
  - 0.1 µg/l voor PFAS individueel
  - 0.5 µg/l voor PFAS totaal (n = 20, C4-C13 PFCA, C4-C13 PFSA)
- Bepalingsgrenseis OW (WAC/VI/A/001): 50 ng/l
- Bepalingsgrenseis AW (Vlarem II bijlage 4.2.5.2): 100 ng/l

## PFAS

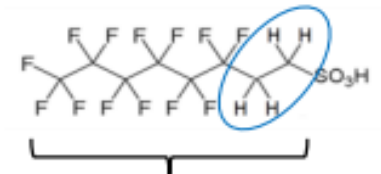
- Per- en polyfluoroalkylverbindingen
- OECD: > 4000 verbindingen met CAS n°
- Meestal zuur: bepaling met LC-MS
  - PFCA
  - PFSA
  - FTA
  - FTS
  - Esters van fosfor/fosforig/fosfienzuren
  - ...
- Soms neutraal: bepaling met GC-MS/LC-MS
  - FTOH
  - Sulfonamidederivaten
  - Iodides
  - Acrylaten
  - ...

Geperfluoreerd:



volledig gefluoreerd

Gepolyfluoreerd:



gedeeltelijk gefluoreerd

## PRECURSOREN

- **Stoffen die in het milieu kunnen afbreken naar PFSA's en PFCAs**
  - fluorotelomeren (polyfluoreerde stoffen)
  - perfluorsulfonamido carboxylzuren
  - perfluorbetaines
  - perfluorsulfonamides
  - perfluorsulfonamidoethanol
  - perfluorthioamido amino carboxylaten
  - perfluorsulfonamido amines
  - perfluoralkyl amido betaines
  - perfluorsulfonamido amine oxides
  - perfluorthioamido sulfonaten
  - perfluorthiohydroxyl ammonium
  - perfluorsulfonamide ketonen, aldehyden and ethers
  - zijketen gefluoreerde polymeren (bv. polyfluortelomeeracrylaten).
- **Belangrijke groep van PFAS wordt met standaard LC-MS/MS methode niet gedetecteerd**
  - > TOPA
  - > organische fluor (TOF, EOF, AOF) met bv. CIC
  - > identificatie via non-target analyse

## PFAS TOEPASSINGEN

- Brandblusschuimen
- Metaalbewerkingsprocessen
- Vuil-, vet- en waterafstoting (papier, textiel, leder, tenten, tapijten, meubels, ...)
- Cosmetica
- Hydraulische vloeistoffen
- Smeermiddelen
- Pesticideformuleringen
- Waxen (o.a. skiwax, autowax)
- Anti-aanbaklagen
- ...

## NORMMETHODEN

- **WAC/IV/A/025:**
  - DW, GW, OW, AW
  - C4-C18 PFCAs, C4-C10 PFSA's, FOSA
  - SPE (Oasis WAX), elutie met MeOH en MeOH (1% NH<sub>3</sub>)
  - LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op C18-kolom
  - Interne standaard methode
  - LOQ: 10 ng/l
- **ISO 21675:2019**
  - DW, GW, OW, AW
  - C4-C18 PFCAs, C4-C10 PFSA's, FOSA, MeFOSA, EtFOSA, FOSAA, MeFOSAA, EtFOSAA, 6:2 FTS, 8:2 FTS, 9Cl-PF3ONS, 8:2 FTUCA, 8:2 diPAP, HFPO-DA en ADONA (totaal van 30 PFAS)
  - SPE (Oasis WAX), elutie met MeOH en MeOH (1% NH<sub>3</sub>)
  - LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op C18-kolom
  - Interne standaard methode
  - LOQ : 0.2 ng/l



## NORMMETHODEN (vervolg)

### ▪ EPA method 537.1 (12/2018)

#### ▪ DW

- C4-C14 PFCAs, C4-C8 PFSA's, MeFOSAA, EtFOSAA, 11Cl-PF3OUdNS, 9Cl-PF3ONS, 8:2 FTUCA, 8:2 diPAP, HFPO-DA en ADONA
- SPE (PS-DVB) elutie met MeOH
- LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op C18-kolom
- Interne standaard methode
- LOQ: 0.5-6 ng/l

### ▪ EPA method 533 (12/2019)

#### ▪ DW

- C4-C12 PFCAs, C4-C8 PFSA's, 4:2 FTS, 6:2 FTS, 8:2 FTS, 11Cl-PF3OUdNS, 9Cl-PF3ONS, 8:2 diPAP, HFPO-DA, ADONA, PFMPA, PFMBBA, PFEESA en NFDHA
- SPE (PS-DVB anion exchange), elutie met MeOH (1% NH<sub>3</sub>)
- LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op C18-kolom
- Interne standaard methode
- LOQ: 10 ng/l

## NORMMETHODEN (vervolg)

- EPA method 8327:2019
  - GW, OW, AW
  - C4-C14 PFCAs, C4-C10 PFSA's, 4:2 FTS, 6:2 FTS, 8:2 FTS, FOSA, MeFOSAA, EtFOSAA
  - Directe injectie na verdunning met MeOH/water 1/1 en aanzuren met HAC
  - LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op C18-kolom
  - Externe standaard methode
  - LOQ: 10 ng/l
- ASTM D7979 – 19 (11/2019)
  - DW, GW, OW, AW
  - C4-C14 PFCAs, C4-C8 PFSA's, PFECHS; 6:2 FTA, 8:2 FTA, 10:2 FTA, 6:2 FTUA en 8:2 FTUA
  - Directe injectie na verdunning met MeOH/water 1/1
  - LC-(ESI-)MS/MS met H<sub>2</sub>O/MeOH/NH<sub>4</sub>Ac gradient op phenyl-hexyl-kolom
  - Interne of externe standaard methode
  - LOQ: 10 ng/l

## VOORSTEL UITBREIDING PFAS PARAMETERLIJST WAC

- Selectie obv. parameters normmethoden, voorkomen, beschikbaarheid/kostprijs standaarden, regelgeving
- Perfluoralkaancarbonsuren (PFCA): C4-C18
- Perfluoralkaansulfonzuren (PFSA): C4-C12, PFECHS
- Fluortelomeersulfonzuren (n:2 FTS): 4:2 FTS, 6:2 FTS, 8:2 FTS, 10:2 FTS
- Fluorooctaansulfonamideverbindingen: FOSA, MeFOSA, EtFOSA, FOSAA, MeFOSAA, EtFOSAA
- Fluortelomeerfosfaatesters (PAPs): 6:2 PAP, 8:2 PAP, 6:2 diPAP, 6:2/8:2 diPAP, 8:2 diPAP
- Perfluorethercarbonsuren (PFECA): HFPO-DA (Gen-X), ADONA
- *Opm.: Tijdelijk handelingskader PFAS (hergebruik grond NL): C4-C18 PFCA, C4-C10 PFSA, 4:2 FTS, 6:2 FTS, 8:2 FTS, 10:2 FTS, MeFOSAA, EtFOSAA, FOSA, MeFOSA, 8:2 diPAP, GenX (optioneel)*

