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Science with Purpose

Optimising the Workflow for Microplastic Analysis by FTIR Microscopy

Microplastics for Breakfast

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What is IR Microscopy used for?

- To IDENTIFY defects, flaws in solids down to ~1/10 width of a human hair. Chemical ID usually supports a visual ID

- To ID very small or trace quantities of samples (eg forensics) e.g. ID a single paint flakes

- To study degradation in materials or across layers

  e.g. how effective is a packaging material

- To study how chemical constituents change across a sample

  e.g. to understand a competitor's product

IR Microanalysis and Sample Size

Smaller

Macro IR

Micro IR

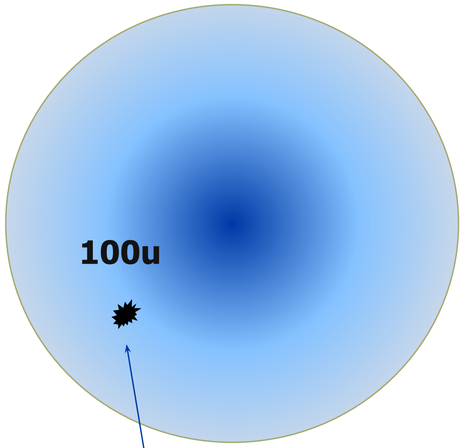
Smallest

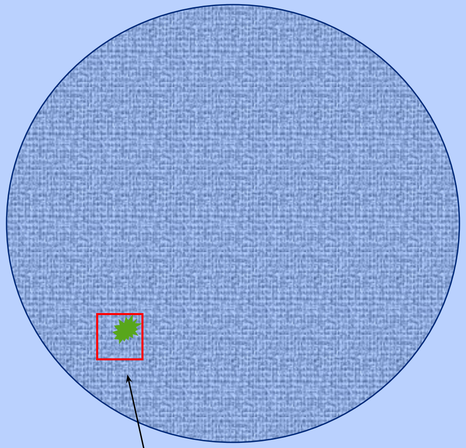
Imaging IR

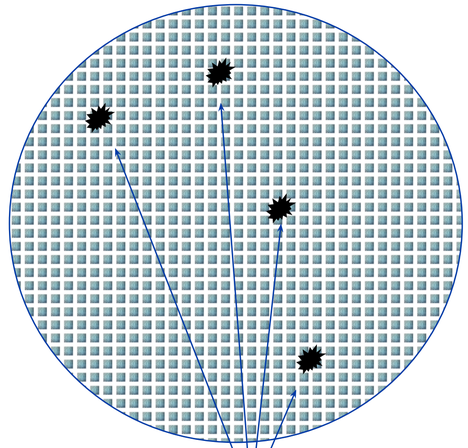
Beam size

1 cm

<1 mm







Contaminant?

You usually can't detect it

You need to see it first

It can find it for you

Spectrum Two, Spectrum 3
100u-yes, 100u in 1cm - No

Spotlight **Aurora**

Spotlight Aurora-I

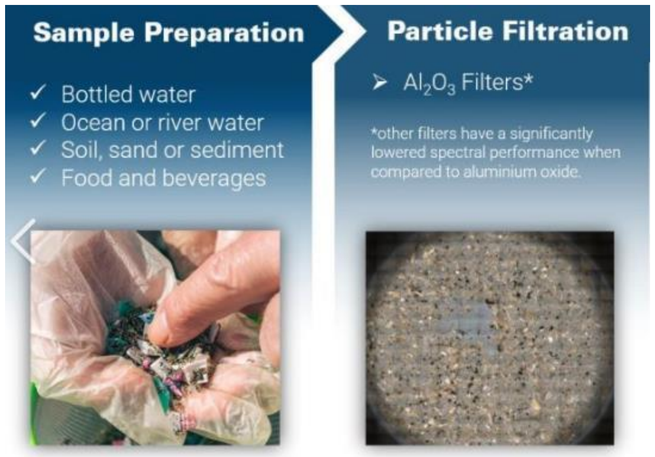
Application: Find/identify contaminants, measure v. small samples, characterise spatial distribution

