

Developments in QCL-Based Spectroscopy for Rapid Identification of Microplastics

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Agilent Technologies



Agilent 8700 LDIR Chemical Imaging System

Routine, robust, automated microplastics analysis by non-experts

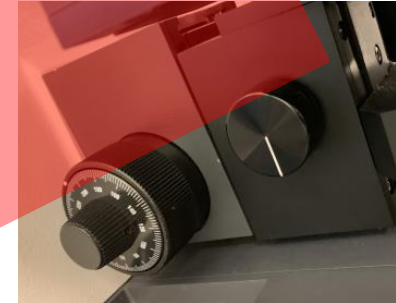
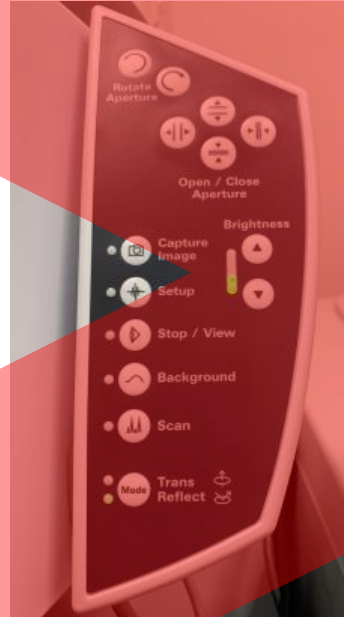
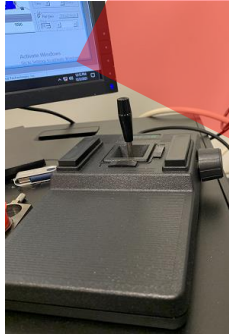


The Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system

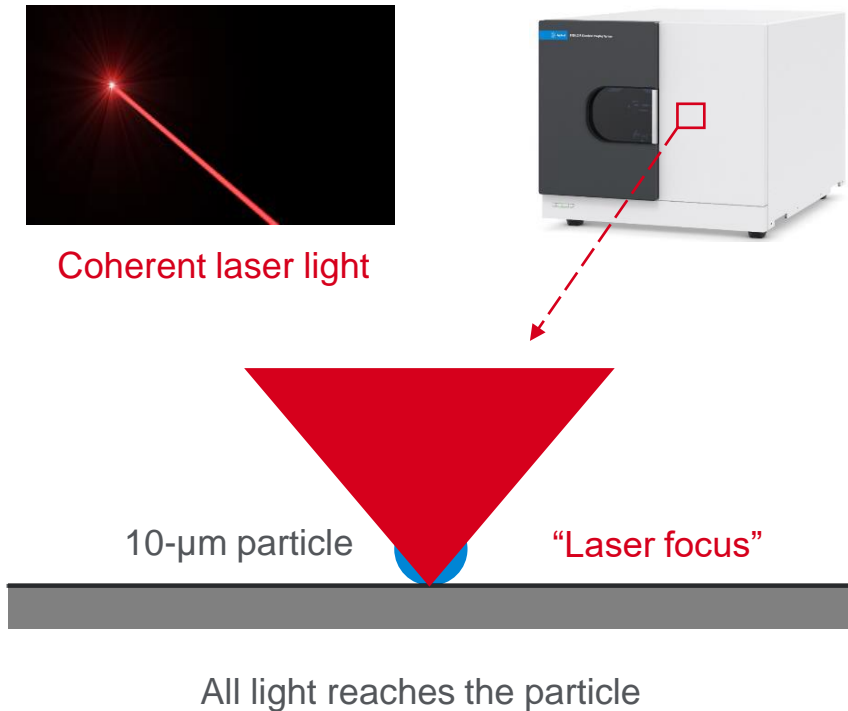
- **A sophisticated new approach to chemical imaging and spectral analysis.**
 - With the 8700 LDIR, you can analyze more samples, in greater detail, in less time
 - Uses Quantum Cascade Laser (QCL) technology & rapidly scanning optics to provide fast, clear, high-quality images and spectral data.
 - 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

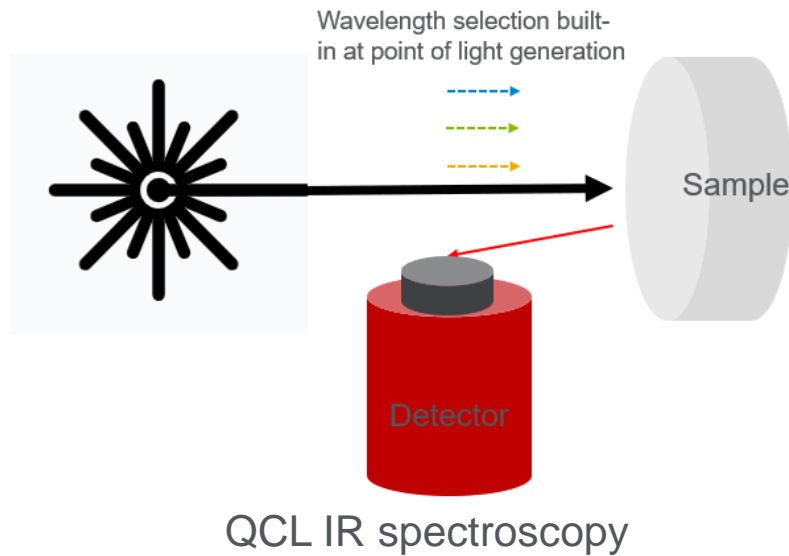


Laser Direct Infrared (LDIR) Spectroscopy



- 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



Modes of Action

Proprietary Agilent quantum cascade laser (QCL) technology

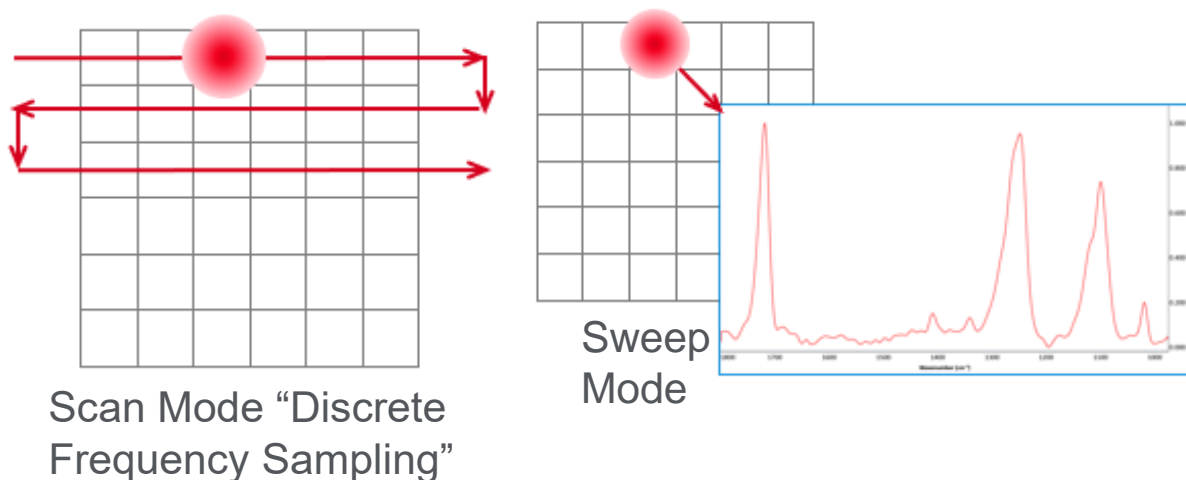
- Bright, coherent light source. More power, directional: Focus all laser power onto a particle
- Rapidly tunable across the mid-infrared for spectroscopy.

