



# Methods used to determine macro -, micro- and nanoplastics in the environment

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Arctic Plastic Symposium,  
Reykjavik 22<sup>nd</sup> November 2023

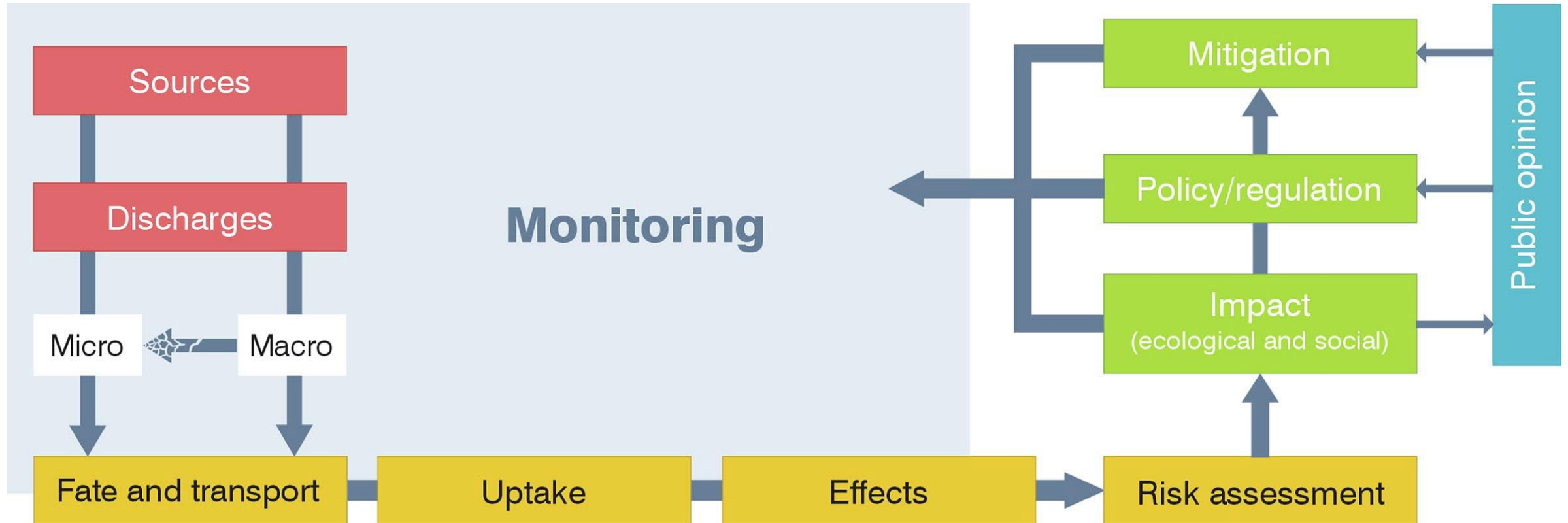


# Defining what we mean:

Developing methods to fit research or monitoring aims is a key step in plastic pollution control and management.

**Harmonisation of methods is paramount for monitoring.**

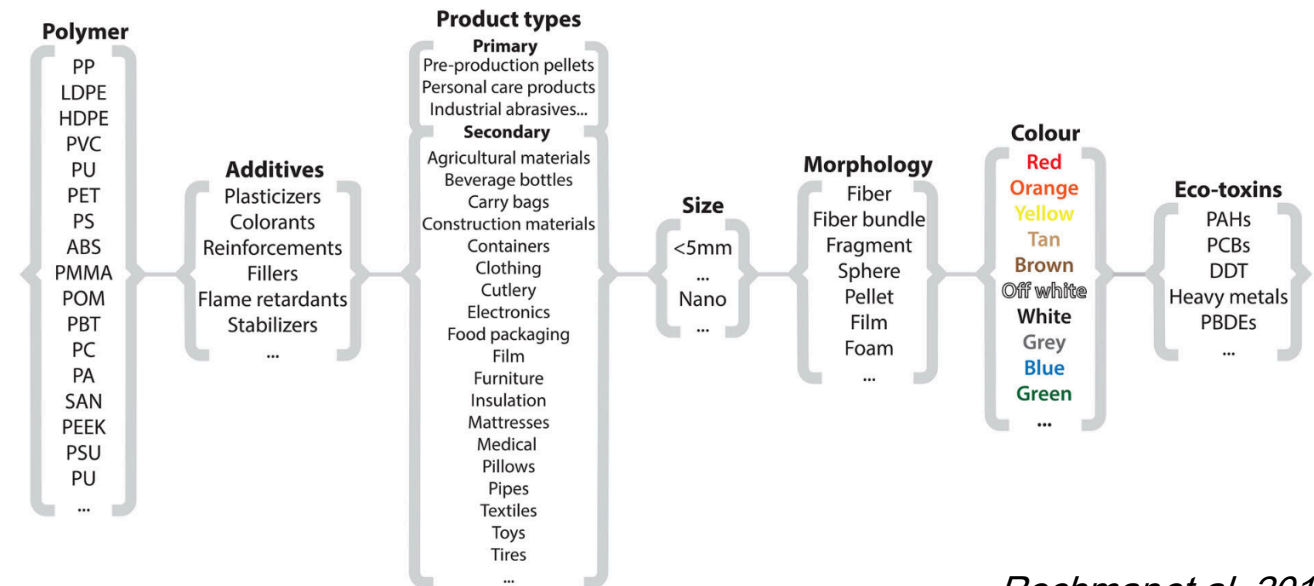
# Different research questions, different approaches



