"Microplastics"

Environmental Pollution

PRESENTED BY

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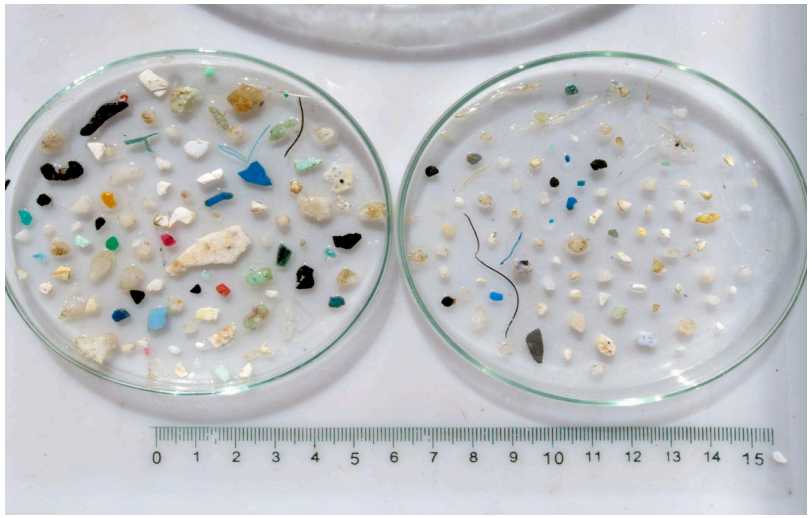
GC GCMS Product Specialist

Topic

- Microplastics (MPs)
- Analytical Method for MPs
- Microplastic Analysis by TD-GC/MS
- Microplastic Analysis by Py-GC/MS

Microplastics (MPs)

Microplastics are any type of plastic polymer less than 5 mm in length*



*Source: NOAA

Primary MPs

Particles designed for commercial use

Secondary MPs

Particles that result from the breakdown of larger plastic items

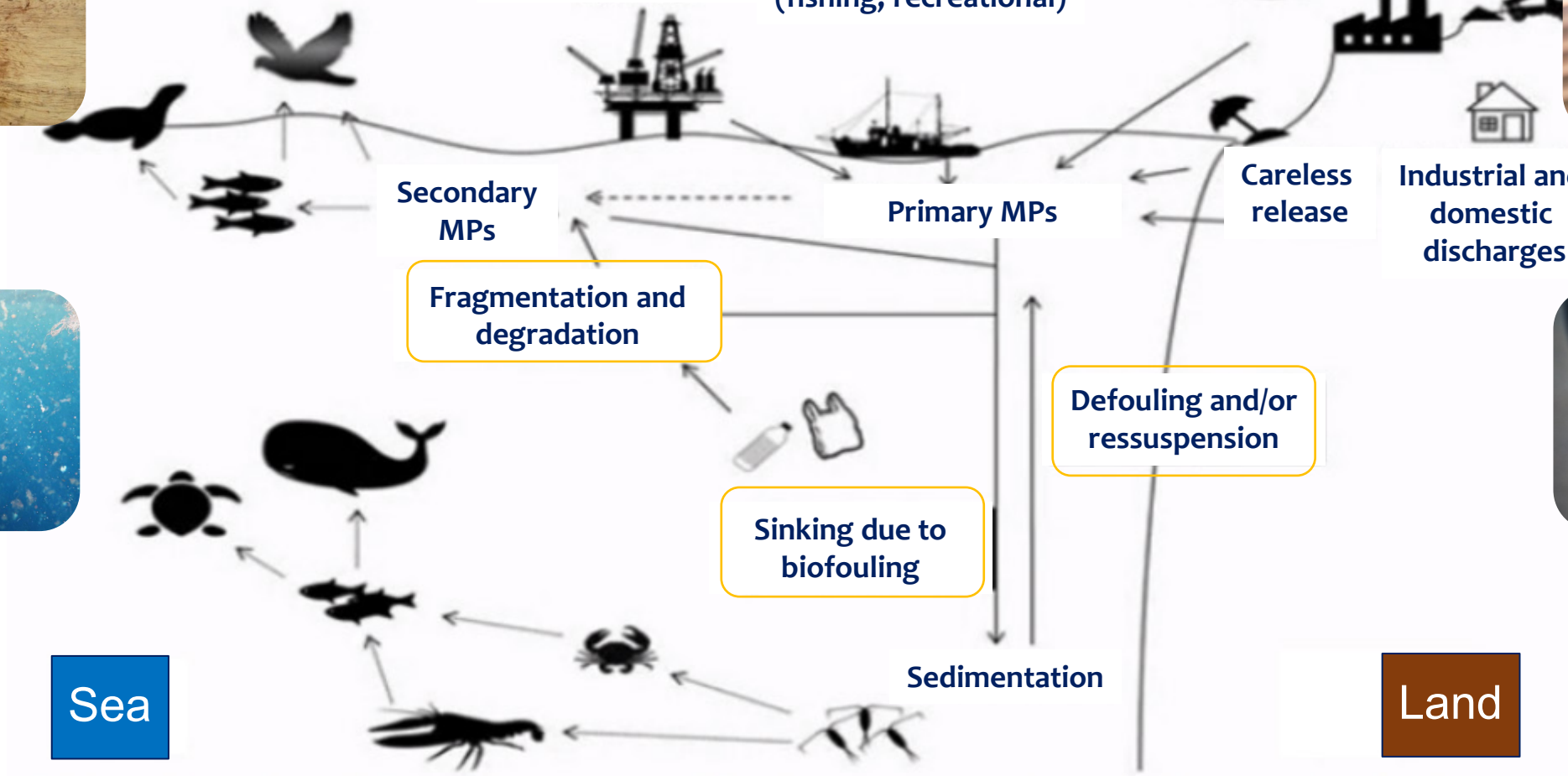
Fate and pathways of MPs

in food

in air

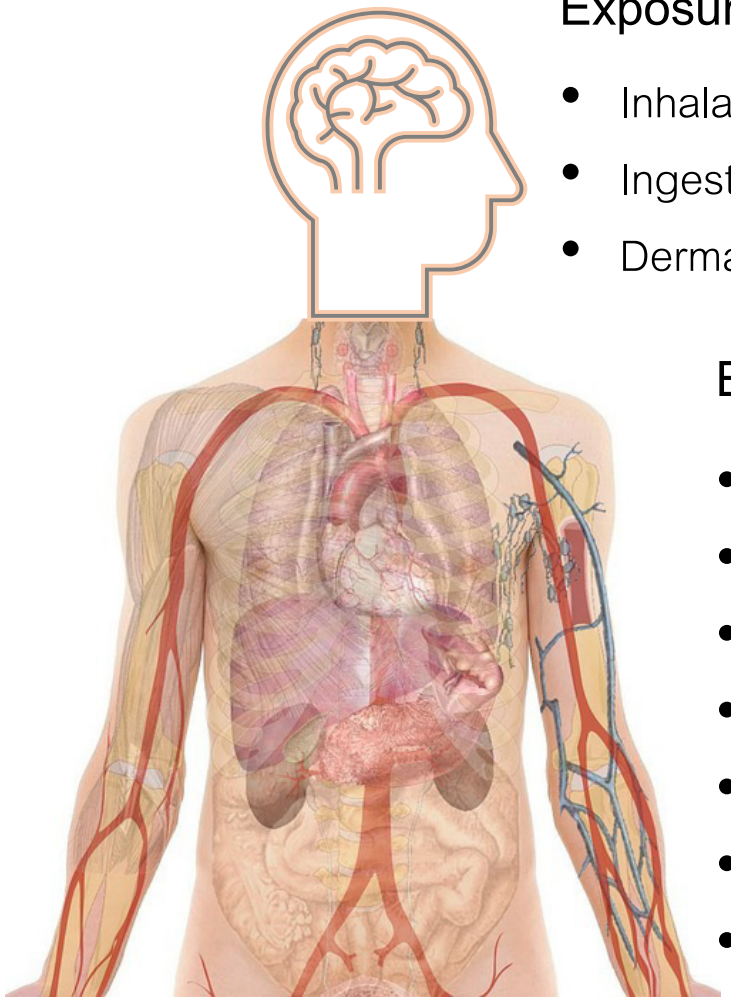
in water

in soil



Adapted from W.C. Li, H.F. Tse, L. Fok, Plastic waste in the marine environment: a review of source, occurrence and effects, Sci. Total Environ. 566-567 (2016) 333-349.

