# Efficient tools for detecting micro- and nanoplastics in surface waters

Interreg
Co-funded by the European Union
IPA Hungary - Serbia
Microplastics

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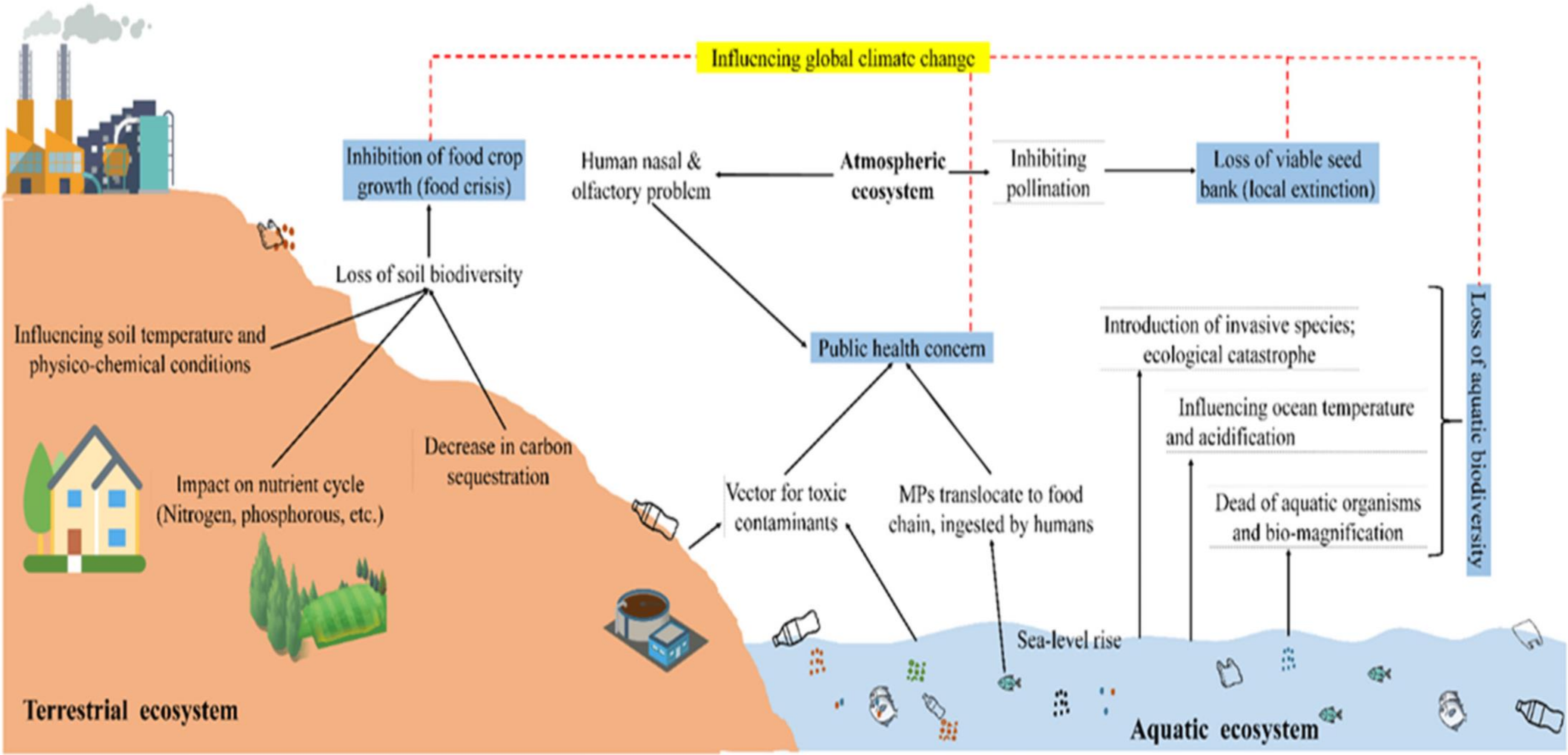
# Main sources of MP and NP

# How much is getting into our environment?




About 20 million t / year → increasing !!!

Source: 1. https://iucn.org/resources/issues-brief/plastic-pollution, 2. Sustainability 2021, 13(17), 9963; https://doi.org/10.3390/su13179963

# Where is microplastics (MP) ?

| Type of plastic | Density (t/m3) |
|---|---|
| polietilén/ polyethlylene | 0.917-0.965 |
| polipopilén/polypropylene | 0.90-0.91 |
| polisztirol/ polystyrene | 1.04-1.10 |
| poliamid (nylon)/ polyamide (nylon) | 1.02-1.05 |
| poliészter/ polyester | 1.24-2.30 |
| akril/ acrylic | 1.09-1.20 |
| polioximetilén/ polyoxymethylene | 1.41-1.61 |
| polivinilalkohol/ polyvinyl alcohol | 1.19-1.31 |
| polivinilklorid/ polyvinylchloride | 1.16-1.58 |
| polimetilakrilát/ poly methacrylate | 1.17-1.20 |
| polietilén tereftalát/ polyethylene terephthalate | 1.37-1.45 |
| alkid/ alkyd | 1.24-2.10 |
| poliuretán/ polyurethane | 1.2 |
| Forrás: Bordós G, Reiber J: Mikroműanyagok a környezetben és a ... 2021 | |

