

Ultra korte keten PFAS

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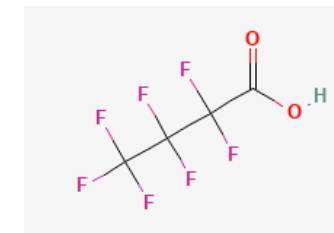
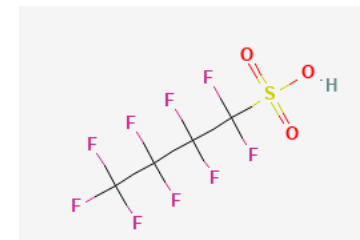
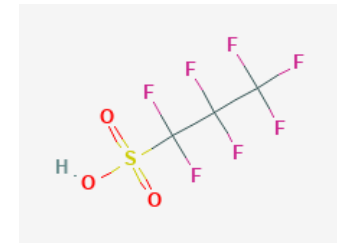
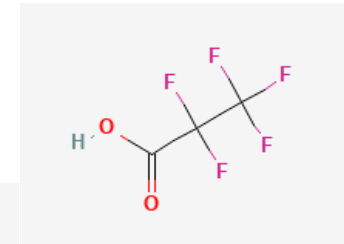
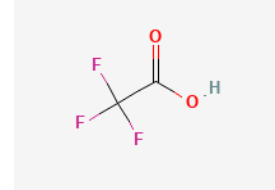
Werkgroep Water (OR) - 12/06/2023

Methode ontwikkeling ultra korte keten PFAS

- PFAS van C1 tot C4 (zie volgende slides voor structuren)
- Ontwikkeling van nieuwe methode
- Belangrijk: stockoplossingen en stalen moeten in CH_3CN gemaakt worden, met MeOH kan verestering gebeuren

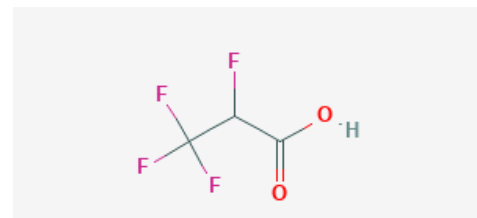
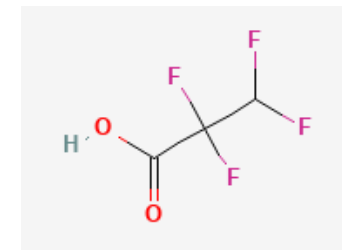
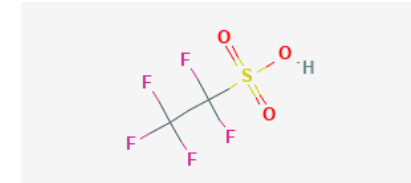
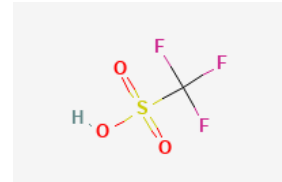
Ultra korte keten PFAS

- Trifluoroacetic acid TFA 76-05-1
- pentafluoropropionic acid PFPrA 422-64-0
- Perfluoropropanesulfonic acid PFPrS 423-41-6
- Perfluorobutanesulfonic acid PFBS 375-73-5
- perfluoro-n-butanoic acid PFBA 375-22-4



Ultra korte keten PFAS

- Trifluoromethanesulfonic acid TFMS 1493-13-6
- pentafluoroethanesulfonic acid PFES 354-88-1
- 2,3,3,3-tetrafluoropropanoic Acid 2,3,3,3-TFPA 756-09-2
- 2,2,3,3-tetrafluoropropanoic Acid 2,2,3,3-TFPA 359-49-9