Toxicological Effects in Human Health

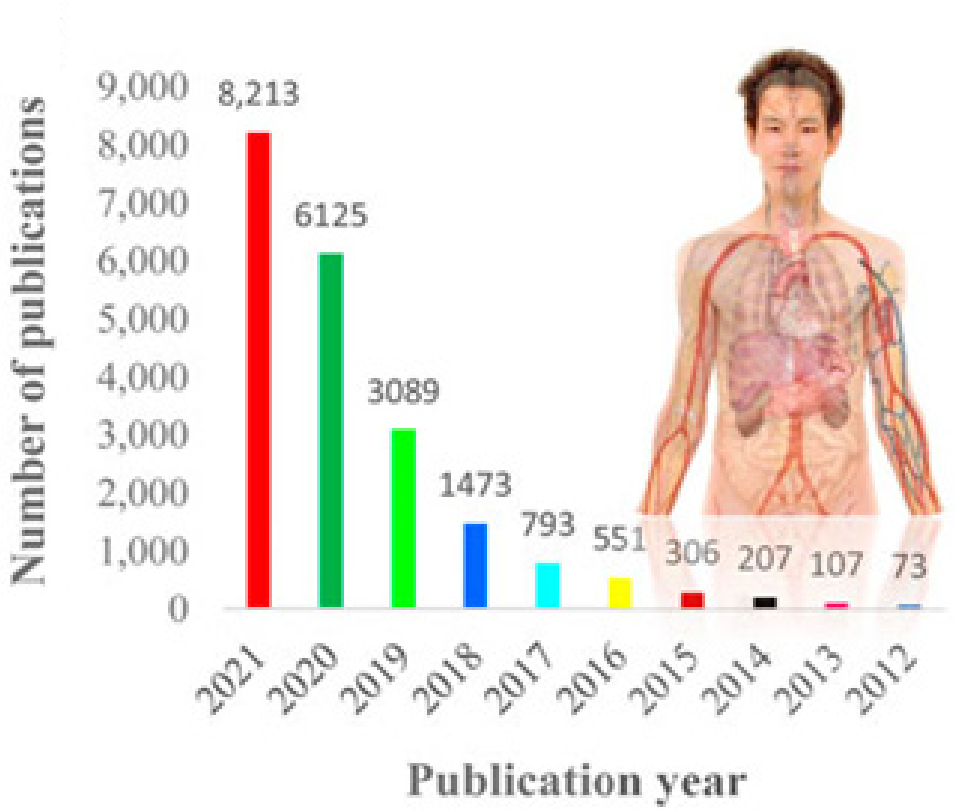


Exposure routes of MPs in body

- Inhalation
- Ingestion
- Dermal contract

Effect of MPs in human

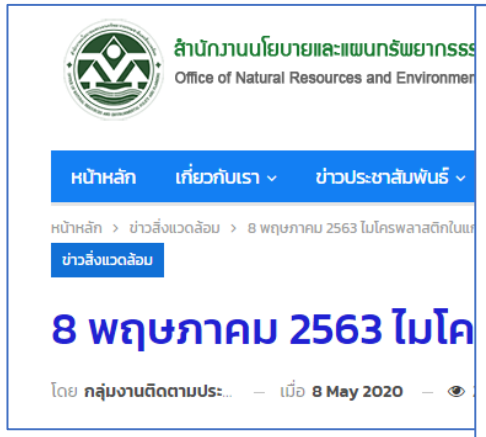
- Translocation to distant tissues
- Disruption of immunity
- Metabolism alteration
- Oxidative stress
- Cytotoxicity
- Neurotoxicity
- Carcinogenicity
- Reproductive toxicity

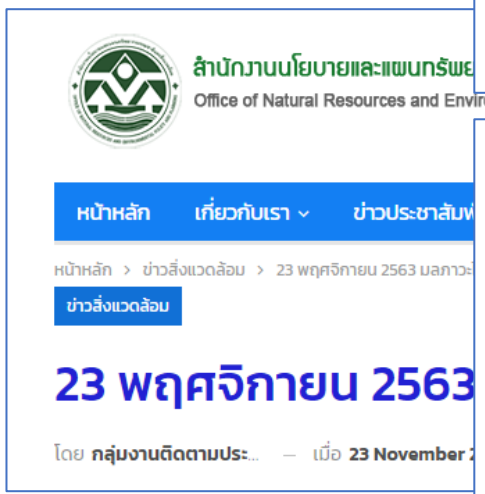


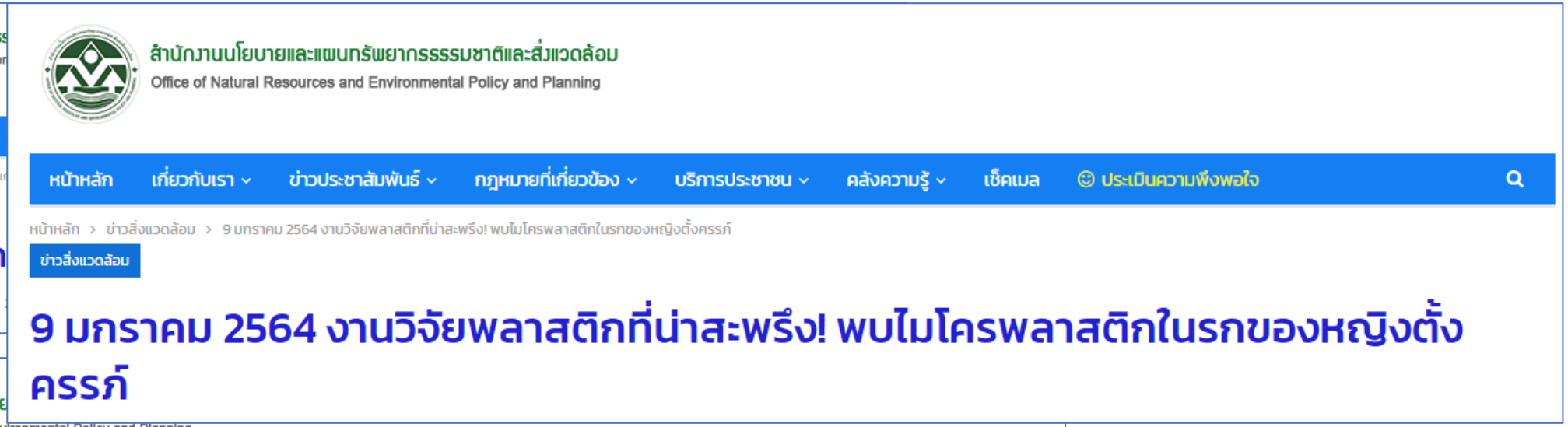
MPs effect related research trends on human.

