

Navigating Global Microplastics Regulations in Drinking Water with Vibrational Spectroscopy

Ensuring accurate and reliable microplastics characterization with the 8700 LDIR



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Introduction

Access to clean and safe drinking water is a fundamental human right and a critical public health priority. With growing concerns about environmental pollution, emerging contaminants such as microplastics have become a major focus for regulatory bodies worldwide. Microplastic particles, originating from sources such as industrial waste, packaging, and everyday consumer products, have been detected in diverse water sources, raising concerns about both their potential health risks and environmental impact.

The accurate and reliable characterization of microplastics presents significant analytical challenges due to the diversity in size, shape, and composition of these materials. Particles range from less than 20 μm to visible fragments and can exist in various forms including spheres, fibers, and irregular shapes. Microplastics are typically composed of various types of polymer, each with distinct physical and chemical properties and unique degradation histories. Because of this complexity, analyzing microplastics requires advanced analytical techniques.

To address these challenges, regulatory bodies and standardization organizations have actively been developing guidelines and testing methods to monitor and control microplastics contamination in drinking water. The analytical techniques used to characterize microplastics are critical in achieving accurate and reliable outcomes when studying the impact of this emerging class of contaminant. The primary techniques used to analyze microplastics fall into two categories:

- Thermo-analytical methods such as thermal extraction desorption gas chromatography-mass spectrometry (TED-GC/MS) and pyrolysis-GC/MS, which provide information about polymer type and mass¹
- Vibrational spectroscopy methods including Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy, which provide information about polymer type, number of particles, and size

There has been an ongoing effort for several years to standardize methods for microplastic analysis using vibrational spectroscopy. These standard methods provide structured approaches that help to ensure consistent, accurate, and reproducible microplastics analysis across different laboratories and regulatory environments.

Three such regulatory frameworks have been established by the following bodies:

- Commission Delegated Decision (EU) 2024/1441:
 Supplementing Directive (EU) 2020/2184 of the
 European Parliament and of the Council by laying down a methodology to measure microplastics in water intended for human consumption.²
- California Water Boards: Policy handbook establishing a standard method of testing and reporting of microplastics in drinking water, August 9, 2022.³ The policy handbook was developed in response to State of California legislation (SB1422) requiring monitoring for microplastics. As noted in the introductory text, the method is designed to "[set] forth the requirements for conducting monitoring and reporting of microplastics in drinking water." Attachment C to that document (SWB-MP1-rev1) is a Standard Operating Procedure that describes the method as relevant to IR techniques.⁴
- ISO/DIS 16094-2: Water Quality Analysis of Microplastic in Water, Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water (under development).⁵

ASTM WK87463 (New Test Method for Spectroscopic Identification and Quantification of Microplastic Particles in Water Using Infrared (IR) Spectroscopy) is also under development. As this method has not yet been published, it is not covered by this white paper.⁶

Table 1 provides an overview of the three standardized methods for microplastic analysis, detailing their specifications for sample type, target polymers, size classification, and permitted analytical techniques.

Table 1. Summary of microplastic analysis requirements across three regulatory frameworks using vibrational spectroscopy.

Method	EU 2024/1441	California Water Boards	ISO/DIS 16094-2
Type of Sample	Water intended for human consumption	Treated drinking water	Ultrapure waterWater intended for human consumptionRaw groundwaters
Polymers of Interest	- Polyethylene (PE) - Polypropylene (PP) - Polyethylene terephthalate (PET) - Polystyrene (PS) - Polyvinyl chloride (PVC) - Polyamide (PA) - Polyurethane (PU) - Polymethyl methacrylate (PMMA) - Polytetrafluoroethylene (PTFE) - Polycarbonate (PC)	Particle of any composition with a continuous polymer surface coating of any thickness or Particle of any composition with a polymer content of ≥ 1% by mass	- Polyethylene (PE) - Polypropylene (PP) - Polyethylene terephthalate (PET) - Polycarbonate (PC) - Polystyrene (PS) - Polytetrafluoroethylene (PTFE) - Polyvinyl chloride (PVC) - Polyamide (PA) - Polymethyl methacrylate (PMMA) - Polyurethane (PU)
Results	Number of particlesPolymer ID for each particleParticle sizeParticle shape	- Number of particles - Polymer ID for each particle - Particle size - Particle morphology - Particle color	- Number of particles - Polymer ID for each particle - Particle size
Minimum Measurable Particle Size	20 μm (particles and fibers)	– 20 μm (Raman) – 50 μm (IR)	– 1 to 10 μm (Raman) – 15 to 20 μm (IR)
Maximum Measurable Particle Size	- 5 mm (particles) - 15 mm (fibers)	5 mm	5 mm
Analytical Techniques	Vibrational spectroscopy methods such as μ-FTIR, μ-Raman, or equivalent variations such as quantum cascade laser infrared microscopy (QCL-IR).	- IR spectroscopy can include, but is not limited to, FTIR, Laser Direct Infrared (LDIR) Imaging, optical-photothermal IR (Ο-PTIR), and other techniques capable of measuring microplastic particles as small as 50 μm - Raman spectroscopy	- IR apparatus coupled to a microscope such as FTIR or QCL-IR microscopy (Ref. Annex F) - Raman coupled to a microscope