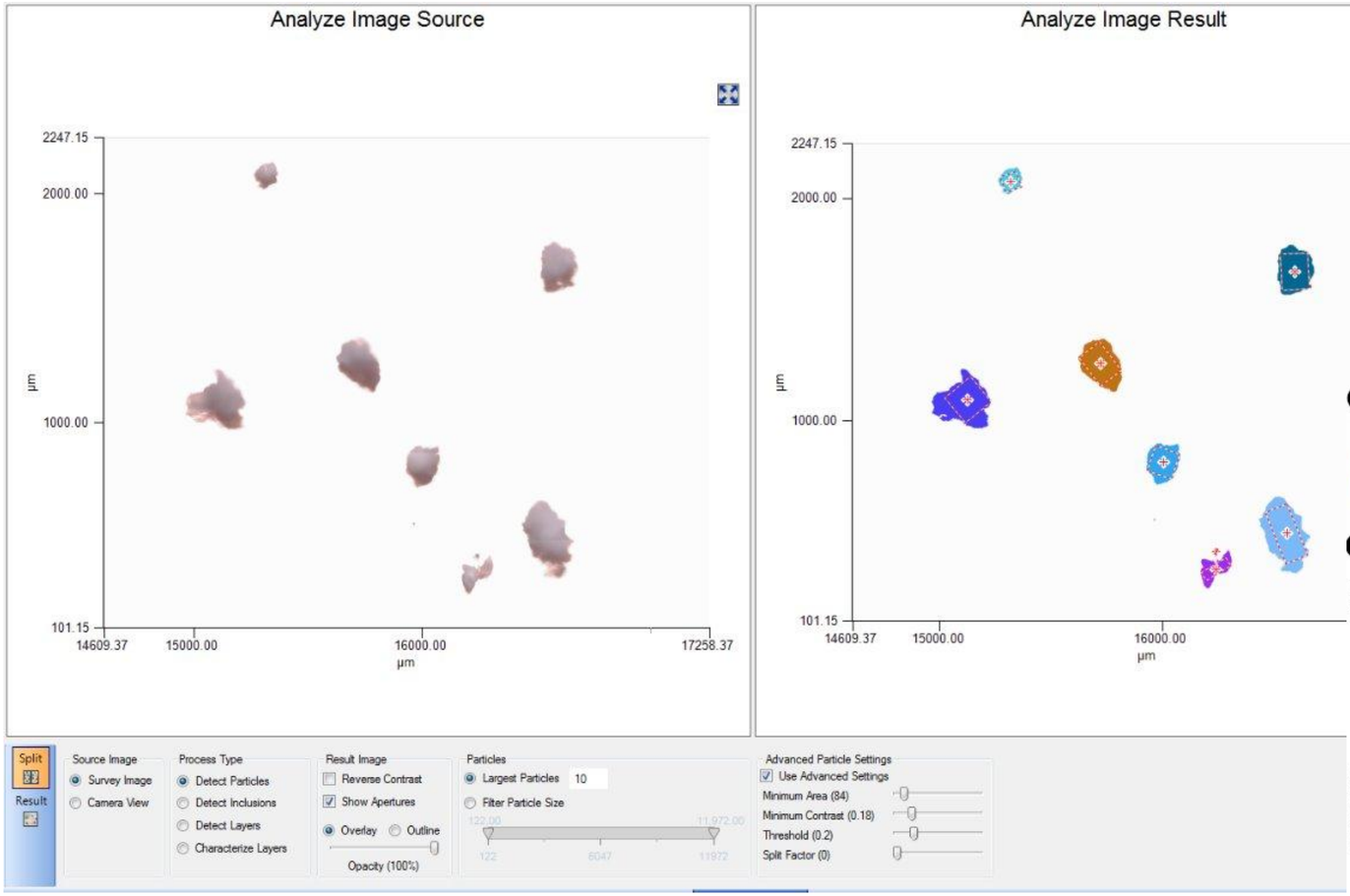
The Microplastics Analysis Workflow

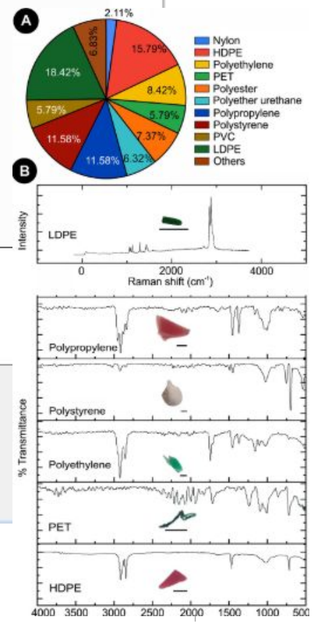
Sample Types and Collection	Sample Cleanup	Sample Preparation for IR Microscopy	Data Collection and Analysis

• Seawater
• River water
• Sediments
• Animal digested
• Consumer related
  ◦ Cosmetics
  ◦ Domestic
  ◦ Food and beverages
	• Flotation
	• Digestion
	  ◦ Acid/alkali
	  ◦ Peroxide
	  ◦ Enzymatic
	• Filtration
		• Filter choice
		• Compatibility
		• Sample size
			Sampling Modes
			• Transmission
			• Reflectance
			• ATR
			
			Measurement modes
			• Point Mode
			• Particle detection
			• Mapping
			• Imaging

IR Microscopy workflow

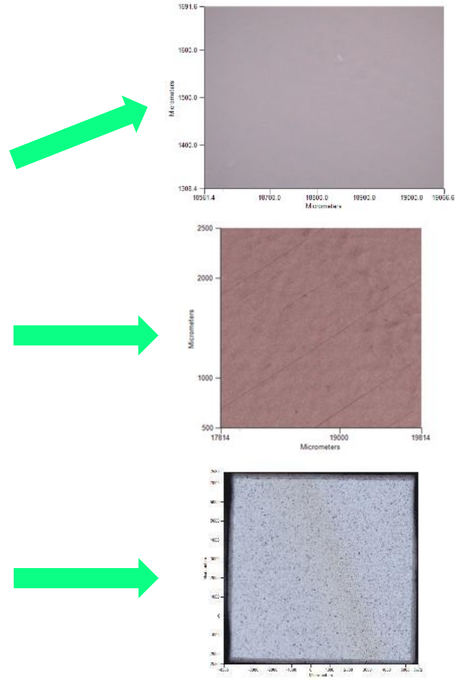






Filter Characteristics

Filter	Diameter	Pore Size	Cost/Filter
Alumina Oxide	13mm	0.2 microns	$8
Glass microfiber	21mm	mixed	$0.2
Gold coated polycarbonate	13mm	0.8 microns	$8
PVDF	25mm	0.45 microns	$2
Silver	13 and 25mm	5 microns	$8
Silicon	10mm square	5 microns	$10



- Pore size
  - Filtration times
  - Blocking
- Diameter
  - Filtration
  - Imaging time
    - 13mm, 50 minutes
    - 25 mm, over 3 hours
- Infrared compatibility
  - Spectral Range
  - Sample size ~5 microns – diffraction limited

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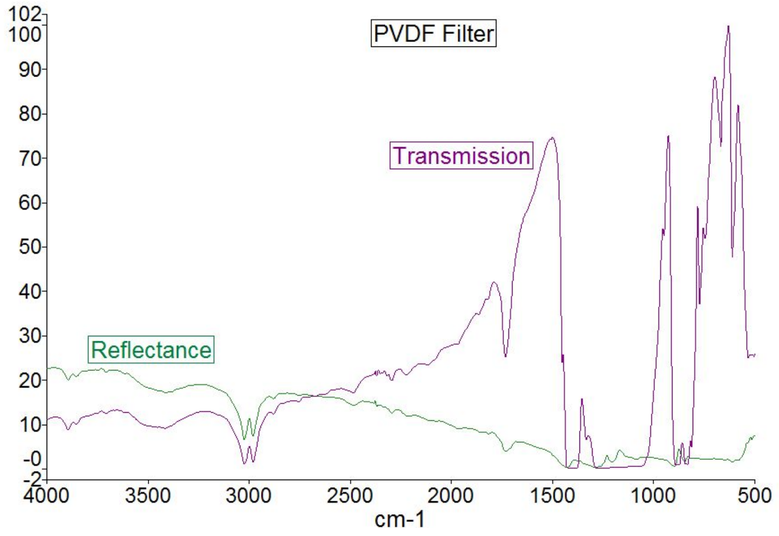
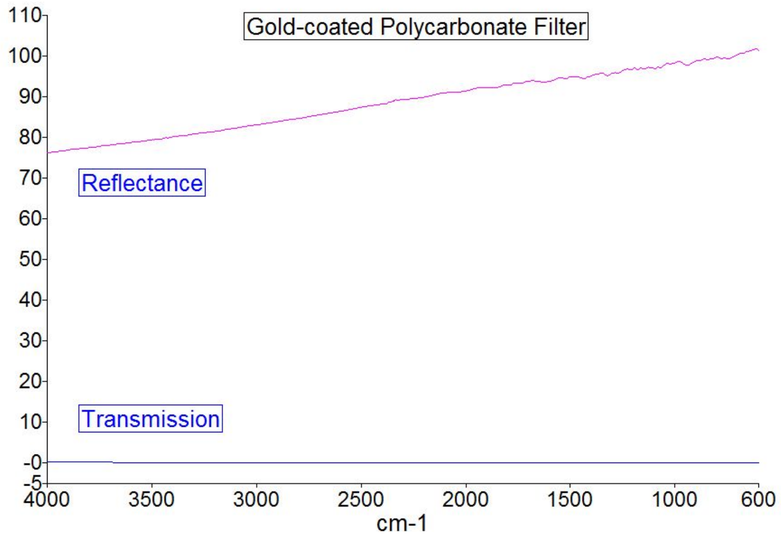
Example Filter Spectra



Poor in both Transmission and Reflectance



Poor in Transmission, Excellent in Reflectance

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IR Spectral Range of Filter Materials

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Alternative after filtration

