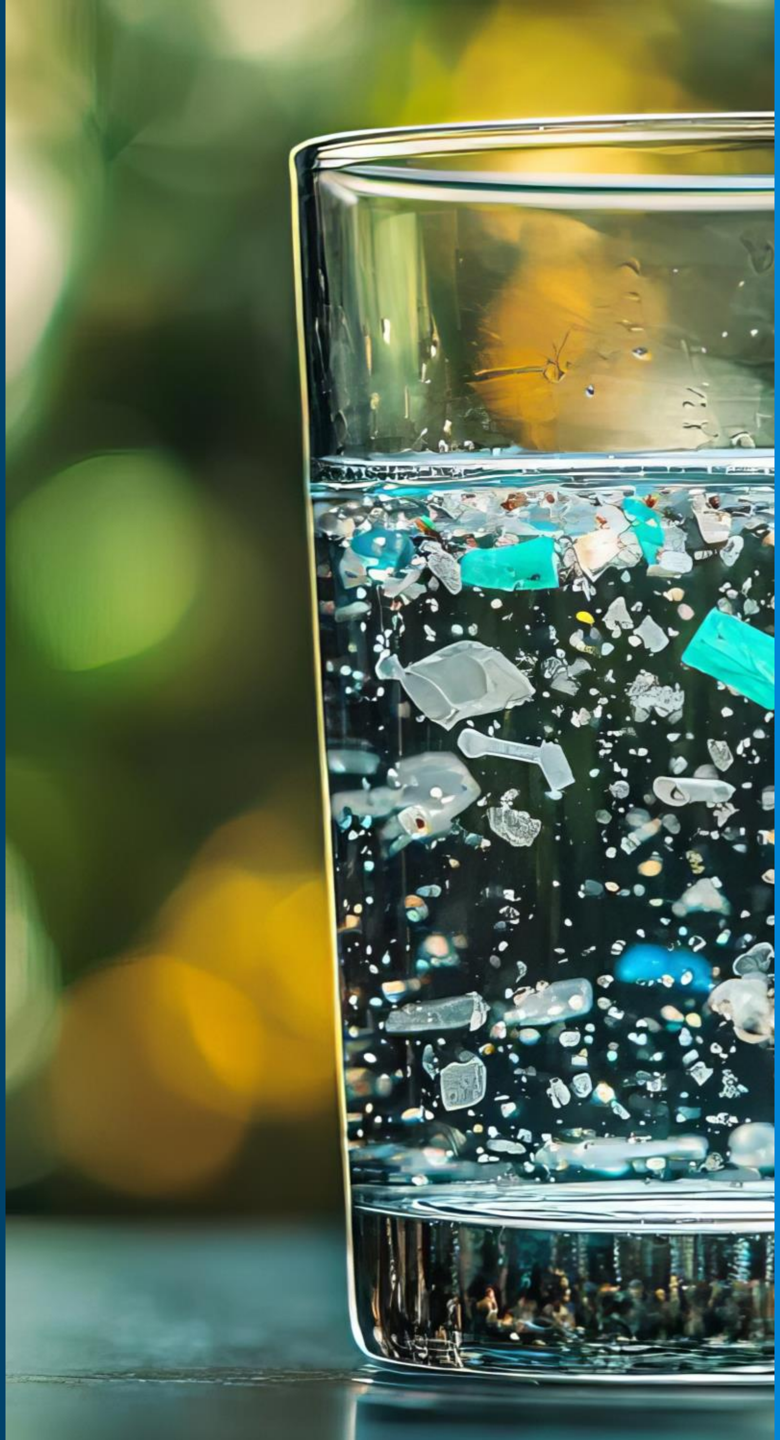
How Critical is Spectral Range to Identification of Microplastics?

Wesam Alwan, Ph.D. and Louis Tisinger, Ph.D.,
Applications Scientists, Molecular Spectroscopy
Agilent Technologies



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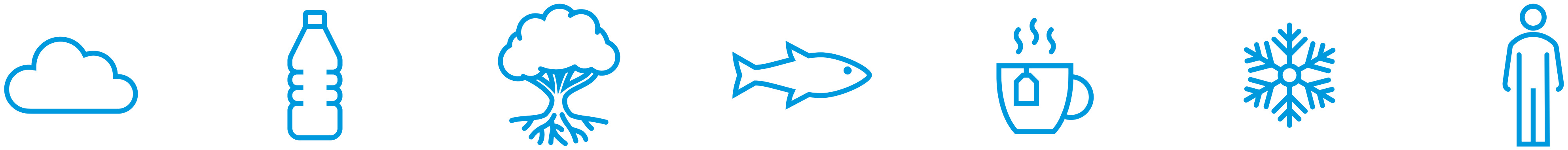
Introduction



Air Water Soil Food Beverages Snow Human

Microplastics are everywhere!

Microplastics are a growing environmental and health concern

Improved detection and characterization

Standardized methods

Development of reference materials

Challenges in Microplastic Analysis

Size Variability

Surface Chemistry Changes

Color Variability

Degradation & Weathering

Shape Variability

Density Differences

Diverse Polymer Types

Additives & Fillers

Characterization is complex!