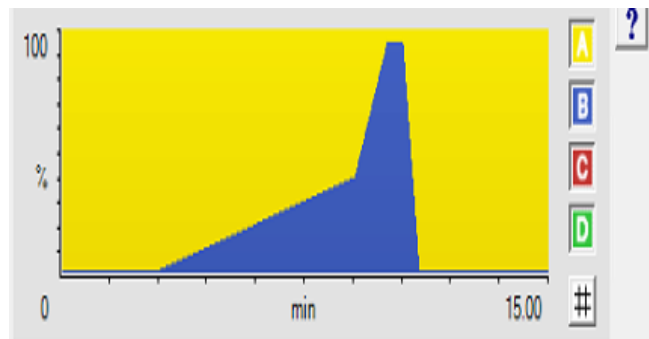


Parameters voor methode ontwikkeling

- Voorkeur voor reversed phase over Hilic
 - Robuustheid
 - Gebruiksgemak
- Geteste kolommen
 - Waters Atlantis T3 150 x 4.6 mm
 - Waters HSS T3
 - Prevail organic acid 150 x 4.6 mm
 - Atlantis premier c18AX
 - Te testen: Raptor Polar X (Hilic, ion exchange)
- Verschillende solventen en pH getest
- Optimalisatie van waters Xevo TQ-XS

Finale HPLC-MS/MS methode voor Ultra korte keten PFAS

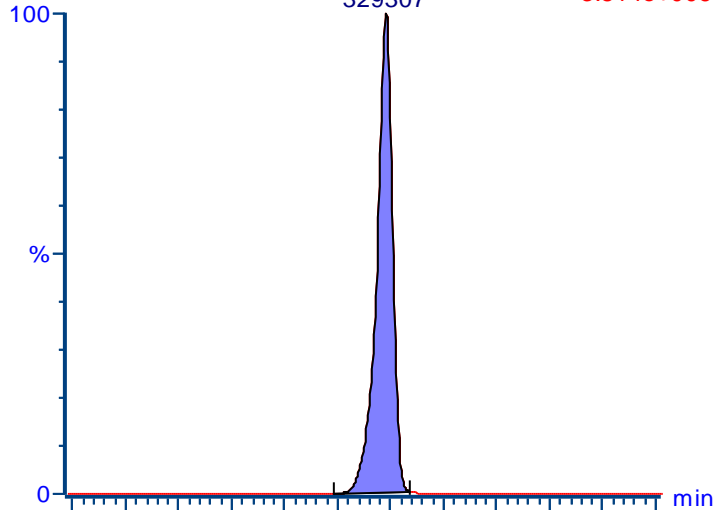
- Kolom: Prevail Organic acid 5u, 150 mm x 4.6 mm (De UPLC versie is in bestelling)
- Kolom temperatuur: 45 °C
- Staal temperatuur: 10 °C
- Injectievolume: 10 µl
- Mobiele fase
 - A: 10 mM NH₄Ac in MQ water pH = 5
 - B: CH₃CN
 - Strong wash: Water/MeOH/CH₃CN/ IPA 25/25/25/25
 - Weak wash: Water/MeOH 80/20
 - Seal wash: Water/MeOH 80/20
- Gradient



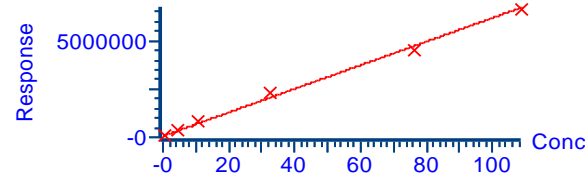
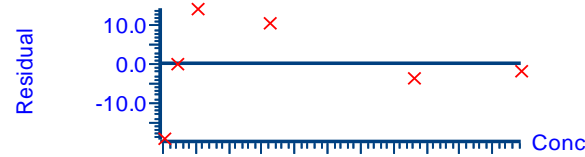
Time	Flow (ml/min)	A (%)	B (%)	Curve
Initial	0.6	98	2	Initial
3	0.6	98	2	6
9	0.6	60	40	6
10	0.6	5	95	6
10.5	0.6	5	95	6
11	0.6	98	2	6
15	0.6	98	2	6

Calibratie resultaten: PFES, TFMS

test1a020 Smooth(Mn,2x2) PFES F6:MRM of 2 channels,ES-cal05
 6.73 198.7 > 79.9
 329307 3.814e+006



