Agilent

Trusted Answers

Solving Our Plastic Problem: Advances in Microplastics Analysis

Contents

Introduction: Our Plastic Problem

Modern society relies on plastic—it touches almost all aspects of our lives, used in everything from packaging, clothing, the cars we drive, to our toothbrushes. Despite the vast amount of plastic production, it remains a mostly nonbiodegradable material and can take up to an estimated 400 years to break down depending on the type of plastic.

However, our reliance on the substance is only increasing. For example, worldwide plastic production increased from 2.3 million tons in 1950, to 448 million tons by 2015, and this figure is expected to double by 2050.[1] Microplastics are a consequence of this global consumption of plastic and the subsequent plastic pollution it causes.

Microplastics are minuscule pieces of plastic, which measure between 1 µm to 5 mm in size,[2] approximately the size of a sesame seed.[3] These tiny plastic particles have the potential to spread to all corners of our environment—in the land, water, air, and ultimately our bodies.[4] Current research believes that microplastics will also degrade into smaller particles on a nanoscale,[5] called nanoplastics, which measure in the range of 1 to 1000 nm.[6] Invisible plastic pollution is a growing global concern that is receiving increasing attention from government bodies and academic institutions. The drive to understand more about the impacts of micro- and nanoplastics is rooted in the lack of expert knowledge we have on the implications of plastic pollution for our health and the environment. Moreover, significantly less is known about the consequences of nanoplastics, but their size and subsequent ability to penetrate even more areas in our ecosystem means that their presence has the potential for more severity.

Where do microplastics come from?

There are two categories of plastic particle pollution to be aware of:

1. Primary micro- and nanoplastics — These are very small plastic pieces that have deliberately been manufactured in products, such as in shower gel and toothpaste.

2. Secondary micro- and nanoplastics — These are small plastics originating from larger plastics that have since degraded. Examples include paints, abraded tires from driving, and textiles.

How Agilent is tackling the problem

Through collaborations with key organizations and opinion leaders across the globe, Agilent continue to create innovative tools and technologies to help better characterize microplastics, and their impact on our environment and health. There are currently two widely accepted analytical pathways to characterize microplastics, which provide complementary yet differing information, that are being developed for standardization—both of which Agilent are well-positioned in.

On the spectroscopy side, Agilent's innovative approach to developing new solutions for microplastics testing has earned them multiple awards, notably for the 8700 Laser Direct Infrared (LDIR) Chemical Imaging System, a chemical imaging tool that provides "rapid processing" and analysis of samples, including microplastics.[6] Through the company's expertise in infrared imaging, Agilent has also developed highly sensitive Fourier transform infrared (FTIR) scanners that are used for mobile and on-site characterization of microplastics in experimental studies.[7]

Agilent is also a leader in the development of gas chromatography-based instruments, which can be used in the field of microplastics testing. Thermal extraction desorption gas chromatography-mass spectrometry (TED-GC-MS) is a new and fast method for the identification and quantification of microplastics in environmental samples without requiring sample preparation.[8]

References

1. National Geographic. The World's Plastic Pollution Crisis Explained. Accessed: November 2019. https://www.nationalgeographic.com/environment/habitats/plastic-pollution/.

2. Frias, J. P. G. L.; Nash, R. Mar. *Pollut.* Bull. 2019, 138, 145–147.

3. National Ocean Service. What Are Microplastics? Accessed: November 2019. https://oceanservice.noaa.gov/facts/microplastics.html

4. Plastic Oceans. The Facts. Accessed November 2019. https://plasticoceans.org/the-facts/

5. Gagné, F. J. *Xenobiot.* 2019, 9(1), 8147.

6. Agilent. Agilent Technologies Spectroscopy Product is Named a "Top Innovation." Accessed February 1, 2024. https://blog.agilent.com/2019/01/22/agilentspectroscopy-productis-named-a-top-innovation/

7. Agilent. Analysis of Microplastics using FTIR Imaging. Accessed February 1, 2024 https://www.agilent.com/cs/library/applications/5991- 8271EN_microplastics_ftir_application.pdf

8. Wiley Analytical Science. Polymer Analysis and Microplastics in the Environment. Accessed February 1, 2024. https://www.laboratory-journal.com/science/chemistry-physics/polymeranalysisand-microplastics-environment

Challenges in Microplastics Analysis: From Routine Laboratory Testing to Pushing the Boundary on Particle Sizes

Practical aspects of analyzing microplastics with the Agilent 8700 LDIR and dealing with the difficulties of analyzing particles smaller than 20 µm.

By Dr. Julia Jaeger

Current challenges in microplastics characterization

Even though microplastics are considered emerging contaminants, plastic debris in the environment has been reported since 1972. This discovery initiated the first wave of research in this area, which took place between 1980 and 2010, finding that microplastics were ubiquitous in the environment, specifically in the ocean environment. The second wave of research, which has occurred during the last decade, has been heavily influenced by the rising social awareness of microplastics as a threat to the environment. To manage this challenge, several governments have instituted some level of action. Australia, for example, banned the use of microbeads from personal care products in 2018 and later banned single-use plastic bags; in 2021, California enacted legislation requiring a period of analysis of microplastics in drinking water. Still, the quality of research surrounding microplastics has hit a standstill in recent years due to several challenges in the field of characterization, including a lack of standard established policies and procedures, a specialized spectral database, interlaboratory studies, and reliable analytical techniques (Figure 1).