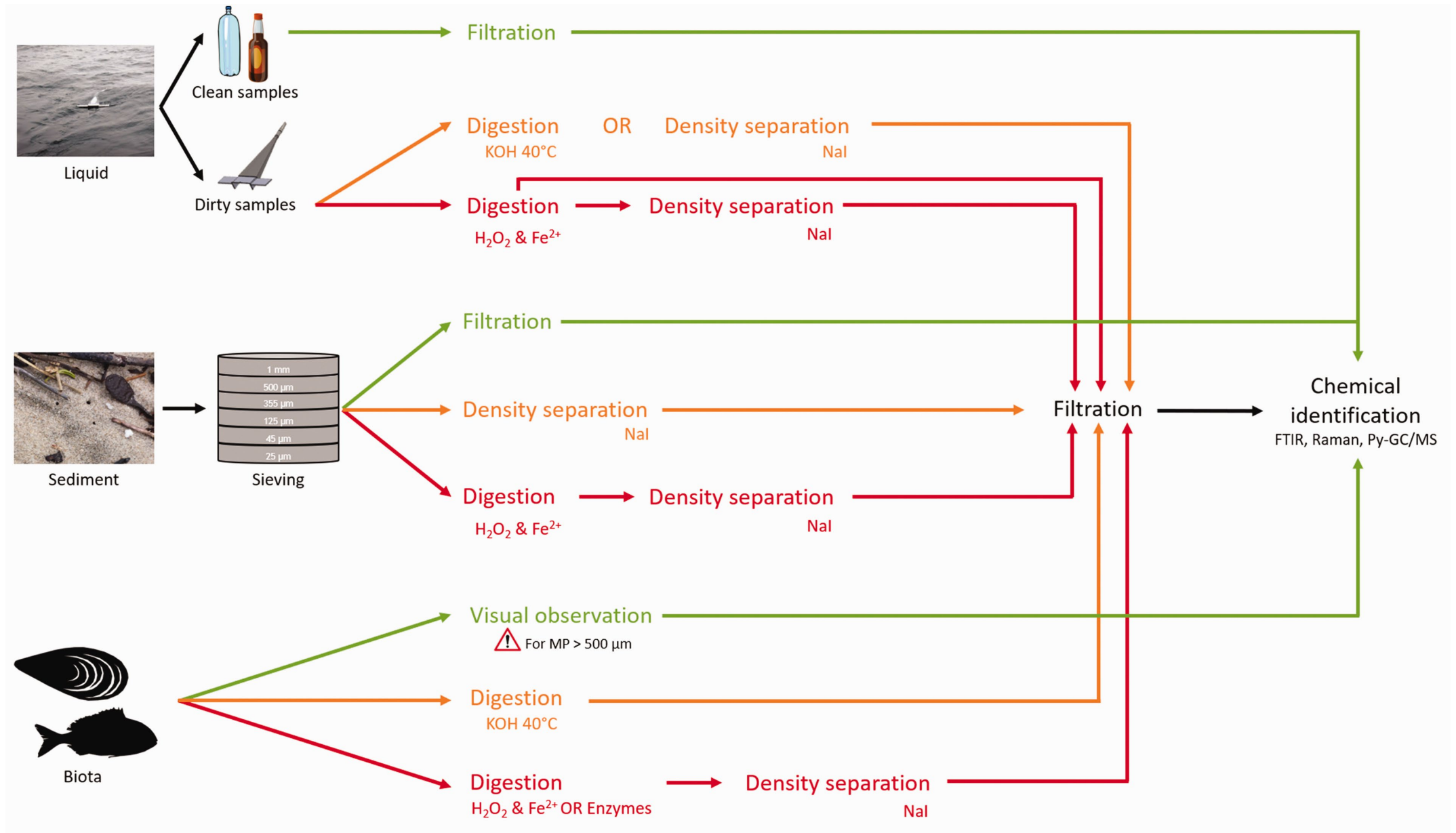
# Plastics research is increasing exponentially

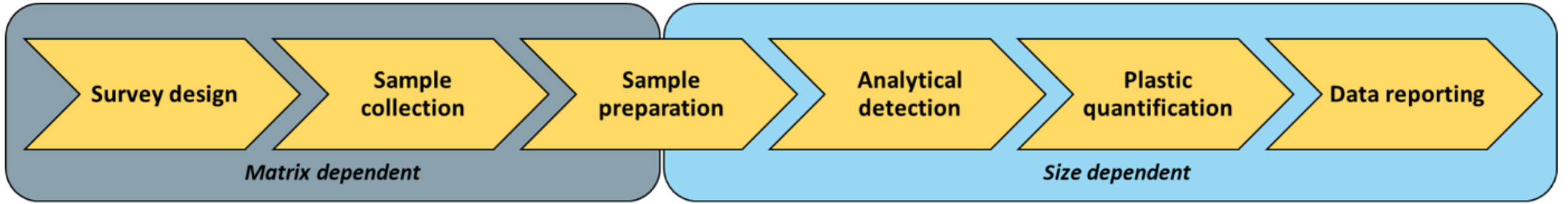
- Plastics are a ubiquitous contaminant - different ecosystems, matrices...
- Highly diverse nature of plastic pollution
- Still an evolving field

