Water Workshop 2025

Applied sampling, preparation and determination protocol of microplastic contamination in HUSRB/23R/12/089 project

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Microplastics

Why microparticles are difficult to study

Microparticles are very small and occur in complex environments, making them hard to prepare and analyse reliably.

- Mixed with many other substances
- High risk of error or misidentification
- Require strict laboratory control

Contamination Risk

External contamination is one of the biggest problems in microparticle analysis.

- Fibres from clothing and air dust
- Plastic residues from tools
- Risk of false positives

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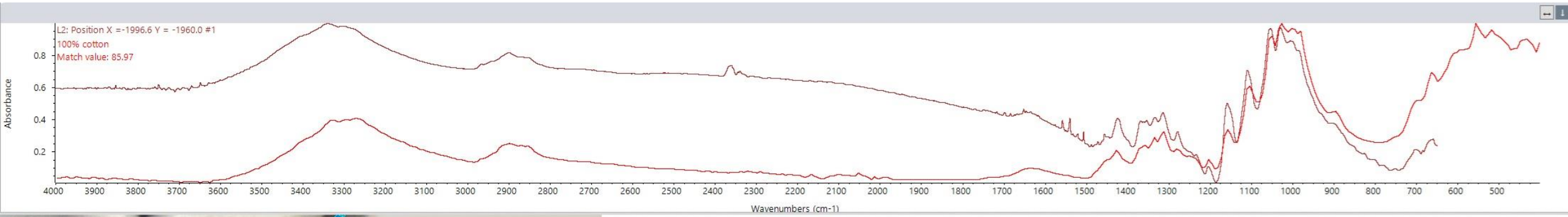
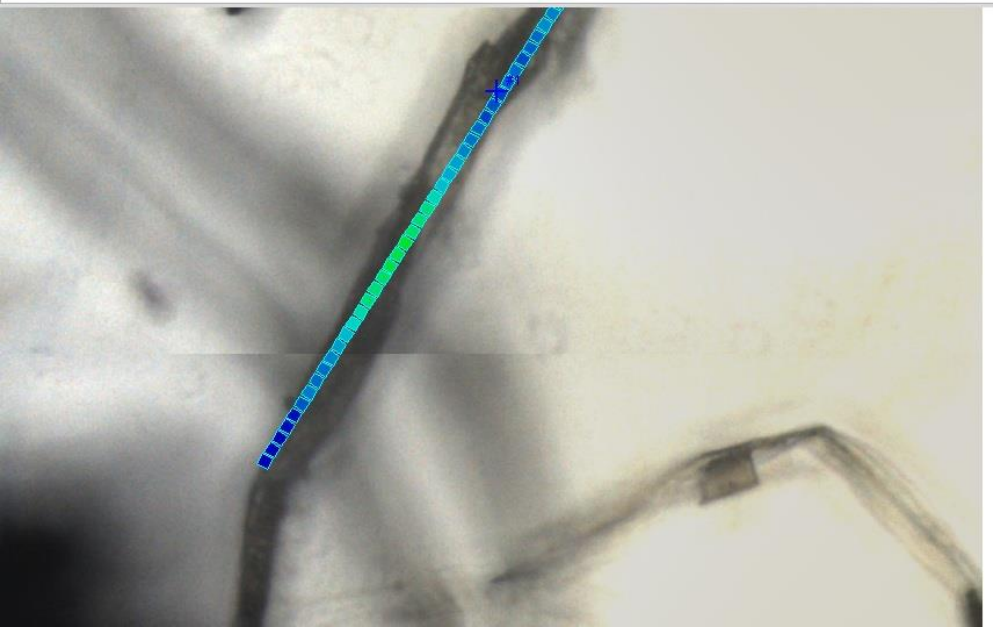
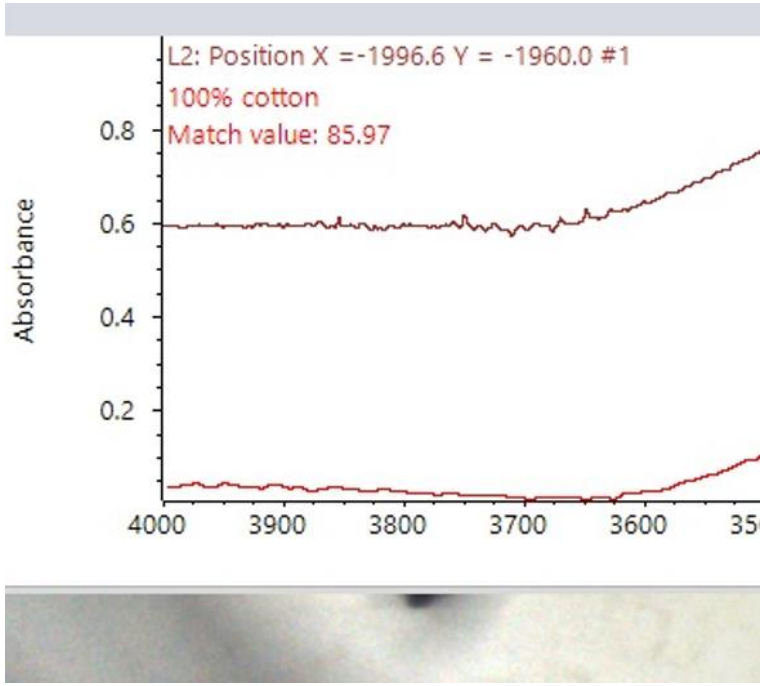






