# Automated Analysis of Microplastics Using a Laser-Based Analyzer

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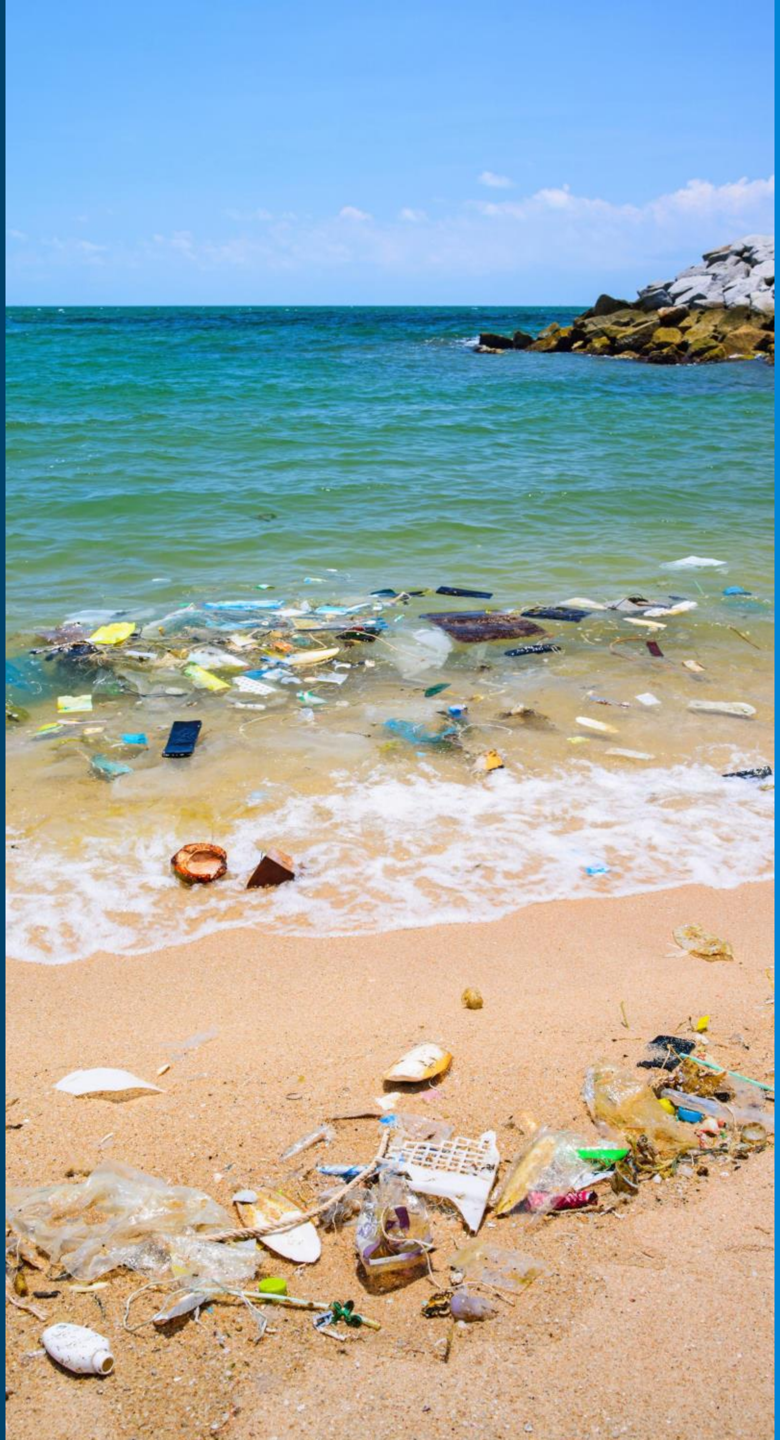

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Trusted Answers

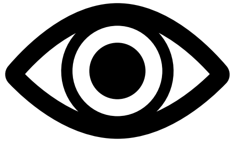


DE53342247

# What are microplastics?

- Small particles 1µm to 5000µm.
- Area of interest 300 µm – 10 µm (and smaller)
- Small plastics can translocate inside the tissue of organisms
- The smaller the particle, the higher the risk
- Microplastics can be vectors for various pollutants
- No established standard methods (yet!)
- Various techniques depending on size

# Options for Microplastics Analysis



### Manual microscopy, by-eye approach, needle test etc.

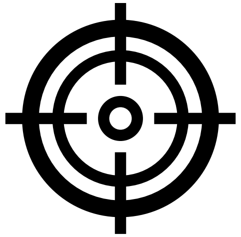
- Inexpensive
- Simple

- Inaccurate / subjective
- Unlikely to determine chemical identity

### Wet chemistry & related methods, e.g., GC/MS + Pyrolysis

- Accurate measurement of mass

- Physically intensive and time-consuming
- Difficult for small or individual particles
- Destructive

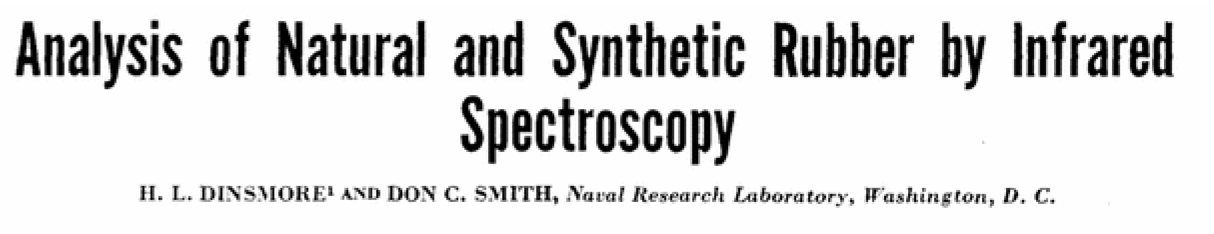


### Molecular Spectroscopic methods

- Chemically-specific (get particle identities)
- Non-destructive (leave the door open to further analysis)
- Can be highly automated