Global Regulatory Frameworks for Microplastic Analysis Using Vibrational Spectroscopy

- Commission Delegated Decision (EU) 2024/144 Method
- ISO/DIS 16094-2

- California Water Boards Policy Handbook
- ASTM WK87463

Industry and group standards

- GH/T 1378-2022 (MPs residues from farmland mulch film)
- T/CSTM 00884—2024 (wastewater)
- T/CSTM 00885—2024 (soil)
- T/CSTM 00886—2024 (groundwater)
- T/CSTM 00887—2024 (seafood)
- T/CSTM 00911-2024 (drinking water)

Global Regulatory Frameworks for Microplastic Analysis Using Vibrational Spectroscopy

Method	EU 2024/1441	California Water Boards	ISO/DIS 16094-2
Analytical techniques	• Vibrational spectroscopy methods such as µ-FTIR, µ-Raman or equivalent variations such as **Quantum Cascade Laser Infrared Microscopy (QCL-IR)**.	• IR spectroscopy can include, but is not limited to, FTIR, **Laser Direct Infrared (LDIR) Imaging**, Optical-Photothermal IR (O-PTIR), and other techniques capable of measuring microplastic particles as small as 50 µm. • Raman spectroscopy	• IR apparatus coupled to microscope, such as FTIR, **QCL-IR microscopy (Ref. Annex F)** • Raman coupled to microscope

Regulatory frameworks recommend various technologies, each differing in:

Wavenumber range

Optical design

Sample introduction substrates

Signal-to-noise ratio

Measurement mode

Library selection

Data processing methods

The Question We Asked

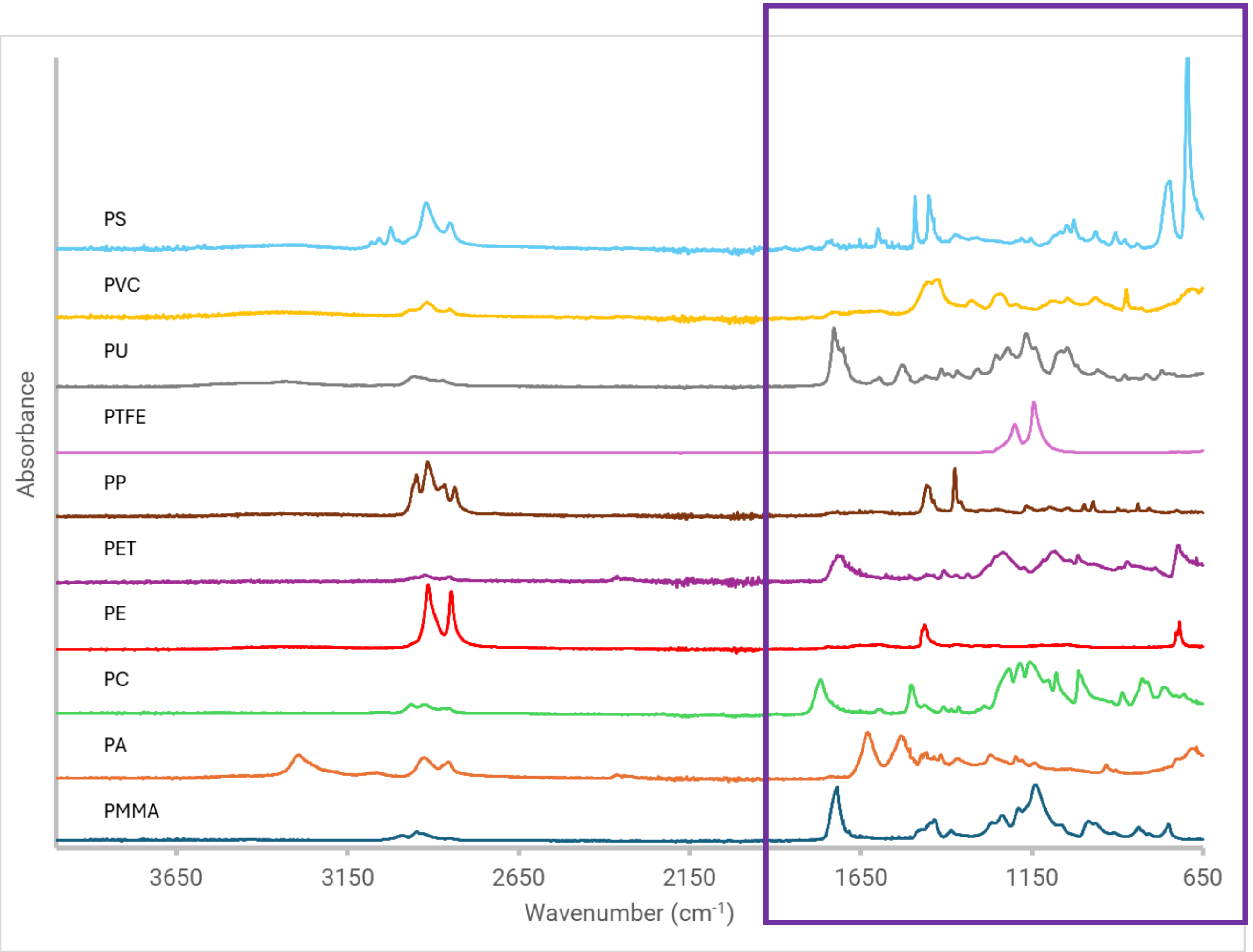
Wavenumber range | Optical design | Sample introduction substrates | Signal-to-noise ratio

Measurement mode | Library selection | Data processing methods

Since only a few aspects of testing variations can be controlled, such as sample introduction substrates, library selection, and data processing, an important question arises:

Can narrowing the **IR spectral range** impact the accuracy of microplastics identification?

