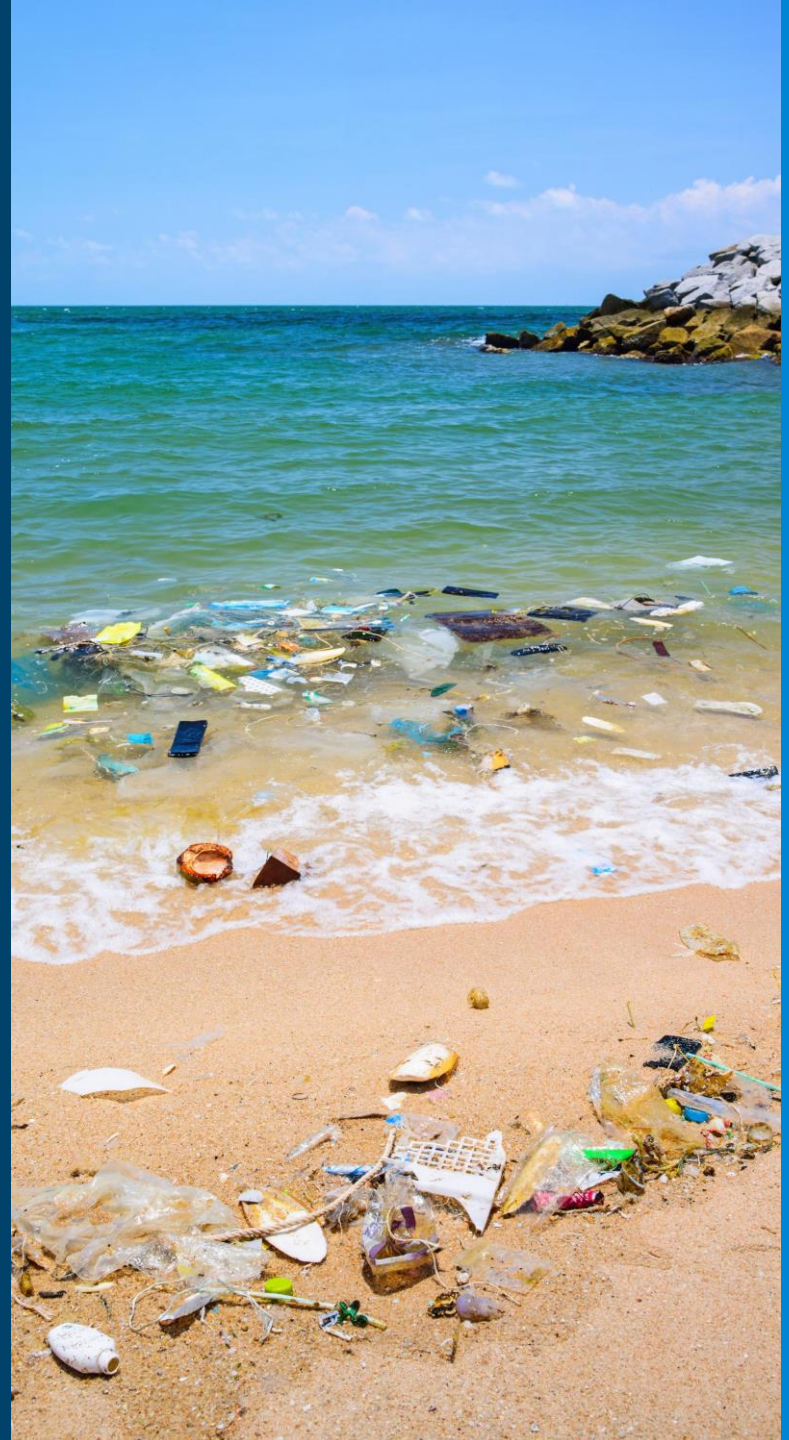
Developments in QCL-Based Spectroscopy for Rapid Identification of Microplastics

Darren Robey, Wesam Alwan, Ph.D, Louis Tisinger, Ph.D.

Agilent Technologies



Agilent
Trusted Answers

DE90196871

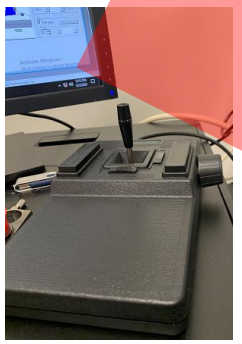
Agilent 8700 LDIR Chemical Imaging System

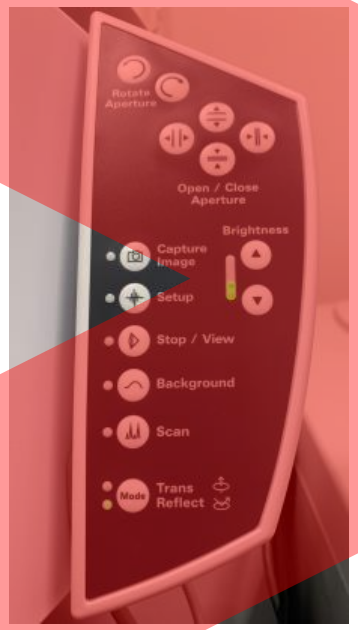
Routine, robust, automated microplastics analysis by non-experts

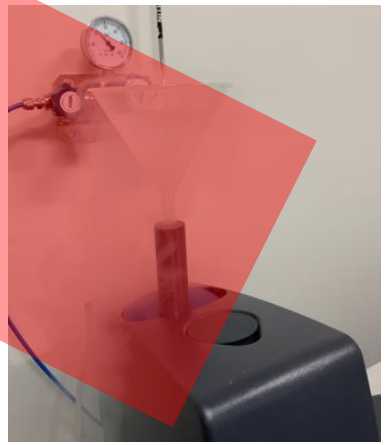
The Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system

- **A sophisticated new approach to chemical imaging and spectral analysis**.
  - With the 8700 LDIR, you can analyze more samples, in greater detail, in less time
  - Uses Quantum Cascade Laser (QCL) technology & rapidly scanning optics to provide fast, clear, high-quality images and spectral data.
  - Intuitive Agilent Clarity software provides rapid and detailed imaging of large sample areas with minimal instrument interaction.
  - Provides compositional analysis of microplastics, tablets, laminates, materials
  - Survey large sample areas and then explore smaller areas of interest in more detail without changing any optics.
  - No requirement for liquid nitrogen reduces operating costs and simplifies maintenance.

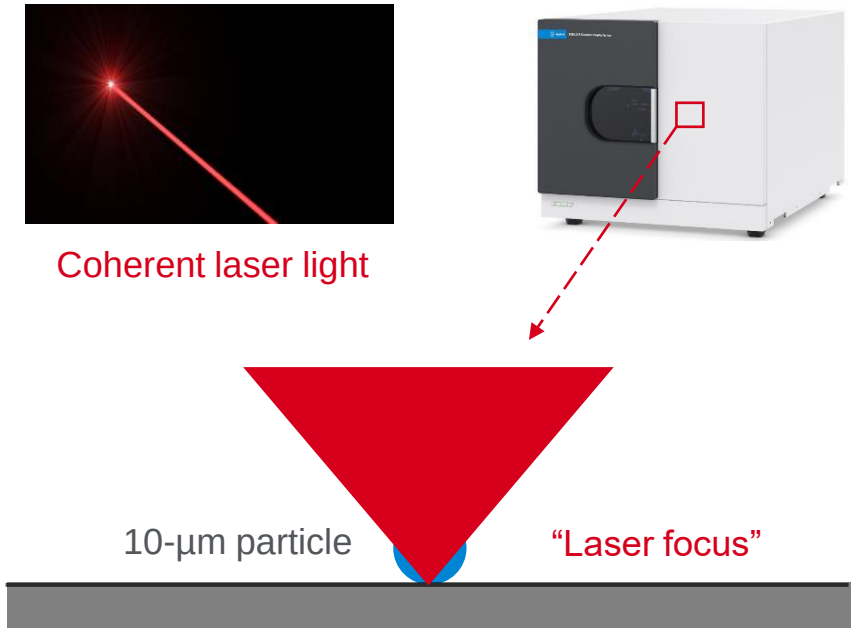
Traditional FTIR-based systems: More Manual Tasks







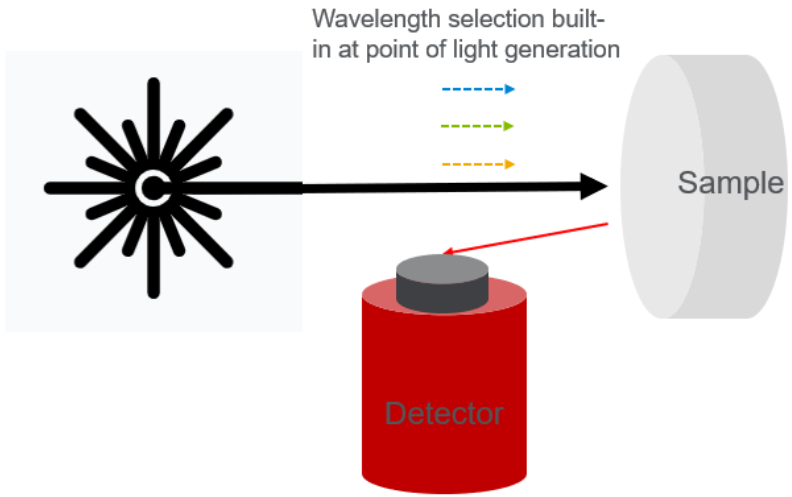
Laser Direct Infrared (LDIR) Spectroscopy



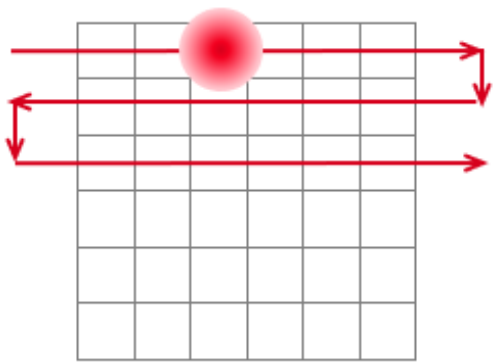
All light reaches the particle

- Bright, coherent light source
- Focus all laser power onto a particle
- New instrument architecture
- Proprietary Agilent quantum cascade laser (QCL) technology
- Rapidly tunable across the mid-infrared fingerprint region for spectroscopy

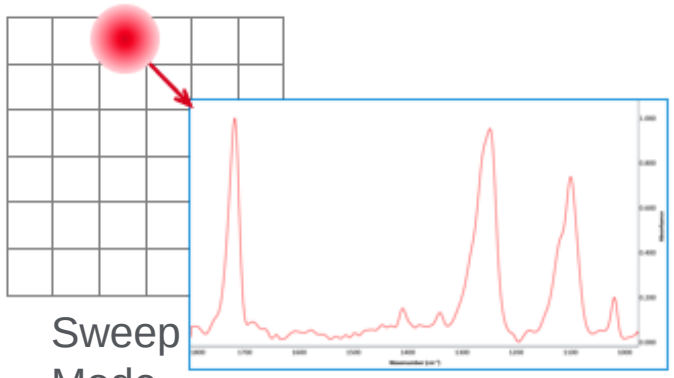
Analyzing Microplastics using Agilent 8700 LDIR



QCL IR spectroscopy



Scan Mode “Discrete Frequency Sampling”



Sweep Mode

Modes of Action

Proprietary Agilent quantum cascade laser (QCL) technology

- Bright, coherent light source. More power, directional: Focus all laser power onto a particle
- Rapidly tunable across the mid-infrared for spectroscopy.