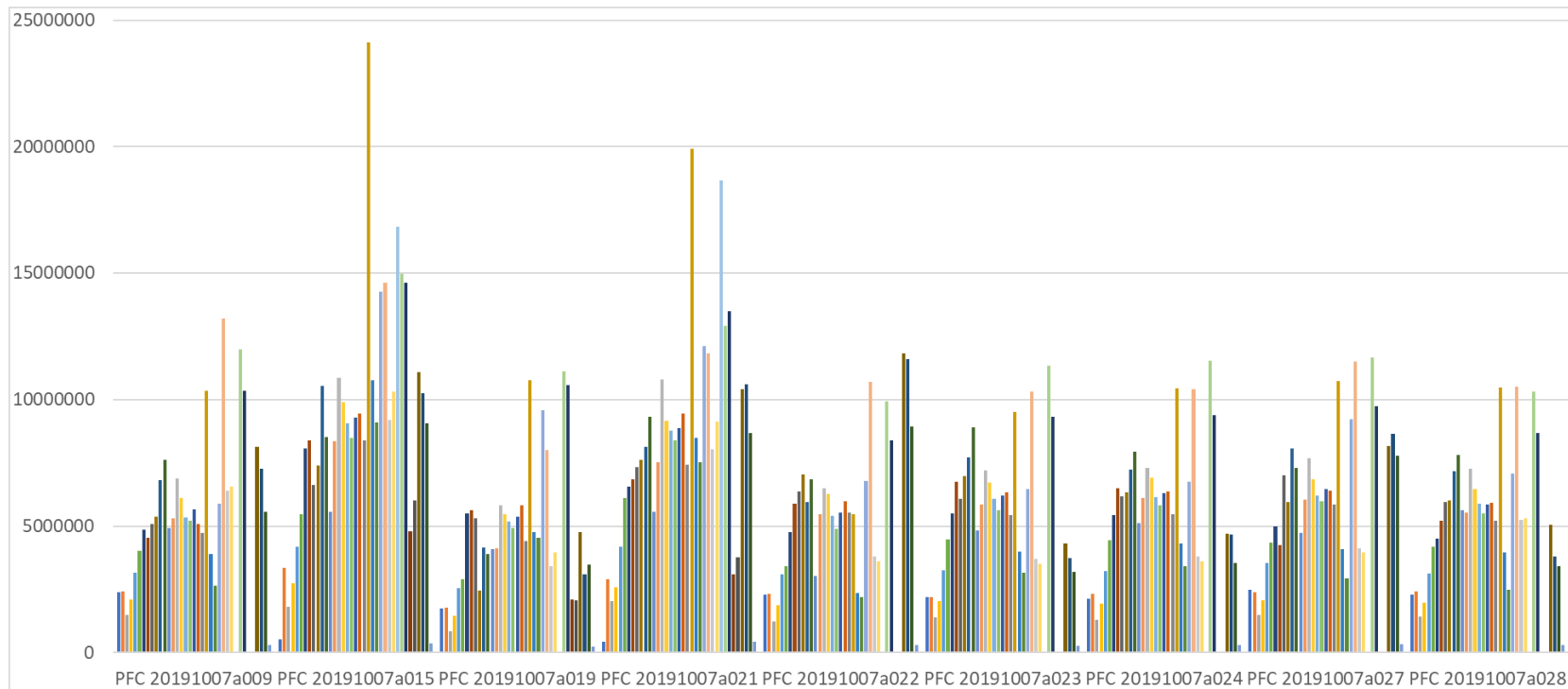
## INTERNE STANDAARDEN

1	PFBA	13C-PFBA	22	4:2FTS	13C-6:2FTS
2	PFPeA	13C-PFPeA	23	6:2FTS	13C-6:2FTS
3	PFHxA	13C-PFHxA	24	8:2FTS	13C-6:2FTS
4	PFHpA	13C-PFHxA	25	10:2FTS	13C-6:2FTS
5	PFOA	13C-PFOA	26	FOSA	13C-FOSA
6	PFNA	13C-PFNA	27	N-MeFOSA	13C-FOSA
7	PFDA	13C-PFDA	28	N-EtFOSA	13C-FOSA
8	PFUdA	13C-PUdA	29	FOSAA	13C-MeFOSAA
9	PFDoA	13C-PFDoA	30	N-MeFOSAA	13C-MeFOSAA
10	PFTTrDA	13C-PFDoA	31	N-EtFOSAA	13C-MeFOSAA
11	PFTeDA	13C-PFTeDA	32	6:2PAP	13C-6:2 PAP
12	PFHxDA	13C-PFHxDA	33	8:2PAP	13C-6:2 PAP
13	PFODA	13C-PFHxDA	34	6:2diPAP	13C-6:2 diPAP
14	PFBS	13C-PFHxS	35	6:2/8:2diPAP	13C-6:2 diPAP
15	PFPeS	13C-PFHxS	36	8:2diPAP	13C-6:2 diPAP
16	PFHxS	13C-PFHxS	37	HFPO-DA	13C-HFPO-DA
17	PFHpS	13C-PFHxS	38	ADONA	13C-HFPO-DA
18	PFOS	13C-PFOS	39	PFECHS	13C-PFOS
19	PFNS	13C-PFOS			
20	PFDS	13C-PFOS			
21	PFDoS	13C-PFOS			

## LC-MS/MS INSTELLINGEN

- MRM transities
- Evaluatie LC-condities en invloed op respons
  - Kolomselectie: BEH C18, HSS T3, CSH Phenyl-hexyl
  - Mobiele fase: water/MeOH/NH<sub>4</sub>Ac
    - Begingradient
    - Bufferconcentratie
    - Additie HCOOH aan injectievial
    - *Opm*: geen signaal voor mono-PAPs en FOSAA
  - Alkalische mobiele fase: water (NH<sub>3</sub>, pH 10)/MeOH
    - Goed signaal voor alle PFAS, echter retentietijdshifts, PFBA en PFPA geen goede piekvorm
    - Grootste responsen (in geval van BEH C18 kolom)
  - Post-kolom additie NH<sub>3</sub>: geen effect
  - Besluit: LC-instellingen weinig invloed, dus LC-MS/MS methode vrij robuust

# EVALUATIE LC-CONDITIES



## FINALE UPLC-CONDITIES

- NH<sub>4</sub>Ac buffer
  - Kolom: BEH C18, 130Å, 1.7 μm, 2.1 mm x 100 mm + prekolom
  - Kolomtemperatuur: 40°C
  - Injectievolume: 10 μl
  - Gradiënt:
    - A : Water/MeOH 95/5 + 2mM NH<sub>4</sub>Ac
    - B : MeOH + 2mM NH<sub>4</sub>Ac
    - 70% A > 90% B
- Alkalische buffer
  - Kolom: idem
  - Gradiënt:
    - A : Water + 2mM NH<sub>4</sub>Ac, pH op 9 brengen met NH<sub>3</sub>
    - B : ACN/MeOH 1/1 + 5mM MP (=1-methylpiperidine, 0.614 ml /l)
    - C : Water/MeOH/ACN/IPA 25/25/25/25
    - 90% A/10% B > 95% B/5% C

