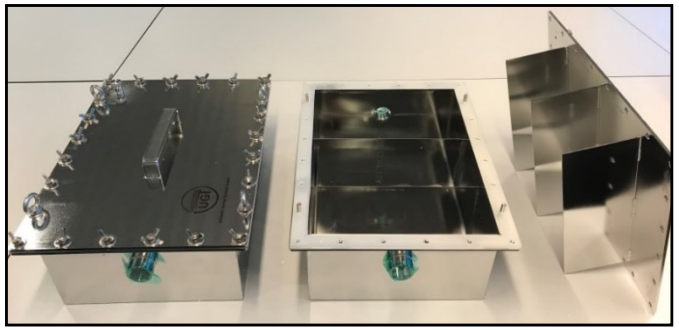
Do not underestimate the effect of sampling!



300 µm

100 µm

20 µm

Rijkswaterstaat
*Ministry of Infrastructure and Water Management*

# Monitoring and modeling microplastics in Dutch rivers and possibilities of discharge permits

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RWS WVL & CIV

14 July 2026

# Sampling performance determines data quality
## (and must be validated cf NEN 7777 & 7779)

**What is the goal?**

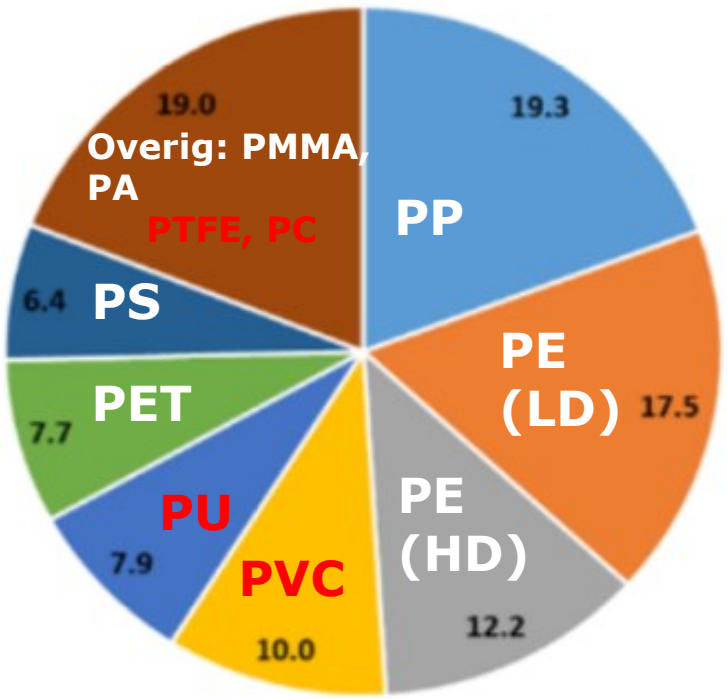
Monitor microplastics concentrations through a reliable 4 week average concentration of microplastics in the Meuse with as few microplastics analyses as possible.

**What are the most important variables for a representative sample?**

**Representativeness:** the degree to which a small set of samples accurately reflects the characteristics of a river or effluent: **sampling the right amount @ the right place and time, in the right frequency**

- **Identification, "Trueness" and variation**
  - **Repeatability:** Measure of the spread between simultaneous samples (or in close succession).
  - **Reproducibility:** Degree to which long-term sampling matches. Indispensable for monitoring, because data is compared over several years.

# RWS analyses 8 of the most commonly produced plastics en rubbers:




In RWS method (white):

| | *Used in:* |
|---|---|
| • PE = polyethylene | *plastic bags, packaging* |
| • PP = polypropylene | *bottles, toys* |
| • PET = polyethyleenterephtalate | *textile, bottles* |
| • PS = polystyrene | *packaging* |
| • PMMA = polymethylmethacrylaat | *Perspex* |
| • PA = polyamide | *Nylon* |
| • NR = Natural rubber | *Tyres* |
| • SBR = Styreenbutadiene rubber | *Tyres* |

(Not yet) analysed in RWS method (red):

