Definition of micro and nanoplastics & analytical challenges

Joint Research Centre
Consumer Products Safety Unit (F.2)

Douglas Gilliland

The general problem – plastic waste

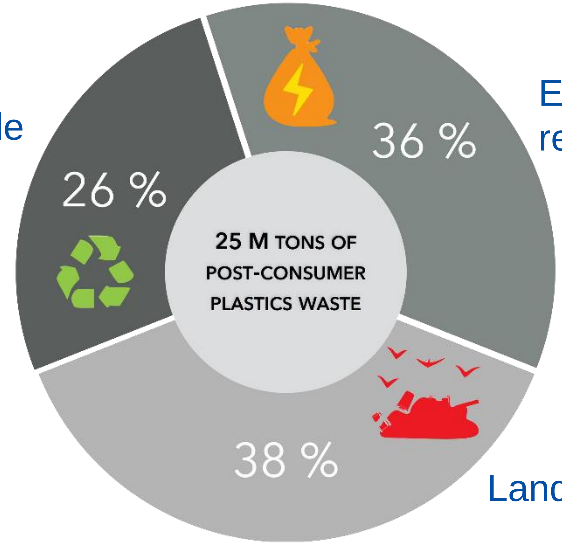
Microplastics and their origins

Globally, 5 to 13 million tons of plastics leak into the environment every year.

Slow plastic degradation
(up to 400 years) => Continuous build-up

Recycle

Energy recovery



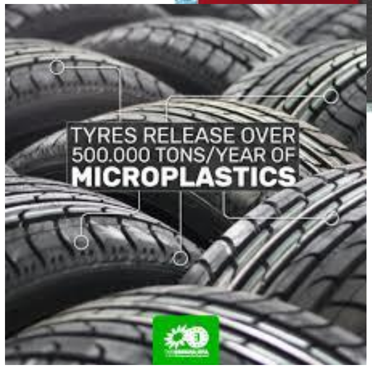
Land fill

Treatment of post-consumer plastics waste in 2012, in the European Union
https://www.plasticseurope.org/en

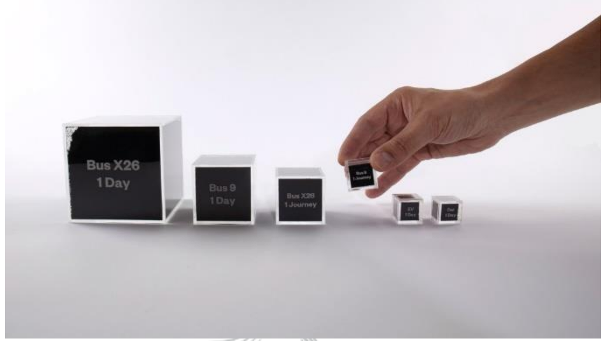
Garbage patch "Islands" of plastic

Land fill outflow

Textile Fibres

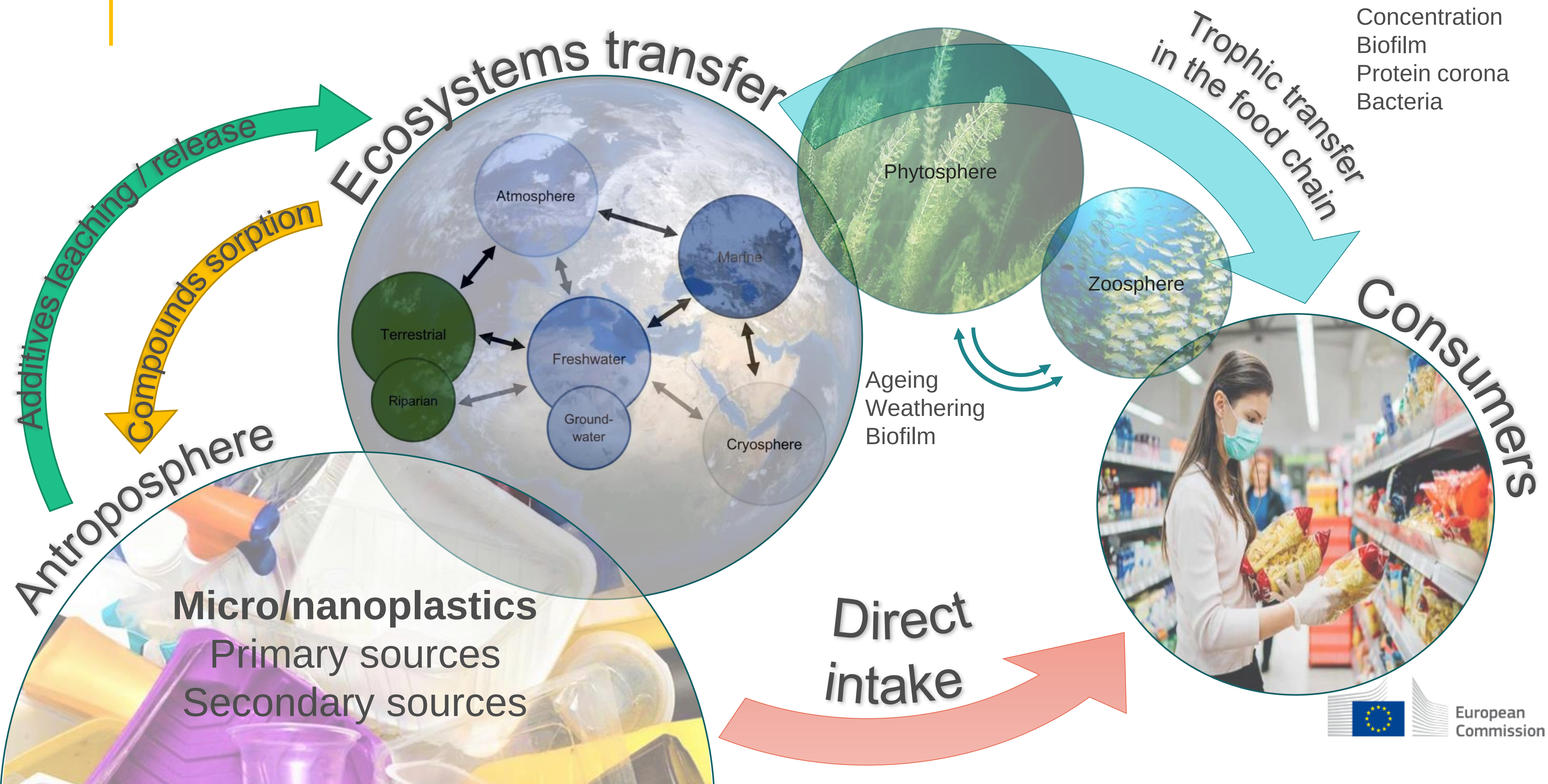


Tyre Wear



Tyre release in use

The dynamic journey of microplastic's towards the consumer



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Intense Media Attention

Generating public alarmism but ….

- Exposure levels not known
- Effects and hazards poorly understood

Balanced and informed scientific reality check required

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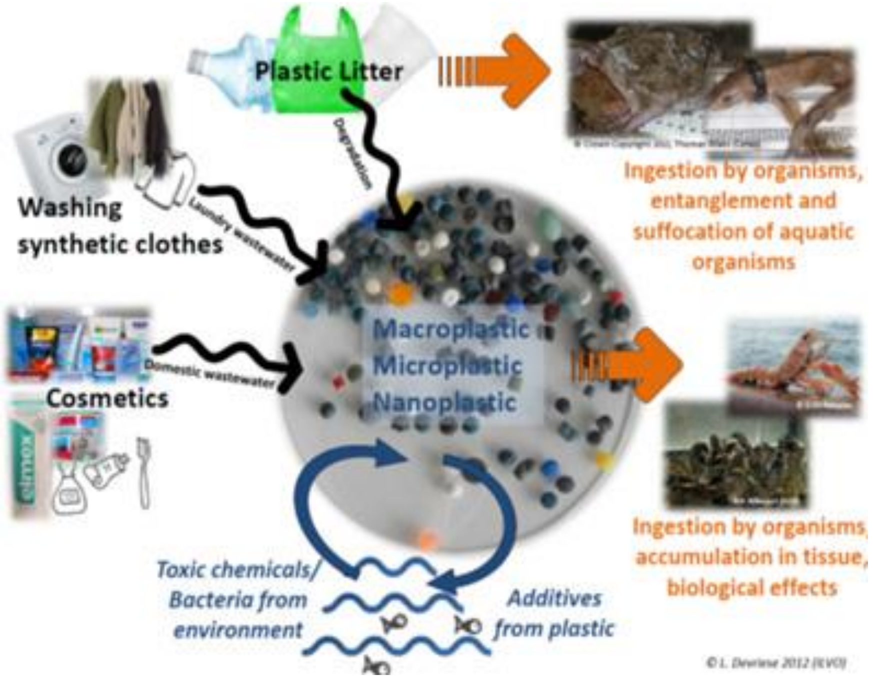
Micro and Nano-plastics pollutants

What are they?

- Primary particles (<5mm):deliberately manufactured particles from e.g. printing inks, paints spray, cosmetics, injection mouldings and abrasives etc.
- Secondary particles (<5mm): from mechanical, chemical and light induced breakdown of bulk and microplastics litter as well as tyre wear debris and fibres from textiles



Lisa Devriese : The Institute for Agriculture and Fisheries Research (ILVO) .

Chemistry and Physics may not be the only natural generators of nanoplastics : Arctic Krill - natural digestive "mill“.

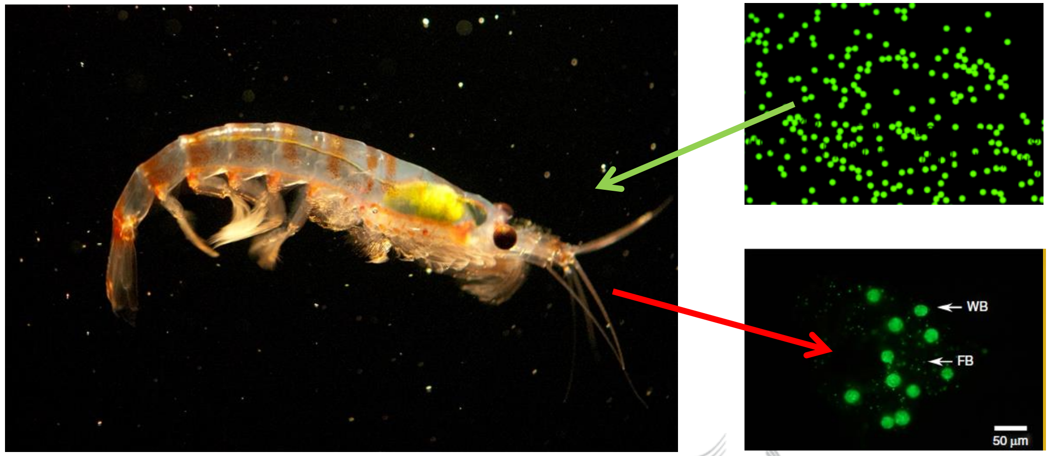
ARTICLE

DOI: 10.1038/s41467-018-03465-9 OPEN

Turning microplastics into nanoplastics through digestive fragmentation by Antarctic krill

Amanda L. Dawson[1], So Kawaguchi[2], Catherine K. King[2], Kathy A. Townsend[3], Robert King[2], Wilhelmina M. Huston[4] & Susan M. Bengtson Nash[1]

NATURE COMMUNICATIONS | (2018)9:1001 | DOI: 10.1038/s41467-018-03465-9 | www.nature.com/naturecommunications



Fragmentation of microplastics => nanoplastics

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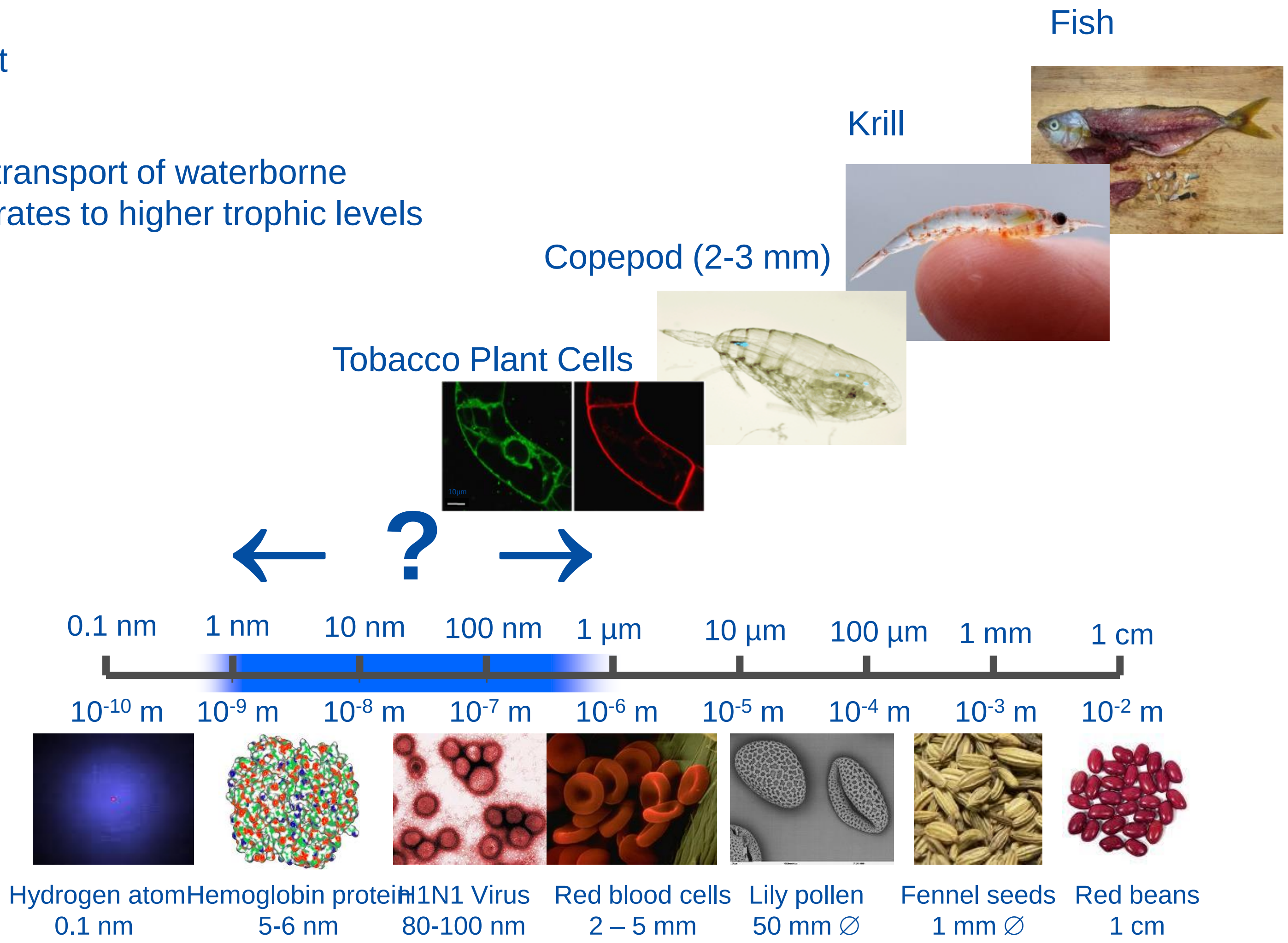
Micro and Nanoplastic pollutants

What are the main concerns?

- Harmful to wildlife and fish in the environment
  - Directly or via the food web
- Vector for the absorption, concentration and transport of waterborne chemical pollutants (eg. PAHs) from invertebrates to higher trophic levels
- Trojan horse for ingestion of plastic additives
- Transporter of bacteria
- Potential effects on human health
  - Enter via the food web
  - Presence in drinking water
  - Air pollution



Major knowledge gaps

- Environmental and human exposure levels
- Internalization and biological effects
- Interaction at cellular level

Independent Scientific Reviews: Key scientific issue - analytics

Knowledge Gaps – general considerations

- *"……there is a need for improved quality of methods and a need for international harmonisation of the methods that are used to measure and assess MP concentrations and exposure …."*

- *"…..The presence of nanoplastics in nature is generally considered highly plausible; however, there is very limited evidence from measurements, as adequate analytical methodology is still lacking….."*

- *"……For nanoplastics, the information gap is even larger. As the nature and concentrations of nanoplastics in the environment have not been measured yet, we do not know anything about the importance of nanoplastics for the total chemical risks posed by fragmenting microplastic…"*

WHO

EFSA

SAM

SAPEA

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Microplastics in food

Literature: Many diverse reports.

