Technical Overview

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

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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.

Depending on how microplastics are produced, they can be classified as either primary or secondary microplastics.

- Primary microplastics are small plastic particles that are released directly into the environment through domestic and industrial effluents, spills, and sewage discharge, or indirectly released by runoff.
- Secondary microplastics are formed as a result of fragmentation of larger plastic particles that are already present in the environment. Fragmentation takes place due to ultraviolet (UV) radiation (photo-oxidation), mechanical transformation (e.g., by wave abrasion), and biological degradation by micro-organisms. These small plastic particles, often less than 5 mm in size, are pervasive across the world's oceans, rivers, soil, and air.

Vibrational spectroscopy techniques, such as Fourier-transform infrared (FTIR) spectroscopy and Raman spectroscopy, have commonly been used to chemically identify microplastics in environmental samples. These methods rely on the unique vibrational modes of chemical bonds in plastics, allowing researchers to differentiate between various types of polymers. However, these techniques can be complex and slow, which may limit their practicality for large-scale and real-time monitoring studies of environmental samples.

The Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system represents an innovative approach to imaging and spectral analysis. LDIR is useful in the context of microplastic identification and other applications that require the chemical characterization of materials. LDIR uses Quantum Cascade Laser (QCL) technology with rapid scanning optics to enable efficient and high-quality imaging and spectral data acquisition.

Depending on the level of suspended solids present in a sample, industry standard practice documents describe the steps required for the preparation of water samples for the identification and quantification of microplastic particles and fibers.[2–4] Regardless of the analysis technique used, the samples will eventually require a filtration step. This filtration step can be performed directly on gold-coated filters that are then inserted into the 8700 for the characterization analysis of microplastics by LDIR.[5] However, gold-coated filters are expensive, so aluminum-coated polyester filters (25 mm, 100/0 nm coating, 0.8 µm pore size) were evaluated as alternatives.

This technical overview outlines the performance of the 8700 LDIR and Agilent Clarity software in achieving accurate microplastics characterization on aluminum-coated filters (Figure 1). Attributes discussed in this technical overview include:

- Useability and handling of aluminum-coated filters
- Detection of particles on aluminum-coated filters by LDIR
- Repeatability of particle count, size, and identification data
- Particle size accuracy
- Identification of common microplastics

Figure 1. The Agilent 8700 LDIR Chemical Imaging System allows the high-speed routine analysis of microplastics on aluminum-coated polyester filters (25 mm, 100/0 nm coating, 0.8 µm pore size).

Usability and handling of aluminum-coated filters

Aluminum-coated filters are delicate, so careful handling of the filters is required. As shown in Figure 2, the filters should be transferred onto the filtration system using the tweezers that are supplied. Once the filters are in position, the filtration assembly must be secured using the clamp before the sample can be filtered. After filtration, the filter can be placed onto the elevated platform of the filter holder and secured using the brass retaining ring. The procedure is described more fully in another publication.[5]

Aluminum-coated filters are less prone to folding than gold-coated filters. They are also easier to position on the filter holder due to increased rigidity of the coating.

Filter pore size and microplastics loading from the sample will directly impact the filtration rate. Using an absolute ethanol fresh sample, a filtration rate of ~1 mL per second was achieved with the aluminum-coated filters (0.8 µm pore size) and a gentle pressure of 700 mbar (–30 kPa). No deformation to the filter was observed.

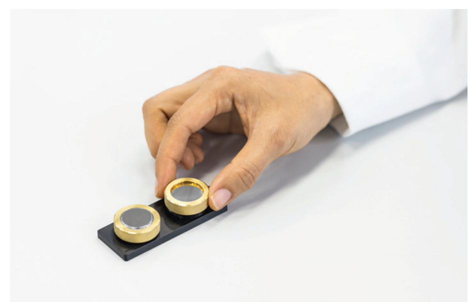
1 Place the filter using the supplied tweezers

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

Figure 2. Sample filtration equipment and steps for the preparation of microplastic samples on aluminum-coated filters, ready for analysis by Agilent 8700 LDIR.

Detection of particles on aluminum-coated filters

The particle detection capabilities of LDIR on aluminum-coated filters were assessed using an automated and manual method. Clear polystyrene microspheres (20 µm) were suspended in ethanol (10 mL). The mixture was then filtered through the aluminum-coated filter, as outlined in Figure 2.