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

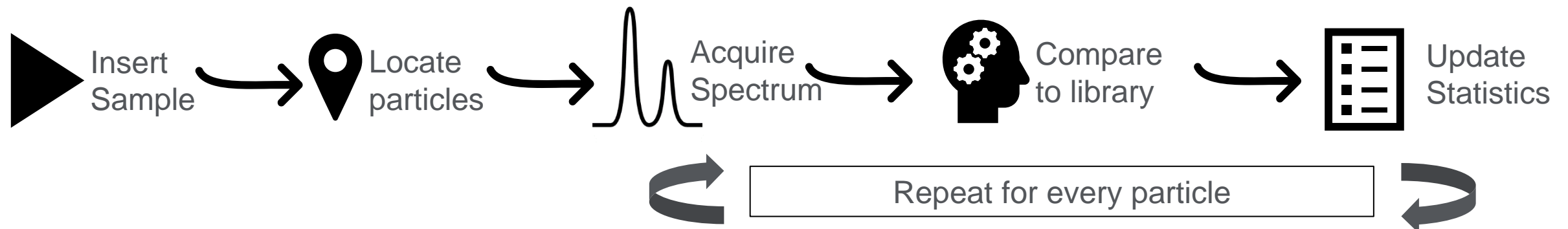
- Single Position
- Full Sweep available wavelengths
- Utilize full spectrum for library matching



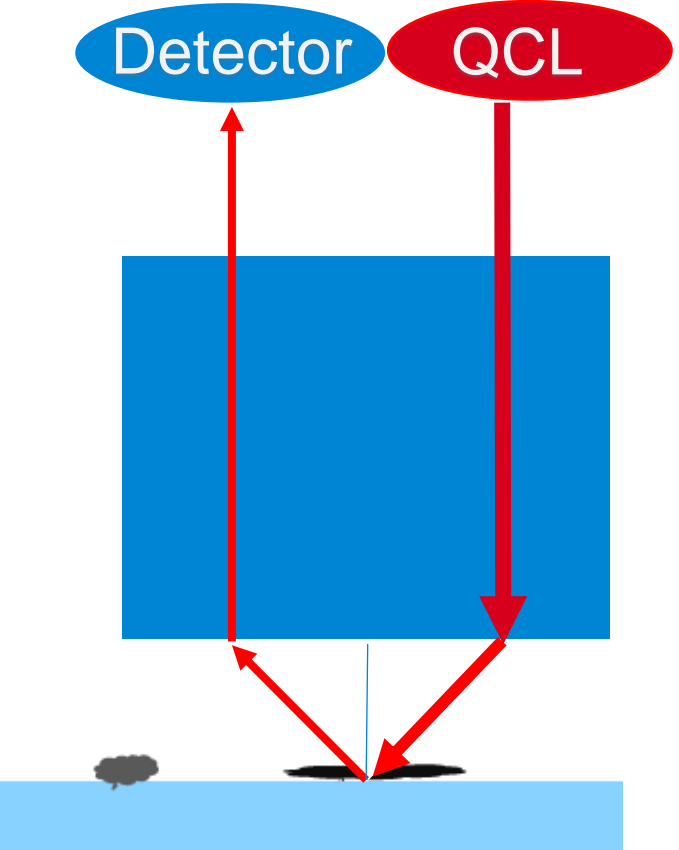
Agilent Solution: 8700 LDIR chemical imaging system



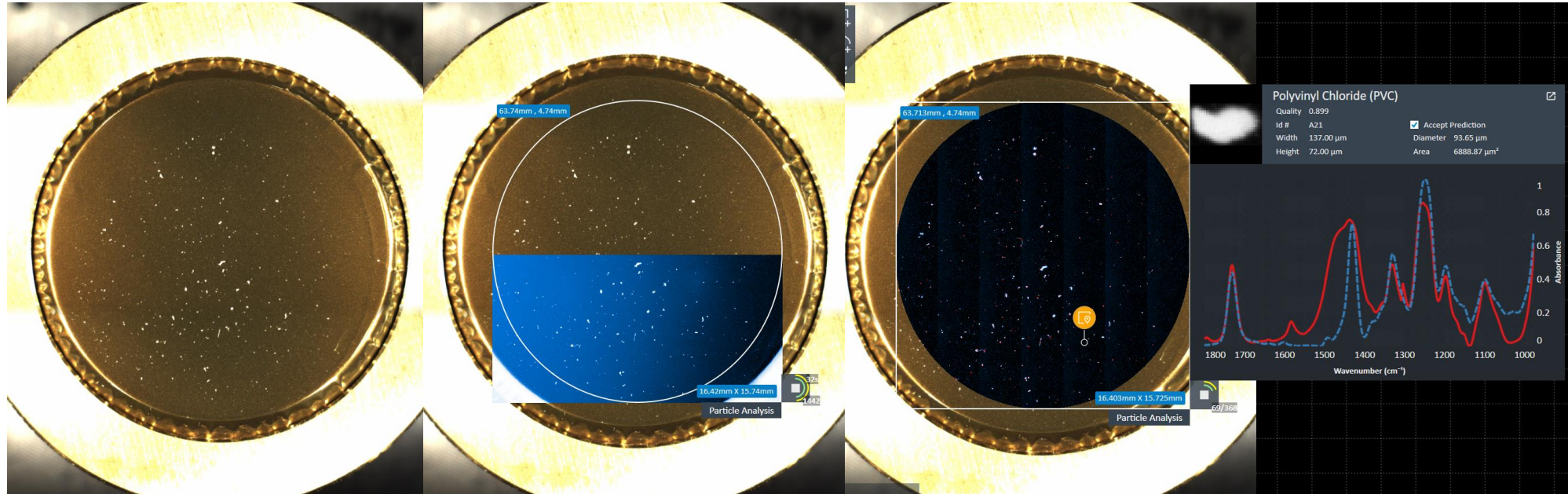
Analysis Workflow – 8700 LDIR + Clarity software



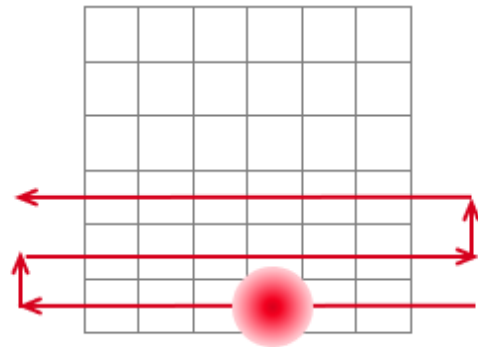
Obtain spectra by passing infrared beam thru sample reflecting off low-E slide or reflective filter (transflectance) and then back thru sample to the detector.



