# Detection of manufactured nanomaterials in complex environmental compartments – An expert review







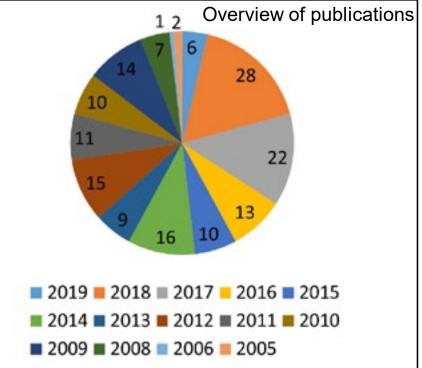
Stefan Schymura<sup>1</sup>, Heike Hildebrand<sup>1,2</sup>, Doris Völker<sup>2</sup>, Kathrin Schwirn<sup>2</sup>, Karsten Franke<sup>1</sup>, Cornelius Fischer<sup>1</sup> <sup>1</sup> HZDR, Institute of Resource Ecology, Leipzig, Germany <sup>2</sup> UBA, Dessau-Roßlau

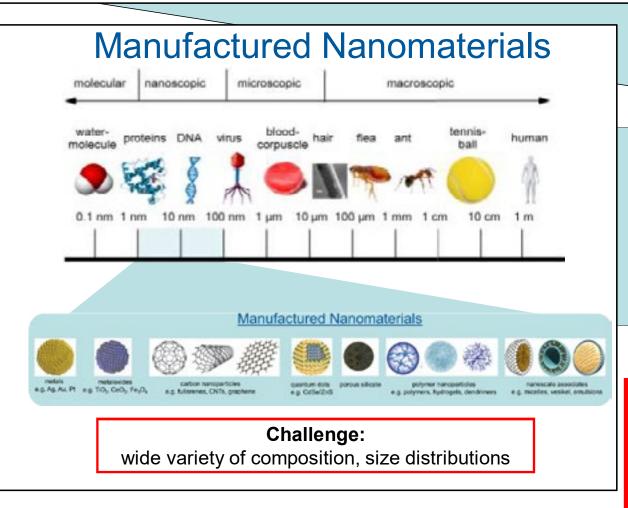
## **Motivation**

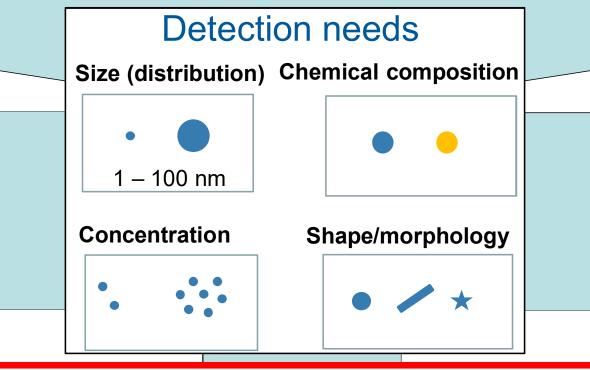
Despite considerable scientific efforts, the selective detection of manufactured Nanomaterials (NMs) in environmental compartments is still a very complex and challenging task. An expert review of the literature has been conducted on behalf of the German Environment Agency (UBA) to identify relevant methods for nanomaterial detection in complex media in the context of environmental monitoring and a need for action was concluded from the existing body of work.

## Methodology

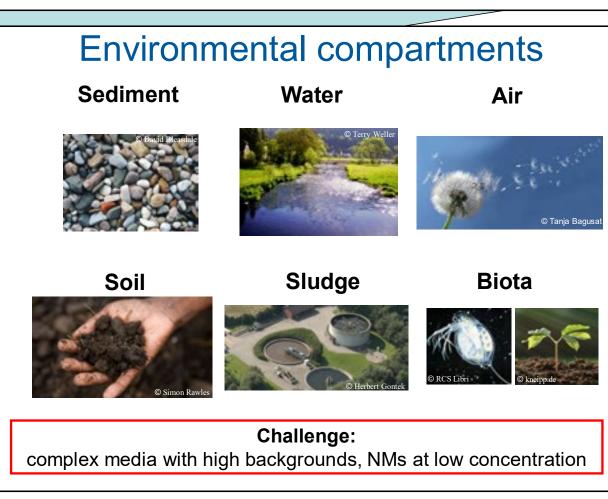
- •Web of Science Literature Review:
  - →> 150 publications
  - →> 10 000 sources
- Expert interviews
- Conference proceedings
- Project reports





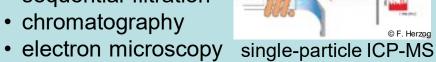


**Needs for environmental monitoring:** Methods with high throughput for the detection, characterization and quantification of nonspecific NMs at minimal concentrations in complex media against high particulate and elemental backgrounds.



# Size (distribution)

- sp-ICP-MS
- light scattering
- sequential filtration chromatography



## Concentration

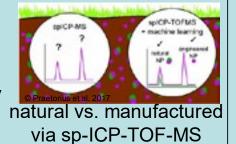
- sp-ICP-TOF-MS
- sp-ICP-MS • ICP-MS/AES
- light scattering
- electron microscopy

# sp-ICP-TOF-MS

## sp-ICP-TOF-MS

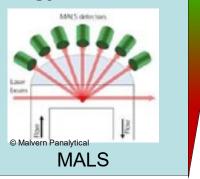
**Chemical composition** 

- sp-ICP-MS
- ICP-MS/AES
- electron microscopy + EDX



# Shape/morphology

 multiple angle light scattering (MALS) electron microscopy



→ Complete information can only be provided with a combination of different detection methods ← → Extraction of NMs from complex matrix necessary ←

→ Size fractionation advisable ←

#### **Extraction**

- Cloud point extraction (CPE) → micelle mediated extraction of specific size ranges
- Ultrasound assisted extraction → extraction media: water,
- alcohol, surfactant solution, toluene, etc. → validated recovery rates
- needed
- Digestion
  - → destruction of complex matrix
  - → conc. acids/bases
  - → enzymes
  - → possible destruction/ alteration of NMs

## Comprehensive scheme for NM detection in a combined approach 3) Size fractionation (e.g. AF4) cross flow 1) Sampling 4) Multi-method analysis 2) Extraction TEM/SEM **MALS** sp-ICP-TOF-MS number concentration • shape information • aggregation elemental composition · surface charge 5) Computational analysis

#### **Size fractionation**

- Chromatography
  - → asymmetric flow field-flow fractionation (AF4)
- → hydrodynamic chromatography
- → size exclusion chromatography
- → NM need to be extracted and stabilized
- Centrifugation
  - → simple technique for removal of NM from solution
  - → sensitive to NM shape, surface coatings
- Filtration
- → sequential filtration of extracted samples
- → simple technique
- → risk of loss of NMs

## **Need for action** Short term

- Identification of most relevant NM/compartment
- Development of extraction techniques for specific NM
- Choice of combined approach/SOP for specific NM
- Standard reference samples

### Medium term

- Development of extraction techniques for NM classes
- Combined approach for wider NM classes International round robin tests for validation
- Automation of experimental steps/combined data handling

### Long term

- Library of extraction techniques
- Automation/combination in single technical solution
- Library of natural/manufactured NM data for routine detection

H. Hildebrand, S. Schymura, K. Franke, C. Fischer: "Analysis of studies and research projects regarding the detection of nanomaterials in different environmental compartments and deduction of need for action regarding method development", UBA Texte 133/2019.

available from: https://www.umweltbundesamt.de/publikationen/