The filter was transferred into the LDIR and the analysis was performed as follows:

- A 9 × 9 mm area of the filter was analyzed using the LDIR automated Particle Analysis workflow based on an IR image generated at 1,442 cm^{-1}.
- A high-magnification visible image of the same area was generated by LDIR, and the number of particles was counted manually.

As shown in Figure 3, 31 particles were detected using the automated Particle Analysis workflow, while only 30 were counted in the high-magnification visible image. The results show the excellent detection capability of LDIR of particles on aluminum-coated filters. The automated IR-image-based particle detection method provided a more comprehensive view of the actual number of particles present compared to the visible-image particle detection method, which is prone to error. As shown in Figure 3B, small particles are difficult to detect by visible imaging.

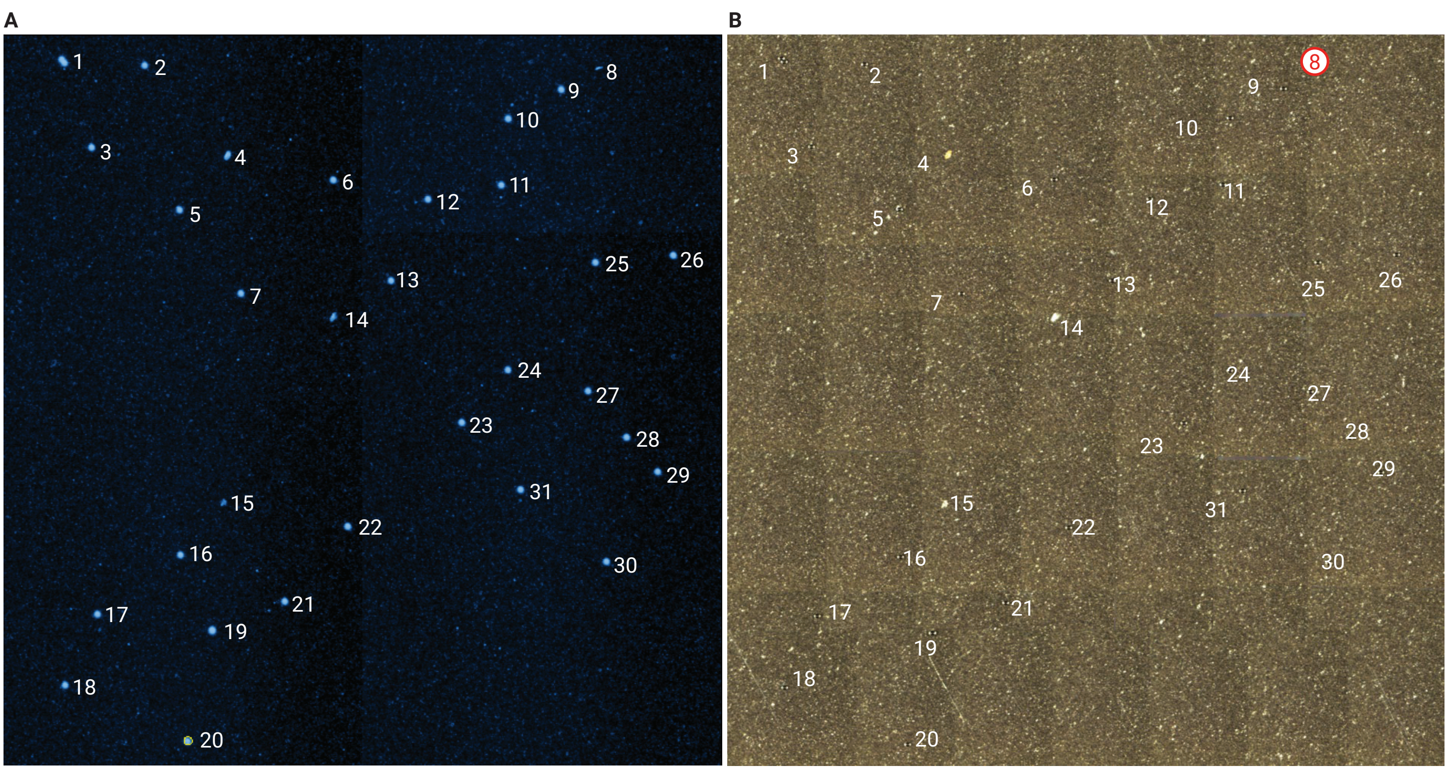


Figure 3. Particle detection capabilities of LDIR of particles on aluminum-coated filters. Both images were collected over the same area. (A) IR image at 1,442 cm^{-1} collected via the automated Particle Analysis workflow. (B) High magnification visible image showing where the missing particle (8, highlighted in red) should be located.