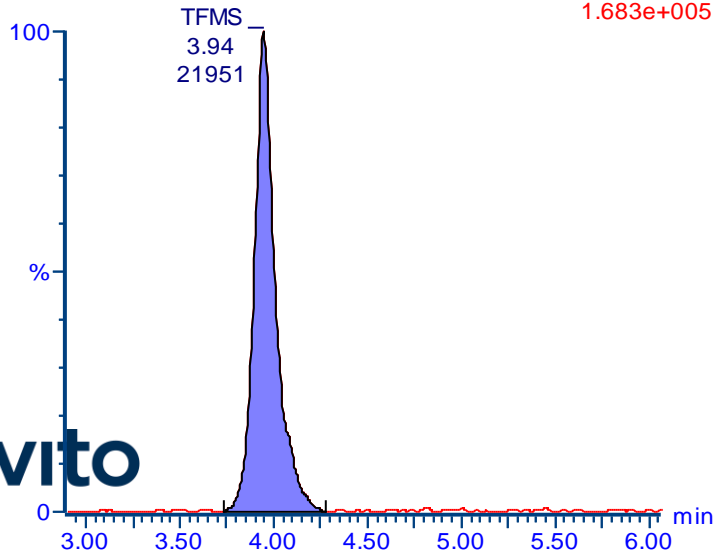
Compound name: PFES
 Correlation coefficient: $r = 0.998314$, $r^2 = 0.996632$
 Calibration curve: $61290 * x + 62125.3$
 Response type: External Std, Area
 Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



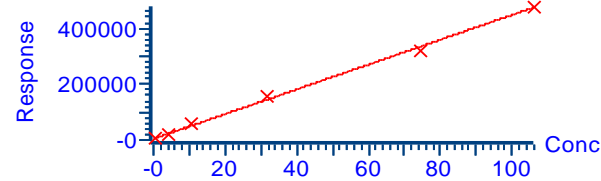
Laagst geteste standard: 0.3 µg/L

MRM: PFES: 199-80

test1a020 Smooth(Mn,2x2) TFMS 7.F4:MRM of 1 channel,ES-cal05
 3.94 149 > 80
 21951 1.683e+005



Compound name: TFMS
 Correlation coefficient: $r = 0.998531$, $r^2 = 0.997064$
 Calibration curve: $4437.63 * x + 3977.75$
 Response type: External Std, Area
 Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



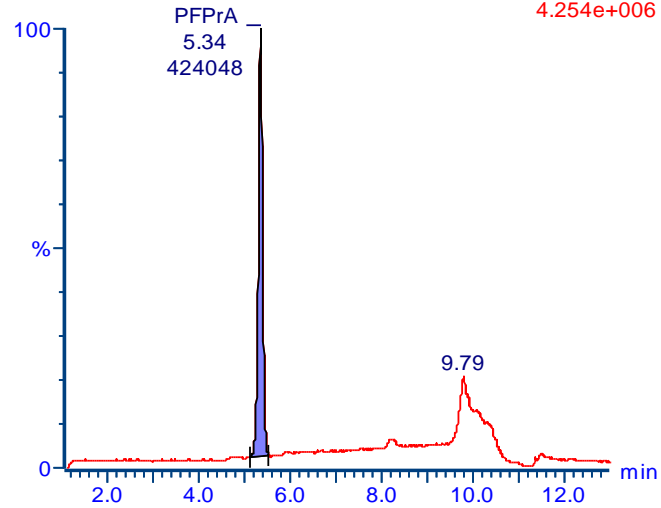
Laagst geteste standard: 0.3 µg/L

MRM: TFMS: 149-80

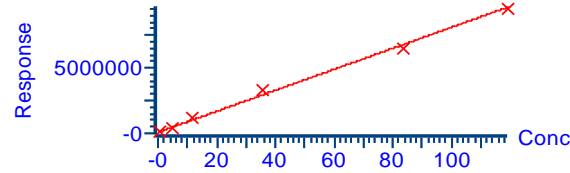
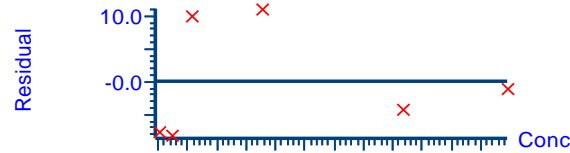
Calibratie resultaten: PFPrA, PFBA