Identifying Microplastics Using IR Spectroscopy



2,980 to 2,780 cm^{-1} (stretching vibrations of $CH/CH_2/CH_3$ groups)

1,260 to 1,087 cm−1 (CF_2 stretching vibrations)

1,480 to 1,400 cm^{-1} (CH_2 bending vibrations)

1,760 to 1,670 cm−1 (C=O stretching vibrations)

1,800 to 1,740 cm−1 (C=O stretching vibrations)

Experimental Approach - Comparative Study of FTIR & LDIR Systems

Polymer samples

- PE
- PP
- PET
- PS
- PVC
- PA
- PU
- PMMA
- PTFE
- PC

Identification

- FTIR-ATR
- μFTIR imaging equipped with an FPA detector.
- QCL-IR

Data processing

- Exporting spectra
- Converting reflection data to absorbance
- Calculating the first derivative

Reference Library

- siMPle library, containing 326 ATR spectra of commonly encountered environment al polymers

Matching algorithm

- Pearson's method (4 ranges)

Cary 630 FTIR-ATR

Cary 620 μFTIR imaging system

8700 LDIR Chemical Imaging System

Cary 630 FTIR-ATR

Polymer	650–3945 cm^{-1}	950–3590 cm^{-1}	1250–3590 cm^{-1}	975–1800 cm^{-1}
PS	0.95 Polystyrene	0.78 Polystyrene	0.78 Polystyrene	0.84 Polystyrene
PE	0.81 Polyethylene_low_density	0.81 Polyethylene_low_density	0.81 Polyethylene_low_density	0.82 Polyethylene_low_density
PET	0.61 Poly(ethylene_terephthalate)	0.54 Poly(ethylene_terephthalate)	0.47 Fibre_polyester	0.62 Poly(ethylene_terephthalate)
PC	0.89 Polycarbonate	0.90 Polycarbonate	0.87 Polycarbonate	0.91 Polycarbonate
PVC	0.28 Poly(vinyl_chloride)_carboxylated	0.30 Polyvinylchloride_with_plasticizer	0.26 Polyvinylchloride_with_plasticizer	0.60 Polyvinylchloride_with_plasticizer
PP	0.79 Fibre_polypropylene_dyed	0.80 Fibre_polypropylene_dyed	0.80 Fibre_polypropylene_dyed	0.94 Fibre_polypropylene_dyed
PTFE	0.96 Poly(tetrafluoroethylene)	0.97 Poly(tetrafluoroethylene)	0.36 Poly(tetrafluoroethylene)	0.98 Poly(tetrafluoroethylene)
PA	0.51 Nylon_6_6	0.51 Nylon_6_6	0.50 Nylon_6_6	0.72 Nylon_6_6
PU	0.77 Polyurethane	0.78 Polyurethane	0.76 Polyurethane	0.82 Polyurethane
PMMA	0.85 Polymethyl methacrylate	0.86 Polymethyl methacrylate	0.83 Polymethyl methacrylate	0.88 Polymethyl methacrylate

Correlation	<0.4	0.4 – 0.6	0.6 – 0.8	>0.8

All polymers were identified correctly

- **PTFE** performs best at 975–1,800 cm^{-1}.
- Fingerprint region improves correlation for **PA**, **PP**, and **PVC**.
- **PET** and **PS** show highest correlation in both full and narrow ranges.
- Minimal correlation variation for **PMMA**, **PC**, **PE**, and **PU** across all spectral ranges.

Cary 620 µFTIR imaging system

Polymer	650–3945 cm^{-1}	950–3590 cm^{-1}	1250–3590 cm^{-1}	975–1800 cm^{-1}
PS	0.41 Polystyrene	0.80 Polystyrene_expanded	0.79 Polystyrene_expanded	0.84 Styrene_acrylonitrile
PE	0.41 Polyethylene_low_density	0.61 Polyethylene_low_density	0.63 Polyethylene_low_density	0.69 Polyethylene_foamed
PET	0.54 Polyethylene terephthalate	0.63 Polyethylene terephthalate	0.57 Polyethylene terephthalate	0.63 Polyethylene terephthalate
PC	0.37 Polycarbonate	0.55 Polycarbonate	0.60 Polycarbonate	0.58 Polycarbonate
PVC	0.17 Vinyl_chloride_vinyl_acetate_hydroxypropyl_acrylate	0.38 Vinyl_chloride_vinyl_acetate_hydroxypropyl_acrylate	0.38 Polyvinylchloride	0.47 Vinyl_chloride_vinyl_acetate_hydroxypropyl_acrylate
PP	0.62 Polypropylene	0.71 Polypropylene	0.70 Fibre_polypropylene	0.88 Polypropylene
PTFE	0.14 Polytetrafluoroethylene	0.44 Polytetrafluoroethylene	0.37 Poly(tetrafluoroethylene)	0.67 Polytetrafluoroethylene
PA	0.43 Nylon_6_6	0.71 Nylon_6_6	0.71 Nylon_6_6	0.74 Nylon_6_6
PU	0.46 Alkyd_varnish	0.61 Alkyd_varnish	0.70 Alkyd_varnish	0.61 Alkyd_varnish
PMMA	0.31 Polymethyl methacrylate	0.38 Polymethyl methacrylate	0.36 Polymethyl methacrylate	0.39 Polymethyl methacrylate