➔ various types of sampling and analysis approaches



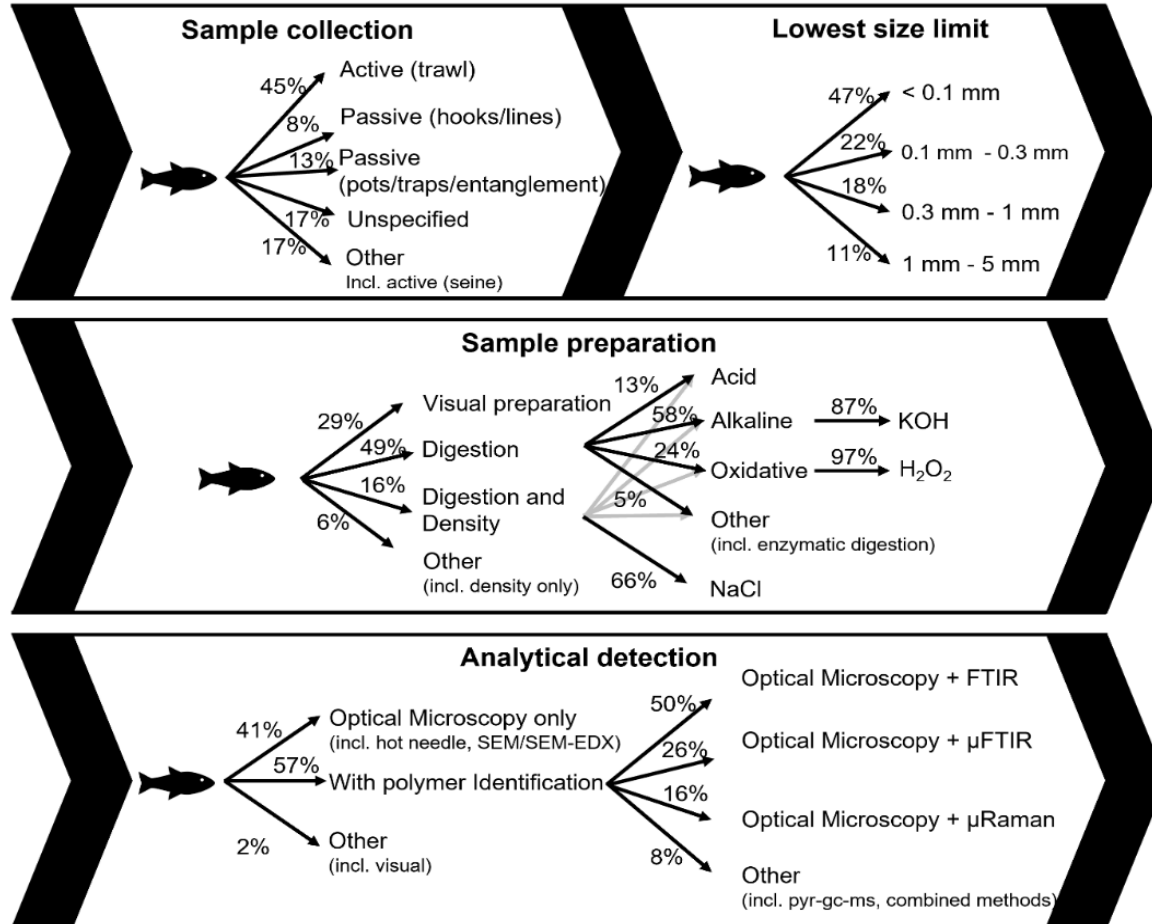
*Rochman et al. 2019*

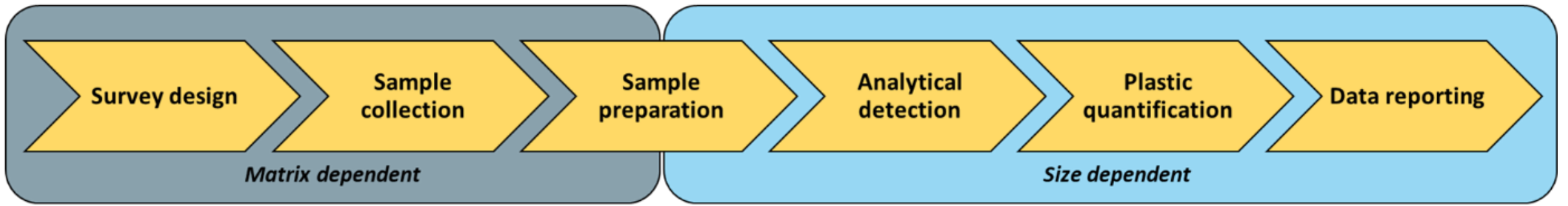




(Aliani et al., 2023) **EURO CHARM**

# Using biota as an example:





(Aliani et al., 2023) **EURO CHARM**

## First quantification of semi-crystalline microplastics in industrial wastewaters

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<sup>1)</sup> Department of Civil and Environmental Engineering Sciences, Institute IWAR, Chair of Wastewater Engineering, Technical University of Darmstadt, Germany



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### Plastic Debris in the Aquatic Environment