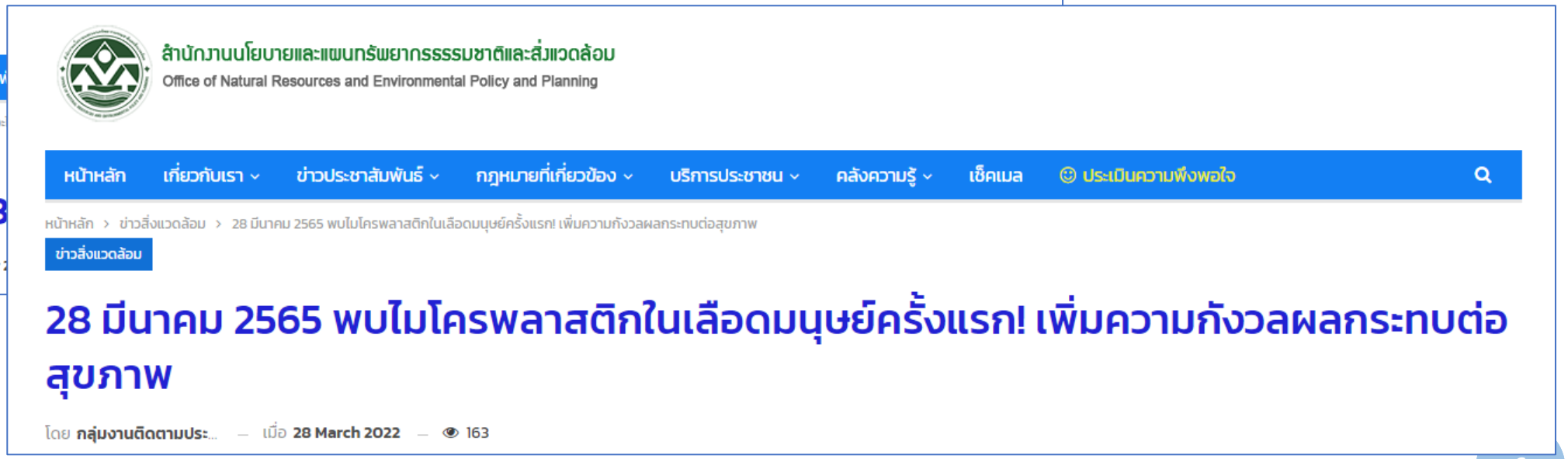
Source: https://www.frontiersin.org/articles/10.3389/fenvs.2022.827289/full

Microplastic contamination









Regulatory & standards landscape

- **UN**: Commissioned a report - Legal **Limits on** ***Single-Use Plastics*** and ***Microplastics***: A Global Review of National Laws and Regulations

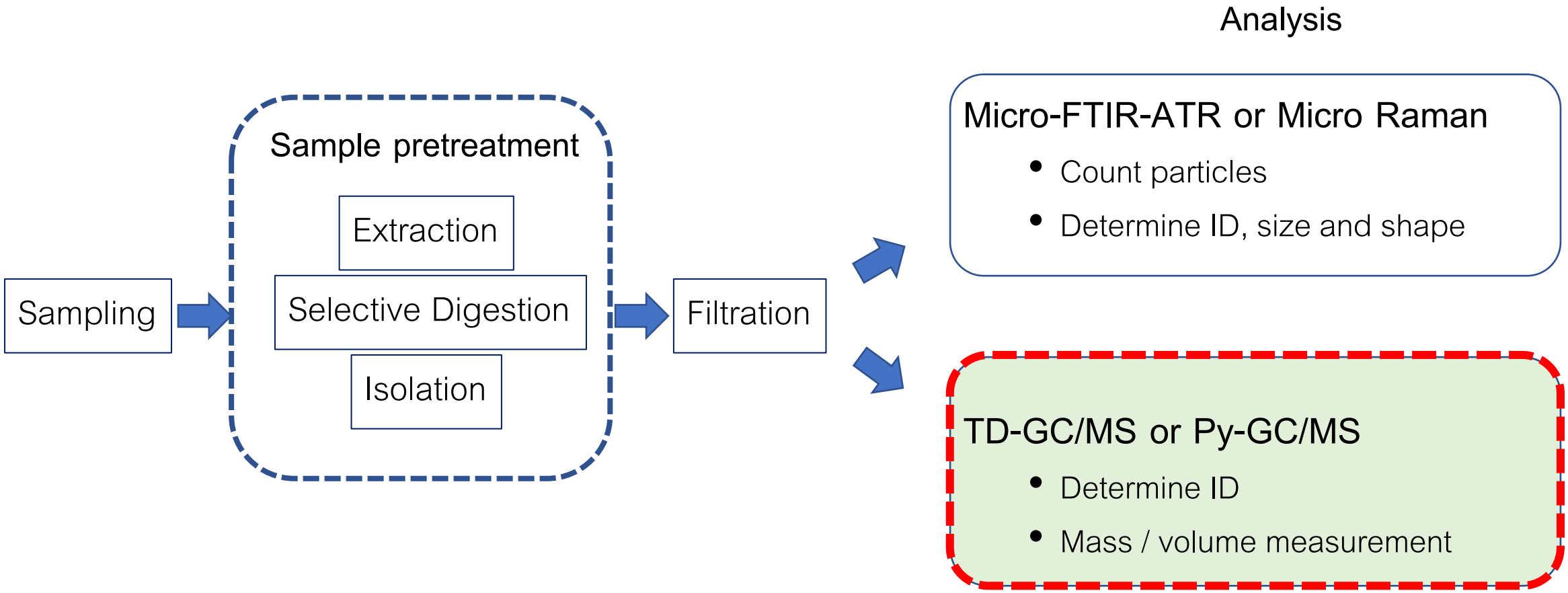
- **Europe**: ECHA's Committee for Risk Assessment (RAC) currently in a consultation period. Expected to amend REACH Annex XV to include some MPs.

- **WHO**: Released report in 2019, covers risk to human health, identifies knowledge gaps, recommendations for management actions.

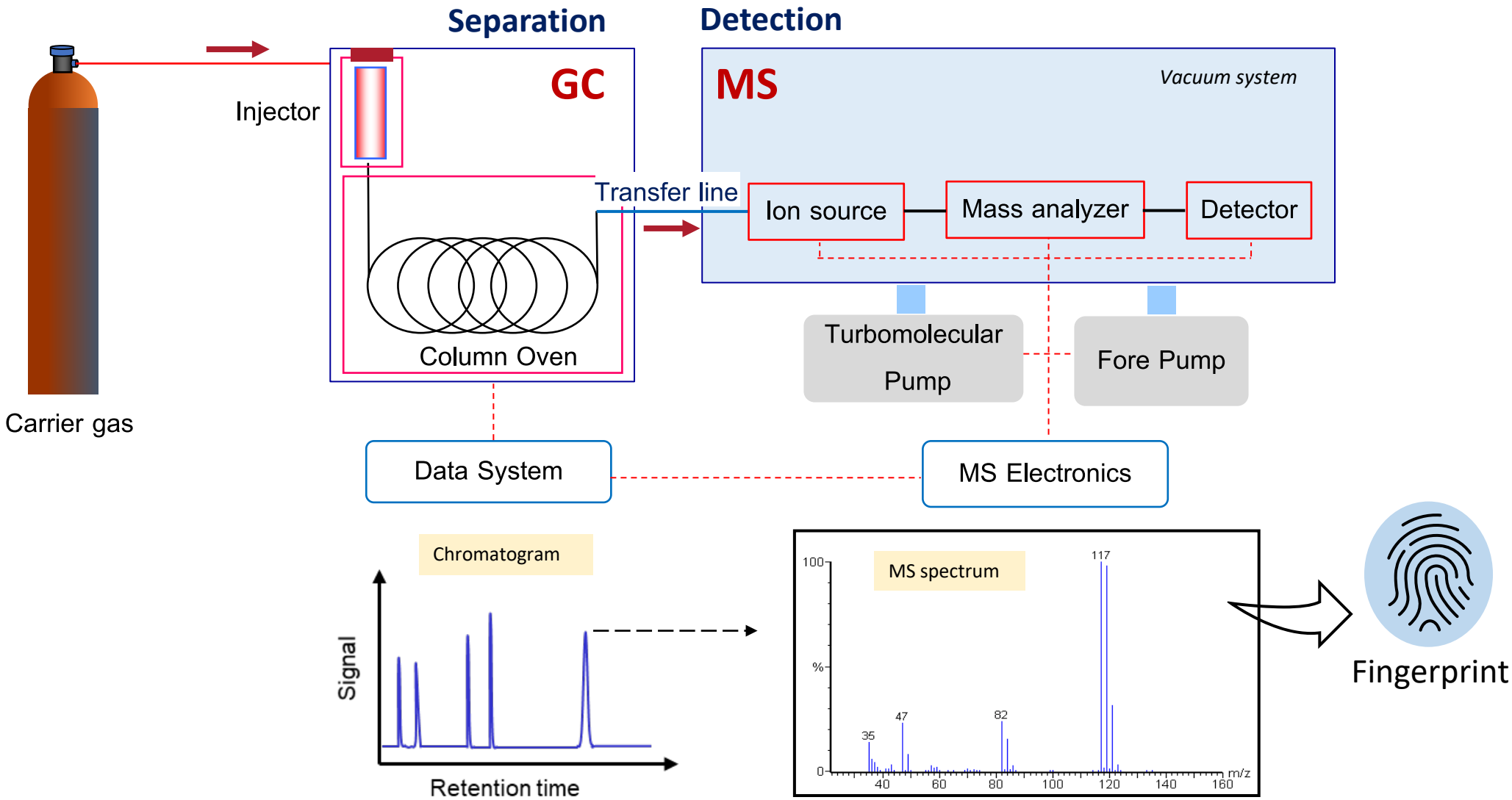
- **ISO/CD 24187**: "Principles for the development of standards for investigation procedures of plastics in environmental matrices and related materials"