- High sample prep requirement

## 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.*

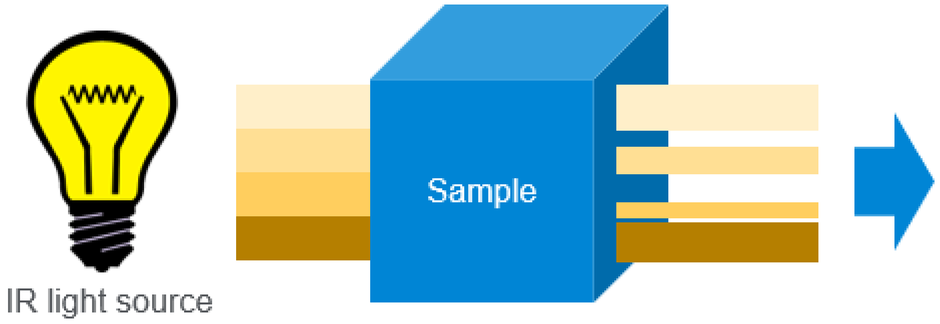
Analytical Chemistry - 1948

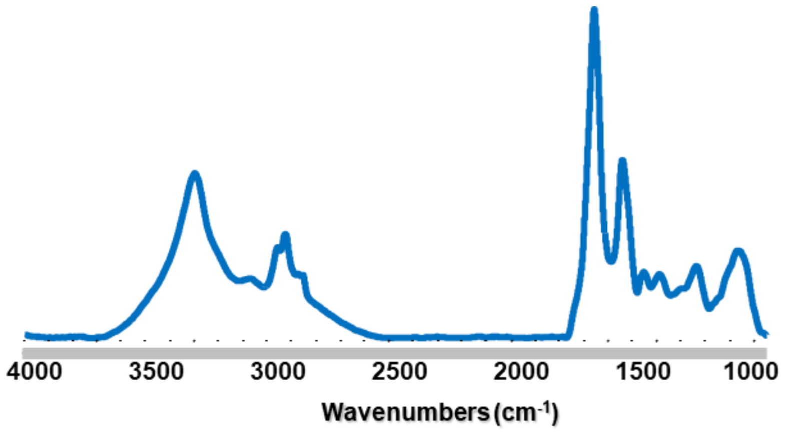
"*Fourier Transform Infrared Spectroscopy (FT—IR) is developing as a ubiquitous tool for use in the characterization of polymers*".

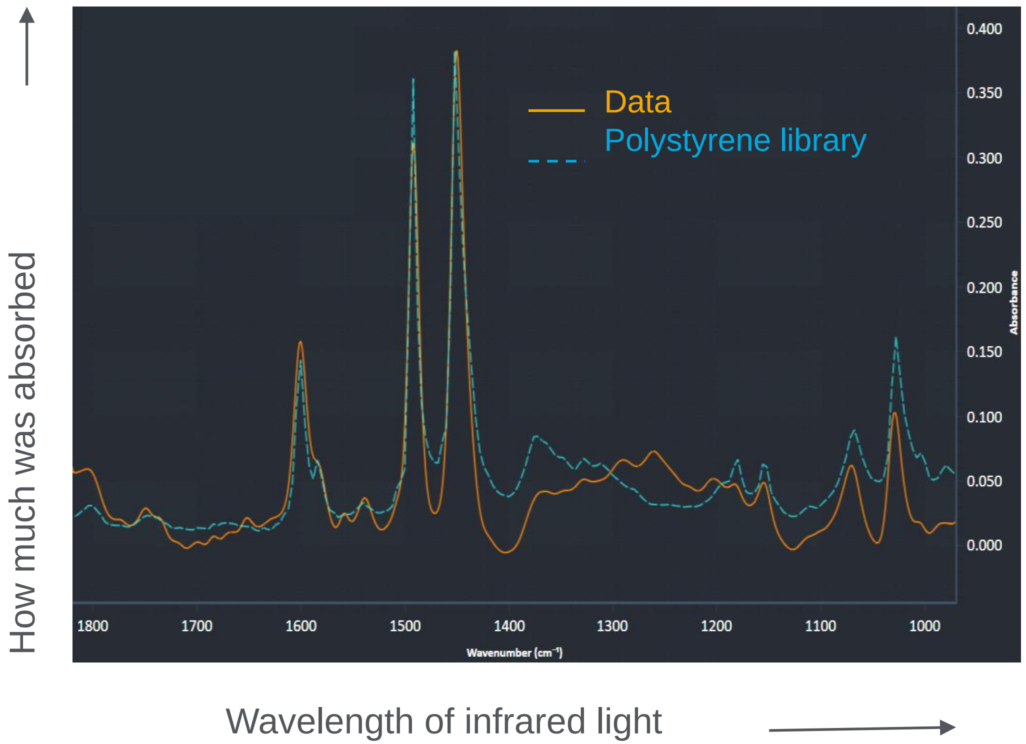
Jack L. Koenig -1985

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

# Infrared spectroscopy

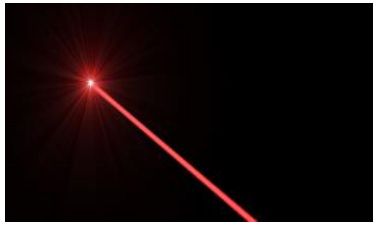


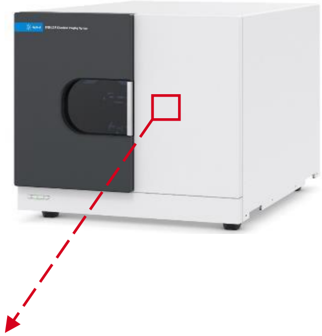



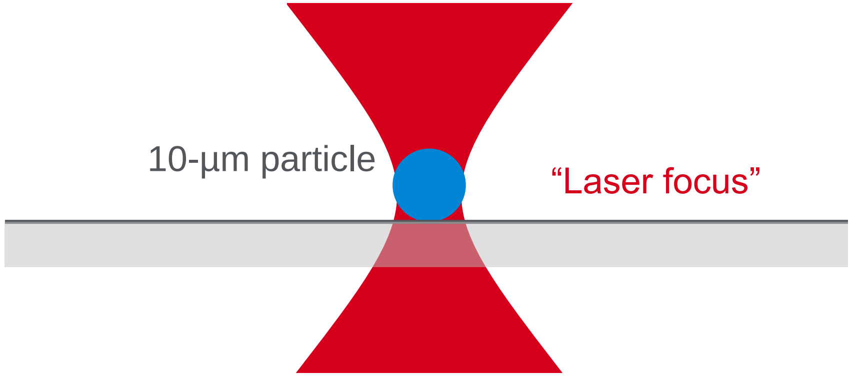

- direct infrared light on a particle and see which wavelengths it absorbs
- The IR spectrum of a sample is a plot of the amount of IR energy (y-axis) that is absorbed at frequencies (x-axis) in IR the region of the electromagnetic spectrum
- Every sample has a unique IR spectrum; an IR spectrum can serve as a compound’s fingerprint.
- Compare spectrum to library for match