Correlation	<0.4	0.4 – 0.6	0.6 – 0.8	>0.8

All polymers were identified correctly

- Full range shows lowest correlation due to low S/N at spectrum edges.
- 975–1,800 cm^{-1} improves correlation for **PP** and **PTFE**.
- **PA**, **PC**, **PE**, **PET**, **PMMA**, **PS**, **PU**, and **PVC** show minimal variation across ranges.

8700 LDIR Chemical Imaging System

Polymer	975–1800 cm^{-1}
PS	0.94 Polystyrene
PE	0.92 Polyethylene_low_density
PET	0.82 Polyethylene_terephthalate
PC	0.92 Polycarbonate
PVC	0.74 Polyvinylchloride
PP	0.96 Polypropylene
PTFE	0.63 Polytetrafluoroethylene
PA	0.90 Nylon_6_6
PU	0.75 Polyurethane
PMMA	0.73 Polymethyl methacrylate

Correlation	<0.4	0.4 – 0.6	0.6 – 0.8	>0.8

Polymer	975–1800 cm^{-1}
PS	0.978 Polystyrene
PE	0.987 Polyethylene
PET	0.954 Polyethylene terephthalate
PC	0.953 Polycarbonate
PVC	0.878 Polyvinylchloride
PP	0.984 Polypropylene
PTFE	0.976 Polytetrafluoroethylene
PA	0.964 Polyamide
PU	0.943 Polyurethane
PMMA	0.952 Polymethyl methacrylate

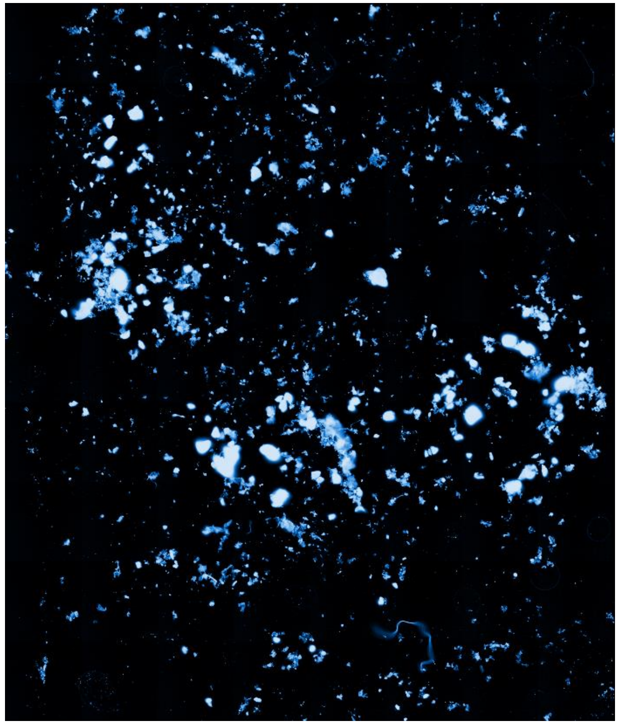
✓ Using Pearson's correlation, LDIR data accurately identified all polymers.

✓✓ Microplastics Starter 2.1 library and Clarity software accurately identified all polymers.

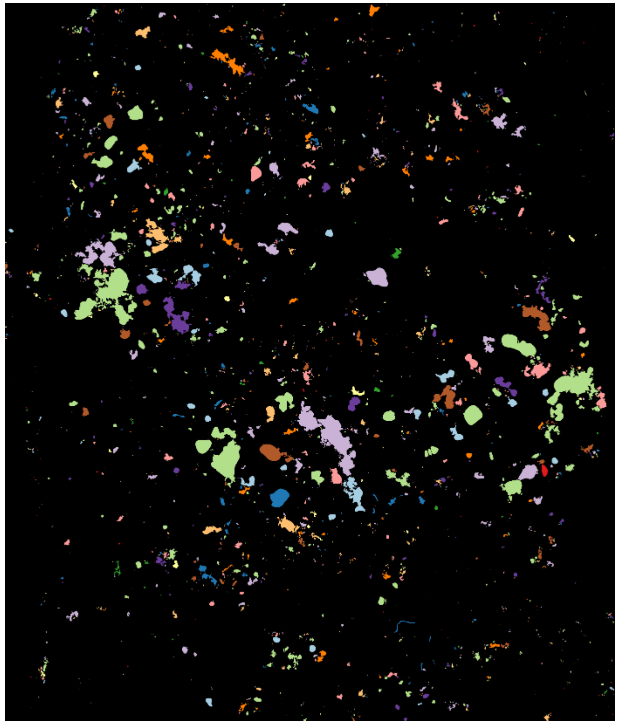
8700 LDIR Chemical Imaging System Automated Workflow

Identification and classification data for a microplastics mixture (39 × 23 mm area).