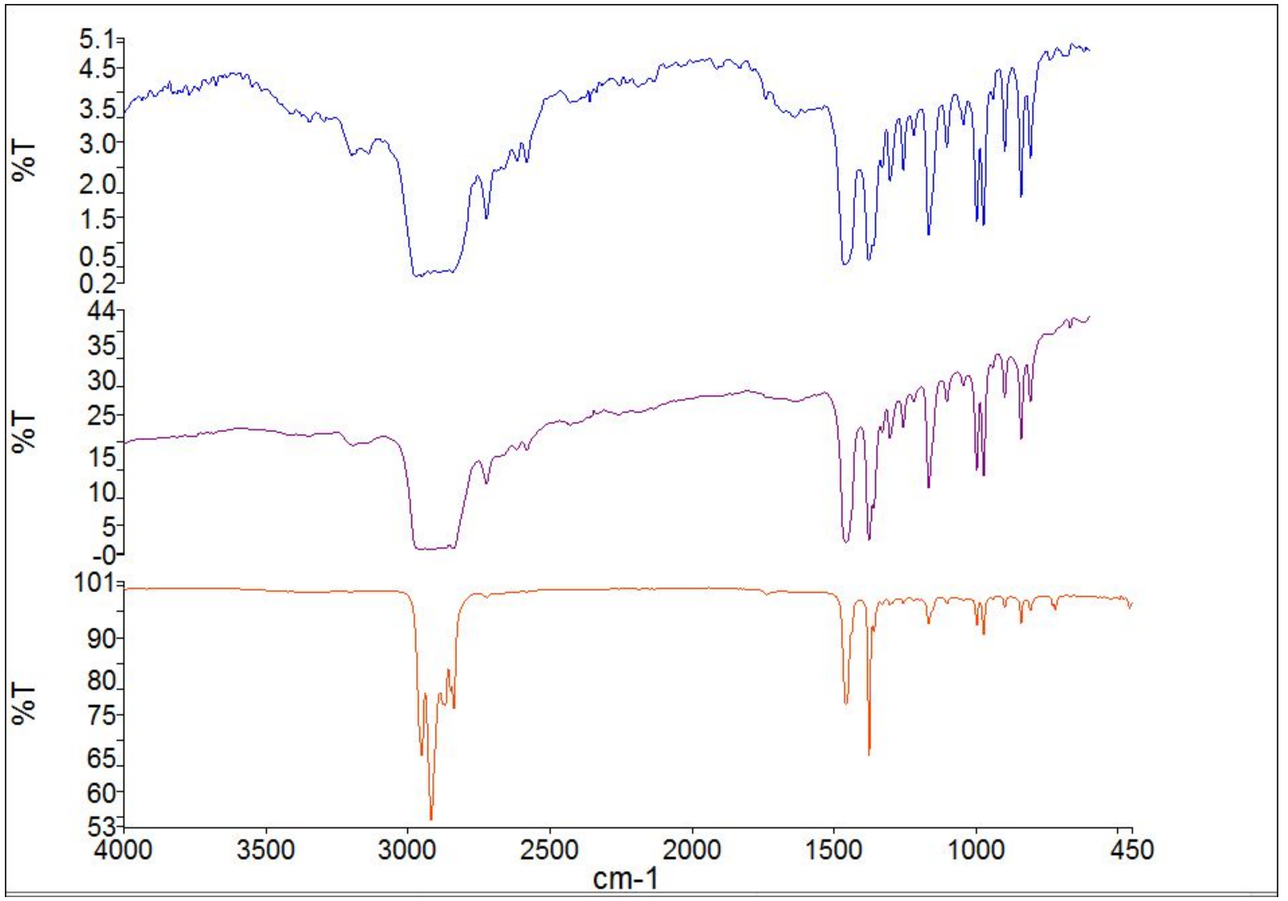
Manually transfer particle(s) onto
suitable substrate

- Transmission measurement on IR compatible window
- Reflectance on low e-glass slide or gold mirror

Not the preferred option as potential loss of
particle(s) and requires an additional step
that could also lead to contamination

FTIR and FTIR Microscopy Sampling Modes for Microplastics

- Reflectance
  - Fast and easy
  - Prone to reflectance artefacts
- Transmission
  - Fast and easy
  - Particles may be too thick leading to poor (too strong) spectra
- ATR
  - Best IR spectra
  - Particles may move under force
  - Particles may stick to ATR crystal



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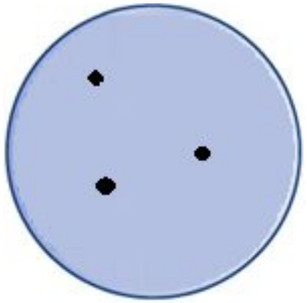
FTIR Microscopy - Measurement Modes

Point Mode

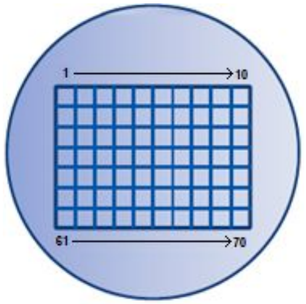


Only measure points of interest.

Fastest for small numbers of particles.

Use with particle detection for automation.

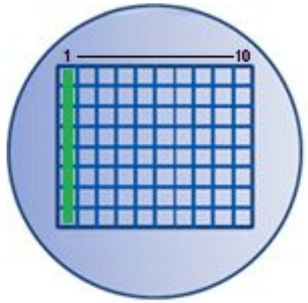
Mapping



Scan-move-scan each point over large region.

Very slow for large regions.

Linear Imaging



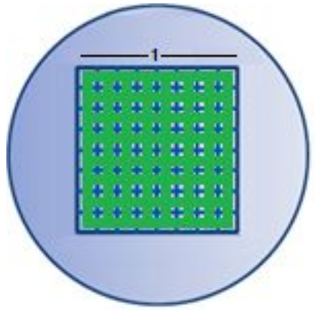
Multiple detector array measures multiple points simultaneously

Fast for large regions

Full spectral range

High Cost system

FPA Imaging



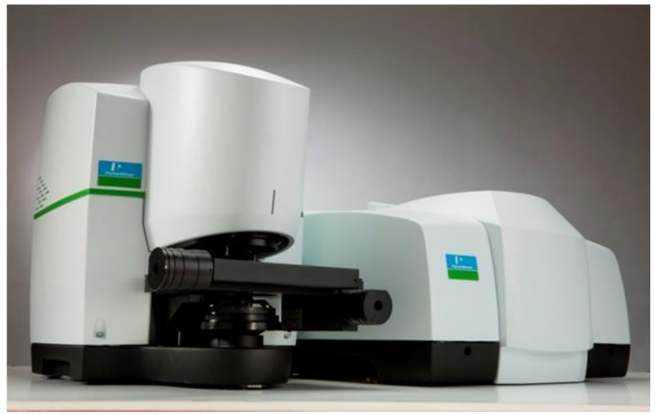
Full X,Y array with largest number of detector pixels

