# Automated In-Vial Extraction of Surface Waters for the Analysis of Mineral Oil by GC-FID

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Analytical support and method development

SampleQ

# Sample Prep

## Manual Procedure

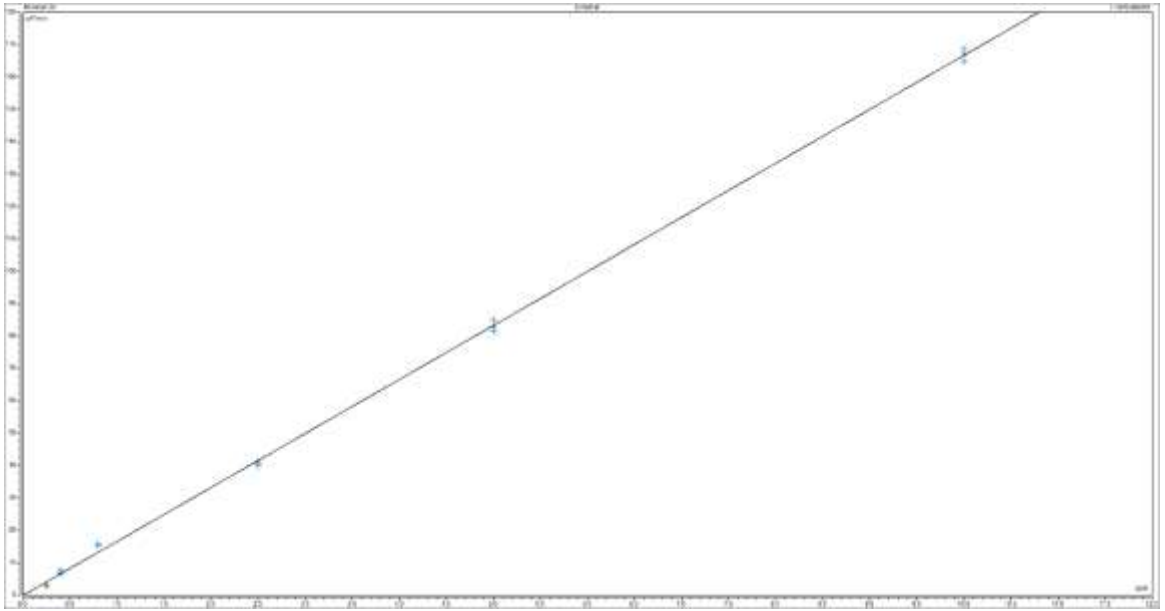
- Add 12 mL of surface water to a 20 mL headspace vial
- Place on the Triplus RSH robotic sampler
- Prep 2ml vials with  a scoop of Florisil

## Automated Procedure

- 3 (or 4) mL of hexane is added
- C10 and C40 ISTD markers are added by the RSH
- Vortex
- Inject 50 uL onto GC-FID (LV-PTV injection)
- Transfer 1.5 mL to a 2 mL vial containing  a scoop of Florisil
  (can be done for positives only or all samples)
- Vortex
- Inject 50 µL onto the GC-FID

# Calibration Curve

A calibration curve is made between 0.0625 ppm and 2.5 ppm (3 mL extraction)
Or a calibration curve between 0.0833 ppm and 3.33 ppm (4 mL extraction)



**$R^2$ = 0.9994**

**No offset!**

# Calibration Curve

| | Std 1 | Std 2 | Std 3 | Std 4 | Std 5 | Std 6 |
|---|---|---|---|---|---|---|
| **Rep 1** | 0.046 | 0.116 | 0.239 | 0.620 | 1.274 | 2.509 |
| **Rep 2** | 0.045 | 0.118 | 0.230 | 0.596 | 1.239 | 2.473 |
| **Rep 3** | 0.045 | 0.113 | 0.230 | 0.612 | 1.222 | 2.529 |
| **Average (ppm)** | 0.046 | 0.115 | 0.233 | 0.609 | 1.245 | 2.504 |
| **St. Dev. (ppm)** | 0.00069 | 0.002537 | 0.005104 | 0.012302 | 0.026522 | 0.028784 |
| **RSD (%)** | 1.5 | 2.2 | 2.2 | 2.0 | 2.1 | 1.1 |
| **LOD (ppm)** | 0.002 | | | | | |
| **LOQ (ppm)** | 0.007 | | | | | |

LOD = 3 * St. Dev.