- **ASTM WK67788**: New Test Method for Identification of Microplastic Particles and fibres in Municipal Wastewate.

Analytical Method for MPs

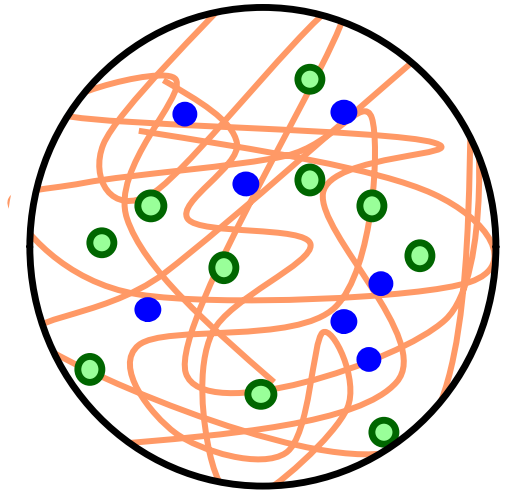


Gas Chromatography / Mass Spectrometry; GC/MS component



Microplastics analysis by GC/MS

Pattern diagram of
typical polymeric material



: Additives

: Polymer

: Inorganics

1.Thermal
Desorption, TD

Heat energy

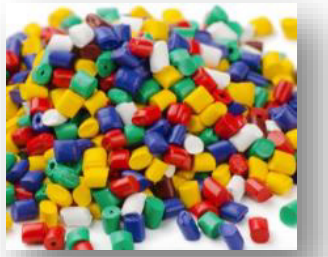
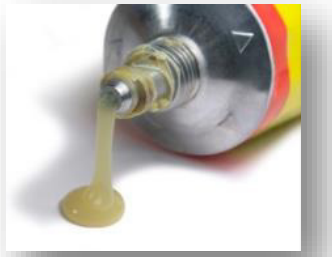
2. Pyrolysis, Py

- Evolved VOCs are separated and analyzed.
- Marker compounds used to identify the presence, and measure the concentration, of specific plastics.

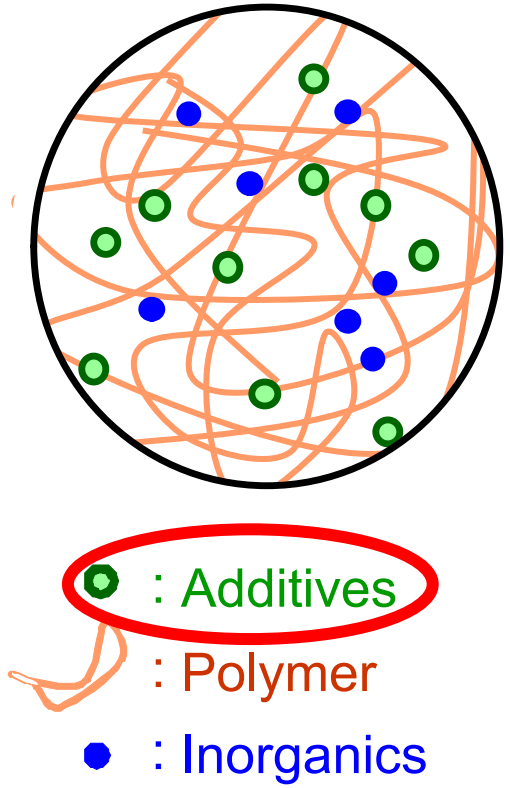
Thermal desorption (TD)-GC/MS

- Thermal desorption is arguably the world's most versatile, readily-automated injector mechanism for gas chromatography.

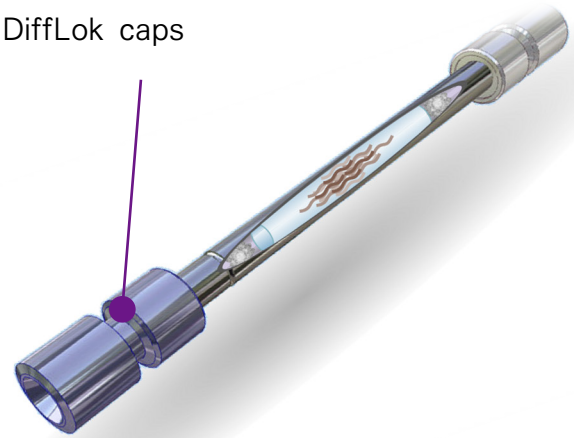
- An alternative to solvent extraction for measuring VOC and SVOC compounds in many different sample matrices – solids, liquids or gases


Pattern diagram of typical polymeric material



Sample Preparation for analysis MPs (PET) in drinker water



- Hundreds of mL to liters of water are filtered
- The filter is then washed with multiple reagents to remove organic matter
- Dried filter is then placed directly into an empty thermal desorption tube for analysis