# Laser Direct Infrared (LDIR) Spectroscopy



Coherent laser light

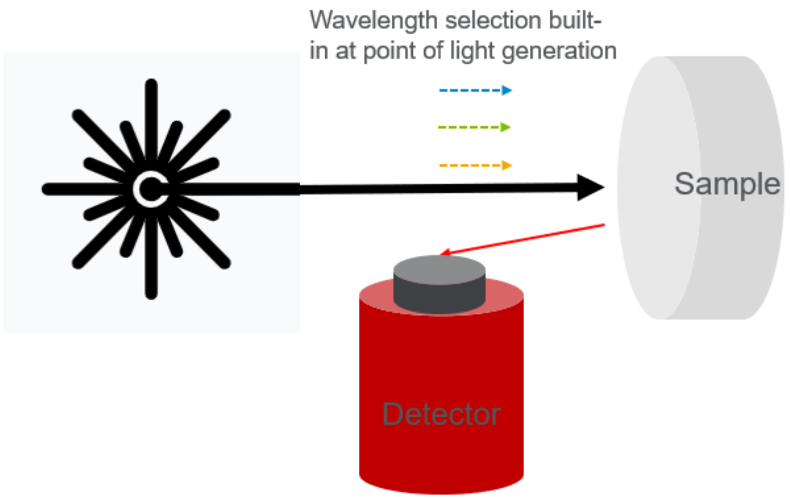




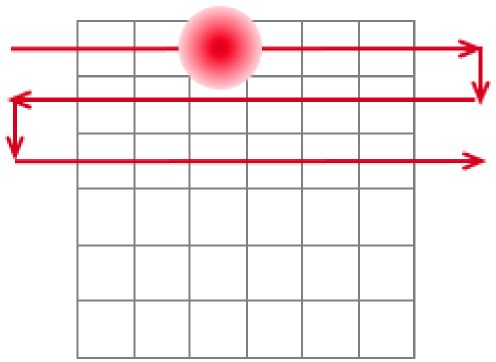

All light reaches the particle

- 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

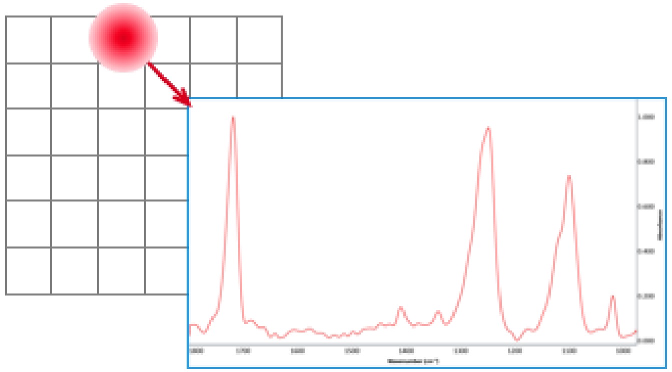
# Application Note: Analyzing Microplastics using Agilent 8700 LDIR




QCL IR spectroscopy



Scan Mode



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.

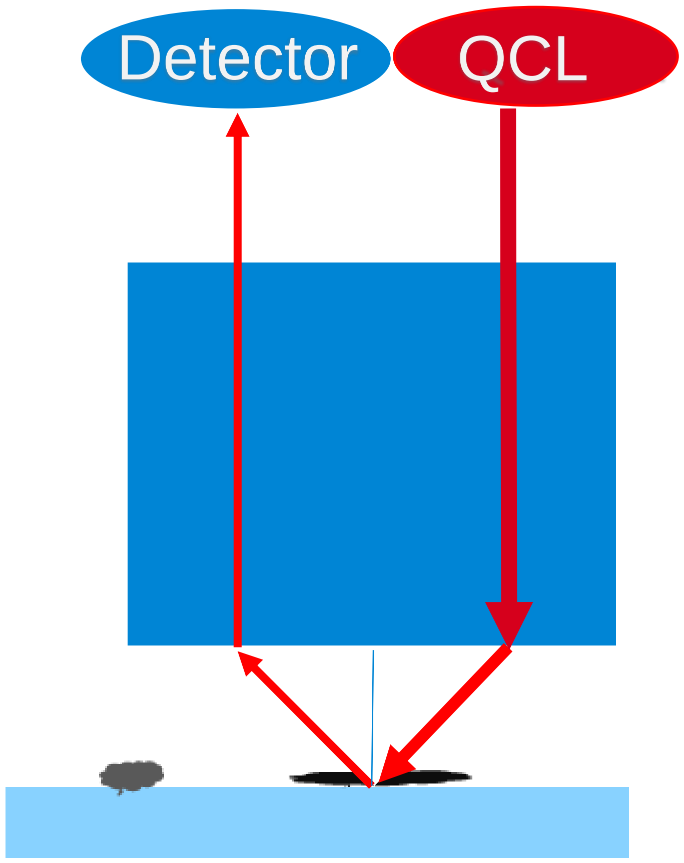
Scan Mode

- 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

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



# Automated Operation

| Feature | Benefit |
|---|---|
| No LN2 | LN2 is expensive and requires special handling |
| Auto backgrounding | New analysis (optics, etc.) would normally require new (manual) background acquisition |
| Sample loading / positioning | No getting lost driving with a joystick, peering through oculars |
| Auto focus / detect illegal moves | No chance of smashing into your specimen |