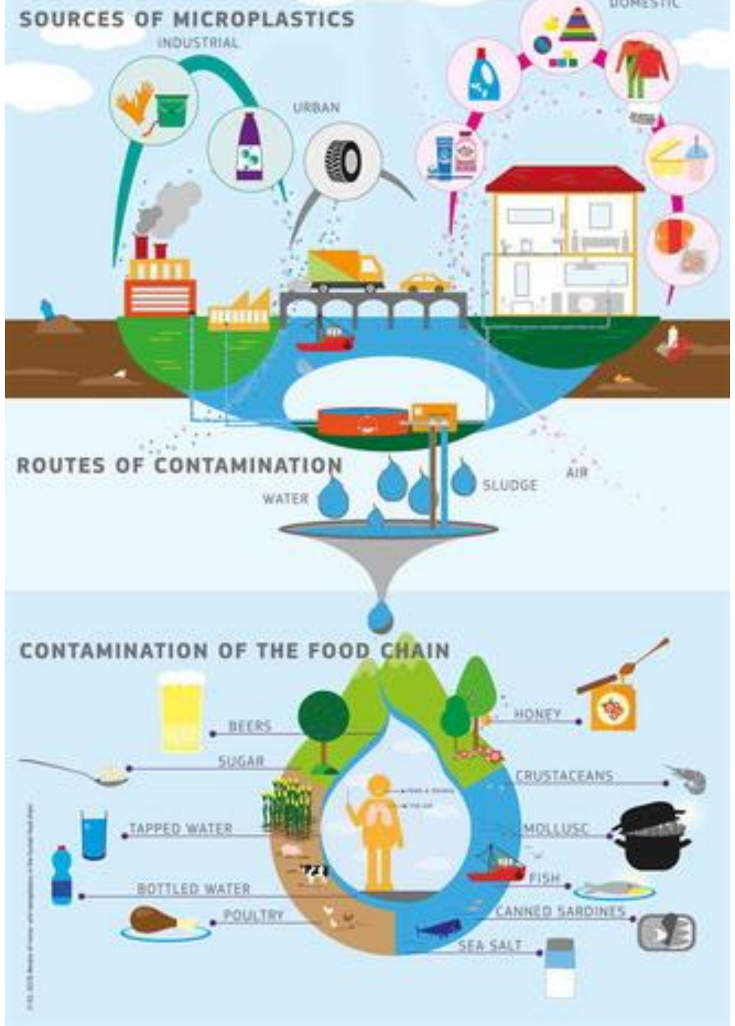
Honey, sugar, beer milk table salt vegetables nori seaweed edible parts of seafood, bottled water, tap water, energy drinks, soft drinks and tea infusions etc. etc……

If you look closely enough you will probably find (some) MPs!

Full picture remains unclear:

- Different sampling methods, sample size, different size ranges collected
- Variable metric, size distribution information lacking
- Different instrumental methods used
- Uncertain quality control – systematic use of blanks
- Contamination in
- No standardised sample preparation or verified recovery rates

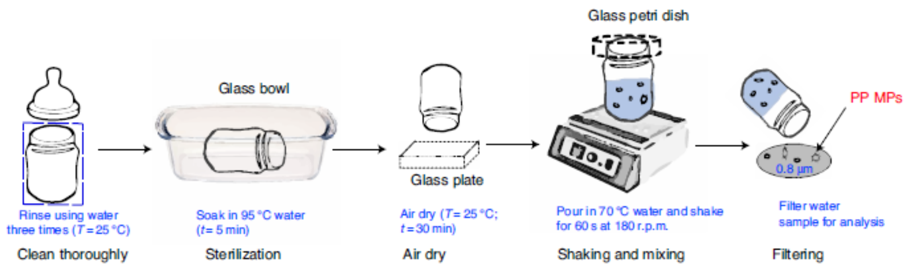
Review of micro- and nanoplastic contamination in the food chain
Toussaint et al. Food Additives & Contaminants: Part A
Volume 36, 2019 - Issue 5



Microplastics from food contact materials during use

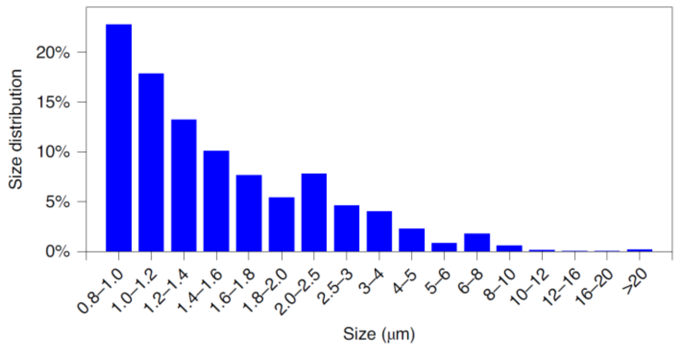
Literature Reports – increasing number of examples

Microplastic release from the degradation of polypropylene feeding bottles during infant formula preparation



Dunzhu Li et al. Nature Food | VOL 1 | **746** November 2020 | 746–754

Release >1x10^6 particles /L



But calculating mass…

0.17mg/L

Case studies - JRC

- Ice bags – colorant*
- Boil-in-the-bag rice: heating => low release*
- Tea bags: heating => release (≈0.2mg nylon)*
- Bottled water (PET); tap opening abrasion=> release**

Infant (1y) exposure to TiO_2(E171) is 3.6mg/day ≈5x10^{11} particles/day

* Manuscript in preparation
** Manuscript submitted

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Criteria and issues in defining MPs

There is a need to move from “common understanding” of microplastics to broadly accepted definition/definitions – detailed considerations by Verschoor and by Hartmann

Some Key Criteria -

Compositions

- **Polymers :** Plastics vs elastomers, Inorganic vs organics, synthetics vs modified “natural”
- **Additives** : Up to 50% weight can be additives
- **Copolymers**
- **Composites** : inorganic core with polymer coating,

Properties

- **Solid state** :
- **Solubility** : usually water but what occurs elsewhere (food, gastric fluid, lung fluid etc)
- **Size** : some consensus on range but nomenclature varies
- **Shape and structure** : some consensus of fibre – otherwise not well
- **Colour** : uncertain relevance

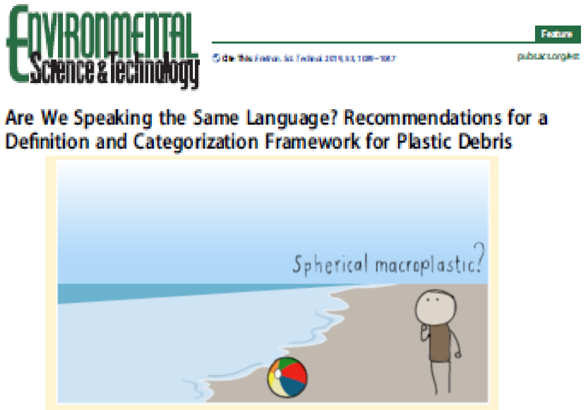
Other

- Origin : relevant for remediation, legislation (e.g. polluter pays),


National Institute for Public Health and the Environment
Ministry of Health, Welfare and Sport

Towards a definition of microplastics
Considerations for the specification of physico-chemical properties

RIVM Letter report 2015-0116
A.J. Verschoor



Hartmann et al.
Environ. Sci. Technol. 2019, 53, 1039−1047

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Definitions: an evolutionary process

Development of a common understanding and terminology

Microplastic: “Microplastic” term coined: At that time it was used in reference to small optically detectable granular or fibrous fragments of plastic materials found in the marine environment.
Thompson et al (2004): Science,Vol. 304, Issue 5672, pp. 838.

MicroLitter (2008) EU Marine Strategy Framework Directive (2008/56/EC) makes a distinction between macro- and micro-particles of litter, defined as objects with largest measurement over or below a limit of 5 mm - during an international workshop by NOAA, 5 mm was chosen “to focus the plastic littered discussion on possible ecological effects other than physical blockage of gastrointestinal tracts”.

Microplastic origins: Differentiate primary and secondary particles
Cole et al. (2011): Mar. Pollut. Bull. 62, 2588–2597

Nanoplastic: “…define nanoplastics as particles unintentionally produced (i.e. from the degradation and the manufacturing of the plastic objects) and presenting a colloidal behavior, within the size range from 1 to 1000 nm”
Gigault et al. 2020 Current opinion: What is a nanoplastic? Environ Pollut. 2018 Apr;235:1030-1034.

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Definitions – evolution for legislative needs

Ban of plastic micro-beads:

- Any intentionally added, 5 mm or less, water insoluble, solid plastic particle used to exfoliate or cleanse in rinse-off personal care products.

Drinking water: California State Senate Bill 1263 and subsequent SB 1422

- Microplastics in Drinking Water' are defined as solid, polymeric materials to which chemical additives or other substances may have been added, which are particles which have at least three dimensions that are greater than 1nm and less than 5,000 micrometers (µm)3. Polymers that are derived in nature that have not been chemically modified (other than by hydrolysis) are excluded. (Size-based nomenclature : "nanoplastics" (1 nm to <100 nm); "sub-micron plastics" (100 nm - <1 µm); "small microplastics" (1 µm to < 100 µm); "large microplastics" (100 µm to <1 mm); "mesoplastics" (1 mm to <2.5 cm); "macroplastics" (>2.5 cm).

ECHA proposed restrictions on intentionally-added microplastics

- "microplastic' means particles containing solid polymer, to which additives or other substances may have been added, and where ≥ 1% w/w of particles have (i) all dimensions 0.1 µm ≤ x ≤ 5 mm, or (ii), a length of 0.3 µm ≤ x ≤ 15 mm and length to diameter ratio of >3" : Pragmatic approach to lower size range

RECAST- European Drinking Water Directive (2020) :

- Measurement methodology to be adopted (Jan 2024) - no definition yet specified

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Micro and nano-plastics: Analytical Status

Microplastics
- Analytical methods exist but need standardization

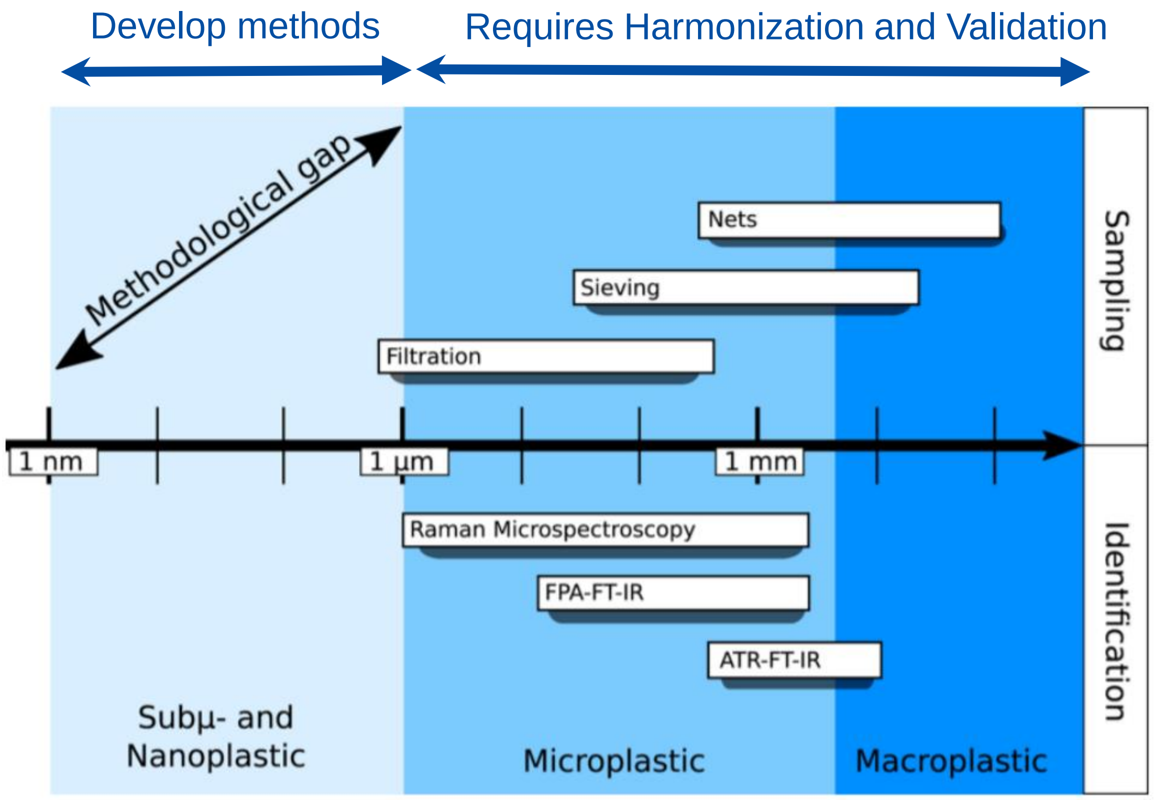
Nanoplastics
- Analytical methods need be developed and standardized

Technical Challenges

Instrument resolution
- Particles (1-10µm) : Optical methods approach resolution limits, slow to measure representative number
- Particles (<1µm) no established methods

Complexity and variety of sample matrixes
- Increasing difficulty - drinking water, environmental water (river/sea/lake), waste water, sediment, soil, alimentary, biota

Concentration and sampling
- from few tens of plastic particles/litre in drinking water to thousands per gram in water treatment sediments
- exponential increase in number with decreasing size

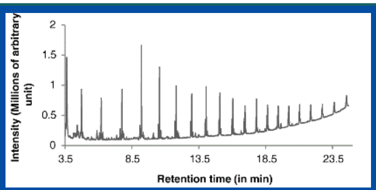


Schwaferts, C., et al. (2019) TrAC Trends in Analytical Chemistry **112**: 52-65.

Developing new analytical methods for sub/micron and harmonizing existing are critical to reducing knowledge gaps about environmental and human exposure levels and biological effects

Conventional Analytics: Microplastics detection and identification by common optical/chemical methods

Integral Methods
Pyro-GCMS and LC-MS
Polymer identity and total mass



µ-Raman

Size range >1µm
Polymer identity, number and size

µ-FTIR

Size range >(2-5µm) typically >10-20µm
Polymer identity, number and size

Fluorescence Microscopy

Size range (>1µm size)
Screening number and size only
No conclusive identity.

Newer variants: AFM-FTIR, AFM-Raman, CARS, Cascade Laser Raman

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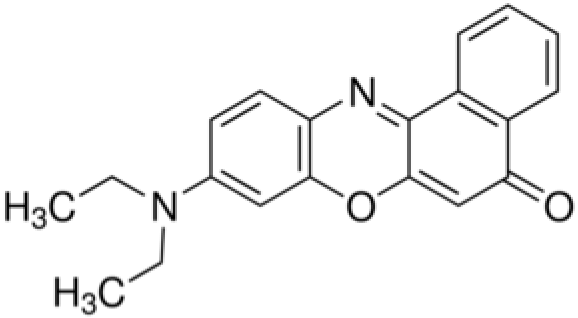
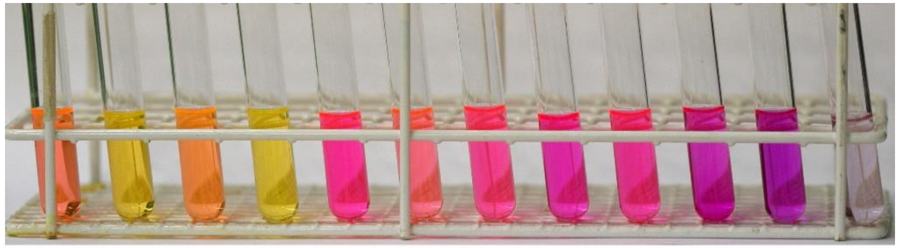
Microplastics detection by fluorescence microscopy

Fluorescence Microscopy

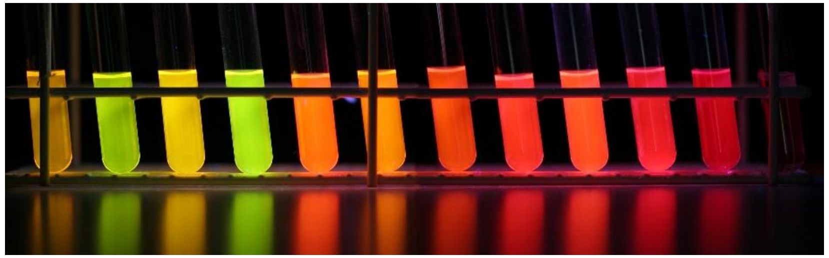


Nile Red Dye:
Liphophilic dye - almost non-fluorescent in water and other polar solvents but undergoes fluorescence enhancement in non-polar environments (absorbed on plastic)



Visible light illumination of Nile red in range of solvents

Illuminated with UV light



Fluorescence colour/intensity varies with solvent (local hydrophobicity)

1. Water, 2. Methanol, 3. Ethanol, 4. Acetonitrile, 5. Dimethylformamide, 6. Acetone, 7. Ethylacetate, 8. Dichlormethane 9. n-Hexane, 10. Methyl-tert-Butylether, 11. Cyclohexane, 12. Toluene.

