SHIMADZU
Excellence in Science

Automatic Sample Preparation Device for Monitoring Microplastics in Water

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Today’s presentation

1. Comprehensive workflow
2. Automated sample preparation
3. Experimental plan
4. Results
5. Take-home messages

Comprehensive Workflow for Microplastics Analysis

Microplastics

Nondestructive

FT-IR

Raman Infrared Microscope

EDX

Destructive

Py-GCMS

Thermal analyzer

MALDI

Trace and sorbed components

Direct analysis

Nondestructive

EDX

FT-IR

Destructive

Py-GCMS

Analysis of extracted solvent

LCMS

GCMS

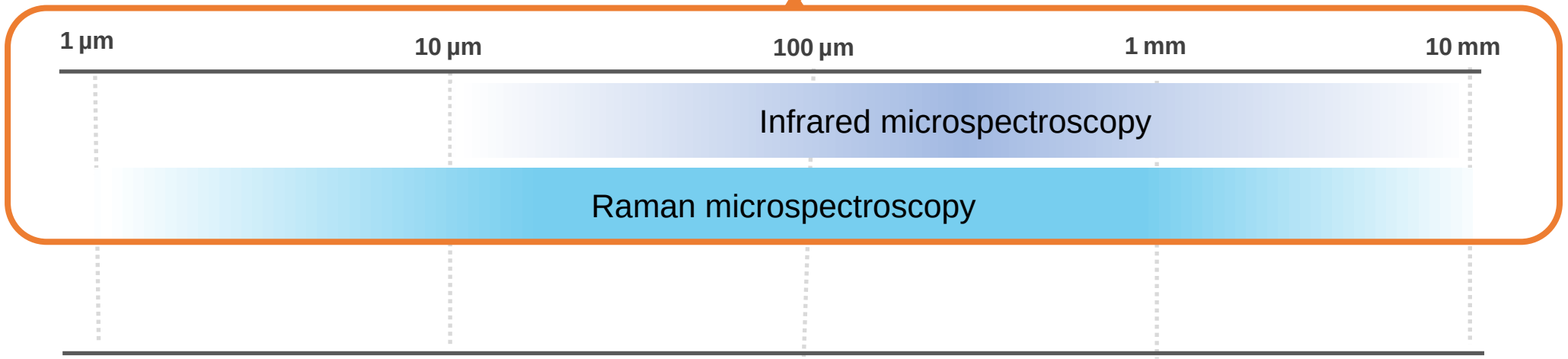
Presented at NEMC 2021; follow-up presentations on specific techniques at NEMC 2022

Comprehensive Workflows – Practical Approach

Sample Collection → Sample Preparation → Non-destructive Sample Characterization → Destructive Sample Characterization

Comprehensive workflows – Practical Approach

Sample Collection → Sample Preparation → Non-destructive Sample Characterization → Destructive Sample Characterization



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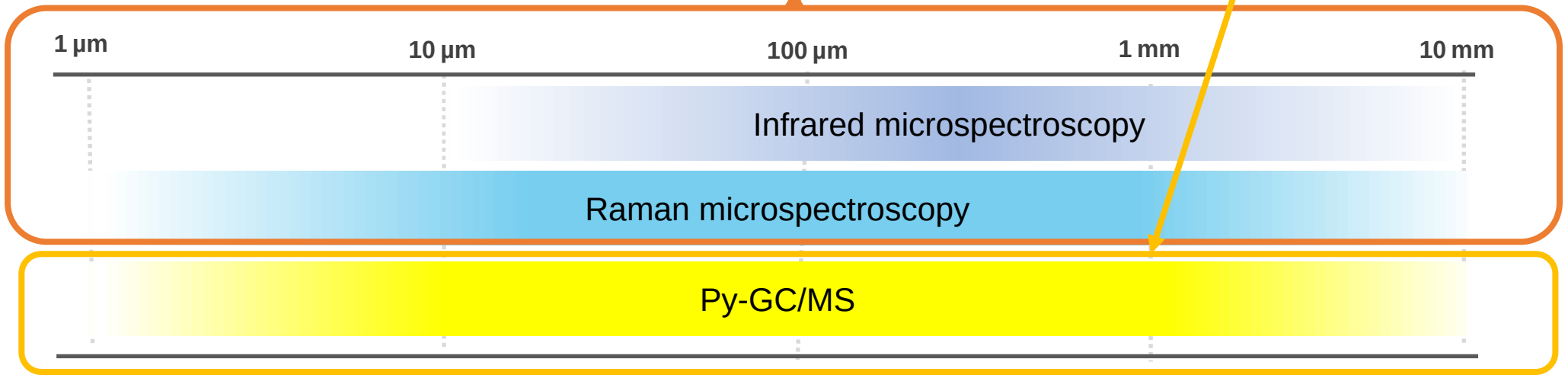