# Traditional FTIR-based systems: More Manual Tasks

# Sample preparation

Clean water: drinking water

Clean sediment: sand

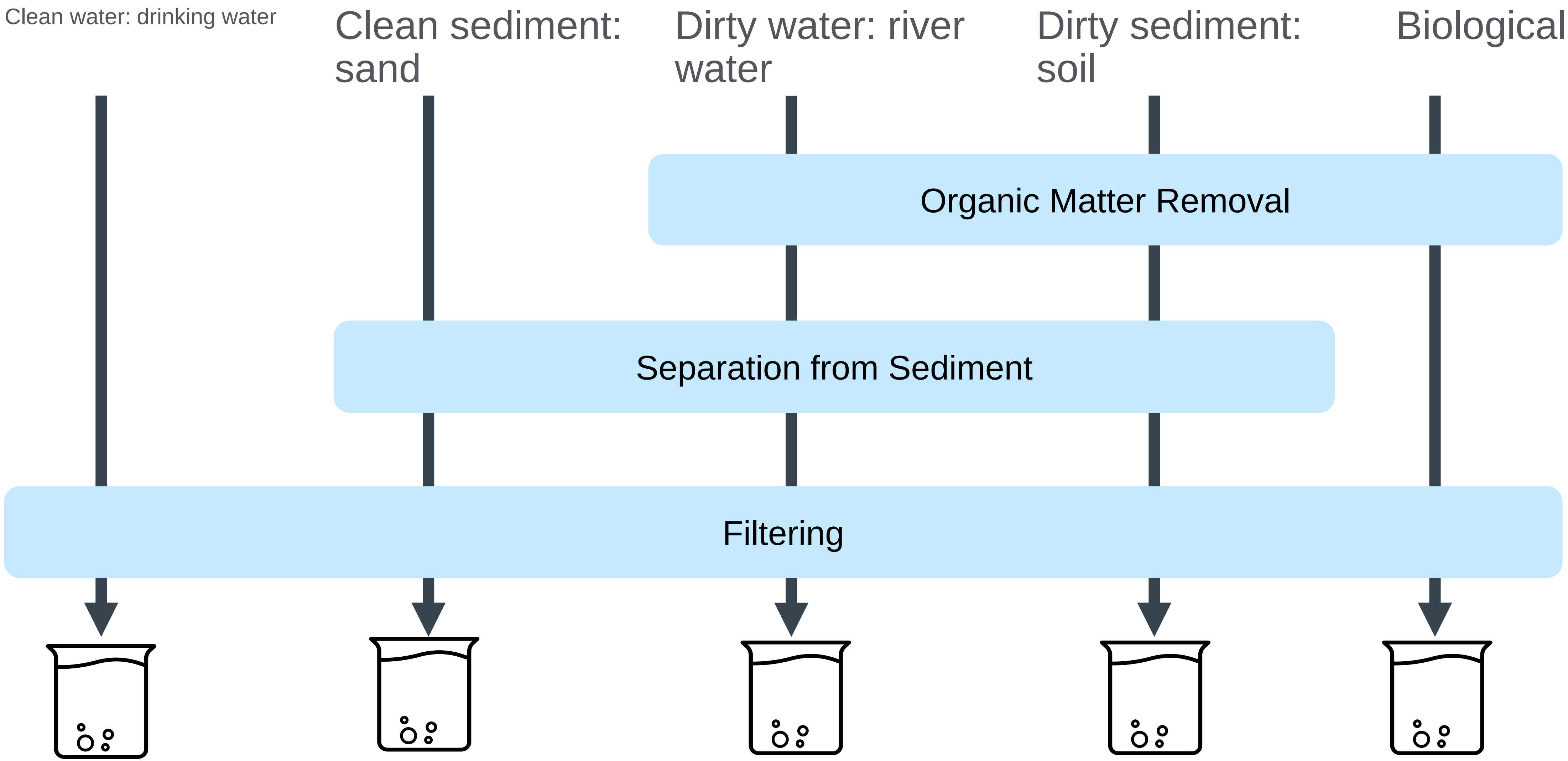
Dirty water: river water

Dirty sediment: soil

Biota

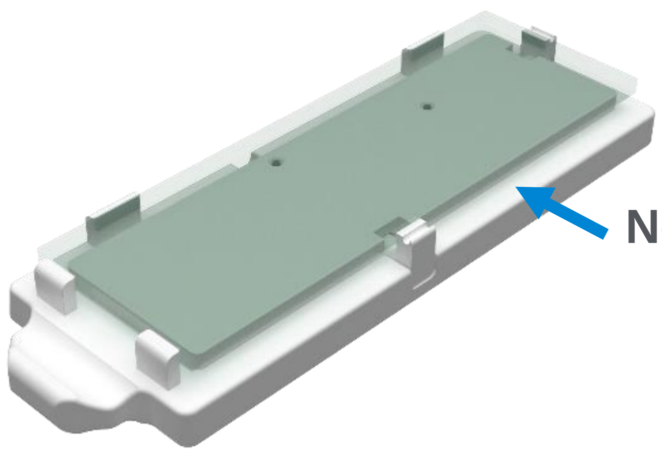
Complexity of sample preparation

# Sample preparations



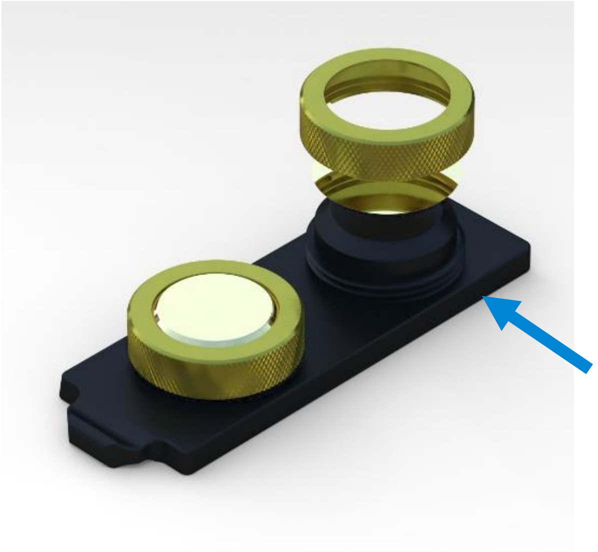
# Particle Analysis on different substrates

## Low-e Slide Analysis




- ✓ Characterizing large number of particles
- ✓ Minimal interference from the analyst.
- ✓ Cheap