## KALIBRATIE EN INSTRUMENTELE DETECTIELIMIETEN

- Lineair bereik (Waters Xevo-TQ-S): 0.1-30  $\mu\text{g/l}$
- Residuele afwijking <20%,  $r^2 >0.996$
- IDL obv. S/R (peak-to-peak): <0.02  $\mu\text{g/l}$ , behalve HFPO-DA 0.1  $\mu\text{g/l}$



## EVALUATIE SPE

- SPE werkwijze WAC en ISO
- Vaststelling: onvoldoende terugvinding lange keten PFAS, MeFOSA, EtFOSA
- Analyse afzonderlijke fracties:
  - Water na SPE
  - NH<sub>4</sub>Ac wasoplossing
  - MeOH eluaat
  - MeOH (1% NH<sub>3</sub>) eluaat (x2)
  - MeOH (2 % NH<sub>3</sub>)/ACN 20/80 eluaat
- Bijna alle PFAS in 1ste MeOH (1% NH<sub>3</sub>) fractie
- FOSA, MeFOSA, EtFOSA partieel in MeOH eluaat
  
- Conclusie: SPE werkt goed, verliezen zijn gevolg van adsorptie aan recipientwanden en injectienaald
  - => herneming SPE met extra spoelen/vortexen wanden monsterfles en extractierecipienten

## SPE RESULTATEN VOOR ultrapuur WATER (250 ng/l, n =3)

	Additie ng/l	Gemidd. ng/l	%RSD	Terugvinding
PFBA	261	273	3.6%	105%
PFPeA	266	268	5.4%	101%
PFHxA	267	275	5.7%	103%
PFHpA	263	246	4.5%	93%
PFOA	263	283	9.8%	107%
PFNA	265	265	2.3%	100%
PFDA	260	243	10.6%	93%
PFUdA	263	210	10.6%	80%
PFDoA	248	228	7.5%	92%
PFTrDA	258	174	11.6%	67%
PFTeDA	264	252	12.6%	95%
PFHxDA	263	337	4.5%	128%
PFODA	274	157	9.8%	57%
PFBS	232	242	2.5%	104%
PFPeS	253	248	1.5%	98%
PFHxS	249	256	4.0%	103%
PFHpS	247	227	5.8%	92%
PFOS	248	253	3.1%	102%
PFNS	247	202	5.8%	82%
PFDS	260	180	8.3%	70%
PFDoS	245	97	6.6%	40%

	Additie ng/l	Gemidd. ng/l	%RSD	Terugvinding
4:2 FTS	249	238	7.2%	96%
6:2 FTS	264	246	3.3%	93%
8:2 FTS	258	224	4.9%	87%
10:2 FTS	255	175	7.5%	69%
FOSA	250	245	4.4%	98%
MeFOSA	253	282	2.7%	111%
EtFOSA	264	292	3.4%	110%
FOSAA	264	229	14.0%	87%
MeFOSAA	259	239	8.5%	92%
EtFOSAA	271	233	9.8%	86%
6:2 PAP	241	227	5.7%	94%
8:2 PAP	239	110	12.1%	46%
6:2 diPAP	255	240	3.6%	94%
6:2/8:2 diPAP	248	262	9.8%	106%
8:2 diPAP	254	500	17.8%	197%
HFPO-DA	259	283	15.6%	110%

## VALIDATIE UITBREIDING PARAMETERLIJST

### ■ DW/GW/OW:

191022-0002_DW Vito	Drinkwater VITO
191022-0003_GW Lommel	Grondwater bemonsterd in Lommel (Kerkhoven)
191022-0004_GW Olen	Grondwater bemonsterd in Onze-Lieve-Vrouw Olen
191022-0005_OW	Oppervlaktewater aangeleverd door VMM (OW 1+2)
191022-0006_OW	Oppervlaktewater aangeleverd door VMM (OW 11 + 12)

### ■ AW:

191022-0007_AW	VMM AW 25+26
191022-0008_AW	VMM AW 30+31
191022-0009_AW	VMM AW 29+32
191022-0010_AW	VMM AW 34+35
191022-0011_AW	VMM AW 38+39

- Analyse in enkelvoud
- Analyse in duplo na additie op dag 1
- Analyse in enkelvoud na additie op dag 2
- Addities: DW/GW/OW: 50 en 250 ng/l, AW: 100 en 500 ng/l

## PFAS GEHALTEN IN NIET-GEADDEERDE STALEN

ID	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTTrDA	PFTeDA	PFHxDA	PFODA
PBL	9	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0002_DW Vito	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0003_GW Lommel	7	<5	<5	<5	11	<5	<5	<5	<5	<5	<5	<5	<5
191022-0004_GW Olen	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0005_OW	10	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0006_OW	7	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0007_AW	<5	<5	<5	<5	<b>52</b>	<5	<5	<5	<5	<5	<5	<5	<5
191022-0008_AW	<b>13</b>	<b>12</b>	<b>14</b>	6	8	<5	<5	<5	<5	<5	<5	<5	<5
191022-0009_AW	<b>13</b>	10	<b>13</b>	<5	7	<5	<5	<5	<5	<5	<5	<5	<5
191022-00010_AW	<5	10	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-00011_AW	9	<5	<b>11</b>	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5

## PFAS GEHALTEN IN NIET-GEADDEERDE STALEN (vervolg)

ID	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
PBL	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0002_DW Vito	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0003_GW Lommel	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0004_GW Olen	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
191022-0005_OW	<5	<5	<5	<5	<5	<5	<5	<5	<5	<b>14</b>	<5	<5
191022-0006_OW	<5	<5	<5	<5	6	<5	<5	<5	<5	<5	<5	<5
191022-0007_AW	6	<5	<5	<5	<5	<5	<5	<5	<5	8	<5	<5
191022-0008_AW	7	<5	9	<5	<b>24</b>	<5	<5	<5	<5	<b>28</b>	<5	<5
191022-0009_AW	8	<5	8	<5	<b>16</b>	<5	<5	<5	<5	<b>12</b>	<5	<5
191022-0010_AW	<5	<5	<5	<5	<5	<5	<5	<5	<5	9	<5	<5
191022-0011_AW	<5	<5	<5	<5	<5	<5	<5	<5	<5	8	<5	<5

## PFAS GEHALTEN IN NIET-GEADDEERDE STALEN (vervolg)

ID	FOSA	MeFOSA	EtFOSA	FOSAA	MeFOSAA	EtFOSAA	6:2 PAP	8:2 PAP	6:2 diPAP	6:2/8:2 diPAP	8:2 diPAP	HFPO-DA
PBL	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0002_DW Vito	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0003_GW Lommel	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0004_GW Olen	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0005_OW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0006_OW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0007_AW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0008_AW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0009_AW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0010_AW	<5	<5	<5	-	<5	<5	-	-	<5	<5	<5	<5
191022-0011_AW	<5	<5	<5	-	9	<5	-	-	<5	<5	<5	<5

