Automated Workflow for the Analysis of Microplastics

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Trusted Answers

How large can a Microplastic be?

Infrared Spectroscopy for identification of polymers is not new

Analysis of Natural and Synthetic Rubber by Infrared Spectroscopy

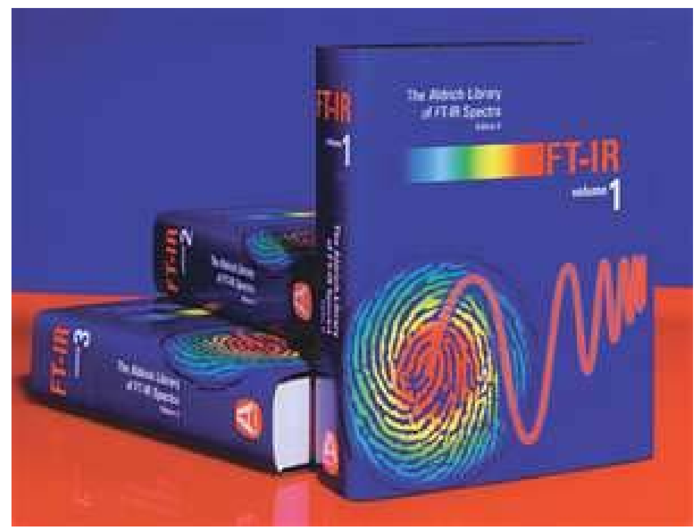
H. L. DINSMORE[1] AND DON C. SMITH, *Naval Research Laboratory, Washington, D. C.*

Analytical Chemistry - 1948

"Fourier Transform Infrared Spectroscopy (FT—IR) is developing as a ubiquitous tool for use in the characterization of polymers".

Jack L. Koenig -1985

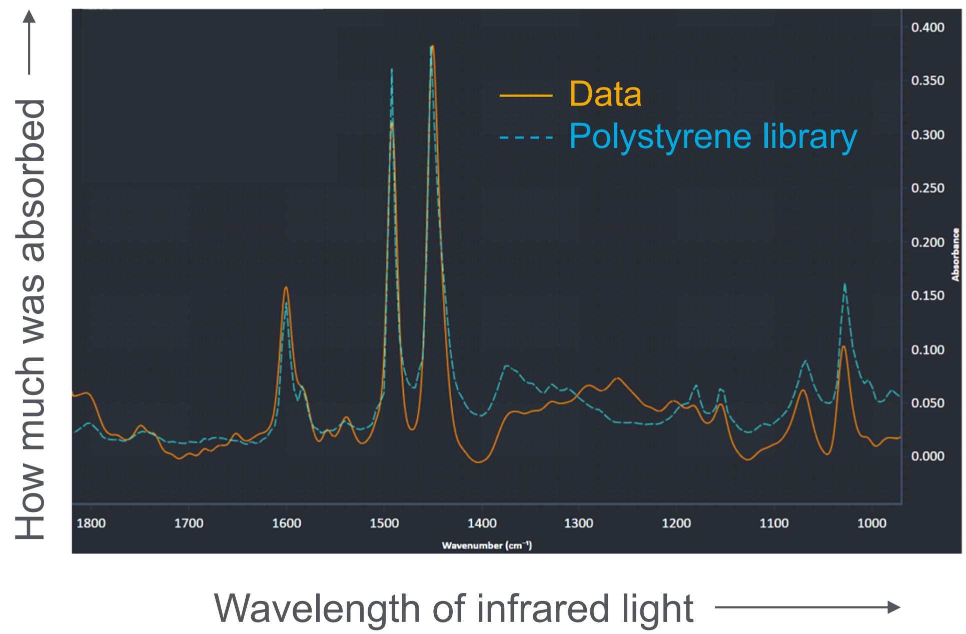
Department of Macromolecular Science, Case Western Reserve University, Cleveland, Ohio 44106



Infrared spectroscopy

Shine infrared light on a particle and see which wavelengths it absorbs

- Every substance has a unique fingerprint and extensive libraries exist
- Robust and reliable
- Non-destructive



Fourier Transform Infrared (FTIR) spectroscopy is the traditional choice

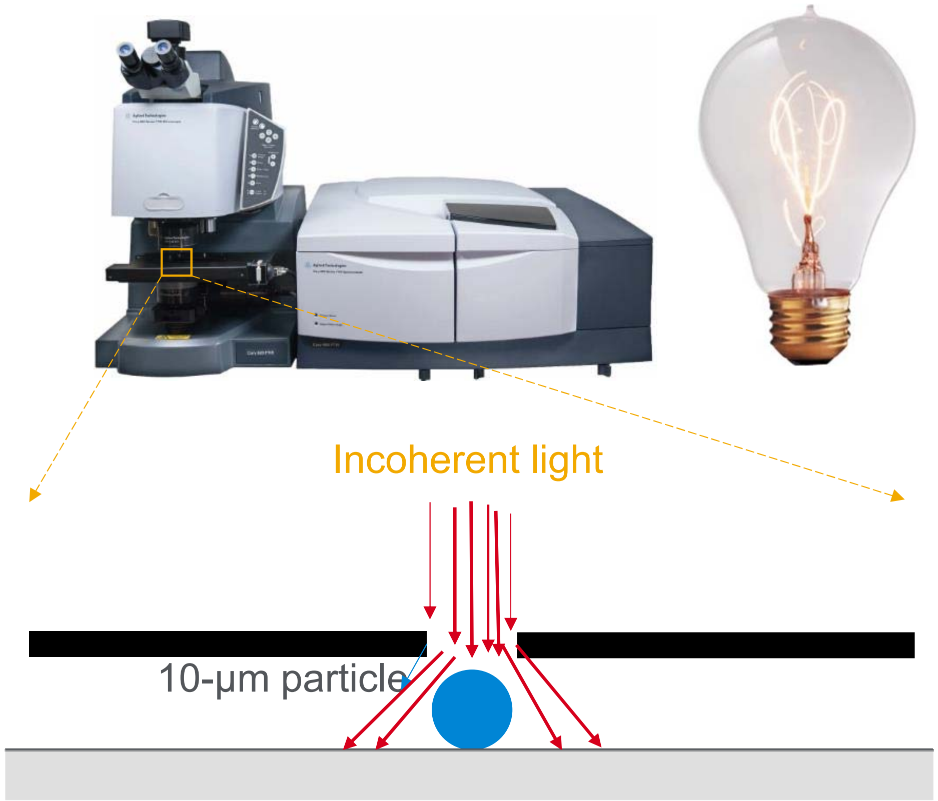
FTIR Micro-Spectroscopy

To measure small things, combine FTIR spectrometer + microscope

Problem: a large incoherent source cannot be focused onto a small microparticle

- Weak signals, slow analysis
- 30 seconds per spectrum typical

Solution: use a laser!



Only a tiny fraction of the light can be absorbed.