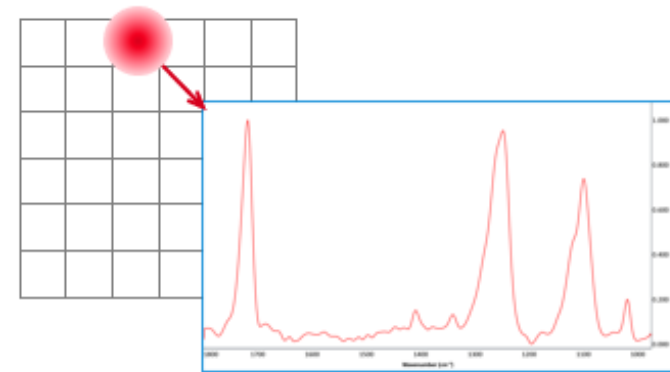
Data Collection



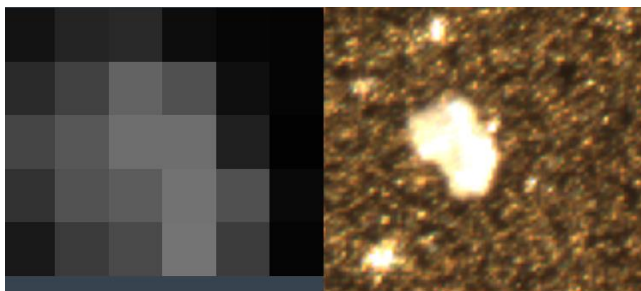
Visible Image



Discrete Frequency Mode



Sweep Mode



Natural Polyamide

Quality 0.795

Id # A6

Accept Prediction

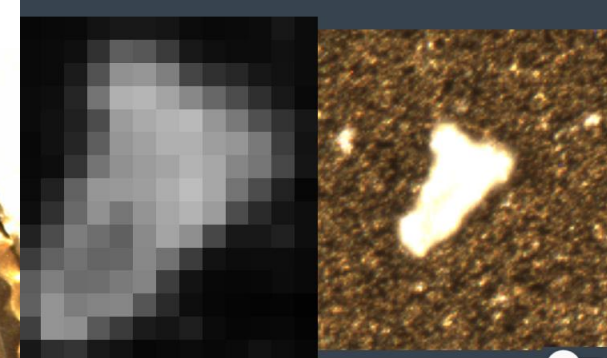
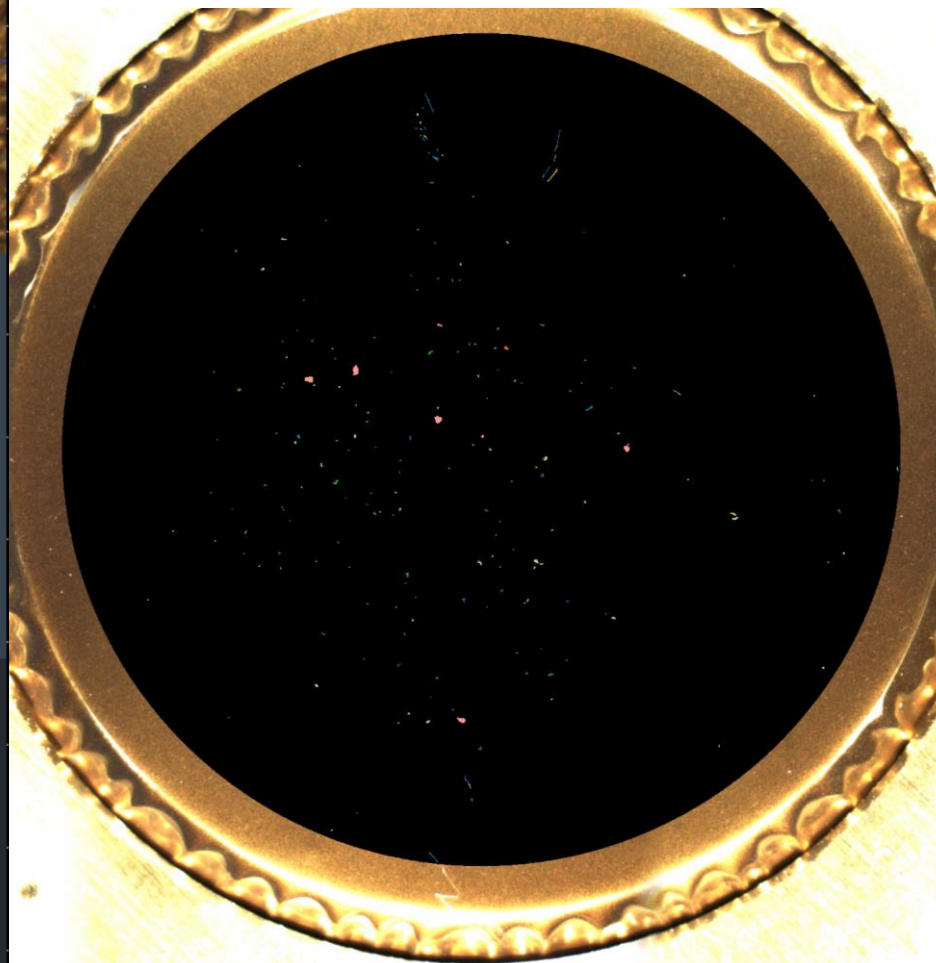
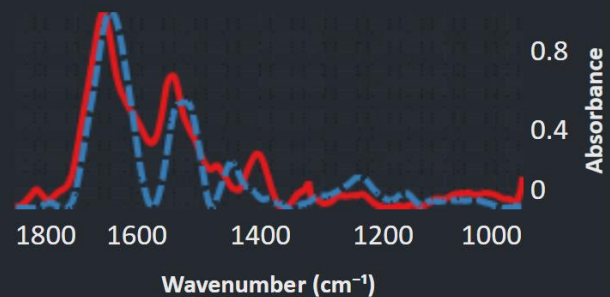
Width 29.00 μm