#### RELEASE OF PRIMARY MICROPLASTICS FROM CONSUMER PRODUCTS TO WASTEWATER IN THE NETHERLANDS

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<sup>†</sup>KWR Watercycle Research Institute, Nieuwegein, The Netherlands

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Environment International

journal homepage: [www.elsevier.com/locate/envint](http://www.elsevier.com/locate/envint)



Full length article

### Discovery and quantification of plastic particle pollution in human blood

Heather A. Leslie<sup>a</sup>, Martin J.M. van Velzen<sup>a</sup>, Sicco H. Brandsma<sup>a</sup>, A. Dick Vethaak<sup>a,b</sup>, Juan J. Garcia-Vallejo<sup>c</sup>, Marja H. Lamoree<sup>a,\*</sup>

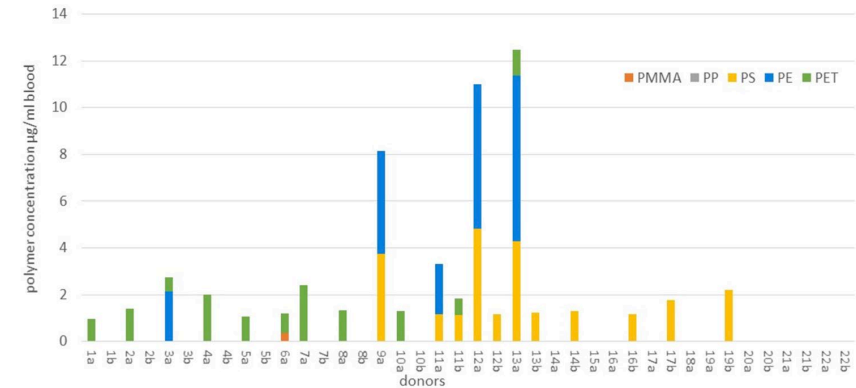
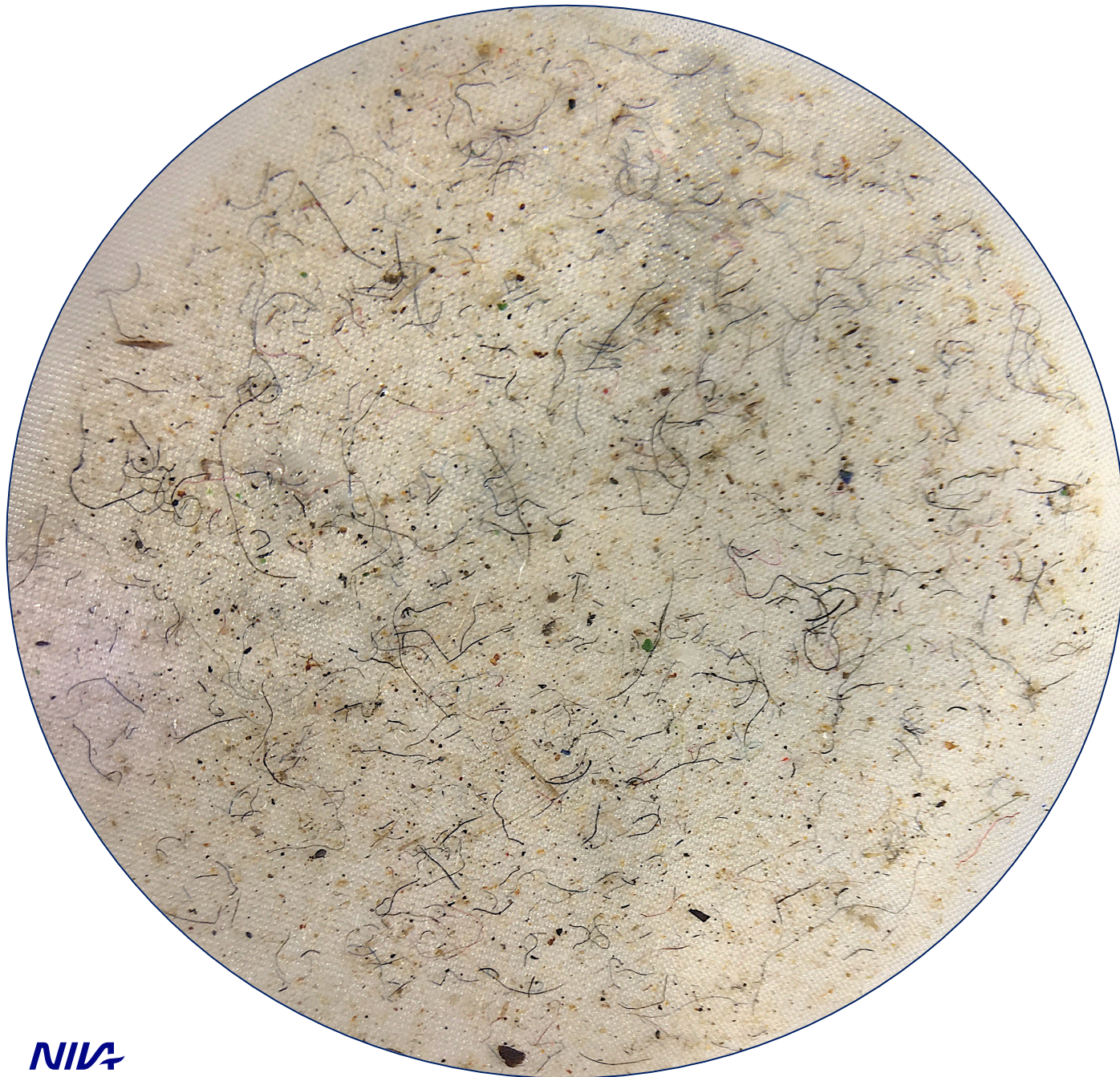