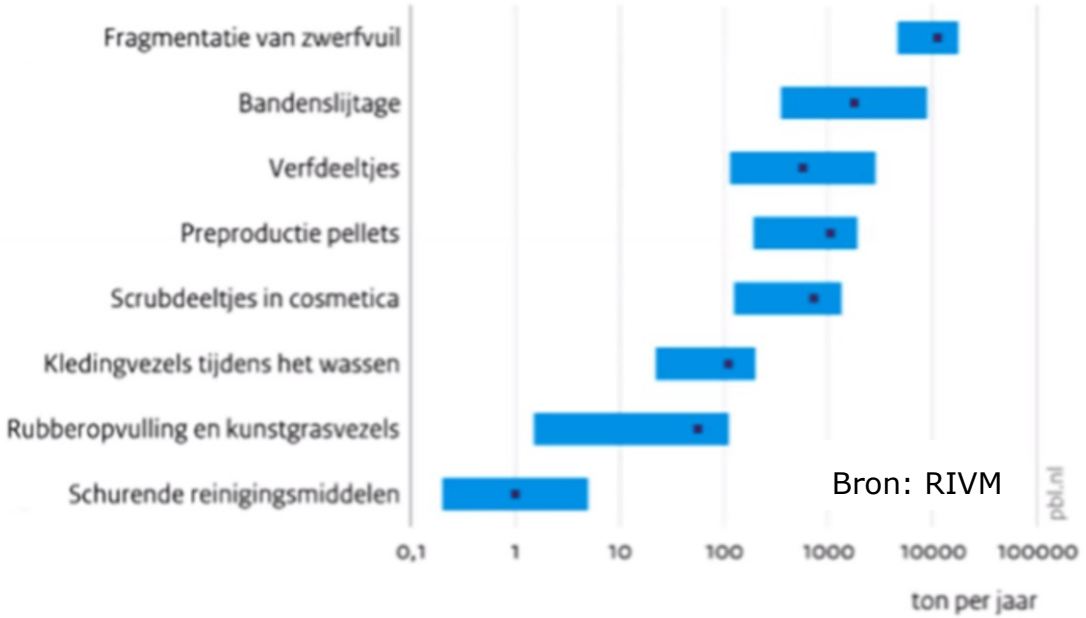
- PVC, PU, PC, PTFE

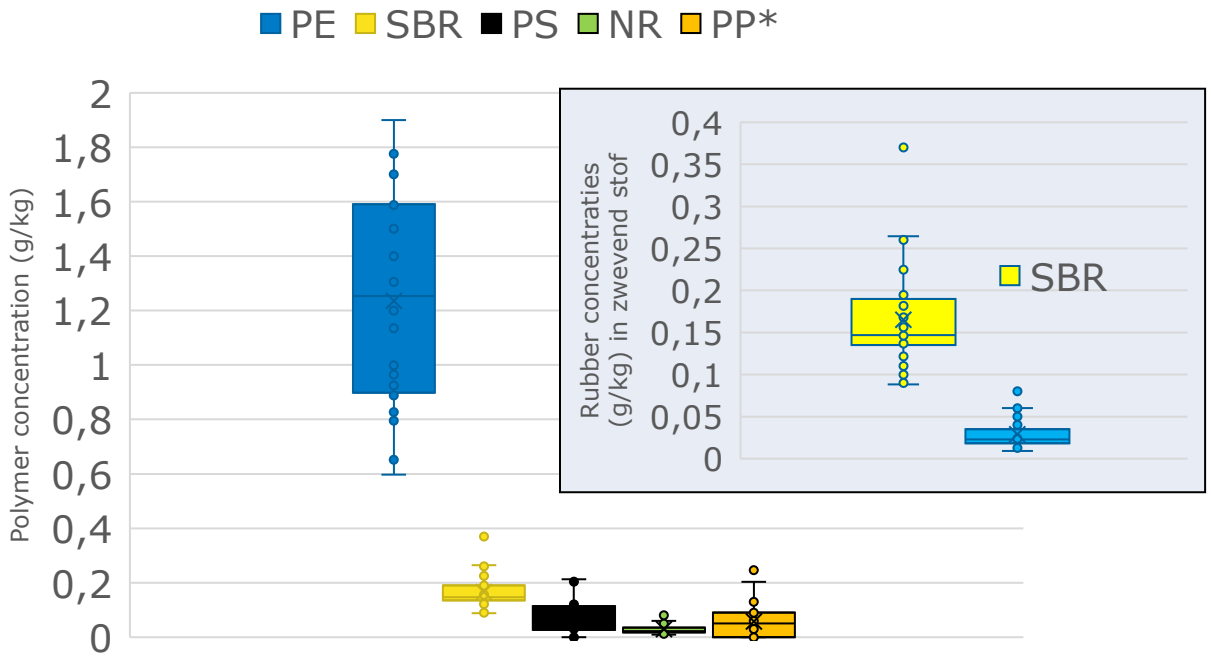
Production of tyre wear is not included in this image

**>75% (mass) of the most used plastics in RWS analysis**

PlasticsEurope, Plastics - the Facts 2019 – An analysis of European plastics production, demand and waste data.

# Emissions vs. measurements:
## Waste (PE,PP) and tyre wear (SBR and NR) dominant




**Expectation based on emissions:**
Waste (PE > PP > other) and tyre wear (SBR > NR) produce more microplastics than the other sources.