test1a020 Smooth(Mn,2x2)
cal05

F5:MRM of 1 channel,ES-
163 > 119
4.254e+006



Compound name: PFPrA
Correlation coefficient: $r = 0.998458$, $r^2 = 0.996918$
Calibration curve: $80168.9 * x + 72806$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



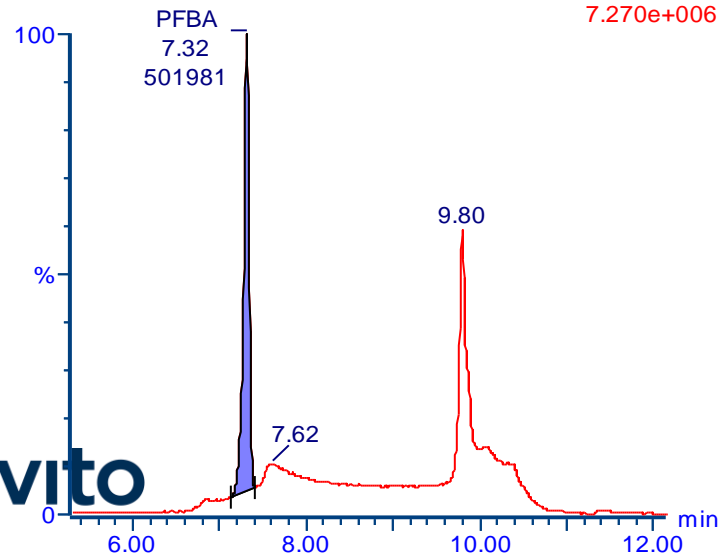
Laagst geteste standard: 0.3
µg/L

MRM: PFPrA: 163-119

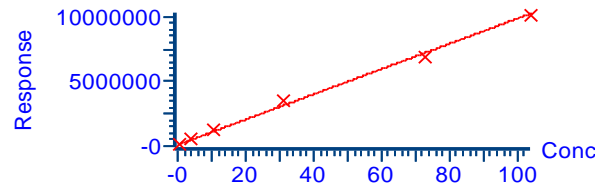
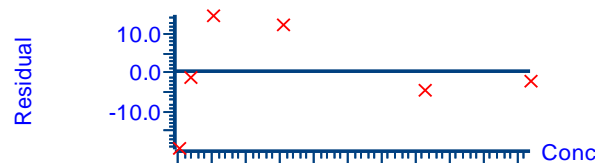
Piek 9.79 ook in procedure
blanco

test1a020 Smooth(Mn,2x2)
cal05

F7:MRM of 1 channel,ES-
213 > 169
7.270e+006



Compound name: PFBA
Correlation coefficient: $r = 0.997895$, $r^2 = 0.995795$
Calibration curve: $97608.6 * x + 100691$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