Fig. 1. Concentrations of plastic particles by polymer type in whole blood samples of 22 donors (duplicates a and b, except for No. 6, 9, 15 and 18). All values >LOQ.

**0.2-66 µg/l in wastewater effluent**

**1.6-12.5 µg/ ml = 1600-12500 µg/l in human blood (!!!)**

**Just one of the many  
challenges with plastic  
pollution research.....**





# Methods are important for (micro)plastic monitoring

- Require tailored methods for reliable detection and environmental enumeration
- Necessary to choose appropriate tool, or combination of tools
- Many standardised methods are developed south of the Arctic and ill-suited (Melvin et al. 2021)

## Methods must be adapted to the ecosystem

*Local site conditions*



*Proximity to anthropogenic activity*



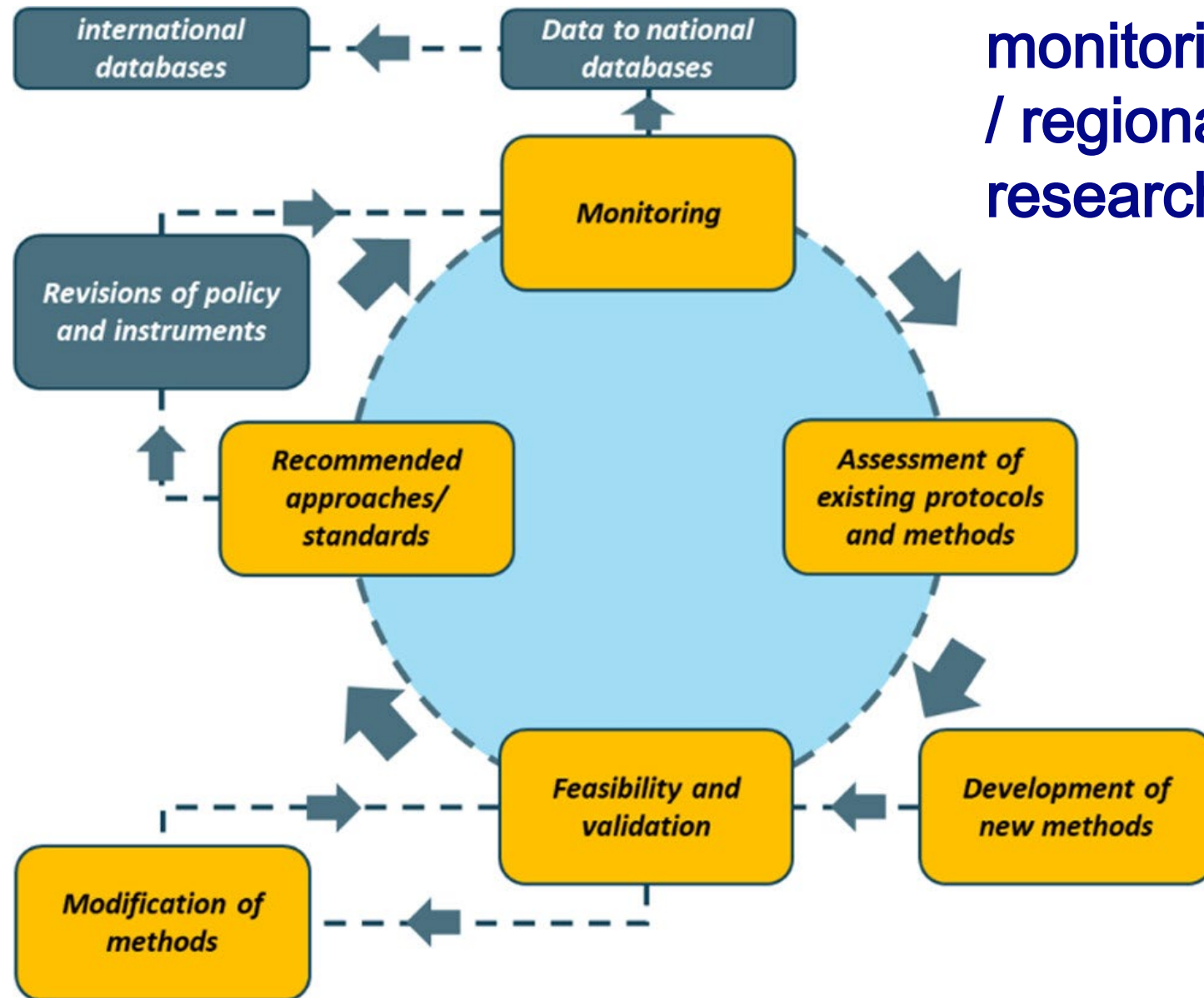
*J. Falk Anderssen/NIVA*

*Presence of fauna*



*Eric Baccega/NPL*

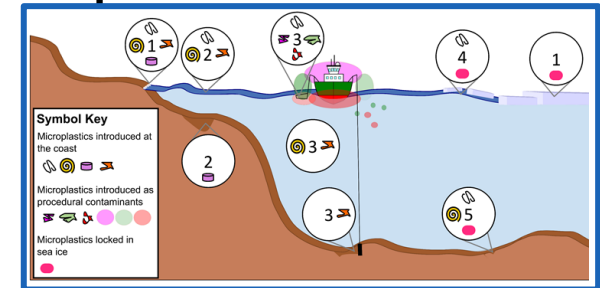
# Balance between monitoring (nationally / regionally) and research



# Value of multi -matrix monitoring

AMAP recommend a joint sediment and water approach is adopted.

- ✓ Can be carried out in same sampling campaign
- ✓ Provide complementary, but not overlapping, information
- ✓ Provide the most complete picture of plastic pollution



*Martin et al. 2022, Arctic Science*

## Water

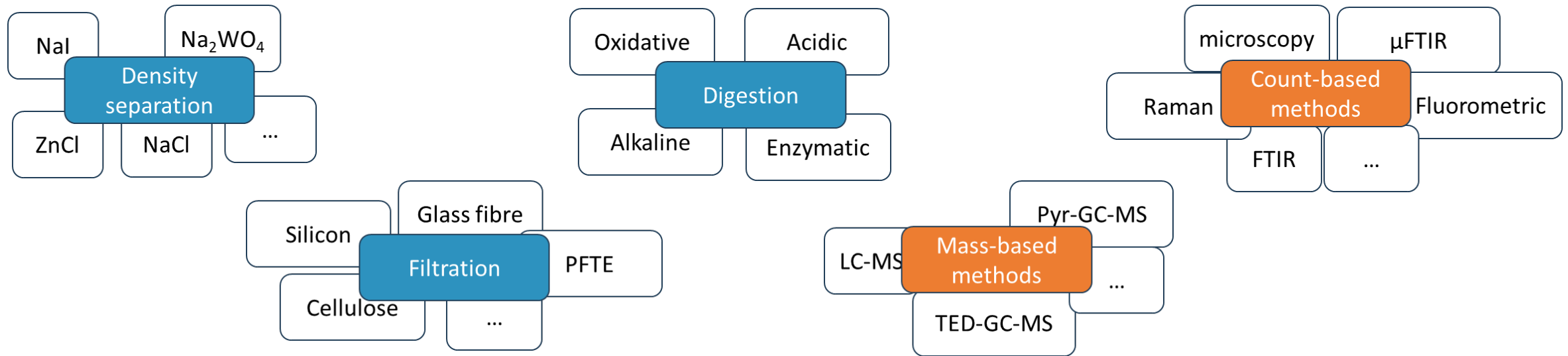
– potential to track rapid fluctuations

## Sediment

– spatially and temporally integrated signal

✗ Still biased to marine – integration of freshwater and terrestrial samples is important

# What method should we choose?

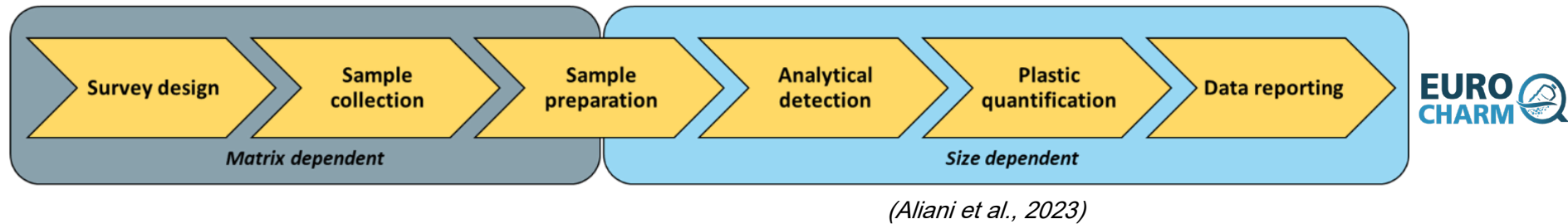


- How do we decide what is best?

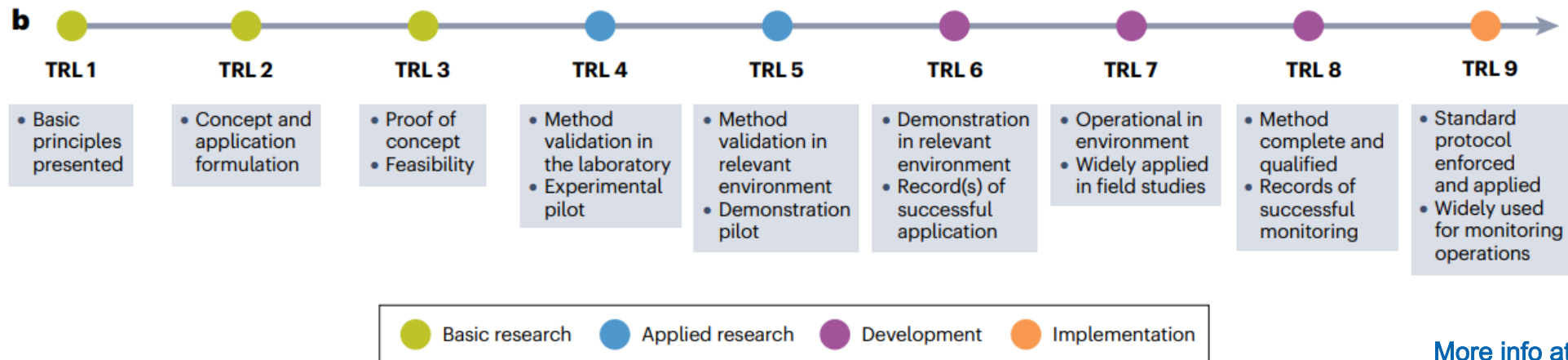
➔ Decisions for monitoring guidelines need to be informed by sound science, meet a minimum criteria, and allow data comparisons.