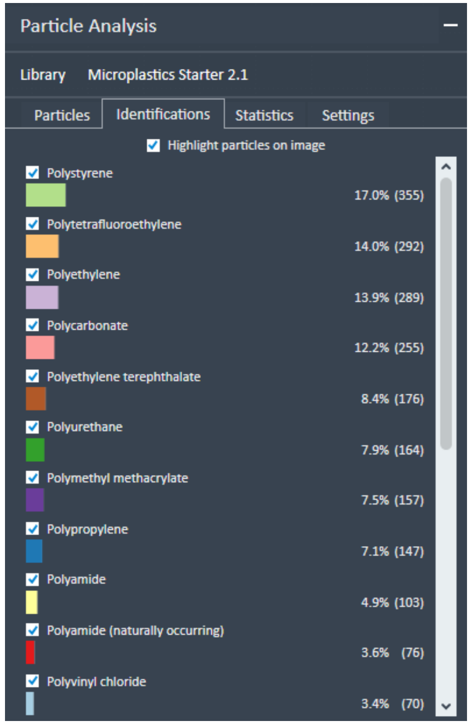
Visible image of the analyzed area



IR image scanned at 1,442 cm^{-1}



Particles colored based on the ID of microplastics

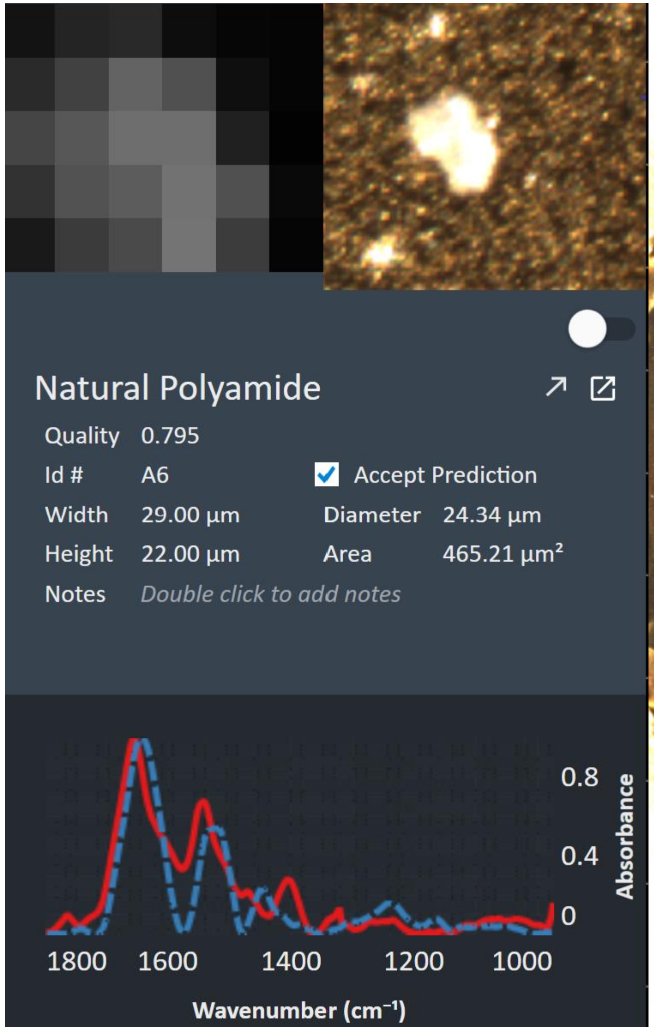