# HERHAALBAARHEDEN VOOR PFAS IN DW/GW/OW (50, 250 ng/l) EN AW (100, 500 ng/l)

## PFCAs

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDaA	PFTTrDA	PFTeDA	PFHxDA	PFODA
Herhaalbaarheid OW/GW/DW (RSD%) - 50 ng/l	2.2	2.5	6.9	2.5	6.4	9.3	13.0	14.4	14.3	20.0	14.0	22.7	29.2
Herhaalbaarheid OW/GW/DW (RSD%) - 250 ng/l	4.1	4.4	5.2	6.3	10.9	7.7	11.9	7.2	18.5	31.0	6.9	8.8	72.7
Herhaalbaarheid AW (RSD%) - 100 ng/l	5.4	4.7	3.7	7.2	10.9	11.3	8.5	13.3	16.3	22.3	13.4	8.5	56.1
Herhaalbaarheid AW (RSD%) - 500 ng/l	3.7	4.6	3.2	4.0	3.8	7.5	15.6	17.4	7.6	26.4	18.6	13.0	57.3

## PFSAs

	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
Herhaalbaarheid OW/GW/DW (RSD%) - 50 ng/l	2.4	1.1	1.6	1.2	2.6	3.1	8.5	28.7	2.6	1.9	2.3	10.2
Herhaalbaarheid OW/GW/DW (RSD%) - 250 ng/l	7.5	6.4	4.2	5.6	6.0	18.7	13.2	80.6	1.8	4.3	9.5	71.6
Herhaalbaarheid AW (RSD%) - 100 ng/l	3.6	5.2	6.8	5.4	2.5	10.2	18.3	39.0	6.4	12.6	10.0	24.7
Herhaalbaarheid AW (RSD%) - 500 ng/l	5.5	5.2	4.4	3.9	3.7	7.3	18.7	22.5	2.4	3.5	5.0	17.3

## Andere PFAS

	FOSA	MeFOSA	EtFOSA	MeFOSAA	EtFOSAA	6:2 diPAP	6:2/8:2 diPAP	8:2 diPAP	HFPO-DA
Herhaalbaarheid OW/GW/DW (RSD%) - 50 ng/l	3.5	22.7	26.8	4.4	7.0	4.5	44.1	57.0	10.9
Herhaalbaarheid OW/GW/DW (RSD%) - 250 ng/l	5.7	121.5	124.5	10.2	9.5	7.6	80.8	91.6	3.3
Herhaalbaarheid AW (RSD%) - 100 ng/l	4.9	43.8	44.7	8.1	12.5	5.3	18.2	62.3	7.8
Herhaalbaarheid AW (RSD%) - 500 ng/l	6.5	24.7	26.0	7.9	10.9	5.1	43.1	45.9	5.5

# REPRODUCEERBAARH. VOOR PFAS IN DW/GW/OW (50, 250 ng/l) EN AW (100, 500 ng/l)

## PFCAs

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTTrDA	PFTeDA	PFHxDA	PFODA
Reproduceerbaarheid OW/GW/DW (RSD%) - 50 ng/l	12.2	13.4	10.8	2.3	8.6	5.5	15.0	11.4	14.5	15.2	13.0	16.3	31.7
Reproduceerbaarheid OW/GW/DW (RSD%) - 250 ng/l	18.7	19.0	8.6	7.1	11.3	14.7	13.1	10.3	18.7	20.8	15.0	15.0	60.2
Reproduceerbaarheid AW (RSD%) - 100 ng/l	16.8	17.7	15.3	6.5	6.4	6.9	11.8	16.1	16.6	28.0	16.3	14.3	33.5
Reproduceerbaarheid AW (RSD%) - 500 ng/l	19.5	22.6	8.8	7.4	13.8	10.1	15.5	10.1	16.5	33.4	17.8	17.2	71.9

## PFASs

	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
Reproduceerbaarheid OW/GW/DW (RSD%) - 50 ng/l	9.6	9.3	4.4	7.0	8.4	6.7	21.4	49.3	6.3	8.1	6.7	14.8
Reproduceerbaarheid OW/GW/DW (RSD%) - 250 ng/l	4.5	4.5	4.5	6.8	8.5	7.3	12.5	46.9	4.3	6.7	11.3	17.3
Reproduceerbaarheid AW (RSD%) - 100 ng/l	5.3	6.6	5.1	7.4	7.1	9.5	16.8	43.9	8.4	10.3	5.6	14.8
Reproduceerbaarheid AW (RSD%) - 500 ng/l	6.9	7.0	4.5	6.7	4.4	9.5	22.6	70.1	8.4	7.7	8.6	22.4

## Andere PFAS

	FOSA	MeFOSA	EtFOSA	MeFOSAA	EtFOSAA	6:2 diPAP	6:2/8:2 diPAP	8:2 diPAP	HFPO-DA
Reproduceerbaarheid OW/GW/DW (RSD%) - 50 ng/l	7.5	31.5	32.8	12.7	14.5	8.8	29.0	45.2	16.7
Reproduceerbaarheid OW/GW/DW (RSD%) - 250 ng/l	7.6	52.0	52.0	13.8	13.0	5.6	59.0	89.9	23.9
Reproduceerbaarheid AW (RSD%) - 100 ng/l	5.9	66.1	69.1	10.0	12.8	6.9	24.5	27.8	12.9
Reproduceerbaarheid AW (RSD%) - 500 ng/l	5.1	23.0	30.5	24.0	29.7	5.0	23.1	62.7	16.1



## TERUGVINDINGEN VOOR DW/GW/OW (dag 2)

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTrDA	PFTeDA	PFHxDA	PFODA
191022-0002_DW Vito_50ng/l_dag2	95%	103%	102%	98%	93%	94%	108%	98%	127%	82%	85%	89%	309%
191022-0003_GW Lommel_50ng/l_dag2	134%	106%	105%	92%	91%	106%	106%	114%	110%	71%	94%	139%	206%
191022-0004_GW Olen_50ng/l_dag2	99%	110%	104%	105%	94%	110%	114%	113%	115%	89%	105%	116%	240%
191022-0005_OW_50ng/l_dag2	101%	118%	107%	101%	88%	110%	101%	93%	95%	78%	117%	111%	103%
191022-0006_OW_50ng/l_dag2	85%	90%	86%	93%	70%	96%	108%	80%	106%	100%	101%	103%	156%
191022-0002_DW Vito_250ng/l_dag2	105%	105%	108%	101%	105%	126%	132%	135%	134%	73%	155%	97%	57%
191022-0003_GW Lommel_250ng/l_dag2	108%	96%	108%	96%	76%	88%	107%	102%	143%	82%	118%	110%	199%
191022-0004_GW Olen_250ng/l_dag2	88%	92%	104%	93%	89%	92%	107%	110%	116%	121%	101%	118%	248%
191022-0005_OW_250ng/l_dag2	102%	100%	94%	97%	92%	92%	102%	114%	112%	103%	105%	84%	154%
191022-0006_OW_250ng/l_dag2	99%	104%	90%	93%	92%	109%	132%	97%	137%	112%	105%	105%	227%
Gemiddelde terugv. voor OW/GW/DW	102%	102%	101%	97%	89%	102%	112%	106%	120%	91%	109%	107%	190%

