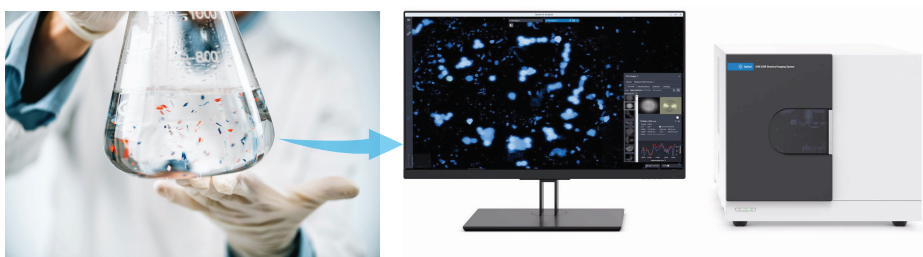**Agilent**

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Performance Attributes of the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System

For microplastics characterization

Introduction

Contamination of waterways, soil, air, and drinking water by microplastics is gaining significant public interest, due largely to its emergence as an environmental threat. Researchers are now working towards standardized analytical solutions to best characterize these small particles in terms of chemical identity, size, shape, and total mass.

Challenges for reliable and fast microplastics characterization are only partially addressed with the current techniques. For example, Fourier transform infrared (FTIR) point-mapping microscopes require small apertures for this work. The small aperture degrades the signal-to-noise ratio, and each microplastic particle requires more than one minute to analyze. FTIR array microscopes and Raman microscopes are also slow for this type of analysis. Laser direct infrared (LDIR) addresses the speed and workflow constraints of traditional vibrational spectroscopy. It overcomes some of the key limitations of FTIR systems, such as eliminating the need to collect data in empty spaces. This can be fully automated and can result in significantly faster analysis times.

The Agilent 8700 LDIR chemical imaging system provides a sophisticated new approach to chemical imaging and infrared spectral analysis. Using quantum cascade laser (QCL) technology and designed to be used by experts and nonexperts alike, the 8700 LDIR provides simple, highly automated operation. This system is ideally suited to the analysis of microplastic particles in environmental samples (such as water). The 8700 LDIR can analyze more samples, in greater detail, in minutes versus hours. The automated workflow within the software reduces costs and potential errors and simplify microplastics characterization process delivering fast and reliable solution.