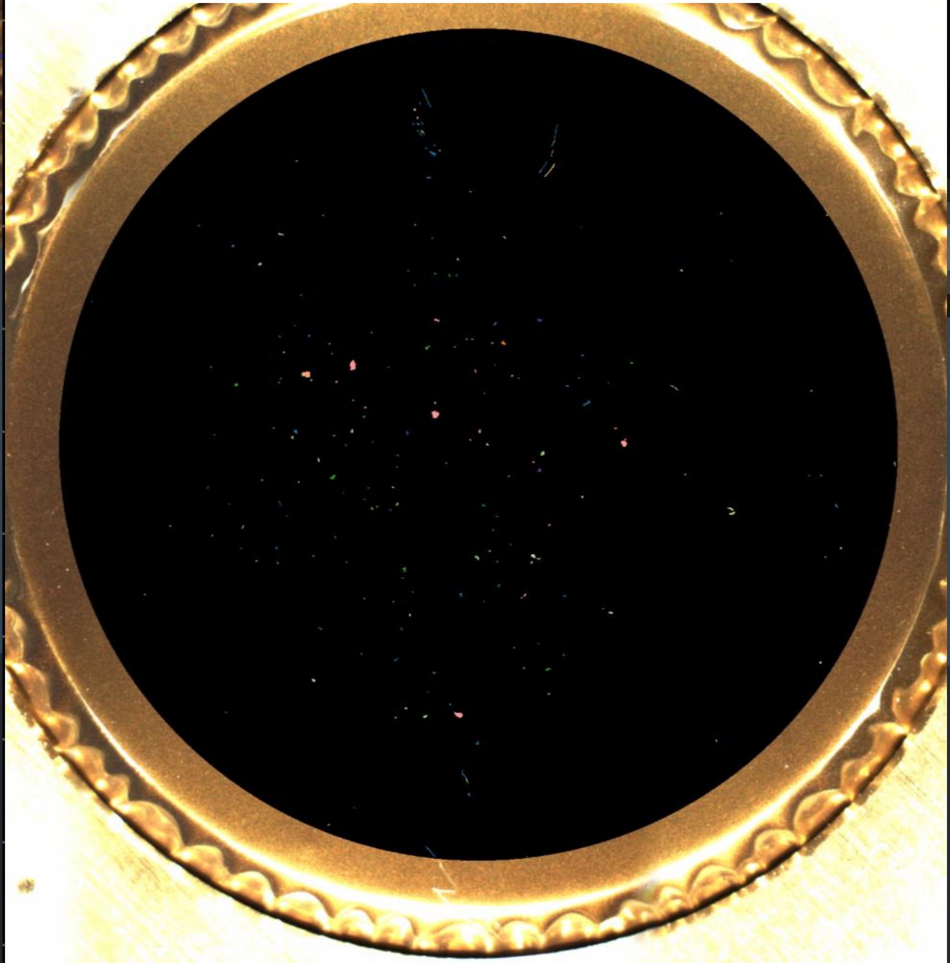
Automatic statistical data generated based on the ID

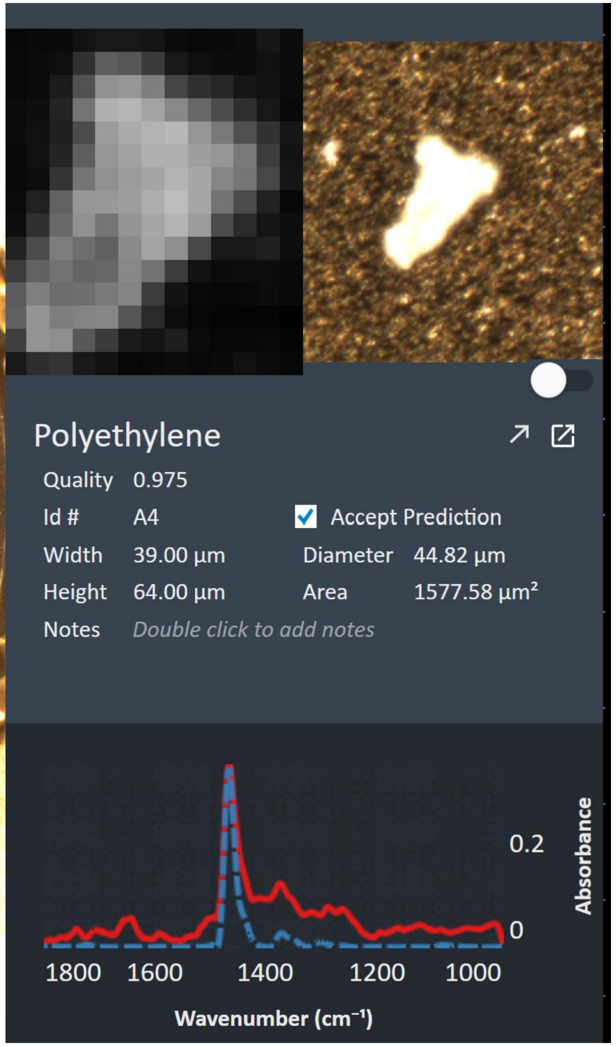
The workflow and library are optimized for microplastics analysis, ensuring robust and accurate identification.