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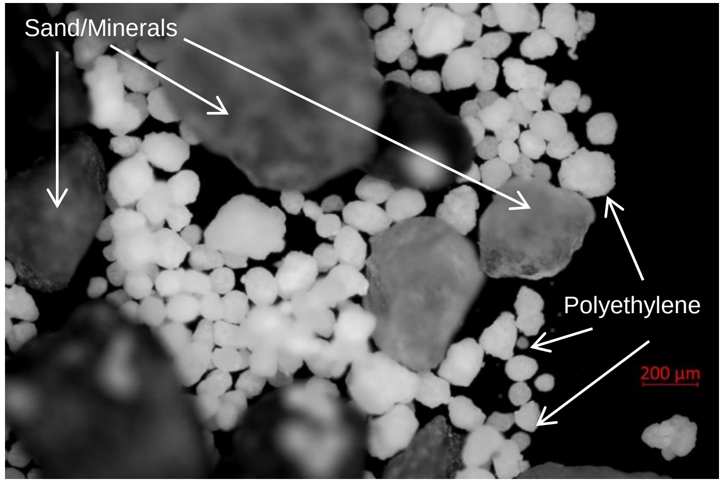
Microplastics detection by fluorescence microscopy

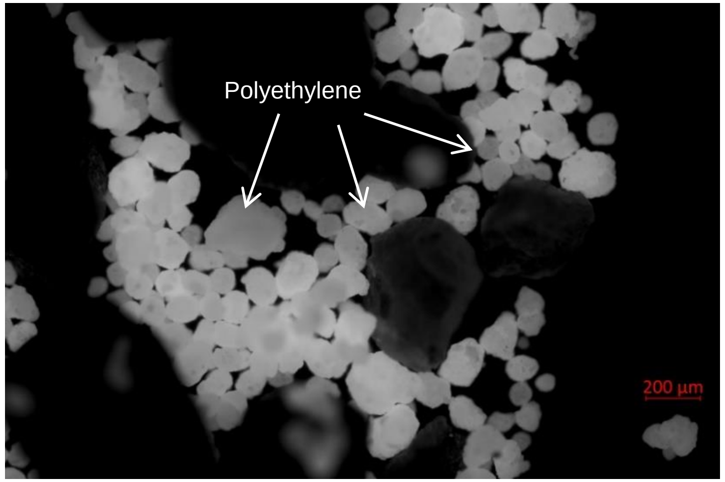
Mix of beach sand and polyethylene



Illuminated by visible light

Apply Nile Red dye to sample

Nile red absorbs preferentially to hydrophobic (plastic) surfaces and fluorescence is enhanced



Illuminated by UV-light

Strong fluorescent emission only from plastic

Overall

- Simple, rapid, moderate cost for screening
- Risk of false positives/negatives
- Little/No information on type of plastic

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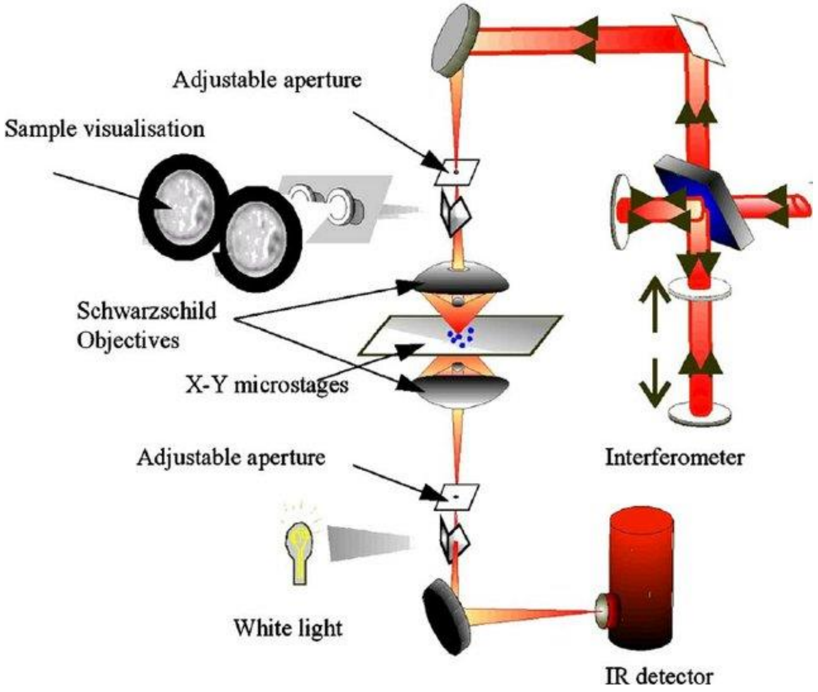
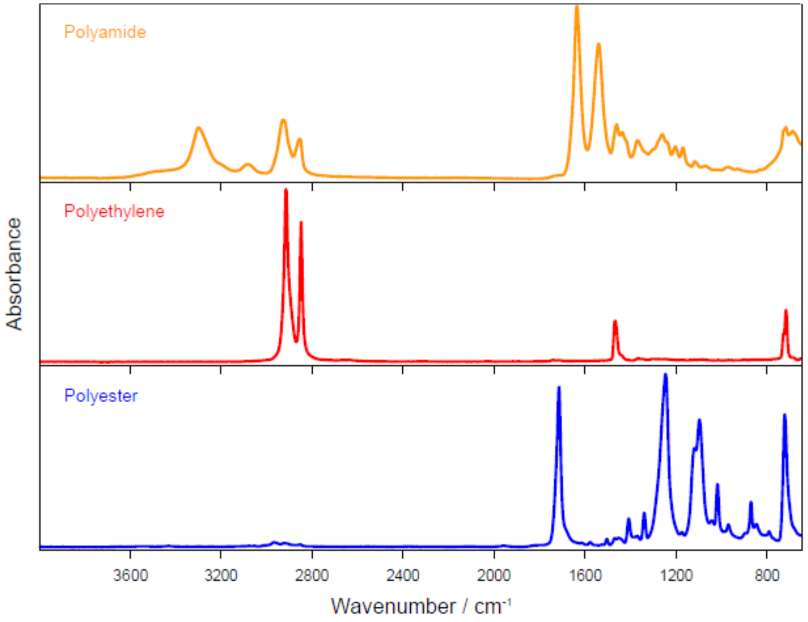
FT-IR microscopy: Localised measurement of Infra-Red absorption spe



FTIR Microscope

Measure IR absorption spectra
from individual particles
Transmission, Reflection, µATR

Measure Spectral "fingerprint"
from individual particles



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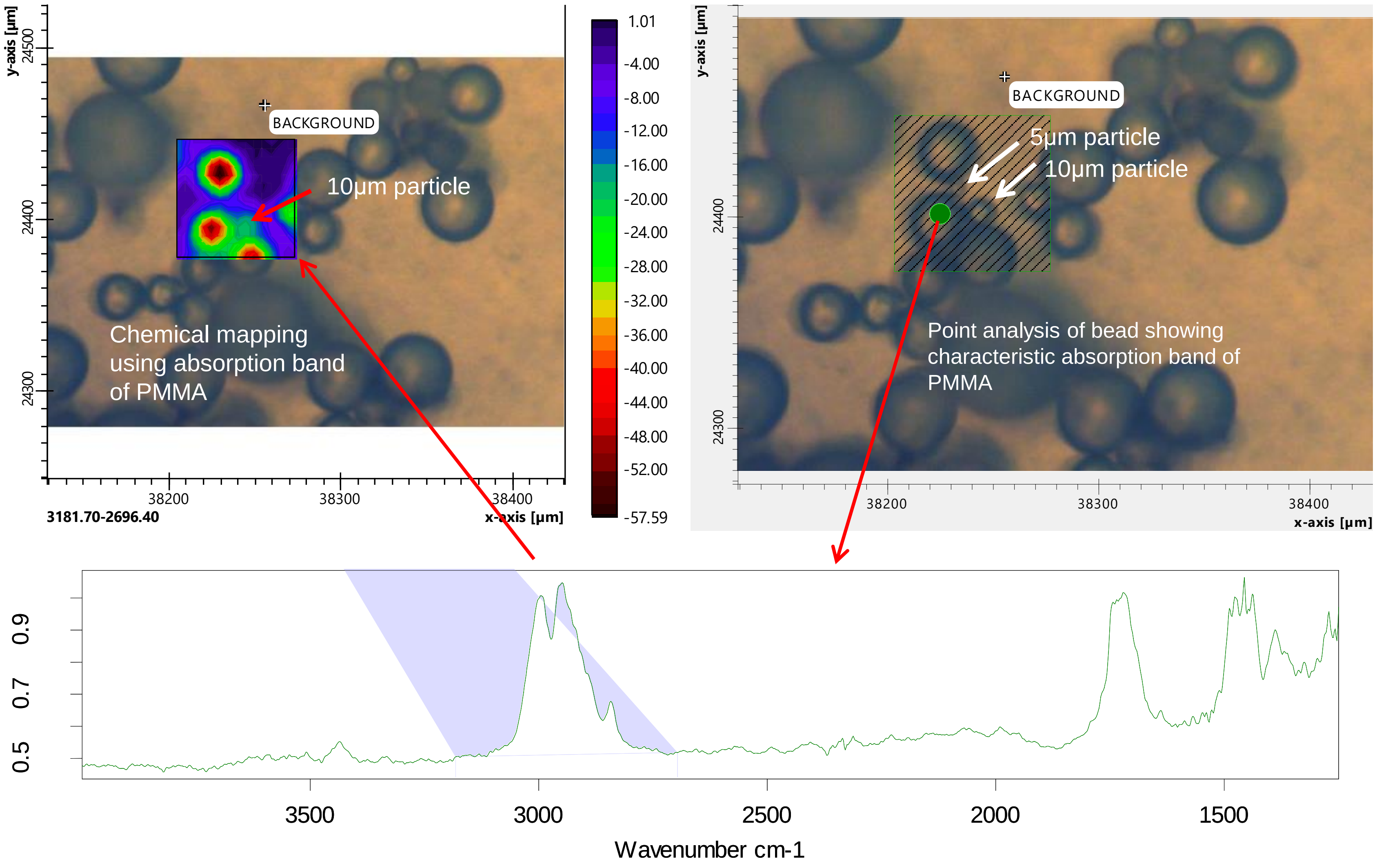
FT-IR microscopy: Chemical imaging of microplastics – limitations

Overlayed Chemical imaging

Optical imaging



Optical and µ-FTIR chemical imaging of PMMA microbeads
- Single point detector

Optical

- 10 µm object detected
- 5 µm object detected

Chemical imaging

- 10 µm object detected
- 5 µm object NOT detected
- Minimum size 2-4um achievable with µ-ATR or FPA options

Overall

- Powerful method, complex with medium-high investment cost
- Identification of polymers
- Slow mapping

Options

- Increase throughout – linear array detector 8-16 spectra simultaneously
- Focal plane array 64x64=4096 spectra simultaneously

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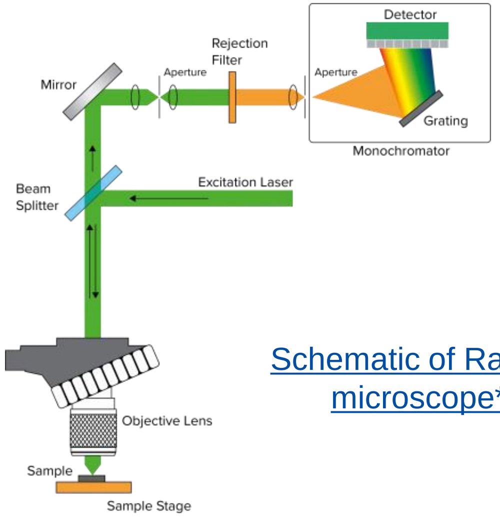
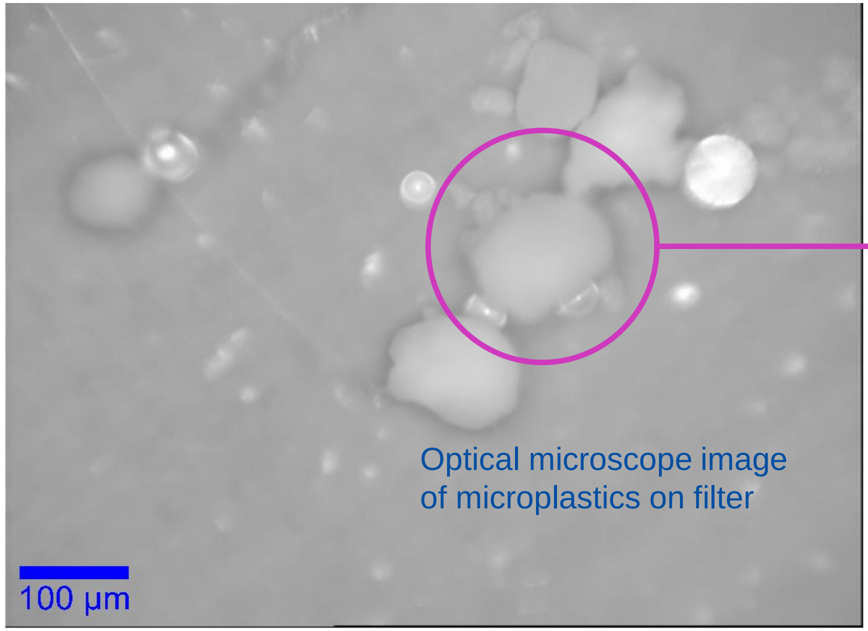
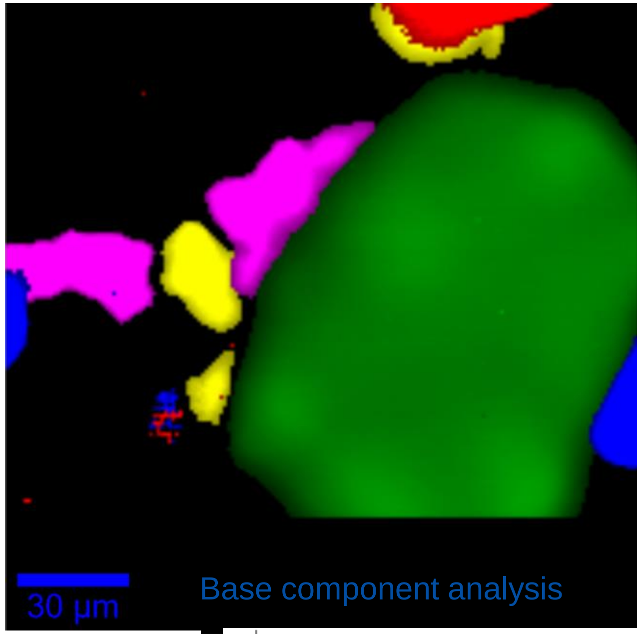
Confocal Raman microscopy : Chemical imaging of microplastics



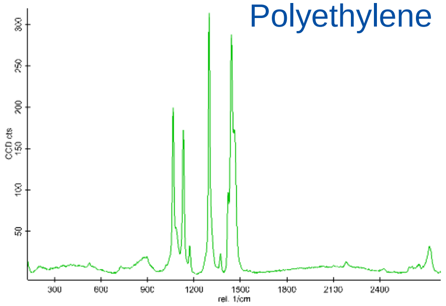
Schematic of Raman microscope*

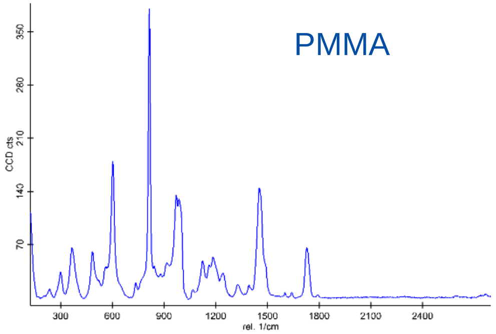


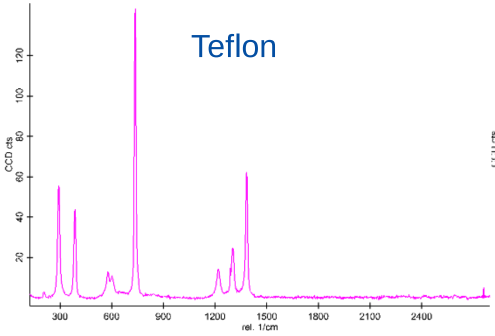
Optical microscope image of microplastics on filter

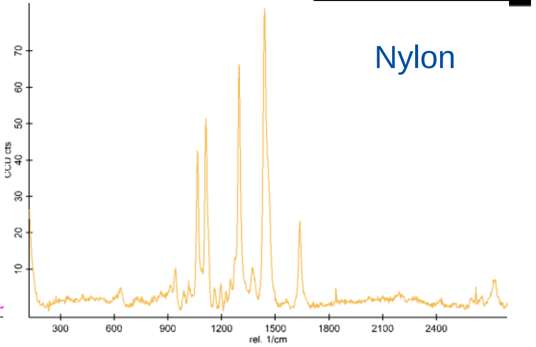


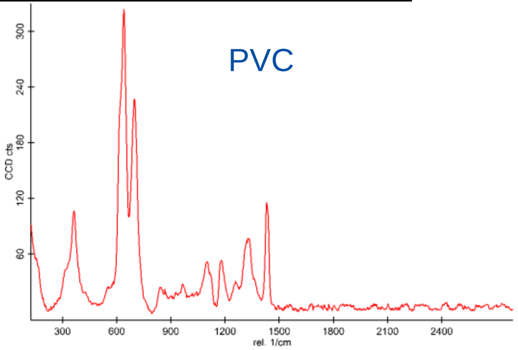
Base component analysis











- Powerful method, complex with medium-high investment cost
- Minimum size <1um and identifies specific plastics
- Slow if particles are numerous, additives/colourant may auto fluoresce covering the plastic spectrum

*https://jascoinc.com/learning-center/theory/spectroscopy/confocalramanmicroscopy/

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Example: Microplastics in sea salt

Salina di Marsala (Image: Fidelity Viaggi)

- Solar evaporation
- Mechanical harvesting
- Processing
  - Washing
  - Drying
  - Packaging