This technical overview describes the easy automated workflow of the 8700 LDIR in characterizing microplastics. It also evaluates the performance of the 8700 LDIR in terms of particle size accuracy and precision, as well as size-ranging precision and particle count repeatability.
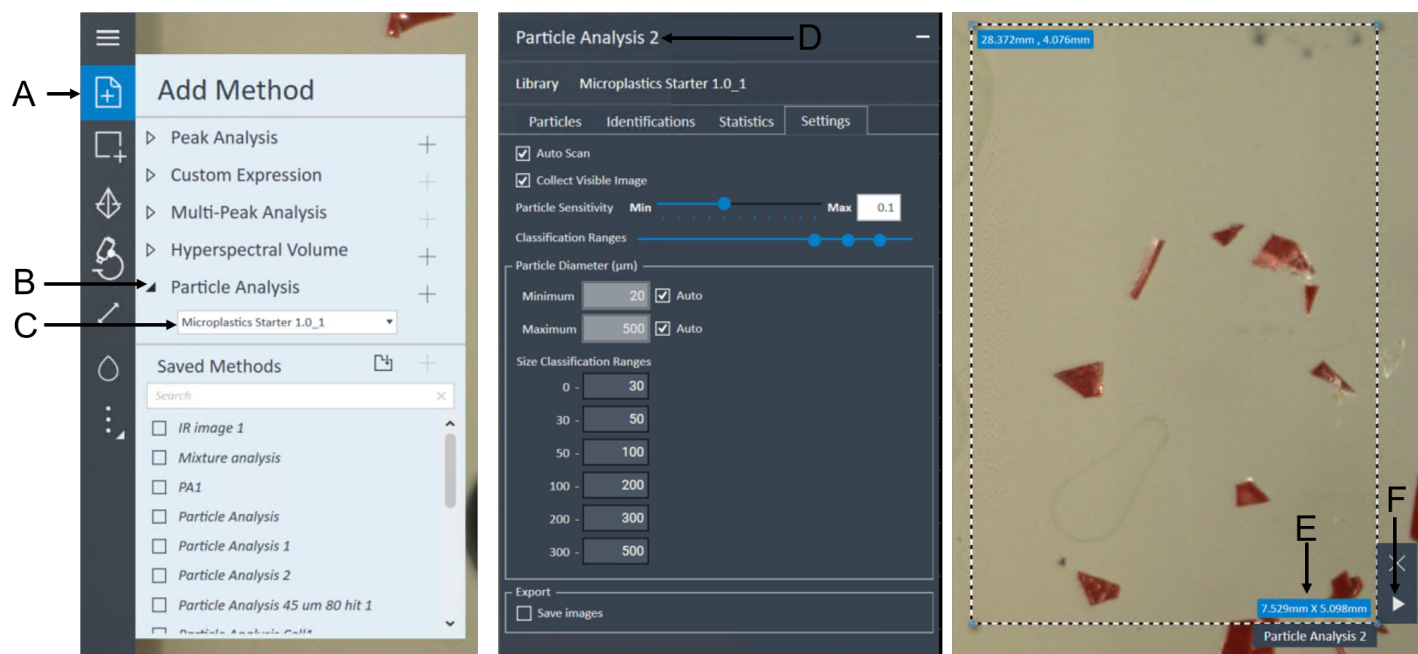
Particle analysis automated workflow

Agilent Clarity software is an intuitive software allowing easy analysis of microplastics. The automated workflow is well suited to identify and analyze particles. The steps to create and run particle analysis starts with inserting the prepared sample into the 8700 LDIR then focusing the infrared (IR) laser on the sample. To perform the automated workflow, particle analysis method can be selected with the desired library to perform the analysis as described in Figure 1. The automated workflow allows users to define certain parameters in the setting depending on the customer needs such as:

– **Auto Scan:** when enabled, the software will automatically proceed through the entire workflow or the user can preview the found particles and adjust the setting accordingly.

– **Collect visible images:** when enabled, the software will automatically collect visible image for each particle.

– **Particle sensitivity:** this will adjust particle sensitivity detection; increased sensitivity will detect fainter and smaller particles in the image.

– **Classification ranges:** customers can choose the range in which particles are classified based on the quality of identification.

– **Particle diameter:** to set the minimum and maximum particle diameter to be detected.

– **Size classification range:** this will allow users to set a maximum particle size to be categorized into the bucket.

After defining these parameters, an area can be selected, and the particle analysis workflow starts by hitting the play button (Figure 1).


Figure 1. Agilent Clarity software automated particle analysis workflow. (A) Addition of new method. (B) Particle Analysis selected. (C) Library to be used in the analysis can be selected from the dropdown menu. (D) Particle Analysis workflow settings. (E) Area selected to analysis. (F) Play button to start the analysis.