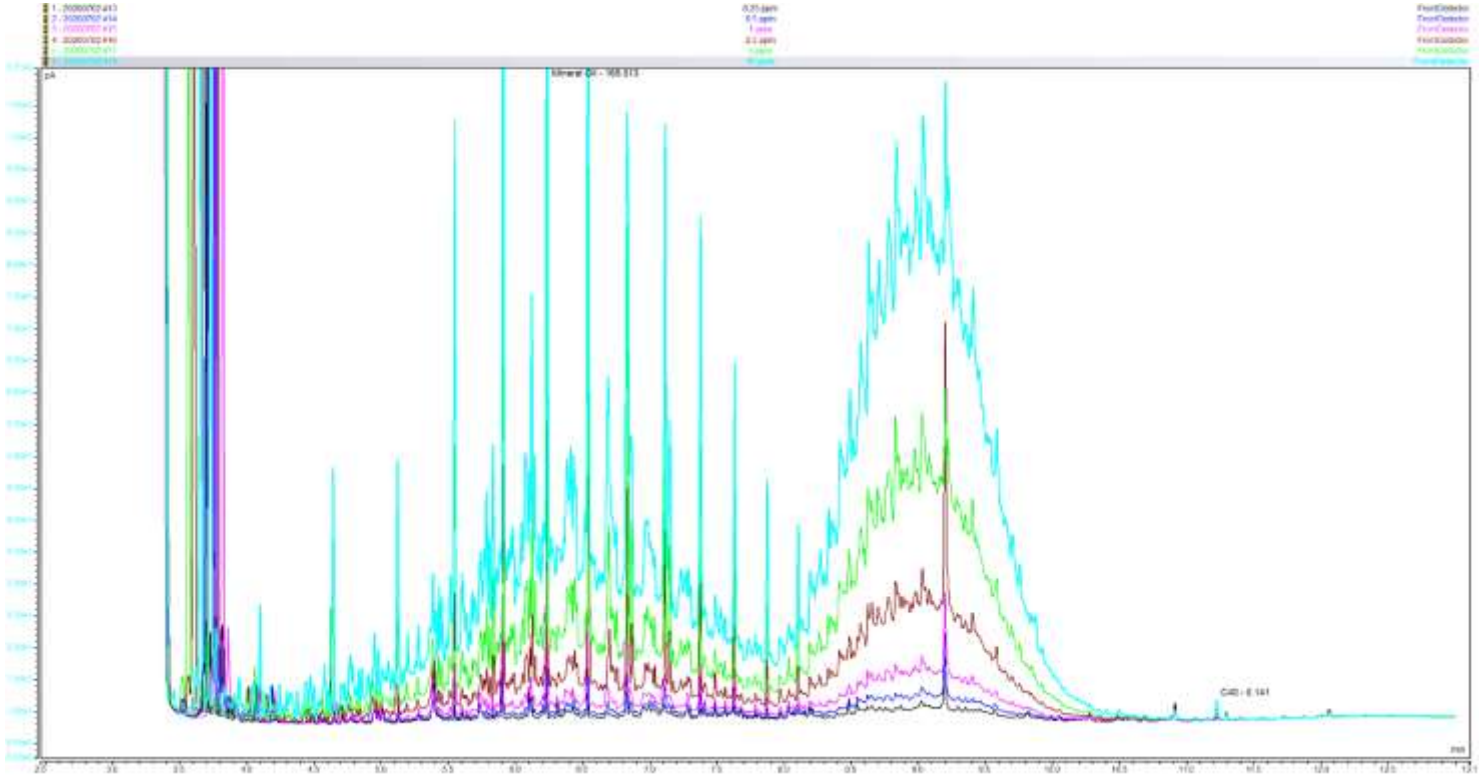
LOQ = 10 * St. Dev.

# Residuals

| | Std 1 | Std 2 | Std 3 | Std 4 | Std 5 | Std 6 |
|---|---|---|---|---|---|---|
| **Rep 1** | -25.72 | -7.4 | -4.37 | -0.77 | 1.93 | 0.37 |
| **Rep 2** | -27.8 | -6 | -7.85 | -4.63 | -0.84 | -1.09 |
| **Rep 3** | -27.4 | -10 | -7.96 | -2.03 | -2.22 | 1.17 |

# Overlay chromatogram

## Benelux RIVM Hydrocarbon Standard



# Method Summary

- Cycle time = 21 min! -> 68 analysis/24h
- No pre-column, 15 m column direct from PTV to FID
- Only 3 or 4 mL Hexane per sample
- RSD < 3%
- $R^2 > 0.999$
- Low LOD 0.002ppm
- Low LOQ 0.007ppm

# Conclusions

- Fast method with Prep Ahead function
- PTV analysis: Simple maintenance and more robust against matrix compared to Oncolumn
- Low solvent use
- Excellent initial validations
- No SPE necessary
- Only 1 software: **Chromeleon**
- Completely automated due to smart sequencing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