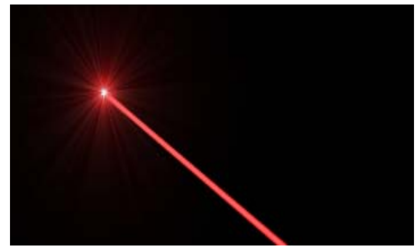
Laser Direct Infrared (LDIR) Spectroscopy

New instrument architecture

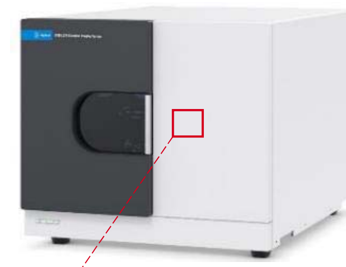
- Bright infrared laser source
- Proprietary Agilent quantum cascade laser (QCL) technology
- Rapidly tunable across the mid-infrared for spectroscopy

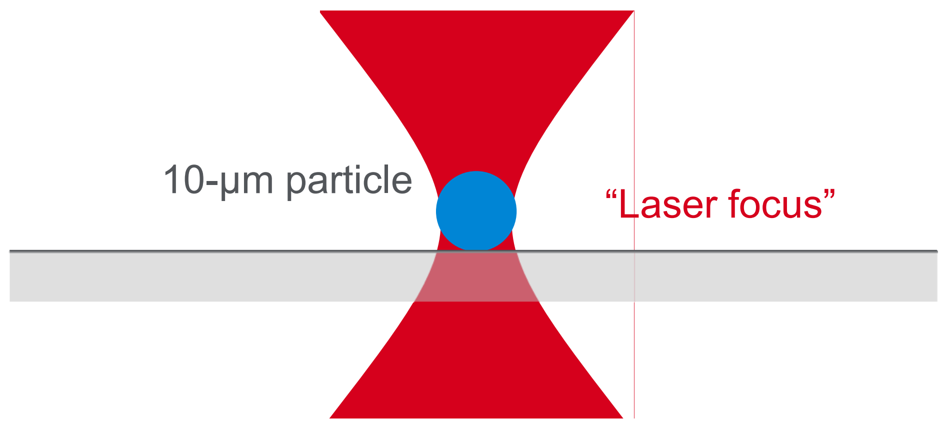
Focus all laser power onto a particle

- One second per spectrum



Coherent laser light





All light reaches the particle

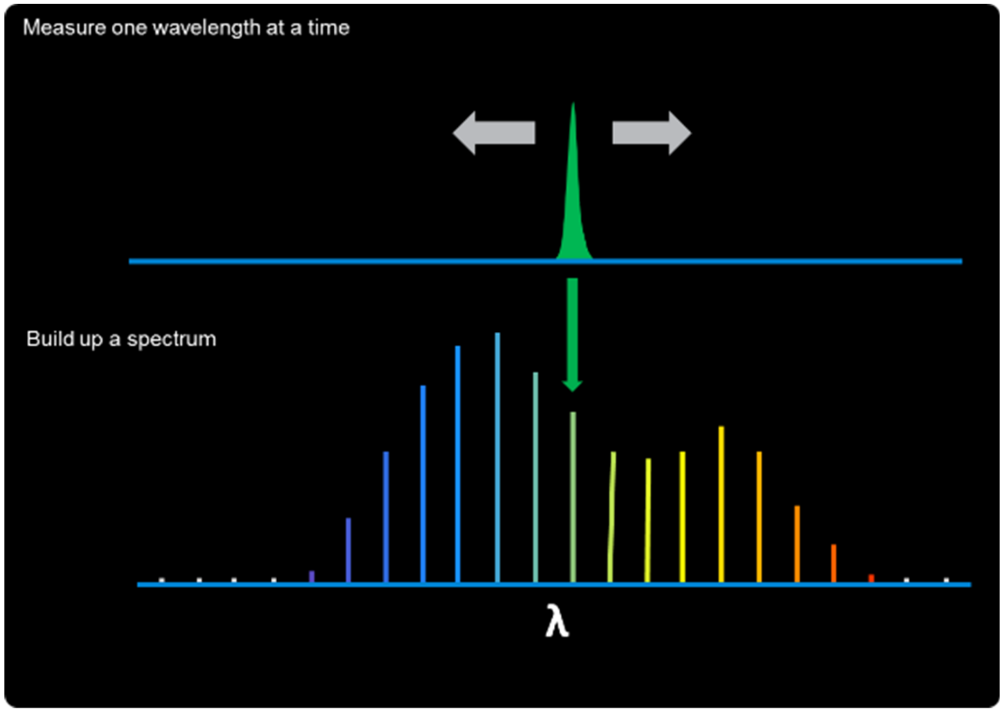
QCL imaging systems

- More light (~1000x)
- Highly directional (it's a laser)
- Diffraction Limited Spot size

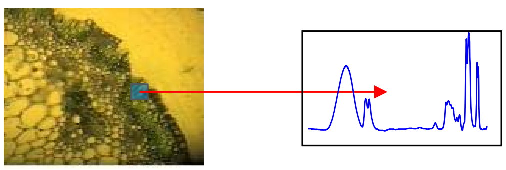
- Operates like a monochromator instrument (measures one wavelength at a time)

- Spectral range covers most important frequencies (fingerprint)

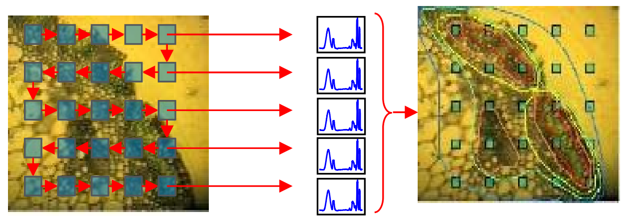
Sweep the whole spectrum one wavelength at a time just like a traditional monochromator



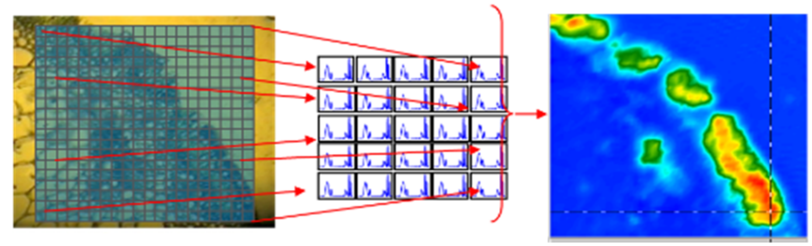
Conventional FTIR microscope modes VS LDIR