Quantum cascade laser infrared microscopy

Quantum cascade laser infrared microscopy (QCL-IR) is a well-established technology that has been used extensively for microplastics analysis. This white paper demonstrates how an Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system (Figure 1), which uses a QCL and rapidly scanning optics, aligns with the requirements of the standardized methods for microplastics.



Figure 1. The Agilent 8700 LDIR chemical imaging system enables the rapid routine analysis of microplastics in drinking water, providing data on the number of particles present in the sample, their size, and their chemical composition.

Requirements of regulatory methods

Sample volume

The various regulatory methods specify different sample volumes and collection procedures for assessing microplastics levels in water.

The EU 2024/1441 method requires a minimum sample volume of 1,000 L, whereas the California Water Boards method follows ASTM-approved procedures, which recommend collecting sample volumes of up to 1,500 L. However, the State Water Board is evaluating the suitability of an alternative sampling methodology to eliminate the possibility of contamination during sample collection and the need for a sample blank.3 The EU method specifies that water should be collected using filtration, where water is passed directly through filters at the sampling site to minimize contamination from intermediate collection or storage vessels.² ASTM procedures require in-line sieving of water, assuming that samples can be processed through further sieving and filtration in the laboratory, typically in relatively low volumes of up to 20 L. This includes samples collected through in-line sieving into containers, which can then undergo further processing.4

ISO/DIS 16094-2 mandates testing the entire sample volume by default. However, if a high number of particles is expected, the filtered volume should be adjusted to ensure representative results while maintaining the ability to analyze the filter.⁵

Microplastics extracted from large volumes of filtered river and lake water have successfully been analyzed using LDIR, producing precise results, as long as the samples are not excessively concentrated with particles.⁷

Filtration

As mentioned in the previous section, large volumes of samples should be collected using filtration by passing through filters. If a sample analysis cannot be done directly on the collection filter, the particulate materials may be re-suspended in liquid and transferred to an alternative support for subsequent analyses.

Typically, a representative sample or subsample of particles should be prepared either on a filter surface or on a glass slide.

For the direct analysis of microplastics, LDIR can analyze samples on two types of substrates as follows:

- Gold- or aluminum-coated filters: Samples containing microplastics (~ 1,000 particles) can be filtered through 25 mm diameter gold- or aluminum-coated filters (Figures 2A and 2B) using a vacuum filtration apparatus (as shown in Figure 3). Compared to transferring particles onto slides, this approach offers more sample representation, easy sample preparation, reduced contamination, and the ability to analyze two filters sequentially. Agilent Clarity software allows the entire filter to be selected for analysis (~ 16 mm). However, if subsampling is required, the software allows the analysis of a user-defined area. The area-selection tool allows users to define the analysis region in a circular or rectangular shape.
- Low-emissivity (low-e) IR reflective slide: For the analysis of a high number of particles (~ 5,000 particles) by LDIR, samples can be prepared for analysis on a large-area, low-emissivity (low-e) IR reflective slide. A 25 x 75 mm slide (MirrIR, Kevley Technologies) is shown in Figure 2C.



Figure 2. (A) Gold- and (B) aluminum-coated filters on holders, (C) low-e IR reflective slide. Samples can be prepared on either substrate, ready for direct analysis using an Agilent 8700 LDIR chemical imaging system.