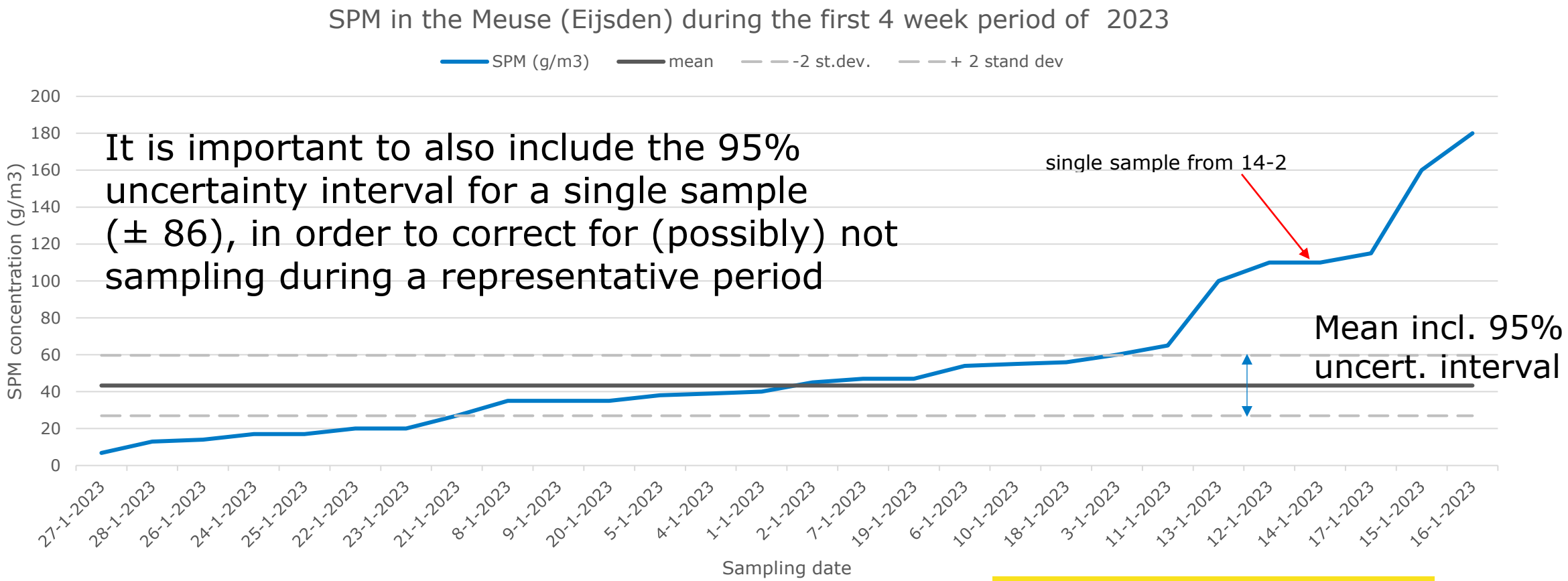
**Test results match expectations:**
PE, PP, PS, and SBR are the most common.
At all locations: the total microplastic concentrations from waste (PE, PP) are higher than from rubbers.
Rubber from car tires (SBR) is found more often than rubber from truck tires (NR).

# Representative and robust sampling

- **Sampling location, frequency, sampling depth**, ……?
- **Discrete sampling vs. "time integrated" sampling**
- **Sampling performance (validated repeatability)**
- **Sampling volume**
  1 $m^3$ or 100.000 $m^3$ of river water?
- **SPM range**
  *NL: ≤ 5 g/$m^3$ (=detection limit) during 50% of year*
- **Particle size range**

  *Based on definition (1 µm - 5mm) or particle size distribution?*

# **Sampling representativeness:** 1x vs 28x

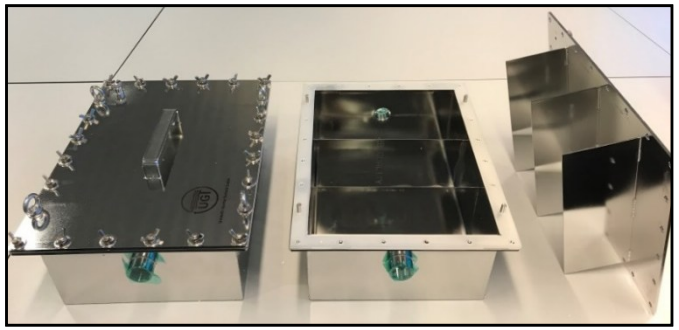



Also an important topic for discharge permits

# Tested sampling methods:

| Method | Duration | Water volume |
|---|---|---|
| Sedimentation box | Up to 4 weeks | Large |
| Continuous flow centrifuge | hrs- days | Medium |
| Sieve cascade | ~1 hr | Small when sampling small particles |
| Filter cascade | hrs – days | Small when sampling small particles |

**Sedimentation box**



*Bannick et al: https://doi.org/10.1021/acsestwater.1c00439*

**Sieve cascade**

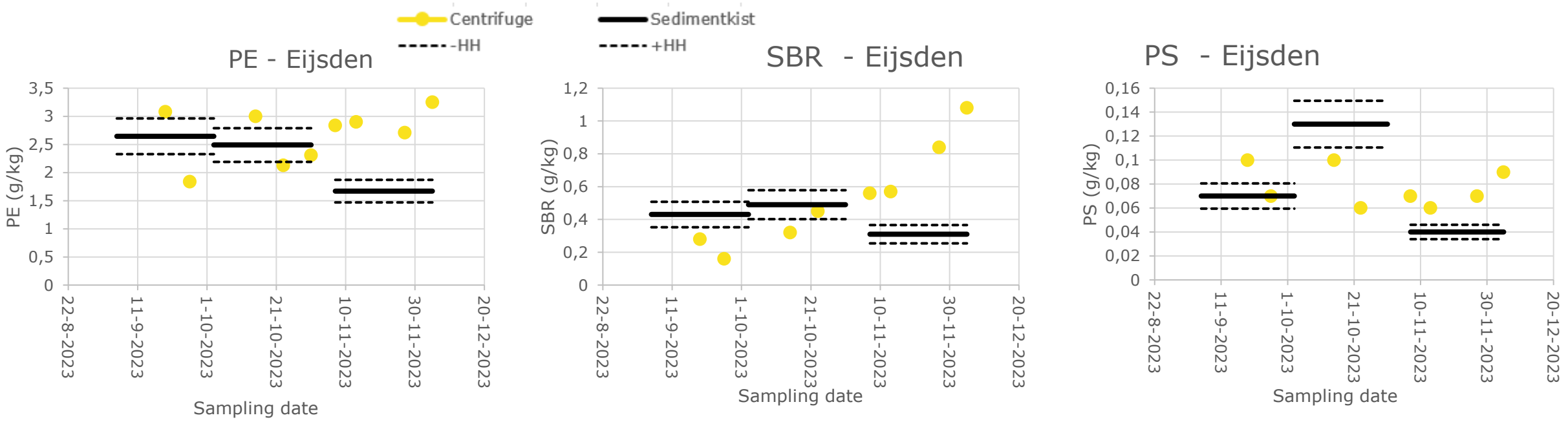
**Continuous flow centrifuge** (used for routine SPM monitoring)