Single Point

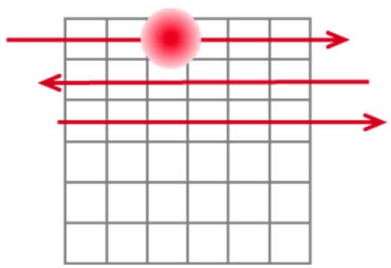


Single Point – Automated Stage



FPA Imaging

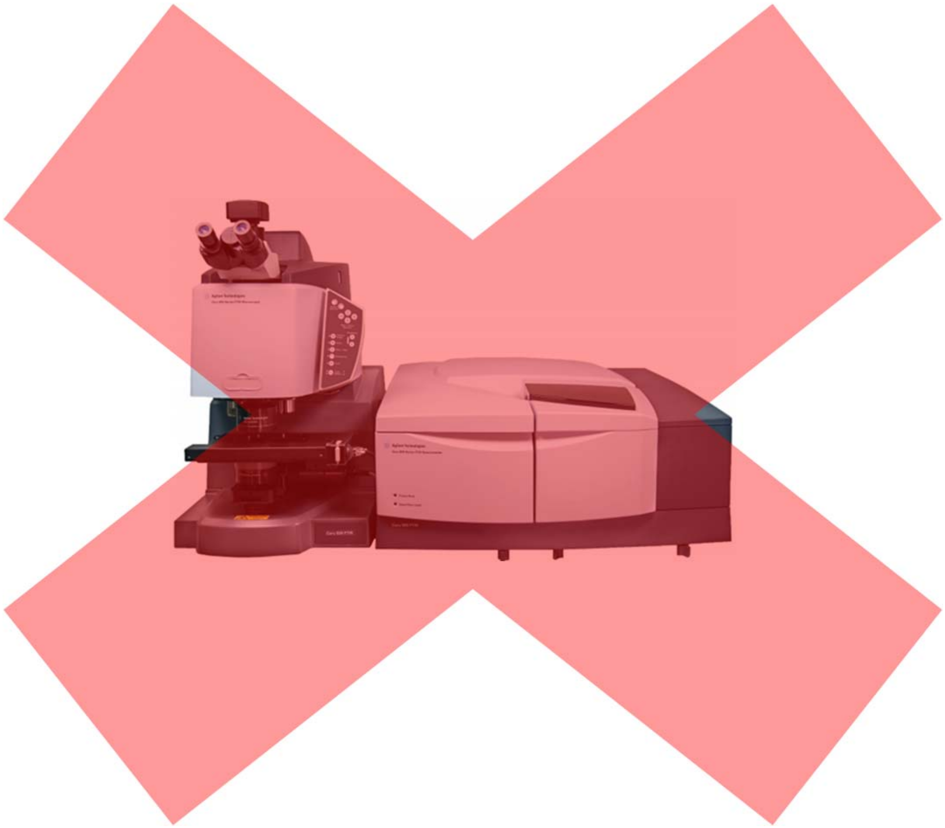
LDIR



Scan laser beam across sample

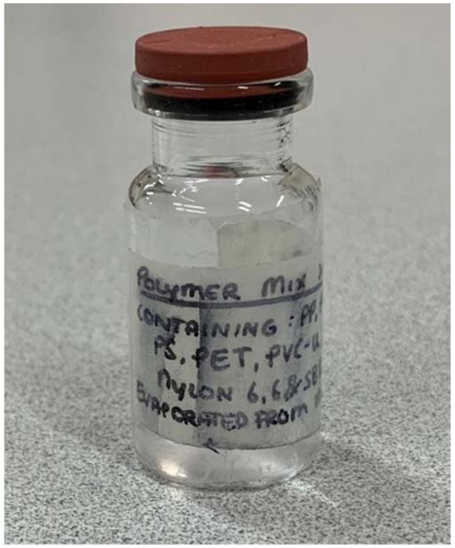
- Measure one pixel at a time (very quickly)
- Take a spectrum at a single pixel (in a very reasonable amount of time)
- Focus all light to a single point and rapidly sweep across the sample

Eliminate complex manual microscope manipulations

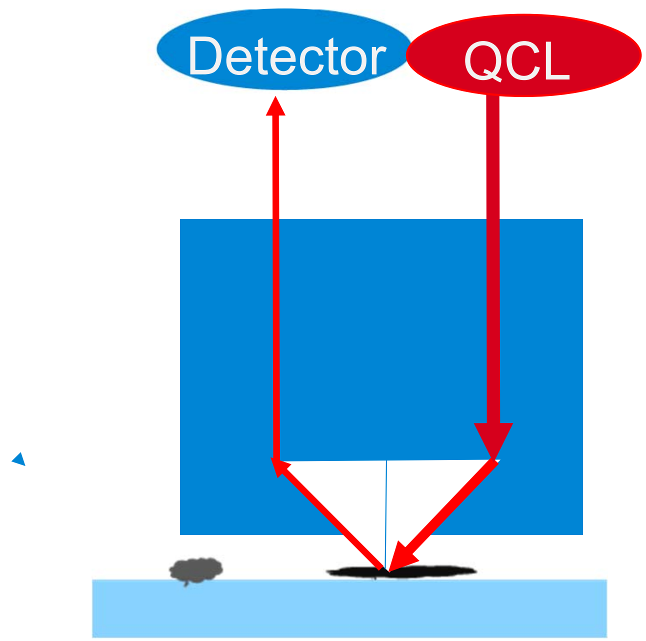


LDIR Analysis of Microplastics

Wash off filtered sample → Sonicate → Pipette on Kevley Slide



Obtain spectra by passing infrared beam thru sample reflecting off Kevley slide (transflectance) and then back thru sample to the detector.