Sample loss during preparation

Particles are so small that they often get lost during handling or filtration.

- Adhesion to surfaces and tools
- Retention inside the filter housing
- Reduced sample accuracy

Challenges with filter materials

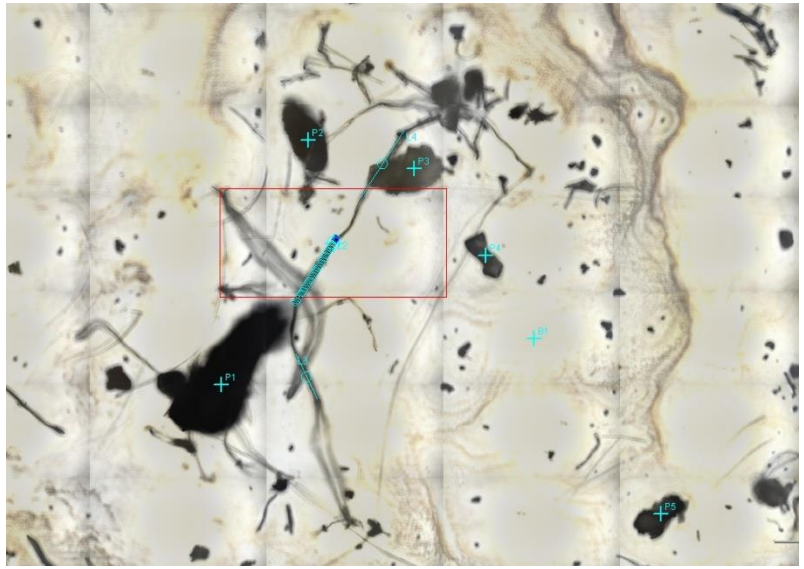
The filter must be stable but should not interfere with the analysis.

- Release of fibres into the sample
- Own IR absorption bands
- Background noise in spectra

Heterogenity of samples

Microparticles are diverse in size, shape, and composition, which complicates analysis.

- Broad size distribution
- Mixed polymers and morphologies
- Poor representativeness after filtration



Chemical pre-treatment

Organic matter must be removed carefully to avoid damaging the particles.