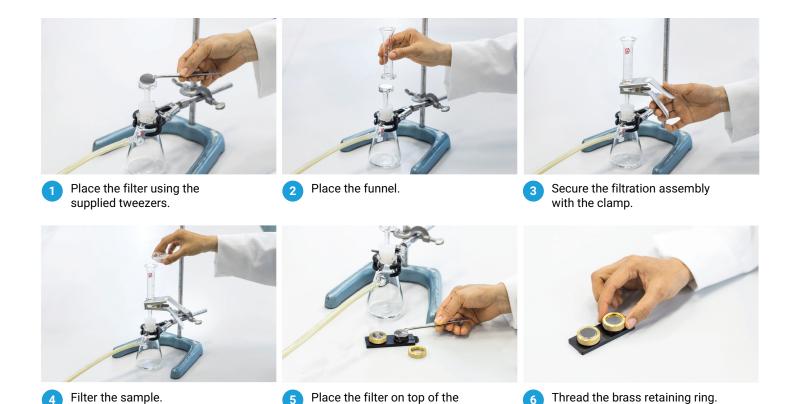


Figure 3. Sample filtration equipment and an outline of the steps for the preparation of microplastic samples on aluminum-coated filters, ready for analysis by an Agilent 8700 LDIR chemical imaging system.

raised platform.

Procedural blanks

To ensure accurate and reliable analytical results, strict quality control (QC) measures must be implemented, including the use of blanks to assess contamination from equipment, reagents, and the environment. For example, EU 2024/1441 requires at least 10 procedural blanks per filter type to establish background levels (mean (μ) and standard deviation (σ)) of microplastic contamination. The methodology also mandates periodic monitoring of procedural blanks, with investigations required if contamination exceeds the mean background contamination (μ) by more than three times the standard deviation.

The California Water Boards protocol requires that the laboratory must analyze at least seven laboratory-fortified blanks (LFBs), spiked with particles > 50 mm before conducting the analysis. Average recovery efficiency by visual microscopy of particles (> 212 μ m) must be 50%, with a precision of 40% RSD. A laboratory reagent blank sample (LRB) must also be analyzed, and the results must be less than the minimum reporting level (MRL).

ISO/DIS 16094-2 requires an analytical control blank for each sample sequence, ensuring identical conditions (operator, equipment, environment, and time frame) as the test samples. Also, the laboratory must determine a reporting limit (RL) for a claimed particle size by analyzing a minimum of 10 laboratory blanks.

The specially designed filter holder for the 8700 LDIR features two positions (i) and (ii), allowing sequential measurements of the blank and sample (Figure 4). This enables automatic characterization for each filter, streamlining QC in microplastics analysis.



Figure 4. The Agilent 8700 LDIR filter holder features two positions for sequential measurement of the blank and sample, enabling robust QC in microplastics analysis.

Minimum and maximum particle size

It is necessary to check the accuracy of any particle-sizing data generated by the analytical method used for the analysis of microplastics. The three regulatory frameworks summarized in Table 1 specify a minimum detectable particle size of 10 to 20 µm using IR spectroscopy.

To demonstrate the performance of the 8700 LDIR, 10, 20, and 50 μm NIST-traceable polystyrene latex beads were measured on a low-e slide. As shown in Figure 5, particles were detected using the automated Particle Analysis workflow within the Clarity software. The method also correctly identified the type of particles, with a hit quality index (HQI) of > 0.95, demonstrating the characterization capabilities of LDIR for particles as small as 10 μm . Additional details on particle sizing using gold- and aluminum-coated filters are available in other publications.

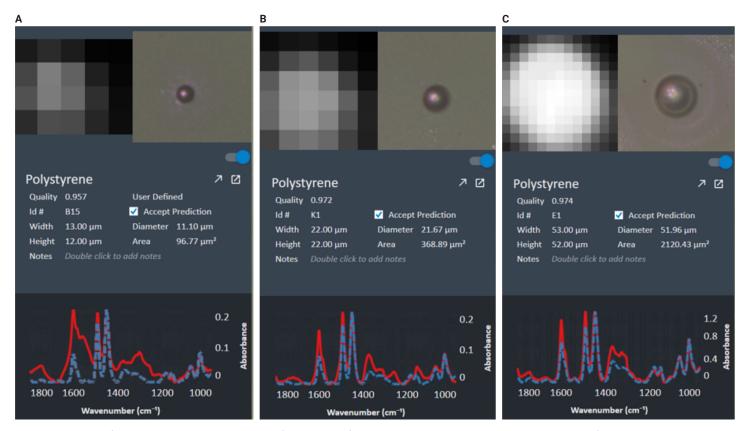


Figure 5. Examples of 10, 20, and 50 µm polystyrene beads (NIST-traceable) measured using the automated Particle Analysis workflow on an Agilent 8700 LDIR chemical imaging system.