Laagst geteste standard: 0.3
µg/L

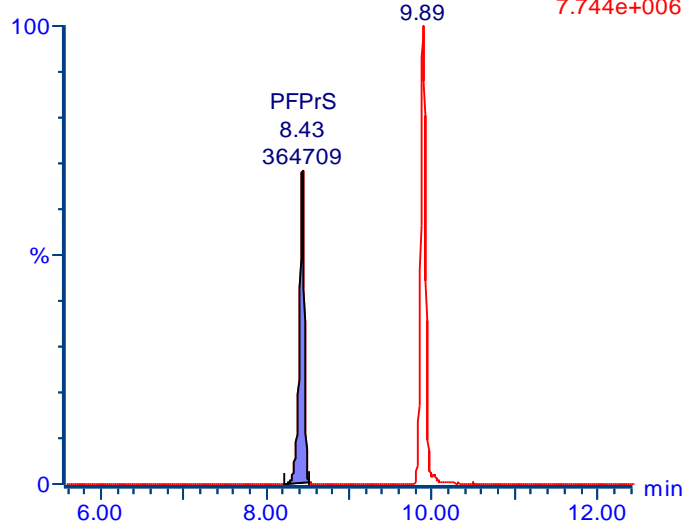
MRM: PFBA: 213-169

Piek 9.80 ook in procedure
blanco

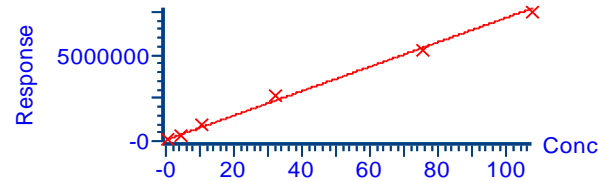
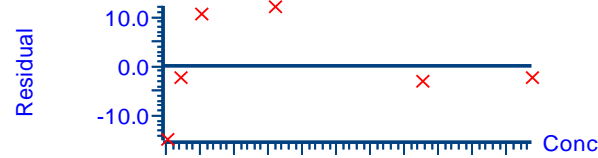
Calibratie resultaten: PFPrS, PFBS

test1a020 Smooth(Mn,2x2)
cal05

F8:MRM of 1 channel,ES-
249 > 80
7.744e+006



Compound name: PFPrS
Correlation coefficient: $r = 0.998321$, $r^2 = 0.996646$
Calibration curve: $70964 \cdot x + 66217.7$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



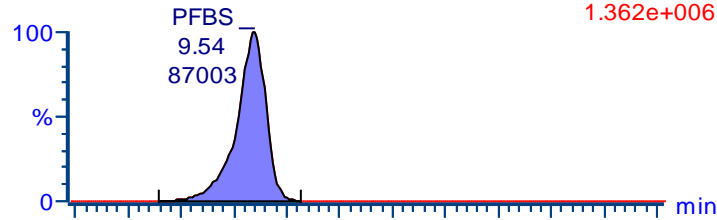
Laagst geteste standard: 0.3 $\mu\text{g/L}$

MRM: PFPrS: 249-80

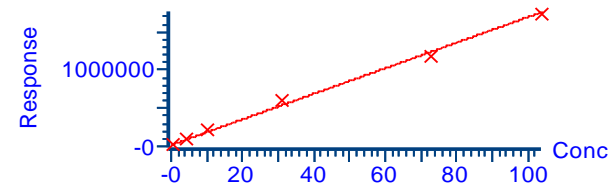
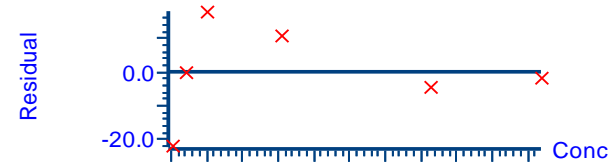
Piek 9.89 ook in procedure blanco

test1a020 Smooth(Mn,2x2)
cal05

F9:MRM of 2 channels,ES-
299 > 99
1.362e+006



Compound name: PFBS
Correlation coefficient: $r = 0.997892$, $r^2 = 0.995788$
Calibration curve: $16752.2 \cdot x + 17463.2$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



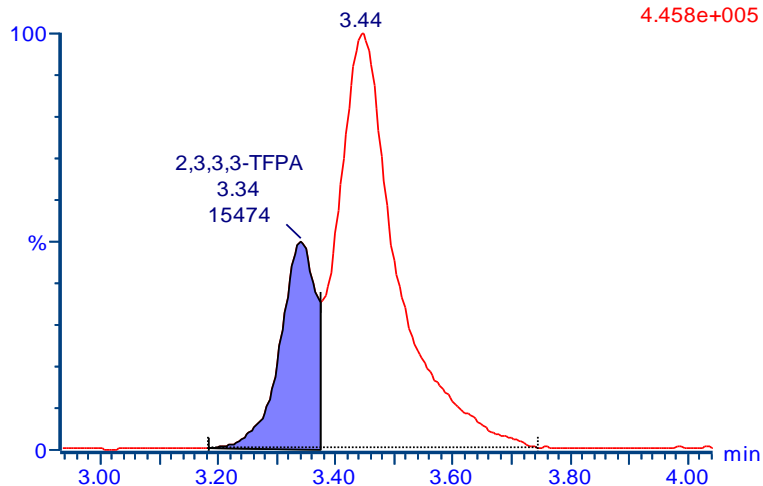
Laagst geteste standard: 0.3 $\mu\text{g/L}$