Scan Mode

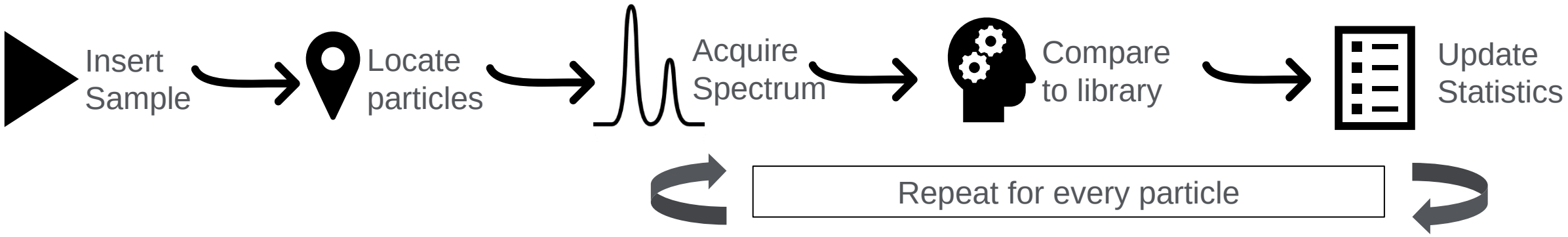
- Single wavelength, scan the sample quickly
- Can be done multiple times for multiple wavelengths at high speed.
- Understand the spatial distribution of known components
- Locate discrete particles

Sweep Mode

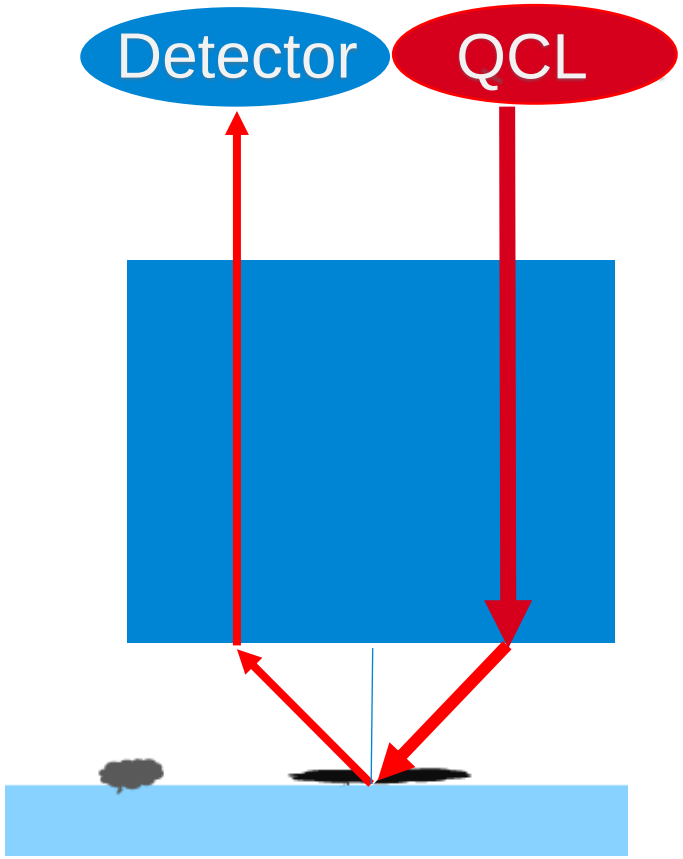
- Single Position
- Full Sweep available wavelengths
- Utilize full spectrum for library matching

Agilent Solution: 8700 LDIR chemical imaging system

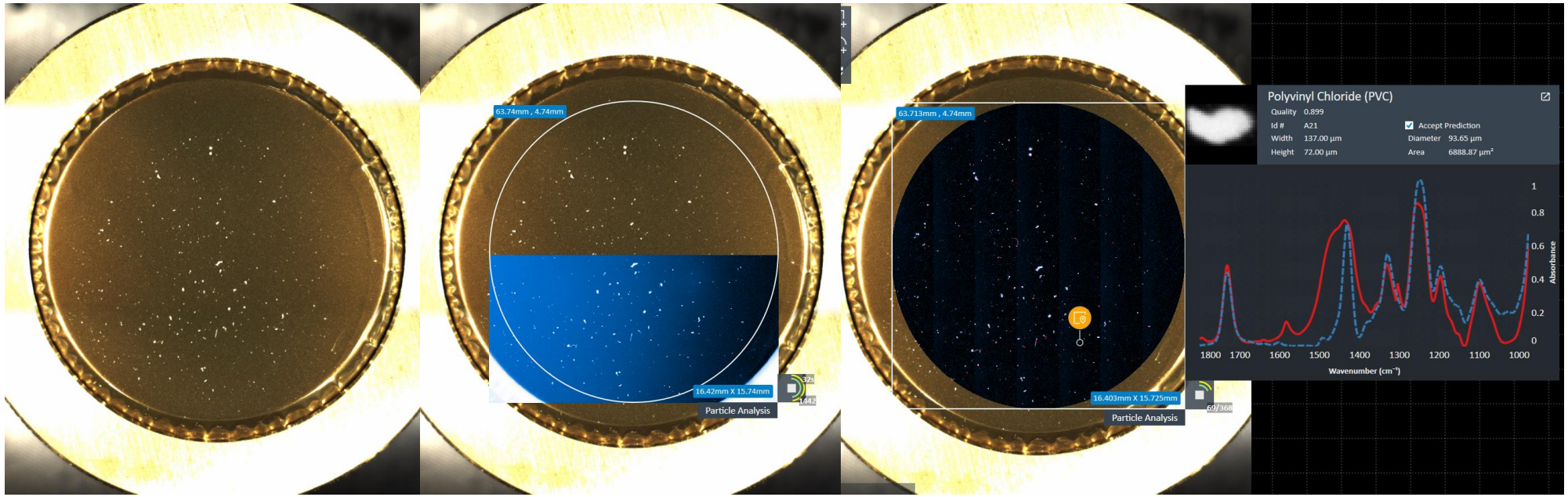
Analysis Workflow – 8700 LDIR + Clarity software



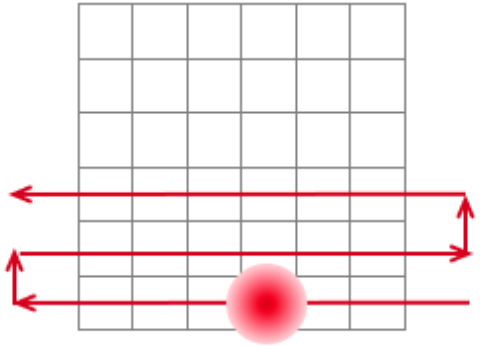
Obtain spectra by passing infrared beam thru sample reflecting off low-E slide or reflective filter (transflectance) and then back thru sample to the detector.