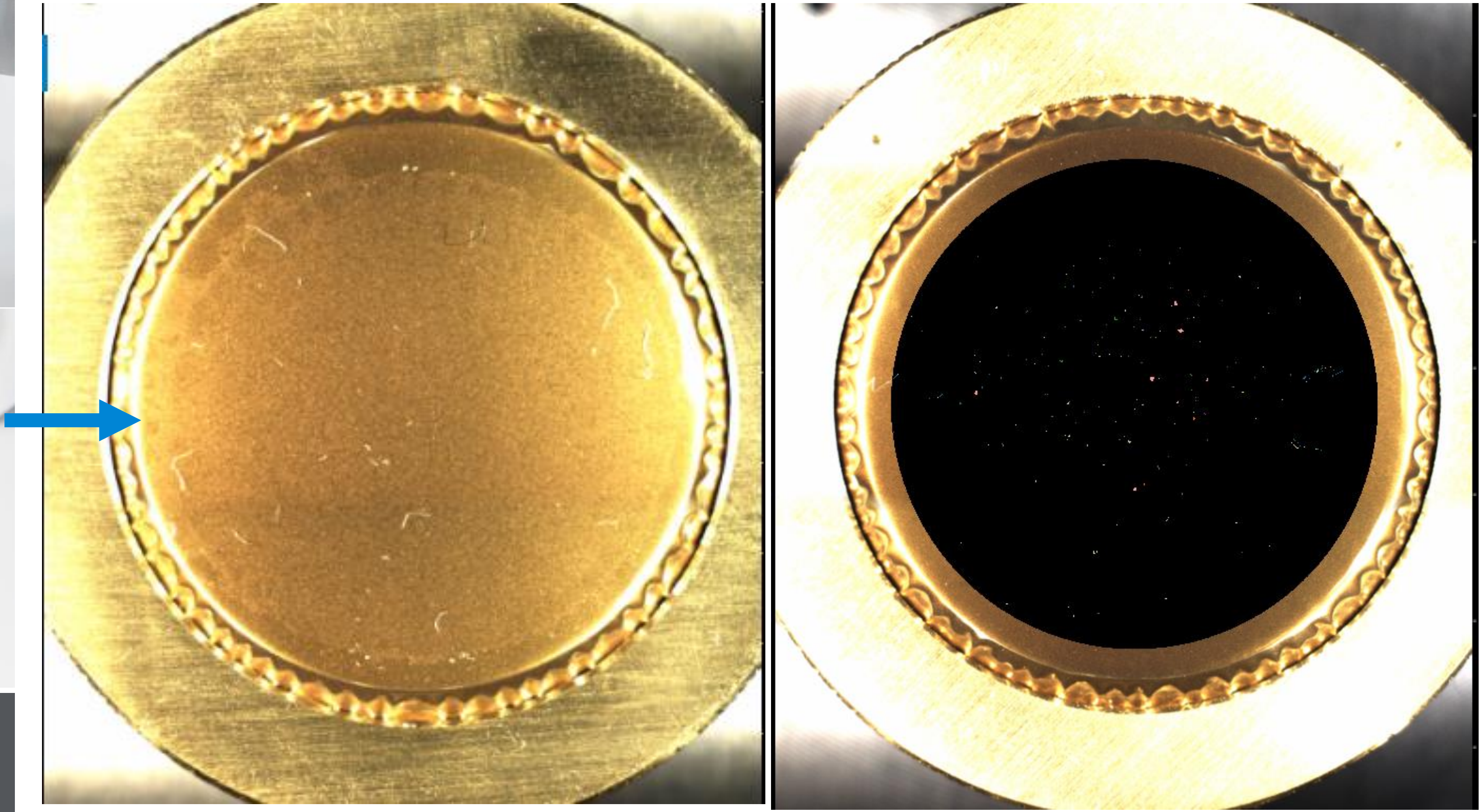
## On-Filter Analysis




- ✓ Less laborious
- ✓ Reduce the potential for contamination
- ✓ Analyzing 2 samples – time saving
- ✓ Broad sample matrices

# Direct microplastics using gold-coated filters

- Minimal sample preparation
- Reduced contamination
- Improved sample representation



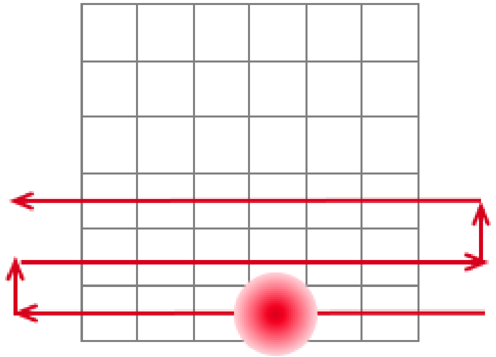
## Agilent solution: 8700 LDIR chemical imaging system

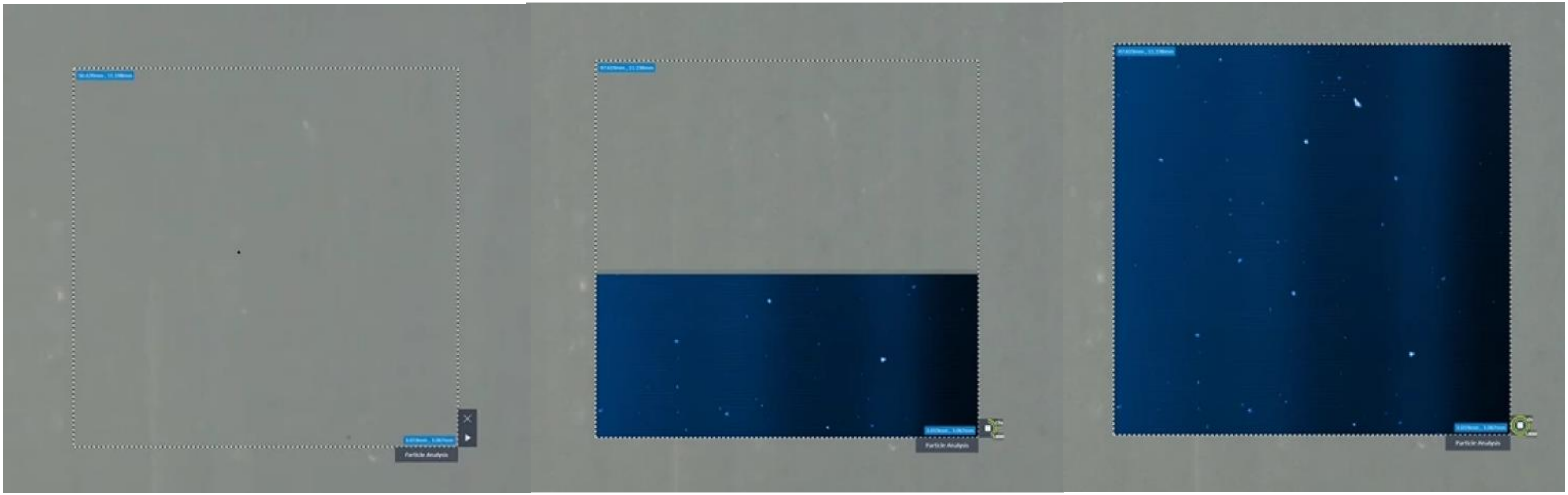
### Analysis Workflow – 8700 LDIR + Clarity software

Insert Sample → Locate particles → Acquire Spectrum → Compare to library → Update Statistics