# Solutions for addressing the useability of methods

(1) Break the analytical elements into useable pieces: steps



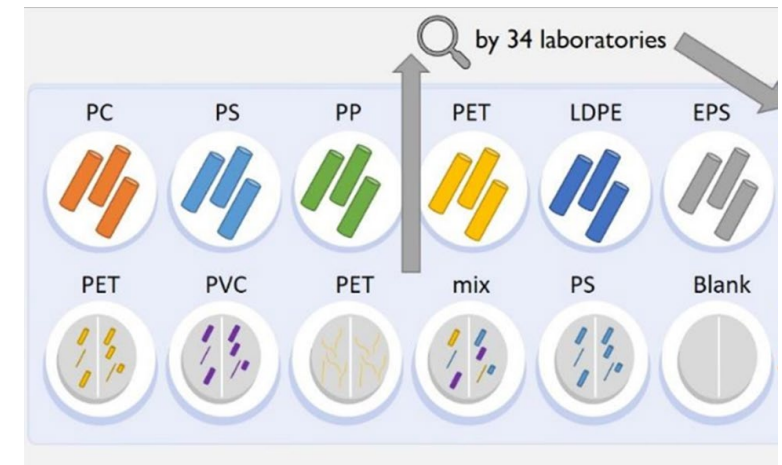
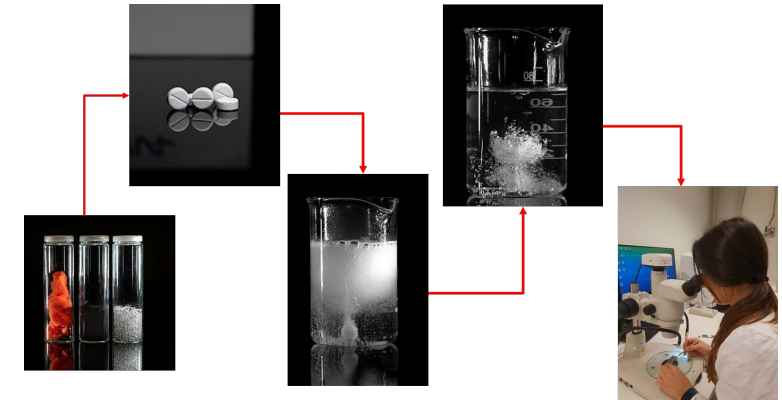
(2) Assess the reproducibility of each approach and the requirement for further research/development, or recommend for monitoring programmes



# Tools for validating methods

## (1) Recovery test / positive controls

- Spiking samples with a known quantity of polymers
- Challenge: generation of relevant reference materials
- Aim of the study, target size, shape, polymer

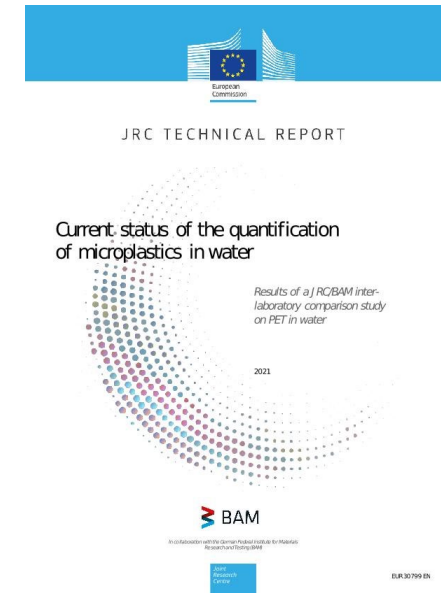


# Tools for validating methods

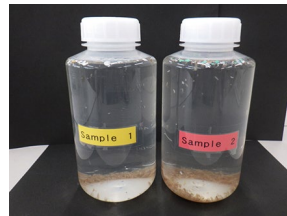
## (2) Interlabaotry comparison exercises (ILCs)

- Compare methods through ILCs looking at different aspects of the analytical approach

- Sampling
- Processing
- Analysis



PLASTIC BUSTERS MPAs  
Tsangaris et al., 2021, MPB, 164:111992



Ministry of the Environment – Government of Japan (2017)  
Isobe et al., 2019, MPB, 146:831-837



WEPAL-QUASIMEME/  
NORMAN network  
Van Mourik et al., 2021, STOTEN, 772: 145071

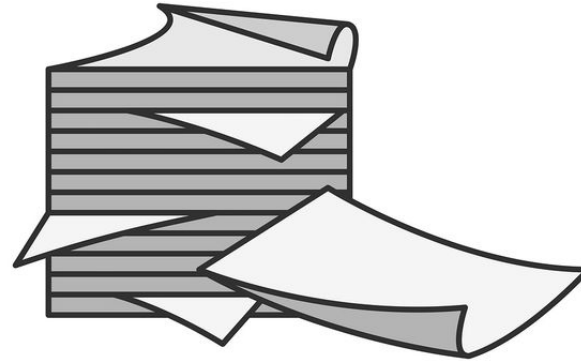


SOUTHERN CALIFORNIA COASTAL WATER RESEARCH PROJECT

De Frond et al., 2022, Chemosphere, 298:134282.

# Tools for assessing available methods

## (1) Critical reviews



## (2) Guidelines for QA/QC steps

54 S. E. SHUMWAY ET AL.

Special Issue: Microplastics

### Sampling and Quality Assurance and Quality Control: A Guide for Scientists Investigating the Occurrence of Microplastics Across Matrices

Susanne M. Brander<sup>1</sup>, Violet C. Renick<sup>2</sup>, Melissa M. Foley<sup>3</sup>, Clare Steele<sup>4</sup>, Mary Woo<sup>4</sup>, Amy Lusher<sup>5</sup>, Steve Carr<sup>6</sup>, Paul Helm<sup>7</sup>,Carolynn Box<sup>8</sup>, Sam Cherniak<sup>9</sup>, Robert C. Andrews<sup>9</sup>, and Chelsea M. Rochman<sup>10</sup>

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Table 2. The quality assurance and control (QA/QC) criteria used to assess the quality of research papers in Table 1.