MRM: PFBS: 299-99

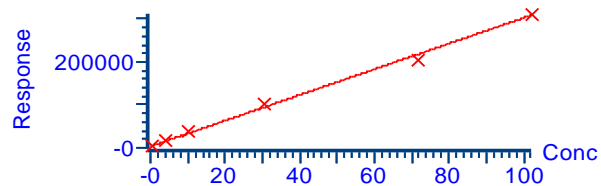
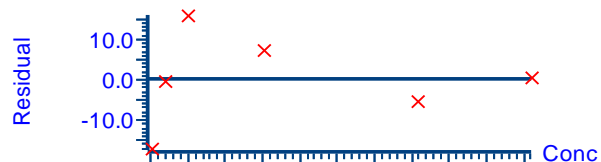
Calibratie resultaten: 2.3.3.3-TFPA en 2.2.3.3-TFPA

test1a020 Smooth(Mn,2x2)
cal05

F2:MRM of 1 channel,ES-
144.9 > 81
4.458e+005



Compound name: 2,3,3,3-TFPA
Correlation coefficient: $r = 0.998444$, $r^2 = 0.996890$
Calibration curve: $2992.99 \cdot x + 3283.04$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra



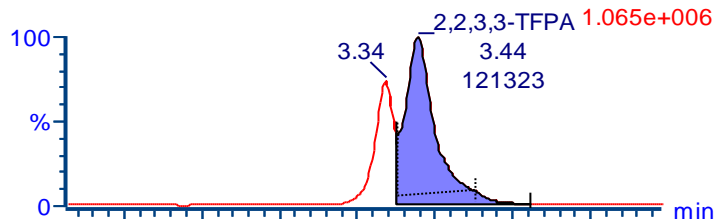
Laagst geteste standard: 0.4 µg/L

Afwachten tot Prevail UPLC tests voor betere scheiding

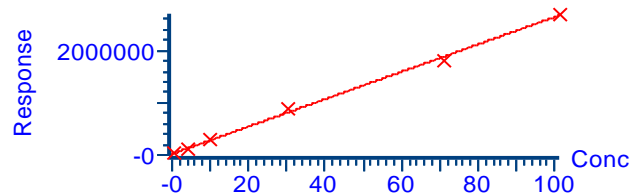
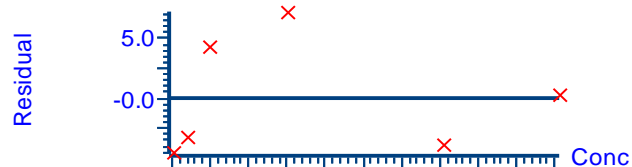
Lichte modificatie van mobiele fase of gradient is ook nog mogelijk

test1a020 Smooth(Mn,2x2)
cal05

F3:MRM of 2 channels,ES-
145.1 > 100.8
1.065e+006

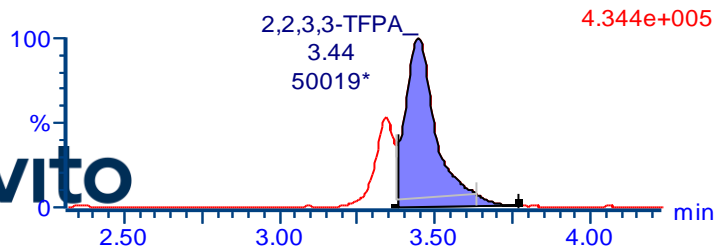


Compound name: 2,2,3,3-TFPA
Correlation coefficient: $r = 0.999301$, $r^2 = 0.998603$
Calibration curve: $26245.8 \cdot x + 18378.8$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis tra