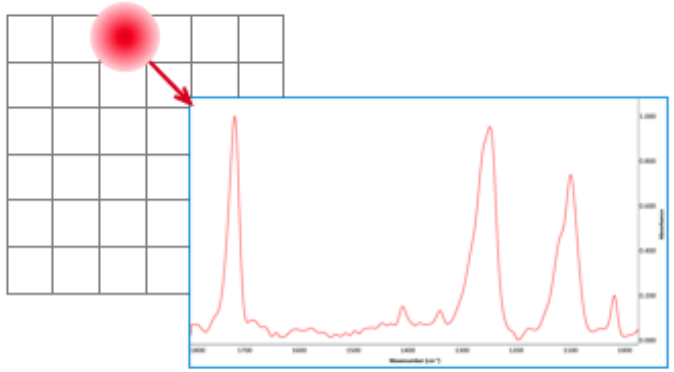
Data Collection



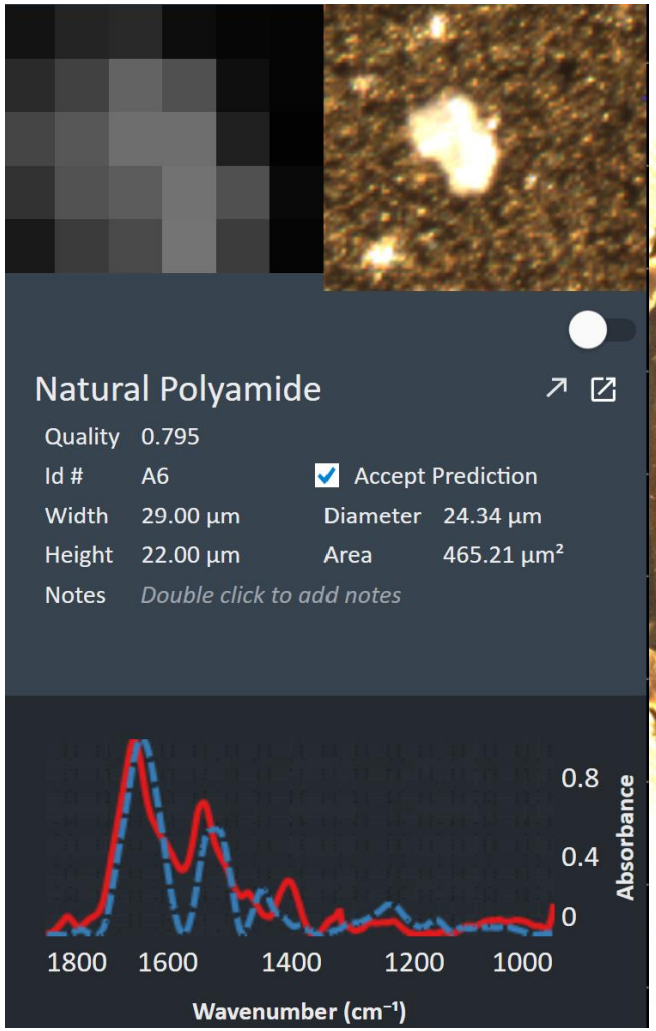
Visible Image

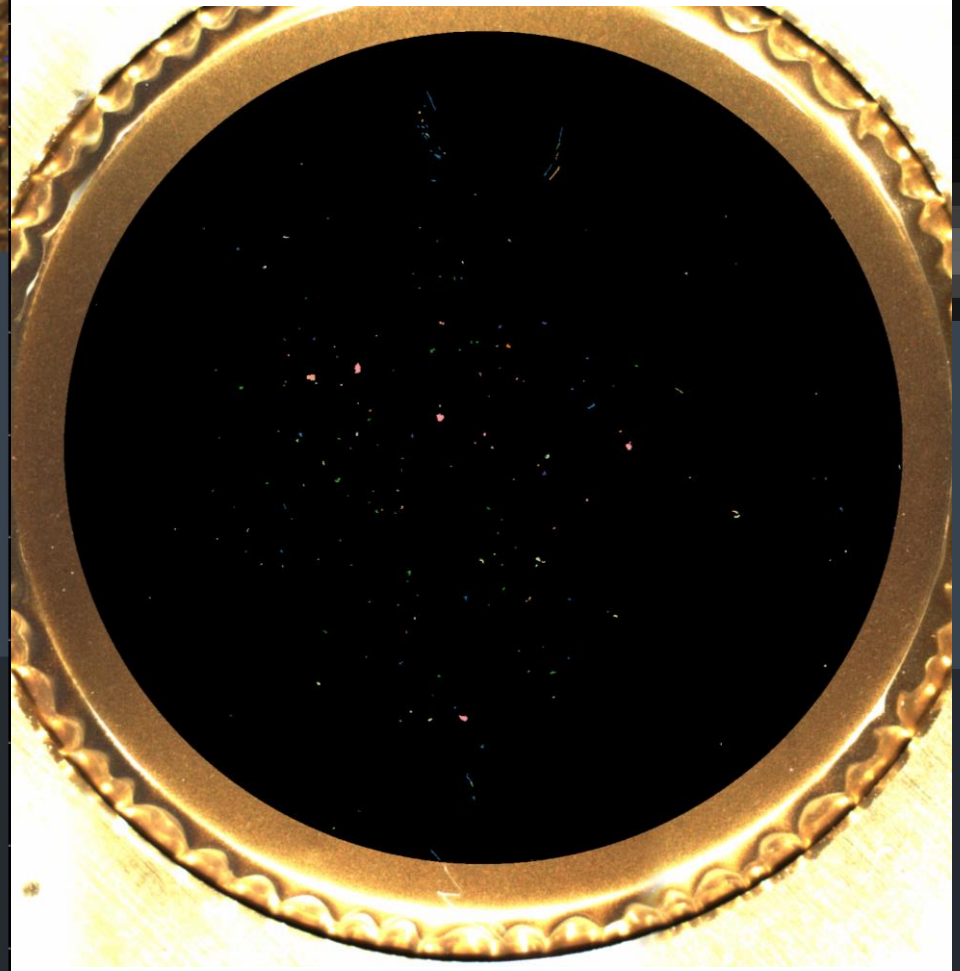


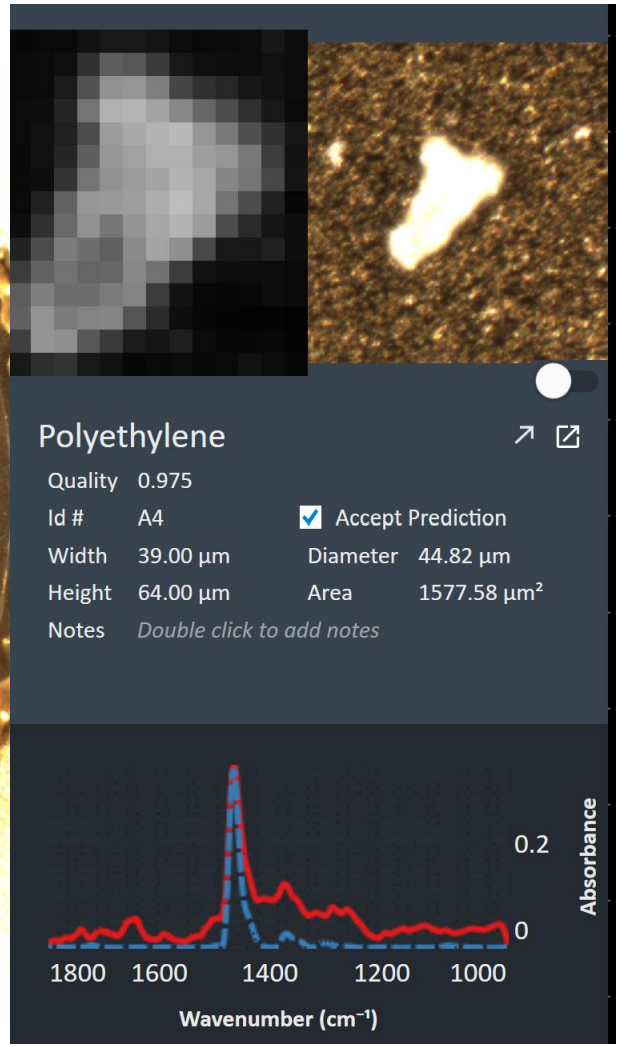
Discrete Frequency Mode



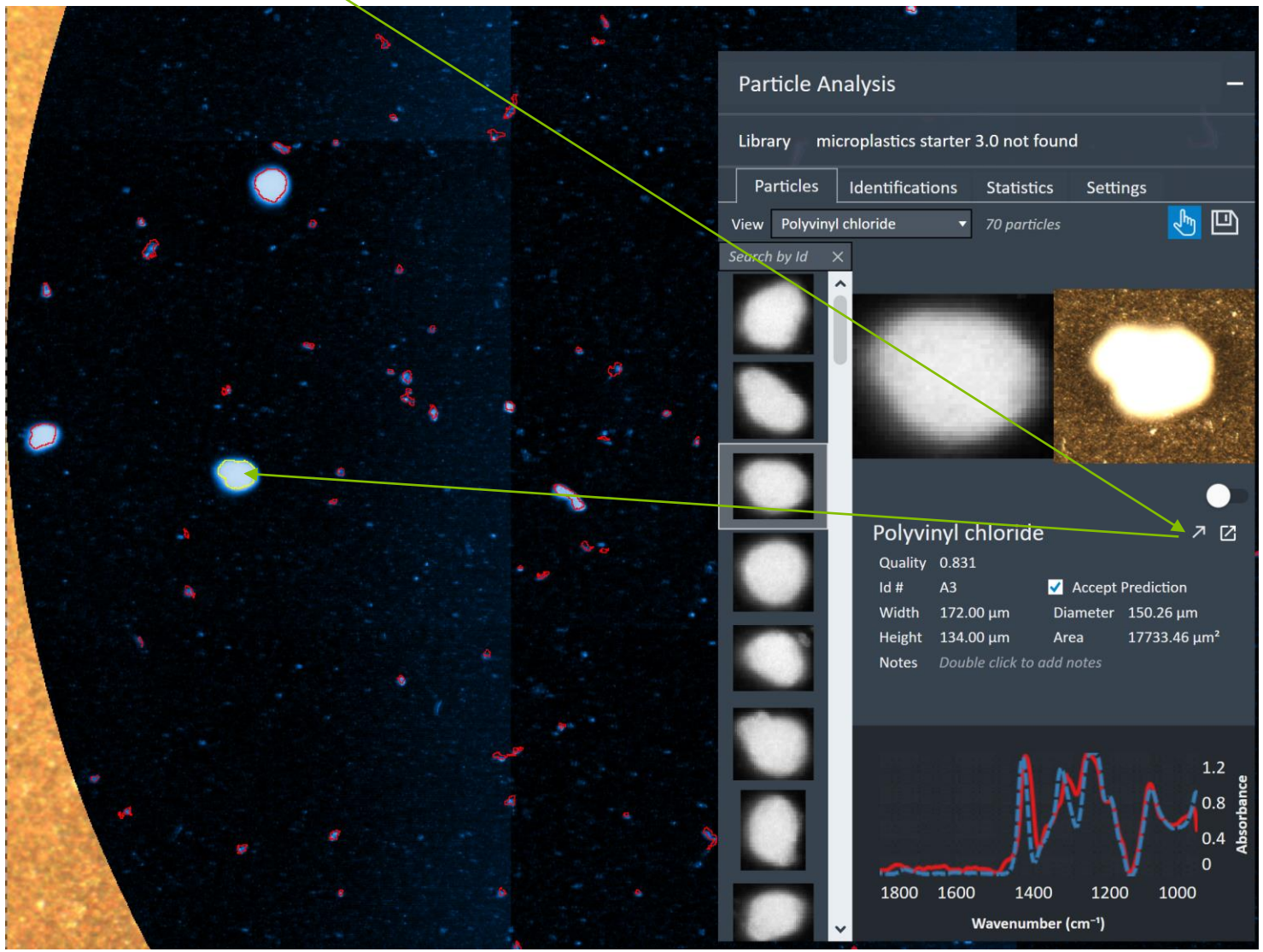
Sweep Mode


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)