8700 LDIR Chemical Imaging System

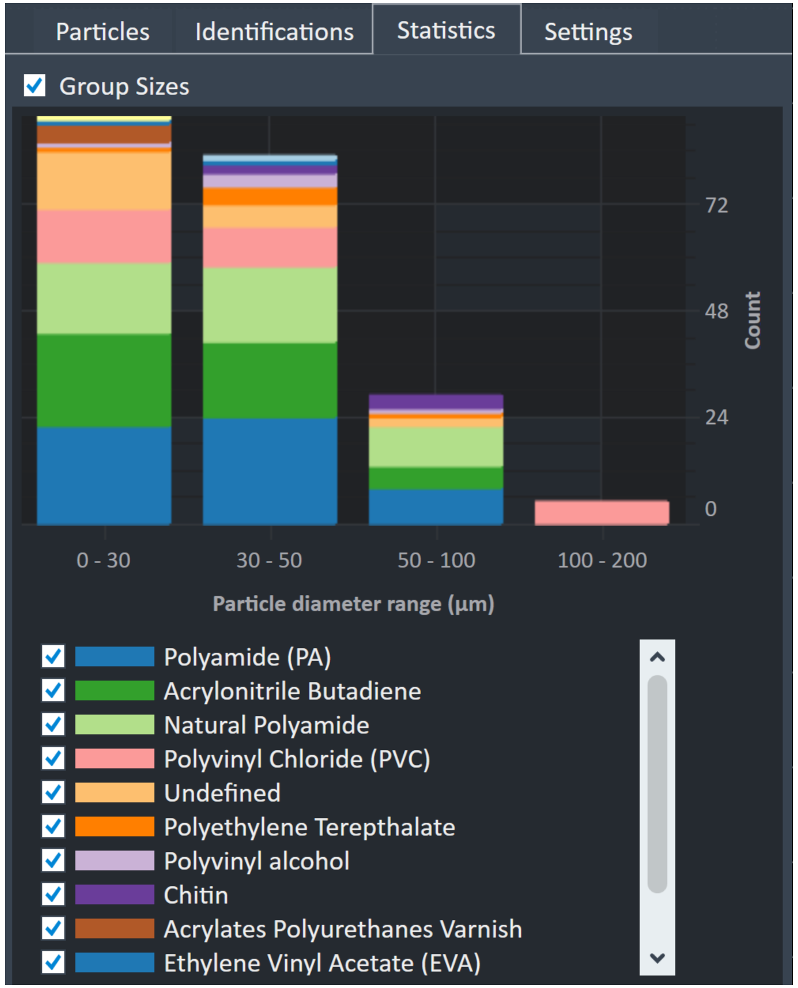
- The LDIR fingerprint region is adequate for identifying most polymers.
- Dedicated for microplastic characterization
- Fully automated microplastics workflow
- Significant time-saving
- Higher sample throughput
- Tailored microplastics library and easy spectral libraries generation

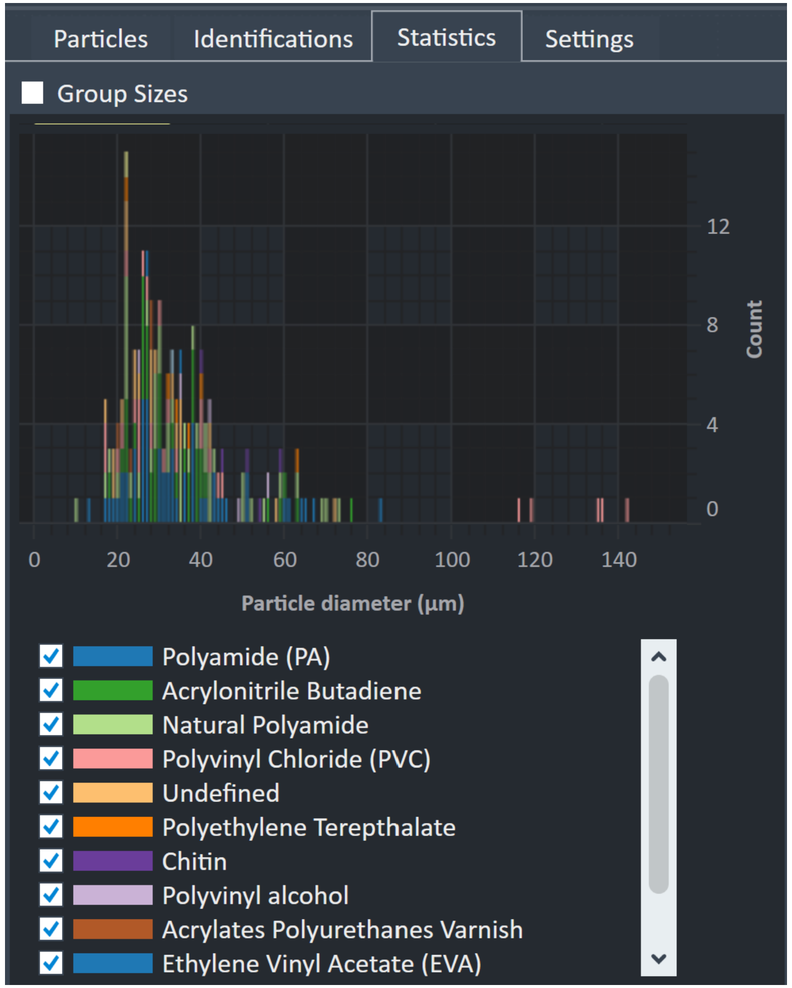

Natural Polyamide
Quality 0.795
Id # A6
Accept Prediction
Width 29.00 µm
Diameter 24.34 µm
Height 22.00 µm
Area 465.21 µm²
Notes Double click to add notes
0.8
0.4
0
Absorbance
1800 1600 1400 1200 1000
Wavenumber (cm⁻¹)




Polyethylene
Quality 0.975
Id # A4
Accept Prediction
Width 39.00 µm
Diameter 44.82 µm
Height 64.00 µm
Area 1577.58 µm²
Notes Double click to add notes
0.2
0
Absorbance
1800 1600 1400 1200 1000
Wavenumber (cm⁻¹)