## TERUGVINDINGEN VOOR DW/GW/OW (dag 2)(vervolg)

PFSA's	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
191022-0002_DW Vito_50ng/l_dag2	107%	100%	92%	80%	104%	78%	58%	23%	94%	116%	102%	79%
191022-0003_GW Lommel_50ng/l_dag2	113%	104%	101%	87%	107%	74%	54%	18%	134%	99%	102%	66%
191022-0004_GW Olen_50ng/l_dag2	113%	109%	100%	89%	123%	87%	65%	21%	111%	101%	104%	77%
191022-0005_OW_50ng/l_dag2	115%	104%	98%	86%	107%	78%	58%	27%	136%	83%	98%	83%
191022-0006_OW_50ng/l_dag2	87%	84%	85%	78%	85%	78%	72%	40%	98%	83%	88%	79%
191022-0002_DW Vito_250ng/l_dag2	110%	105%	103%	95%	126%	99%	73%	18%	103%	95%	91%	61%
191022-0003_GW Lommel_250ng/l_dag2	95%	92%	93%	90%	93%	81%	68%	21%	125%	86%	82%	61%
191022-0004_GW Olen_250ng/l_dag2	96%	92%	94%	90%	103%	99%	94%	68%	94%	87%	86%	83%
191022-0005_OW_250ng/l_dag2	109%	105%	98%	95%	98%	94%	90%	77%	130%	87%	86%	92%
191022-0006_OW_250ng/l_dag2	100%	92%	93%	95%	96%	90%	86%	59%	98%	87%	82%	83%
Gemiddelde terugv. voor OW/GW/DW	104%	99%	96%	88%	104%	86%	72%	37%	112%	92%	92%	76%

## TERUGVINDINGEN VOOR DW/GW/OW (dag 2)(vervolg)

<b>Andere PFAS</b>	<b>FOSA</b>	<b>MeFOSA</b>	<b>EtFOSA</b>	<b>MeFOSAA</b>	<b>EtFOSAA</b>	<b>6:2 diPAP</b>	<b>6:2/8:2 diPAP</b>	<b>8:2 diPAP</b>	<b>HFPO-DA</b>
191022-0002_DW Vito_50ng/l_dag2	107%	115%	99%	85%	80%	94%	64%	63%	105%
191022-0003_GW Lommel_50ng/l_dag2	103%	104%	92%	80%	73%	103%	34%	35%	112%
191022-0004_GW Olen_50ng/l_dag2	109%	106%	89%	96%	95%	112%	78%	52%	131%
191022-0005_OW_50ng/l_dag2	107%	54%	51%	111%	104%	99%	106%	168%	109%
191022-0006_OW_50ng/l_dag2	90%	93%	85%	85%	88%	88%	66%	76%	96%
191022-0002_DW Vito_250ng/l_dag2	129%	9%	6%	150%	144%	113%	-	-	112%
191022-0003_GW Lommel_250ng/l_dag2	102%	66%	55%	90%	82%	96%	58%	197%	99%
191022-0004_GW Olen_250ng/l_dag2	102%	197%	193%	99%	103%	100%	90%	53%	112%
191022-0005_OW_250ng/l_dag2	98%	24%	26%	133%	140%	105%	103%	110%	116%
191022-0006_OW_250ng/l_dag2	98%	92%	88%	99%	103%	105%	81%	74%	99%
<b>Gemiddelde terugv. voor OW/GW/DW</b>	<b>104%</b>	<b>86%</b>	<b>79%</b>	<b>103%</b>	<b>101%</b>	<b>102%</b>	<b>76%</b>	<b>92%</b>	<b>109%</b>

## TERUGVINDINGEN VOOR AW (dag 2)

PFCAs	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTTrDA	PFTeDA	PFHxDA	PFODA
191022-0007_AW_100ng/l_dag2	104%	115%	103%	98%	87%	97%	99%	107%	80%	78%	115%	98%	334%
191022-0008_AW_100ng/l_dag2	105%	95%	119%	102%	82%	115%	99%	98%	114%	118%	78%	98%	232%
191022-0009_AW_100ng/l_dag2	87%	106%	103%	95%	92%	115%	108%	125%	114%	118%	88%	107%	206%
191022-00010_AW_100ng/l_dag2	113%	106%	111%	98%	98%	115%	117%	107%	114%	91%	115%	125%	240%
191022-00011_AW_100ng/l_dag2	127%	115%	122%	125%	86%	106%	108%	134%	132%	109%	98%	116%	129%
191022-0007_AW_500ng/l_dag2	95%	96%	95%	91%	95%	90%	134%	110%	81%	76%	136%	131%	384%
191022-0008_AW_500ng/l_dag2	101%	94%	87%	86%	92%	111%	119%	82%	83%	87%	108%	99%	201%
191022-0009_AW_500ng/l_dag2	90%	96%	99%	102%	82%	111%	111%	101%	93%	80%	95%	91%	130%
191022-00010_AW_500ng/l_dag2	85%	92%	93%	82%	68%	90%	90%	84%	123%	111%	95%	80%	152%
191022-00011_AW_500ng/l_dag2	102%	100%	106%	105%	66%	94%	94%	105%	109%	85%	100%	131%	58%
Gemiddelde terugvinding voor AW	101%	102%	104%	99%	85%	105%	108%	105%	104%	95%	103%	108%	206%

## TERUGVINDINGEN VOOR AW (dag 2)(vervolg)

PFSA's	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
191022-0007_AW_100ng/l_dag2	95%	93%	94%	91%	95%	89%	89%	65%	123%	90%	109%	120%
191022-0008_AW_100ng/l_dag2	92%	93%	96%	92%	91%	95%	90%	80%	123%	91%	109%	120%
191022-0009_AW_100ng/l_dag2	104%	102%	96%	95%	99%	95%	99%	80%	104%	96%	109%	111%
191022-00010_AW_100ng/l_dag2	101%	102%	104%	105%	114%	105%	108%	77%	217%	107%	109%	111%
191022-00011_AW_100ng/l_dag2	101%	111%	113%	105%	123%	95%	85%	54%	91%	144%	119%	111%
191022-0007_AW_500ng/l_dag2	96%	93%	94%	91%	97%	93%	92%	74%	113%	85%	87%	94%
191022-0008_AW_500ng/l_dag2	89%	91%	91%	91%	92%	91%	92%	80%	115%	82%	80%	90%
191022-0009_AW_500ng/l_dag2	95%	93%	93%	93%	96%	91%	94%	88%	91%	87%	83%	96%
191022-00010_AW_500ng/l_dag2	88%	85%	86%	83%	87%	83%	83%	65%	165%	80%	74%	77%
191022-00011_AW_500ng/l_dag2	80%	87%	88%	89%	91%	83%	77%	35%	68%	72%	91%	88%
Gemiddelde terugvinding voor AW	94%	95%	96%	94%	98%	92%	91%	70%	121%	93%	97%	102%