- Oxidative or enzymatic digestion
- Risk of polymer degradation
- Altered surface chemistry

Fenton Reaction

$$Fe^{2\oplus} + H_2O_2 \longrightarrow Fe^{3\oplus} + HO\cdot + OH^{\ominus}$$

Ferrous | Hydrogen peroxide | Ferric | Hydroxyl radical | Hydroxyl

$$Fe^{3\oplus} + H_2O_2 \longrightarrow Fe^{2\oplus} + HOO\cdot + H^{\oplus}$$

Hydroperoxyl | Proton

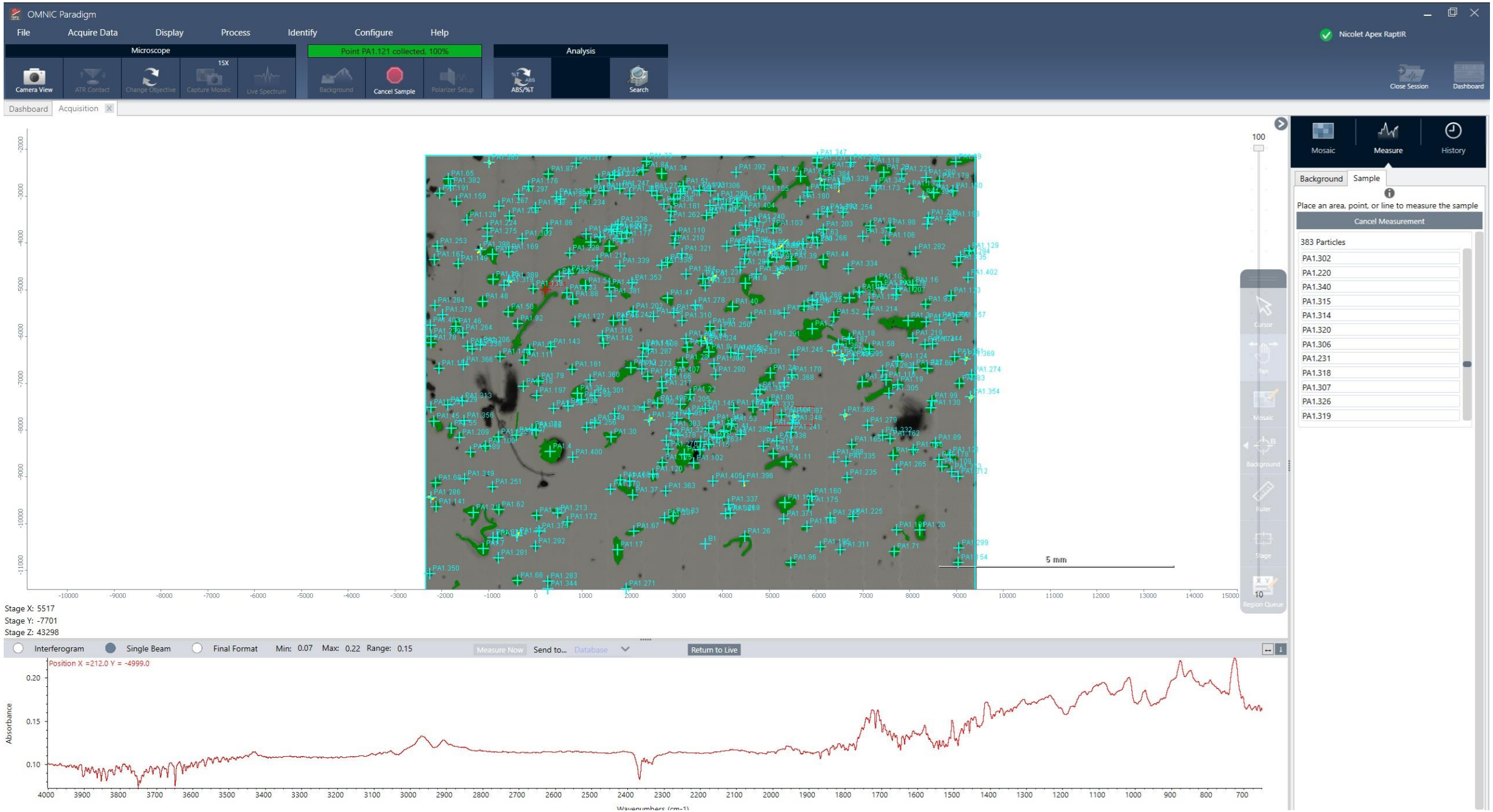
$$2\,H_2O_2 \longrightarrow HO\cdot + HOO\cdot + H_2O$$

ChemistryLearner.com

Micro-FTIR: size limitation

Micro-FTIR cannot measure the smallest microparticles and nanoplastics.