Fastest for large regions

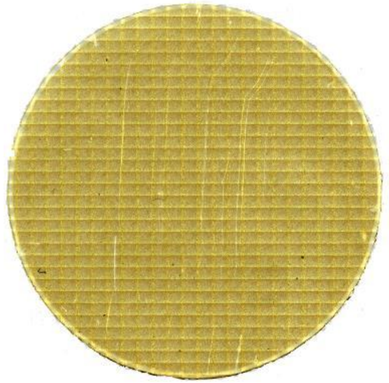
Limited spectral range

Very high cost system

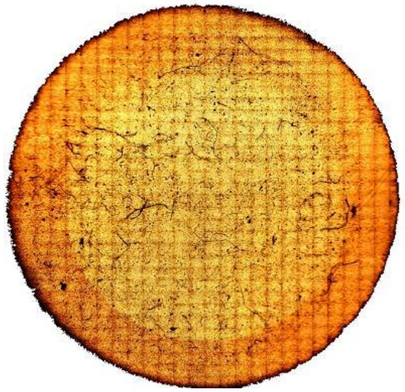
Infrared Microscopy Workflow for Microplastics



1. Collect Visible Image Survey
2. Select Point mode or Imaging
3. Perform IR Data Collection
4. Analyse Data

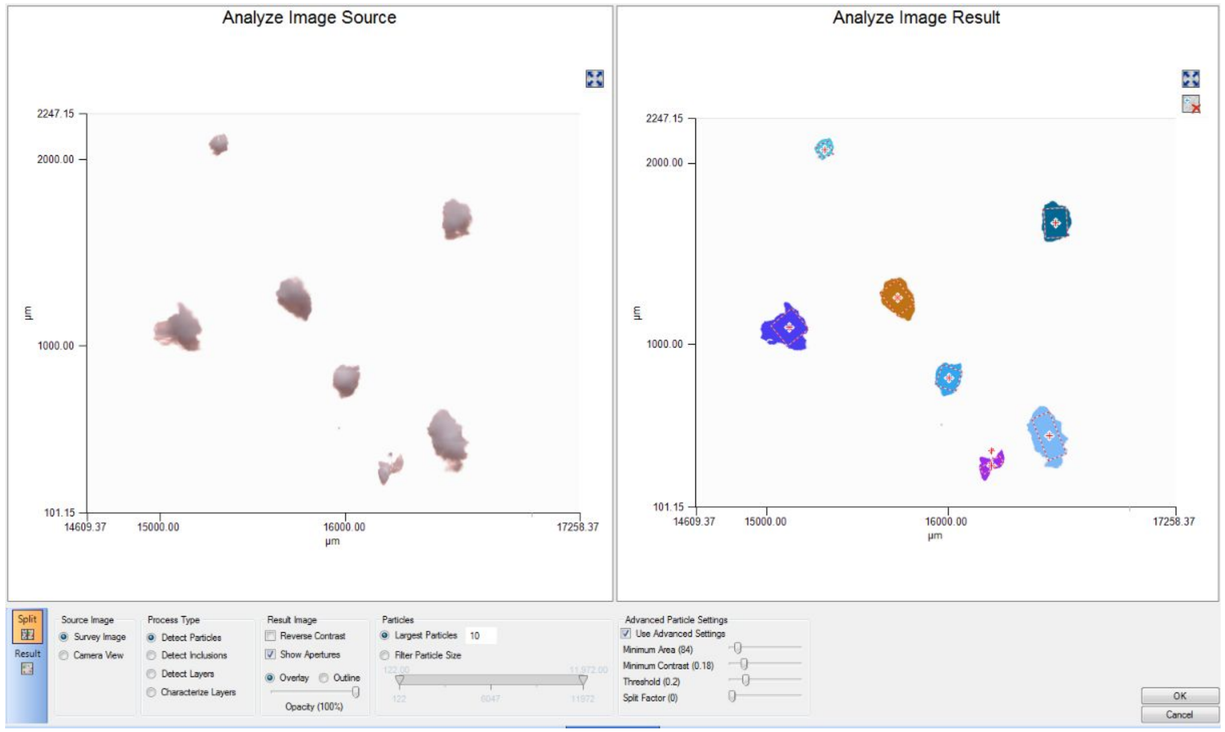


Brand “B” bottled water



Laboratory tap water

Point Mode with Automatic Particle Detection



1. Software algorithm detects particles
2. Displays particles detected
3. Sets maximum aperture to fit inside particle
4. Scans matched aperture backgrounds
5. Scans all particles
6. Classifies particles based on identity from library search

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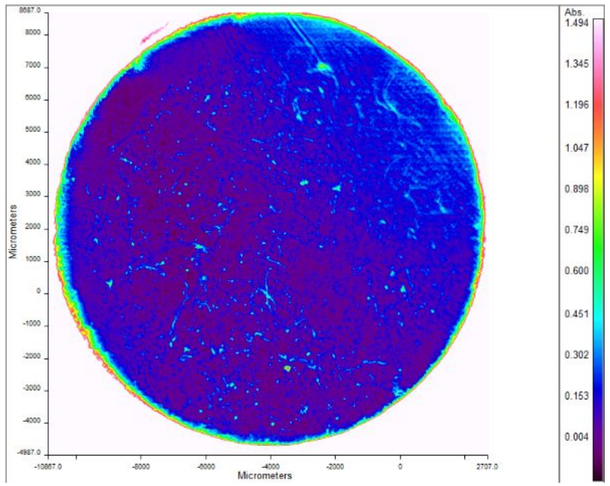
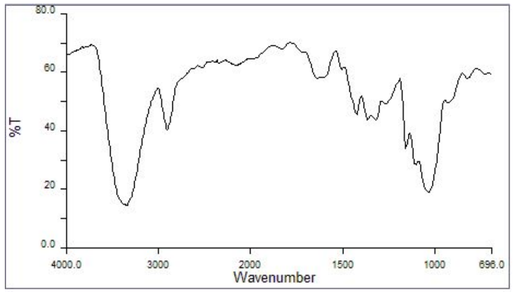
Imaging

- Uses array detectors with multiple detector elements
- Significantly faster than (single detector element) mapping
- Generates chemical images of the sample
- Each pixel represents a spectrum
- Measurement time ~50 minutes for 13mm filter
- ~250,000 spectra