As stated in Table 1, the maximum required measurement size across the three regulatory frameworks is 5 mm for particles and 15 mm for fibers per EU 2024/1441. While LDIR analyzes particles up to 500 µm, larger particles can be detected visually and measured using an Agilent Cary 630 FTIR spectrometer fitted with a diamond attenuated total reflectance (ATR) module (Figure 6). A fast and simple method for identifying plastic debris in this size range is reported elsewhere. 10

Size classification

Classifying microplastics based on their size is crucial for understanding their distribution and potential environmental impact. Different regulatory frameworks have established specific size classification methods to standardize microplastic analysis.

- According to EU 2024/1441, particles are categorized into five area-equivalent diameter ranges: 20 to 50 μm, 50 to 100 μm, 100 to 300 μm, 300 to 1,000 μm, and 1,000 to 5,000 μm.
- The California Water Boards method defines size fractions as < 50 μ m, 50 to 212 μ m, 212 to 500 μ m, and > 500 μ m by IR spectroscopy.
- The ISO/DIS 16094-2 method classifies microplastics not only by size but also by their material composition such as PE, PP, PU, etc. Its size classification ranges include 1 to 5 μm, 5 to 10 μm, 10 to 20 μm, 20 to 50 μm, 50 to 100 μm, 100 to 500 μm, and 500 to 5,000 μm.



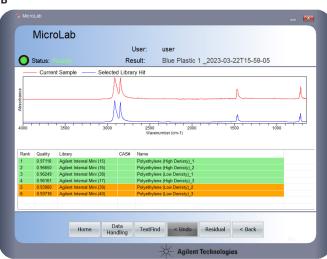


Figure 6. (A) Agilent Cary 630 FTIR spectrometer coupled with a diamond ATR module. (B) Example of Cary 630 FTIR qualitative analysis spectral data of plastic debris (red traces) and library hits (blue traces). The table shows the hit quality, library used, and the hit name for each sample. Color-coding the results based on the HQI score can be used to define confidence levels, helping users to interpret the results and reducing oversights that may lead to errors.

The 8700 LDIR instrument's automated Particle Analysis workflow allows flexible size classification by adjusting classification ranges before analysis. These settings can be predefined and customized to align with specific laboratory needs or regulatory guidelines. Throughout the analysis, the particle count within each size category is continuously updated, and the final classification results are available for review upon completion, ensuring a comprehensive understanding of microplastic size distribution, as shown in Figure 7.

Identification-libraries and polymers of interest

Vibrational spectroscopy uses absorption bands generated by intramolecular vibrational modes to distinguish organic and mineral materials by comparing their spectra with reference libraries. As outlined in Table 1, regulatory frameworks require accurate identification of major polymer types while ensuring differentiation from natural substances such as proteins, minerals, and cellulosic materials.

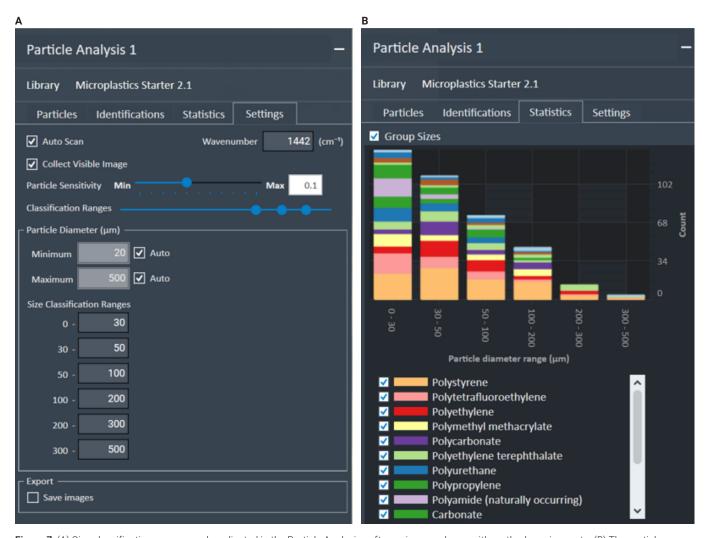


Figure 7. (A) Size classification ranges can be adjusted in the Particle Analysis software in accordance with method requirements. (B) The particle diameter range (μ m) versus count, based on the selected size classification range, is automatically generated after analysis.