Diameter 24.34 μm

Height 22.00 μm

Area 465.21 μm^2

Notes *Double click to add notes*



Polyethylene

Quality 0.975

Id # A4

Accept Prediction

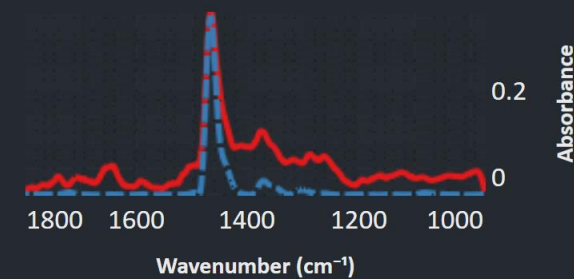
Width 39.00 μm

Diameter 44.82 μm

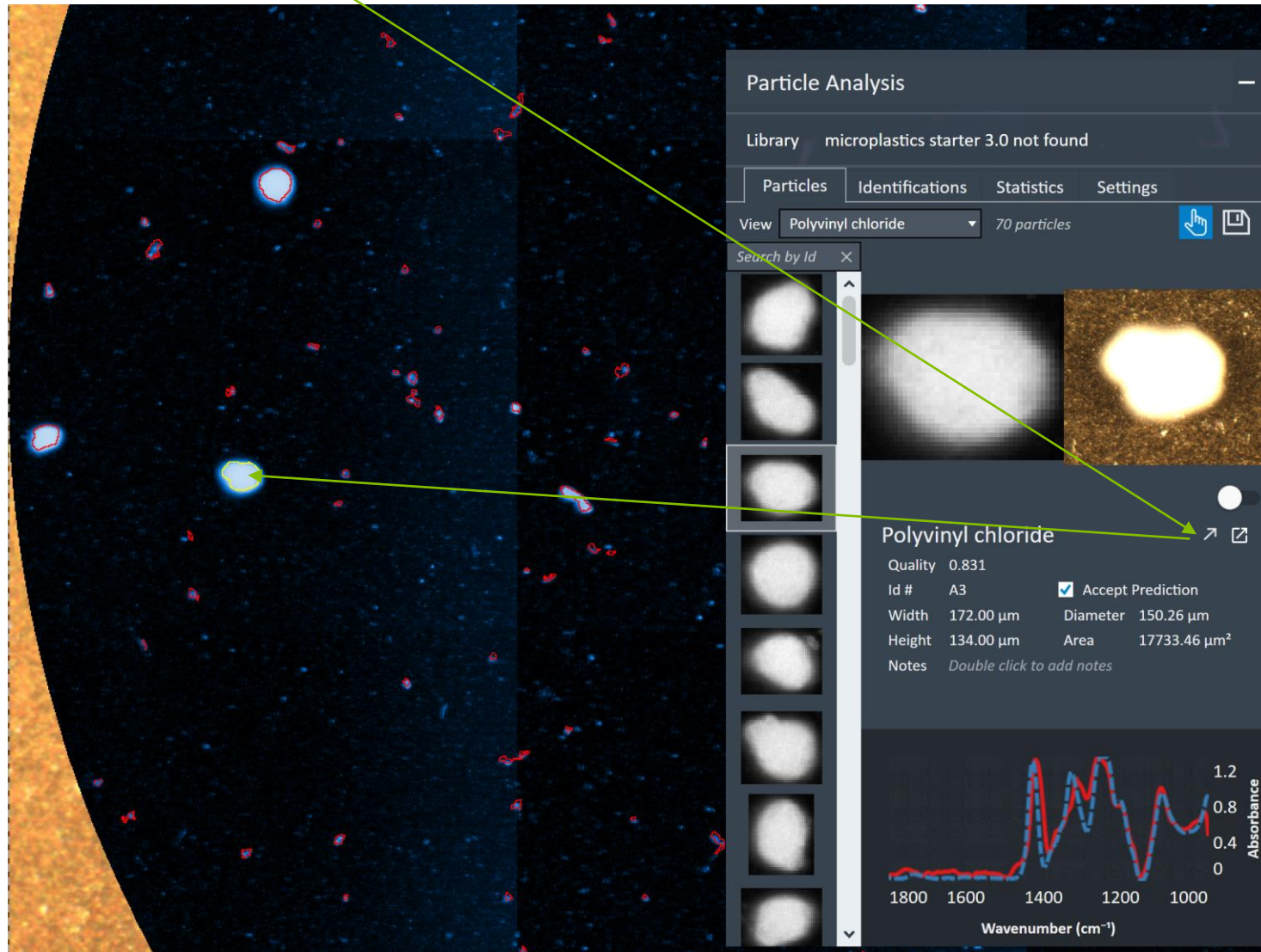
Height 64.00 μm

Area 1577.58 μm^2

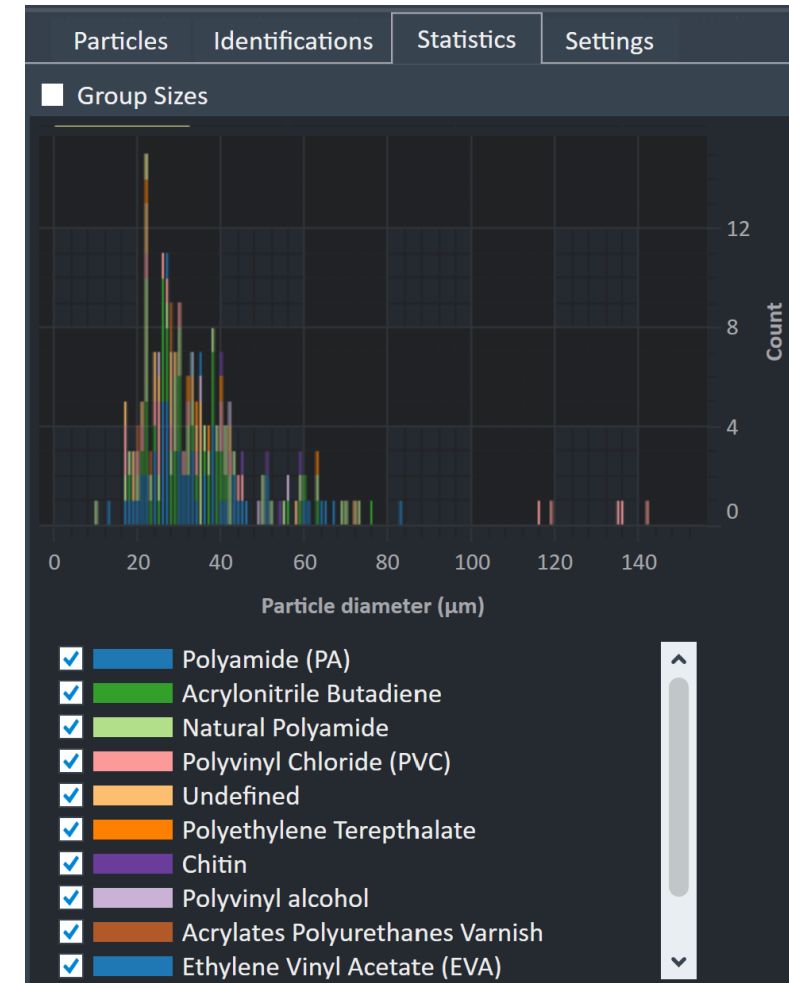
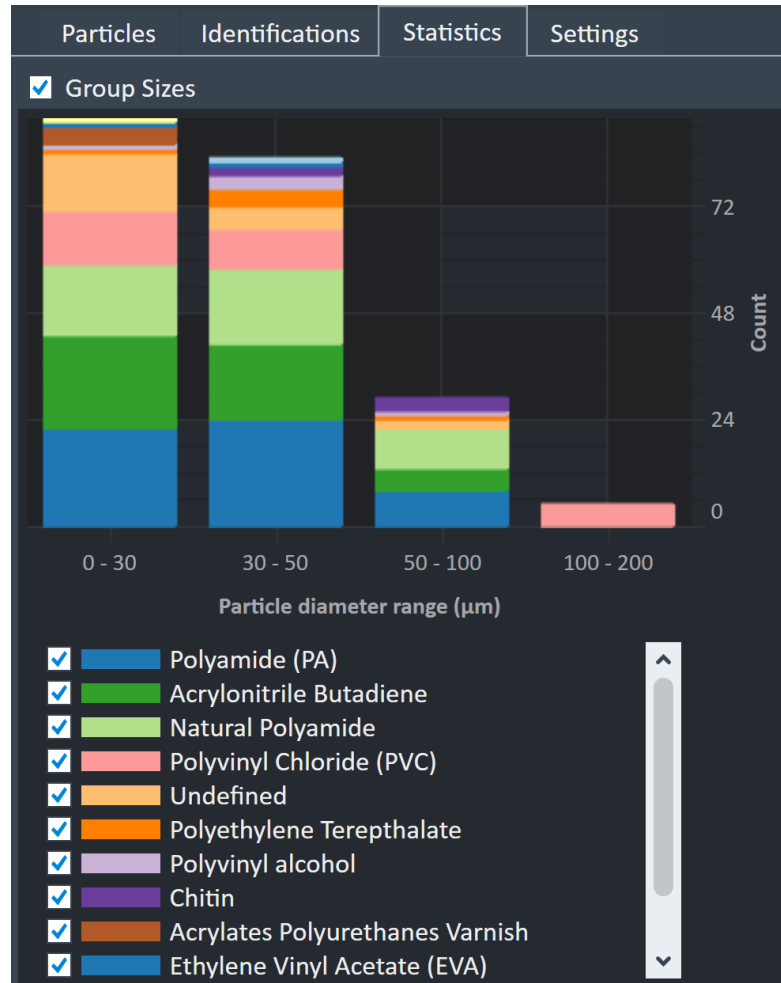
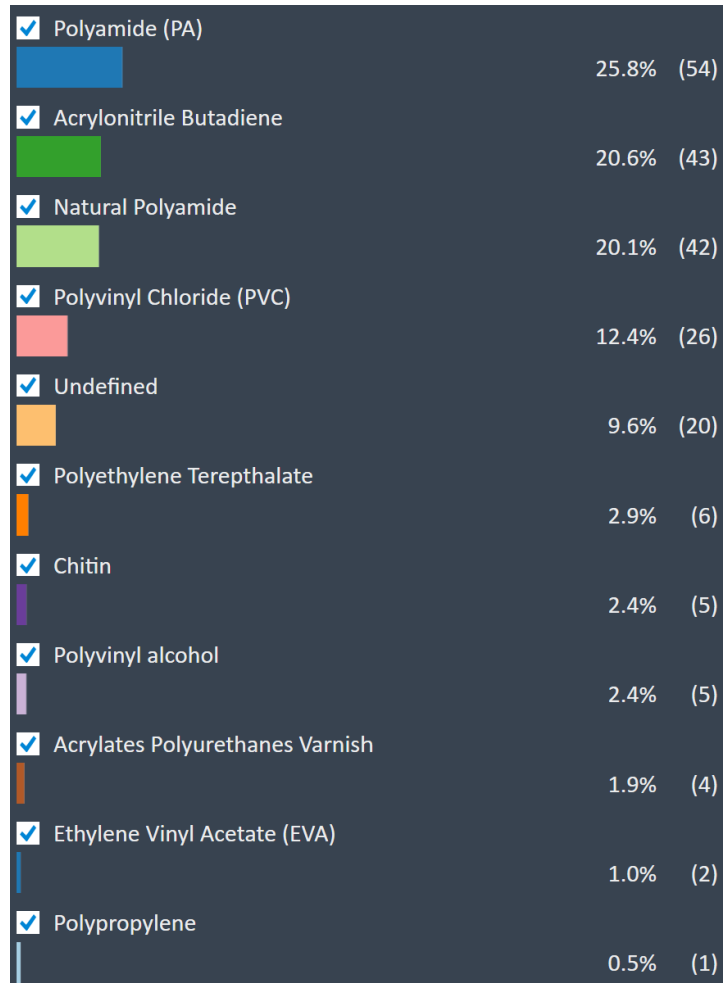
Notes *Double click to add notes*