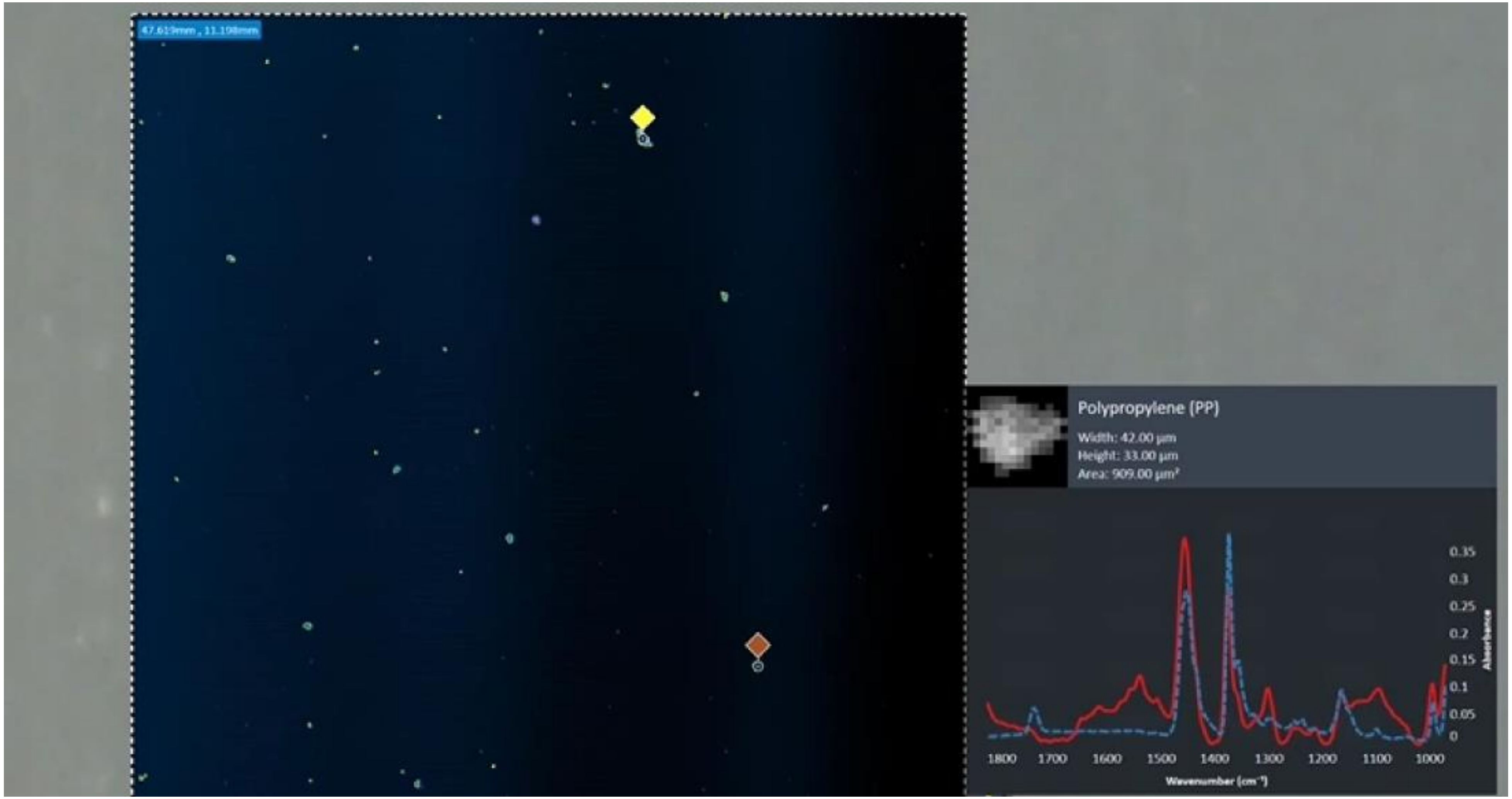
Repeat for every particle

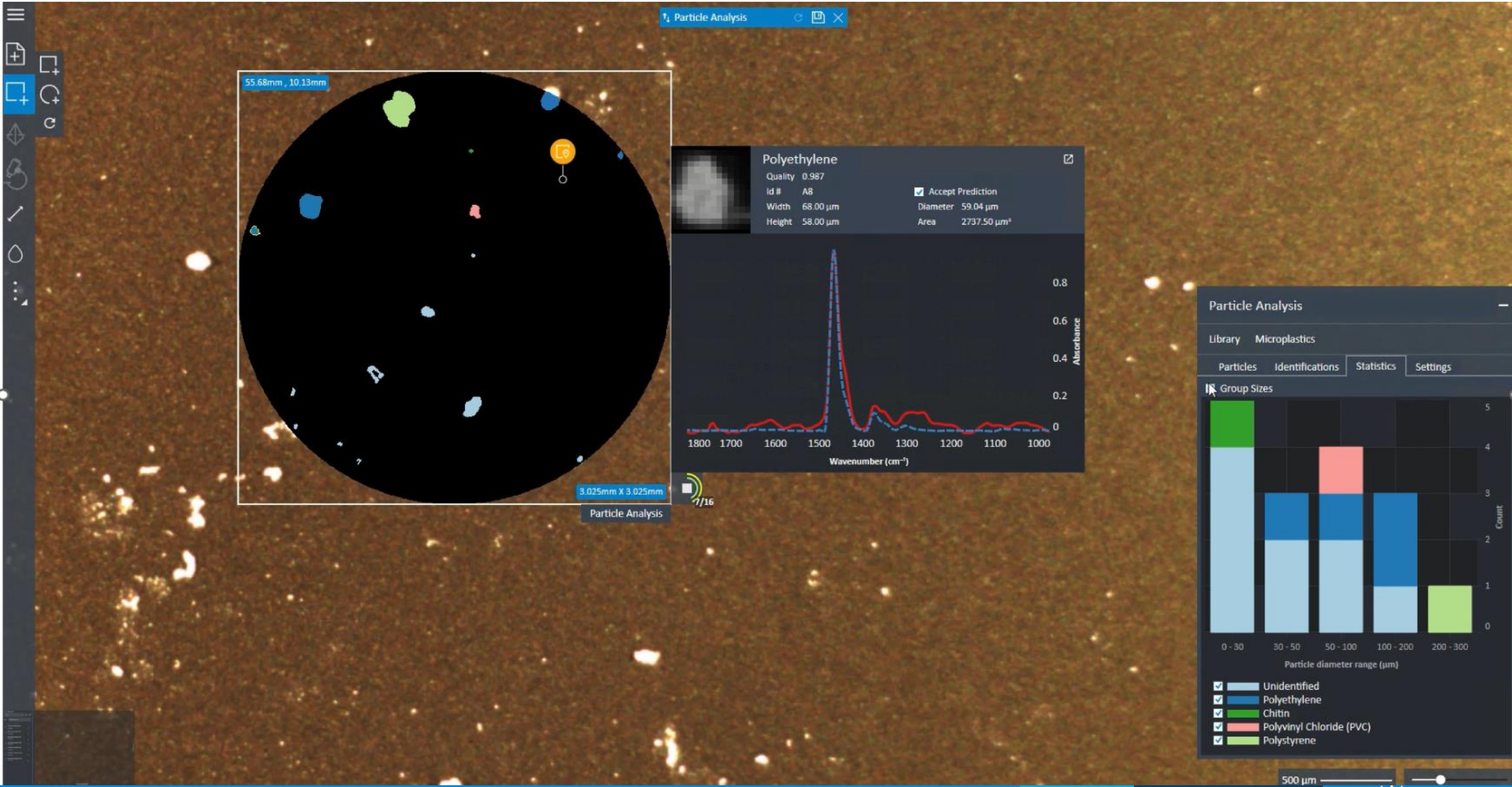