Filtering
0.2µm quartz filter


Washing
with reagents


Drying
30min at 100˚C


Quick Preparation
filter into TD-tube

1 h
Sample
preparation
time

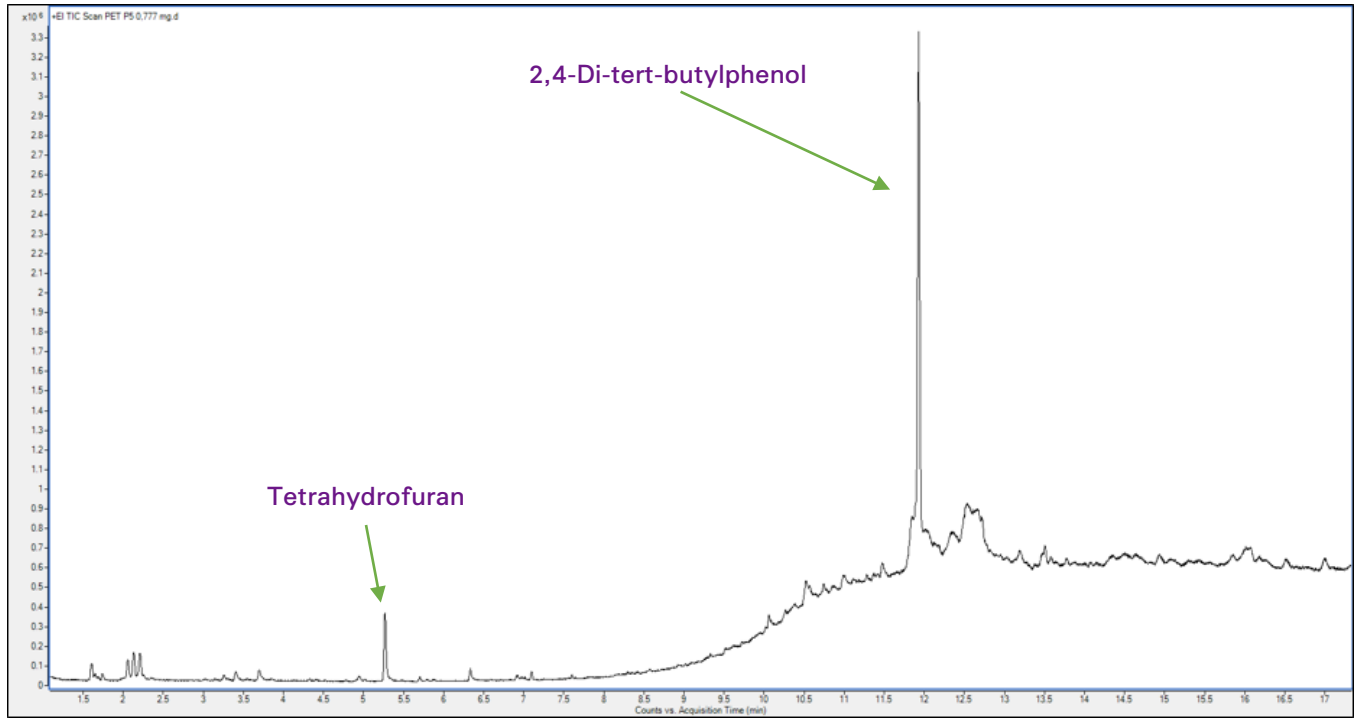

Direct Desorption at
320˚C

30 mins
analytical cycle

Polyethylene terephthalate (PET) in water

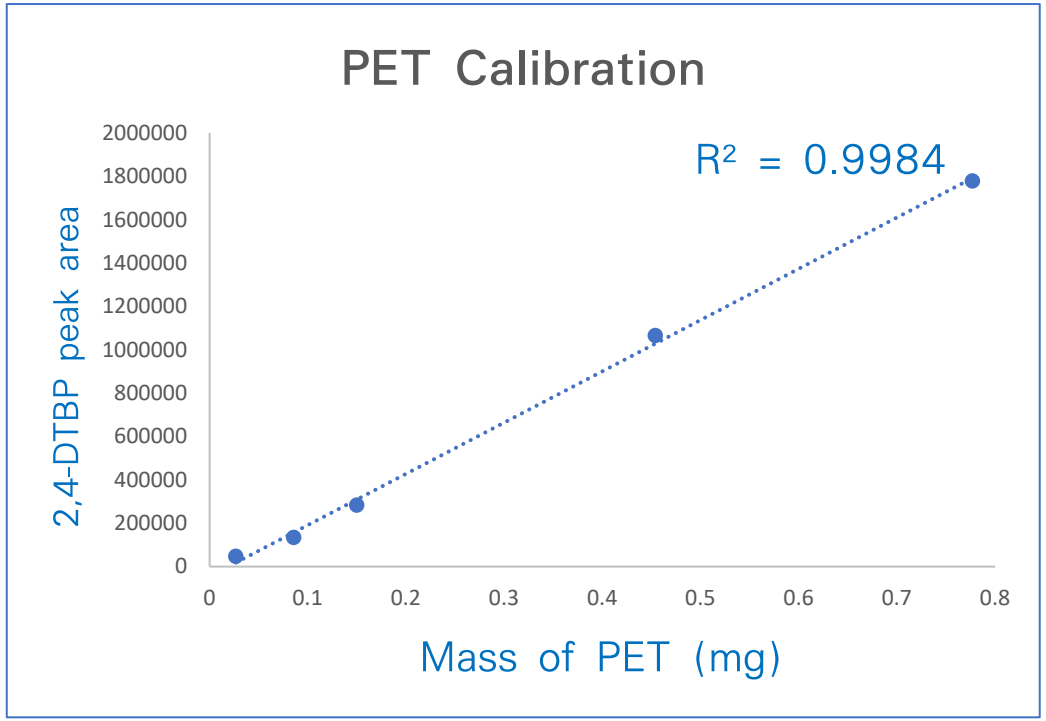
Identify marker compounds for PET by TD–GC–MS analysis of standard pellets.

- 2,4-di-tert-butylphenol (2,4-DTBP) used as quantitation marker
- Tetrahydrofuran is used to confirm presence of PET