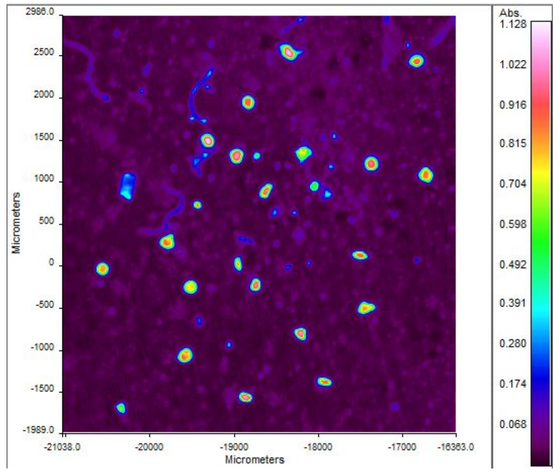


Average Absorbance Image – Tap water sample

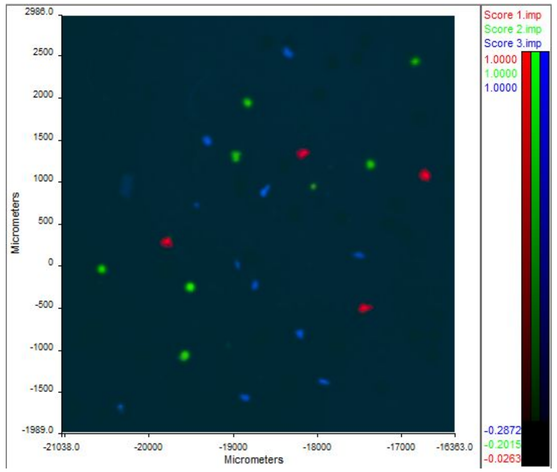


Typical spectra observed - cellulose

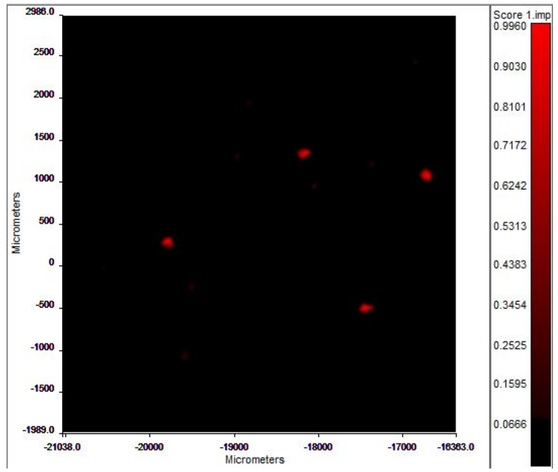
Data Processing – “Show Structure” using Principal Components Analysis



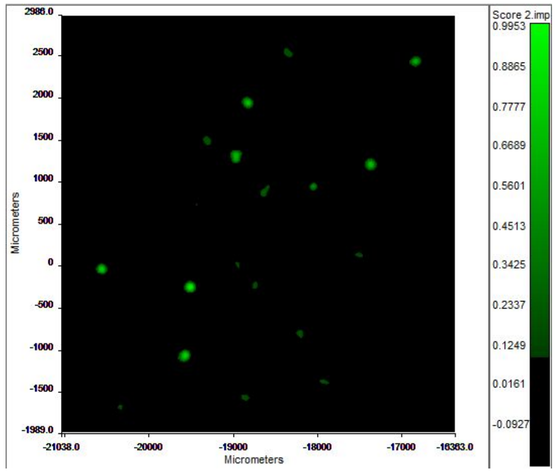
Original Average Abs



Combined Scores

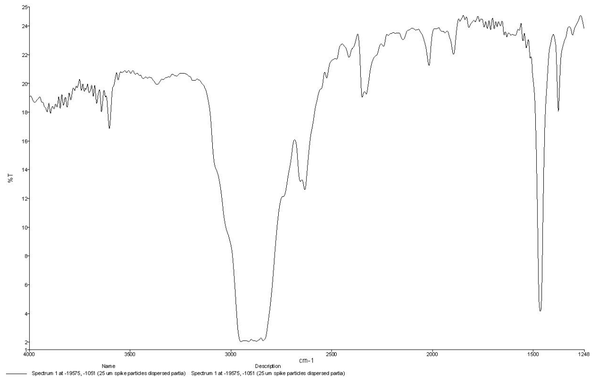


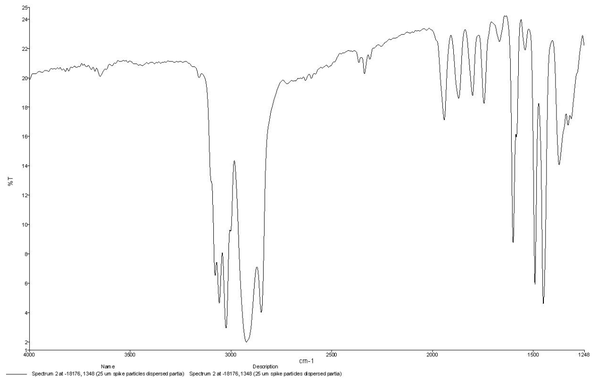
Score 1



Score 2

- Applies PCA to entire dataset
- Ability to pick out different materials
- Different scores represent materials





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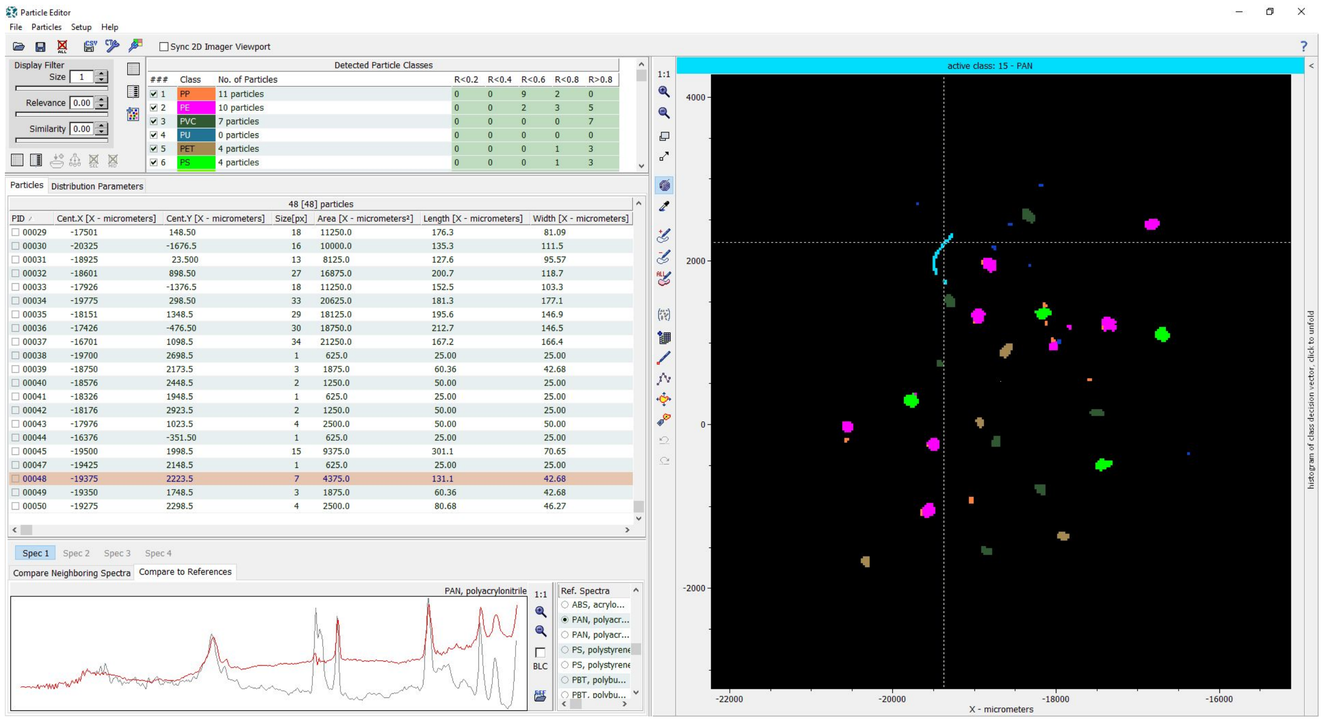
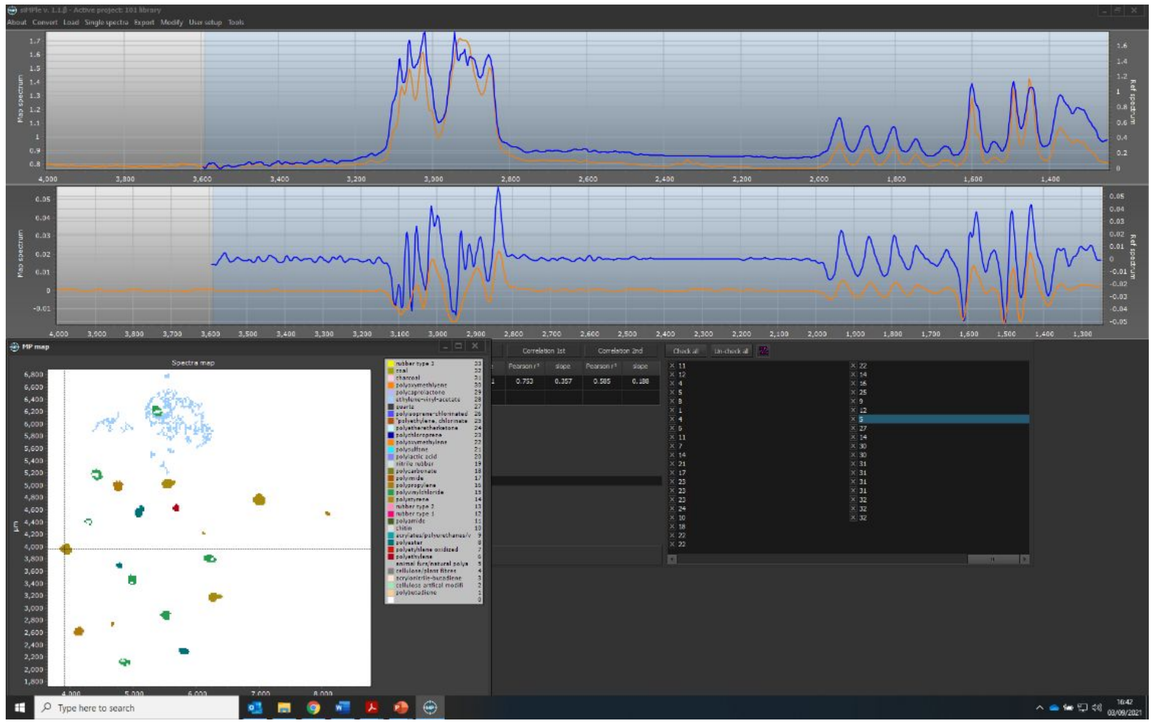
3rd Party Microplastics Data Analysis Packages