Repeatability of particle count, size, and identification

The automated Particle Analysis workflow within the Clarity software was used to evaluate aluminum-coated filters and LDIR repeatability in terms of particle count, size, and identification. This evaluation was performed by filtering 10 mL of mixed microplastics suspended in ethanol. A total of 10 measurements were conducted on a circular region of 9 mm in diameter, without moving the sample.

A total average of 407 particle counts were detected with < 1% variability between the 10 runs (Figure 4A). Particle count repeatability, based on size range and polymer identification, showed a similar performance, with <1% variation across the 10 runs (Figure 4B and 4C). These results show the reliability and accuracy of performing microplastics characterization on aluminum-coated filters by LDIR.

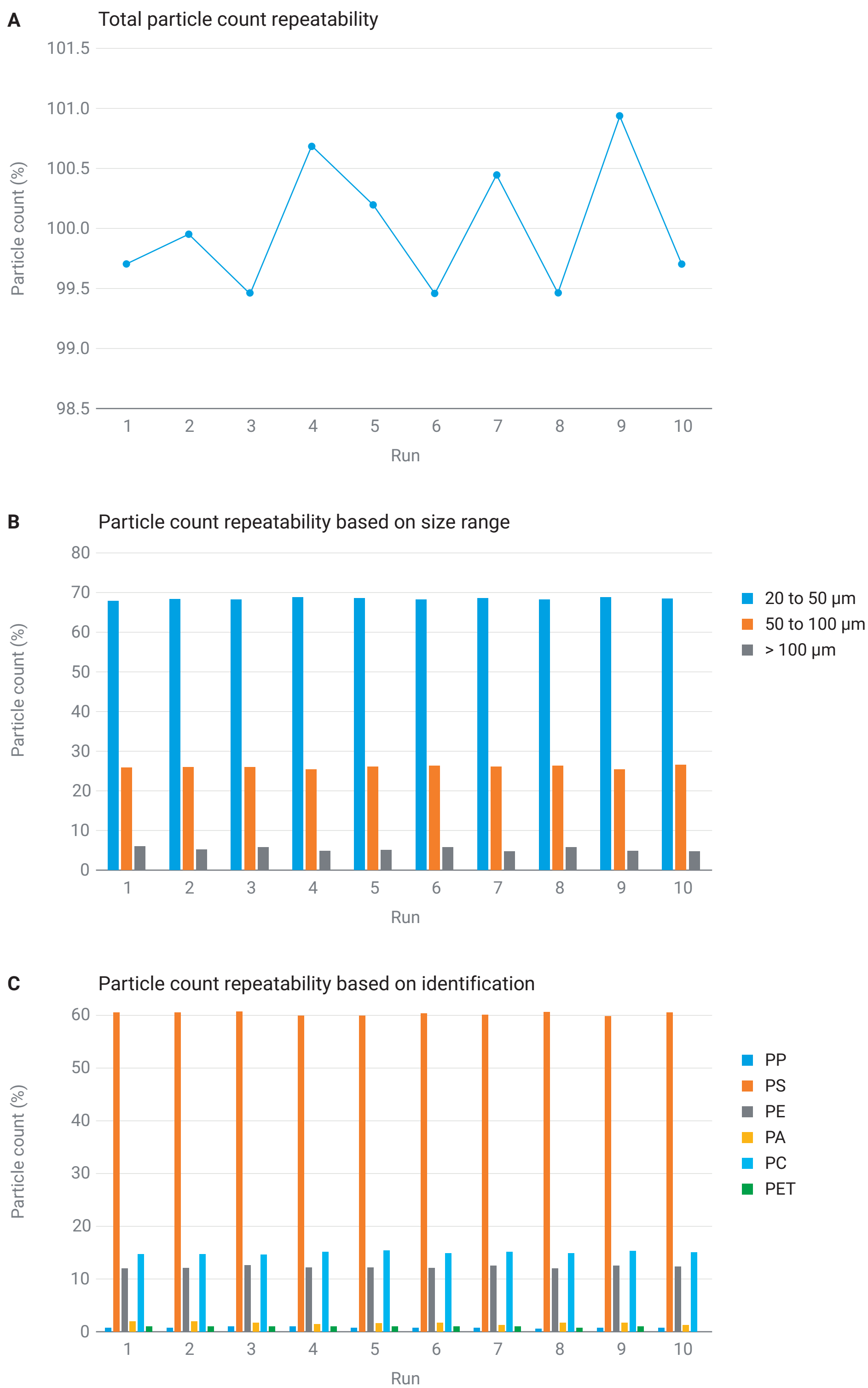


Figure 4. Repeatability of particle counts on aluminum-coated filters based on 10 runs using the LDIR automated Particle Analysis workflow. (A) Total particle counts repeatability. (B) Particle count repeatability based on size range. (C) Particle count repeatability based on identification.

Particle size accuracy

Since accurate particle sizing is essential for obtaining reliable and meaningful results in microplastics research, the accuracy of particle sizing data generated by LDIR using aluminum-coated filters was assessed.

NIST traceable 50 and 20 µm polystyrene latex beads were tested for particle size accuracy on an aluminum-coated filter. As shown in Figure 5, with the 50 µm beads, 37 particles were detected with an average size of 55.10 µm (standard deviation = 3.67 µm), while with the 20 µm beads, 223 particles were detected with an average size of 22.90 µm (standard deviation = 2.3 µm). These results suggest that accurate particle sizing with minimal variation can be achieved on aluminum-coated filters using the LDIR automated Particle Analysis workflow.