## TERUGVINDINGEN VOOR AW (dag 2)(vervolg)

Andere PFAS	FOSA	MeFOSA	EtFOSA	MeFOSAA	EtFOSAA	6:2 diPAP	6:2/8:2 diPAP	8:2 diPAP	HFPO-DA
191022-0007_AW_100ng/l_dag2	94%	111%	98%	100%	113%	92%	85%	59%	136%
191022-0008_AW_100ng/l_dag2	94%	121%	124%	127%	130%	92%	93%	102%	109%
191022-0009_AW_100ng/l_dag2	103%	158%	160%	127%	139%	101%	89%	111%	127%
191022-00010_AW_100ng/l_dag2	113%	167%	169%	172%	182%	92%	114%	167%	136%
191022-00011_AW_100ng/l_dag2	103%	148%	204%	146%	191%	120%	53%	65%	100%
191022-0007_AW_500ng/l_dag2	96%	28%	30%	124%	131%	100%	77%	43%	118%
191022-0008_AW_500ng/l_dag2	94%	28%	34%	191%	203%	94%	105%	116%	104%
191022-0009_AW_500ng/l_dag2	100%	32%	42%	715%	757%	94%	111%	144%	110%
191022-00010_AW_500ng/l_dag2	92%	16%	21%	290%	296%	88%	99%	168%	97%
191022-00011_AW_500ng/l_dag2	88%	61%	78%	133%	172%	96%	34%	35%	85%
Gemiddelde terugvinding voor AW	98%	87%	96%	212%	231%	97%	86%	101%	112%

Kwantificatie tov.  
13C-FOSA

## TERUGVINDING IS (250 ng/l DW/GW/OW en 500 ng/L AW op dag 2)

ID	13C-PFBA	13C-PFPeA	13C-PFHxA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PUdA	13C-PFDoA	13C-PFTeDA
CAL-PFC-255	103%	102%	101%	111%	108%	95%	125%	102%	119%
Procedureblanco	92%	94%	88%	88%	84%	83%	60%	53%	18%
191022-0002_DW Vito_250ng/l_dag2	96%	101%	90%	77%	70%	58%	45%	59%	28%
191022-0003_GW Lommel_250ng/l_dag2	61%	80%	76%	83%	73%	61%	54%	60%	21%
191022-0004_GW Olen_250ng/l_dag2	87%	84%	77%	80%	77%	61%	55%	56%	43%
191022-0005_OW_250ng/l_dag2	76%	86%	81%	83%	81%	73%	75%	67%	48%
191022-0006_OW_250ng/l_dag2	72%	74%	73%	78%	64%	65%	65%	39%	35%
191022-0007_AW_500ng/l_dag2	80%	83%	72%	74%	72%	62%	61%	77%	38%
191022-0008_AW_500ng/l_dag2	34%	56%	64%	67%	60%	60%	67%	76%	43%
191022-0009_AW_500ng/l_dag2	87%	83%	73%	79%	69%	66%	71%	79%	67%
191022-00010_AW_500ng/l_dag2	102%	95%	81%	95%	77%	76%	77%	63%	56%
191022-00011_AW_500ng/l_dag2	45%	85%	47%	73%	55%	46%	48%	37%	20%
CAL-PFC-255	85%	81%	126%	120%	98%	127%	100%	122%	110%

## TERUGVINDING IS (250 ng/l DW/GW/OW en 500 ng/L AW op dag 2) (vervolg)

ID	13C-PFHxDA	13C-PFHxS	13C-PFOS	13C-6:2FTS	13C-PFOSA	13C-6:2 PAP	13C-6:2 diPAP	13C-HFPO-DA
CAL-PFC-255	87%	104%	103%	101%	99%	-	96%	98%
Procedureblanco	19%	86%	72%	96%	83%	-	29%	106%
191022-0002_DW Vito_250ng/l_dag2	46%	97%	69%	95%	52%	-	17%	114%
191022-0003_GW Lommel_250ng/l_dag2	14%	103%	90%	89%	64%	-	12%	109%
191022-0004_GW Olen_250ng/l_dag2	25%	94%	81%	86%	80%	-	70%	97%
191022-0005_OW_250ng/l_dag2	56%	91%	89%	81%	59%	-	67%	101%
191022-0006_OW_250ng/l_dag2	40%	97%	92%	90%	69%	-	65%	86%
191022-0007_AW_500ng/l_dag2	24%	95%	92%	86%	64%	-	73%	91%
191022-0008_AW_500ng/l_dag2	43%	103%	101%	89%	40%	-	120%	99%
191022-0009_AW_500ng/l_dag2	40%	107%	104%	91%	23%	-	125%	101%
191022-00010_AW_500ng/l_dag2	43%	112%	109%	103%	28%	-	88%	107%
191022-00011_AW_500ng/l_dag2	18%	98%	99%	103%	65%	-	144%	73%
CAL-PFC-255	97%	106%	107%	97%	100%	-	127%	101%



## TERUGVINDINGEN EN HERHAALBH. VOOR DW/GW/OW (250 ng/l) EN AW (500 ng/l) MENGSTALEN - ALKALISCHE MOBIELE FASE

### PFCAs

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTrDA	PFTeDA	PFHxDA	PFODA
Mengmonster DW/GW/OW_1	87%	91%	91%	83%	90%	87%	94%	85%	101%	53%	115%	96%	81%
Mengmonster DW/GW/OW_2	103%	103%	103%	95%	106%	95%	118%	110%	133%	69%	159%	129%	240%
Mengmonster DW/GW/OW_3	95%	91%	95%	95%	94%	99%	106%	89%	121%	73%	119%	125%	240%
Mengmonster DW/GW/OW_4	99%	103%	99%	87%	102%	103%	118%	93%	125%	93%	167%	153%	225%
Mengmonster DW/GW/OW_5	103%	103%	103%	99%	102%	103%	127%	106%	121%	61%	155%	145%	267%
Mengmonster AW_1	95%	99%	97%	97%	95%	105%	116%	108%	111%	71%	153%	135%	232%
Mengmonster AW_2	104%	99%	115%	105%	105%	111%	116%	114%	153%	93%	155%	129%	329%
Mengmonster AW_3	106%	99%	109%	101%	101%	107%	129%	104%	137%	124%	125%	147%	232%
Mengmonster AW_4	104%	103%	107%	109%	95%	103%	125%	108%	121%	89%	109%	157%	368%
Mengmonster AW_5	104%	103%	109%	105%	103%	115%	129%	110%	95%	73%	137%	135%	213%
Gemidd. terugvind. OW/GW/DW (%)	97%	98%	98%	92%	99%	97%	113%	97%	120%	70%	143%	129%	211%
Herhaalbaarheid (RSD%)	6.8%	6.6%	5.3%	7.1%	6.6%	6.8%	11.3%	10.9%	9.9%	21.9%	16.8%	16.8%	35.1%
Gemidd. terugvind. AW (%)	103%	100%	108%	103%	100%	108%	123%	109%	123%	90%	136%	140%	275%
Herhaalbaarheid (RSD%)	4.3%	2.3%	7.5%	7.2%	4.3%	3.5%	4.0%	7.9%	14.7%	18.1%	4.7%	7.6%	17.3%