Purency Software



siMPle Software



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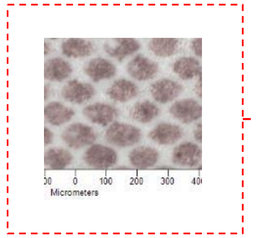
What’s next? Spotlight Aurora

New Optics for Finding defects in real samples – 1. *Wide areas*



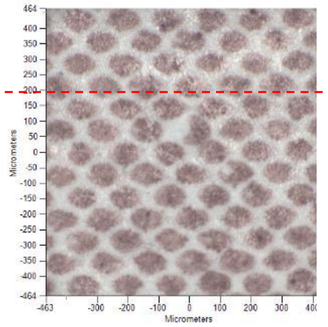
~500x350u

175000

(1x)

Spotlight 200i

Single frame Images

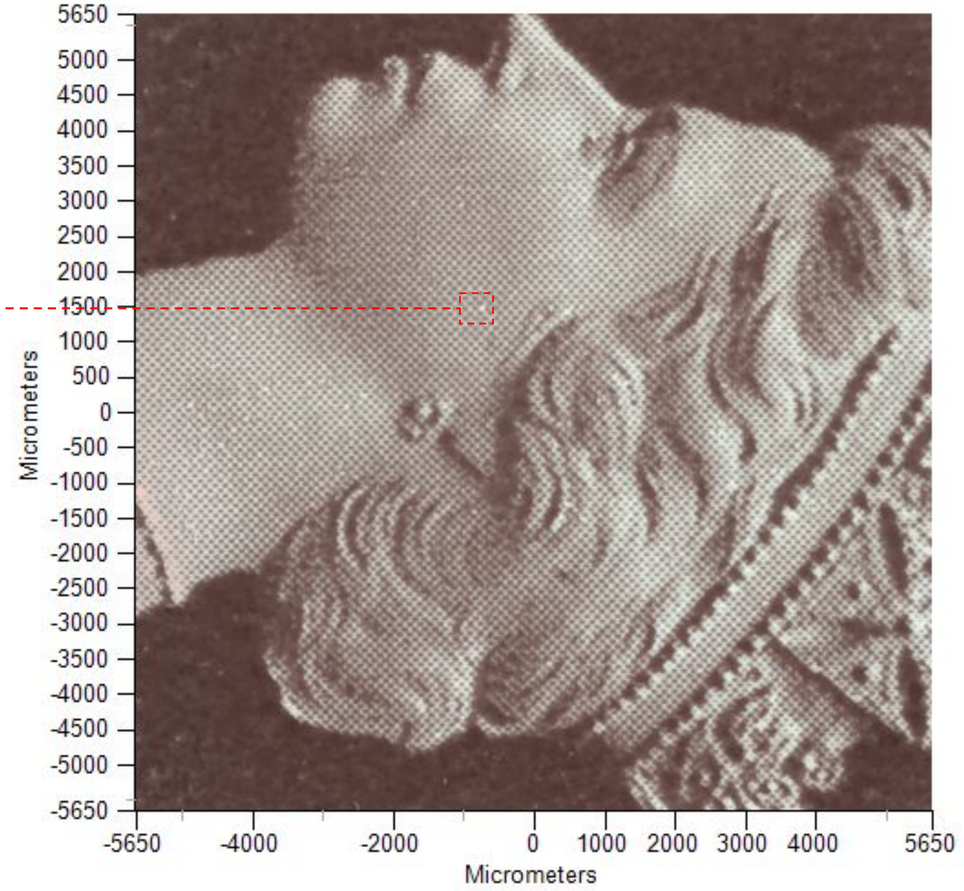


1,000,000

(5.7x)

Spotlight Aurora Hi Def

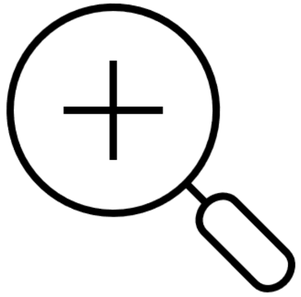
Around 13x faster for a 1x1 cm image survey