QA/QC measures
Monitor contamination
1. Account and monitor for plastic used throughout collection, extraction, and identification
2. Procedural blank samples processed alongside collected samples (negative controls)
3. Monitor airborne contamination with a wet filter and/or an open petri dish in the field and laboratory
Prevent contamination
4. Prefilter liquid reagents into clean glassware
5. Use glass and metal instead of plastic whenever possible
6. Muffle glassware, glass filters, and metal (450 °C for minimum 5 hr) prior to use or rigorously clean glassware and equipment (acid wash protocol, multiple rinses with filtered water and ethanol)
7. Rinse glassware prior to use and in-between samples
8. Cover all containers (e.g., aluminum foil) when not in use
9. Wear cotton laboratory coats or non-synthetic clothing when interacting with samples
10. Use positive controls and test and report method recovery
11. Check filters for contamination under microscope and clean prior to use (not necessary if can be muffled)
12. Perform extractions in clean air environment (laminar flow hood, air-controlled space)
13. Identify polymers with chemical characterization method (e.g., FTIR, Raman, Pyrolysis-GC/MS)

Each QA/QC measure was assigned a number. If the study did not mention a particular QA/QC measure, the appropriate number was added to the criticism column in Table 1. The numbers assigned to each measure do not signify priority over the other measures. One hundred thirty studies were reviewed against this QA/QC list.



# Tools for assessing available data

## (3) Online platform – crowd sourced

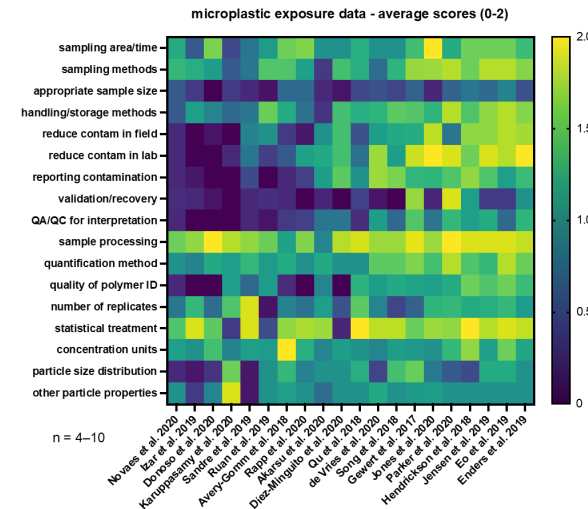
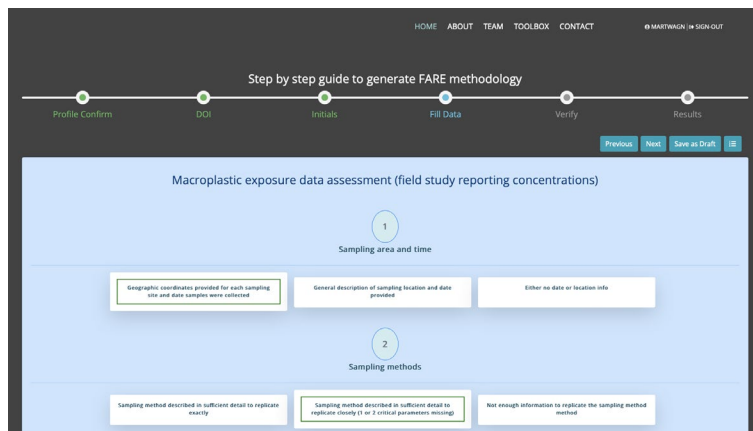


The FARE project has developed and tested a new toolbox specifically designed to assess the quality of existing macro- and microplastic exposure and effects data.

Insufficient quantity of exposure and hazard data for macro- and microplastic

Variable quality of existing macro- and microplastic exposure and hazard data

Lack of tools for assessing the quality of data



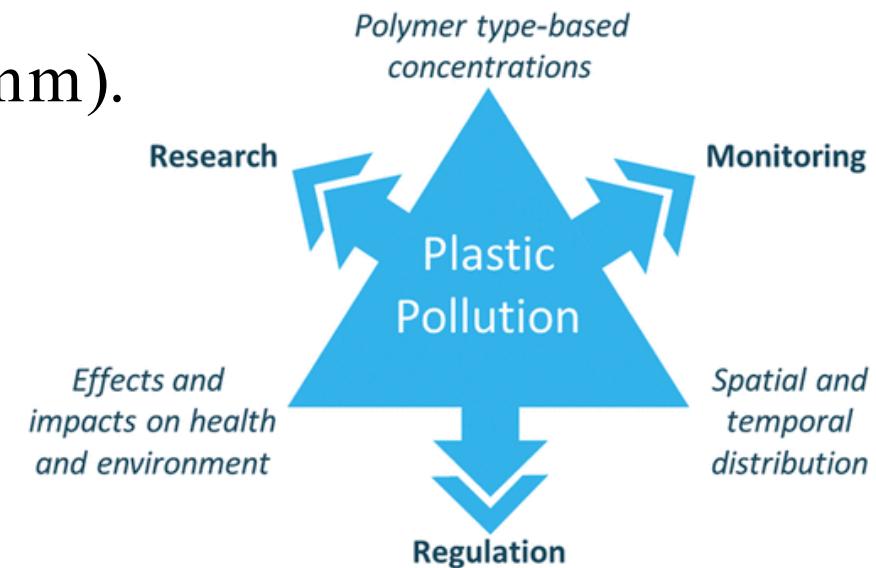
First steps towards an Assessment of plastic Risk to the Norwegian Environment

# Do we have enough methods yet?

Different research questions require different methods.

Method choices depend on rigorous examination of the science being published.

We already have methods ready for monitoring ( $>1$  mm).



# Thank you for listening

# Amy.Lusher@niva.no

**Comment**

<https://doi.org/10.1038/s43017-023-00405-0>

## Reproducible pipelines and readiness levels in plastic monitoring

Stefano Alarri, Amy Lusher, François Gaigani, Dorte Herzke, Vladimir Nikiforov, Sebastian Primpke, Lisa Roseher, Vitor Hugo da Silva, Jakob Strand, Giuseppe Sbaria, David Vanvermeert, Katrien Verlé, Bavo De Witte & Bert van Bavel

Check for updates

Flexible decision-making tools are needed to support action plans for plastics and other pollutants. Reproducible Analytical Pipelines (RAPs) and technological readiness levels (TRLs) will enable systematic validation and global harmonization of plastic pollution monitoring methods.