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



Sample preparations

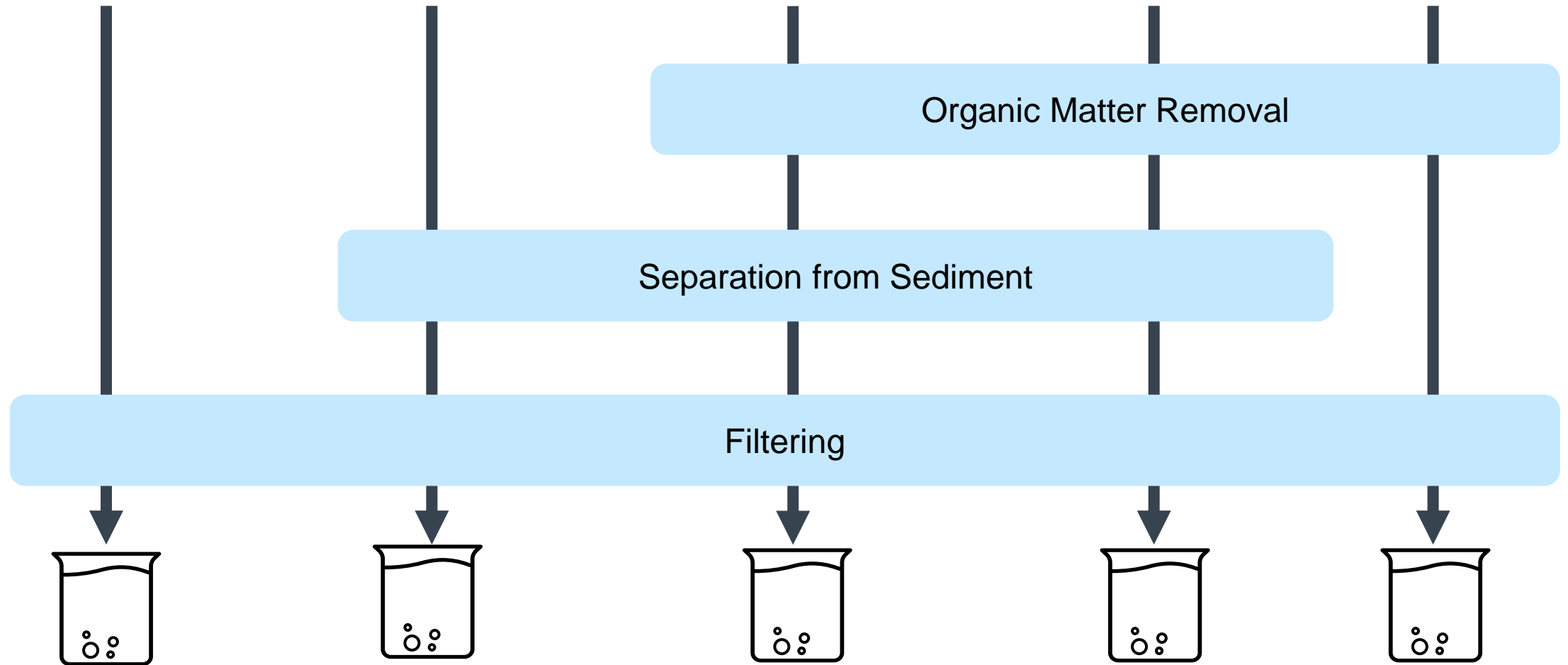
Clean water: drinking water

Clean sediment:
sand

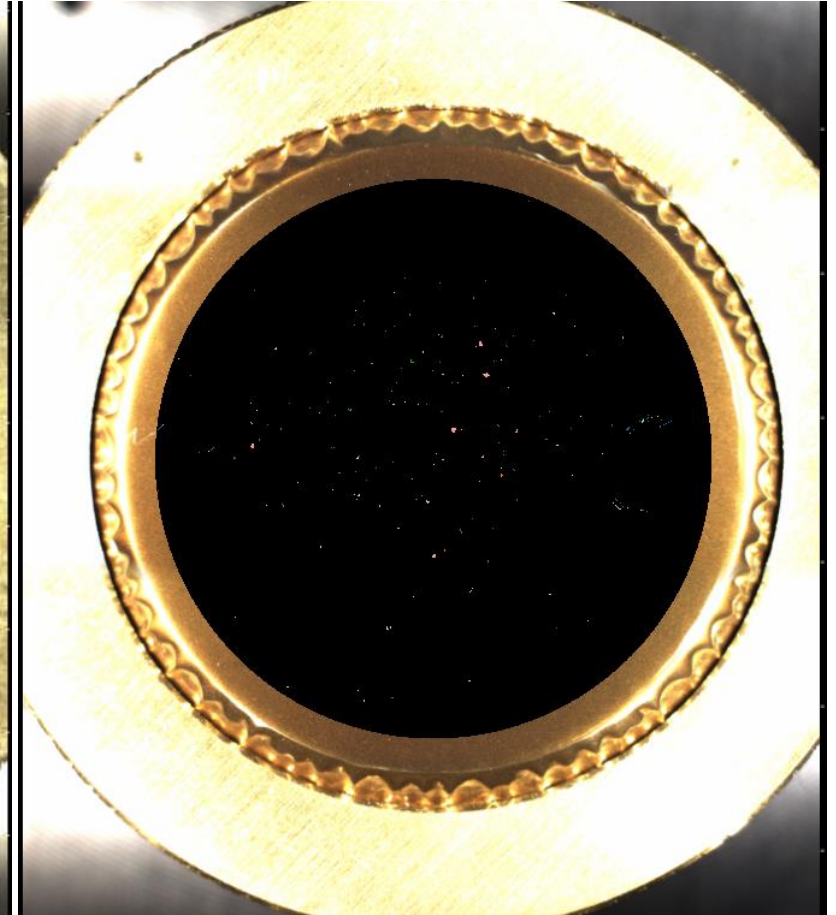
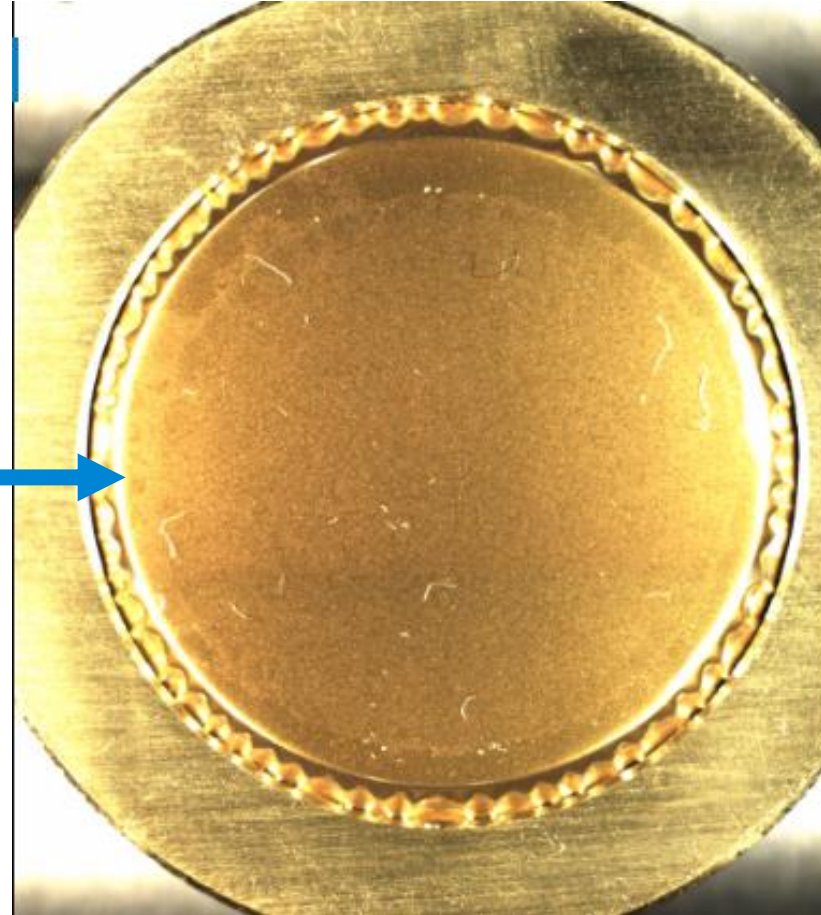
Dirty water: river
water