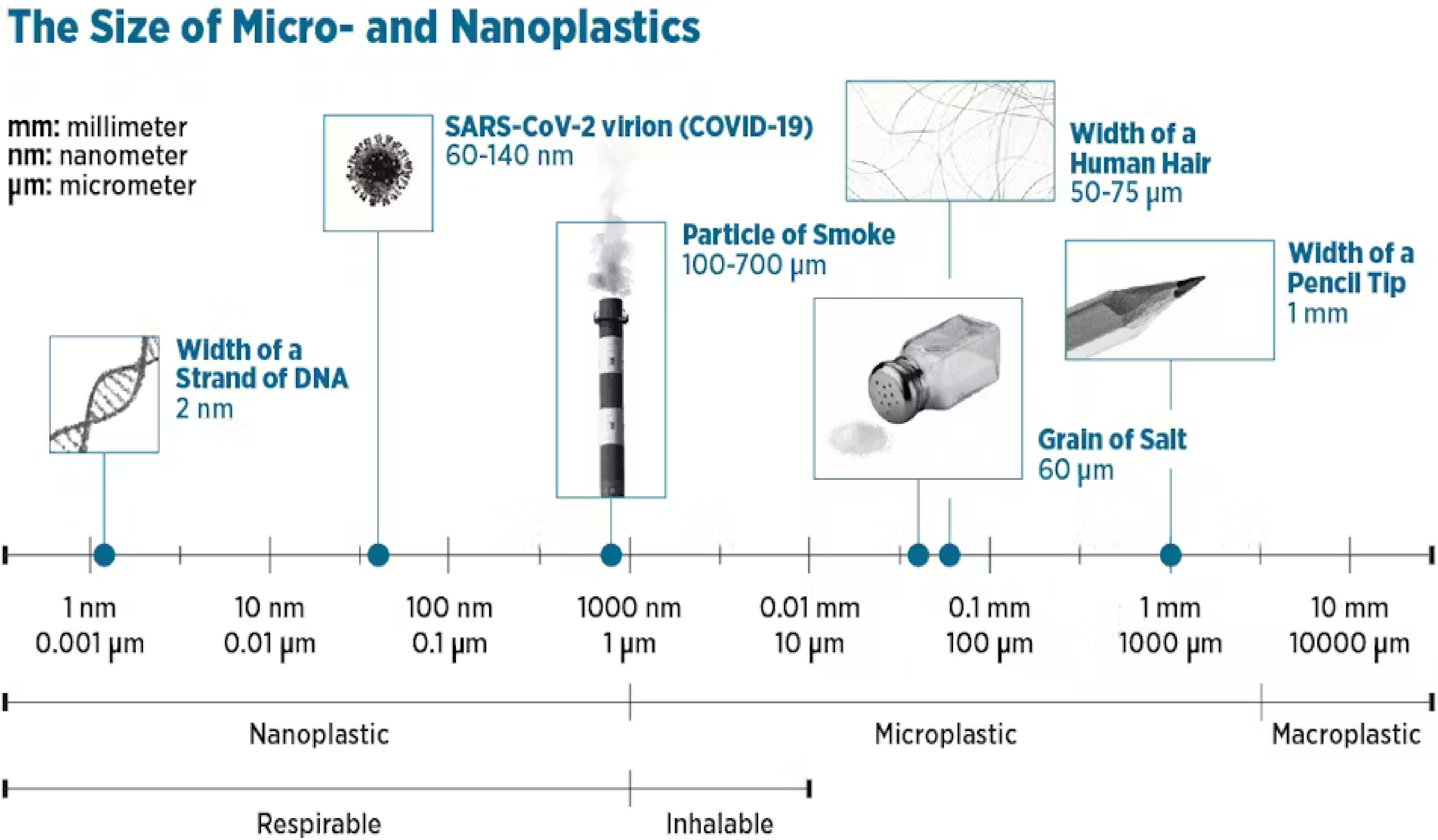
# Hundreds of millions of tons of MP and NP in the ocean and underwater...

# Estimation → Measurement

News: „...we estimate that the mass of nanoplastic may amount to 27 million tonnes (Mt).“ (Source:Hietbrink, Sophie et.al. Nanoplastic concentrations across the North Atlantic,  Nature 2025 07.01.)

- NP pollution is pervasive and exists in concentrations millions of times higher, by particle count, than microplastics! (13-1700 pcs/liter...)
- NP oroginates mainly from the degradation of MP!

What is nanoplastics?




# Why is nanoplastics important?


Is there any correlation between MP and NP??

**YES!**

Depending on:

- 1. Type of MP, Polistyrene UV-degradation (Mattson et.al. Environmental Science & Technology, 2020)
- 2. Sources (eg. Municipal, Industrial..)

- 3. Degradation processes → positive correlation (Alimi et.al. 2018, Investigation of Rhine river Mintenig et.al. 2021 Water Research)

Summary: … →<u>It is indispensable to measure NP and MP!</u>

  – <u>HOW ??</u>

# Samplers for MP 1.

**1. Plankton nets** (Manta nets)

Mesh size: 100 μm – 300 μm

**Advantages:**

- Investigation of large sample
- Efficient in measuring large MP > 100 μm

**Disadvatages**:

- Inaccurate measurement of water quantity
- 1 μm < MP < 100 μm  are not measured…
- Self-contaminantion
- Problematic cleaning