Performance attributes

Particle size accuracy and precision

Particle size accuracy and precision of the 8700 LDIR were assessed using the automated particle analysis workflow and validated using a third-party instrument (the Digital Microscope Leica DVM6). 10 mg of clear polyethylene microspheres (0.96 g/cc, 38 to 45 μm, 5 g) were suspended in 100 μL ethanol, the resulting particle/ethanol suspension (4 × 10 μL) was transferred onto two infrared reflective glass slides (7.5 × 2.5 cm; MirrIR, Kevley Technologies, Ohio, USA) using pipettes. This step was performed in a laminar airflow fume hood to minimize contamination by air. Particle analysis workflow was performed three times on the same area with by collecting visible images (Figure 2).
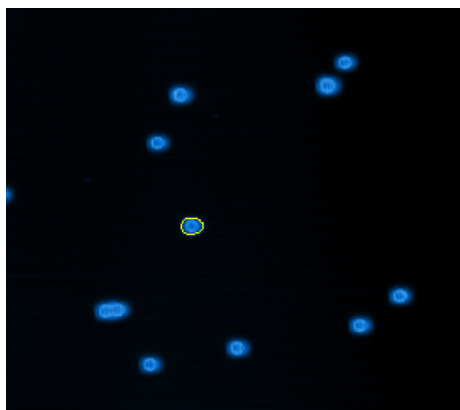
Figure 2. Clear polyethylene microspheres image taken on the Agilent 8700 LDIR chemical imaging system.

Particle size accuracy of A3 to A9 on the 8700 LDIR was found to be within the range of certified values which is specified as >90% of particles should be within 38 to 45 μm (Table 1).

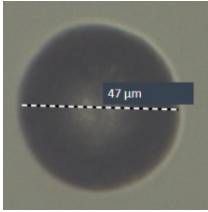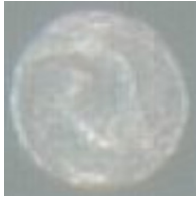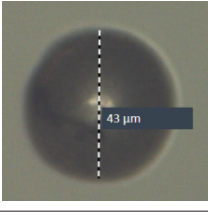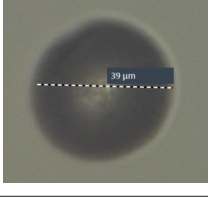This result agreed with the digital microscope data as shown in Table 2. Particle size of A2 was found to be outside the range specified on both LDIR and the digital microscope (Table 2). Particle size precision was determined based on the standard deviation of the diameter obtained from the three scans, excellent repeatability and precision was achieved on the LDIR system ranging from 0.03 to 0.10 μm (Table 1).

Table 1. Clear polyethylene microspheres measured using the Agilent 8700 LDIR chemical imaging system.

Particle ID	A2	A3	A4	A5	A6	A7	A8	A9	A10
Repeat 1	47.03	43.18	42.08	41.61	40.96	41.15	40.1	38.98	38.93
Repeat 2	46.99	43.07	42.05	41.55	40.9	40.44	40.06	39.01	39.25
Repeat 3	47.11	43.21	42.08	41.65	40.45	40.37	40.17	38.97	39.29
Average	47.04	43.15	42.07	41.60	40.77	40.65	40.11	38.99	39.16
Standard Deviation	0.06	0.07	0.02	0.05	0.28	0.43	0.06	0.02	0.20

Table 2. Particle sizes from the Agilent 8700 LDIR chemical imaging system, validated using a third-party instrument. The dashed line is a Clarity software feature that allows users to measure the diameter of particles manually.
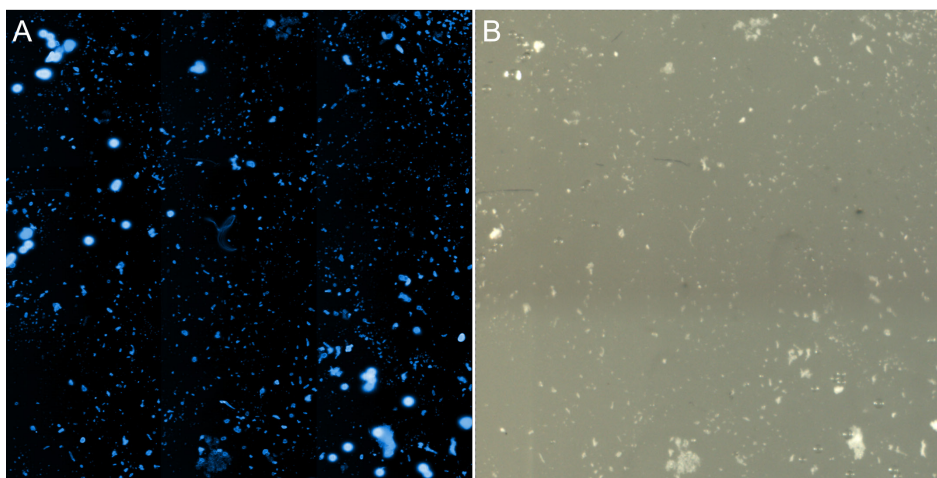
Particle	Agilent 8700 LDIR Chemical Imaging System	Size (μm)	Digital Microscope Leica DVM6	Size (μm)
A2		47.0		46.0
A3		43.2		42.5
A9		39.0		39.5