# Centrifuge vs Sediment Box @ Eijsden

## Microplastic concentrations (GC-MS) in SPM within very narrow range

Differences between time integrated sampling with a sediment box and sampling during “only” several hours with a centrifuge are smaller than expected

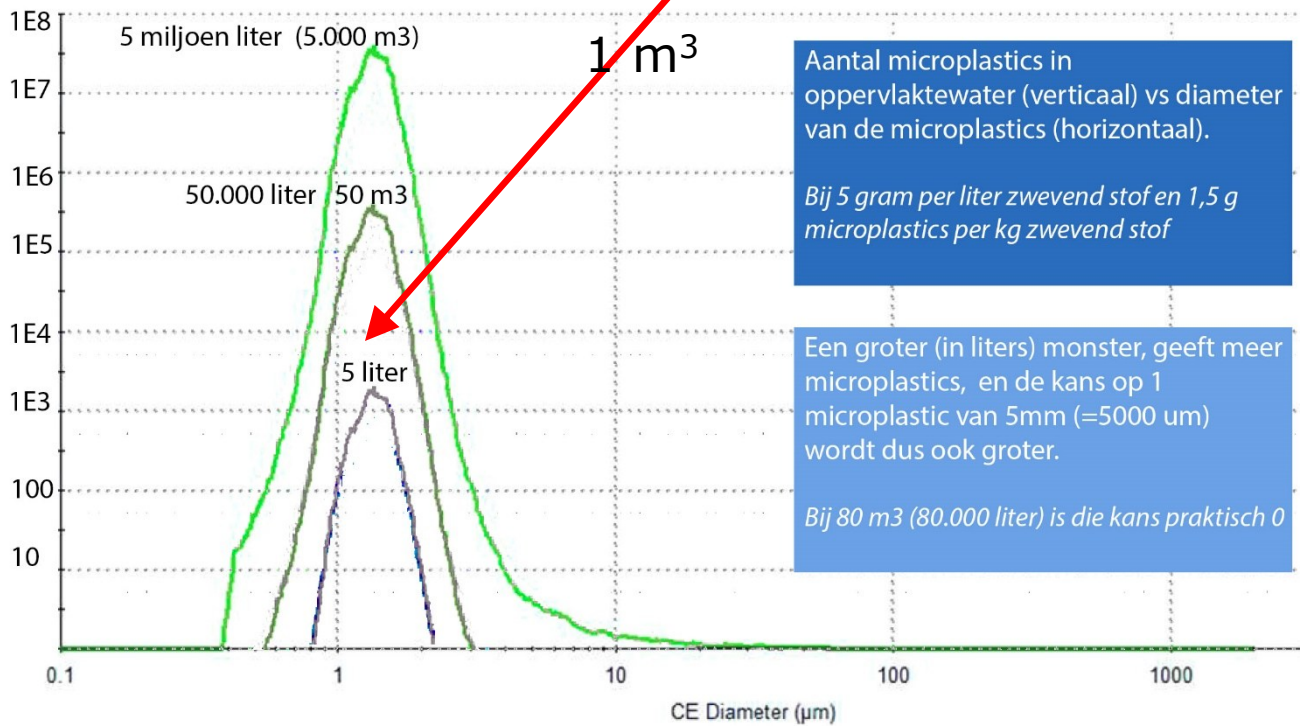
NO NEED FOR DAILY ANALYSES:
For sampling dates without known plastics concentrations, the long-term average mp conc. (g/kg) incl. 95% uncertainty interval can be used



# How many m³ for a sample which includes 5 mm microplastics?

- Pilot tests with sieves, filters, centrifuge and SBs indicate that large microplastics (>300 µm) are rare in the Dutch part of the river Meuse

- For a well-repeatable analysis of invisible microplastics (<300 µm) you need to sample much less river water, than for the analysis of visible (>300 µm) microplastics, but the representativeness decreases