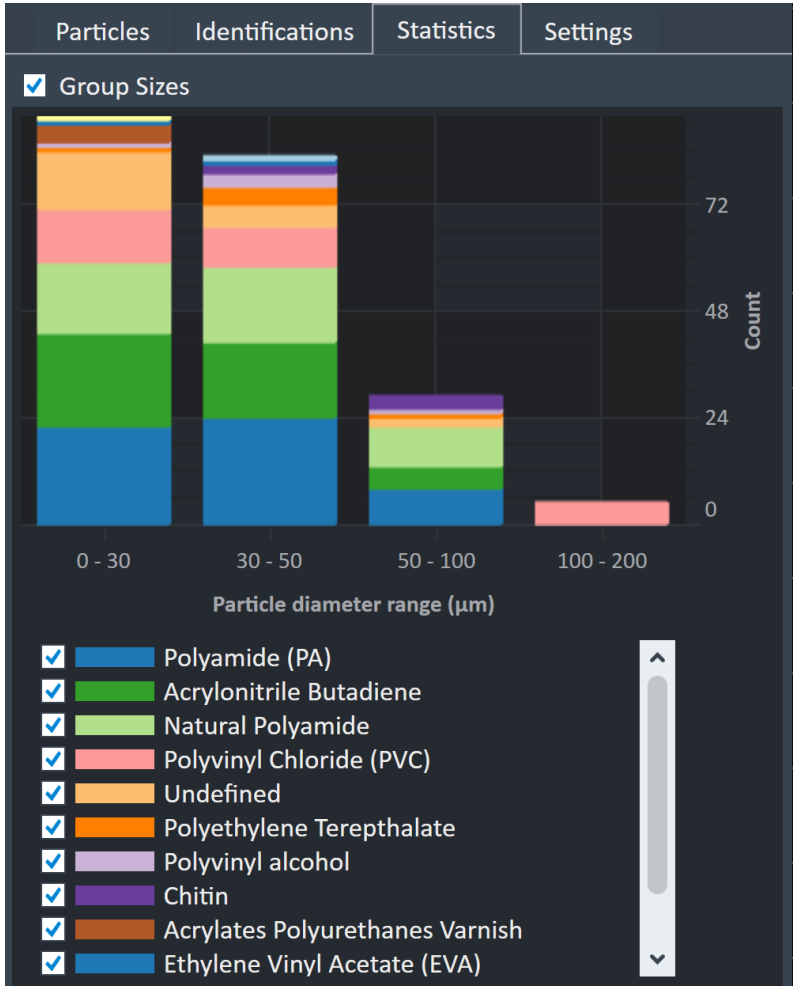


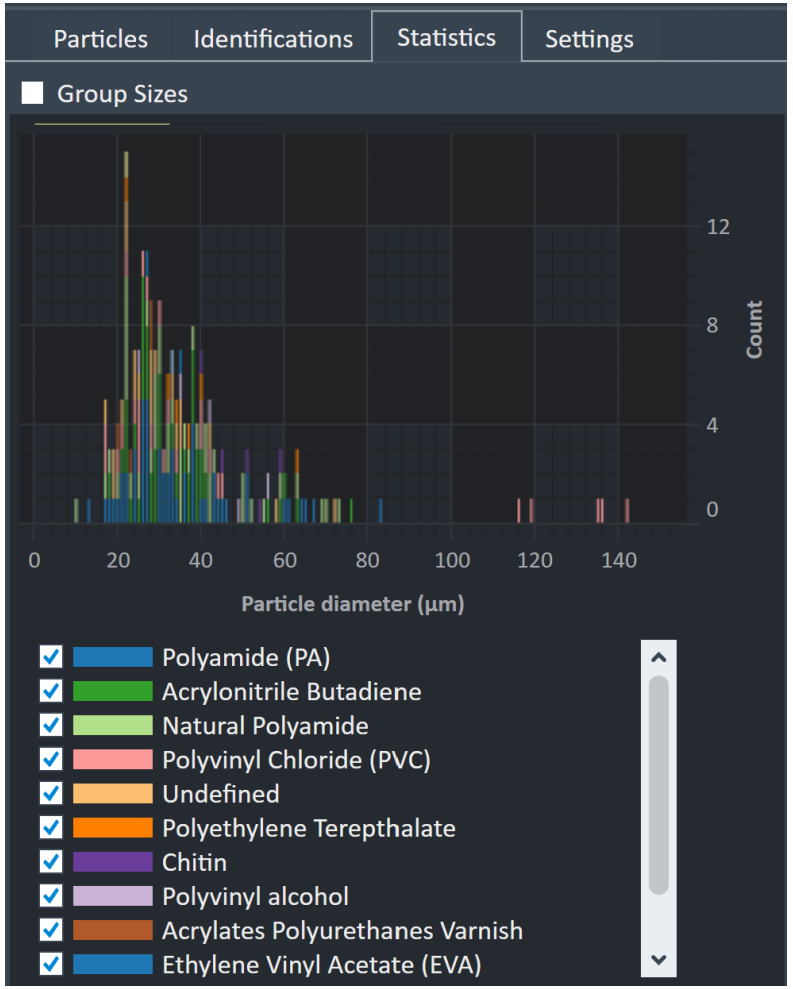

Polyethylene
Quality 0.975
Id # A4
Accept Prediction
Width 39.00 µm
Diameter 44.82 µm
Height 64.00 µm
Area 1577.58 µm²
Notes Double click to add notes
0.2
0
Absorbance
1800 1600 1400 1200 1000
Wavenumber (cm⁻¹)

Example Particle: Particles can be sorted by type and highlighted in image, using the “Zoom” function.



Reported Results: Statistics are updated as analysis proceeds





Improvements To MP Workflow

Outline

- **Microplastics Analysis Validation Using Aluminum-Coated Filters**
  - Sample introduction
  - Useability and handling
  - Detection of particles
  - Repeatability of particle count, size, and identification data
  - Particle size accuracy
  - Identification of common microplastics
  - Spectra quality
- **Clarity 1.6 Improvements**
  - Speed of Analysis
  - Data Archiving
  - Wavenumber selection

Microplastics Analysis Validation Using Aluminum-Coated Filters

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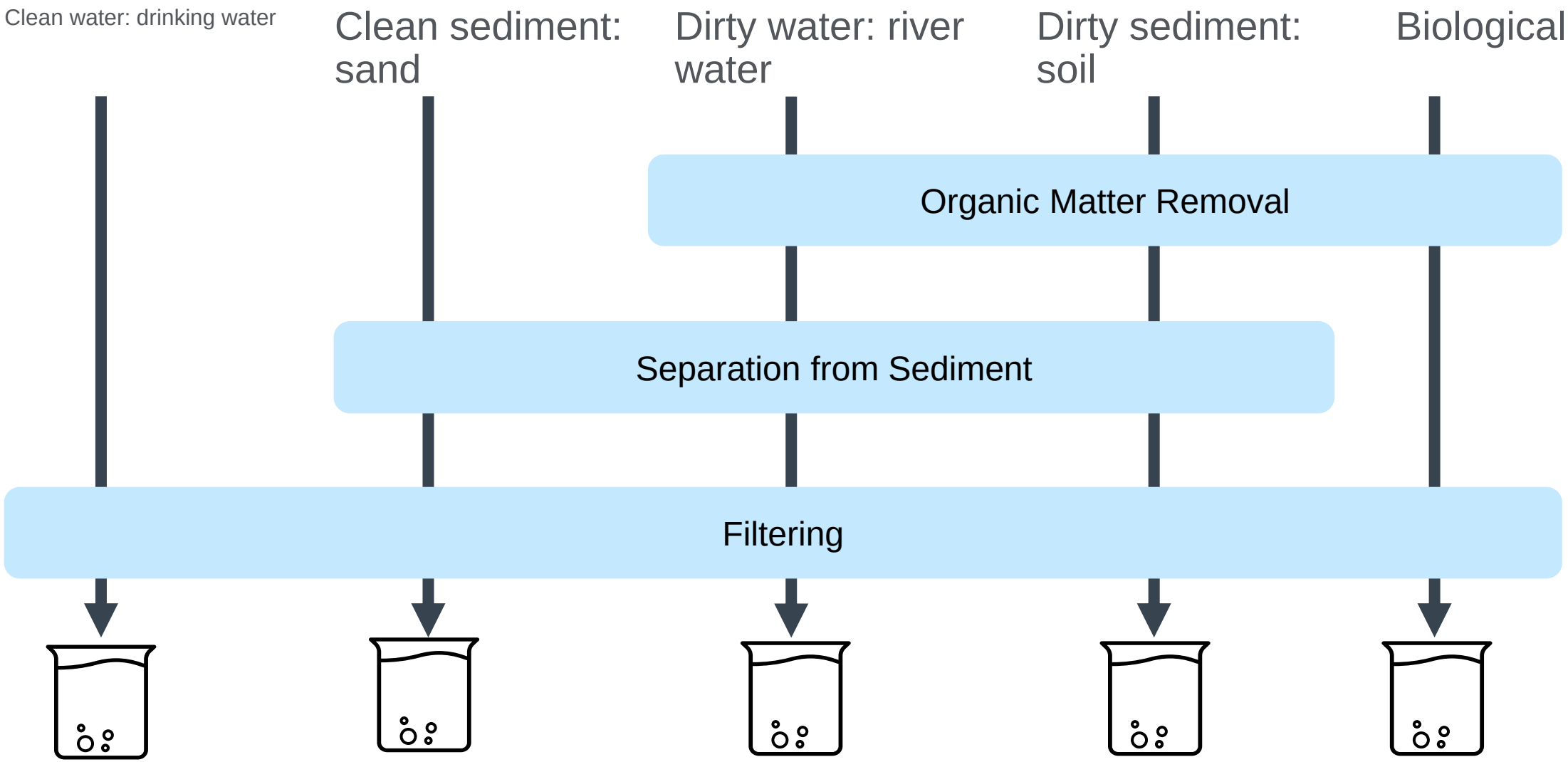
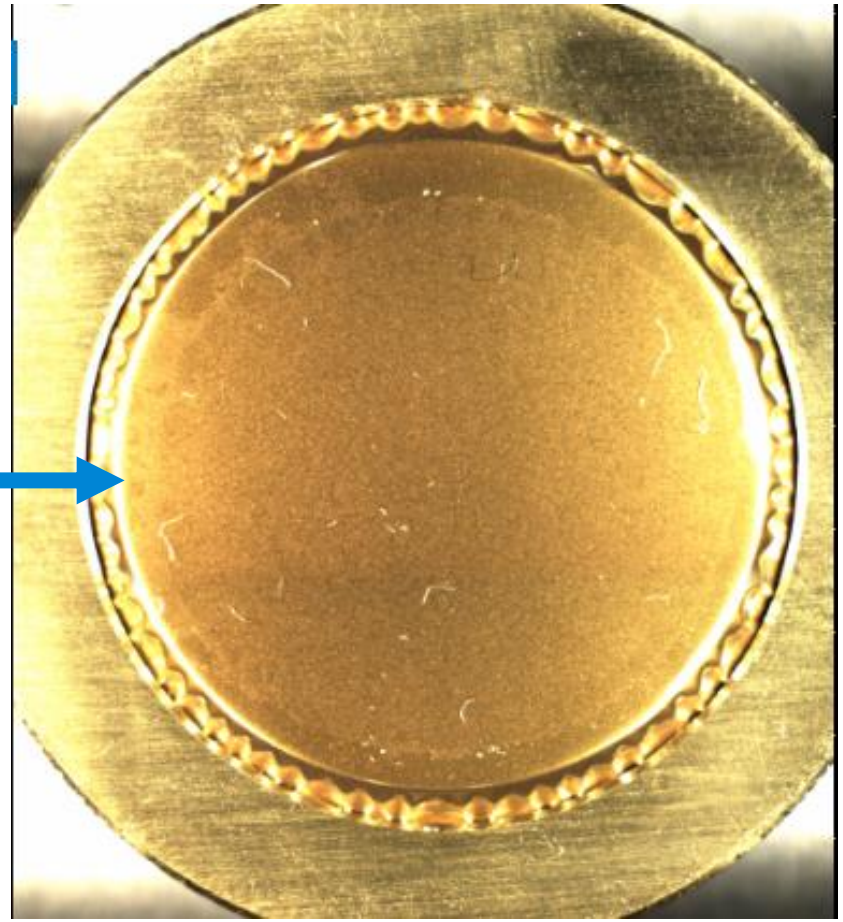
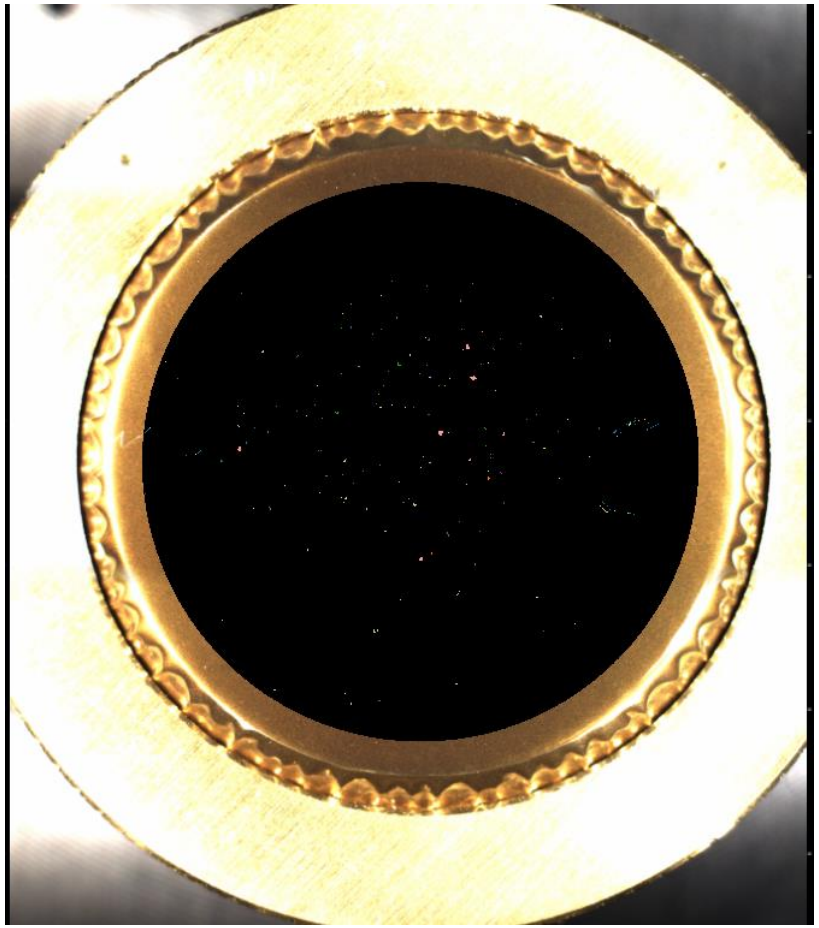
Sample preparations