Dirty sediment:
soil

Biological



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



1 Place the filter using the supplied tweezers



2 Place the funnel



3 Secure the filtration assembly with the clamp



4 Filter the sample



5 Place the filter on top of the raised platform

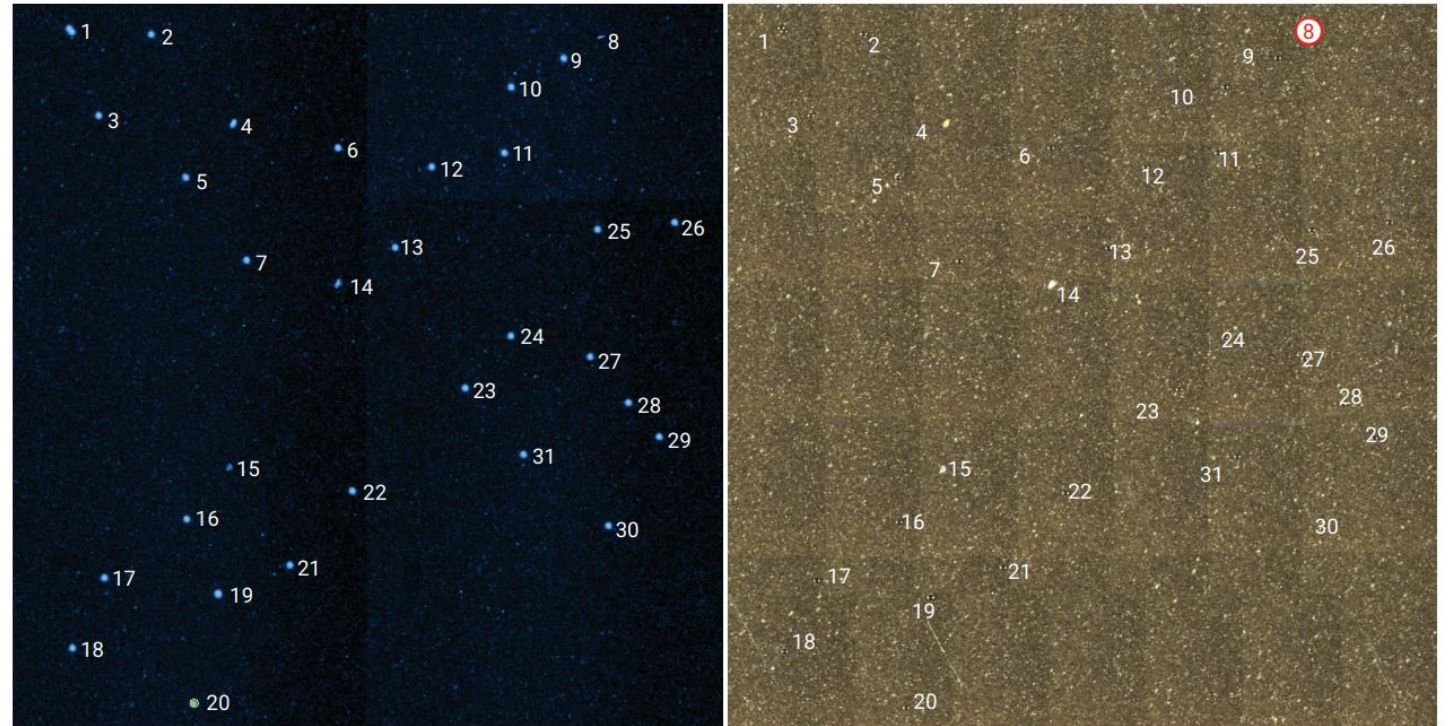


6 Thread the brass retaining ring

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. ✓



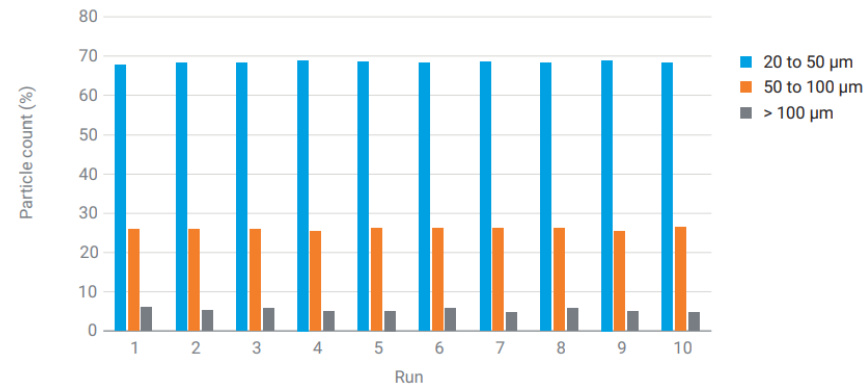
Shows the value of finding particles via IR imaging

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

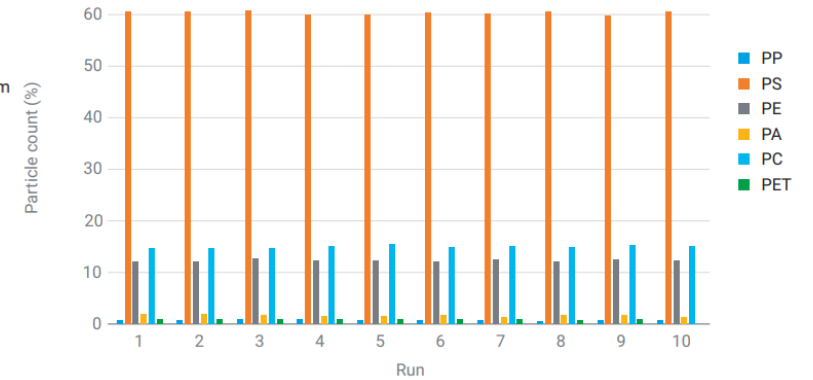
- Particle Analysis Workflow, same area (n = 10)
- < 1% variability based on total particles, size range and polymer identification. ✓
- Excellent reliability and accuracy. ✓