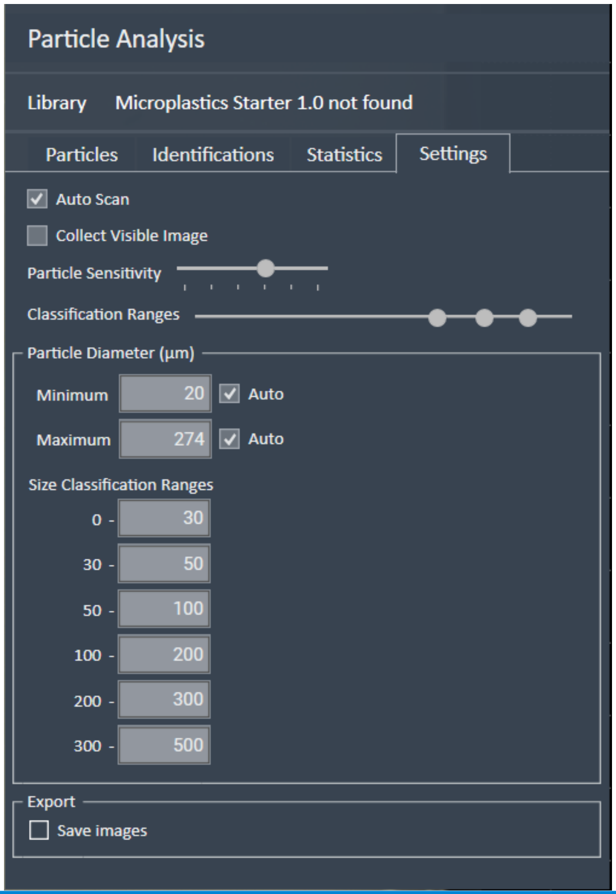
LDIR Microplastics Analysis Workflow

Insert sample → Locate and measure all particles →

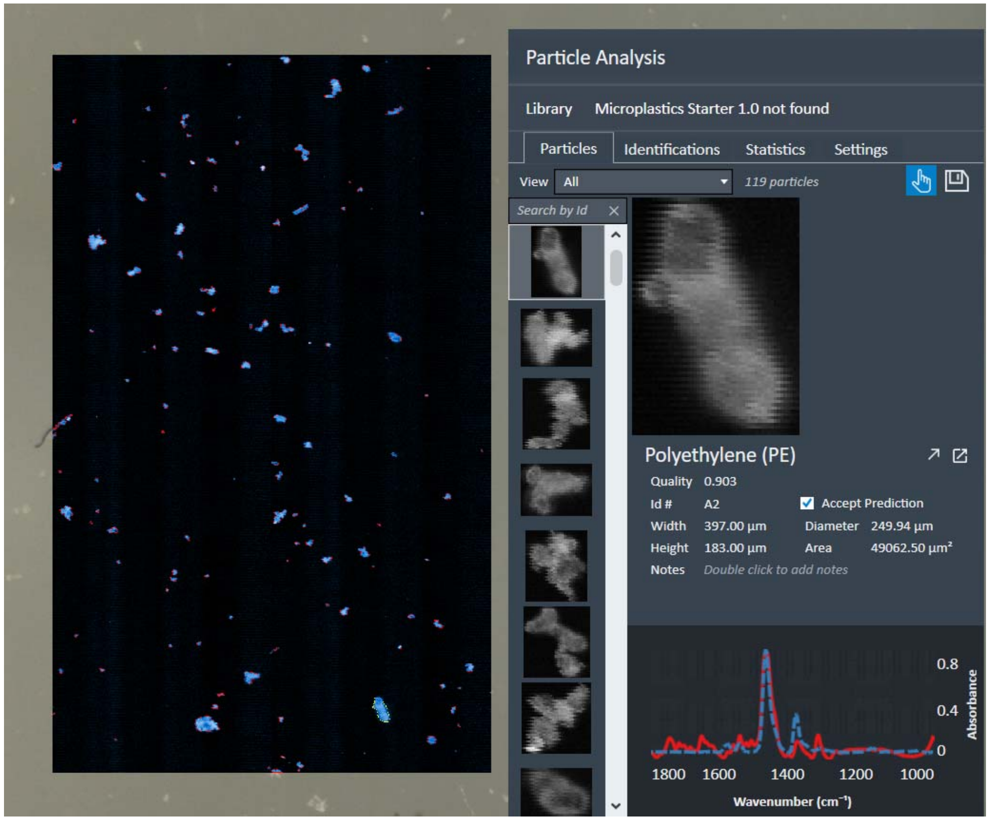
- Acquire spectrum
- Compare to standard IR library
- Update statistics