100,000,000

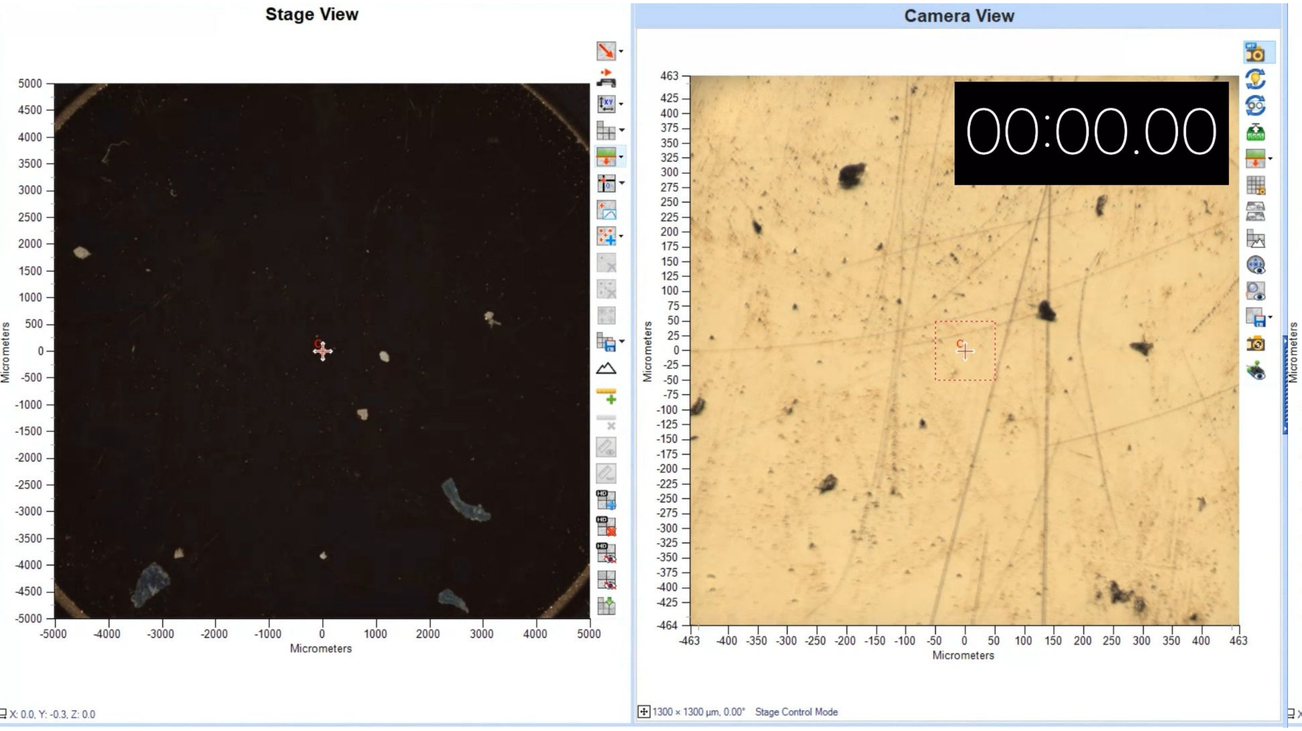
(570x)