Microplastics separation from sea salt: Minimal sample Preparation

5 g sea salt

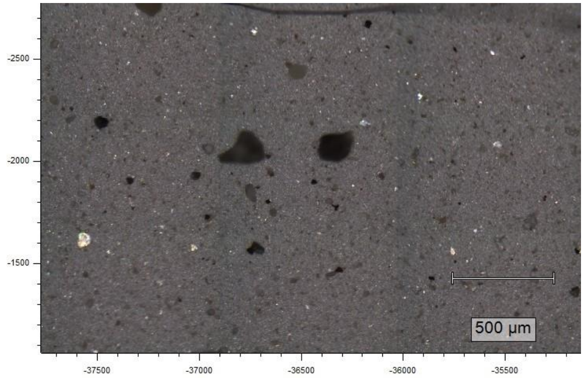
Dissolution

Density Separation

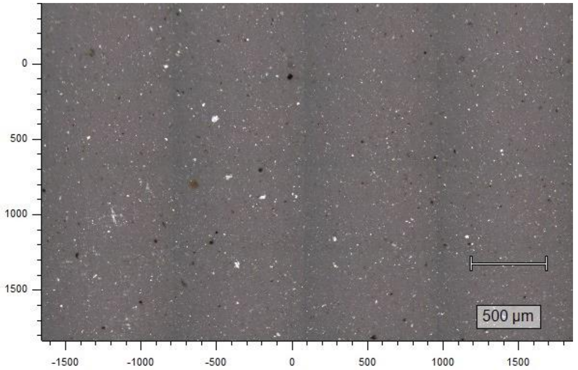
ZnCl2 solution
1.5 g/cm3

Vacuum filtration, Wash with detergent

Anodisc*



17 x more particles in the >5 micron range in optical image



* Alternative filters possible: porosity, flatness sample, analytical method, compatibility

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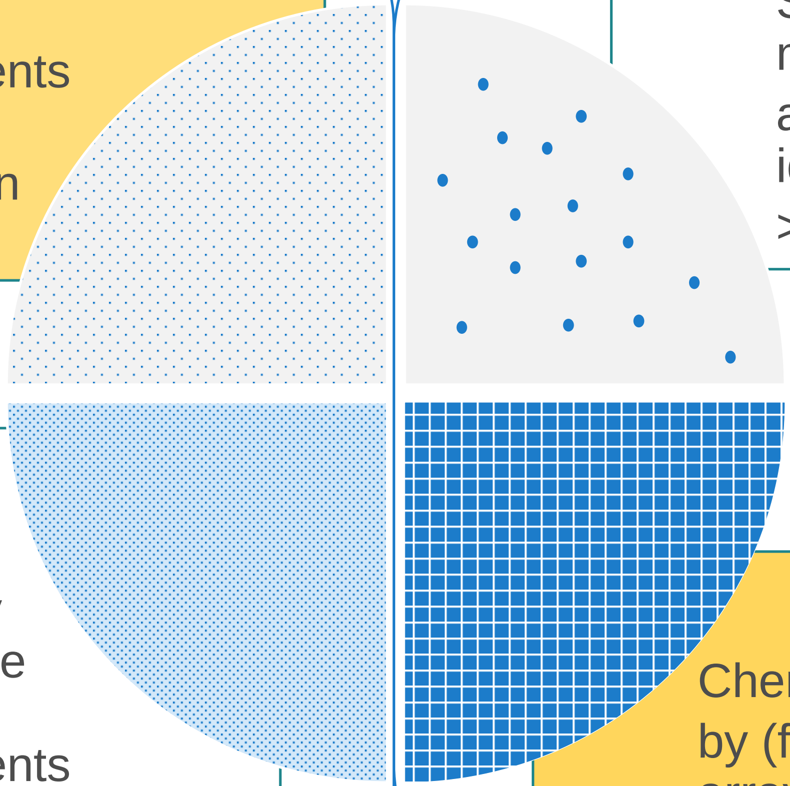
Microplastics analysis in sea salt

Raman microscopy

Single spot measurements after optical identification >1µm

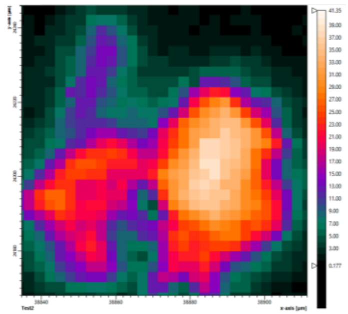
Chemical mapping by scanning the filter, measurements at single spots



FT-IR microscopy

Single-spot measurements after optical identification > 10 µm

Chemical mapping by (focal plane) array detectors



Resolution
vs
TIME

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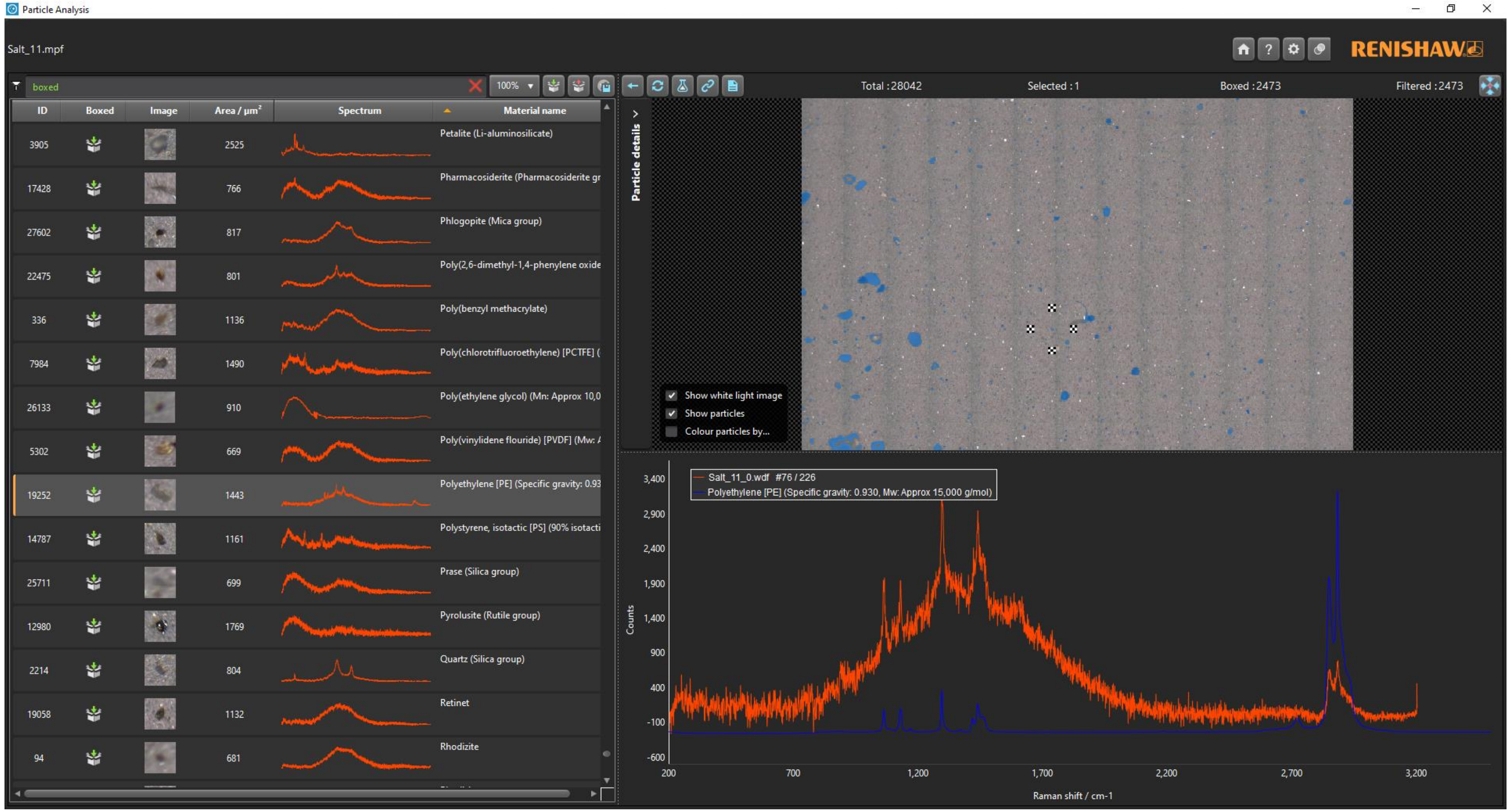
Raman analysis, optically identified single spots

Example: polyethylene particle in sea salt sample on Anodisc filter



- Renishaw inVia + Particle Analyser
- 785 nm laser
- Life track
- 10 x objective
- 5x10s collection time for each spectrum
- Inorganic + Polymer database

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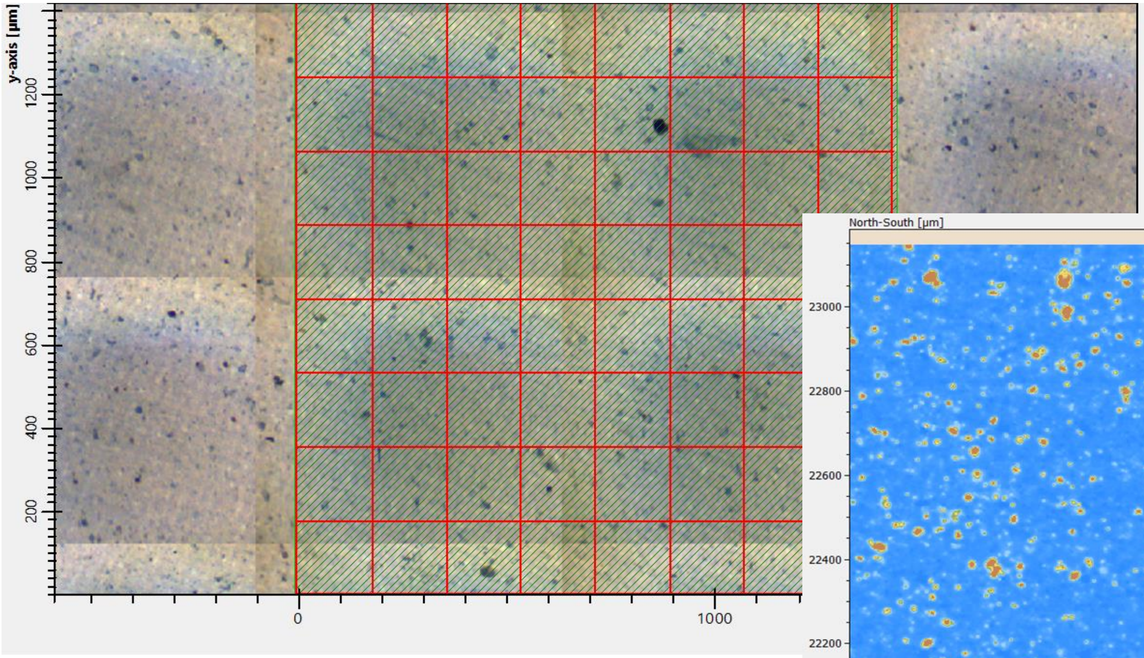
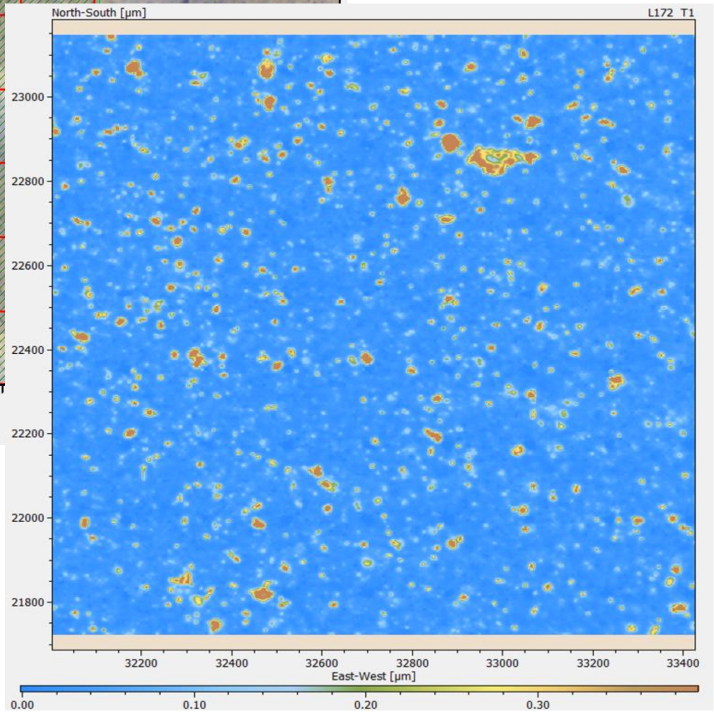
FT-IR analysis, chemical map, focal plane array

Example: sea salt sample on Anodisc filter



Optical image and scanned area (Opus)



Heat map (Purency) – single wavenumber

- Bruker Hyperion mic, Opus
- Transmission
- 15x objective
- 64x64 FP
- Purency software

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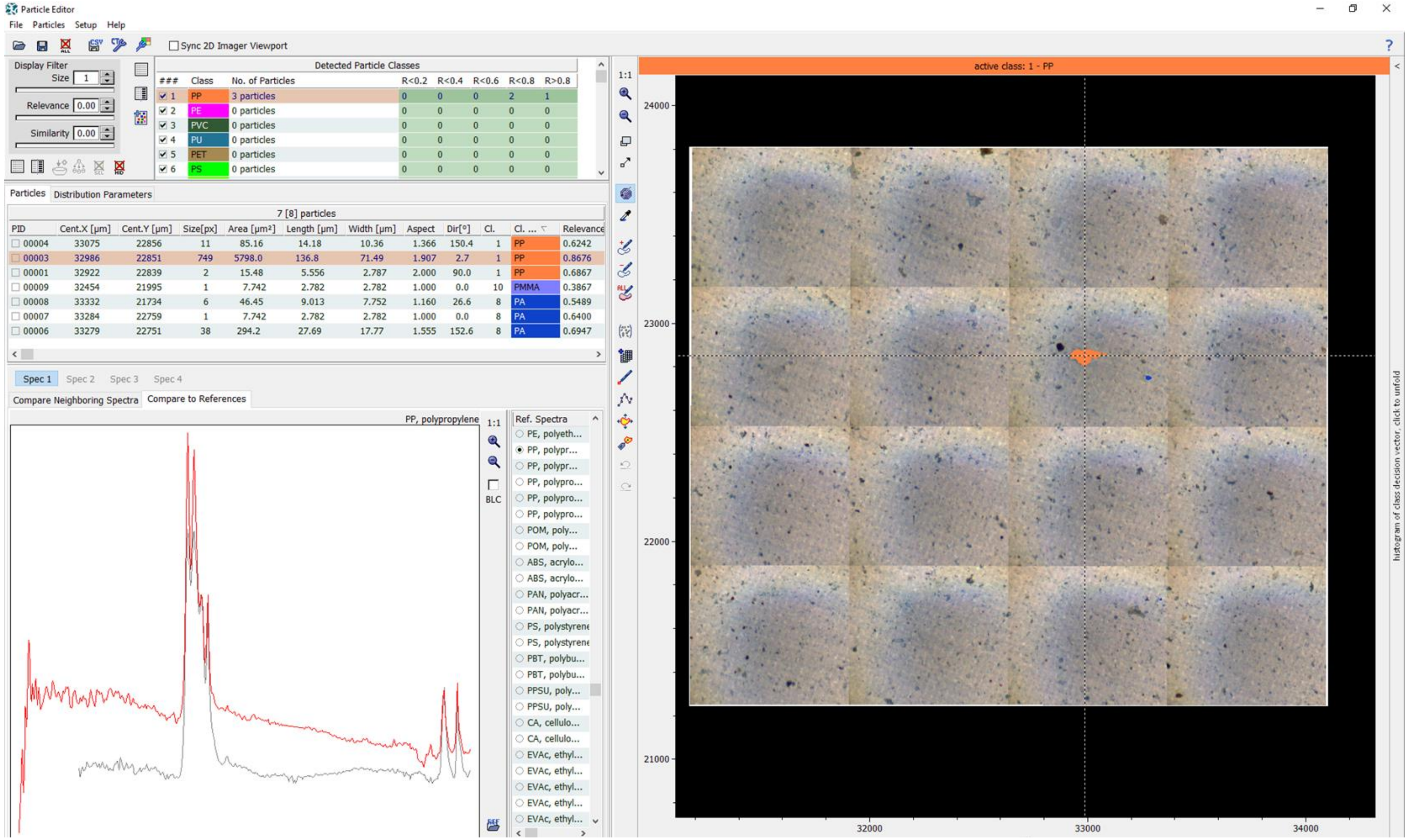
FT-IR analysis, chemical map, focal plane array