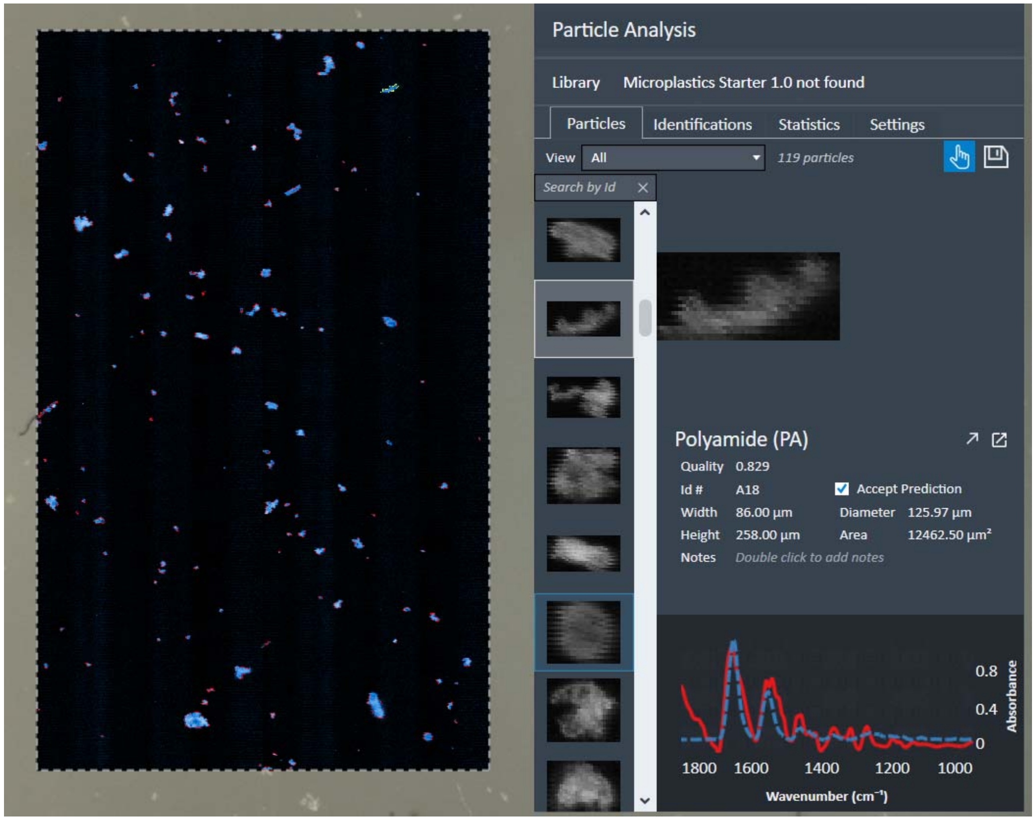
For every particle

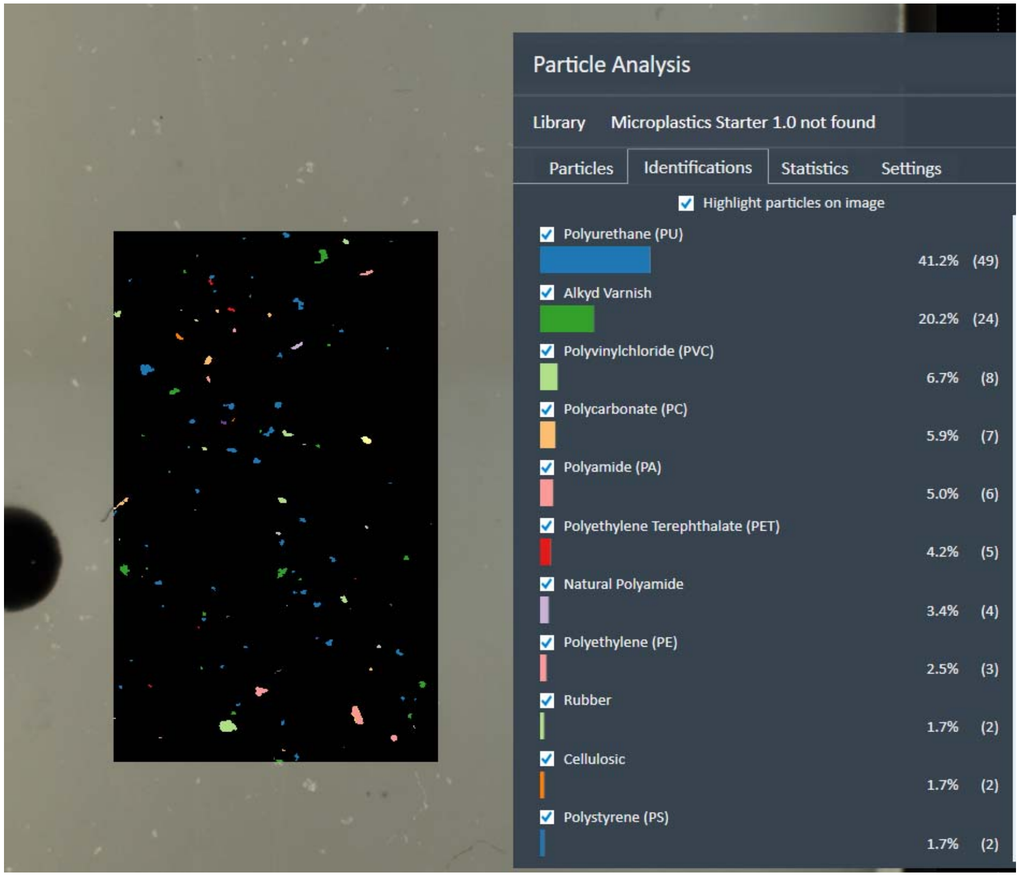
3 Steps for analysis



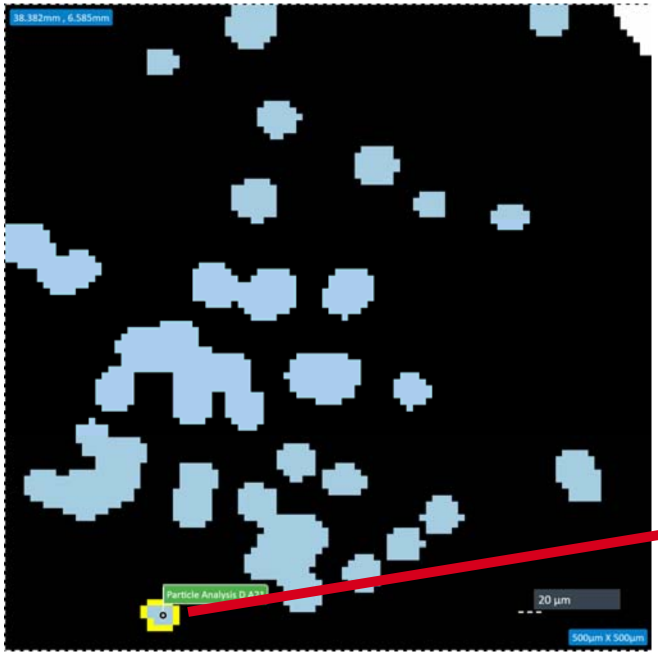
Scan and Identification

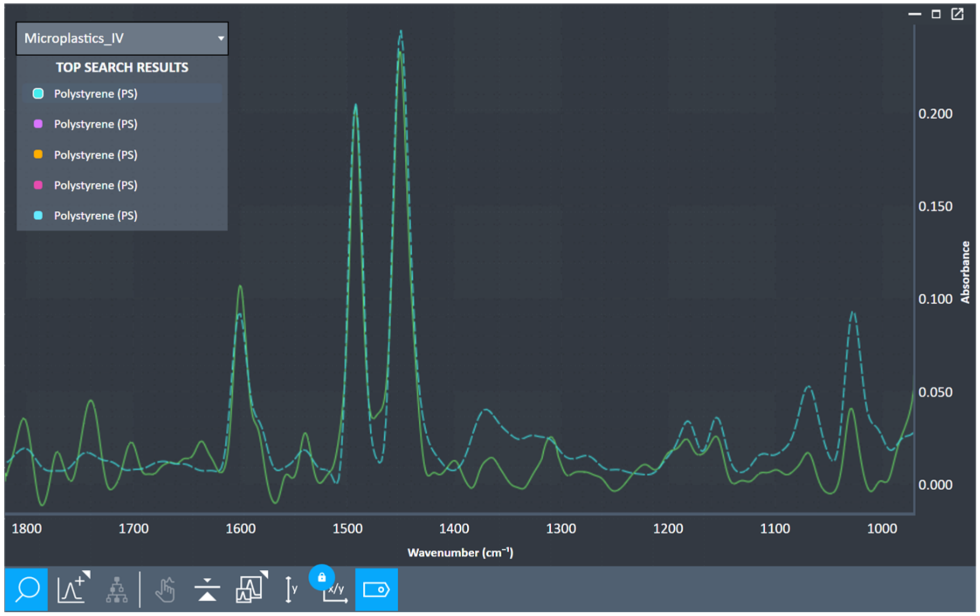




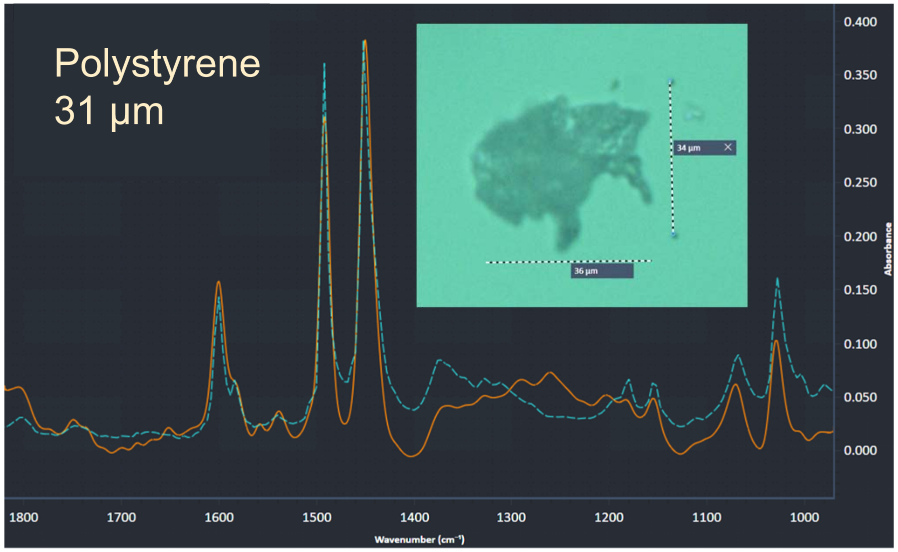

Particle Analysis
Library
Microplastics Starter 1.0 not found
Particles
Identifications
Statistics
Settings
Highlight particles on image
Polyurethane (PU)
41.2% (49)
Alkyd Varnish
20.2% (24)
Polyvinylchloride (PVC)
6.7% (8)
Polycarbonate (PC)
5.9% (7)
Polyamide (PA)
5.0% (6)
Polyethylene Terephthalate (PET)
4.2% (5)
Natural Polyamide
3.4% (4)
Polyethylene (PE)
2.5% (3)
Rubber
1.7% (2)
Cellulosic
1.7% (2)
Polystyrene (PS)
1.7% (2)