## TERUGVINDINGEN EN HERHAALBH. VOOR DW/GW/OW (250 ng/l) EN AW (500 ng/l) MENGSTALEN - ALKALISCHE MOBIELE FASE

### PFSAs

	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
Mengmonster DW/GW/OW_1	104%	97%	89%	81%	89%	77%	38%	12%	81%	84%	62%	21%
Mengmonster DW/GW/OW_2	122%	119%	102%	94%	102%	77%	52%	17%	94%	96%	75%	28%
Mengmonster DW/GW/OW_3	113%	110%	98%	90%	98%	77%	52%	23%	85%	88%	75%	33%
Mengmonster DW/GW/OW_4	113%	110%	98%	90%	107%	73%	48%	19%	94%	92%	66%	27%
Mengmonster DW/GW/OW_5	117%	110%	102%	90%	102%	73%	48%	18%	85%	96%	66%	23%
Mengmonster AW_1	94%	95%	93%	90%	102%	82%	54%	33%	117%	88%	75%	44%
Mengmonster AW_2	106%	108%	103%	99%	113%	86%	60%	28%	119%	94%	72%	44%
Mengmonster AW_3	110%	112%	110%	105%	113%	86%	54%	39%	128%	96%	77%	38%
Mengmonster AW_4	99%	102%	99%	96%	111%	86%	60%	30%	119%	90%	70%	42%
Mengmonster AW_5	108%	108%	106%	99%	113%	86%	60%	33%	123%	94%	70%	46%
Gemidd. terugvind. OW/GW/DW (%)	114%	109%	98%	89%	100%	76%	48%	18%	88%	91%	69%	26%
Herhaalbaarheid (RSD%)	5.9%	6.9%	5.3%	5.3%	6.6%	3.1%	12.4%	21.9%	6.5%	5.7%	8.1%	16.8%
Gemidd. terugvind. AW (%)	103%	105%	102%	98%	110%	85%	58%	33%	121%	92%	73%	43%
Herhaalbaarheid (RSD%)	9.6%	6.8%	4.7%	4.4%	4.6%	7.6%	10.0%	21.5%	14.0%	3.7%	5.9%	26.0%

# TERUGVINDINGEN EN HERHAALBH. VOOR DW/GW/OW (250 ng/l) EN AW (500 ng/l) MENGSTALEN - ALKALISCHE MOBIELE FASE

## Andere PFAS

	FOSA	MeFOSA	EtFOSA	FOSAA	MeFOSAA	EtFOSAA	6:2 PAP	8:2 PAP	6:2 diPAP	:2/8:2 diPA	8:2 diPAP	HFPO-DA	ADONA	PFECHS
Mengmonster DW/GW/OW_1	85%	46%	52%	95%	98%	70%	70%	35%	126%	370%	877%	82%	78%	79%
Mengmonster DW/GW/OW_2	111%	41%	35%	119%	115%	94%	92%	44%	150%	217%	751%	123%	99%	92%
Mengmonster DW/GW/OW_3	94%	28%	25%	107%	111%	90%	84%	43%	130%	212%	668%	98%	82%	84%
Mengmonster DW/GW/OW_4	102%	54%	48%	111%	115%	94%	88%	44%	159%	280%	960%	102%	91%	84%
Mengmonster DW/GW/OW_5	106%	46%	40%	119%	115%	90%	88%	48%	126%	204%	710%	110%	95%	84%
Mengmonster AW_1	98%	56%	56%	127%	110%	96%	79%	35%	128%	155%	355%	84%	82%	94%
Mengmonster AW_2	109%	79%	81%	135%	112%	103%	86%	40%	175%	191%	501%	88%	91%	101%
Mengmonster AW_3	123%	94%	105%	157%	133%	113%	88%	40%	132%	140%	292%	96%	95%	105%
Mengmonster AW_4	106%	64%	66%	137%	121%	107%	86%	40%	134%	119%	271%	86%	86%	101%
Mengmonster AW_5	109%	54%	60%	131%	118%	102%	90%	46%	136%	153%	334%	98%	82%	101%
Gemidd. terugvind. OW/GW/DW (%)	100%	43%	40%	110%	111%	87%	85%	43%	138%	257%	793%	103%	89%	85%
Herhaalbaarheid (RSD%)	10.3%	22.5%	26.6%	9.0%	6.4%	11.2%	10.0%	11.6%	11.0%	27.3%	15.3%	14.7%	10.1%	5.7%
Gemidd. terugvind. AW (%)	109%	69%	73%	137%	119%	104%	86%	40%	141%	151%	351%	90%	87%	101%
Herhaalbaarheid (RSD%)	4.3%	20.5%	24.6%	7.5%	1.8%	5.5%	4.9%	14.2%	16.9%	35.0%	75.0%	13.7%	6.2%	8.5%

tov 13C-MeFOSAA

## GESCHATTE LOQ-WAARDEN

- Obv laagste kalibratiestandaard, rekening houdend met intake en terugvindingen IS

	intake 50 ml		intake 25	
	DW/GW/OW		AW	
	LOD ng/l	LOQ ng/l	LOD ng/l	LOQ ng/l
PFBA	1	3	3	6
PFPeA	1	3	3	6
PFHxA	1	3	3	6
PFHpA	1	3	3	6
PFOA	1	3	3	6
PFNA	1	3	3	6
PFDA	2	3	3	7
PFUdA	2	4	4	8
PFDoA	2	4	4	8
PFTTrDA	3	5	5	10
PFTeDA	3	5	5	10
PFHxDA	3	7	7	13
PFODA	3	7	7	13
PFBS	1	3	3	5
PFPeS	1	3	3	5
PFHxS	1	3	3	5

	intake 50 ml		intake 25	
	DW/GW/OW		AW	
	LOD ng/l	LOQ ng/l	LOD ng/l	LOQ ng/l
PFNS	1	3	3	5
PFDS	1	3	3	5
PFDoS	1	3	3	5
4:2 FTS	1	3	3	5
6:2 FTS	1	3	3	5
8:2 FTS	1	3	3	5
10:2 FTS	1	3	3	5
FOSA	2	3	3	7
MeFOSA	2	3	3	7
EtFOSA	2	3	3	7
MeFOSAA	2	3	3	7
EtFOSAA	2	3	3	7
6:2 diPAP	2	3	3	7
6:2/8:2 diPAP	2	3	3	7
8:2 diPAP	2	3	3	7
HFPO-DA	1	2	2	4

## TOTAL OXIDISABLE PRECURSOR ASSAY (TOPA)

- Werkwijze cfr Houtz en Sedlak (Water Research 95:142-149, 2016)
  - Breng 50 ml waterstaal in PP centrifuge buis
  - Voeg 0.85 g kaliumpersulfaat toe (= 61,7 mM)
  - Voeg 1 ml NaOH 7,65M toe (= 150 mM)
  - Laat overnacht bij 85°C reageren in een geventileerde oven
  - Neutraliseer het mengsel met HCl 5N tot pH 5-9
  - Neem 500 µl van bovenstaande vloeistof, voeg 500 µl MeOH (0,2% HAc) toe
  - Addeer IS en injecteer rechtstreeks in een LC-MS/MS
- De methode werd in 3-voud uitgetest op ultrapuur water met PFAS additie 250 ng/l, gebruikmakend van warmwaterbad resp. oven
- Vaststelling: afname conc. MeFOSA, EtFOSA, diPAPs  
Echter geen toename conc. andere PFAS