# Samplers 2.

**2. Pump-and-Filter Systems**

Mesh size: 10 μm – 300 μm

**Advantages:**

- Accurate measurement of sample size!
- Efficient in measuring smaller

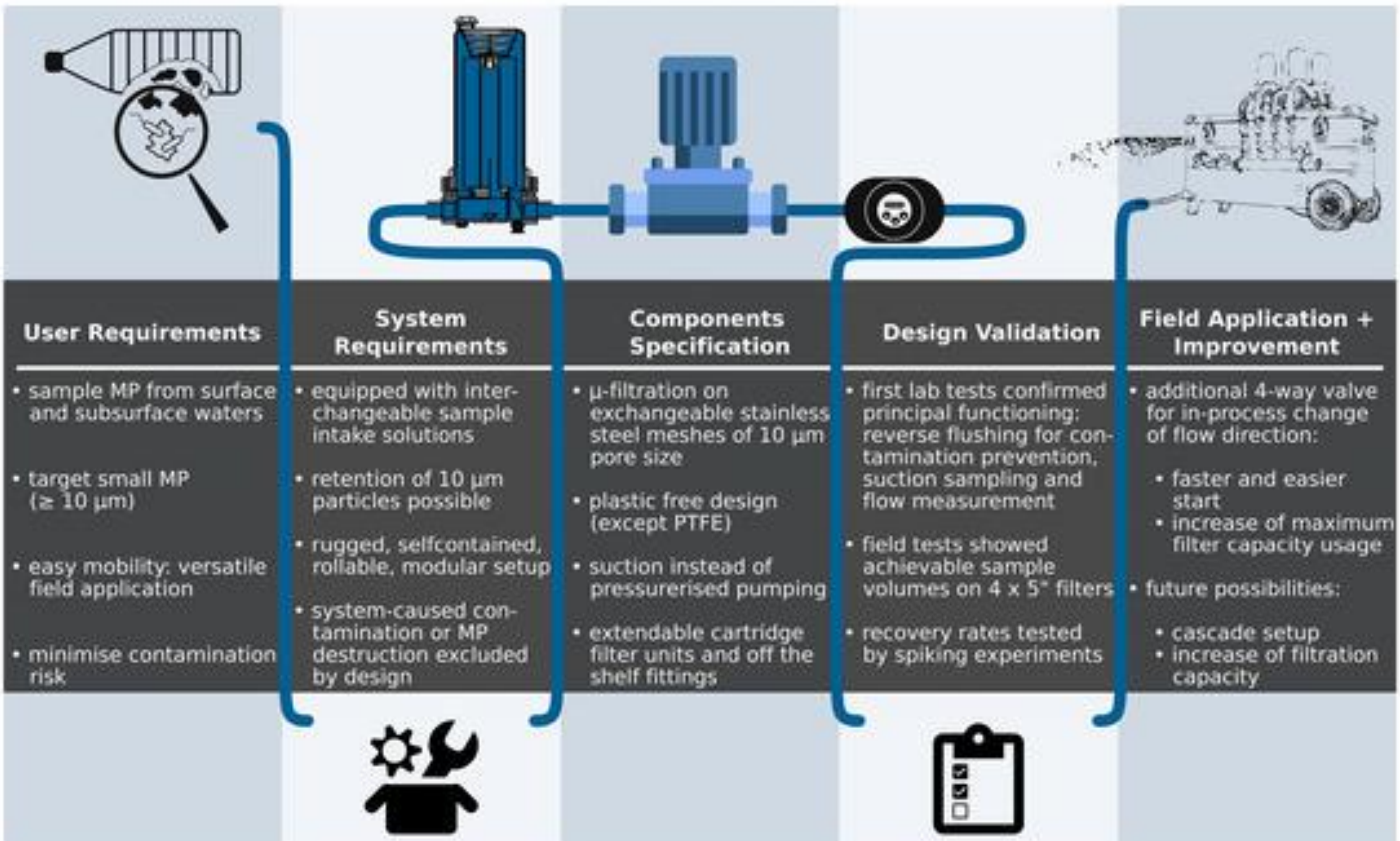
MP < 100 μm as well!

- Minimal danger of self contamination
- Easy cleaning

**Disadvatages**:

- Smaller sample size than with Manta net V < 10 m3

# Pump and filter sampling device (MP) (R, Lenz, M Labrentz, Water 2018, 10(8), 1055; https://doi.org/10.3390/w10081055 )



# Samplers 3.

## Grab samplers for water

**Advantages:**

- Small, light, simple, cheap
- Minimal chance for self contamination

**Disadvantages:**

- Very small sample volume →

not representative for large water bodies

# Samplers 4.

## Grab samplers for sediment

**Advantages:**

- Small, light, simple
- No chance for self contamination

**Disadvantages:**

- Very small sample volume →

not representative for large water bodies

→ Large number of samples needed!

# Samplers 5.

## Passive samplers, sediment traps

**Advantages:**

- Small, simple
- No chance for self contamination
- Long time data
- Low operation cost

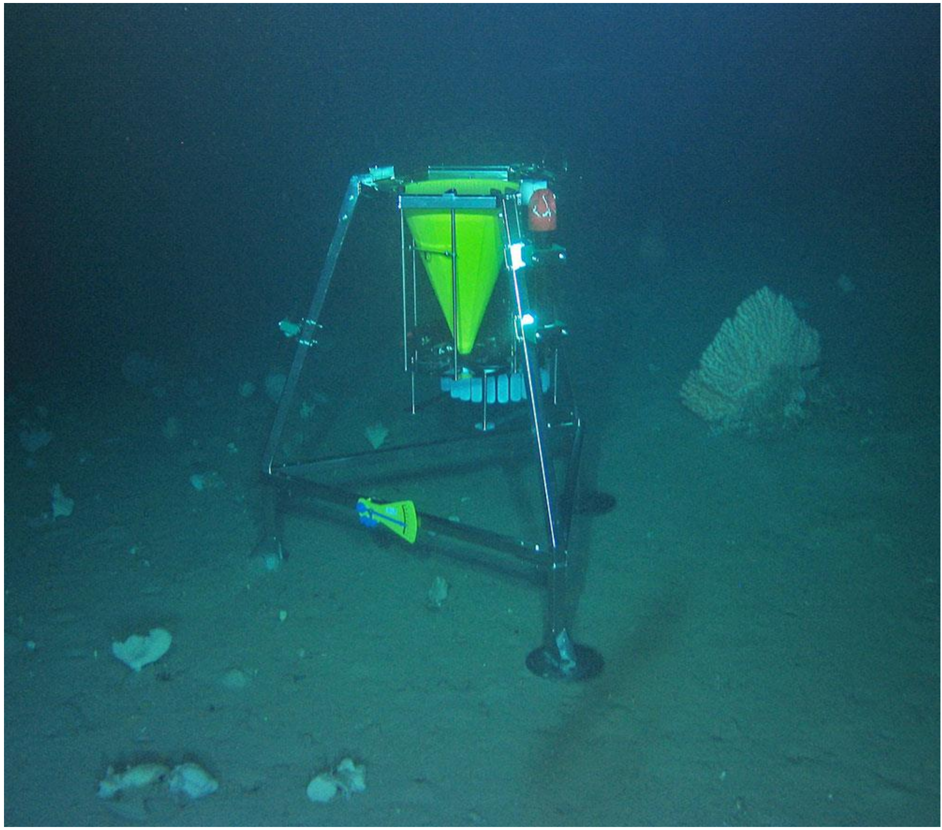
**Disadvantages:**

- Very small sample volume →

not representative for large water bodies

→ Large number of samples needed and

- Long time needed for evaluation



# Measuring nanoplastics 1.

➔ Removing NPs is difficult due to their small size (<1000 nm), diversity, and low environmental concentrations, which are often **masked by** NOM!