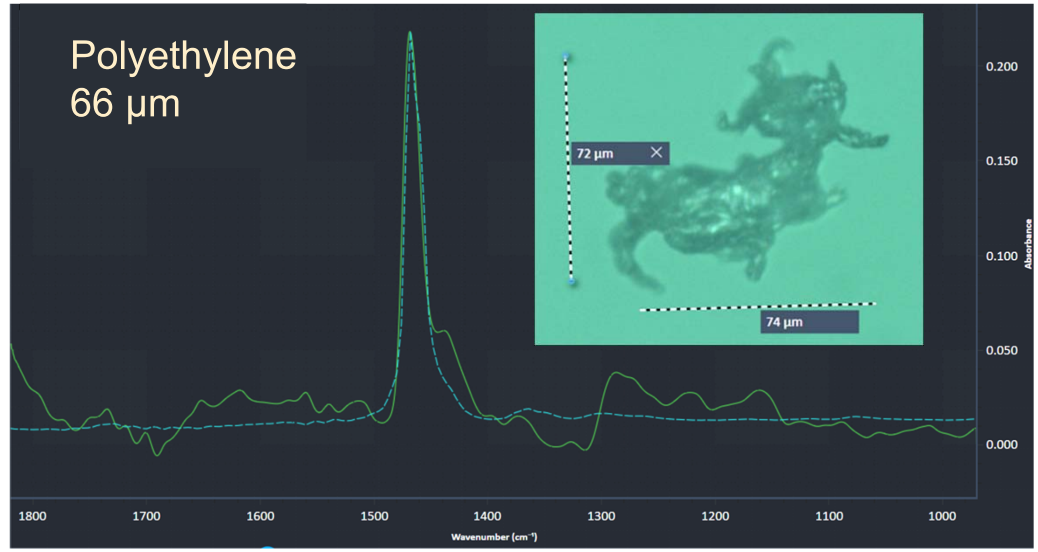
Spectral example

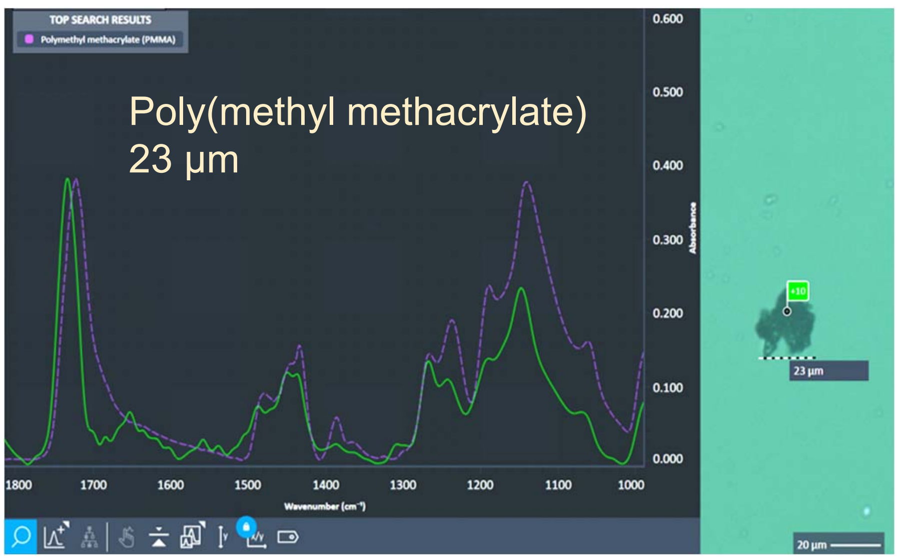


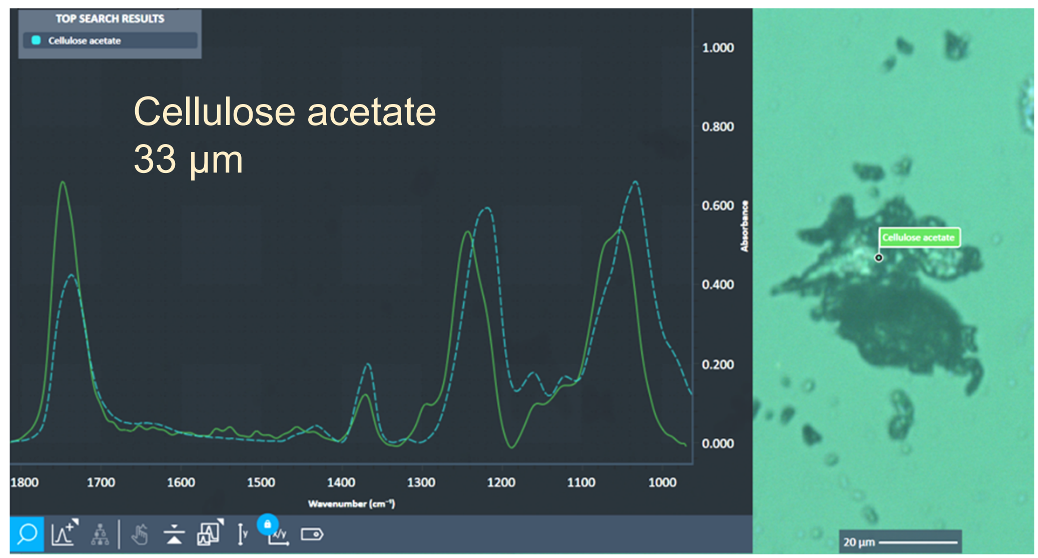


Spectrum from PS bead obtained from automated analysis; very good match with PS in library.


Polystyrene
31 µm
34 µm
36 µm
0.400
0.350
0.300
0.250
0.200
0.150
0.100
0.050
0.000
Absorbance
1800
1700
1600
1500
1400
1300
1200
1100
1000
Wavenumber (cm⁻¹)

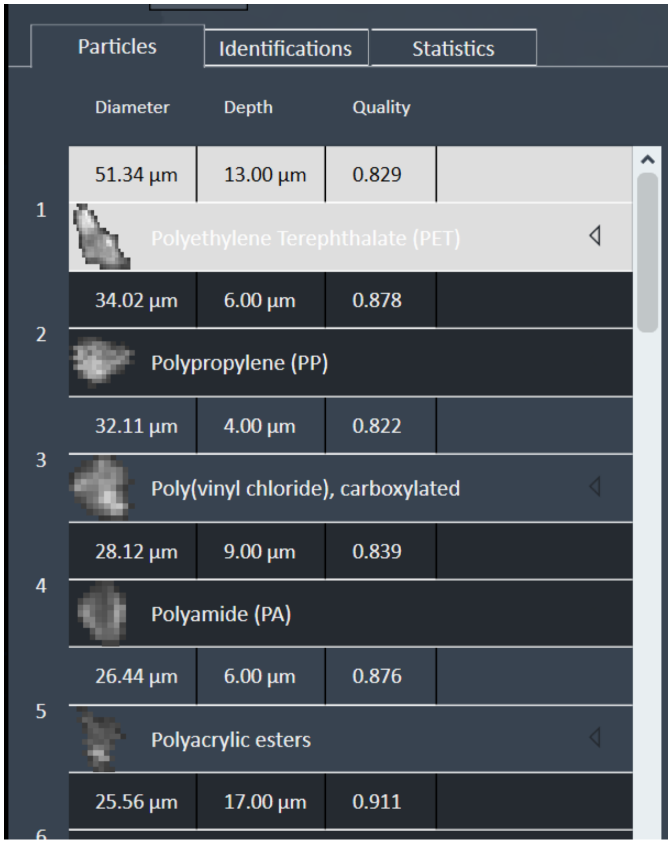

Polyethylene
66 µm
72 µm
74 µm
0.200
0.150
0.100
0.050
0.000
Absorbance
1800
1700
1600
1500
1400
1300
1200
1100
1000
Wavenumber (cm⁻¹)