Spotlight Aurora Widefield

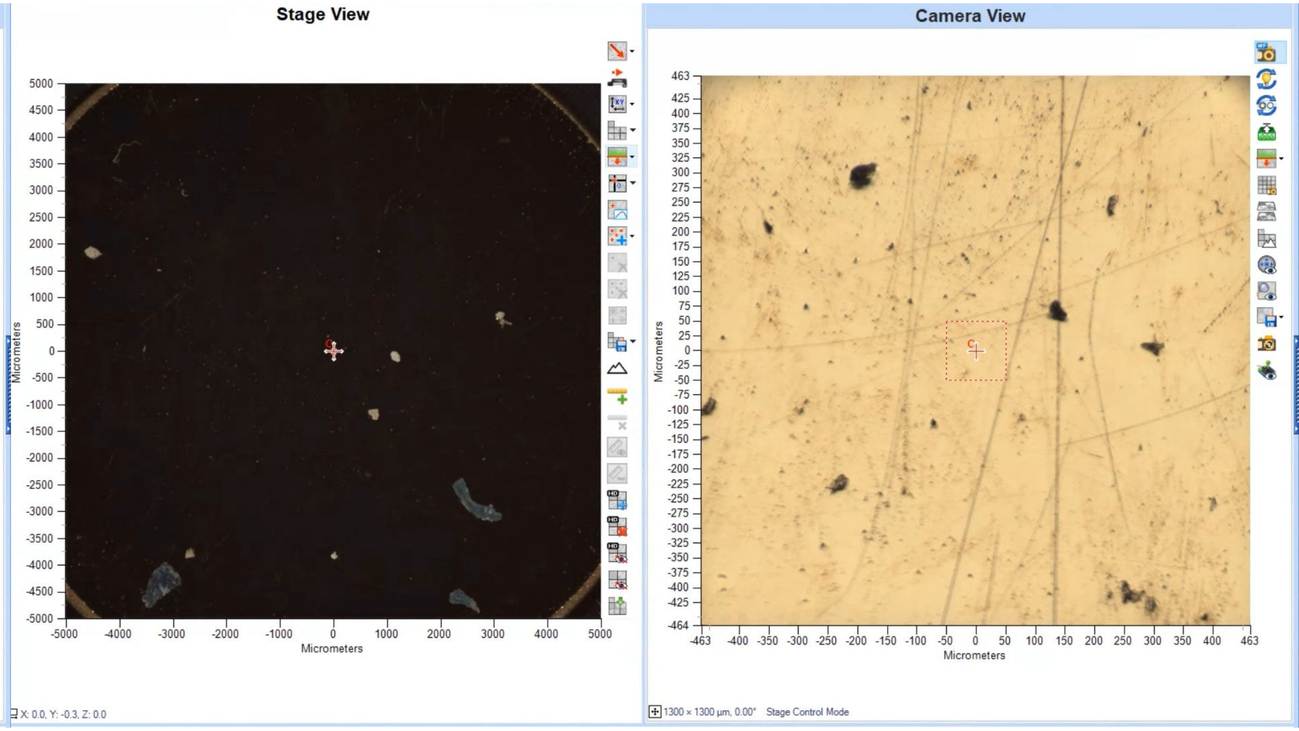


Find and locate particles: Collect a 1x1 cm survey image

Spotlight Aurora



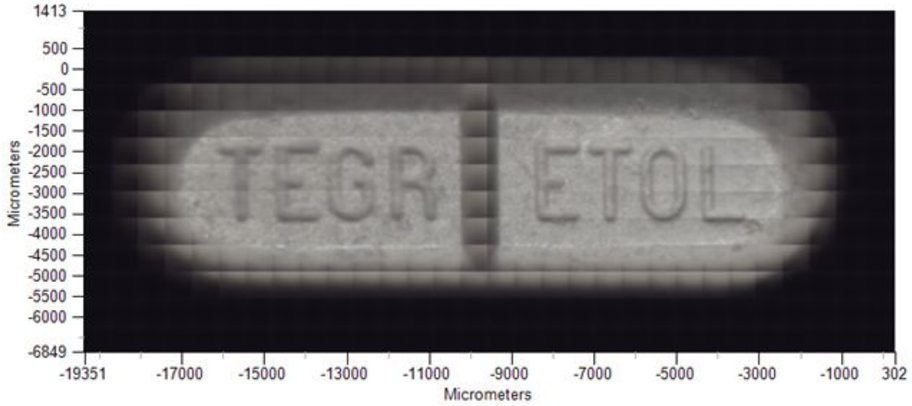
Previous Instrument

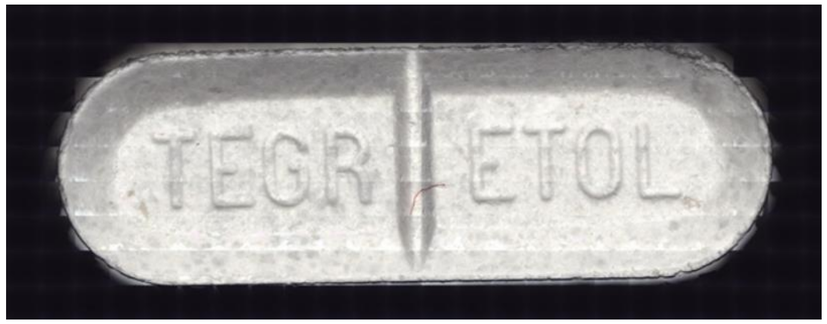


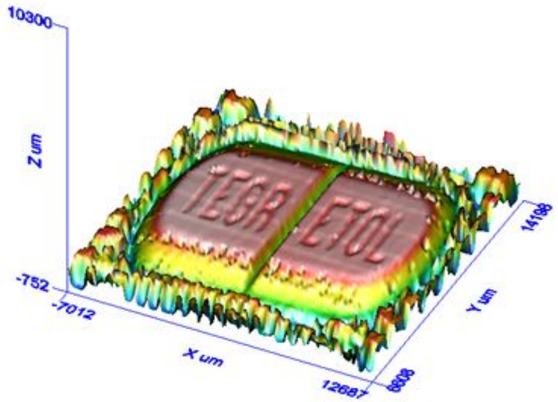
Particles are collected on a reflecting surface for measuring and identifying using IR spectra

Initially – a survey image of a 1x1 cm area is needed to isolate, aperture and collect the individual spectra before IR library search and ID

Pharmaceutical samples

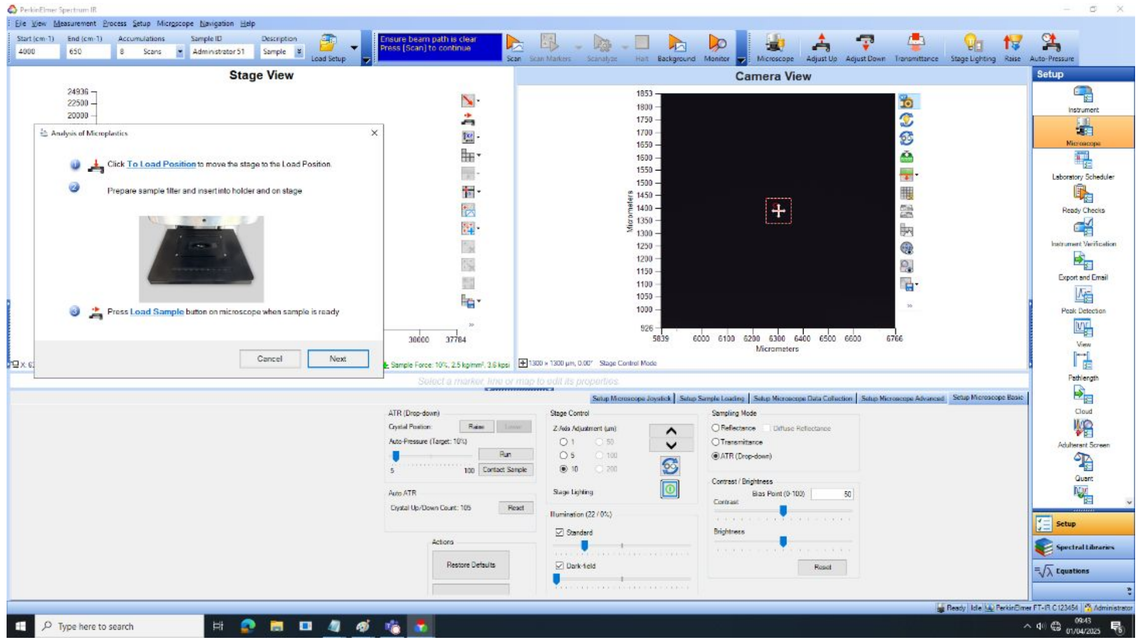


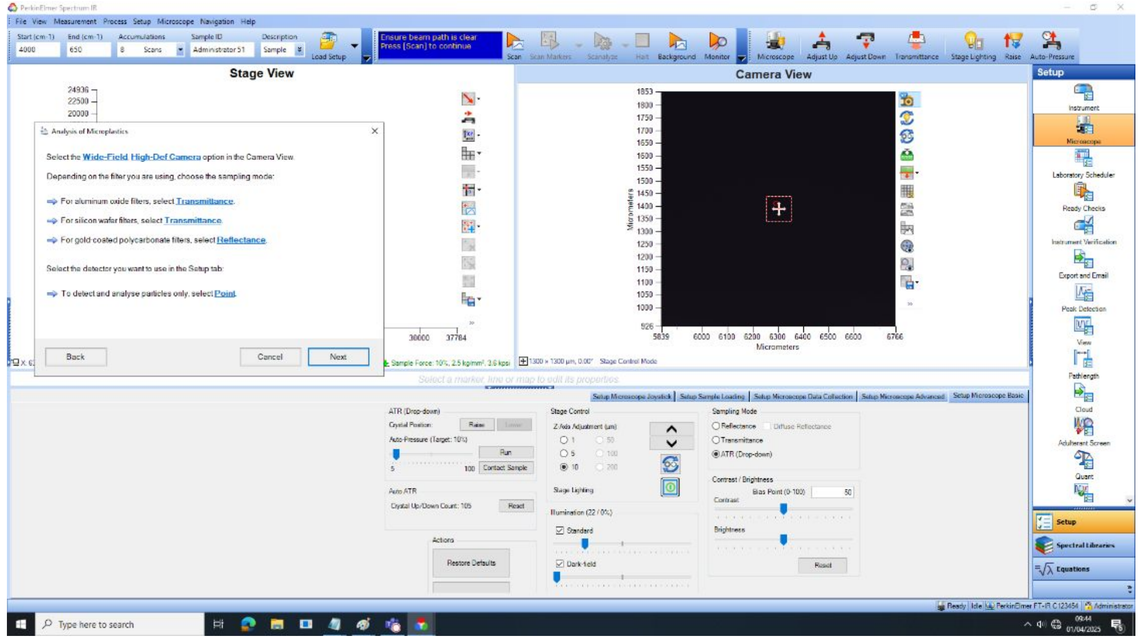




Spotlight Aurora – Microplastics Guided Workflow

Guides the user through the steps required for analysis





Conclusions

- FTIR Microscopy is an important technique for the analysis of microplastics down to a few microns in particle size
- Appropriate sample preparation is essential to eliminate matrix interferences and to generate good quality spectra
- Optimum filters for IR measurements
  - Gold Coated PC or silicon for reflectance, silicon or Alumina Oxide for Transmission
  - 13mm diameter
  - 5 microns pore size
- Optimum sample throughput
  - Point mode measurements with particle detection for small numbers of particles
  - Imaging for whole filters