➔ The most effective filtration techniques **are not simple mechanical filters** but rely on physico-chemical processes, often used in combination:

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**<u>Membrane Filtration:</u>**

Ultrafiltration (UF): Effective for larger NPs (1-100 nm), but smaller particles can pass through, and membranes can foul easily.

Nanofiltration (NF): With pores ~0.5-1 nm, NF removes a significant fraction of NPs but requires higher energy and is also prone to fouling.

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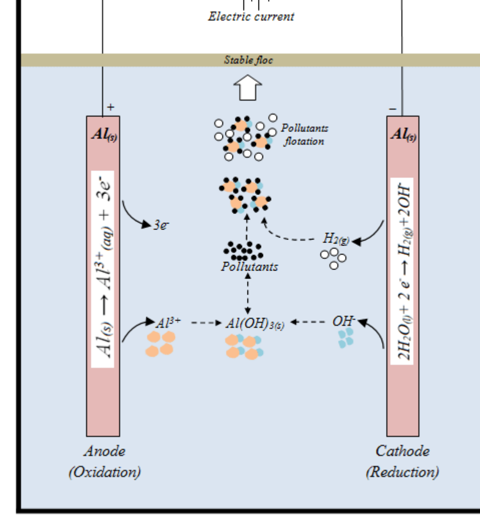
# Measuring nanoplastics 2.




**Activated Carbon:**

- Granular (GAC) or Powdered (PAC) Activated Carbon uses adsorption to trap NPs, especially hydrophobic types like polystyrene (PS) and (PE).

**Electrocoagulation (EC):**

- Applying an electric current using metal (e.g., Al, Fe) electrodes. The dissolving metal ions form coagulants in situ, which form flocs to trap NPs. → Bubble generation ..→ flotation!

**Advantage:** Very effective, uses fewer chemicals, and handles stabilized nanoparticles well.

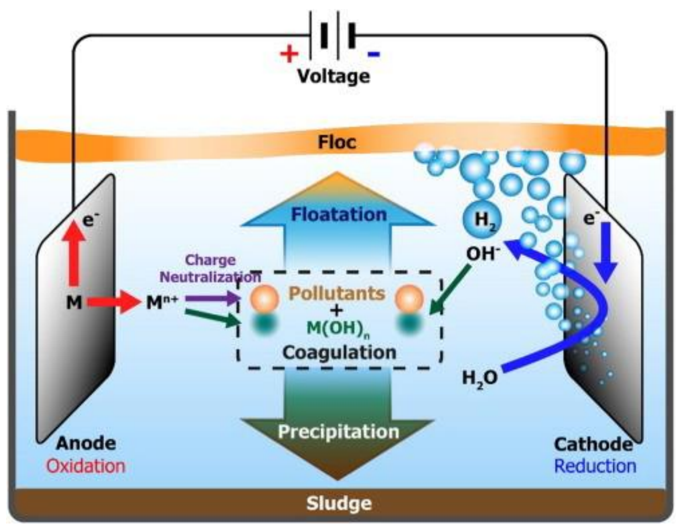
# Efficient practical implementation

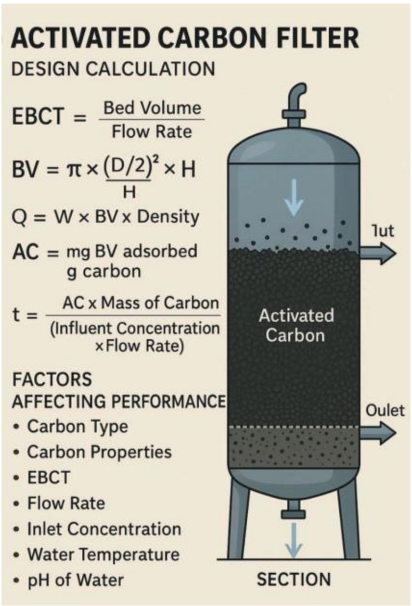
**Multi-barrier approach:**

Pre-filtration ( 300µm→ 100µm→ 10µm) Lab preparation →filtration 1 µm.

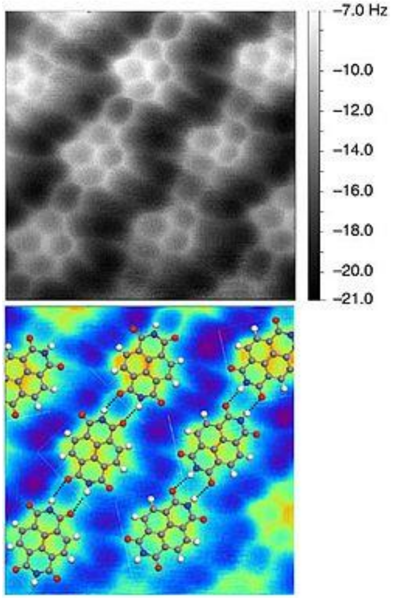
→ Coagulation/Flocculation → Sedimentation or flotation → Activated Carbon Filtration → Nanofiltration (NF, TiO2 ceramic filter)

→ Laboratory analitics!