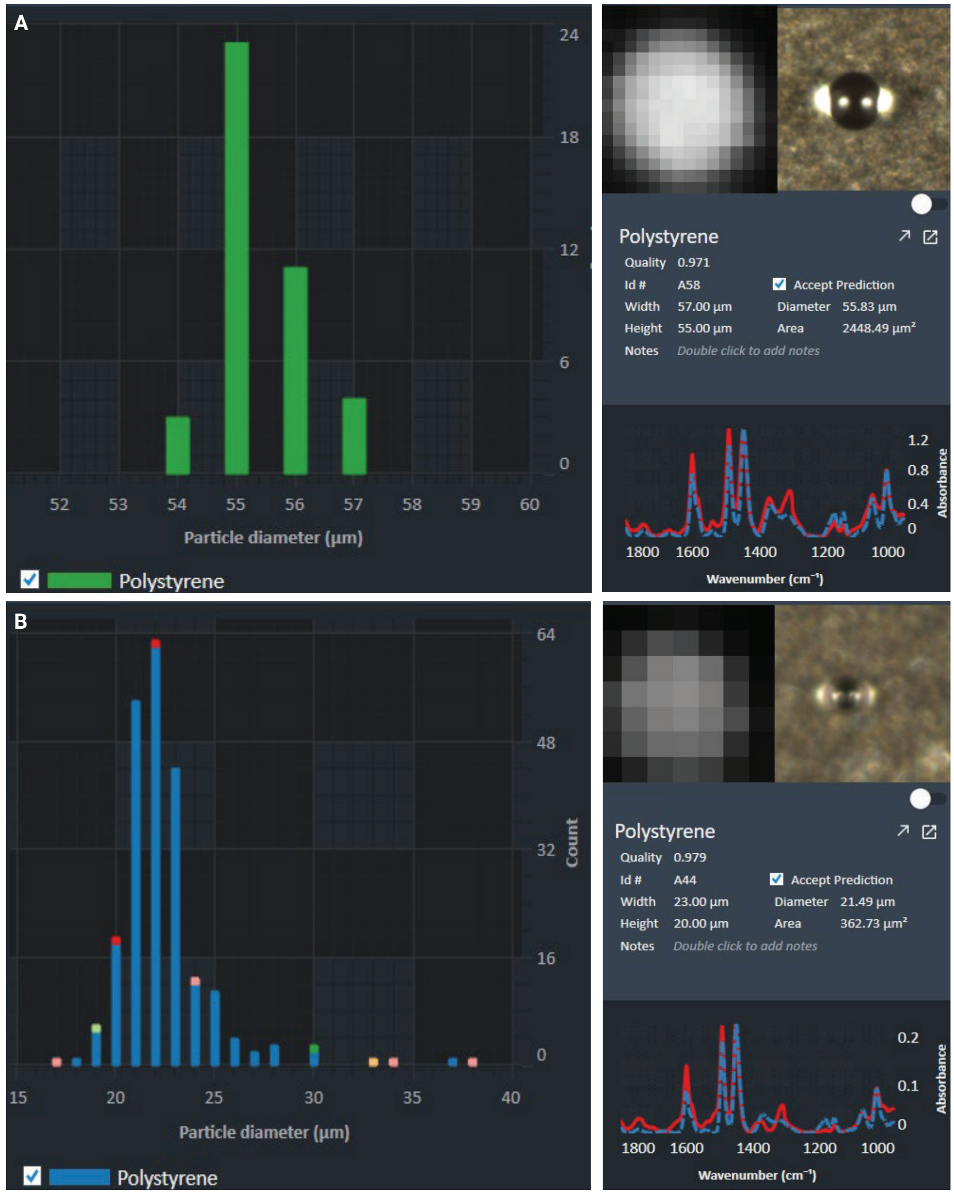


Figure 5. Particle size accuracy of LDIR on aluminum-coated filter using the automated Particle Analysis workflow. (A) 50 µm and (B) 20 µm NIST traceable polystyrene latex beads were tested.

Identification of common microplastics

To aid identification of the microplastics, the Clarity software includes a high-quality microplastics library for LDIR that contains spectral data of polymers and natural materials. The 8700 LDIR microplastics identification capabilities on aluminum-coated filters were examined for the four most common types of polymers (polystyrene, polypropylene, polyethylene, and polyethylene terephthalate).

Plastic materials in the micro range were generated from pellets using a metal file and suspended in ethanol (10 mL). The suspension was then filtered through an aluminum-coated filter using vacuum filtration glassware, as described in Figure 2. LDIR analyzed a selected area of each filter using the automated Particle Analysis workflow.

The classification range used for the identification of microplastics in a sample uses a hit quality index (HQI) score system, where a score of 1 represents the highest-quality result.

As shown in Table 1 and Figure 6, accurate identification of the four major types of microplastics was successfully achieved on aluminum-coated filters. The high HQI scores, which surpassed 0.8 for approximately 95 to 100% of the particles, confirm the accuracy of the method.