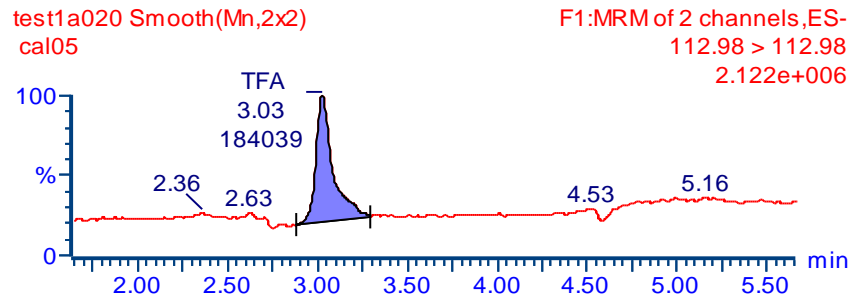
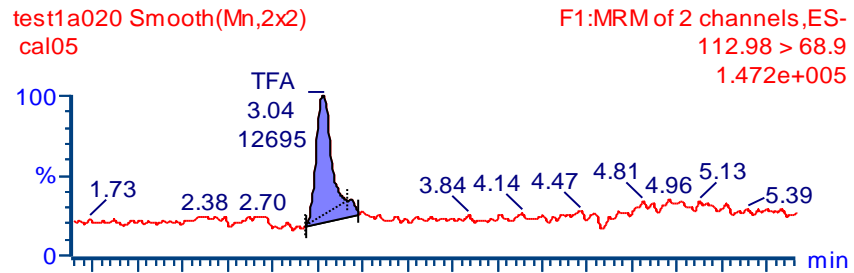


test1a020 Smooth(Mn,2x2)
cal05

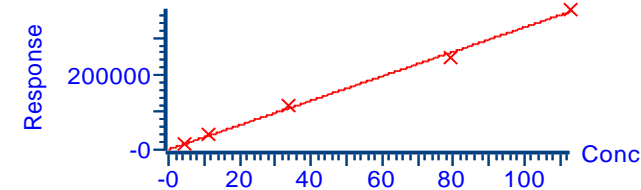
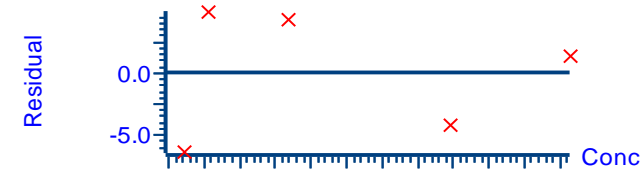
F3:MRM of 2 channels,ES-
145.1 > 80.9
4.344e+005



Calibratie resultaten: TFA



Compound name: TFA
Correlation coefficient: $r = 0.999231$, $r^2 = 0.998462$
Calibration curve: $3290.01 * x + -1227.47$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trar



Voorlopige LOQ: 4 µg/L
Testen met UPLC prevail kolom
Verdere testen met Raptor kolom

Verdere stappen

- Gebruik van Prevail organic acid UPLC kolom
- Vergelijking met de Raptor Polar X (Hilic, ion exchange)
 - Methode volgens application note GNSS3195-UNV

Column: Raptor Polar X (cat.# 9311A52); Dimensions: 50 mm x 2.1 mm ID, Particle Size: 2.7 µm; Temp.: 40 °C; **Sample:** Diluent: 50:50 Water:-methanol; Conc.: 400 ng/L; Inj. Vol.: 10 µL; **Mobile Phase:** A: Water, 10 mM ammonium formate, 0.05% formic acid; B: 60:40 Acetonitrile:methanol, 0.05% formic acid; Gradient (%B): 0.00 min (85% B), 5.00 min (85% B); Flow: 0.5 mL/min; **Detector:** MS/MS; Ion Mode: ESI-; Mode: MRM; **Instrument:** UHPLC.

- Validatie van de methode
- Testen op positieve 3M water stalen