# Locate each particle in X Y space





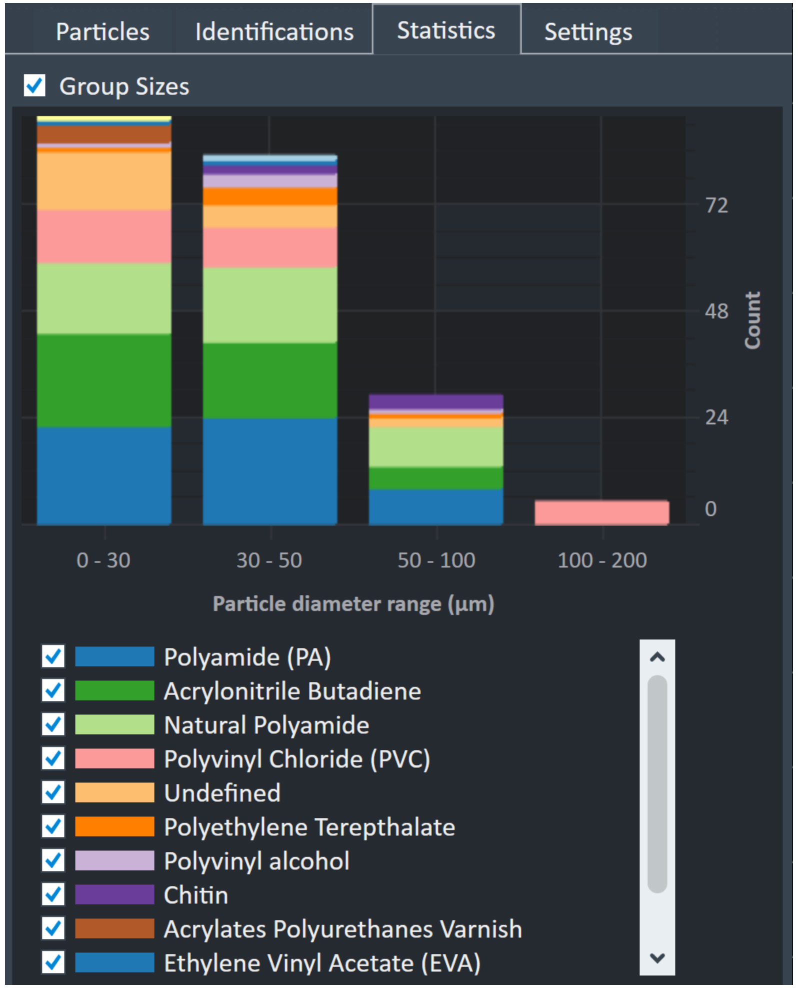
# Microplastics are scanned and identified