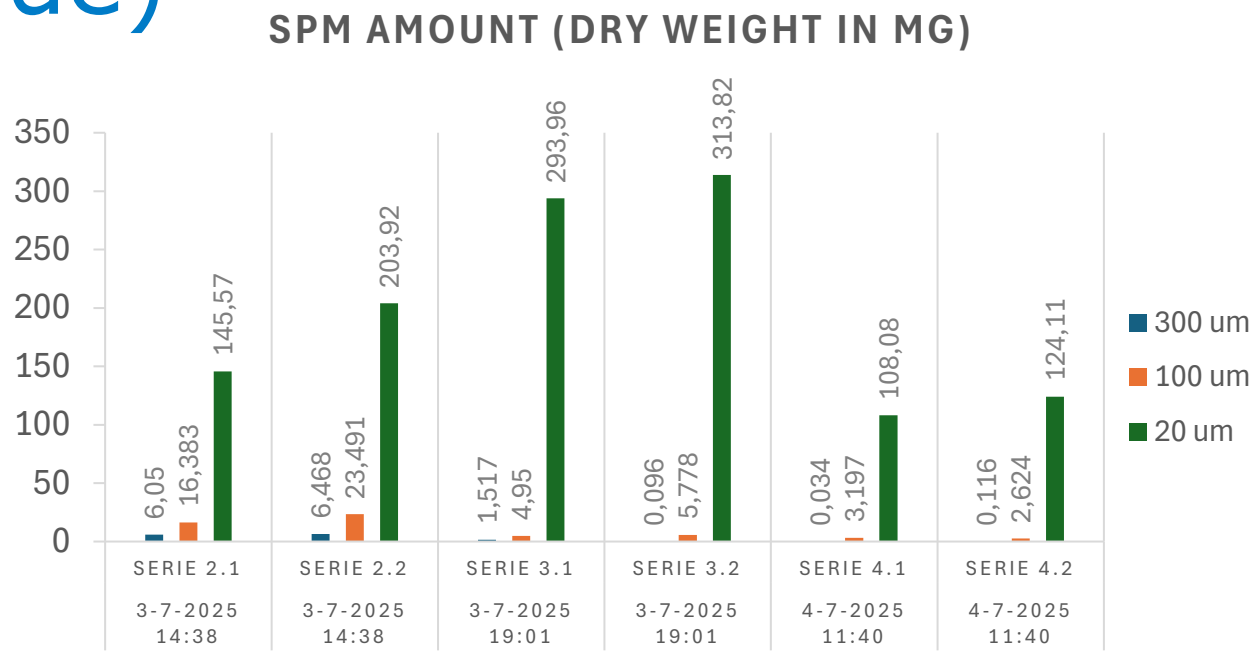


Sieves

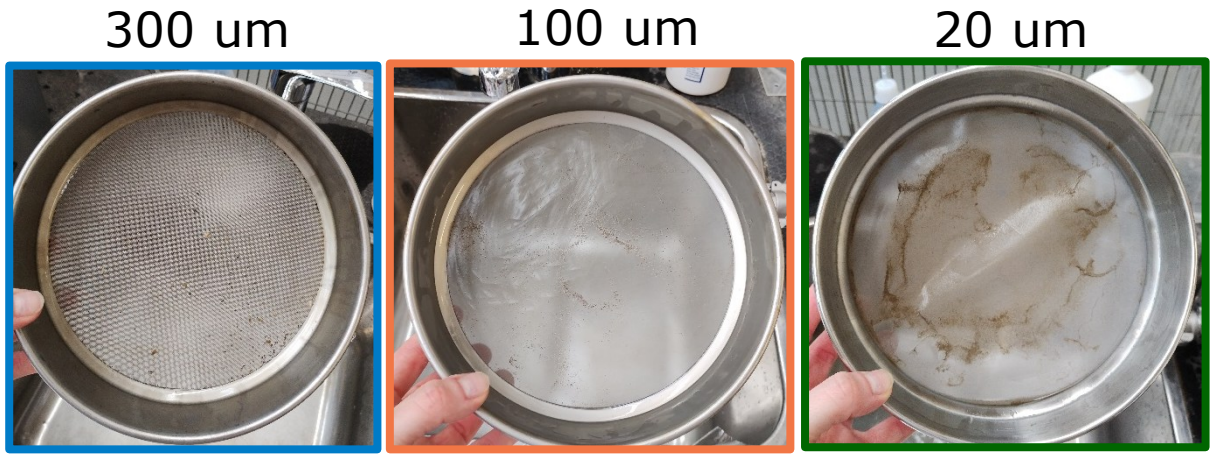
# Sampling by sieve (cascade)

- Repeatability (n=3):

| Meshsize | RSD (%) |
|---|---|
| 300 um | 69 |
| 100 um | 18 |
| 20 um | 13 |




- Not practical for surface water monitoring:
  - Low water volumes
  - Small sample amounts
  - Missing particles <20 µm
  - Susceptible for contamination



# The information need defines the minimum performance characteristics for sampling, analysis and modelling

1. Can we confirm a 10% decrease in microplastic concentrations after 5 years?
2. What's the risk of microplastics in water for aquatic organisms?
3. Can we convert particle distributions in mass distributions (and vice versa)?

Quantitative (accuracy and precision) validation of data and model must be done. Their uncertainty intervals determine whether you can fullfill the information need:

Can you tell me if and how sick I am?

Yes: 16.0

But last week you said 114.21?

I'll measure again.

How often will you measure for a reliable result?

No idea. Are you insured?