Size ranging precision and particle count repeatability

The 8700 LDIR size ranging precision and particle count repeatability were evaluated using a microplastic mixture sample. The sample contained different types, sizes, and shapes of microplastics dispensed on two infrared reflective glass slides (7.5 × 2.5 cm; MirrIR, Kevley Technologies, Ohio, USA). To perform this analysis, an area of 9 × 9 mm was scanned six times using the automated workflow with visible images collections (Figure 3).

The area scanned contained particles ranging from 20 to 2,000 µm in size. Based on standard deviation of six scans, the 8700 LDIR exhibited excellent size ranging precision of 10, 7, 3, and two particles for buckets 0 to 30, 30 to 50, 50 to 100, and 100 to 200 µm, respectively. Larger particle size buckets (200 to 300, 300 to 500, and 500 to 5,000 µm) showed excellent repeatability across the six scans, with 0% deviation (Table 3). The 8700 LDIR showed remarkable particle count repeatability from six scans, which was found to be 2.1%. This was calculated based on the average particle count and the standard deviation from six scans which were found to be 871 and 18 particles respectively (Table 3).

Figure 3. Area scanned for size ranging precision and particle count repeatability study. (A) IR image at 1,800 cm⁻¹. (B) Visible image obtained via Agilent 8700 LDIR chemical imaging system.

Table 3. Size ranging precision and particle count repeatability using the Agilent 8700 LDIR chemical imaging system.

Size Ranges (µm)	Automated Workflow R1	Automated Workflow R2	Automated Workflow R3	Automated Workflow R4	Automated Workflow R5	Automated Workflow R6	Average	Standard Deviation
0 to 30	112	118	112	119	106	88	109	10
30 to 50	393	406	402	404	401	386	399	7
50 to 100	291	290	284	284	286	286	287	3
100 to 200	64	65	68	68	68	62	66	2
200 to 300	7	7	7	6	7	7	7	0
300 to 500	3	3	3	3	3	3	3	0
500 to 5,000	1	1	1	1	1	1	1	0
Total	871	890	877	885	872	833	871	18

Key features and applications of the Agilent 8700 LDIR chemical imaging system

- Enables expert spectroscopists and trained technicians to analyze and characterize samples rapidly and accurately

- Highly automated workflow for characterizing surface distribution of components in pharmaceutical tablets

- Automated chemical imaging workflow for microplastics in environmental samples and drinking water

- Ability to survey and image large sample areas and then interrogate smaller areas of interest in more detail without changing any optics

- Full control using Agilent Clarity software

- Change the field of view from microns to centimeters or the pixel size from 1 to 40 µm.

- Rapidly identify unknowns using either commercial or custom libraries via ATR capabilities

- Obtain relative quantitative information of sample constituents without complex method development

- Simple load-and-go methods require minimal instrument interaction, and small footprint saves laboratory bench space

Conclusion

The Agilent 8700 LDIR is a fast, automated solution for smaller microplastic identification, size measurement, semiquantitation, and report generation. The 8700 LDIR has been shown to exhibit excellent performance in terms of particle size accuracy and precision, as well as size-ranging precision and particle count repeatability. Therefore, it is a first-rate solution for rapid and accurate characterization of microplastics in environmental samples.

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