- Resolution ~10–20 µm
- Small particles remain undetected
- Underestimation of particle numbers

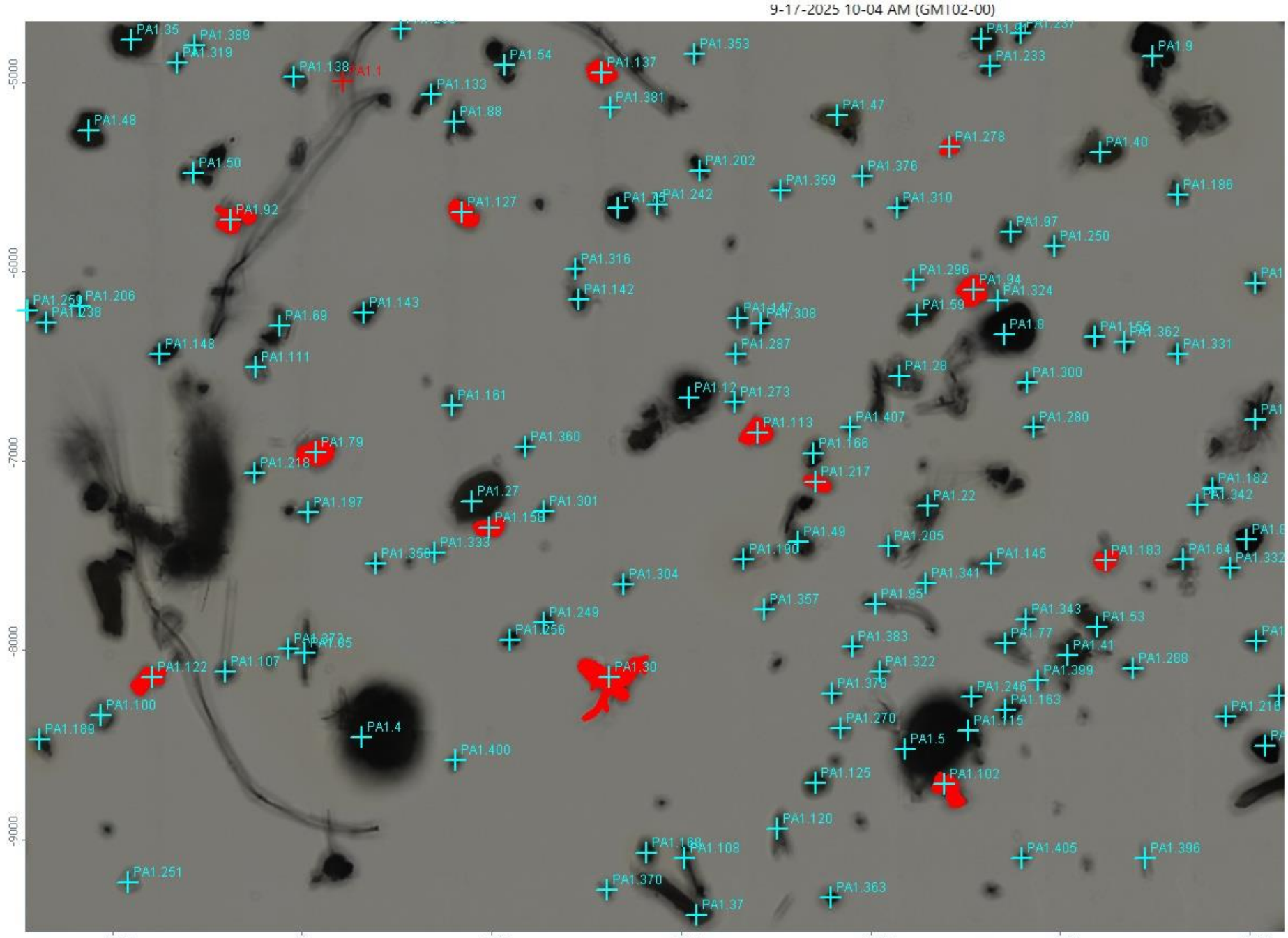

OMNIC Paradigm
File
Acquire Data
Display
Process
Identify
Configure
Help
Nicolet Apex RaptIR
Microscope
Point PA1.121 collected, 100%
Analysis
Camera View
ATR Contact
Change Objective
15X
Capture Mosaic
Live Spectrum
Background
Cancel Sample
Polarizer Setup
ABS/%T
Search
Close Session
Dashboard
Dashboard
Acquisition
Mosaic
Measure
History
Background
Sample
Place an area, point, or line to measure the sample
Cancel Measurement
383 Particles
PA1.302
PA1.220
PA1.340
PA1.315
PA1.314
PA1.320
PA1.306
PA1.231
PA1.318
PA1.307
PA1.326
PA1.319
5 mm
Stage X: 5517
Stage Y: -7701
Stage Z: 43298
Interferogram
Single Beam
Final Format
Min: 0.07
Max: 0.22
Range: 0.15
Measure Now
Send to...
Database
Return to Live
Position X =212.0 Y = -4999.0
Absorbance
Wavenumbers (cm-1)