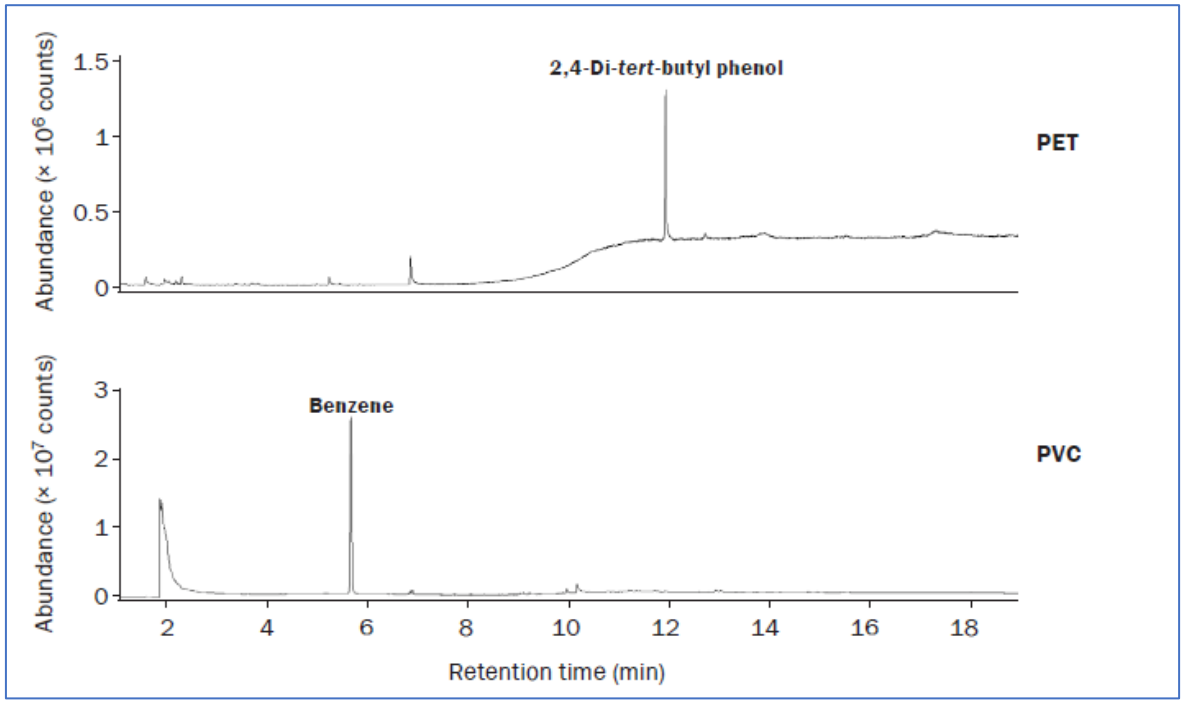


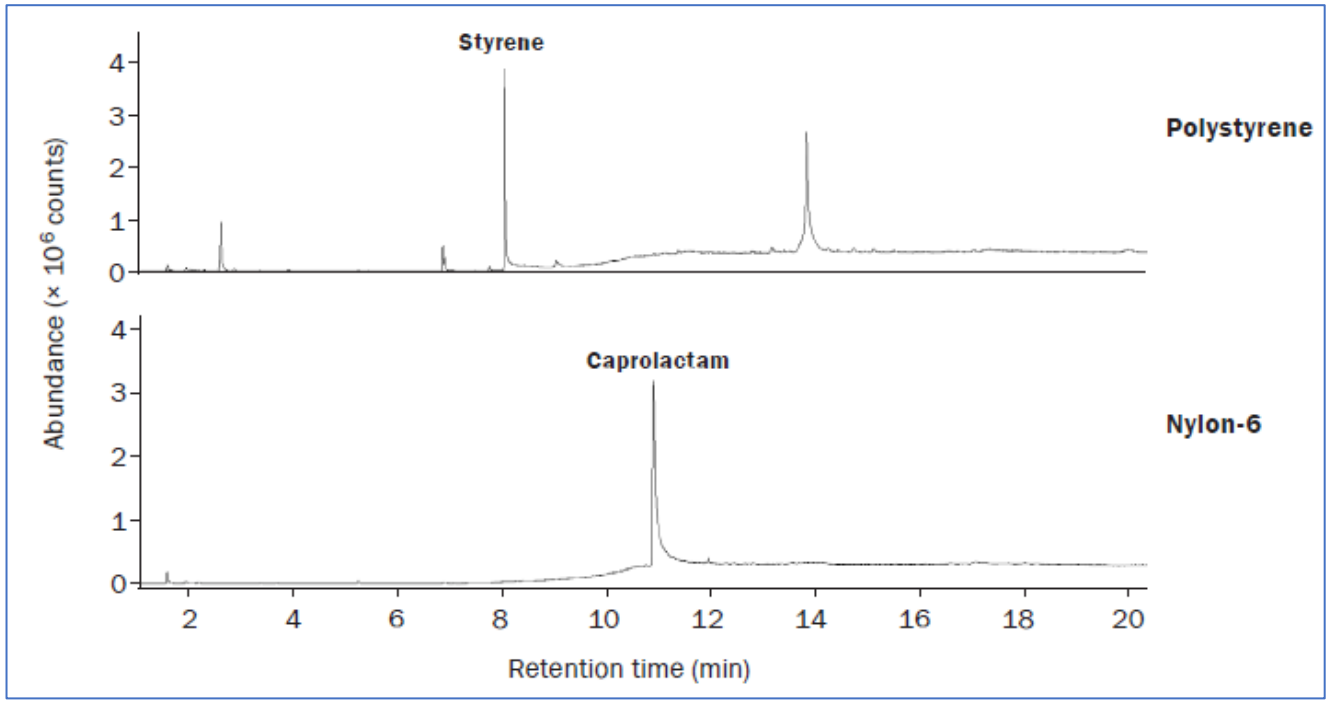
Create calibration curve to quantify PET in samples

- Mass of PET vs DTBP peak area



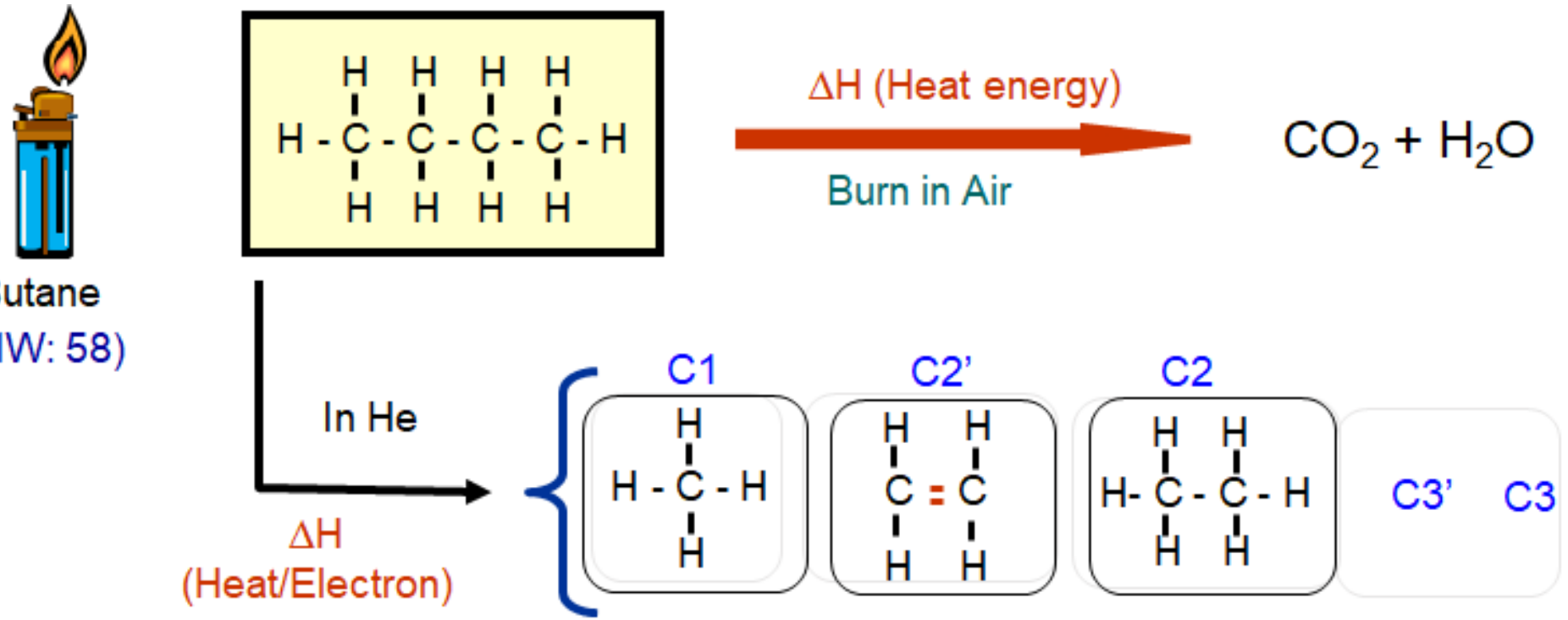
Identification of marker compounds for the four polymer standards displayed





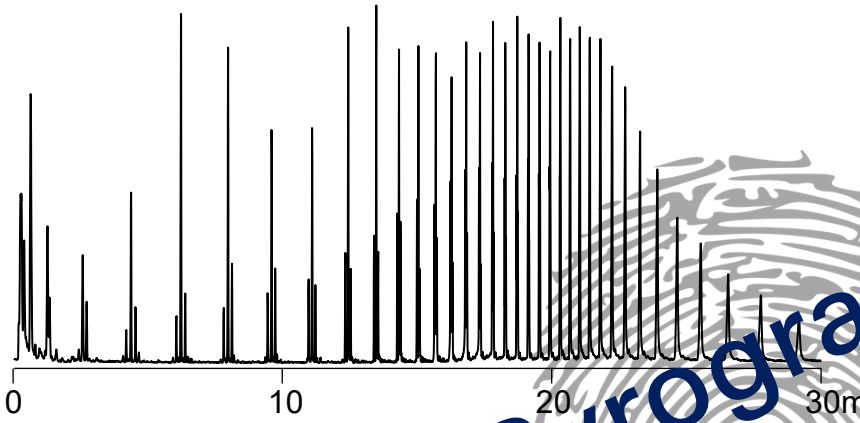
Polymer	Marker compound	Degradation process
Nylon-6	Caprolactam	Depolymerisation of caprolactam
PET	2,4-DTBP	PET glass transition
Polystyrene	Styrene	Depolymerisation of styrene
PVC	Benzene	Dehydrochlorination process of PVC