Plastic pollution is a wicked problem that spans all environmental compartments, with different magnitudes in space and time. A Global Plastic Treaty is under preparation with the ambitious goal of producing a set of legally binding tools aimed at stopping or reducing the flow of plastics into the environment. Policymakers and scientists are looking forward to endorsing monitoring plans based upon ready-to-deploy methods for different analytical scenarios. However, plastic monitoring is facing reproducibility crisis. Despite attempts to define monitoring guidelines, there are still widely accepted monitoring frameworks. Tools and protocols have been developed to quantify plastic pollution, but these methods often provide incompatible results, even if applied to the same environmental matrix.

Expansions and advances in the adoption of best monitoring practices, a flexible method validation framework based on reproducibility, reliability, and regulatory urgency are required. In this Comment, we propose the application of RAPs and TRLs as a tool to support policy and technical decisions about plastic monitoring.

**Reproducible analytical pipelines**

RAPs are a set of assessment processes used to identify best practices needed to assure that coding pipelines and data processing are standard, audited, quality controlled and reproducible. The concept was first introduced to manage workflows in software engineering and it is now widely applied to manage industrial processes. RAPs are especially helpful for multistep workflows like many plastic monitoring methods, providing modularity as a possible solution.

Agreement on plastic monitoring guidelines is traditionally considered a unique, solid and complete path dedicated to a single matrix and particle size. Moving forward, we advocate framing these workflows as modular RAPs, where any methodological step is separately evaluated and then implemented, saving money and time compared with evaluating a full pipeline.

Plastic monitoring can be divided into six modules in the RAP: survey design, sample collection, sample preparation, analytical detection, quantification, and data reporting (Fig. 1). Important information can be extracted when every step in RAPs is investigated

**Technological readiness level**

The TRL scale classifies technology or methods into basic research (TRL1–3), applied research (TRL4–5), in development (TRL6–8) and implementation (TRL9) phases (Fig. 7). Where a technology falls on the scale is usually assessed by experts' opinion. In plastic research and monitoring, TRL can be based on the functionality, reliability, usability, efficiency, maintainability, accessibility, cost, and portability of a method. These aspects could be ranked and assessed using a SWOT (strengths, weaknesses, opportunities, and threats) approach. The outputs of these systematic assessments should be freely available to relevant stakeholders, deposited in suitable open access repositories such as the CPAL digital platform, and reported and updated on a regular basis. This information will support informed decision making, but before implementation, scientific, technical, logistical, environmental, and ethical constraints must be considered.

**Merging RAPs and TRLs**

The TRL approach should be simply applied to create full plastics monitoring guidelines; however, we argue that applied singularly to each step in a RAP, it has the potential to greatly improve and accelerate the selection, evaluation, and adoption of large-scale plastic monitoring programmes.

For instance, no methodological standards exist for microscopic sampling in the air (for example, using active versus passive samplers, monitoring dry versus wet deposition, and appropriate sampling volume and duration). Therefore, air sampling related modules would have a TRL 3, as they are still at a basic research level and not yet ready for monitoring recommendation. Conversely, analysis of samples with Fourier transform infrared (FTIR) spectroscopy and/or wet chemistry sampling method or matrix, and commonly used for plastic polymer identification (FTIR) would have a TRL 9 and could be recommended for air monitoring guidelines. Overall, the low TRL of the sampling modules prevents the definition of full standard pipelines for monitoring microplastics in the air, but breaking the method down into the

**nature reviews** earth & environment

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Findings

## Finding the Balance between Research and Monitoring: When Are Methods Good Enough to Understand Plastic Pollution?

Amy L. Lusher\* and Sebastian Primpke

Cite This: *Environ. Sci. Technol.* 2023, 57, 4033–4039

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**ABSTRACT:** Plastic pollution is an international environmental problem. Despite its scale, the public, policymakers, and scientists are struggling to understand the problem, let alone address it. This Comment provides a framework for understanding the balance between research and monitoring, and offers guidance on how to assess the readiness of monitoring methods. The authors argue that the current state of plastic monitoring is a 'wicked problem' that requires a holistic approach. The authors propose a framework for assessing the readiness of monitoring methods, and offer guidance on how to assess the readiness of monitoring methods. The authors argue that the current state of plastic monitoring is a 'wicked problem' that requires a holistic approach. The authors propose a framework for assessing the readiness of monitoring methods, and offer guidance on how to assess the readiness of monitoring methods.

**KEYWORDS:** plastic litter, debris, environmental pollution, harmonization, microplastics

**Plastics are increasingly reported in environmental samples across the globe. This ubiquitous and heterogeneous environmental contaminant is receiving attention from researchers, citizens, and policymakers. Monitoring plastic pollution is positioned high on agendas of international governing bodies, with the realization of a new legally binding global instrument on plastic pollution during the 2024 Session of the United Nations Environment Assembly (UNEA 5.2). This is mirrored by calls from regional and national agencies to understand the extent of this pollutant in their local environments. Much attention is directed toward the perceived harm plastic pollution could cause to the environment, including animals and humans. It is imperative that any potential risk is assessed appropriately and that mitigation measures can be monitored accordingly.**

Monitoring is necessary to address questions about the presence and abundance of plastics in the environment. As monitoring is the repeated measurement of variables to detect a change, it requires significant data gathering, analysis, and archiving. This cannot be achieved until appropriate approaches are chosen to address well-defined goals which should be subject to regular reports. Frameworks and instruments to conduct environmental assessments must be tailored with a clear purpose before monitoring can be initiated. For example, UNEA suggests many activities concerning

**ACS Publications**

**EUROCHARM**

European Quality Controlled Harmonization Assuring Reproducible Monitoring and assessment of plastic pollution

# EUROCHARM

Tools for assessing the methods and protocols used in the analysis of nano-, micro-, and macroplastic

**EUROCHARM Short Report**

Date: 07.10.2023

This project has received funding from the European Union's Horizon 2020 Coordination and Support Action under Grant Agreement No 101003805. The sole responsibility for the content of this document lies with the author and in no way reflects the views of the European Union.

**EUROCHARM**

European quality Controlled Harmonization Assuring Reproducible Monitoring and assessment of plastic pollution

# EUROCHARM

Standard measuring procedures for policy and legislation (baselines and thresholds) D3.3

Date: 31.08.2023  
Deliverable Identifier: D3.3  
Document Version: 1.0

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**EUROCHARM**

European quality Controlled Harmonization Assuring Reproducible Monitoring and assessment of plastic pollution

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Short report on methods and protocols for the analysis of nano-, micro-, and macroplastic in water samples

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