# If you sampled 200 gram over 14 days, was that collected over the entire period or not?

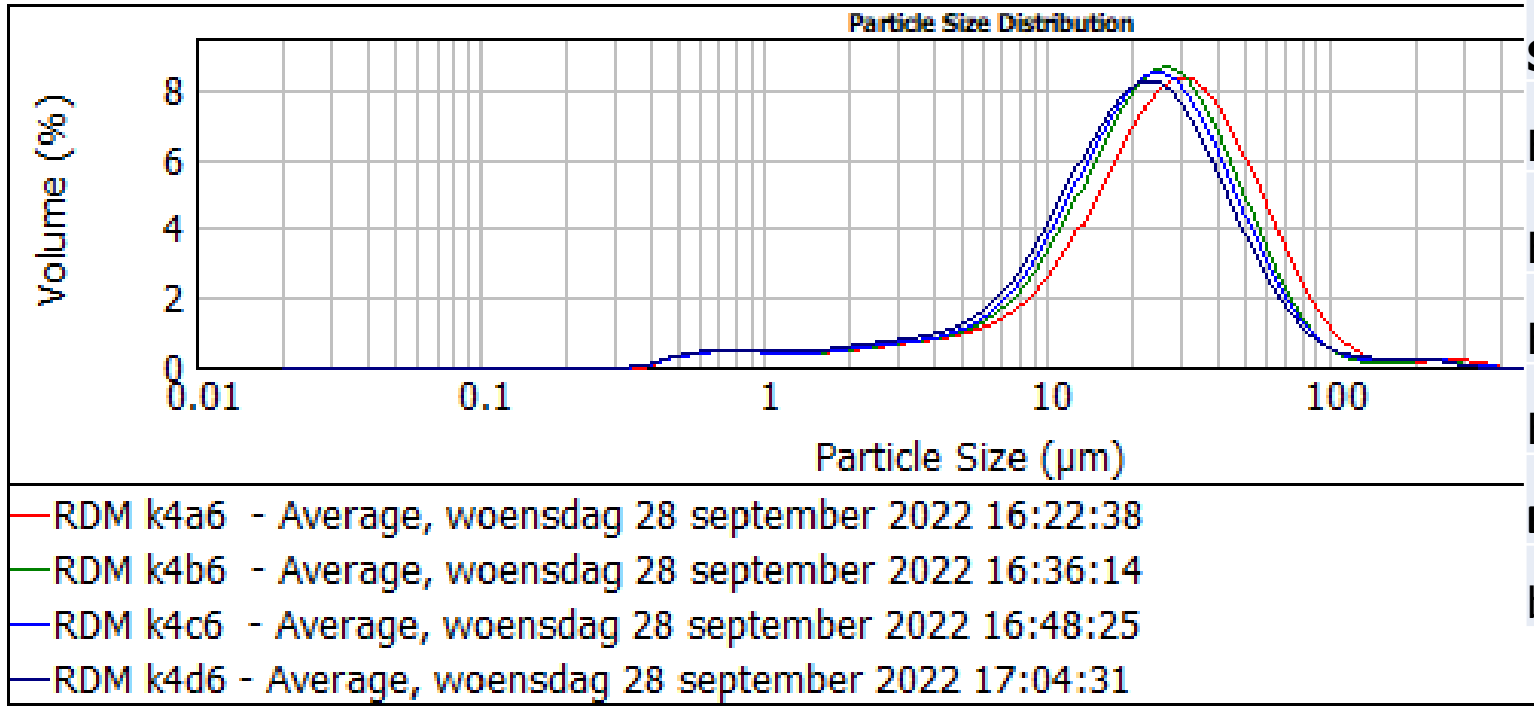
- Added visible microplastics (≥1 mm) present on Day 1 were all still in the bin on Day 14.
- Microplastics <1 mm were also still present in the bin after 14 days.
- But what about the fraction <20 µm?

# Repeatability centrifuge (CFC)

- 6 sampling periods simultaneous sampling in Eijsden
  CFC on the station and a mobile CFC on a trailer.
- 2 sampling periods simultaneous sampling with 3-4 CFCs by boat
- Average RSD of collected SPM: 9%
  - *Based on wet weight SPM*
- Research in progress

Centrifuge

# Does a sediment box take a representative sample ? particle distribution (volume) of 4 consecutive boxes:




| **Sample** | **d (0.1)** | **d (0.5)** | **d (0.9)** |
|---|---|---|---|
| Box a | 6,336 | 26,828 | 64,042 |
| Box a duplo | 6,23 | 25,905 | 61,034 |
| Box b | 6,083 | 23,376 | 54,071 |
| Box c | 5,952 | 22,207 | 52,917 |
| **Box d** | 5,194 | 20,747 | 50,984 |
| box d duplo | 5,263 | 21,203 | 52,344 |

# Conclusions

- **Influence of sampling on monitoring data is often overlooked**
- Sampling a large water volume (>100m$^3$) or sampling @ a high frequency is important to obtain a representative sample.
  - Filter- and sieve cascades are badly reproducible and not effective in sampling small particles. Clogging makes sampling of large volumes impossible.
  - Sedimentation box is suitable for reproducible, time-integrated sampling, but underestimates the smallest particles.
  - Continuous flow centrifugation (CFC) leads to comparable microplastic concentrations in SPM, and yields a higher estimation of the smallest particles.