Direct Analysis of Microplastics Using Gold-coated Filters





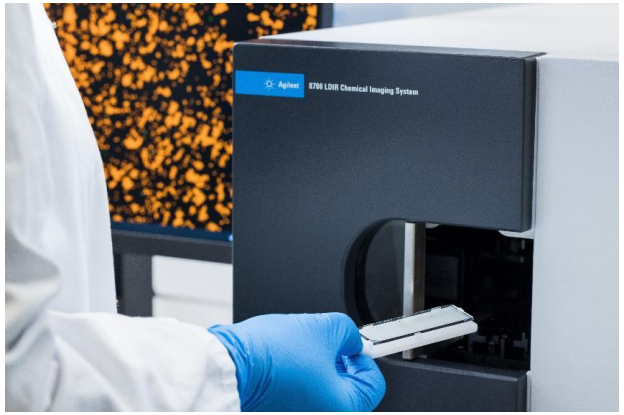
- Minimal sample preparation
- Reduced contamination
- Improved sample representation

Aluminum-Coated Filters: Sample Introduction

Extracted Microplastics

NEW!

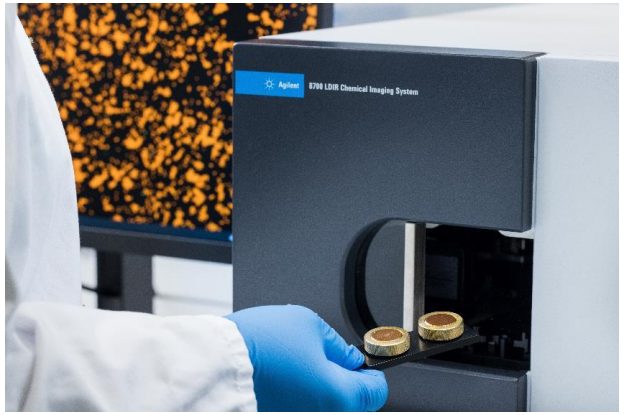
Option 1



Low-e slide

- Ideal for high number of particles
- Large area of analysis (25 x 75 mm)

Option 2



25 mm gold-coated filter

- More sample representation
- Easy sample preparation
- Reduced contamination
- Two filters analyzed sequentially

Option 3

25 mm Aluminum-coated filter

- More sample representation
- Easy sample preparation
- Reduced contamination
- Two filters analyzed sequentially

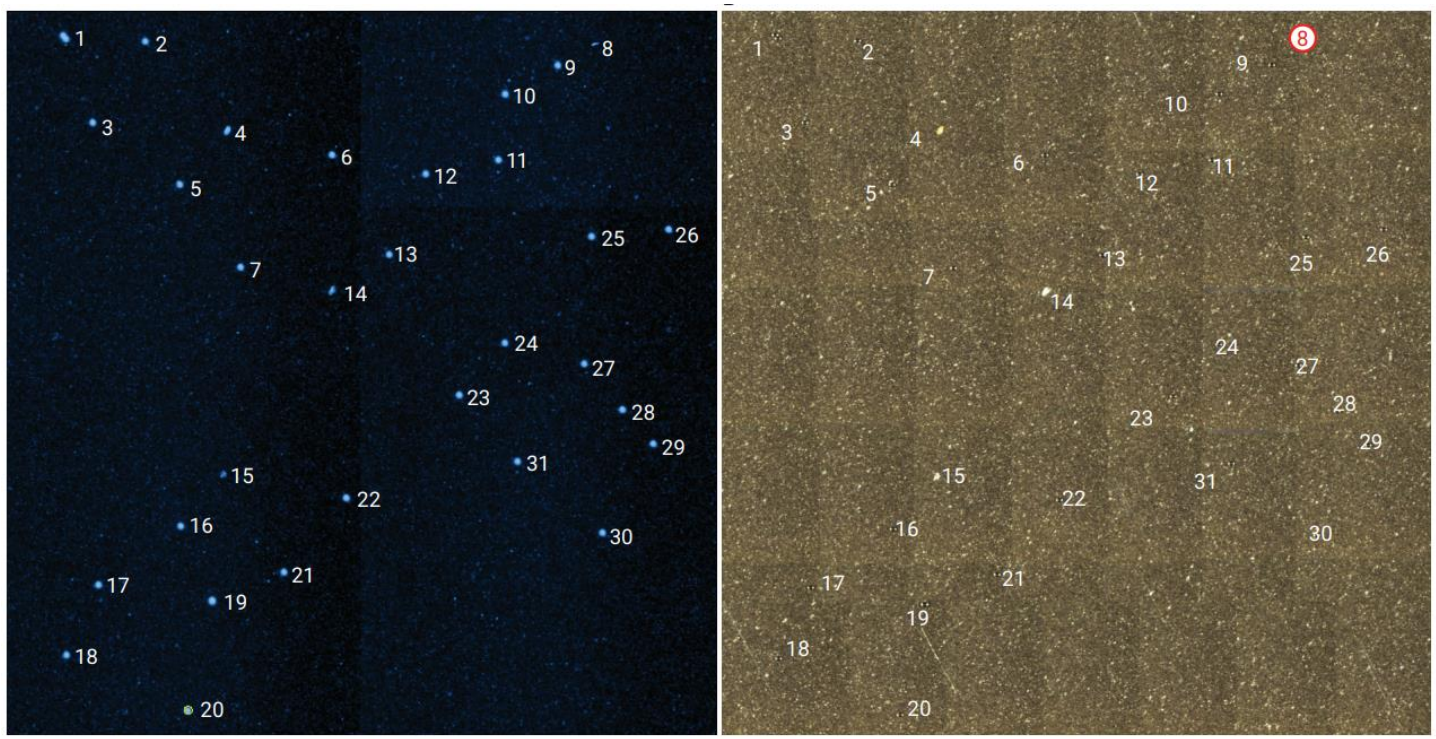
Aluminum-Coated Filters: Useability and handling

- Aluminum-coated polyester - i3 TrackPor PA (25 mm, 100/0 nm coating, 0.8 µm pore size)
  - Cheaper than gold-coated filters
  - Less prone to folding than gold-coated filters.
  - Easier to position on the filter holder due to increased rigidity of the coating
- Orderable via Agilent or directly from i3 Membrane GmbH