Statistics are updated as analysis proceeds





Reported Results: Data Table

#	Id	Width (µm)	Height (µm	Diameter (µm)	Aspect Ratio	Area (µm²)	Perimeter (	Eccentricity	Circularity	Solidity	Identification	Notes	Match Typ	Quality	Is Valid
1	A1	226	230	219.5632762	0.98461529	37862.5	807.193	0.7613484	0.730238	0.921789	Polystyrene		Auto	0.776292	true
2	A2	161	164	163.9065416	0.98203604	21100	576.2742	0.7115964	0.798425	0.948848	Polyethylene		Auto	0.920886	true
3	A3	125	123	125.6509863	1.01818184	12400	440.4163	0.7474169	0.80335	0.967805	Polyethylene		Auto	0.92681	true
4	A4	97	170	121.9224003	0.57009344	11675	446.2742	0.8639478	0.736654	0.934935	Polyethylene		Auto	0.972862	true
5	A5	88	74	80.18640596	1.19047621	5050	271.4214	0.6662479	0.861417	0.975845	Polyvinyl Chloride (PVC)		Auto	0.823328	true
6	A6	75	90	77.97255175	0.83333333	4775	301.4214	0.7362301	0.660443	0.866213	Polyvinyl Chloride (PVC)		Auto	0.893123	true
7	A7	116	82	77.35777828	1.410596	4700	537.6955	0.5146039	0.204284	0.628763	Natural Polyamide		Auto	0.76303	true
8	A8	68	58	59.03806614	1.15873019	2737.5	216.066	0.6668491	0.73687	0.9125	Polyethylene		Auto	0.986766	true
9	A9	35	40	39.29125791	0.875	1212.5	129.4975	0.681444	0.908592	1	Polyethylene		Auto	0.99268	true
10	A10	30	55	37.42410319	0.54545455	1100	140.7107	0.7350755	0.69815	0.916667	Polyethylene		Auto	0.991073	true
11	A11	25	57	36.56366396	0.44578312	1050	146.5685	0.7735358	0.614211	0.893617	Polytetrafluoroethylene (PTFE)		Auto	0.71854	true
12	A12	30	25	27.3501048	1.2	587.5	95.35534	0.6274145	0.811947	0.94	Chitin		Auto	0.658915	true
13	A13	31	22	25.54476985	1.40000009	512.5	89.49747	0.6776102	0.804048	0.953488	Natural Polyamide		Auto	0.929911	true
14	A14	20	30	24.26671155	0.66666667	462.5	85.35534	0.7387094	0.797738	0.948718	Polyvinyl alcohol		Auto	0.827235	true
15	A15	25	20	22.56758334	1.25	400	78.28427	0.6210353	0.820202	0.941176	Acrylates Polyurethanes Varnish		Auto	0.878318	true
16	A16	25	34	21.85096861	0.73529417	375	120.7107	0.7342011	0.323407	0.625	Silica		Auto	0.669237	true

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Microplastics Analysis and the Infrared Spectrum: Is Spectral Range Selection Critical?

Authors
Wesam Alwan, Ciaran Worth, and Philip Wilson
Agilent Technologies, Inc.

Introduction

Microplastic contamination is an emerging threat to environmental and ecological systems across the globe. Microplastics are defined as small (less than 5 mm) solid fragments of synthetic or modified natural polymers that are insoluble in water.[1] Studies have shown that plastic particles cause harm to multiple types of organisms through exposure to toxic substances.[2,3] Despite these known toxic effects, the full extent of the impact of microplastics remains unclear due to challenges in developing reliable methods for characterizing this broad class of micro-sized polymers.

Infrared (IR) spectroscopy is an established technique for characterizing microplastics. It identifies the molecular composition and structure of materials by measuring the absorption of IR radiation. The resulting absorption bands, which are due to intramolecular vibrational modes, help identify organic or mineral materials through comparison of spectra with a library of spectra from known polymers.[1] Current directives and standard methodologies require that major types of polymers are identified correctly and can be distinguished from other natural materials.[1,4]

Microplastics Analysis and the Infrared Spectrum: Is Spectral Range Selection Critical?
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Navigating Global Microplastics Regulations in Drinking Water with Vibrational Spectroscopy

Ensuring accurate and reliable microplastics characterization with the 8700 LDIR

Author
Wesam Alwan
Agilent Technologies, Inc.

Introduction

Access to clean and safe drinking water is a fundamental human right and a critical public health priority. With growing concerns about environmental pollution, emerging contaminants such as microplastics have become a major focus for regulatory bodies worldwide. Microplastic particles, originating from sources such as industrial waste, packaging, and everyday consumer products, have been detected in diverse water sources, raising concerns about both their potential health risks and environmental impact.

Navigating Global Microplastics Regulations in Drinking Water with Vibrational Spectroscopy
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Resources: www.Agilent.com

- Best Practice for On-Filter Analysis of Microplastics Using the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System
- Rapid, Large-Area, Analysis of Microplastics from Plastic Bottles Using Laser Direct Infrared Imaging
- Performance Attributes of the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System
- Distinguishing Between Polyamide Microplastics and Natural Polyamide
- Fast, Automated Microplastics Analysis Using Laser Direct Chemical Imaging
- Agilent 8700 LDIR Chemical Imaging System
- Agilent Clarity Software
- Microplastics Technologies FAQs
- Microplastics Analysis in Water
- Agilent Cary 630 FTIR Spectrometer
- Agilent MicroLab Software
- ATR-FTIR Spectroscopy Overview

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