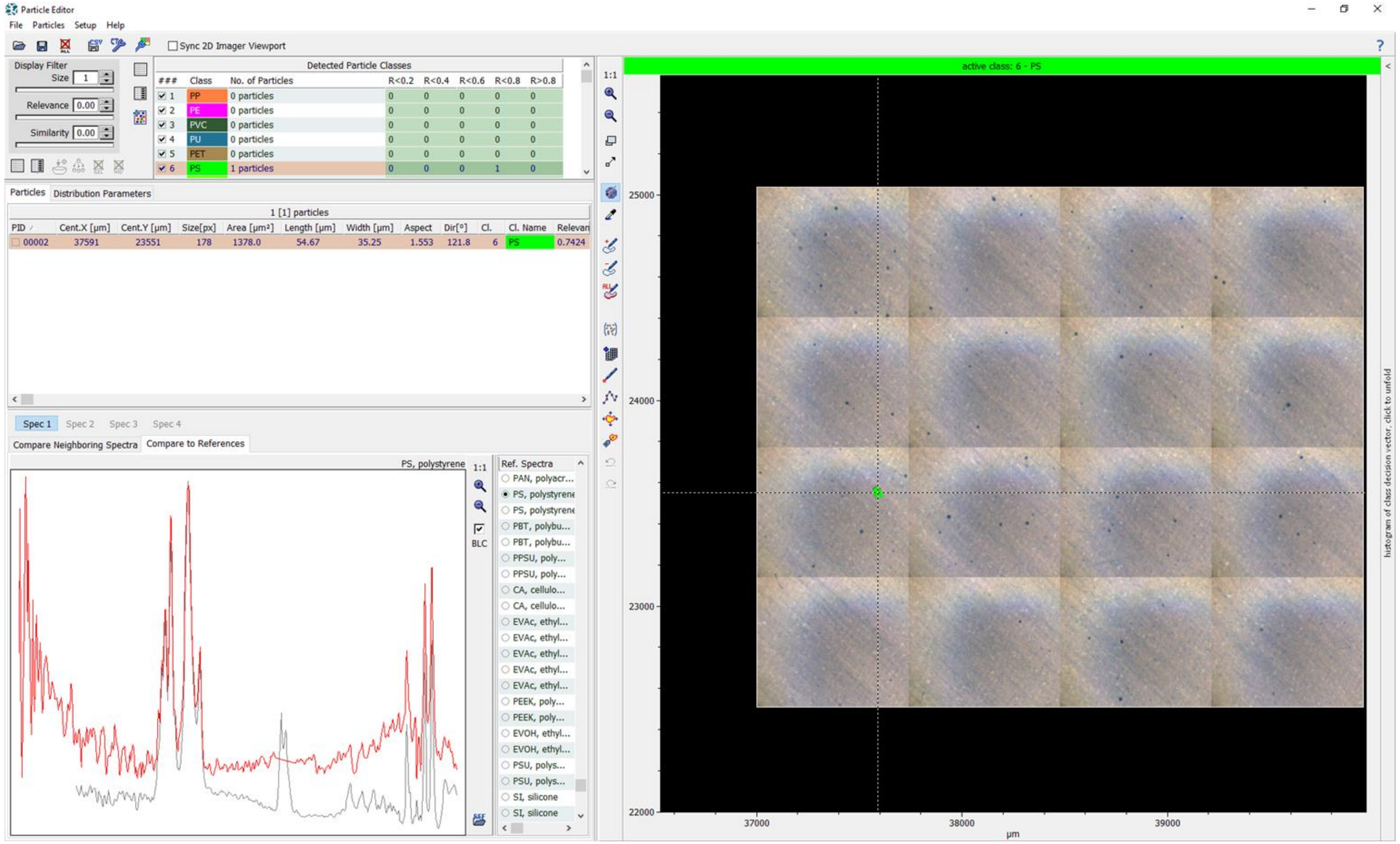
Example: polypropylene particle in sea salt sample on Anodisc filter



- Purency software
- In-built database, created by machine learning
- No info on non-plastic in the database

FT-IR analysis, methodological blank

Example: polystyrene contamination in methodological blank sample on Anodisc filter

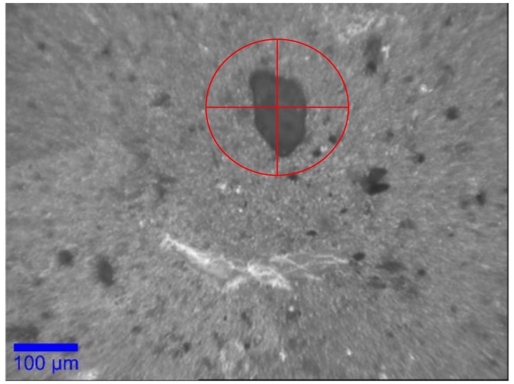


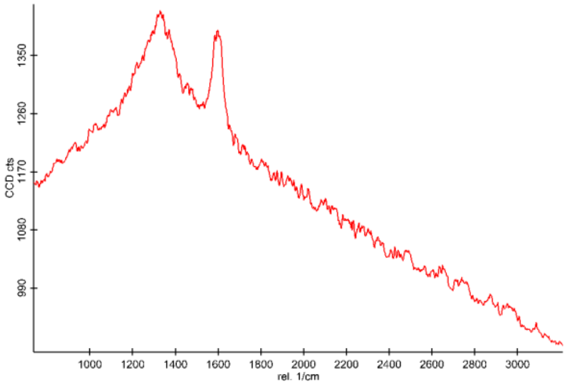
- Bruker Hyperion mic
- Purency
- Possible source of contamination:

filtration system for filtering detergent solution.

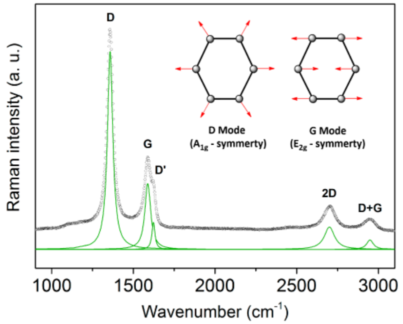
But what are the other particles in sea salt?

Optical image	Spectrum	Assignment

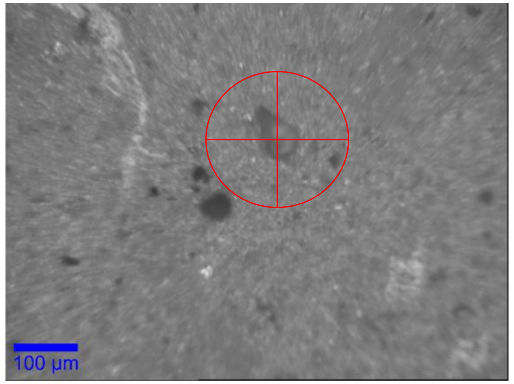


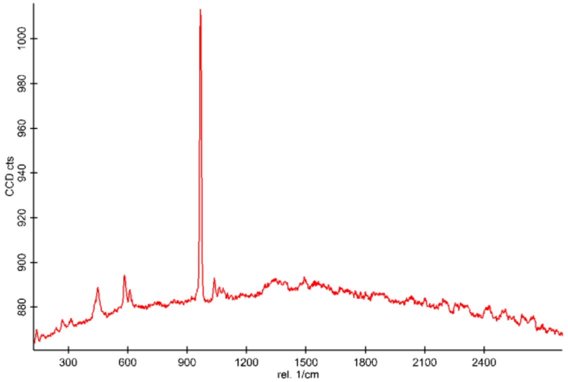


Carbon black

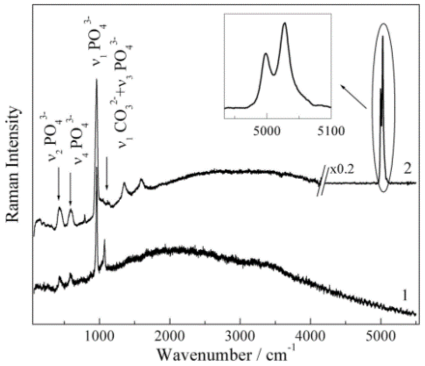


Materials **2018**, *11*(1), 93; https://doi.org/10.3390/ma11010093





Hydroxyapatite



Nosenco et al., 2016 Raman Spectroscopy
https://doi.org/10.1002/jrs.4883

- Raman microscopes

  (WiTec, Renishaw)

  532 nm, 785 nm
- Calcium carbonate(s)
- Hydroxyapatite
- Carotenoids
- Carbonised organic compounds
- …

Complex extractions - PVC from mussels

Exposure/uptake study in mussels – extraction of MPs

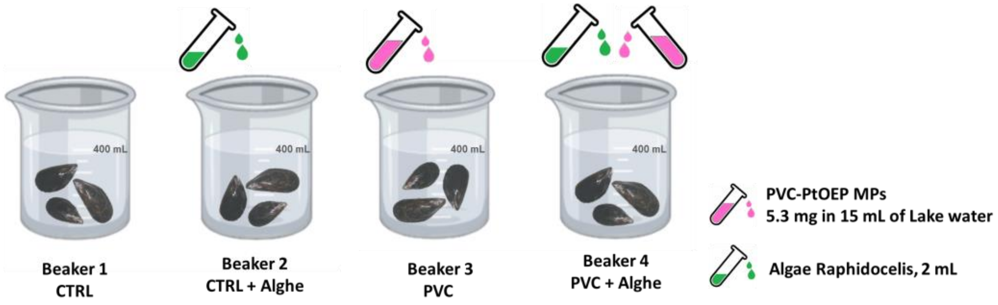
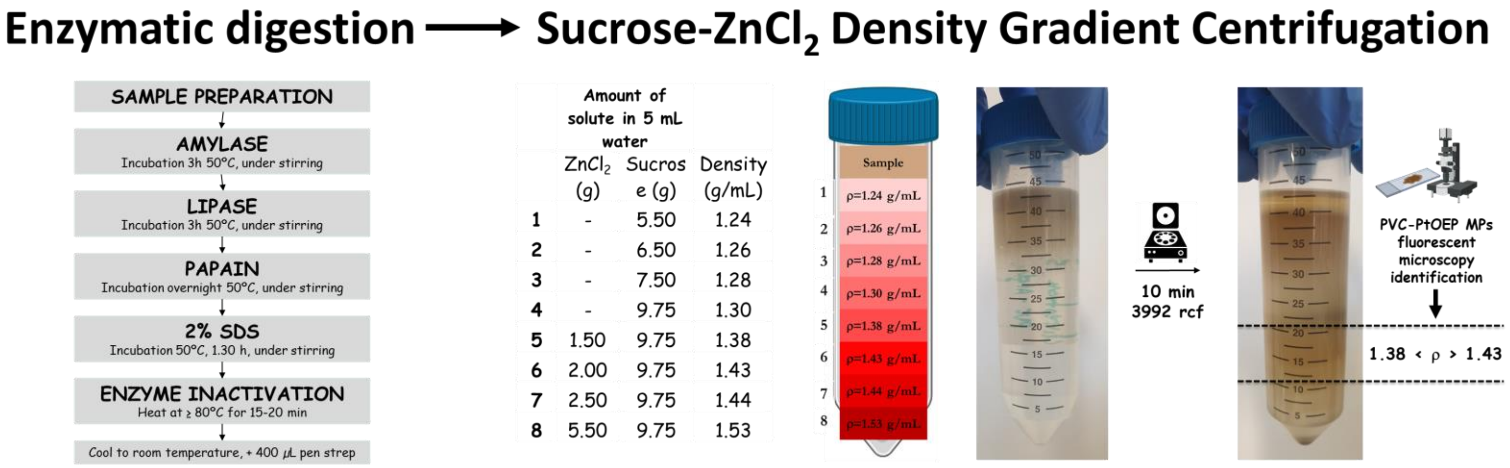


Figure 2. Schematic illustration of the exposure experiments preparation.



	Amount of solute in 5 mL water		
	$ZnCl_2$ (g)	Sucrose (g)	Density (g/mL)
1	-	5.50	1.24
2	-	6.50	1.26
3	-	7.50	1.28
4	-	9.75	1.30
5	1.50	9.75	1.38
6	2.00	9.75	1.43
7	2.50	9.75	1.44
8	5.50	9.75	1.53

Figure 3. (**a**) Flowchart of the optimized enzymatic digestion protocol used. (**b**) Separation, according to density, of the sample digested through a sucrose-$ZnCl_2$ gradient by centrifugation. Black dotted lines show the gradient layer in which the PVC-PtOEP MPs particles internalized by the animal were found by fluorescent microscopy analysis.

Complex extraction development aided by use of custom synthesised fluorescent PVC MPs

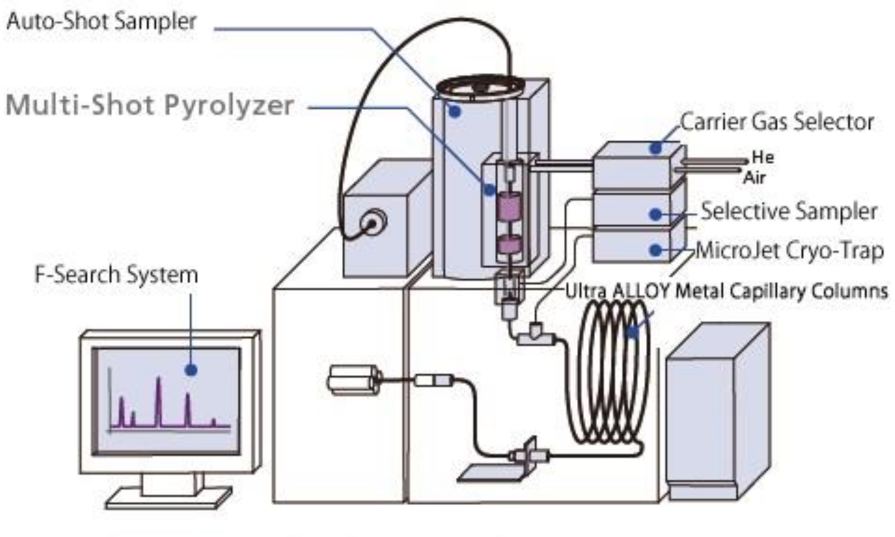
Detection of Metal-Doped Fluorescent PVC Microplastics in Freshwater Mussels
Samantha V. Facchetti et. al Nanomaterials 2020, 10, 2363

European Commission

Pyrolysis-GCMS: Identification of Micro and Nano-plastics

Sample exposed to high temperature – decomposes

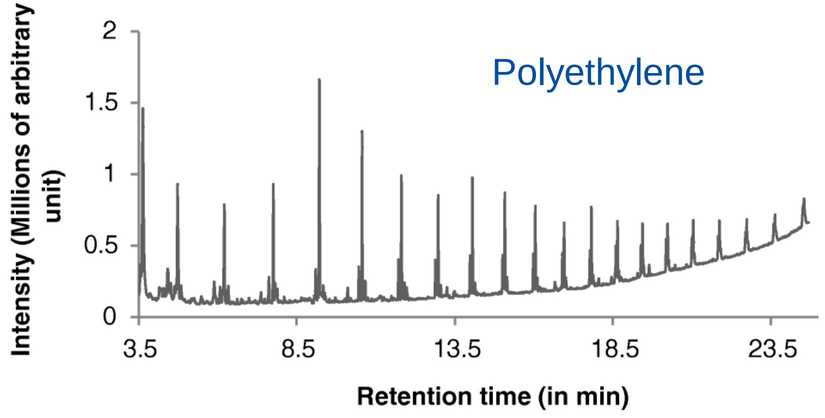
- Decomposition products are characteristic of polymer



Py-GC/MS System (Multi-Shot Pyrolyzer and peripherals)

Analysis of decomposition products

Characteristic spectra



Py-GCMS Characteristics

- Determines quantity and type of polymer
- Lower limit for detection (mass) ≈0.5µg Lower limit of size ≈50-100µm (single particles)
- Information on additives
- But - no direct particle number or size information - to have size information must combine with other methods

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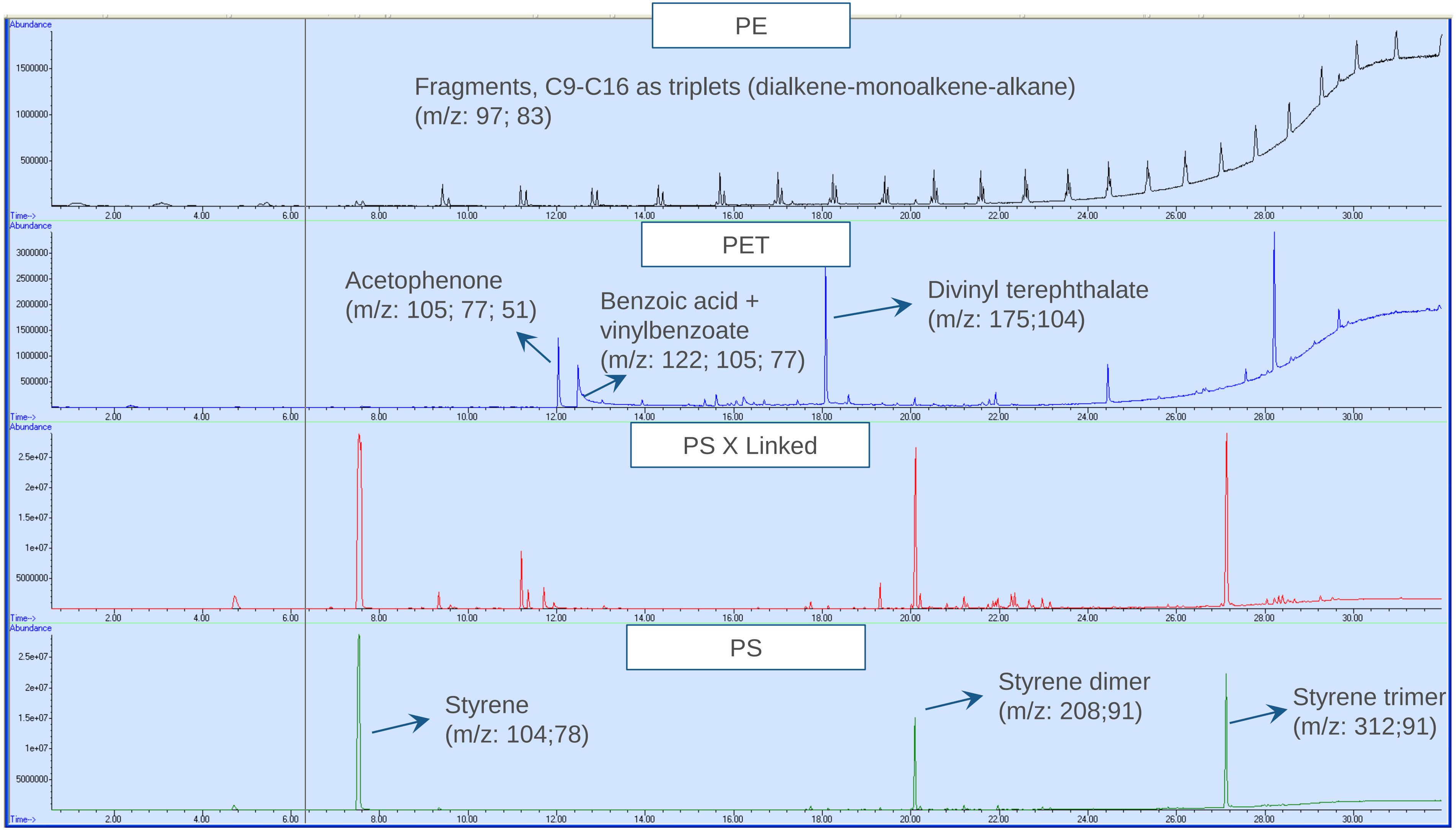
Characteristic Fingerprints – marker peaks