Efforts by different international standardization groups and other organizations to provide guidelines and methods for each step of the workflow

Comprehensive workflows – Practical Approach

Sample Collection → Sample Preparation → Non-destructive Sample Characterization → Destructive Sample Characterization

NEMC 2023 Poster:
Dual Microscopy Technique for the Analysis of Microplastics

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NEMC 2023 Poster:
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Sample Preparation for Microplastics Analysis

Goal: to remove organic and inorganic contaminants

Sampling

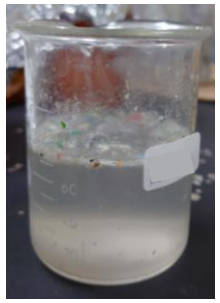
Sieving

Sample preparation

Analysis

- ✓ Manta net
- ✓ Neuston net

Digestion ➤ Separation ➤ Transfer



Different reagents are commonly used

Reference:
https://www.env.go.jp/en/water/marine_litter/guidelines/Guidelines_for_River_Microplastic_Monitoring_Methods_Marge.pdf

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Microplastic Automatic Sample Preparation Device

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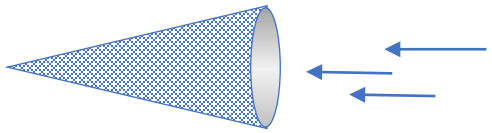
Experimental Plan

River sample



Manta, Neuston or plankton net
Net Mesh 300 µm
18 m^3

1.0945 g

Set in the strainer

Preparation device

Collection

22.25 mg

Cryomilling

Before

After

Analysis by technique of choice (Py-GCMS)

Experimental Plan

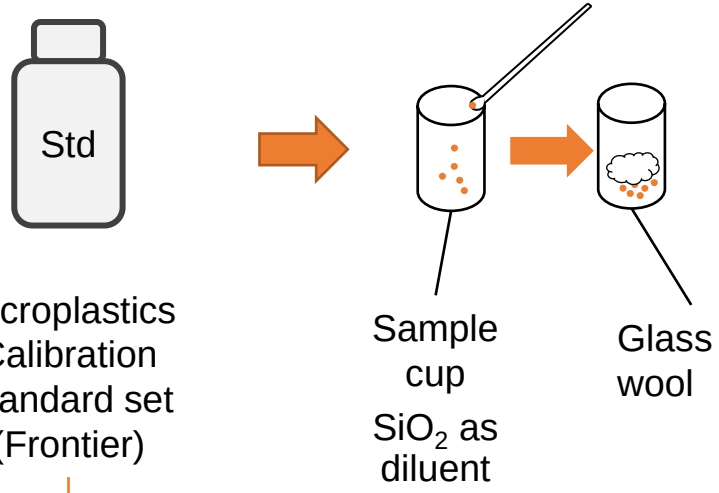
Digestion and density separation are automated resulting in:
- minimization of labor and bias
- Increased safety in laboratory

1. Digestion of organic compounds via a hydrogen peroxide solution in the reaction vessel.
2. Density separation via a sodium iodide solution in the reaction vessel.
3. The supernatant fluid discharged by the overflow is filtered out by the collection filter.
4. Collect microplastics for the next qualification/quantification process.