The Clarity software includes a high-quality microplastics spectral library (Microplastics Starter 2.1 library), which includes both major polymers of interest and natural materials. ^{11,12} As demonstrated in Figure 8, LDIR provides

accurate identification for all polymers specified in the regulatory methods, achieving HQI scores between 0.913 and 0.991 (a HQI score between 0.85 and 0.99 indicates high confidence, with 1 representing the highest quality match).

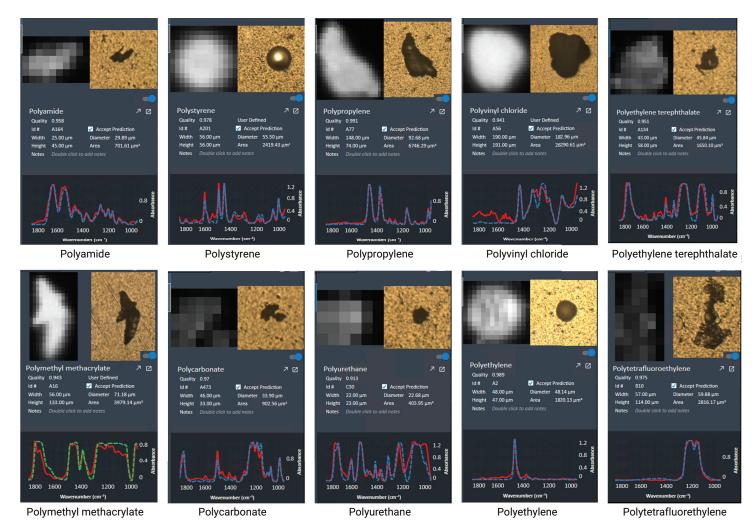


Figure 8. Examples of major types of microplastic polymers analyzed on a gold-coated filter using the automated Particle Analysis workflow on an Agilent 8700 LDIR chemical imaging system.

LDIR also exhibits excellent performance in identifying non-priority materials such as naturally occurring polyamides and cellulosic substances, as shown in Figure 9.

Matching within the workflow occurs in real time, as soon as the spectrum is acquired, significantly reducing analysis time, and eliminating the need for post-analysis processing. To further enhance polymer identification, the software enables users to easily create custom spectral libraries tailored to specific analytical needs.

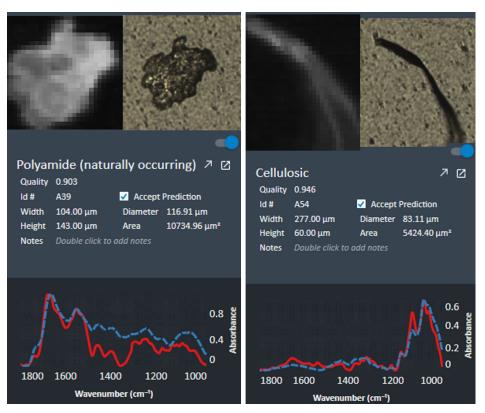


Figure 9. Examples of non-microplastic particles detected and identified with high hit quality index results using an Agilent 8700 LDIR chemical imaging system.

Particle shape classification

The classification of microplastic particle shapes varies across different regulatory methods. According to EU 2024/1441, particle shape is defined based on dimensions, with particles having a maximum dimension of 5 mm and an aspect ratio (length-to-width ratio) of \leq 3. Fibers, in contrast, are classified as having a length of up to 15 mm and an aspect ratio > 3.

Meanwhile, the California Water Boards method groups foams, films, fragments, and pellets under the category of "fragments," while fibers and fiber bundles are categorized as "fibers." The third group is "spheres" and rubbery

fragments, which are often black in appearance. This group is classified separately as "rubbery fragments." The ISO/DIS 16094-2 method, however, does not specify a procedure for determining the geometric shape of microplastics.

LDIR automatically calculates the aspect ratio for each particle, assisting in distinguishing between particles and fragments as outlined in EU 2024/1441. Also, the LDIR instrument's high-magnification visible camera captures individual particle images, supporting visual confirmation of shape classification. These images can be exported at the end of the analysis, providing a comprehensive record of particle morphology.