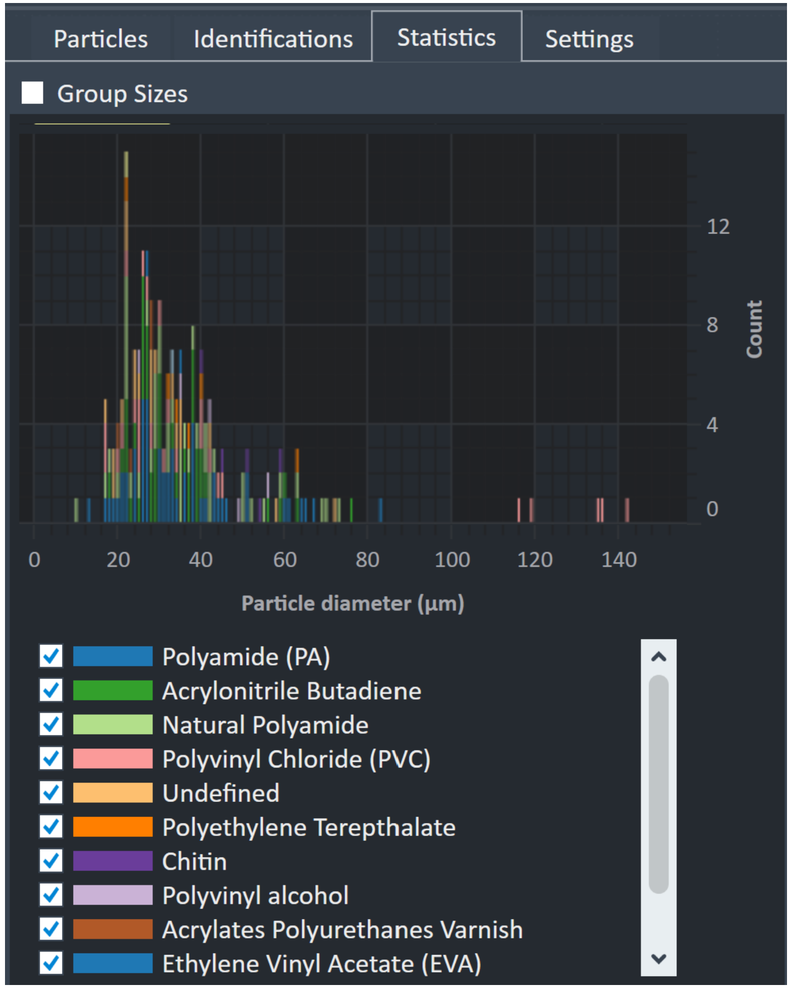




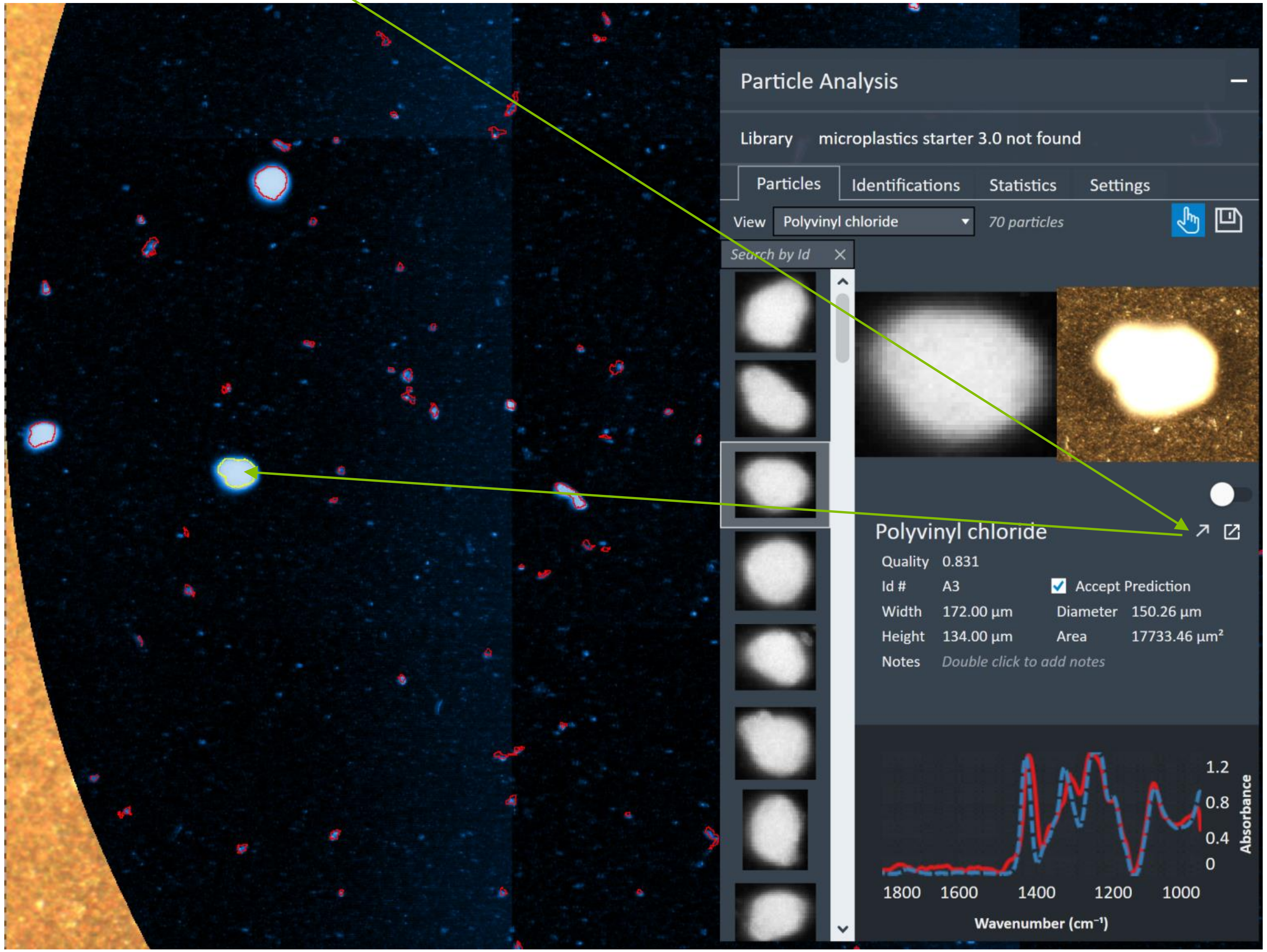
Particle Analysis
55.68mm , 10.13mm
3.025mm X 3.025mm
Particle Analysis
7/16
Polyethylene
Quality 0.987
Id # A8
Width 68.00 µm
Height 58.00 µm
Accept Prediction
Diameter 59.04 µm
Area 2737.50 µm²
Absorbance
Wavenumber (cm⁻¹)
1800 1700 1600 1500 1400 1300 1200 1100 1000
0 0.2 0.4 0.6 0.8
Particle Analysis
Library Microplastics
Particles Identifications Statistics Settings
Group Sizes
Count
0 - 30 30 - 50 50 - 100 100 - 200 200 - 300
Particle diameter range (µm)
Unidentified
Polyethylene
Chitin
Polyvinyl Chloride (PVC)
Polystyrene
500 µm

# Reported Results: Statistics are updated as analysis proceeds

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



# 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 |

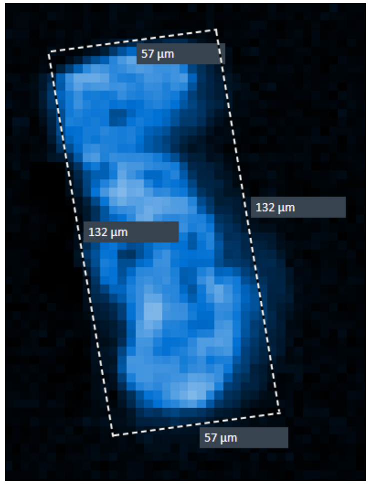
# What do the reported parameters mean? (For use with attached speadsheet)

**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.

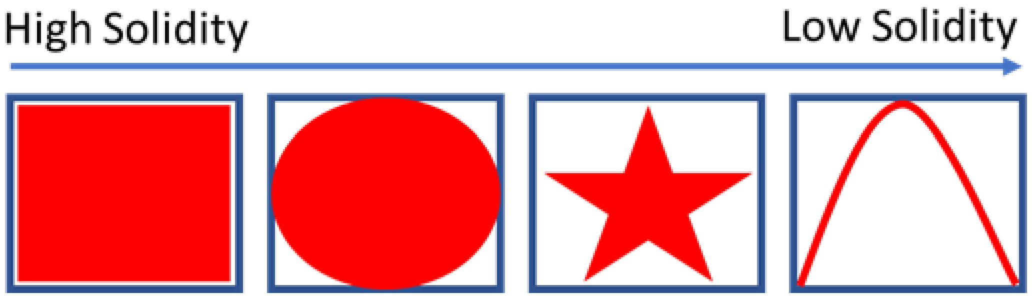


# What do the reported parameters mean?

**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