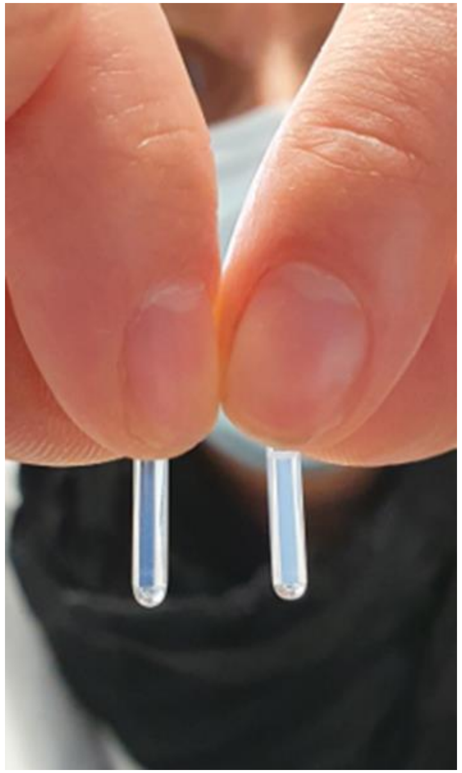


Pros and Cons of the technique

- Powerful tool in the detection/identifications of low mass of plastic (nano)particles (down to less than <0.5-1 µg) in the sample <=> Particles 50-70µm).
- Analysis of properties of the entire particle (not only surface). Less sensitive to interferences such as pigments or surface oxidation phenomena.

- Calibration can be challenging – sample handling
- Matrix interferences. Matrix must be removed, either by size separation techniques or by digestion

Sample Vials



Practical consideration:

- GCMS instrumentation commonly available in analytical laboratories - upgradable to py-GCMS at moderate cost

Pyr-GCMS : recent developments in sample prep

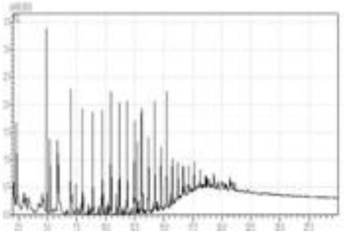
Sample (Sludge) → PLE → Py-GC/MS → Plastic concentrations

PLE

Pressurized Liquid Extraction

Py-GC/MS

Plastic concentrations



High recovery

	Standard Spike		Extract from sample	
Plastic type	Recovery ($n = 5$)[a] (%)	RSD (%)	Recovery ($n = 5$)[b] (%)	RSD (%)
PE	91 ± 8	8.4	83 ± 3	3.6
PMMA	98 ± 3	3.4	91 ± 2	1.9
PS	98 ± 3	13.7	93 ± 3	2.9
PET	93 ± 9	9.4	85 ± 5	6.0
PC	99 ± 8	8.3	95 ± 2	2.3
PP	94 ± 5	5.6	85 ± 4	4.5
PVC	87 ± 8	8.6	84 ± 3	3.1

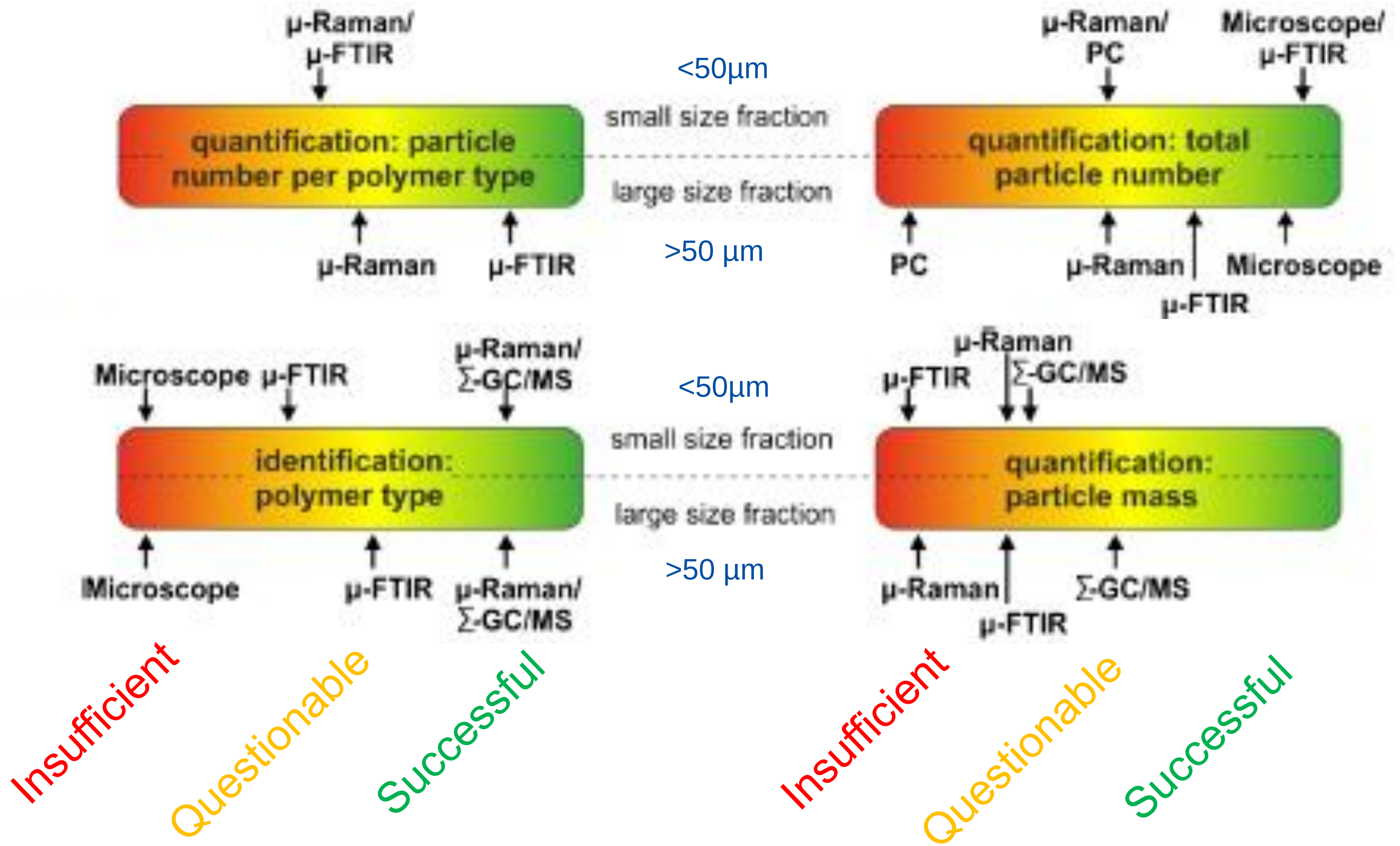
Processing large, complex samples: Could this be applicable to food products?

Identification and quantification of selected plastics in biosolids by pressurized liquid extraction combined with double-shot pyrolysis gas chromatography–mass spectrometry
Okoffo et al 2020 Science of the Total Environment 715 (2020) 136924

European Commission

Relative performance of methods

Sample; 5 polymers types, sizes 8-140 um mixed, 100-900 particles each – 17 laboratories

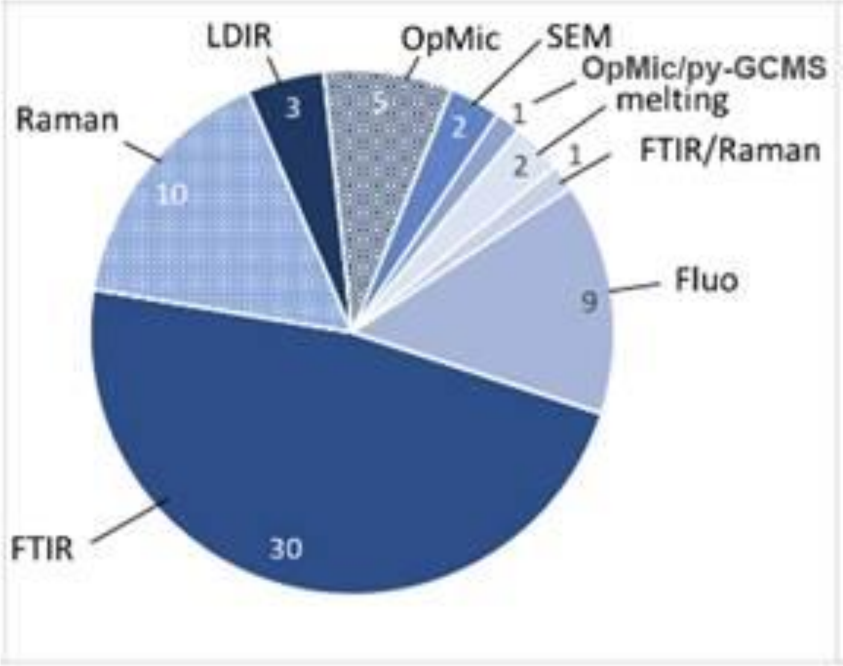


Microplastic analysis—are we measuring the same? Results on the first global comparative study for microplastic analysis in a water sample
Müller et al Analytical and Bioanalytical Chemistry (2020) 412:555–560

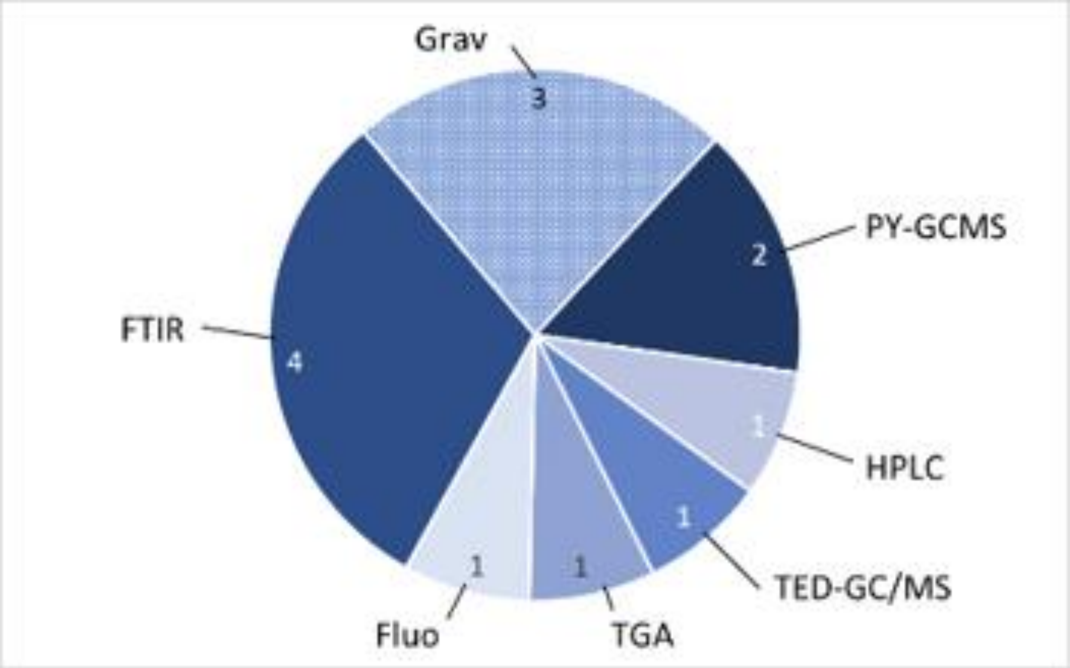
European Commission

Methods use/availability

JRC Inter-laboratory comparison - measurement of microplastic in water



Metric = Particle Number



Metric = Particle Mass

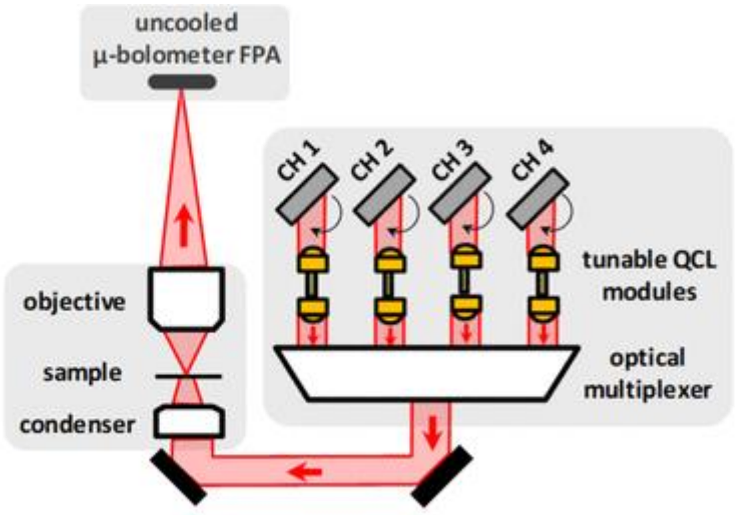
Reported methods dominated by optical spectroscopy methods – FTIR primarily
Particle counting predominates

Some recent/developing methods - micro

- µFTIR: Quantum cascade laser - FTIR - High power light source combined with FPA – rapid FTIR analyses possible*
- µRaman : optical cells for on-line analysis of liquids: coupling of detector to nanoparticle fractionation methods (e.g.FFF)
- GC/MS: TED-GCMS: automation, simplified sample preparation, larger total sample weights up to 1 g
- Pressure Liquid Extraction for py-GCMS – large samples

Data use

- Automated spectra analysis for µRaman/µFTIR – data manipulation, automation of analysis, machine learning
- Increase reliability, minimise manpower, reduce subjective input from operators
- Development towards routine, fast, reliable measurements for monitoring



*Primpke et. al Environ. Sci. Technol. 2020, 54, 24, 15893–15903 Rapid Identification and Quantification of Microplastics in the Environment by Quantum Cascade Laser-Based Hyperspectral Infrared Chemical Imaging

**E.Duemichena et. al
Journal of Chromatography A Volume 1592 (2019), Pages 133-142
Automated thermal extraction-desorption gas chromatography mass spectrometry: A multifunctional tool for comprehensive characterization of polymers and their degradation products

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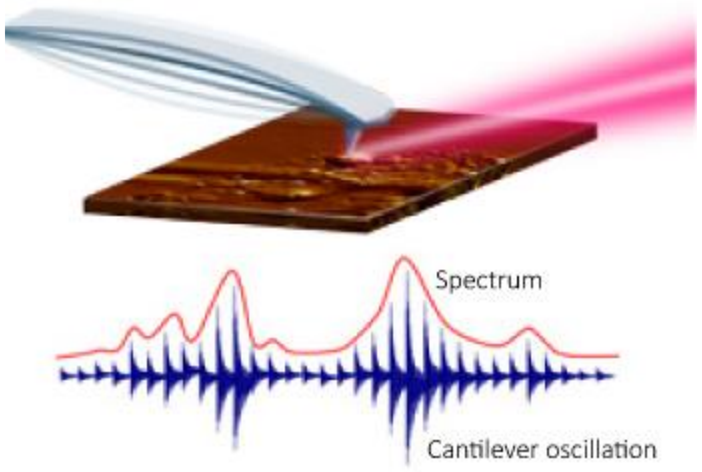
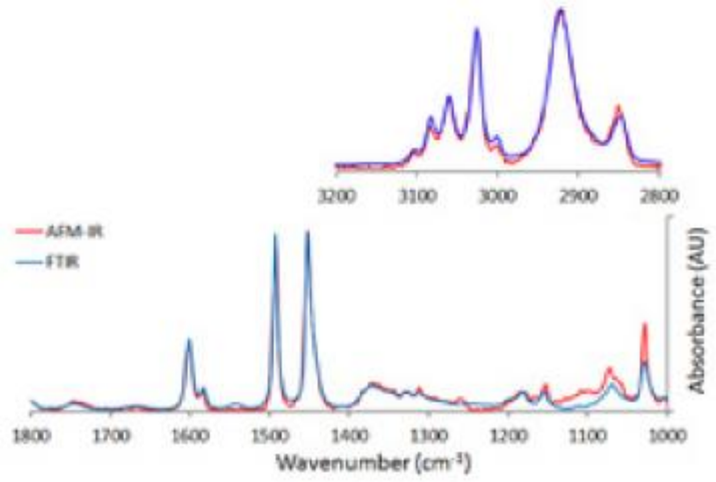
IR with sub-micron particulates – Atomic Force Microscope-IR