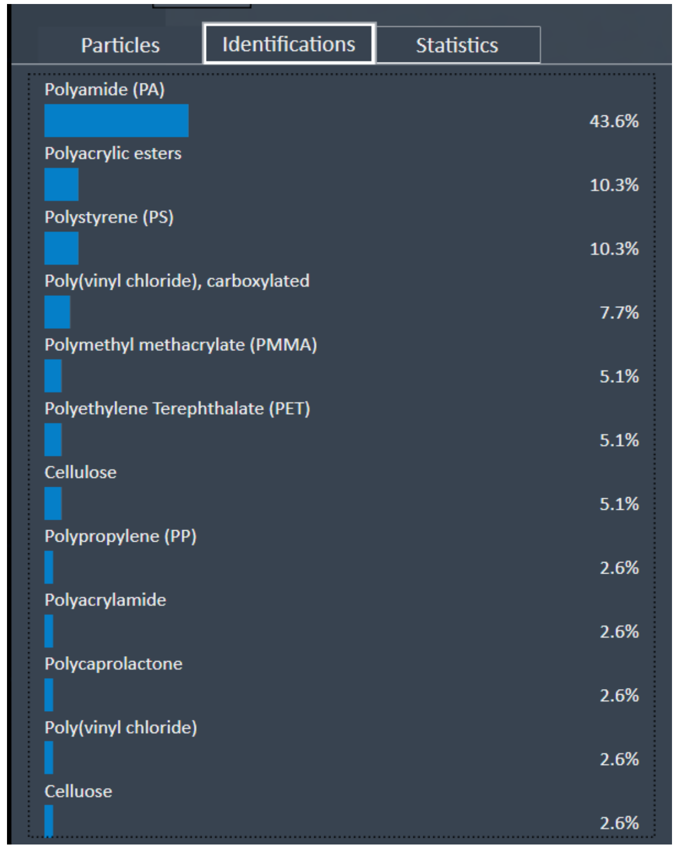

TOP SEARCH RESULTS
Polymethyl methacrylate (PMMA)
Poly(methyl methacrylate)
23 µm
0.600
0.500
0.400
0.300
0.200
0.100
0.000
Absorbance
1800
1700
1600
1500
1400
1300
1200
1100
1000
Wavenumber (cm⁻¹)
23 µm
20 µm

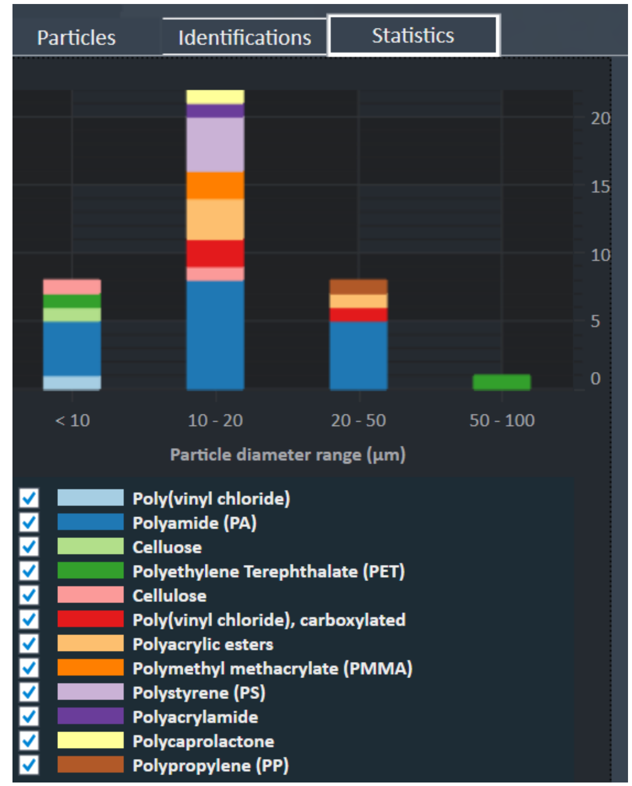

TOP SEARCH RESULTS
Cellulose acetate
Cellulose acetate
33 µm
1.000
0.800
0.600
0.400
0.200
0.000
Absorbance
1800
1700
1600
1500
1400
1300
1200
1100
1000
Wavenumber (cm⁻¹)
Cellulose acetate
20 µm