For microplastic monitoring RWS choses to use the CFC at multiple locations in the Netherlands.

**Combining daily SPM measurements with the average microplastics mass concentrations of a relatively small sample set, yield accurate exposure or annual load estimations for the Meuse**

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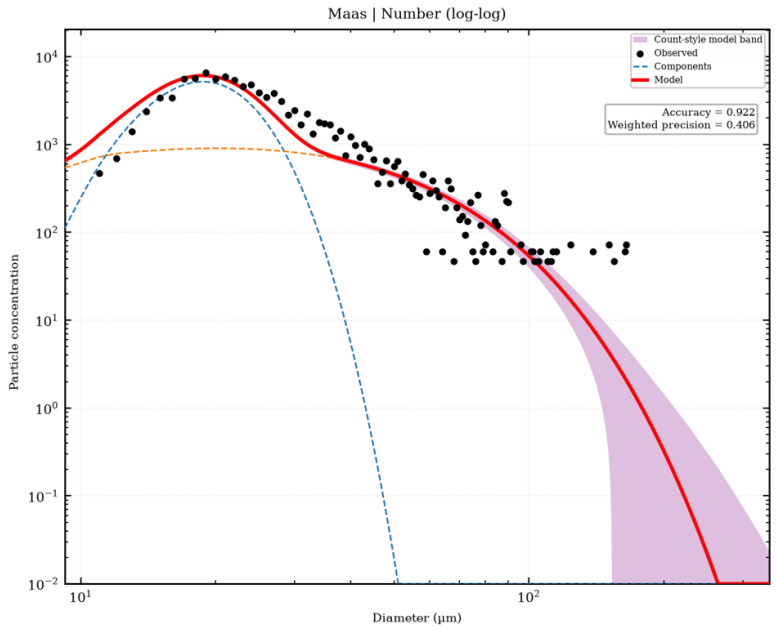
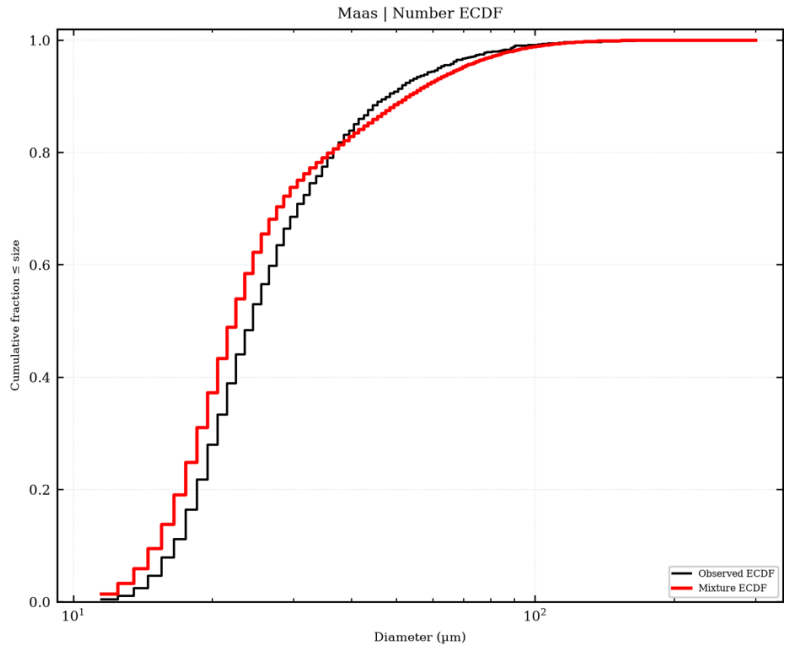
Construct experimentally validated particle distributions (nrs: 1-5000 µm & Volume) for the Meuse catchment area on the basis of large (n> 200.000 MP) FT-IR datasets originating from large volumes (>1000 $m^3$) river water