Reagents recommended in the Guideline from Ministry of Environment (Japan)

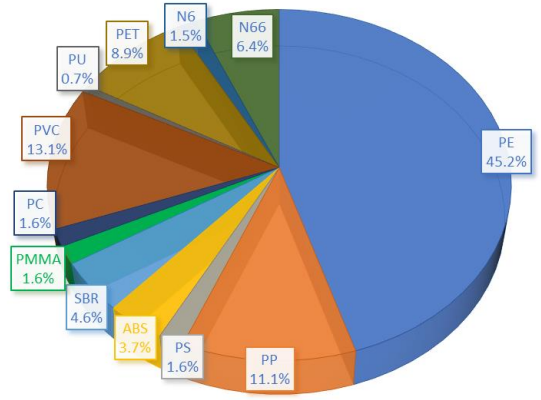
Reference:
https://www.env.go.jp/en/water/marine_litter/guidelines/Guidelines_for_River_Microplastic_Monitoring_Methods_Marge.pdf

Experimental Plan (Analysis by Py-GCMS)

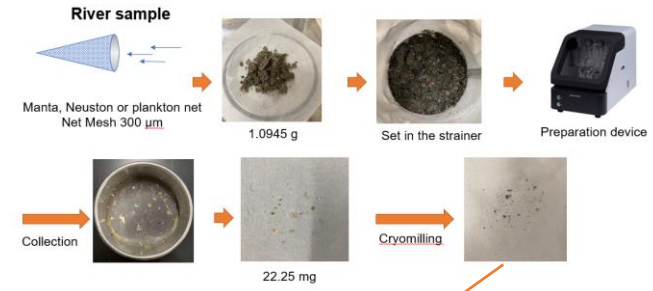


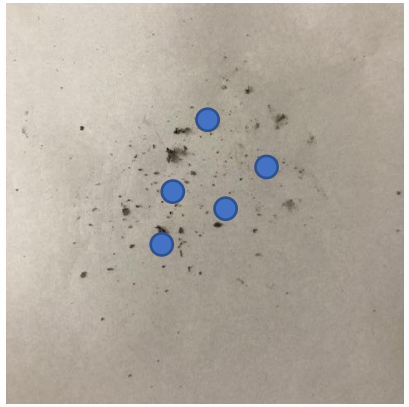
Calibration curve	Weight (mg)
1	0.39
2	2.08
3	4.05

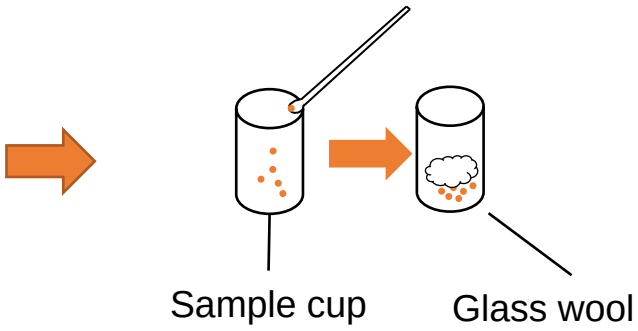
Polymer
PE
PP
PS
ABS
SBR
PMMA
PC
PVC
PU
PET
N6
N66



Experimental Plan (Analysis by Py-GCMS)