## RESULTATEN VERKENNENDE TEST TOPA

### ■ PFCAs

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTrDA	PFTeDA	PFHxDA	PFODA
	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l
geaddeerde concentratie	261	266	267	263	263	265	260	263	248	258	264	263	274
TOPA_voor	258	265	251	268	270	258	232	243	183	218	160	259	299
Terugvinning additie (%)	99	100	94	102	103	97	89	92	74	85	61	98	109
TOPA_na_1 WWB	282	294	274	277	329	277	225	212	227	208	167	225	715
TOPA_na_2 WWB	255	264	275	273	298	255	224	259	207	205	144	165	175
TOPA_na_3 WWB	254	263	254	243	286	217	246	282	253	262	134	104	95
Gemiddelde	264	273	268	264	305	250	232	251	229	225	148	165	328
%RSD	6.1	6.4	4.4	7.0	7.3	12.1	5.4	14.3	10.0	14.1	11.4	36.9	102.7
Terugvinning additie (%)	101	103	100	101	116	94	89	95	92	87	56	63	120
TOPA_na_1 oven	242	260	249	259	229	225	217	232	227	202	159	122	234
TOPA_na_2 oven	241	233	251	240	272	229	213	236	213	229	137	104	169
TOPA_na_3 oven	239	255	242	224	262	227	173	187	181	164	101	63	186
Gemiddelde	241	250	248	241	254	227	201	218	207	198	133	97	197
%RSD	0.7	5.7	2.0	7.1	9.0	0.9	12.1	12.6	11.4	16.3	22.1	31.1	17.0
Terugvinning additie (%)	92	94	93	92	97	86	77	83	83	77	50	37	72

## RESULTATEN VERKENNENDE TEST TOPA (vervolg)

### ■ PFSA's

	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFDoS	4:2 FTS	6:2 FTS	8:2 FTS	10:2 FTS
	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l
Geaddeerde concentratie	232	253	249	247	248	247	260	245	249	264	258	255
TOPA_voor	222	249	240	248	249	243	224	190	267	280	255	220
Terugvinding additie (%)	96	98	97	100	101	99	86	78	107	106	99	87
TOPA_na_1 WWB	275	285	279	270	235	221	219	126	325	288	236	186
TOPA_na_2 WWB	230	260	247	238	232	230	233	141	307	267	247	190
TOPA_na_3 WWB	235	260	251	242	230	230	237	149	302	263	237	226
Gemiddelde	247	268	259	250	232	227	230	139	311	273	240	201
%RSD	10.0	5.5	6.8	6.9	1.2	2.4	4.2	8.3	3.9	5.0	2.6	10.9
Terugvinding additie (%)	106	106	104	101	94	92	88	57	125	103	93	79
TOPA_na_1 oven	232	245	240	231	215	190	201	162	286	257	212	213
TOPA_na_2 oven	239	251	252	238	216	199	208	155	286	257	208	213
TOPA_na_3 oven	221	247	239	226	207	198	203	91	274	260	202	159
Gemiddelde	230	248	244	232	212	196	204	136	282	258	207	195
%RSD	4.0	1.2	3.0	2.6	2.4	2.5	1.7	28.9	2.5	0.6	2.3	15.8
Terugvinding additie (%)	99	98	98	94	86	79	79	56	114	98	80	77

## RESULTATEN VERKENNENDE TEST TOPA (vervolg)

### ▪ Andere PFAS

	FOSA	MeFOSA	EtFOSA	FOSAA	MeFOSAA	EtFOSAA	6:2 PAP	8:2 PAP	6:2 diPAP	6:2/8:2 diPAP	8:2 diPAP	HFPO-DA
	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l	ng/l
Geaddeerde concentratie	250	253	264	264	259	271	241	239	255	248	254	259
TOPA_voor	217	187	192	272	248	247	-	-	196	209	215	221
Terugvinning additie (%)	87	74	73	103	96	91	-	-	77	85	85	85
TOPA_na_1 WWB	237	41	7	199	227	251	-	-	171	44	29	254
TOPA_na_2 WWB	250	23	1	189	246	275	-	-	200	42	20	226
TOPA_na_3 WWB	240	4	0	187	241	259	-	-	171	51	21	235
Gemiddelde	242	23	3	192	238	262	-	-	181	46	24	238
%RSD	2.7	80.2	155.6	3.3	4.2	4.7	-	-	9.3	10.0	21.4	6.1
Terugvinning additie (%)	97	9	1	73	92	97	-	-	71	18	9	92
TOPA_na_1 oven	193	13	0	166	202	211	-	-	173	60	34	185
TOPA_na_2 oven	202	8	0	174	225	240	-	-	168	45	25	191
TOPA_na_3 oven	188	0	0	172	207	212	-	-	85	18	16	178
Gemiddelde	194	7	0	171	212	221	-	-	142	41	25	184
%RSD	3.7	94.2	-	2.3	5.7	7.3	-	-	34.5	52.0	34.3	3.5
Terugvinning additie (%)	78	3	0	65	82	82	-	-	56	17	10	71



## BESLUIT

- Geaddeerd UP water:
  - goede terugvindingen en RSDs voor meeste PFAS
  - behalve: PFTTrDA, PFODA, PFDS, PFDoS, 10:2 FTS, 8:2 PAP, 8:2 diPAP
- Geaddeerde DW/GW/OW en AW: matrixinvloeden
  - goede gemiddelde terugvindingen, behalve PFODA, MeFOSAA (AW) en EtFOSAA (AW)
  - afwijkende RSDs voor PFTTrDA, PFODA, PFDS, PFDoS, 10:2 FTS, MeFOSA, EtFOSA, 6:2/8:2 diPAP, 8:2 diPAP
- Alkalische gradient:
  - goede resultaten voor FOSAA en 6:2 PAP, 8:2 PAP te lage terugvinding
  - afwijkende terugvindingen voor PFTeDA, PFHxDA, PFODA, PFDS, PFDoS, 10:2 FTS, MeFOSA, EtFOSA, diPAPs
- Isotoop dilutie noodz. voor goede resultaten: echter kostprijs, beschikbaarheid  
Bijkomend aangekocht:  $^{13}\text{C}$ -MeFOSA,  $^{13}\text{C}$ -MeFOSAA

## BESLUIT VERVOLG

- Voorstel aanpassing WAC (*ontwerpversie nov. 2020*)
  - Kwantitatieve parameters: C4-C16 PFCAs, C4-C8 PFSAs, n:2 FTS (n = 4,6,8), FOSA, FOSAAs, 6:2 diPAP, HFPO-DA, ADONA, PFECBS
  - Indicatieve parameters: PFTrDA, PFODA, PFDS, PFDoS, 10:2 FTS, MeFOSA, EtFOSA, 6:2/8:2 diPAP, 8:2 diPAP
  - Optioneel (op vraag): FOSAA, 6:2 PAP, 8:2 PAP (alkalische mobiele fase)
  - Inwendige standaarden: verplichte lijst
  - *Opn.:* 8:2 diPAP, MeFOSA, EtFOSA kwantitatief met <sup>13</sup>C-8:2 diPAP en <sup>13</sup>C-MeFOSA als IS
  
- TOPA: voorlopig geen meerwaarde, verder uit te testen
  
- *Discussie ...*