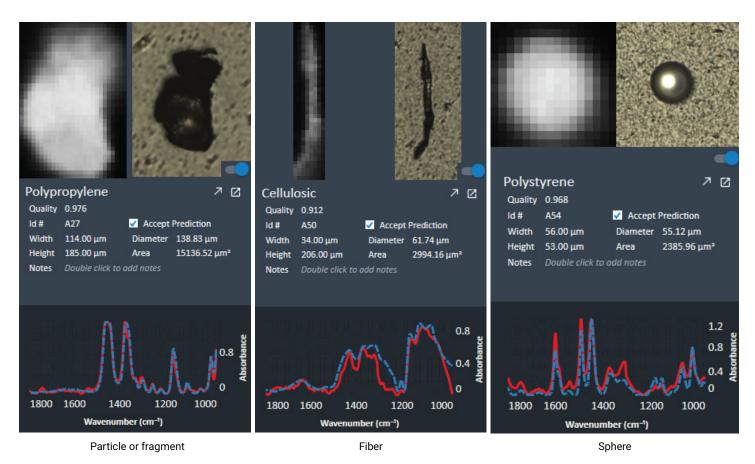


Figure 10. Various particle shapes captured and identified using the automated Particle Analysis workflow within Agilent Clarity software. The height and width measurements are used to calculate the aspect ratio, which is used as a metric to differentiate between particles and fibers.

Data reporting

All standardized methods require comprehensive data reporting at the end of the analysis, including details such as sample matrix, origin, receipt date, processed water volume, and sieve fractions. More detailed reporting includes the number, size, shape, color, and composition of detected particles.

With the 8700 LDIR system, the Clarity software automatically provides the analyst with the following statistical data:

- Total number of particles detected
- Total particle count within each size fraction

- Type of polymer of each particle identified
- Statistical overview of the identified particles, color-coded based on the identification of each particle
- Size ranges
- Infrared and high-magnification visible images for each particle detected

The software generates an Excel sheet containing all relevant information, enabling further statistical analysis and reporting based on method requirements and analytical objectives (Figure 11).

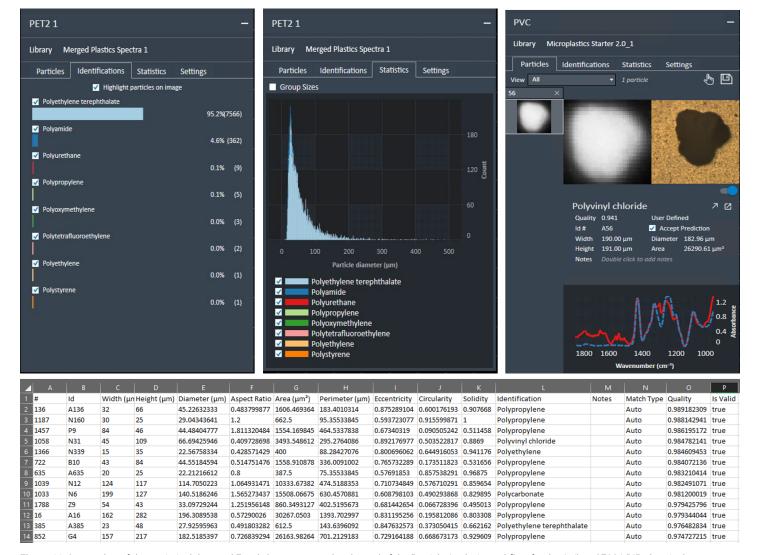


Figure 11. Screenshot of the statistical data and Excel sheet generated at the end of the Particle Analysis workflow for the Agilent 8700 LDIR chemical imaging system.

Conclusion

Microplastics are widespread in the environment, exhibiting significant variation in size, shape, color, and composition, which complicates their analysis. To address these challenges, current regulatory frameworks and emerging analytical methods offer flexibility in sampling equipment, instrumentation, and data analysis techniques, provided they meet the required analytical standards. The aim of the standardized methods is to improve the accuracy and comparability of microplastic assessments.

In addition to thermo-analytical methods and vibrational spectroscopy techniques such as μ -FTIR and μ -Raman, quantum cascade laser-based IR technology has proven to be a suitable technique for microplastic analysis.

This white paper demonstrates how an Agilent 8700 LDIR chemical imaging system combined with Agilent Clarity software meets the requirements of the three global regulatory frameworks, enabling accurate microplastics characterization. The laser-based system offers a fully automated workflow for the direct filter-based analysis of microplastics, resulting in significant time savings and higher sample throughput compared to conventional vibrational spectroscopy techniques.

References

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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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