AFM-IR: How it works



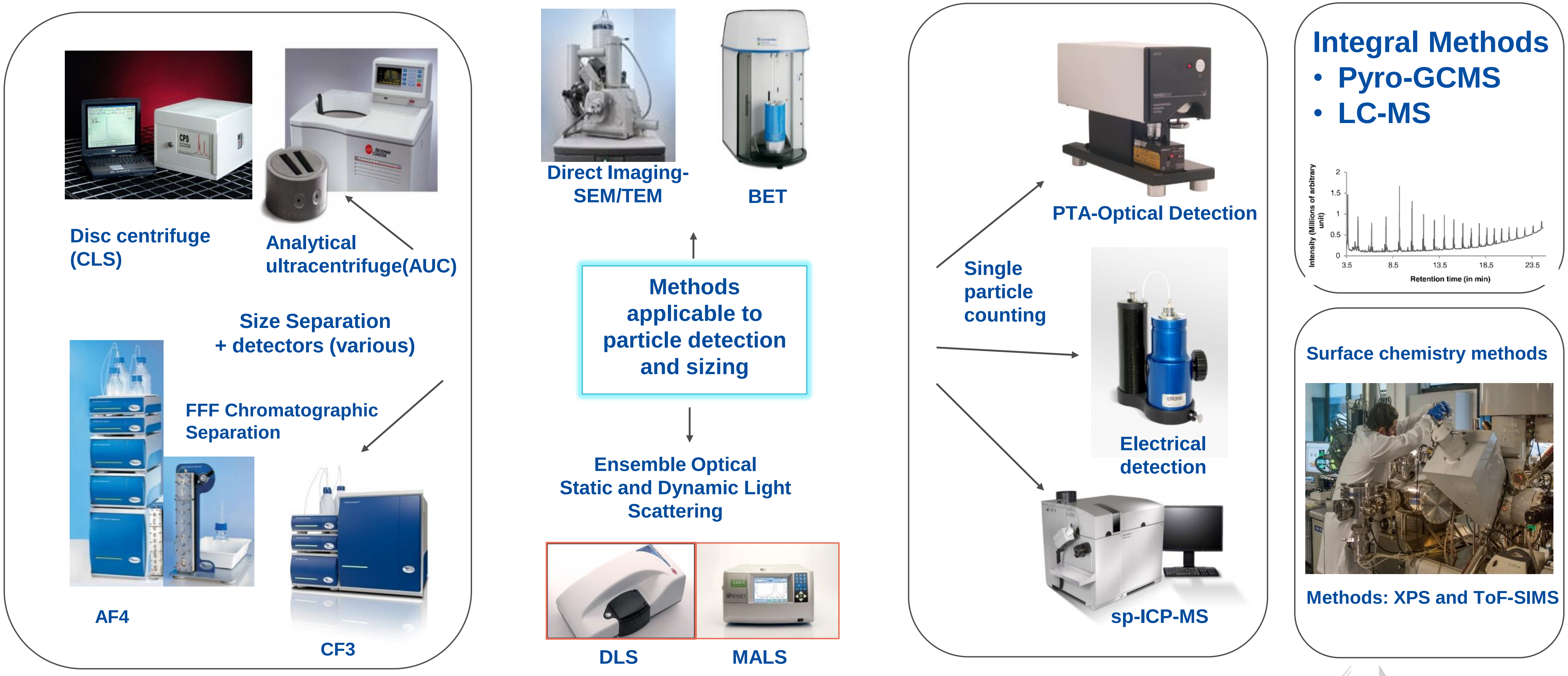


Excellent correlation to FTIR spectra

Pulses of infrared laser radiation tuned to an absorbance band of a material causes an abrupt and short-lived thermal expansion which excites resonant oscillation of the AFM cantilever. An AFM-IR absorption spectrum is created by measuring the cantilever oscillation amplitude while scanning the laser across the spectral range of interest. The resulting absorption spectrum is a unique chemical fingerprint of a nanoscale region of the sample under the AFM probe tip.

https://www.anasysinstruments.com/afm-ir-overview/

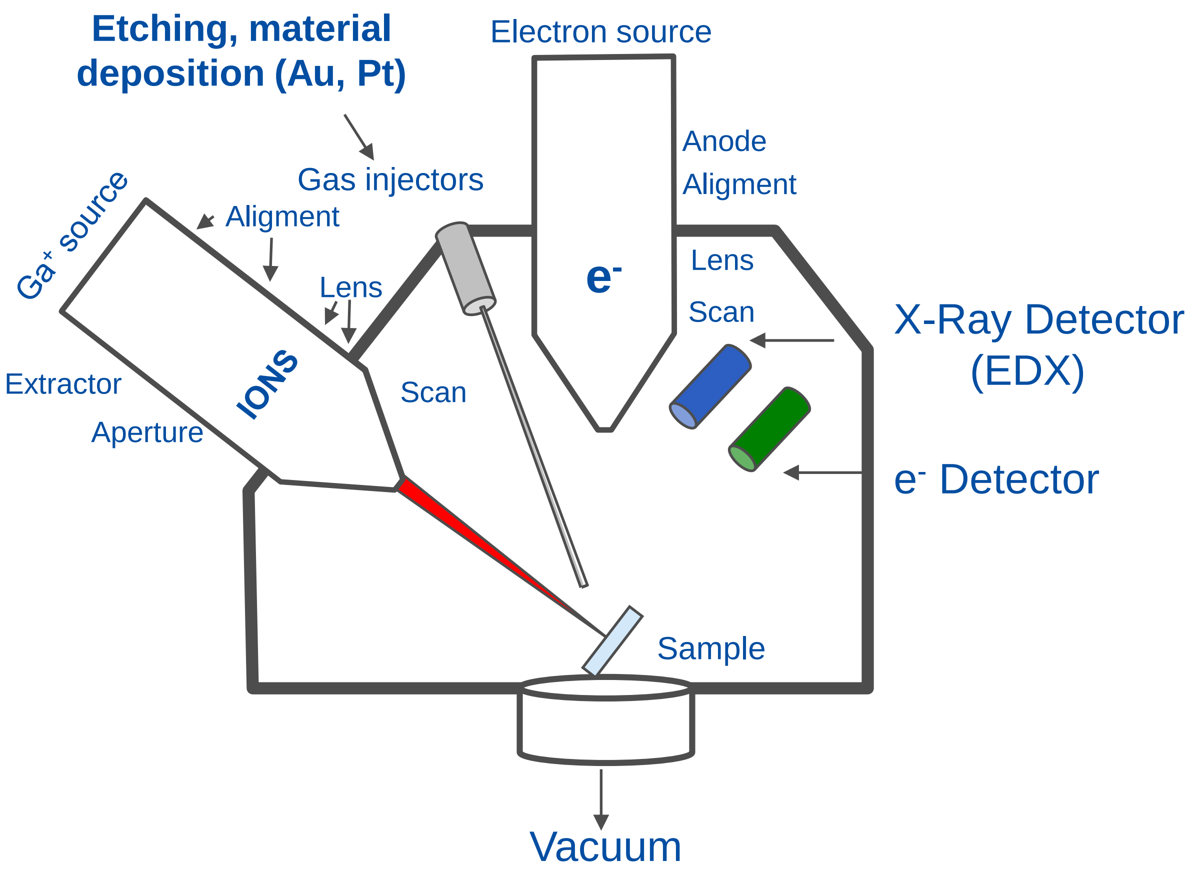
Nanoparticle analytics : Nanoplastics – analytics based on existing nanoscience methods



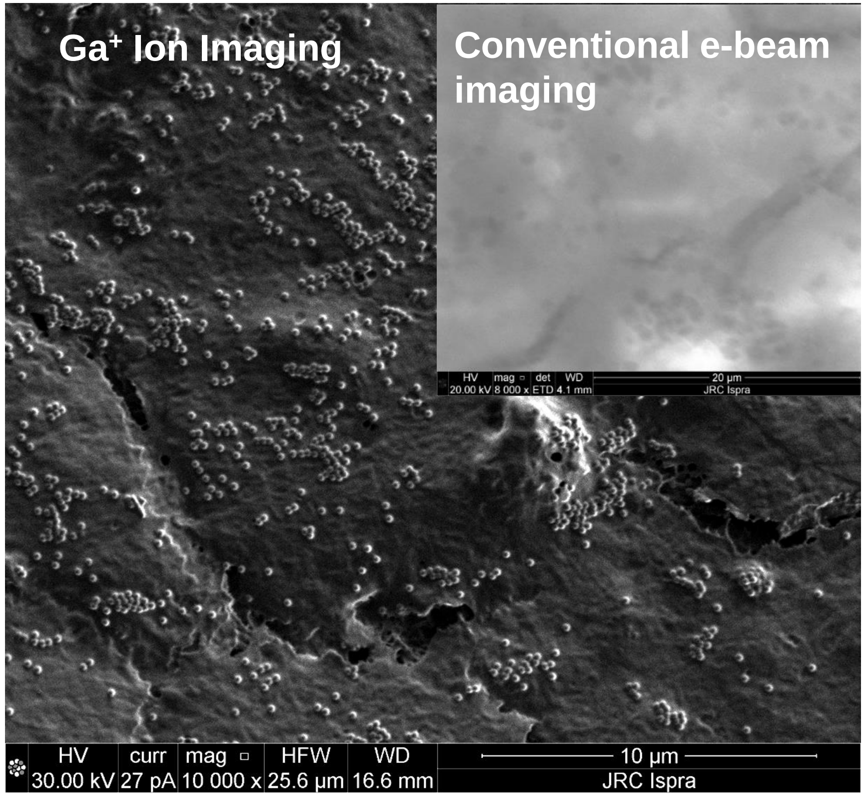
Developing or adapting existing methods to use with micro(nano)plastics (<10µm)

Novel analytics : Novel detection methods (FIB-SEM)

MPs/NPs imaging SEM with Ga^+ Ion-beam



Cell tissue spiked with polymeric particles (PS 200 nm)
Ga^+ ion-beam induced secondary electron image

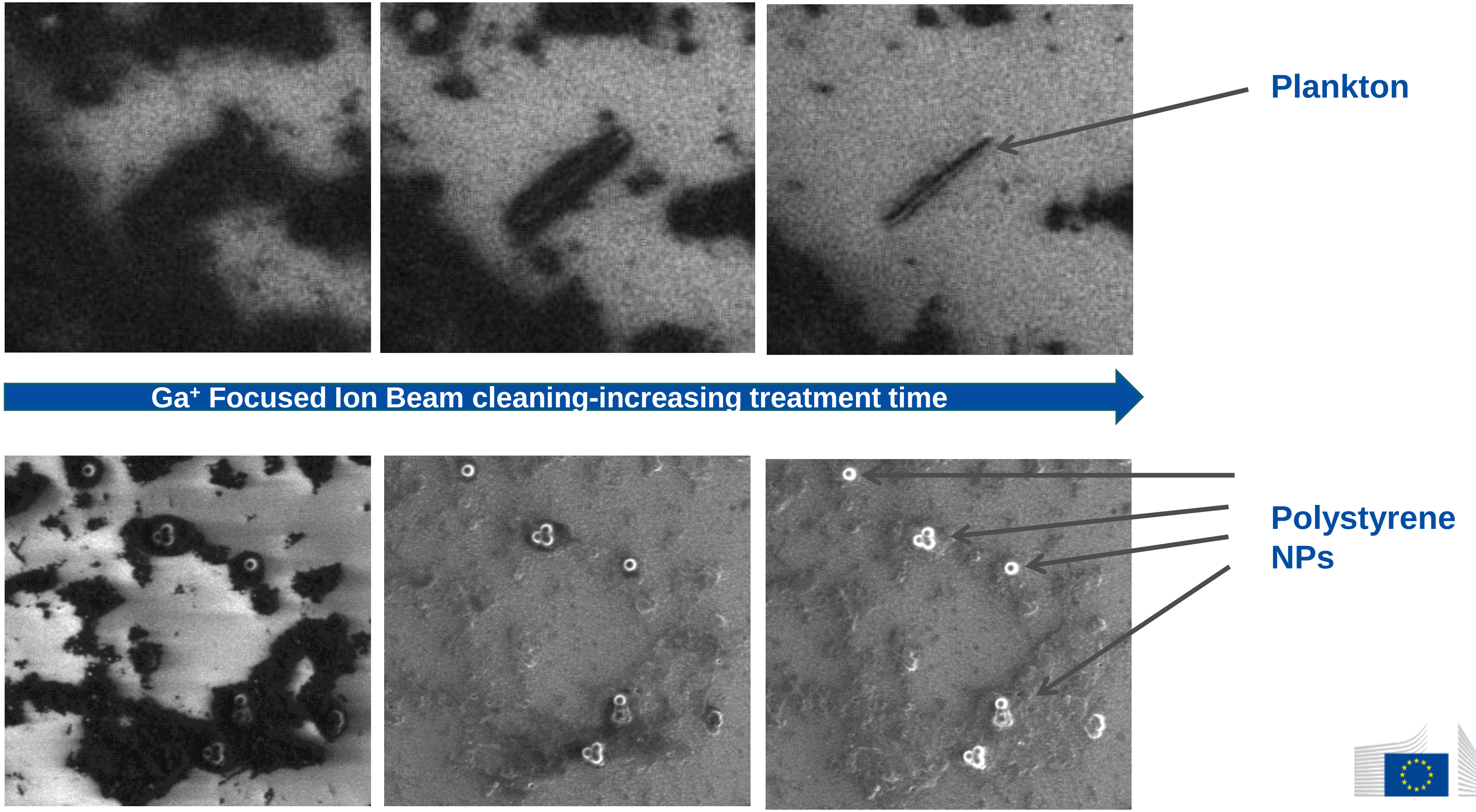


Secondary electrons produced by ion beam impingement (Ion Imaging) give better contrast and resolution for polymeric particles in cells (PS 200 nm)

European Commission

Enhancing imaging quality by FIB cleaning with Ga^+ Ion Beam

- **Selective removal of organic matrix (mussel tissue)**

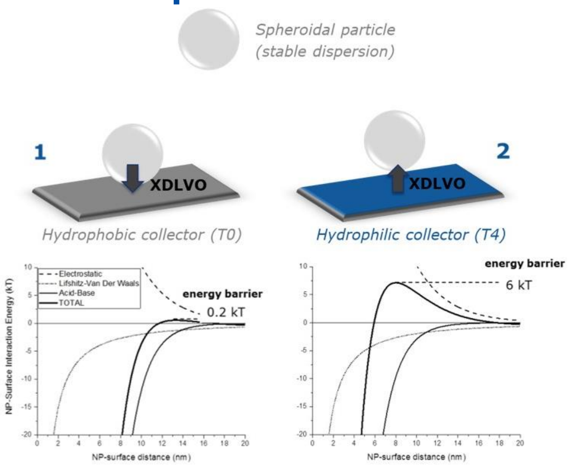


Other specialised characterisation tools

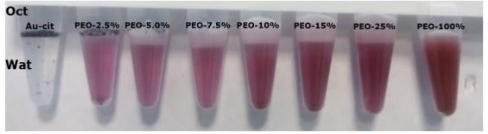
Nanoparticle hydrophobicity measurements

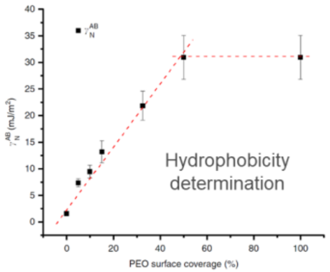
"Direct quantification of nanoparticle surface hydrophobicity"
Valsesia et al.
Communications Chemistry volume 1, Article number: 53 (2018)

- From proof-of-concept



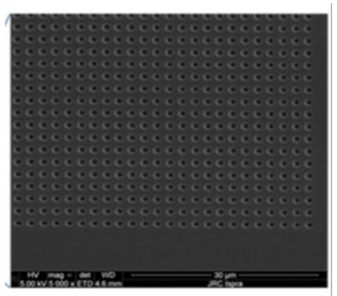
- to OECD Test Guideline
- ➔ Interlab. comparis
  ongoing



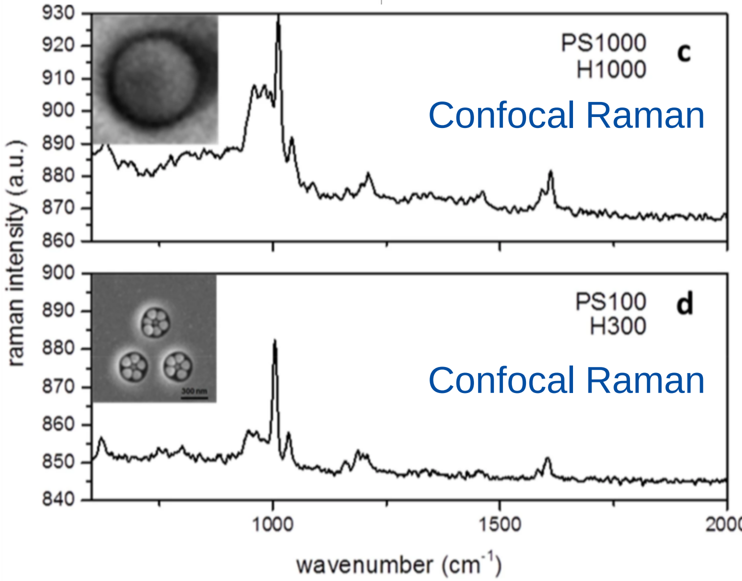


Sub-micron single particle Raman analysis

"Combining microcavity size selection with Raman microscopy for the characterization of Nanoplastics in complex matrices"
Valsesia et al
Scientific Reports volume 11 Article number: 362 (2021)



micro cavity array for size selective particle trapping



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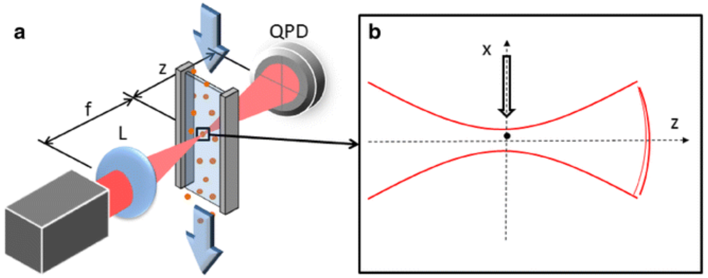
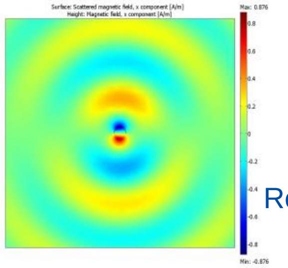
Advanced Single Particle Detection Technology

Single Particle Extinction and Scattering (Mariani et al.)