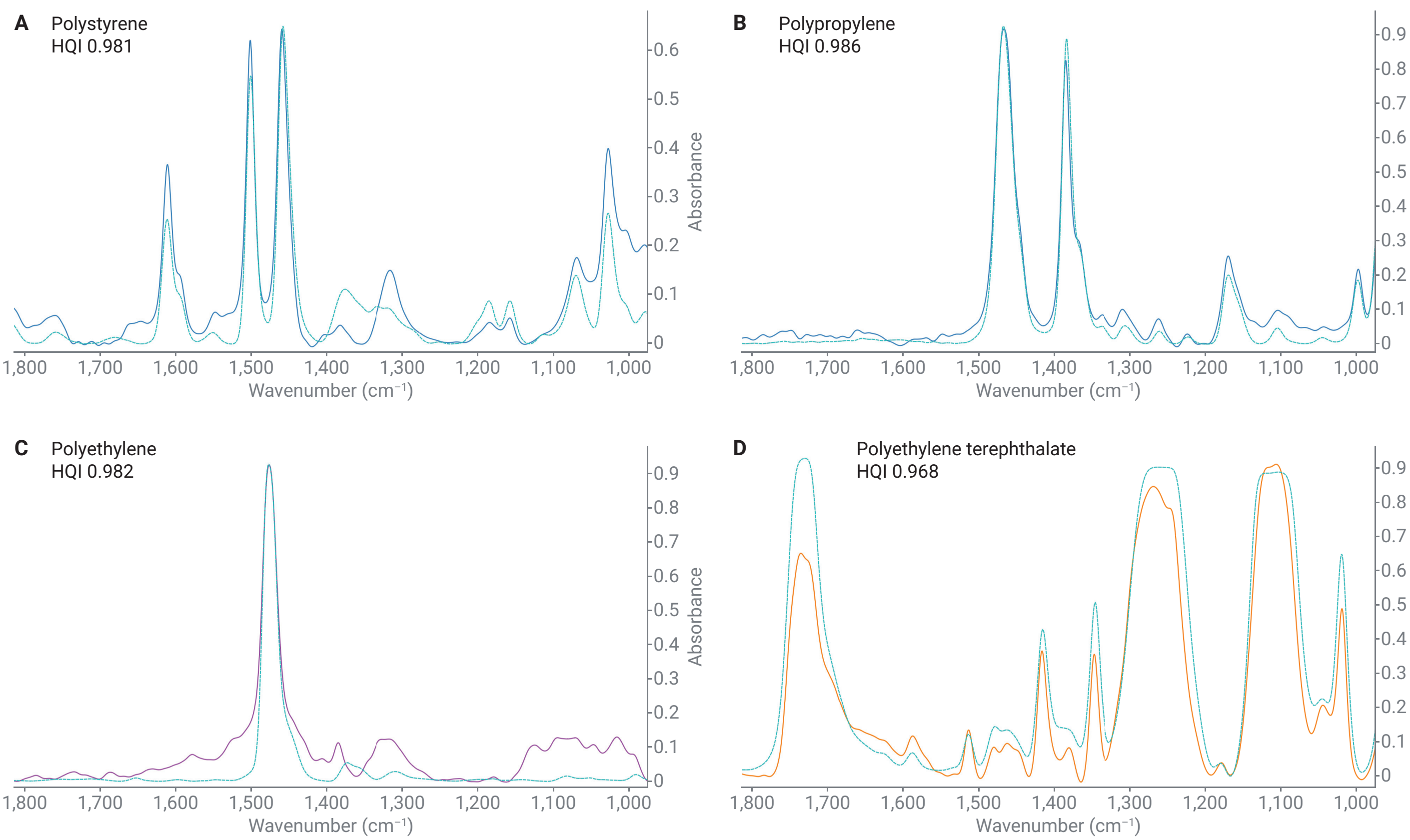
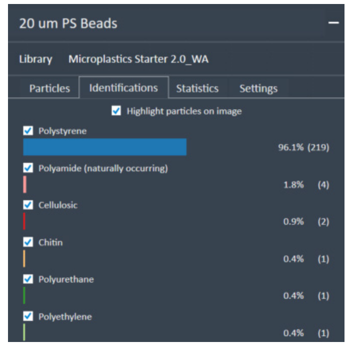
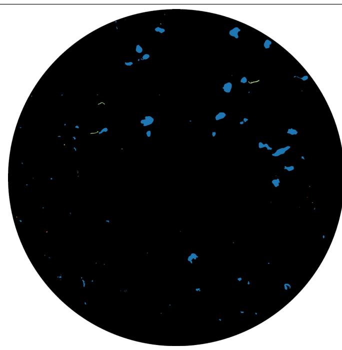
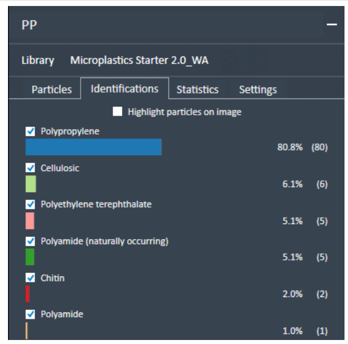