Same filtration workflow as gold-coated filters

Aluminum-Coated Filters: Detection of particles

- Step 1: Particle Analysis Workflow (31 Particles)
- Step 2: Manual Count Using High-Mag Image (30 Particles).
- Excellent detection. ✓
- High quality IR images. ✓

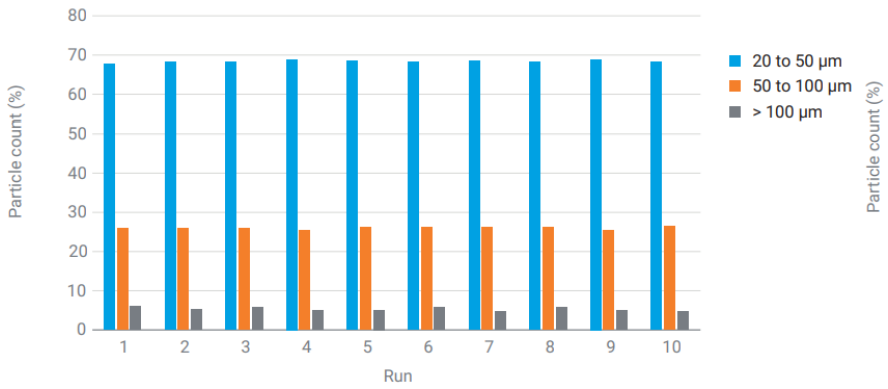


Shows the value of finding particles via IR imaging

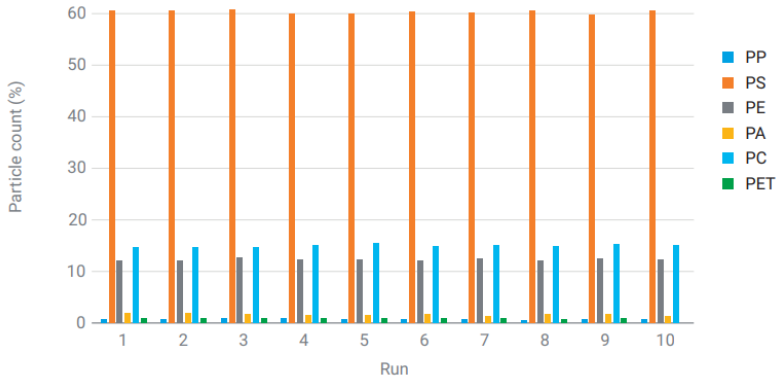
Aluminum-Coated Filters: Repeatability of particle count, size, and identification data

- Particle Analysis Workflow, same area (n = 10)
- < 1% variability based on total particles, size range and polymer identification. ✓
- Excellent reliability and accuracy. ✓

Total particle count



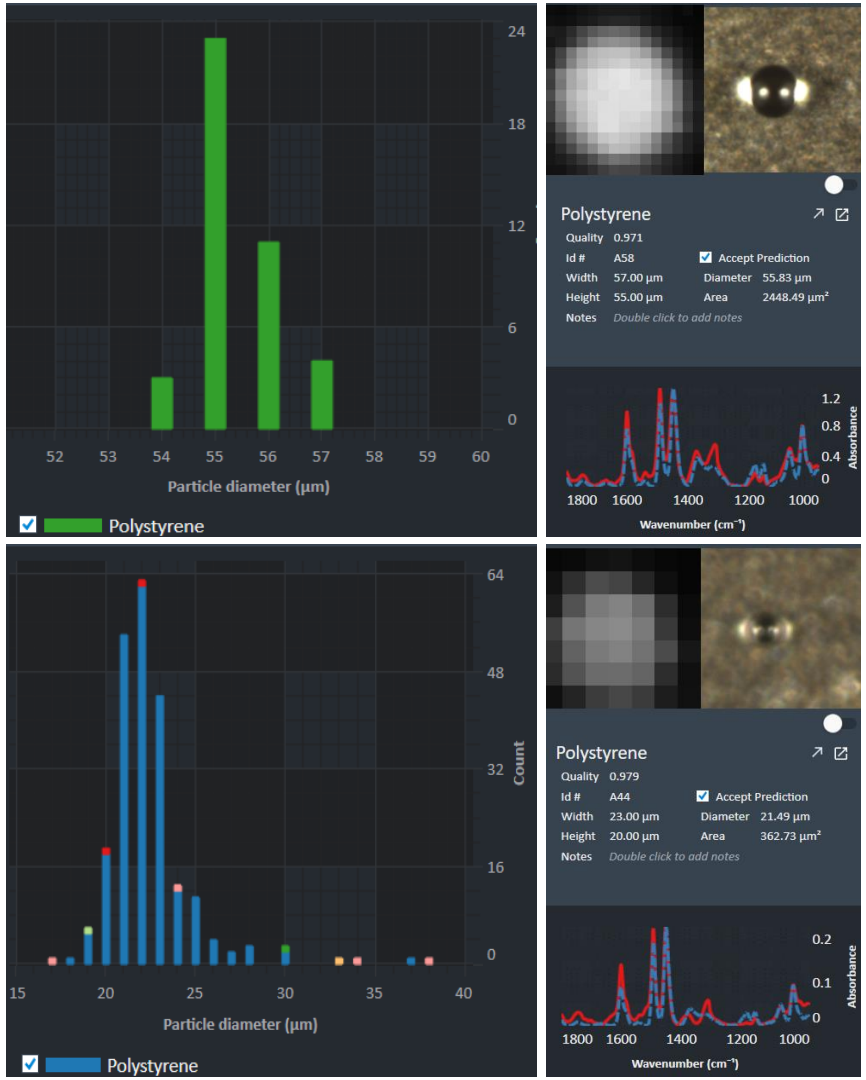
Size range



Identification

Aluminum-Coated Filters: Particle size accuracy

- 50 µm NIST traceable polystyrene latex beads: 37 particles; average size of 55.10 µm (standard deviation = 3.67 µm)
- 20 µm NIST traceable polystyrene latex beads: 223 particles; average size of 22.90 µm (standard deviation = 2.30 µm)
- Accurate particle sizing with minimal variation. ✓



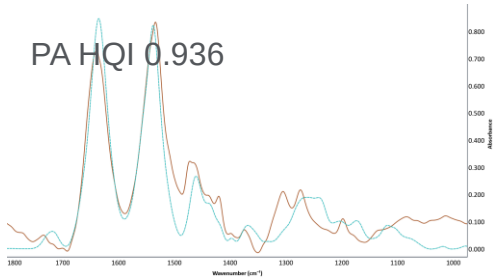
Aluminum-Coated Filters: Identification of common microplastics

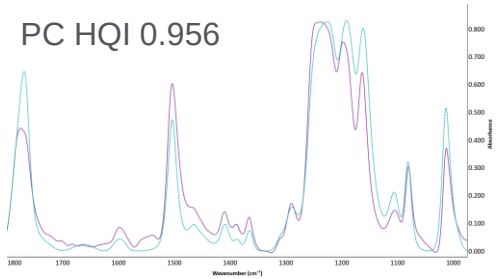
- Accurate identification of major types of microplastics. ✓
- High HQI scores, which surpassed 0.8 for 93 to 100% of the particles. ✓

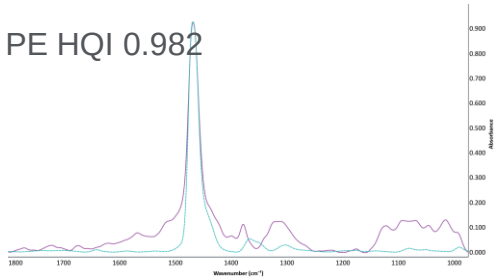
Polymer	Particle count	HQI >0.8	HQI >0.8 (%)
PET	345	330	96%
PC	418	392	94%
PA	506	493	97%
PE	241	241	100%
PMMA	234	227	97%
POM	107	99	93%
PTFE	550	546	99%
PU	504	491	97%
PS	228	227	100%
PVC	40	40	100%
PP	99	96	97%

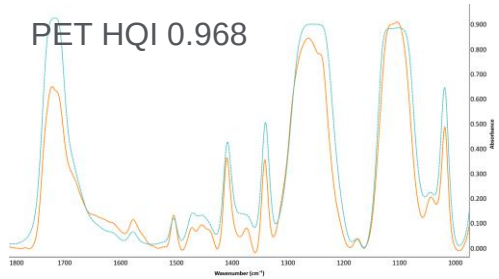
Aluminum-Coated Filters: Spectra quality

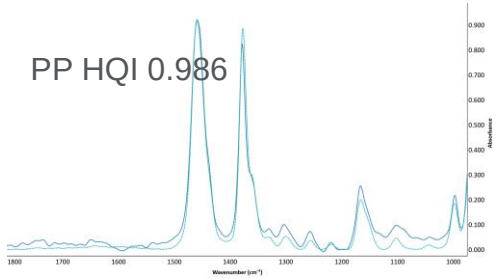
- Excellent acquisition spectra and matches using MPs Starter 2.0 Library. ✓