Single Particle

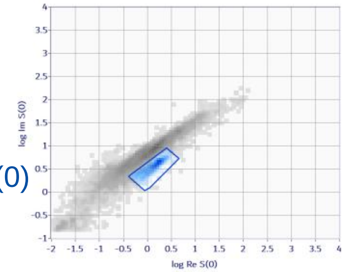


Optical Pattern
(transmitted and scattered fields)

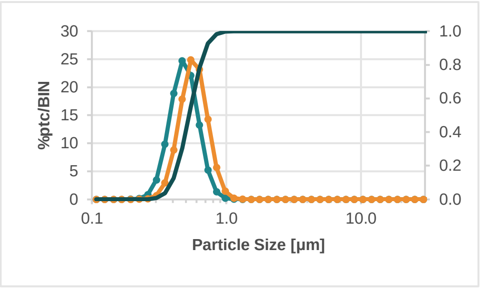
Accumulate Multi-Particle Data

Re S(0) and Im S(0)

SPES "Cloud"
500nm PS particles in River (Tiber) water



2D histograms: the optical fingerprint of the sample- extinction cross section and scattered intensity:
=> size and refractive index



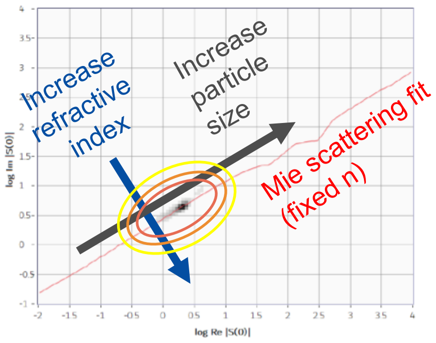
Information is extracted using unique particle data libraries

Analysis of 2D Histogram of independent (optical) parameters of each single measured particle

- Distinguish populations with different compositions
- Determine number based size distributions and concentrations
- Aggregation

Sample analysis possible on-line - potential as detector for size fractionation instruments (CF3, AF4)

SPES "Cloud"



Mariani et. al.
Single Particle Extinction and Scattering allows novel optical characterization of aerosols
Journal of Nanoparticle Research August 2017, 19:291

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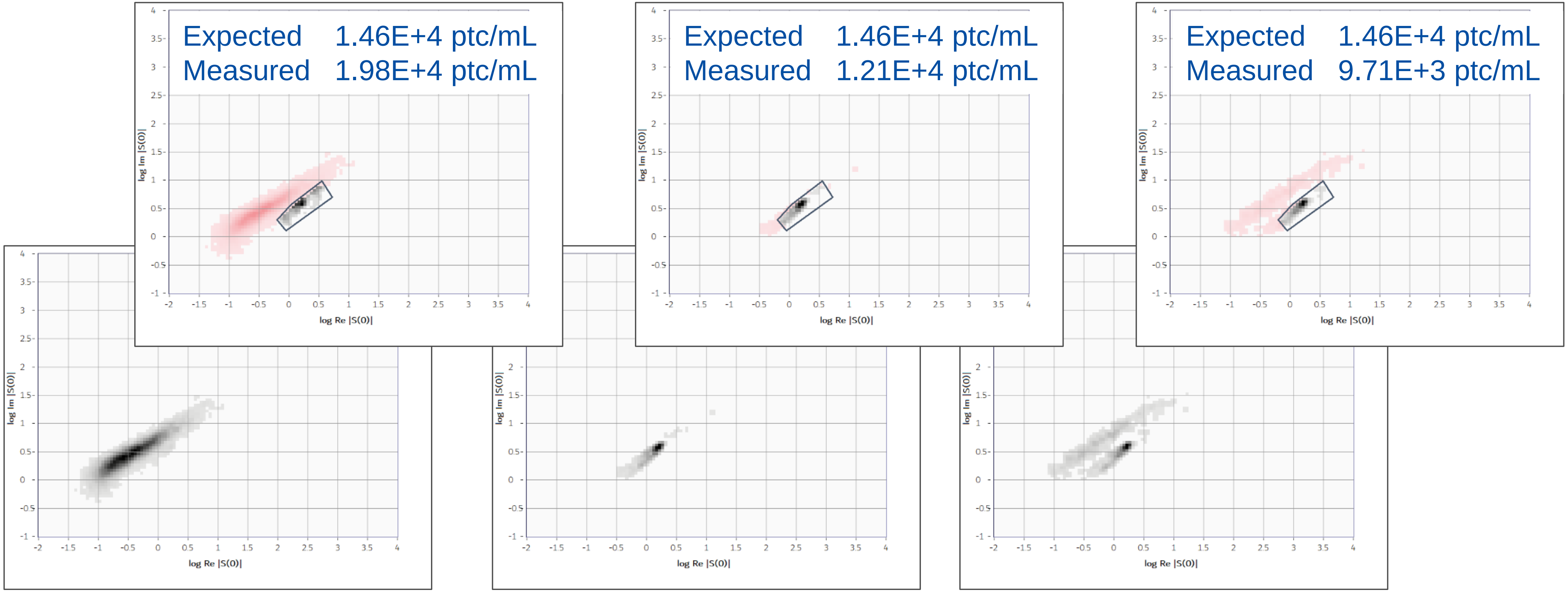
Particle classification – environmental media

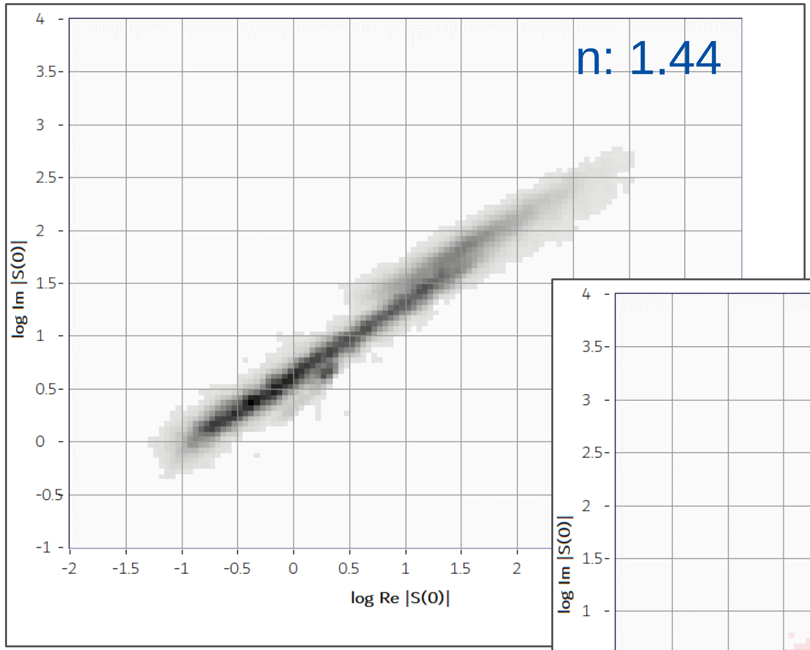
Water samples spiked with PS 0.5 µm

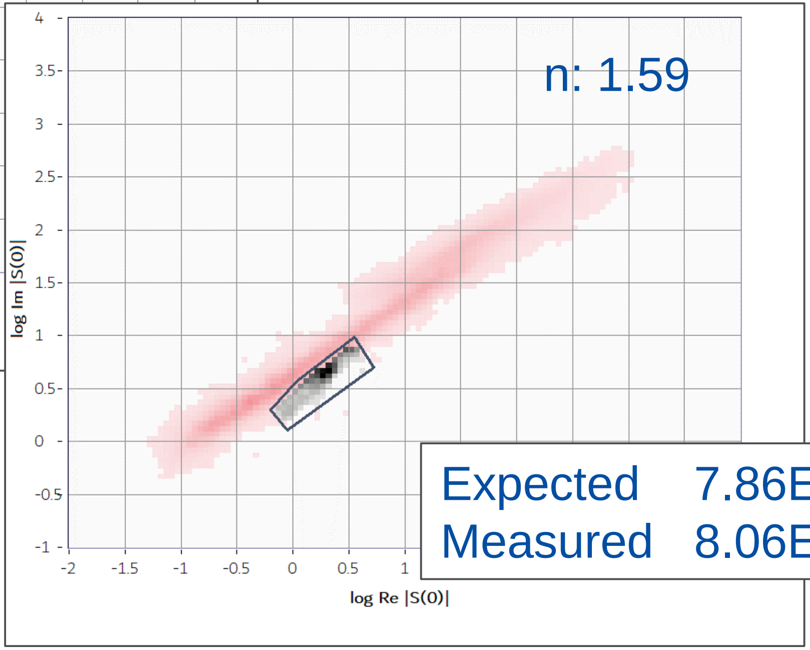


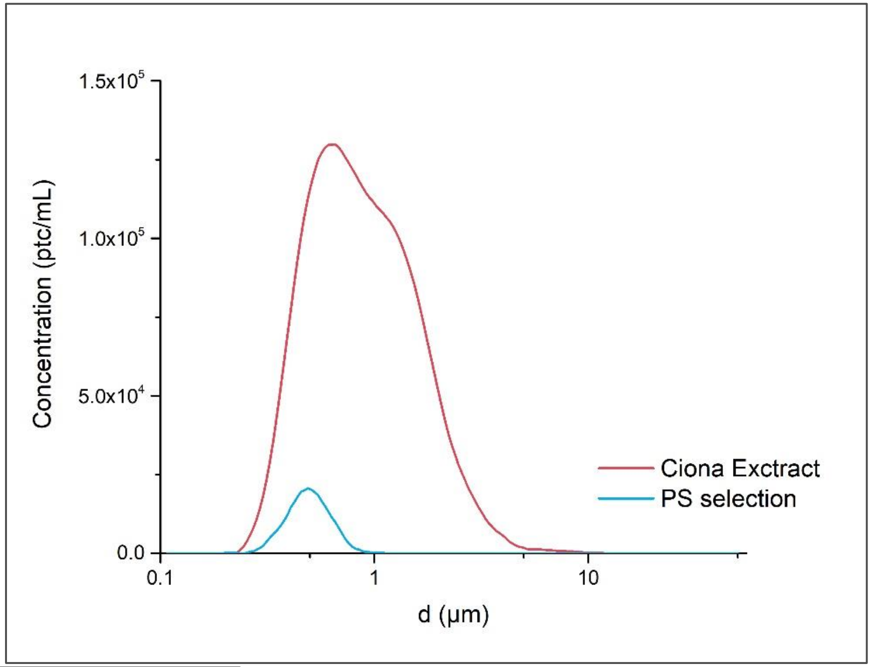
Tap water MilliQ water Mineral water

Particle classification – complex media

Extract from Ciona robusta (dilute in H_2O) spiked with PS 0.5 µm







Expected	7.86E+4 ptc/mL
Measured	8.06E+4 ptc/mL

Thanks to Valsesia A. (JRC) for samples preparation

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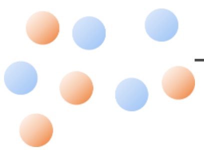
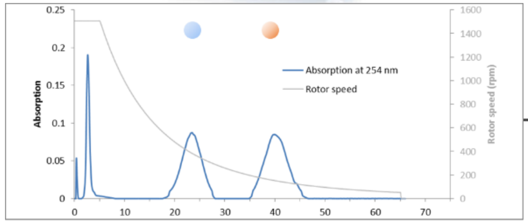
Novel analytics : Preliminary tests of CF3-SPES in-line coupling

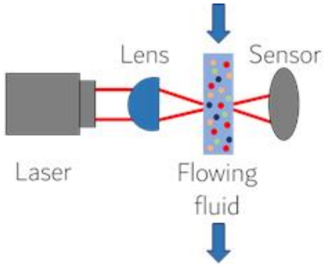
500nm PVC and PS Mix



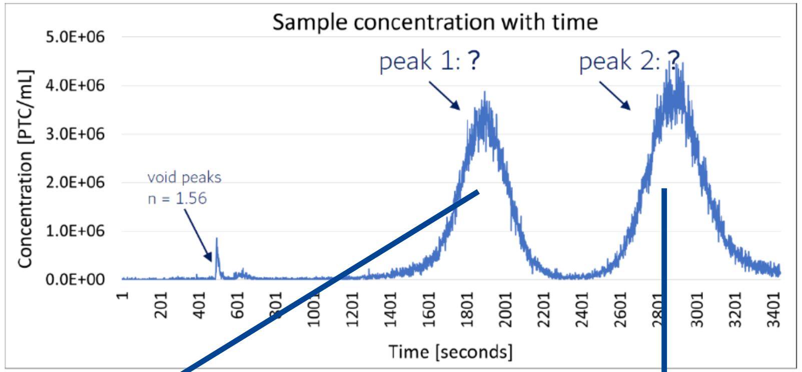
CF3 fractionation



Single Particle Extinction and Scattering)



Time resolved particle number concentrations



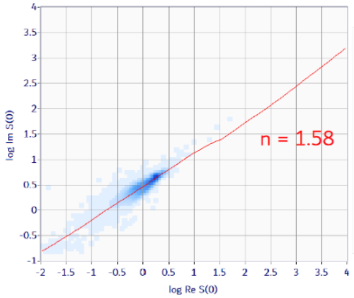
Combination CF3-SPES techniques

- Particle number concentration
- Size, Refractive index (SPES)
- Density(CF3)

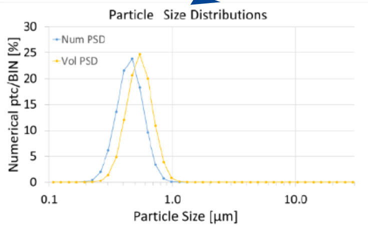
Advantages

- Complimentary to DLS/MALS in FFF methods
- Good sensitivity wrt DLS (on-line)
- On-line analysis with compositional specificity

Elaboration of single particle data in elution peak 1

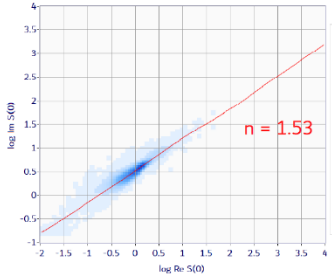


Optical fingerprint

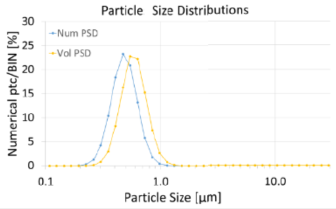


Size distribution

Elaboration of single particle data in elution peak 2



Optical fingerprint



Size distribution

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Concluding remarks

Microplastics- Basic instrumental tools are available for size/number/identity or identity/mass

- Refine, validate and harmonize/standardise methods/protocols – understand the limitations – dependant on sampling and sample preparation methods
- Standards and reference materials critical for method development, quality control and laboratory development
- Methods are slow – need to develop appropriate capacity for measurement – monitoring and/or research
- Reliable end effective sample pre-treatments and extraction methods will be key factor for all methods

Nanoplastics

- No single instrumental method available for nano-range – need combination or hyphenated methods and extensive development
- Knowledge of the critical size ranges for hazards would help prioritize method development.

Key requirements

- Know exactly what questions that we need to answer and consequently what descriptors we must measure
- Know why we are asking the questions – environmental monitoring, legislative control, basic research for exposure/hazard/risk assessment
- Availability of standards and generalised acceptance of minimum quality controls

Thank you

Acknowledgments
D.Mehn, R.La Spina, C.Cella, J.Ponti, A.Valsesia, F.Fumagalli, O.Geiss, I.Bianchi

European Commission

European Commission