Reported results

Particle breakdown and statistical analysis

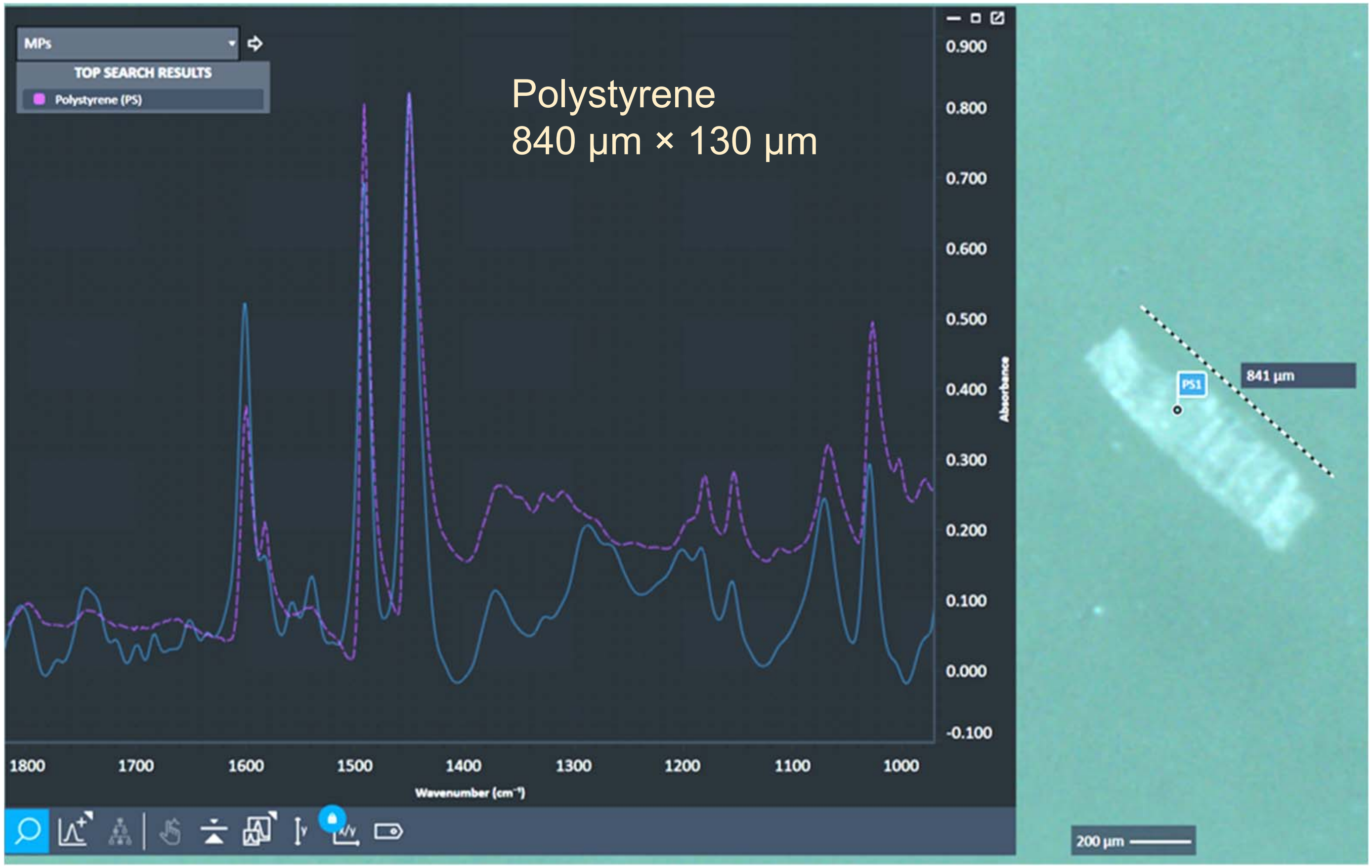






3 mm × 3 mm; 39 particles; 5 minutes

Large microplastic example

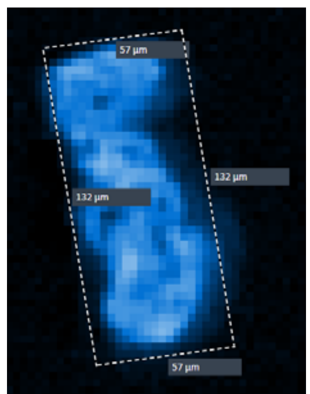


Both large (1 mm) and small (10 µm) particles can be measured in the same analysis

Bright laser light is able to transmit through thick particles and ID them

Physical Properties

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	#	Id	Width	Height	Diameter	Aspect Ra	Area	Perimeter	Eccentricity	Circularity	Solidity	Identification	Quality	Is Valid
2	1	A12	132	57	87.91587	2.315789	6070.5	119.4974736	0.654157982	35.46525598	0.875405581	Polyethylene	0.95073032	true
3	2	A30	87	87	82.39872	1	5332.5	101.8406196	0.766896556	36.55503384	0.905963303	Polyethylene Terepthalate	0.837835667	true
4	3	A20	57	75	67.0223	0.76	3528	78.08326018	0.802531625	31.54336326	0.951456311	Polystyrene	0.974123981	true
5	4	A11	87	30	53.68405	2.9	2263.5	71.21320307	0.774589202	22.19000181	0.952651515	Polyvinyl Chloride (PVC)	0.855147023	true
6	5	A3	54	54	45.03633	1	1593	74.42640603	0.794447413	14.94259711	0.720977597	Polyethylene	0.933734214	true
7	6	A32	42	42	42.34806	1	1408.5	48.87005723	0.679627729	20.12108347	0.951367781	Polyethylene Terepthalate	0.873186413	true
8	7	A10	51	30	39.54965	1.7	1228.5	46.384776	0.783735224	18.49000617	0.975	Polyamide (PA)	0.745801906	true
9	8	A13	45	27	36.30156	1.666667	1035	42.14213526	0.646254678	17.14593496	0.978723404	Chitin	0.688479698	false
10	9	A14	48	27	33.51115	1.777778	882	46.28427052	0.580561718	13.30370238	0.863436123	Polyamide (PA)	0.929741675	true
11	10	A31	36	27	31.20943	1.333333	765	34.97056234	0.686665089	15.27200924	0.977011494	Polyvinyl Chloride (PVC)	0.8618916	true
12	11	A33	36	24	28.92263	1.5	657	32.97056234	0.61435788	13.91157732	0.979865772	Polyester	0.859101809	true
13	12	A21	24	24	25.44489	1	508.5	29.55634892	0.614112504	12.01095476	0.941666667	Polyvinyl Chloride (PVC)	0.727845913	true
14	13	A9	36	21	24.99048	1.714286	490.5	31.55634904	0.446842469	10.85149612	0.923728814	Polyethylene Terepthalate	0.890295468	true
15	14	A16	24	18	21.54288	1.333333	364.5	23.89949465	0.510614829	10.64746384	1	Polyethylene Terepthalate	0.888116627	true
16	15	A35	24	18	21.27526	1.333333	355.5	23.89949465	0.592095545	10.38456349	0.9875	Acrylates Polyurethanes Varnish	0.86158601	true
17	16	A7	21	21	20.31083	1	324	23.31370807	0.539651825	9.7022177	0.972972973	Polystyrene	0.917831107	true
18														



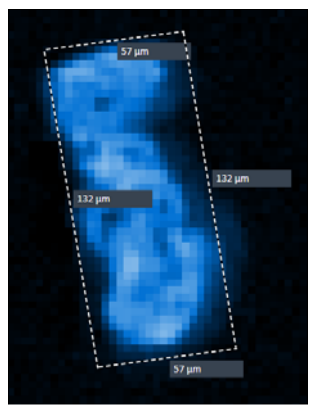
What do the reported parameters mean?

Aspect ratio = the ratio of width/height

Area = calculated based on the pixels enclosed by width and height

Diameter = calculated by equating the calculated particle area to the area of a circle: using the circle area, the diameter is calculated using equation A = pi * (diameter^2)/4

Circularity: Measures how close the shape of the particle is to a circle. A perfect circle will have a circularity of 1. Other shapes will have a value < 1.

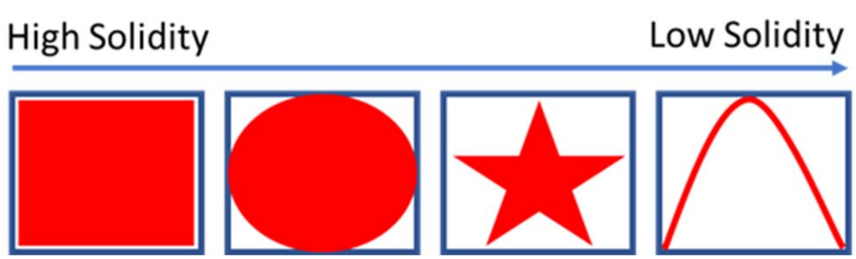


What do the reported parameters mean?

Perimeter: The length of the line that makes up the boundary of the particle.

Eccentricity: Another metric that characterizes the shape. A circle has a value of 0. Ellipses range from 0-1. A value close to 1 suggests a high aspect ratio.

Solidity: The ratio of the particle area over the area of its convex hull. That might be confusing so perhaps easier to say. A particle in the shape of a rectangle will have a high solidity close to 1. A starfish shape, or a fiber that is curving will have a low solidity since its area is small relative to its bounding area



Agilent 8700 LDIR Chemical Imaging System

Routine, robust, automated microplastics analysis by non-experts

Agilent

Thank you!

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