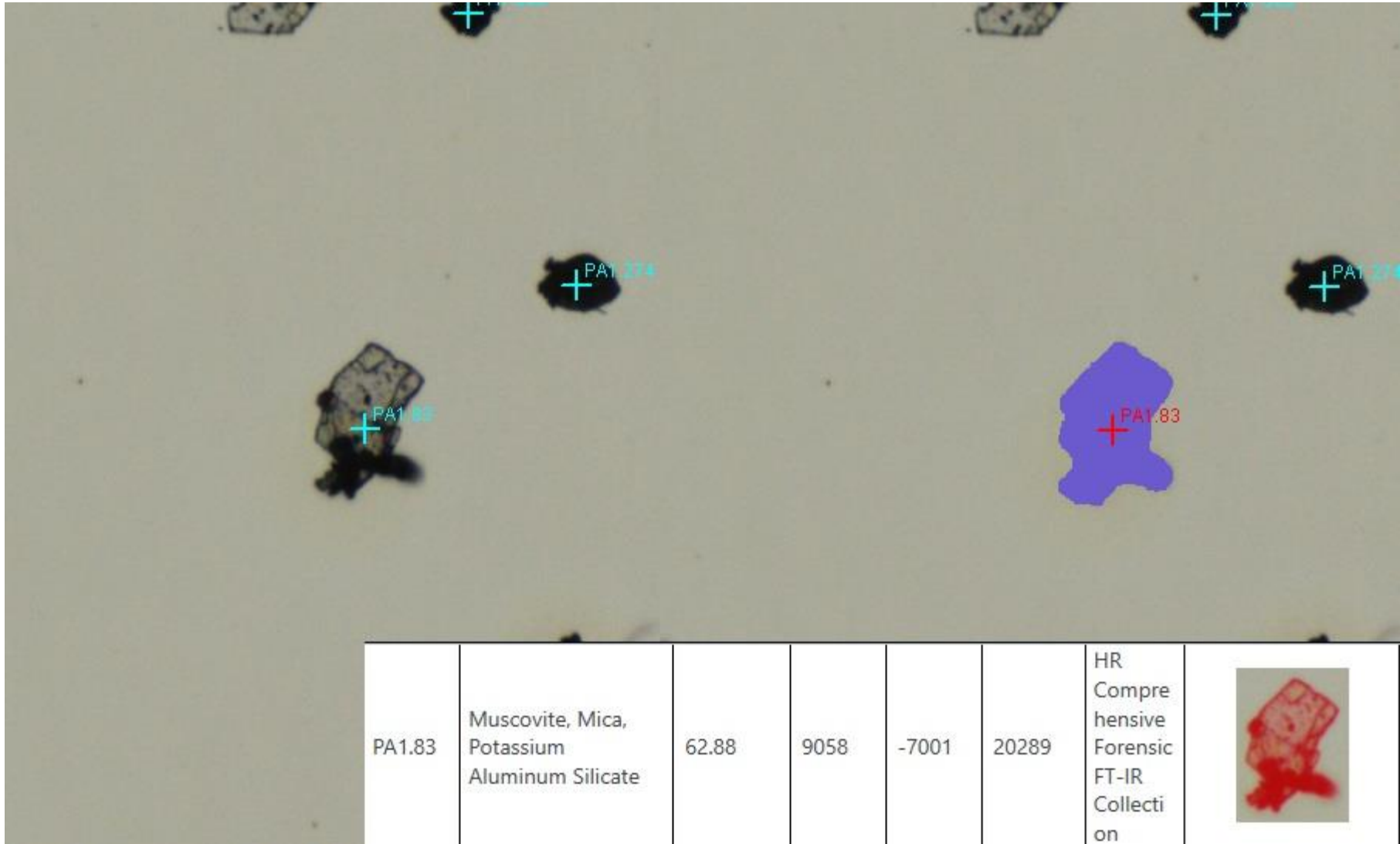
Background and interferences

Background signals from filters and matrices can obscure microparticle spectra.

- Filter substrate interference
- Residual organic matter
- Overlapping absorption bands

Rank	Component Name	Area %	Count	Library	Color
1	Water vapor-without CO2	7.02	33	HR Georgia State Forensic Drugs	





PA1.83	Muscovite, Mica, Potassium Aluminum Silicate	62.88	9058	-7001	20289	HR Comprehensive Forensic FT-IR Collection	