Pyrolysis and pyrolyzates of polymeric materials

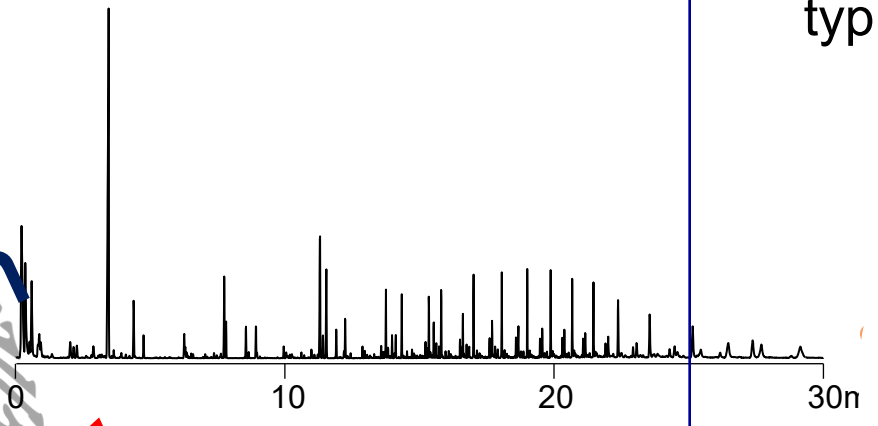


Pyrograms of typical polymers

Polyethylene

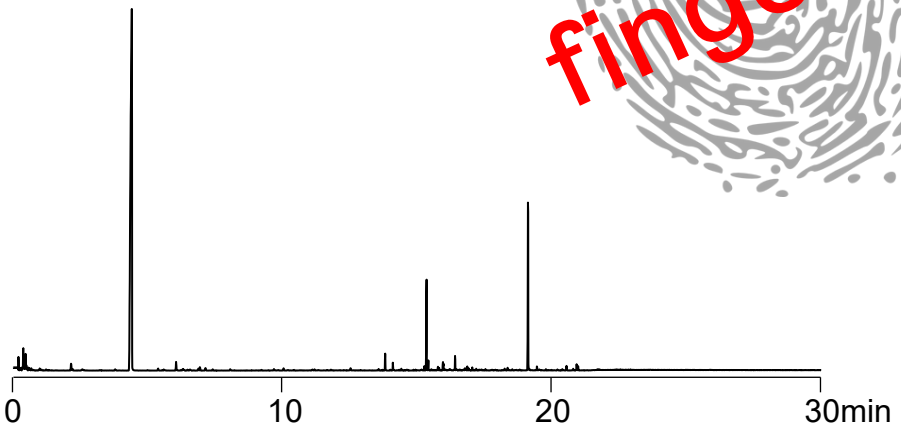


Polypropylene

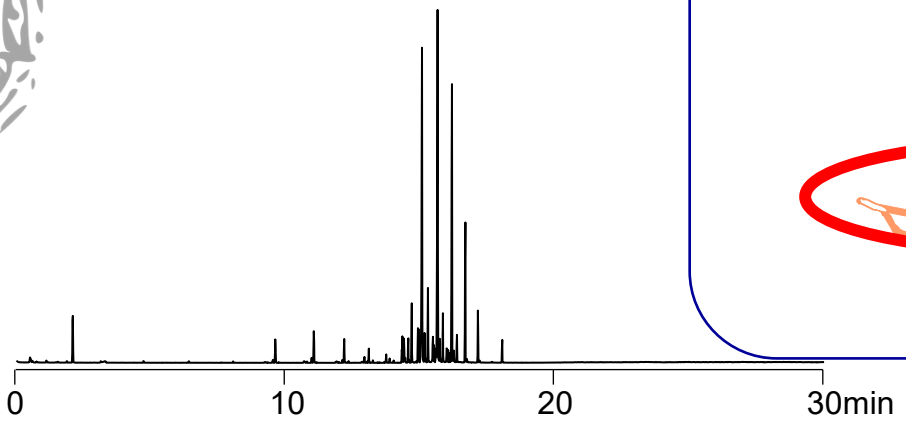


Pyrogram fingerprint

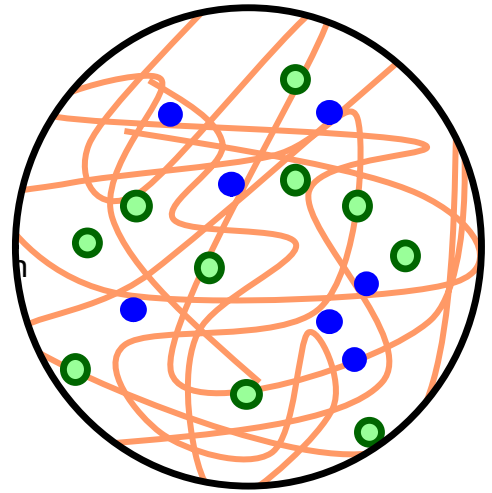
Polystyrene



Higher methacrylate copolymer



Pattern diagram of typical polymeric material



: Additives

: Polymer

: Inorganics

Sample Preparation Protocol for analysis MPs in environment sample

MPs in Sediment

Mass reduction by Sieving

↓

Density separation

(Ns_2WO_4, NaCl, NaI, $ZnCl_2$ aq soln.)

↓

MPs in Water

Sampling with a plankton net (ca. 0.3 mm)

↓

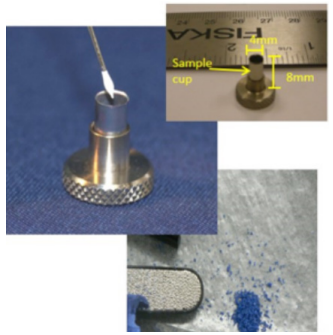
Mass reduction by Sieving

↓

Remove of nature debris from the MPs surface (Chemical; H_2O_2, enzymatic)

↓

Py-GC/MS Analysis



F-search MPs 2.0 : Analytical software for microplastic analysis

*Japan Patent #6683335

Easily identify and quantify unknown microplastic (MPs)

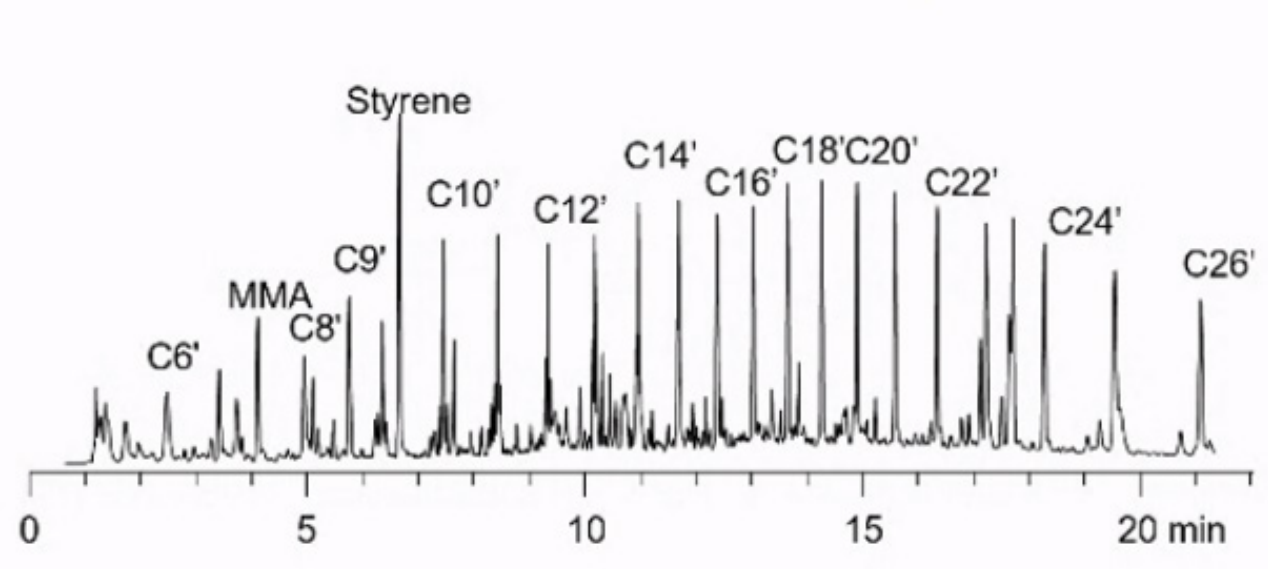
- Quick identification of polymer types for unknown MPs by a patented search algorithm*
- Automatic creation of calibration curve and quick quantification
- Library for 12 commonly used polymers