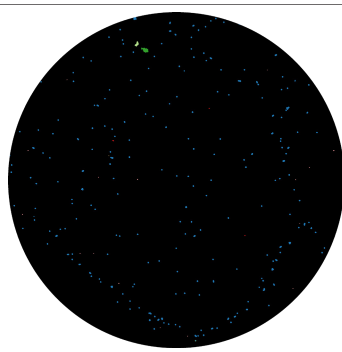
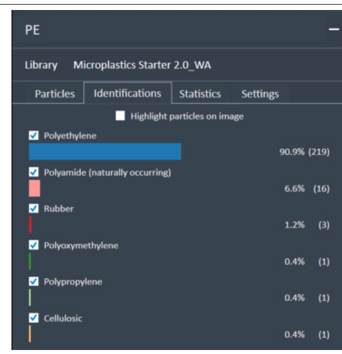
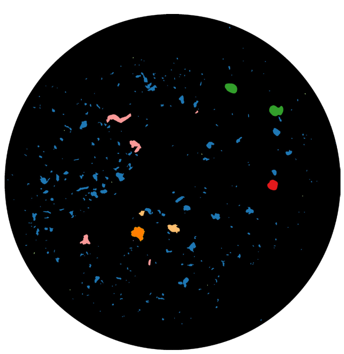
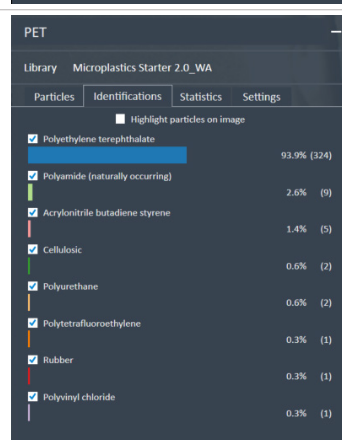