# Analytical Methods for Identification and Characterization




**Core Challenge:** Distinguishing synthetic NPs from natural particles and characterizing them at very low cc.

<u>A combination of anal. techniques is always required!</u>

**Characterization For Size and Shape:** 1. Scan. E. Microscopy (SEM): hr. images of morphology and surface texture! + Energy-Dispersive X-ray Spectroscopy (EDS/EDX), elemental composition!

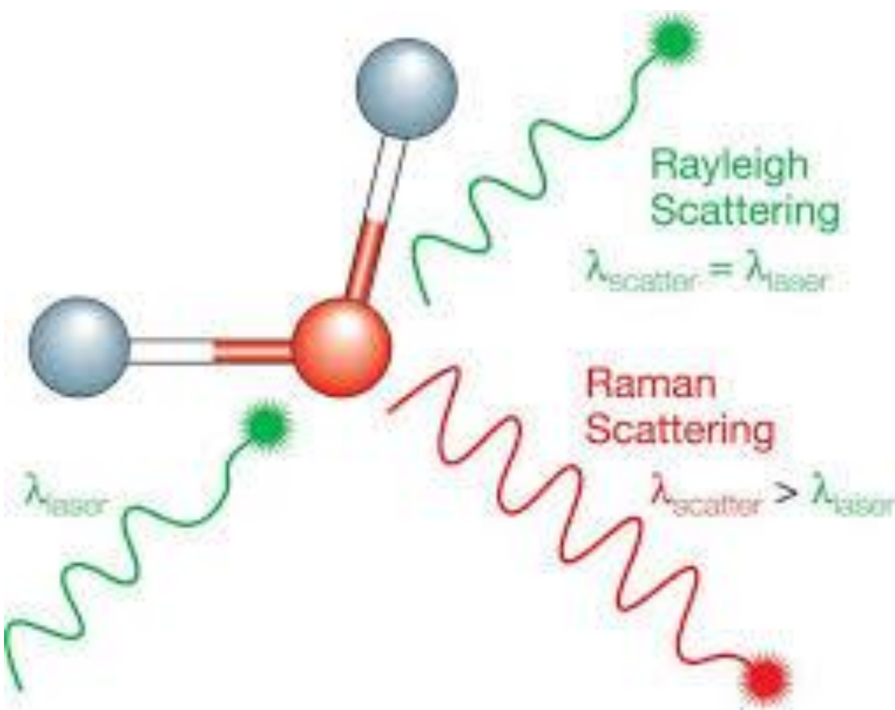
2. Transm. E. Microscopy (TEM): Higher resolution, allowing for the analysis of the internal structure and more precise size measurement of individual NPs.

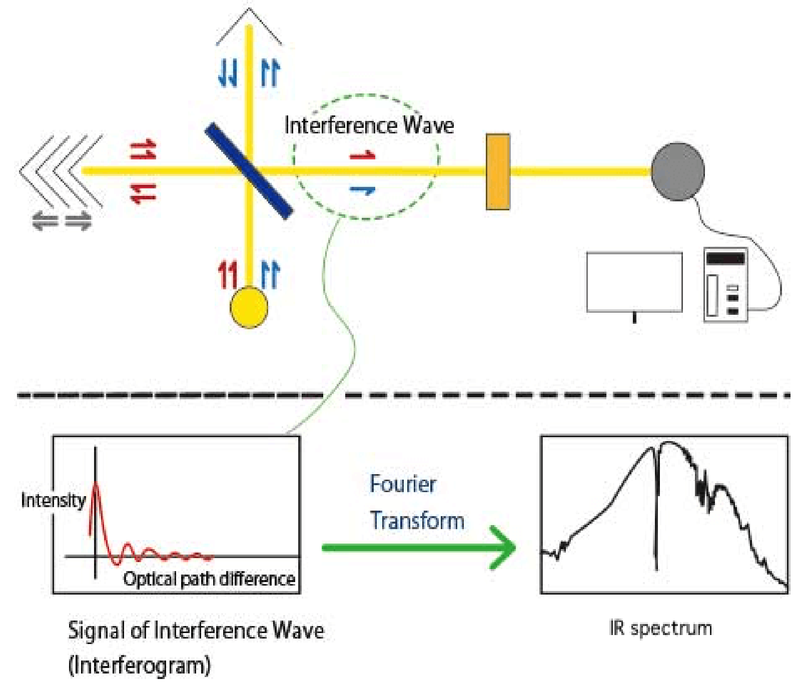
3. Atomic Force Microscopy (AFM): Provides 3D topographic maps, giving accurate information on particle height and shape.

# For Polymer Identification (Spectroscopy):

**Raman Microscopy:** The gold standard (~100nm-1µm). A unique molecular "fingerprint" spectrum for each polymer type (e.g., PET, PE, PS), allowing for precise identification. It can analyze single particles.

**Fourier-Transform Infrared Spectroscopy (FTIR)**: Similarly identifies polymers by their functional groups. Micro-FTIR is used for single-particle analysis. It excels at identifying specific chemical bonds.(10µm - ...)



# Flow cytometry

For counting and analysing MP and NP particles of size: 200nm- 100µm

1. **Fluorescent Staining (hydrophobic) → 2. Hydrodynamic Focusing→ 3. Laser Interrogation → 4. Detection & Analysis**

<u>Forward scatter</u> – particle size, <u>Side scatter</u> - complexity/internal granularity
<u>Fluorescence:</u> material identification based on intensity!