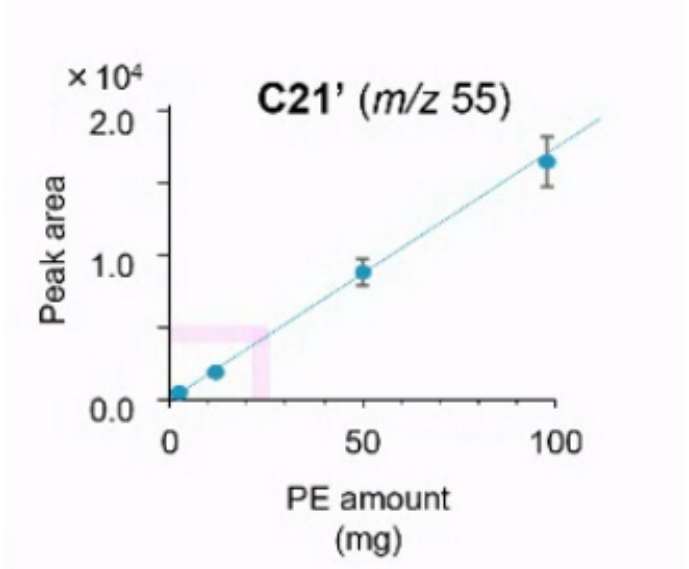
F-search MPs 2.0 Software

Automate qualification and quantification work

1. Search marker peaks for each polymer

2. Quantify each polymer based on calibration curve





Results of Qual/Quant

Library Match Quality (%)

Plastic Name

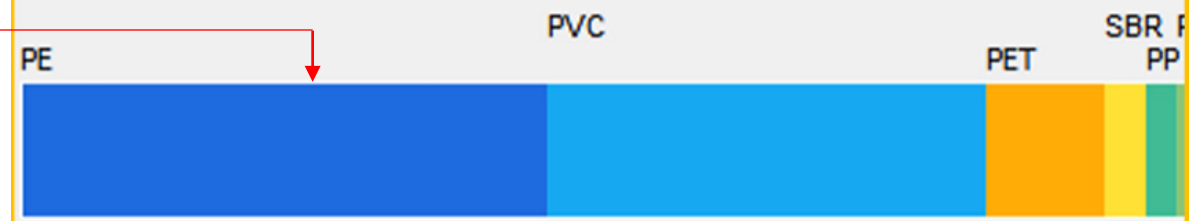
Quant Result (µg)

Relative Amount (%)

Peak Area

Polymer	Prob. [%]	Qnty [ug]	Ratio [%]	Area	RT [m...	LOQ [ug]
PE	99.5	11.20	42.5	31420	16.36	7.60
PVC	92.5	9.355	35.5	146285	10.57	2.70
PET	7.8	2.562	9.73	21353	14.10	1.20
SBR	18.8	0.917	3.48	7107	11.50	1.30
PP	89.9	0.691	2.62	4116	6.46	3.90
PS	98.2	0.601	2.28	75144	21.33	0.51
PMMA	99.2	0.375	1.42	39050	4.82	0.69
PU	96.1	0.276	1.05	81556	18.01	0.69
ABS	57.6	0.150	0.57	2697	18.02	0.76
N66	94.1	0.138	0.52	6349	6.23	0.55
N6	61.6	0.058	0.22	3745	11.50	0.23
PC	69.5	0.018	0.07	5027	11.24	0.67
			(100)			

Bar graph of Relative Amount (%)



Conclusions

- GC/MS technique can analysis MPs in all particle size is only limited by the size of filter chosen
- TD-GC/MS and PY-GC/MS Simple sample preparation workflows that can be applied to wide ranging sample types. And large sample sizes for enhanced repeatability and sensitivity.
- TD-GC/MS technique can identification and quantification of MPs and provides simultaneous information on targets and non-targets compounds. Additional toxicity information and source profiling.
- Py–GC–MS technique provides detailed information about polymers, additives, and even contaminants.
- Py–GC/MS technique And Software F-Search MPs can identification and quantification of MPs is rapid and automated.

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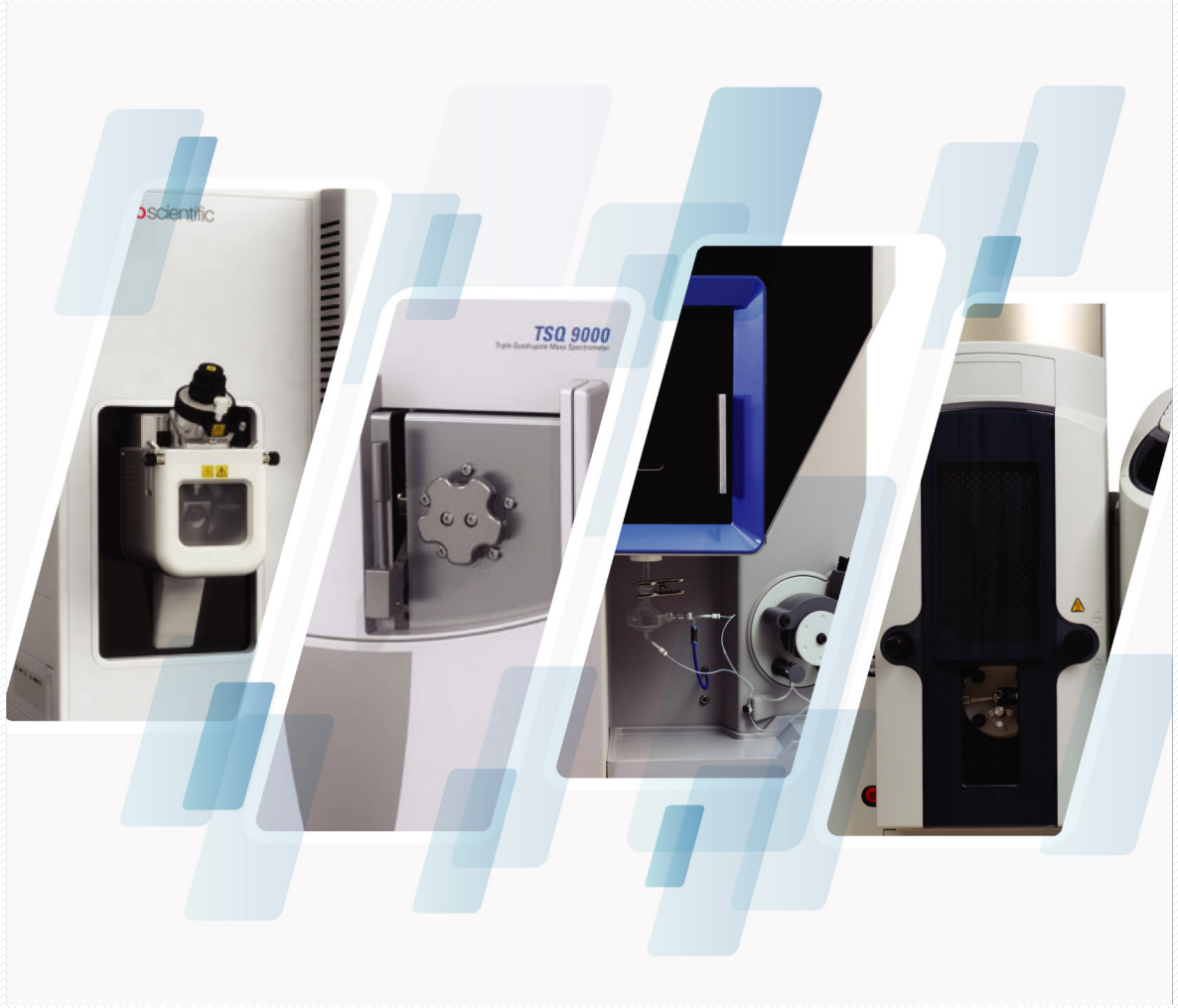

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