Figure 6. Example spectra of major types of microplastics measured on aluminum-coated filters using the automated Particle Analysis workflow. LDIR acquisition spectra are shown as solid lines, and matched library spectra are shown as pale green dashed lines.

Table 1. Identification of major types of polymers on aluminum-coated filters by the Agilent 8700 LDIR Chemical Imaging System.

Type of Polymer	Identification Image	Statistical Overview	Quality of the Data
Polystyrene			Total number of particles: 228 > 0.8 HQI: 227 (99.56%) < 0.8 HQI: 1 (0.44%)
Polypropylene			Total number of particles: 99 > 0.8 HQI: 96 (96.97%) < 0.8 HQI: 3 (3.03%)
Polyethylene			Total number of particles: 241 > 0.8 HQI: 241(100%) < 0.8 HQI: 0 (0%)
Polyethylene terephthalate			Total number of particles: 345 > 0.8 HQI: 330 (95.65%) < 0.8 HQI: 15 (4.35%)

Conclusion

This work has demonstrated the suitability of aluminum-coated polyester filters for the characterization of microplastics using the Agilent 8700 LDIR chemical imaging system. The filters enable the efficient sample preparation (filtration) of environmental and water samples, as well as offering significant cost savings compared to gold-coated filters.

The aluminum-coated filters provided accurate microplastics characterization in terms of particle detection, repeatability of particle counts, size accuracy, and identification.

Direct on-filter microplastics analysis by the 8700 LDIR minimizes sample handling compared to other techniques, reducing the potential for sample contamination and improving sample throughput. The automated Particle Analysis workflow of LDIR provides accurate characterization of microplastics in different matrixes using cost-effective filters. All of these factors improve sample throughput, enabling laboratories to characterize higher numbers of samples in less time. Large-sample studies are vital for understanding the full environmental and health impacts of microplastic pollutants and for developing strategies for reducing the effects of microplastic pollution.

References

1. Gerresten, I. Plastic Pollution is One of the Defining Legacies of Our Modern Way of Life, But It Is Now So Widespread It Is Even Finding Its Way into Fruit and Vegetables As They Grow., *BBC*, **2023**, accessed November 2023, https://www.bbc.com/future/article/20230103-how-plastic-is-getting-into-our-food
2. ASTM D8332, Standard Practice for Collection of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers, ASTM International, Philadelphia, PA, USA, **2020**, accessed November 2023, https://www.astm.org/d8332-20.html
3. ASTM D8333, Standard Practice for Preparation of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers Using Raman Spectroscopy, IR Spectroscopy, or Pyrolysis-GC/MS, ASTM International, Philadelphia, PA, USA, **2020**, accessed November 2023, https://www.astm.org/d8333-20.html
4. Draft Microplastics in Drinking Water Policy Handbook, The Division of Drinking Water, State Water Resources Control Board, State of California, Nov. 10, **2021**, accessed November 2023, https://www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/documents/microplastics/mcrplsts_plcy_drft.pdf
5. Best Practice for On-Filter Analysis of Microplastics Using the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System, *Agilent Technologies white paper*, **2023**, publication number 5994-5467EN.

Further information

- Agilent 8700 LDIR Chemical Imaging System
- Agilent Clarity Software
- Microplastics Technologies FAQs
- Microplastics Analysis in Water

www.agilent.com/chem/8700-ldir

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