**High-Throughput!**

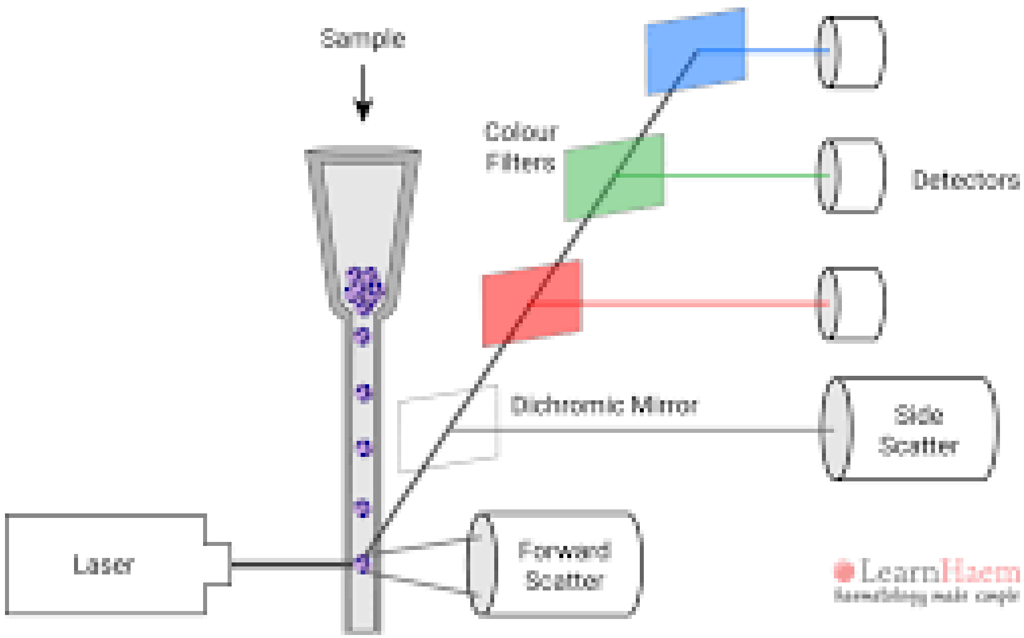
**Hi sensitivity!** (488nm, 405 nm)

**Large size range!**

**For small MP and large NP..**

**Excellent complementer for**

**Pyrolysis-GC-MS !**



# Sensitive Mass Quantification (Mass Spectrometry):

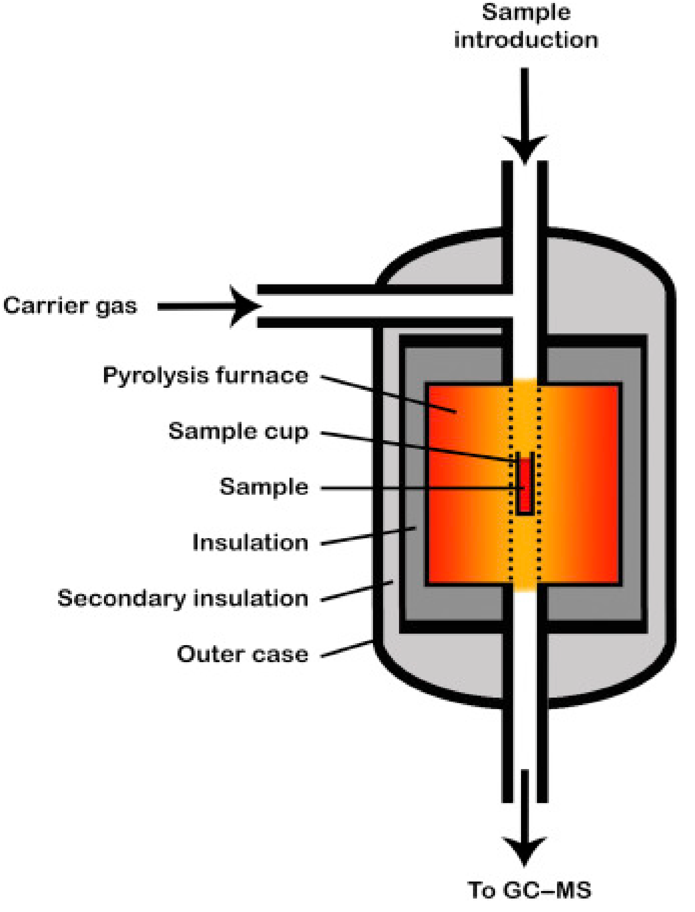
**Pyrolysis Gas Chromatography-Mass Spectrometry (Py-GC-MS):**

The sample is pyrolyzed (certain T) → characteristic polymer fragments are separated by gas chromatography and identified by mass spectrometry.