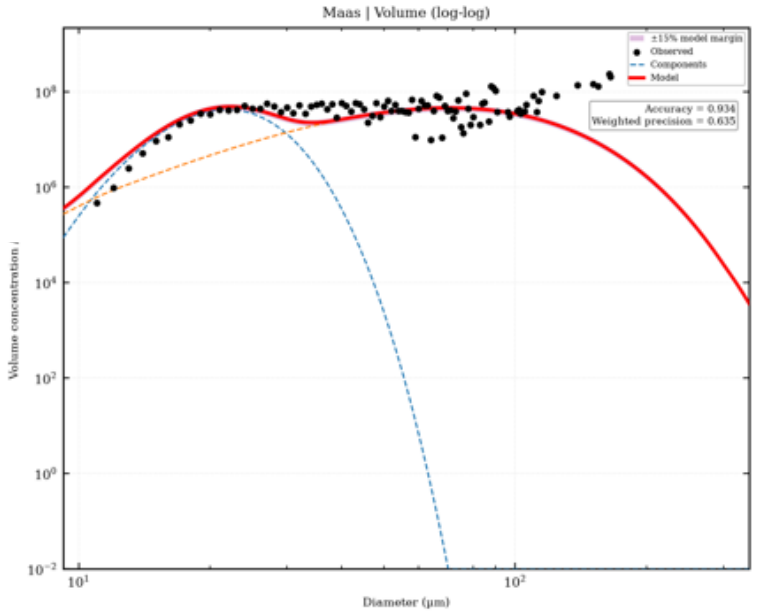
# Modelling

- Validated particle distributions (nrs & volume) for (individual) microplastics

# **Microplastics modelling** (example using 2 functions)
# FT-IR data for catchment areas




Particle nrs distribution

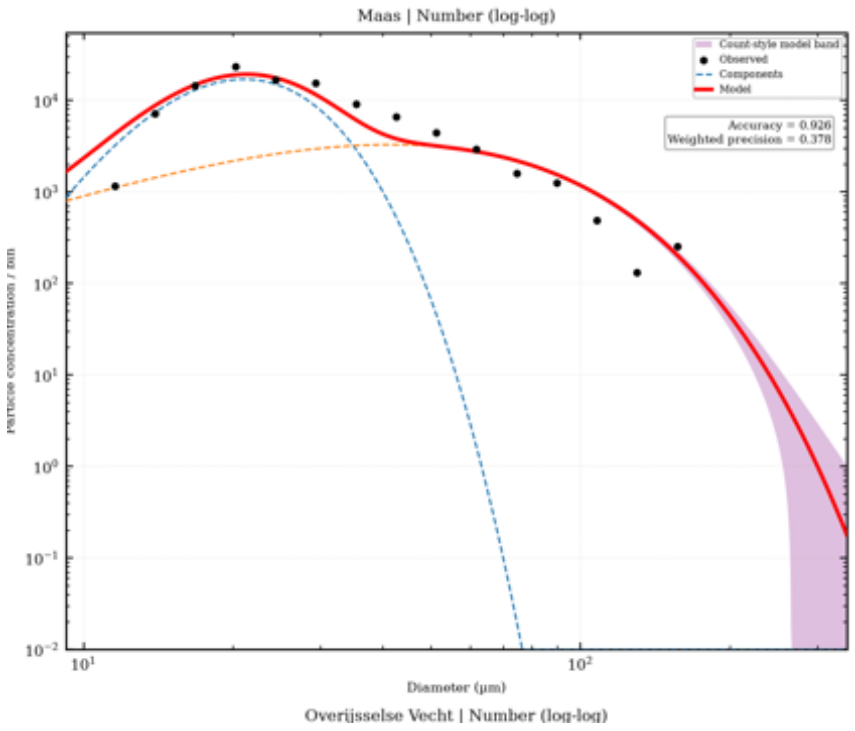
Cumulative particle nrs distribution

Particle Volume distribution

Validated for nrs and mass over entire size range

# Microplastics modelling (binned FT-IR data)

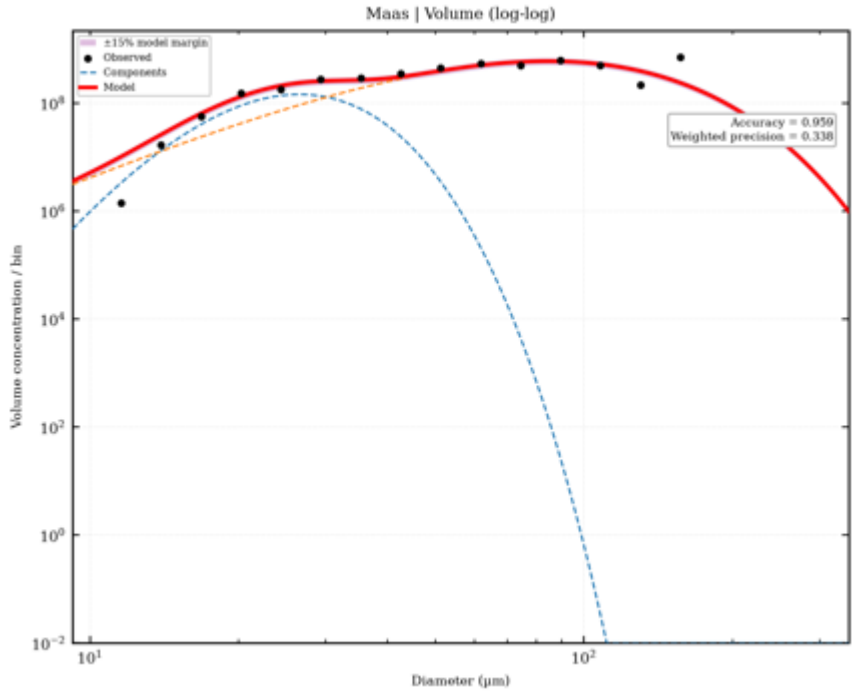
## FT-IR data for catchment areas as a **stone of Rosetta**




Particle nrs distribution

Conversion of mass (GC-MS) in nrs (FT-IR data) and vice versa)

Fill in the measured mass, use a validated model to obtain an accurate estimation of the nrs and size of MP.




Particle Volume distribution

For any discharge,
the guiding principle is always that pollution must be tackled at the source.

Companies are therefore required to make efforts to prevent or reduce pollution.

Monitoring microplastics emissions is the first, and crucial, step.

Monitoring microplastics is a complex process.

Close cooperation between enforcing authority, RWS and companies is vital.

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# Possibilities of discharge permits

- Monitor effluents, preferably using robust, time integrated sampling techniques