Total particle count



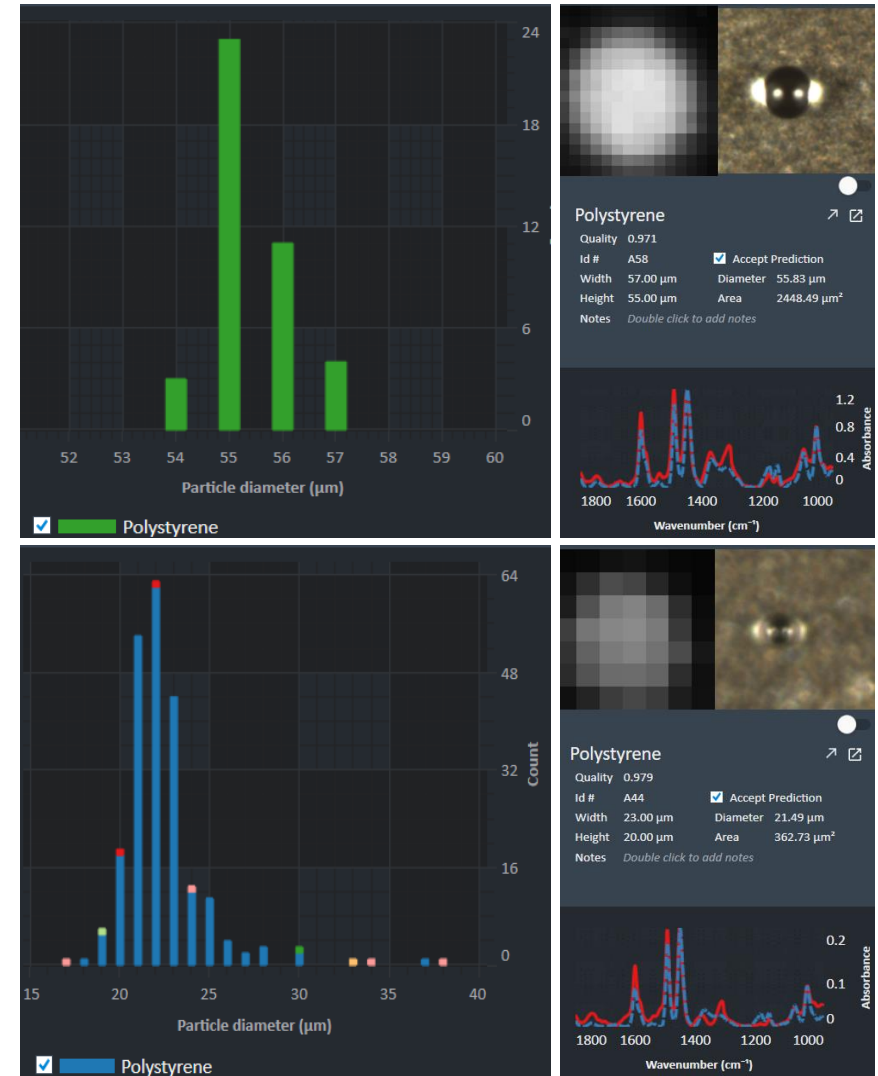
Size range



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. ✓



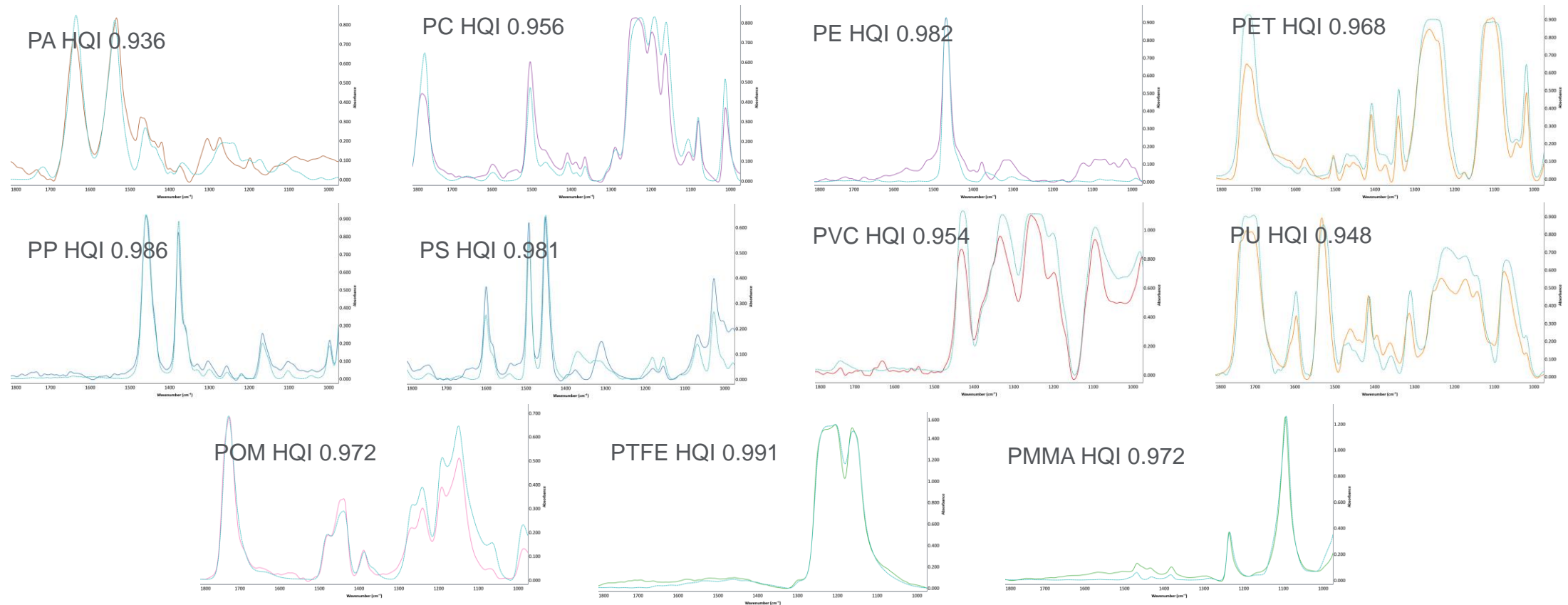
Aluminum-Coated Filters: Identification of common microplastics

- 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



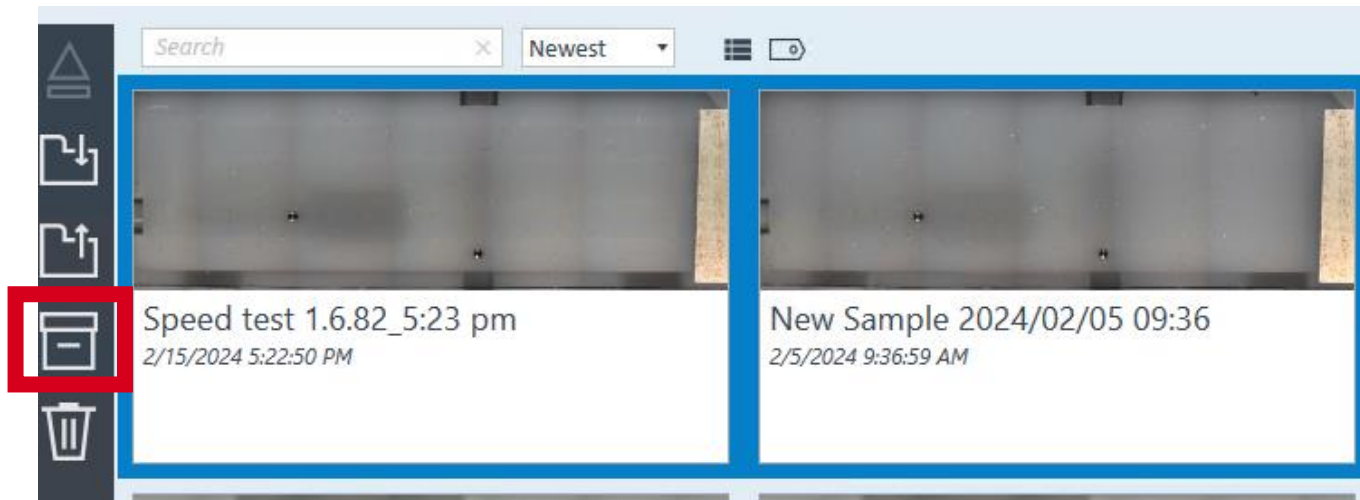
Clarity 1.6 Improvements: Speed of Analysis

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

Particle Analysis 1

Library Microplastics Starter 2.1

Particles Identifications Statistics Settings

Auto Scan Wavenumber 1442 (cm^{-1})

Collect Visible Image

Particle Sensitivity Min Max 0.1

Classification Ranges

Particle Diameter (μm)

Minimum 20 Auto

Maximum 500 Auto

Size Classification Ranges

0 - 30

30 - 50

50 - 100

100 - 200

200 - 300

300 - 500

Export

Save images

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

Technical Overview

Agilent
Trusted Answers

Getting the Most from Your Sample Data with the Agilent 8700 LDIR Chemical Imaging System

Data management and analysis options for microplastic analysis



Introduction

Microplastics have gained significant attention in the field of environmental analysis as the threats associated with their contamination of soil, air, and water are more fully understood. Chemical identification, number, size, and shape of microplastics are key characteristics that researchers look for in analytical samples. Given the diversity of these characteristics, streamlined, easy-to-use, and hands-off analytical software is key for high-throughput sample analysis. The amount of data generated during an analysis is also highly dependent on the matrix and, in some cases, the data from multiple thousands of particles will be collected and stored. As microplastic analysis becomes more established, being able to manage the large amount of data and images generated from a regular analysis is becoming increasingly important.


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

Technical Overview

Agilent
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Analysis of Microplastics on Aluminum-Coated Filters Using the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System

Accurate microplastics characterization performance using cost-effective filters for LDIR



Introduction

The public is increasingly concerned about the long-term impact of plastic waste on the natural world. Media coverage of scientific studies has contributed to increasing awareness of the potential consequences of microplastics on ecosystems and human well-being.¹ Research is continuing apace to better understand the full impact of the emerging class of pollutants.

Authors
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[Analysis of Microplastics on Aluminum-Coated Filters Using the 8700 LDIR](#)

Technical Overview

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Collecting and Preparing Microplastics for Analysis by the Agilent 8700 LDIR

Applying the sample preparation methodology specified in ASTM D8333 and SCCWPR



Authors
Louis Tisinger, Darren Robey,
and Wesam Alwan
Agilent Technologies, Inc.

Introduction

The success of any spectroscopic analysis of microplastic particles depends on the effectiveness of the sample preparation procedure. Inadequate or improper sample preparation can introduce errors into the analysis, leading to unreliable data. Whether using infrared (IR) or Raman microscopy, microplastic particles need to be isolated so they can be discretely distributed on a substrate ready for analysis. The more complex the matrix, the greater the amount of preparation will be required. Standardization of sample preparation methodology is also important to enable the comparison of results from different studies.

[Collecting and Preparing Microplastics for Analysis by 8700 LDIR](#)



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