Particle morphology

Shape and thickness influence spectral quality and measurement success.

- Irregular shapes scatter IR light
- Curved surfaces distort spectra
- Thick particles block transmission

Spectral interpretation

Interpreting spectra is limited by incomplete libraries and particle alterations.

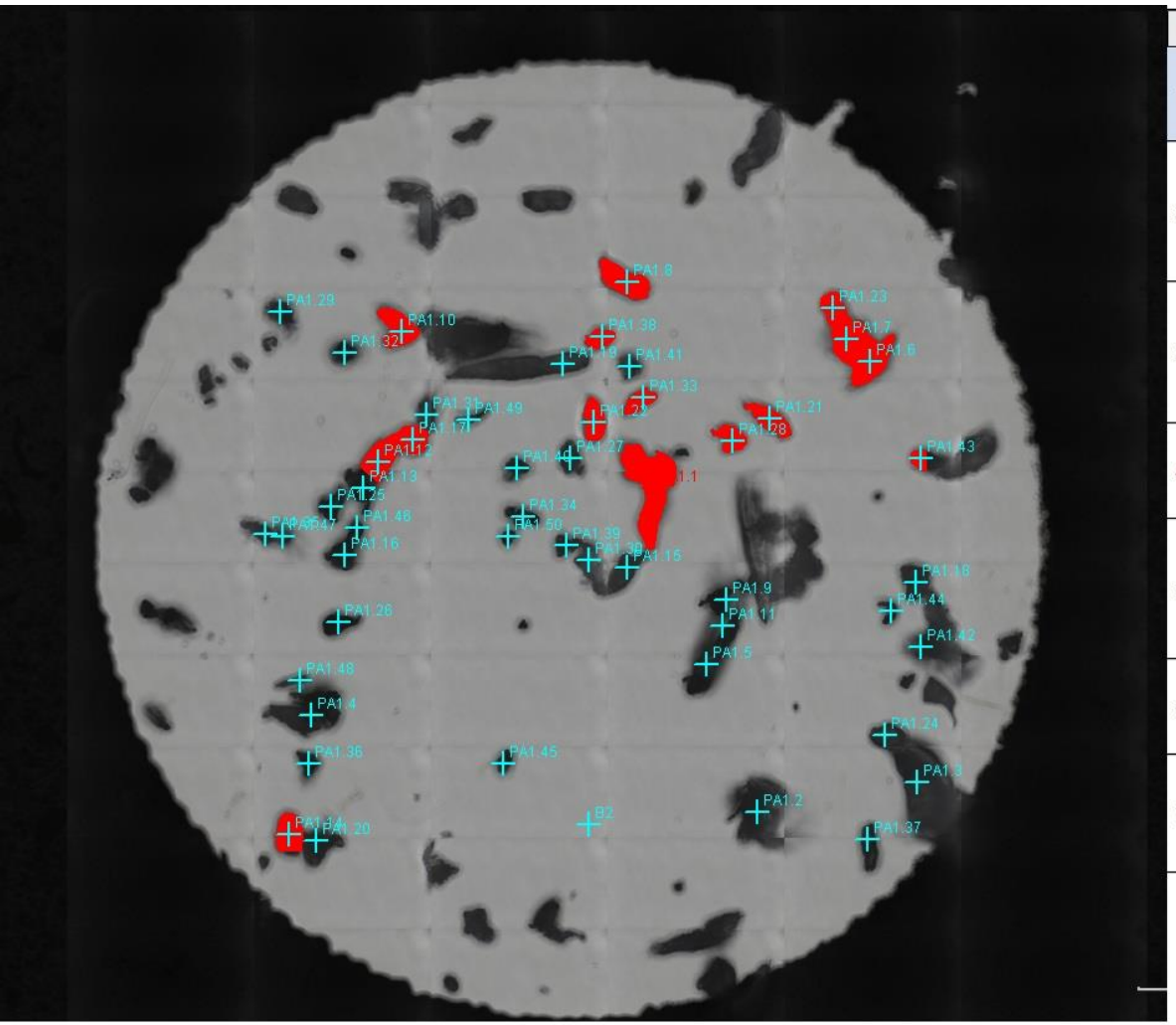
- Missing polymers and additives
- Blends and weathered particles
- Uncertain identification