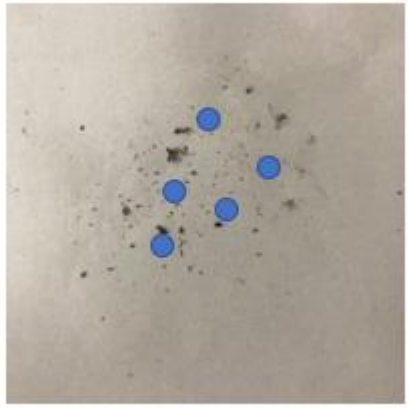


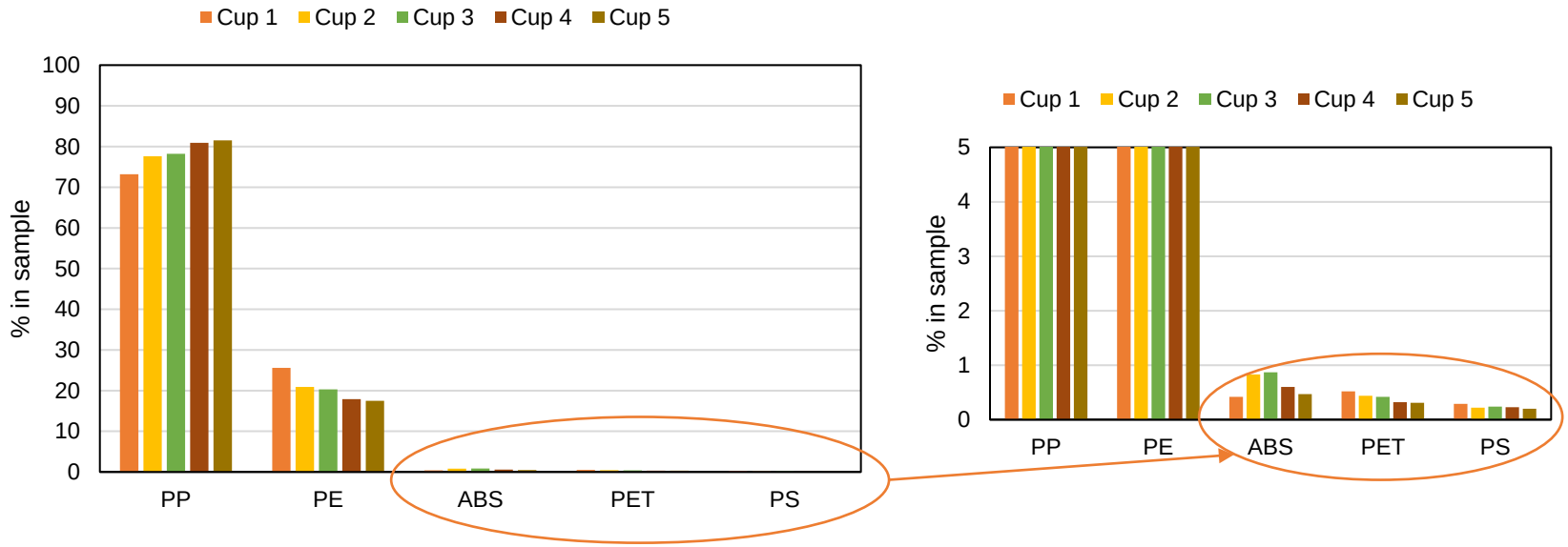




Sample cups	Weight (mg)
1	0.10
2	0.23
3	0.14
4	0.10
5	0.23

Results

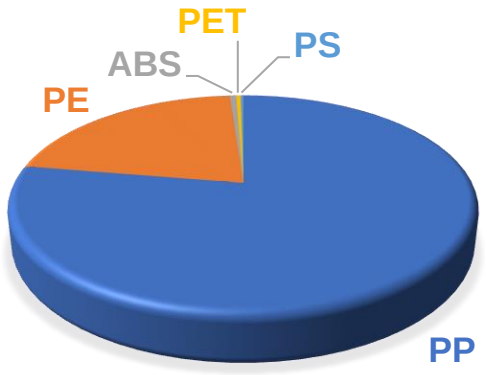


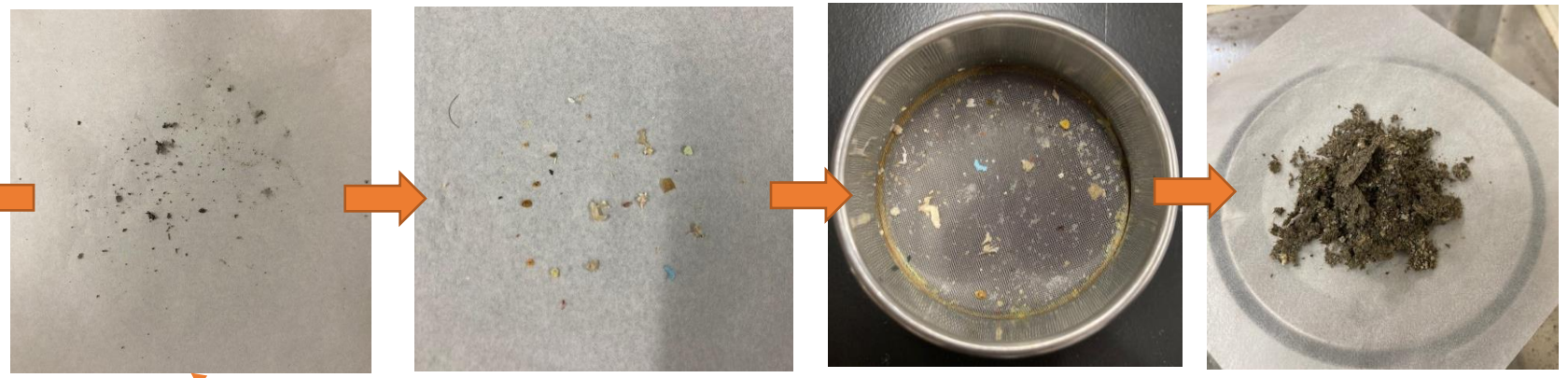


Identified Polymers[1]	Ave	Range	SD	CV
PP	78.28	73.2 - 81.5	3.30	4.2%
PE	20.44	17.5 - 25.6	3.24	15.8%
ABS	0.64	0.42 - 0.87	0.20	32.1%
PET	0.40	0.31 - 0.52	0.09	21.9%
PS	0.24	0.2 - 0.29	0.03	14.2%

[1]Based on ≥85% similarity in F-Search MP Library Software

Results





Digested sample
(22.25 mg)

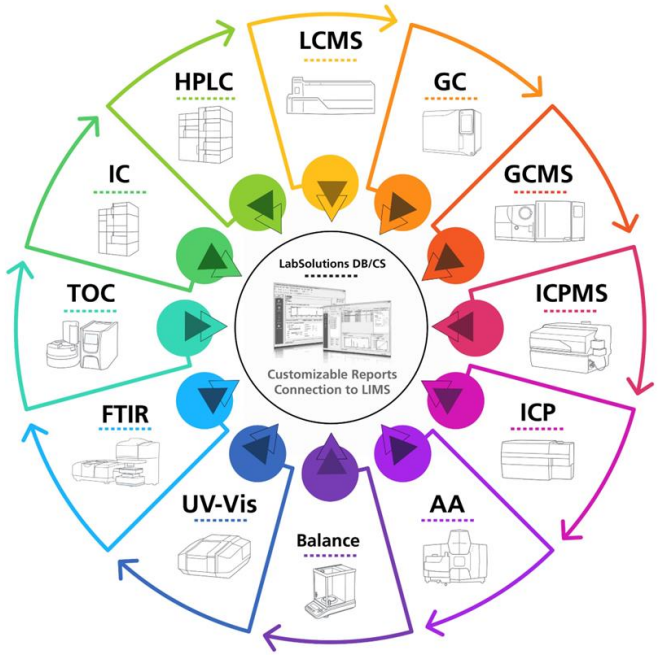
Filtered sample
(1.0945 g)

Polymer	Ratio (%) in standard
PP	77.48
PE	21.22
ABS	0.525
PET	0.377
PS	0.231

Polymer	Ratio (%) in analyzed sample	Weight (mg) in digested sample	Ratio (%) in filtered sample
PP	78.28	17.24	1.57
PE	20.44	4.72	0.43
ABS	0.64	0.12	0.011
PET	0.40	0.08	0.007
PS	0.24	0.05	0.004

Take-home messages

- The suitability of an automated sample preparation device for the analysis of microplastics in environmental samples is demonstrated.
- Additional work on-going to determine specific performance parameters, as part of an international method standardization effort.
- Py-GCMS analysis can be successfully applied to digested samples.



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