**Advantage:** determins the exact polymer

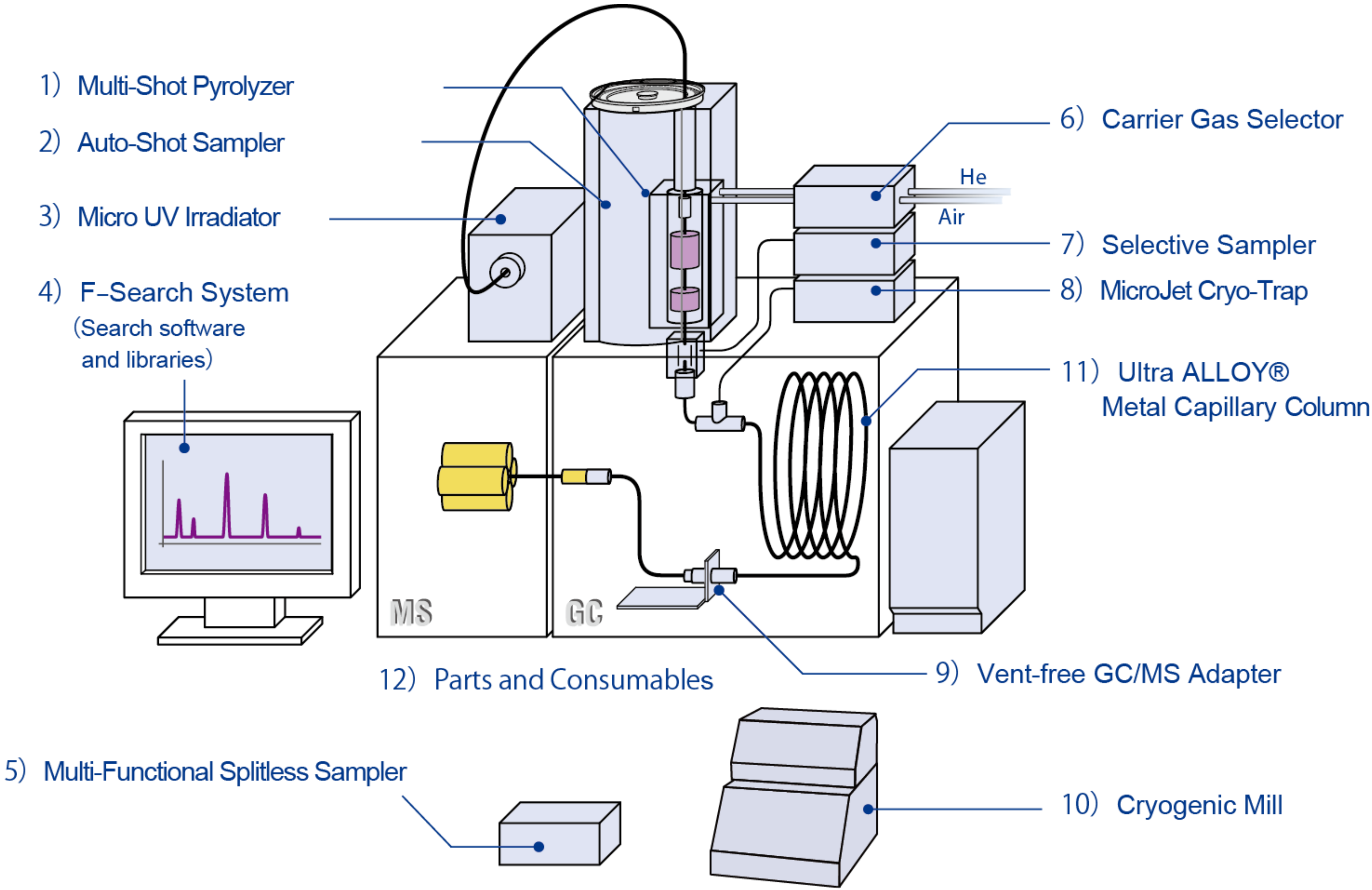
type and its mass concentration,

but it destroys the sample…



# Multi functional pyrolysis system

(https://www.frontier-lab.com/products/multi-functional-pyrolysis-system/200599/)



# Summary

1. Sampling of MP and NP in surface water is complicated and non standardised yet.

2. Lab preparation is crucial and different for each sample type !

3. Analytics of NP is very demanding, particle number and size distribution are often estimated.

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# Thanks for attention

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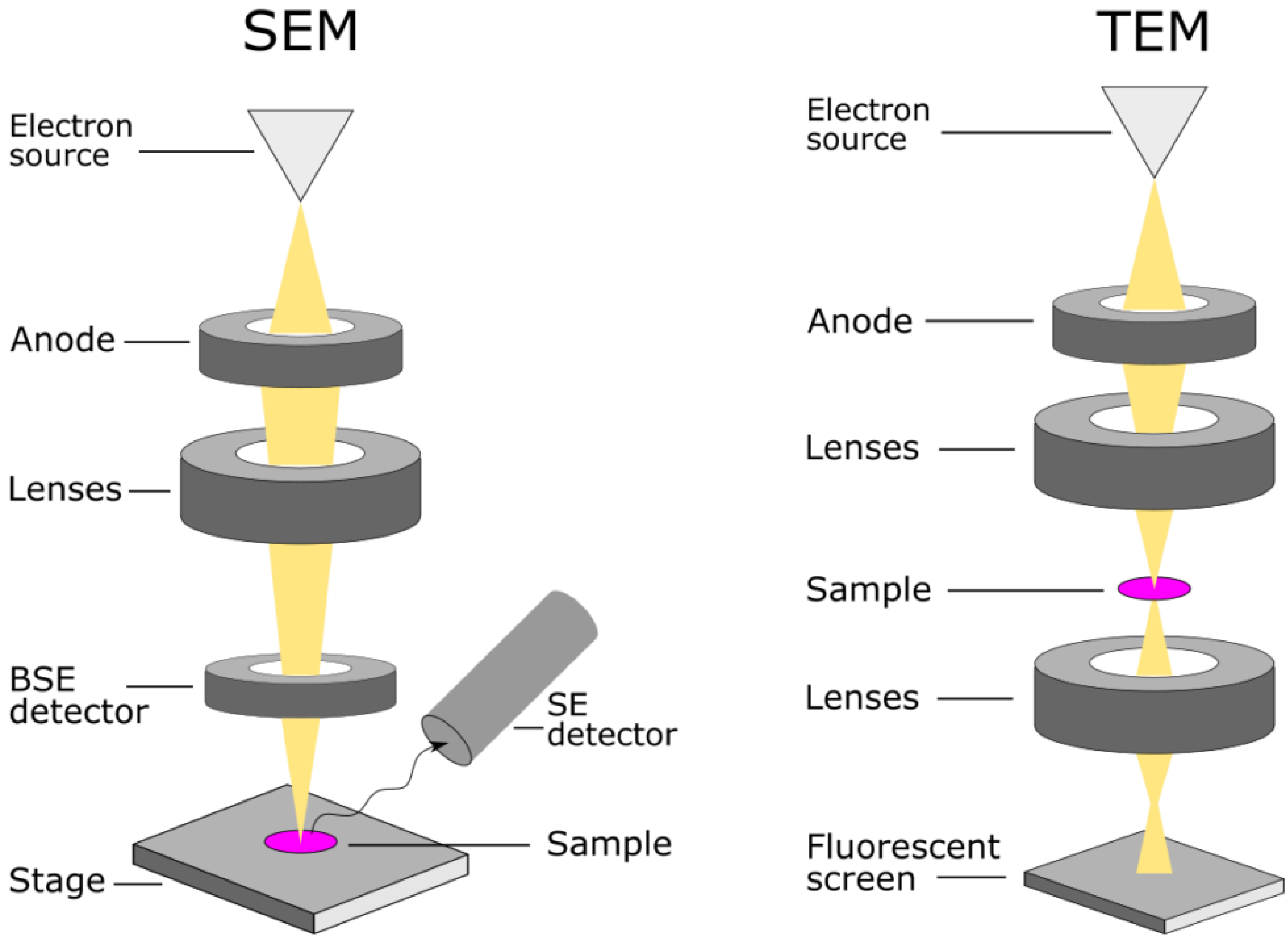