#	Component Name	Match %	X (µm)	Y (µm)	Area (µm²)	Library	Particle thumbnail
PA1.114	POLYPROPYLENE HOMOPOLYMER #1	79.63	-805	-10306	16834	HR Polymer Additives and Plasticizers	

Representativeness

Only a fraction of particles can be analysed, which limits conclusions.

- Limited particle number per sample
- Non-representative particle selection
- Reduced statistical significance



Rank	Component Name	Area %	Count	Library	Color
1	POLYSTYRENE STANDARD, TYPICAL MW 200,000	35.13	15	HR Aldrich Polymers	
2	POLYPROPYLENE #1	12.76	5	HR Polymer Additives and Plasticizers	
3	POLYVINYL CHLORIDE #3	8.59	2	HR Polymer Additives and Plasticizers	
4	Tygon polymer R-3603	1.46	1	HR Nicolet Sampler Library	
5	Poly(ethylene terephthalate)	1.36	1	HR Hummel Polymer and Additives	
6	Vinyl Gloves	0.96	1	Common Materials	
7	Water vapor-without CO2	0.59	1	HR Georgia State Forensic Drugs	

#	Component Name	Match %	X (µm)	Y (µm)	Area (µm²)	Library	Particle thumbnail
PA1.1	POLYSTYRENE STANDARD, TYPICAL MW 200,000	90.41	19270	2919	47494	HR Aldrich Polymers	
PA1.2	POLYVINYL CHLORIDE #3	76.40	19704	1577	46703	HR Polymer Additives and Plasticizers	
PA1.3	Unidentified	0.00	20362	1703	41870		
PA1.4	Unidentified	0.00	17854	1976	41207		
PA1.5	POLYPROPYLENE #1	78.73	19491	2187	25918	HR Polymer Additives and Plasticizers	
PA1.6	POLYSTYRENE STANDARD, TYPICAL MW 200,000	79.56	20169	3440	22484	HR Aldrich Polymers	
PA1.7	POLYSTYRENE STANDARD, TYPICAL MW 200,000	78.62	20069	3530	19586	HR Aldrich Polymers	
PA1.8	POLYSTYRENE STANDARD, TYPICAL MW 200,000	78.20	19164	3767	18887	HR Aldrich Polymers	

Outlook and future directions

Despite the challenges, these methods provide valuable insights, and improvements are ongoing.

- Better contamination control protocols
- Expanded spectral databases
- Automation and complementary methods

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Thank you for your attention!

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