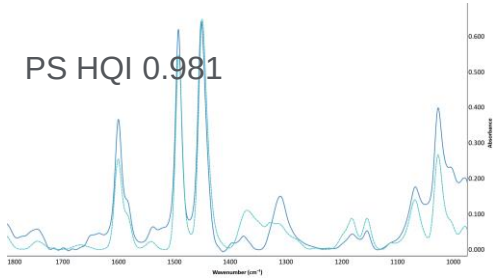


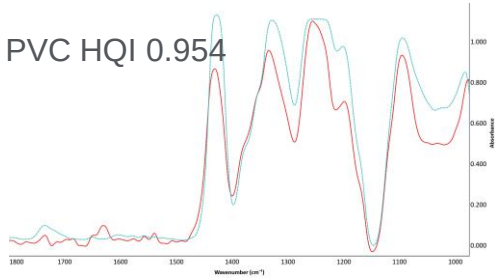


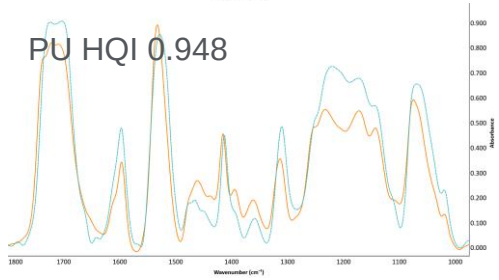


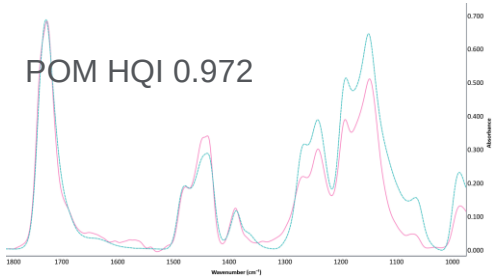


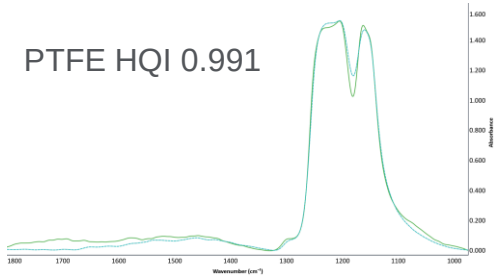


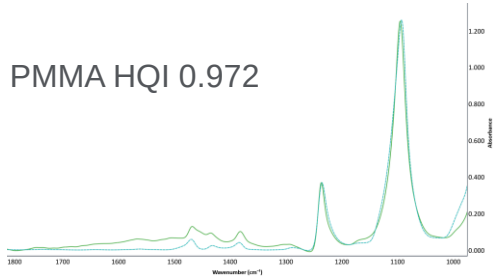




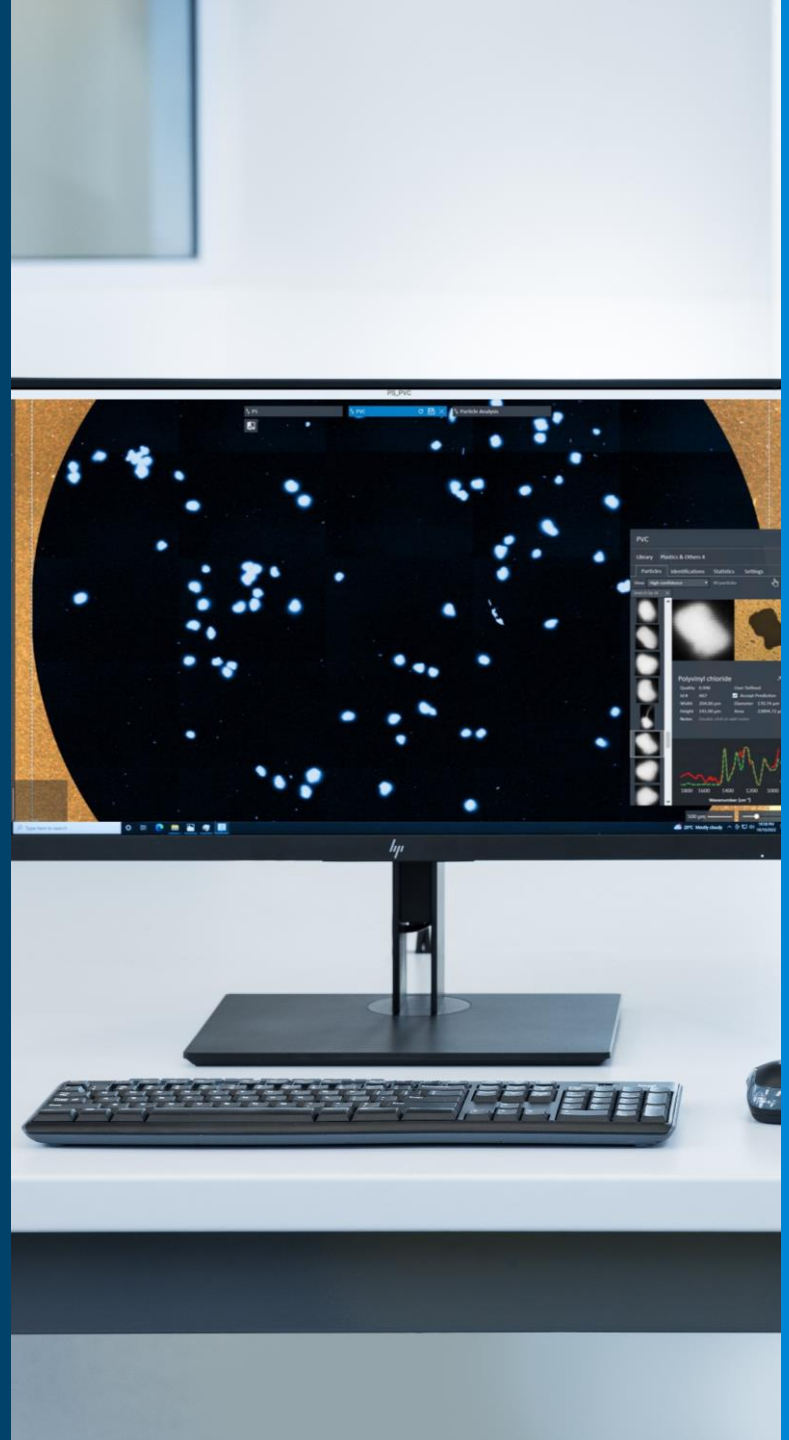








Clarity 1.6 Improvements



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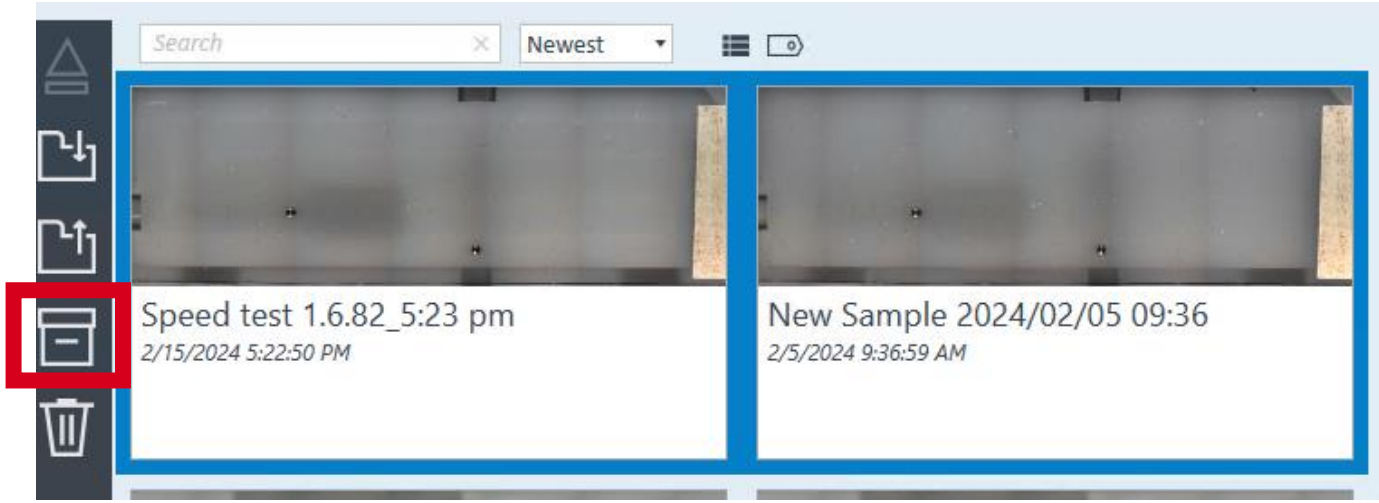
Clarity 1.6 Improvements: Speed of Analysis

System	PP04	LD23
Clarity	1.6.79	1.6.79
Database Status	6.68 GB	4.0 GB
Area Scanned	20 x 20 mm	60 x 20 mm
Number of Particles	6930	14847
Time to detect particles	25 mins	1 hour
High-mag image per particle	5 sec	6 sec
Spectrum per particle	4.4 sec	7.76 sec
Time per particle (Full characterization)	10 sec	13 sec

Improved speed of analysis and consistency between instrument

Clarity 1.6 Improvements: Data Archiving

- Archiving exports samples to a location of the user's choice and then deletes them from the Clarity database
  1. Select one or multiple samples
  2. Select data archiving button
  3. Choose location (PC, external hard drive, network)



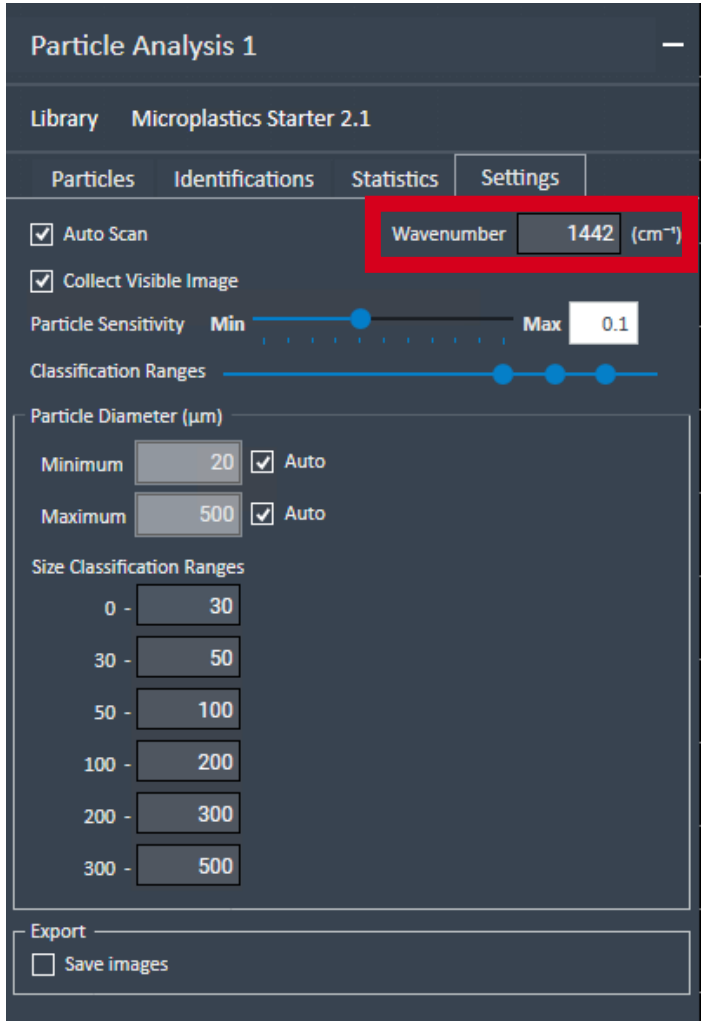
Ensuring that the Clarity data drive does not fill up.