SEM
Electron source
Anode
Lenses
BSE detector
SE detector
Sample
Stage
TEM
Electron source
Anode
Lenses
Sample
Lenses
Fluorescent screen

# Pump and filter device operation

Sample

Sampler with filters: 150 micron and 20 micron

Lake, river..

Typical sample volume: 5-10 m³.

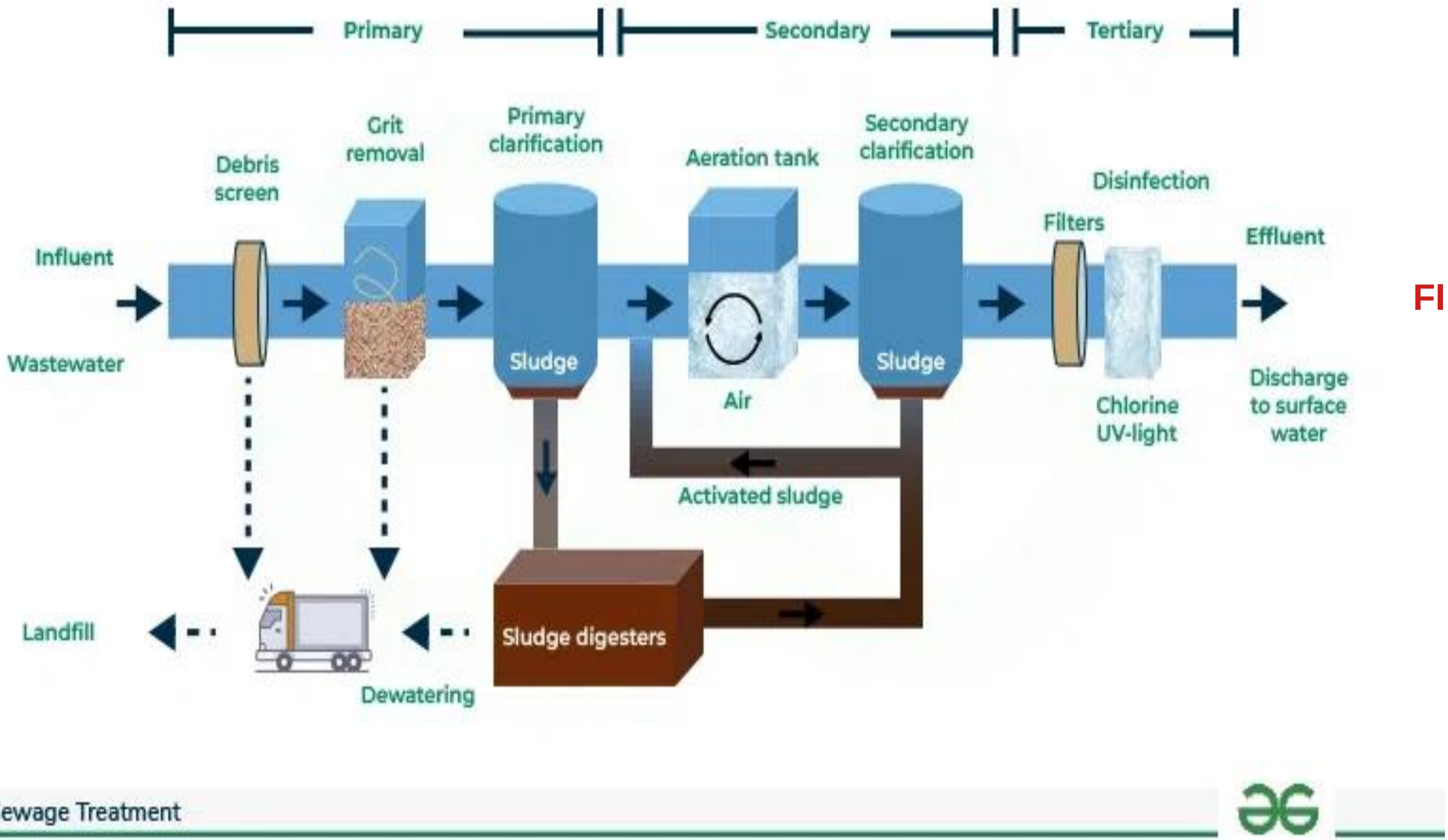
Filters are easy to exchange and serve as samplers with their closable housings.

# Filters for WWTP

Starting considerations:

1. Even small WWTP-s have a huge flow ~ 1000 m3/h ! ⟶ self cleaning device with the possible largest filter area
2. It must be simple and cheap to install and operate!
3. It should be efficient (filter out above 100 micron…)
4. Easy to repair and change filter tissue
5. In the case of havaria easy to eliminate or naturally bypass!

# How to install?




FILTER

# Operational conditions

Filter in the effluent channel

- Up to a certain high..

- self washing with tangential flow

- Only if there is no swimming sludge

Or foaming, no filamentous organisms

In the discharge and the secondary

Sedimentation works well!!

(no denitrification, NOM, foam...etc.)

Mechanical cleaning each month!