Allows users to use their own secure storage solutions.

Clarity 1.6 Improvements: Wavenumber Selection

Clarity 1.5 - Image used to detect and outline particles is always scanned at 1442 cm^{-1}.

Clarity 1.6 - The user can input a custom wavenumber value between 975 and 1800 cm^{-1} for the particle detection image scan.



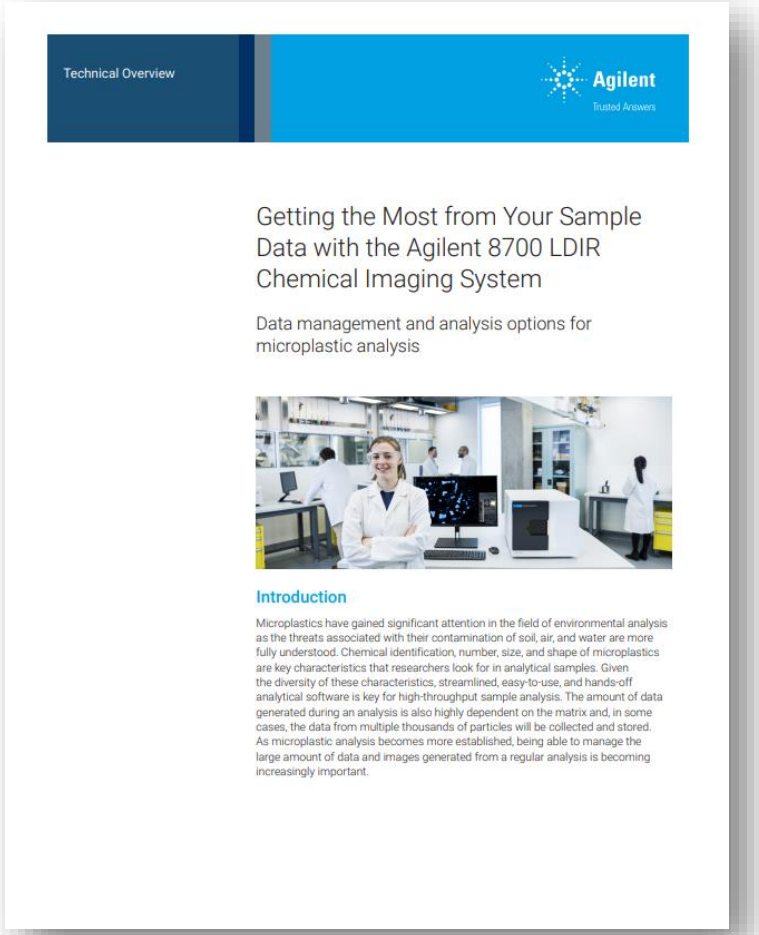
Improved reliability of the detection based on polymer of interest and user needs

Summary

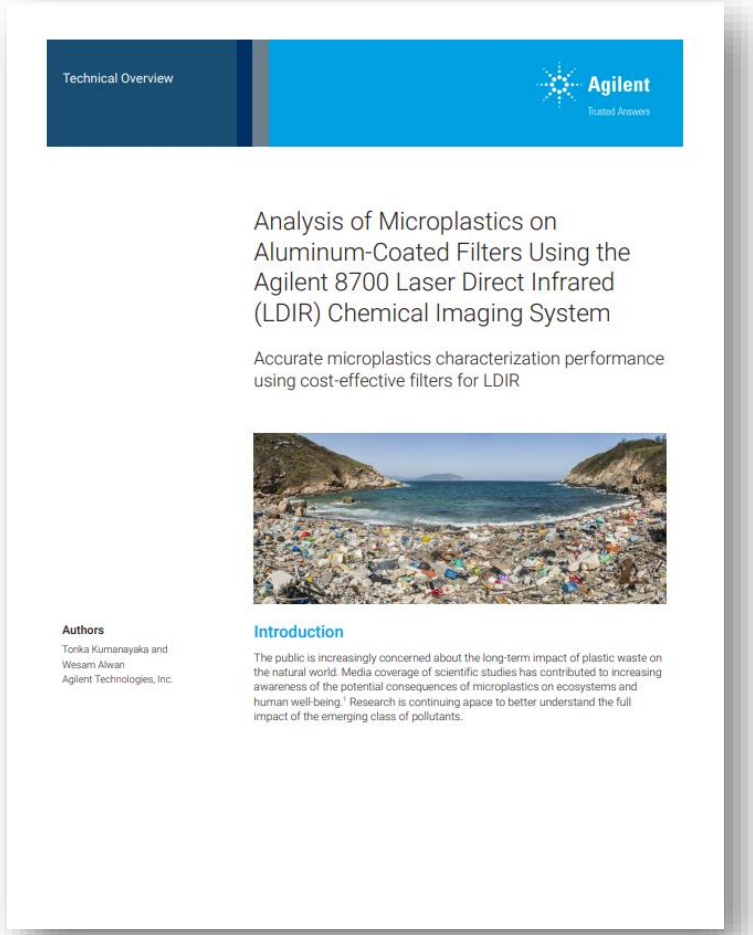
Agilent 8700 LDIR and Clarity 1.6

- Alu filter with same performance as gold at a 50% lower cost
- Overall analysis time reduced
- Easier data archiving and data management

New Resources: www.Agilent.com



[Getting the Most from Your Sample Data with the 8700 LDIR]

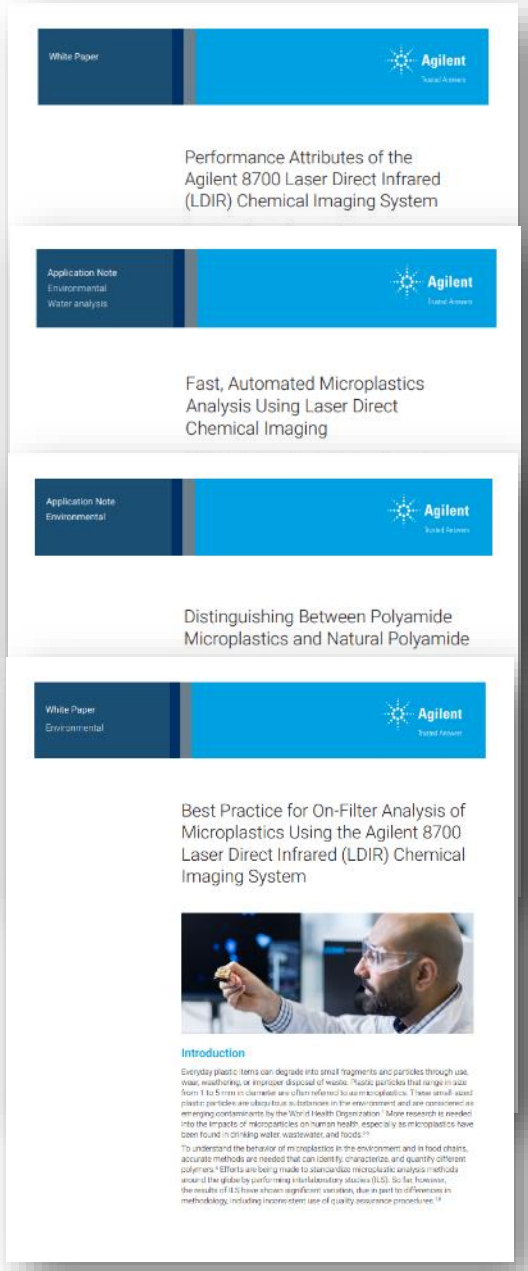


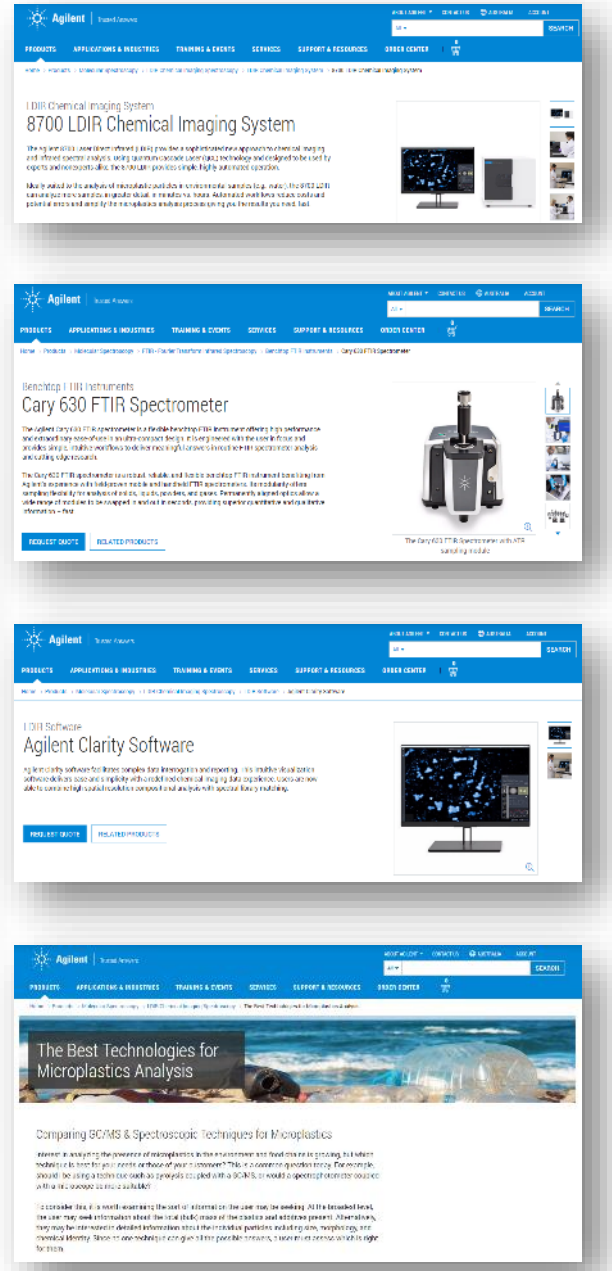
[Analysis of Microplastics on Aluminum-Coated Filters Using the 8700 LDIR]

[Collecting and Preparing Microplastics for Analysis by 8700 LDIR]

Other 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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