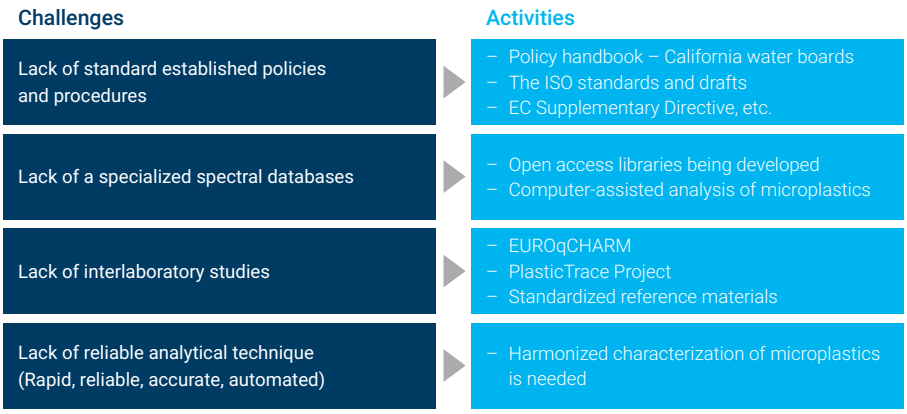


Figure 1. Characterization challenges and solutions.

Article

Micro- and nanoplastics: a deep dive into a global issue

That said, these challenges present a range of opportunities for researchers to contribute to microplastic research. Already, the California State Water Resources Control Board produced a policy handbook describing a standard analytical method for the sound preparation and identification of microplastics; a range of open-access libraries are currently being developed by various labs and groups; scientists are creating a computer-assisted analysis of microplastics; and labs are collaborating to develop interlaboratory studies. That said, there is still a lack of reliable analytical techniques for studying microplastics.

In addition to myriad challenges related to microplastic characterizations, the industry also suffers from challenges in the research itself, including a lack of quality controls and assurances of the published data. For example, among 230 studies published since the early 2000s, only 71 papers have published data with method blanks; just 13 papers published a data recovery. This indicates a possible overestimation and underestimation of results, respectively.

Challenges of using LDIR for routine microplastics analysis

In 2019, Eurofins set up the first commercial laboratory in Australia to use laser direct infrared imaging (LDIR) for the analysis of microplastics, starting with demo models and going fully operational in 2021. The laboratory focuses its LDIR work on the normal particle size range, from 20–500 µm. Jaeger points out that for larger particles, Attenuated Total Reflection (ATR) or IR analysis of pieces cut off from the particles delivers better results. Their LDIR workflow is very similar to other analytical methods, consisting of sample collection, sample preparation, and analysis. In the absence of standard methods, Dr. Jaeger and her team initially used methods published in peer-reviewed journals. More recently, they are using two standard methods, one by ASTM (ASTM D8332-20) and the other by the State Water Resources Control Board of California. Her lab can handle a variety of samples from different environmental matrices, including water from various sources, sand and sediments, sewage and biosolids, and even marine organisms and fish tissue (Figure 2). Jaeger's team also developed preparation methods needed for handling less usual samples, such as air, dust, infant formula, eye drops, and even cheese, milk, and body washes. Most of the particles they analyze are in the size range from 20–100 µm, well within the LDIR capabilities. They report their results in terms of the nine most common polymers and include the number of particles found for each polymer, as well as the total number of particles and the weight of the sample analyzed (Figure 3). They also provide a plot of the particle size distribution for each of the identified polymers. Jaeger mentions that if the client requires additional information, the lab will provide a file with all the data generated by LDIR.

Figure 2. What matrices can we analyze?

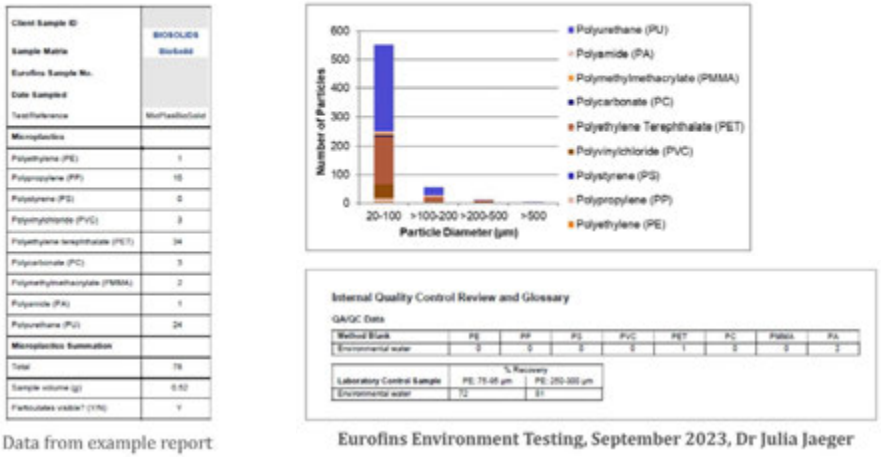


Figure 3. How do we report our results?

Ensuring reliable results

Their quality assurance and quality control procedures are very robust, to ensure that their data has a high confidence level. The backbone of all their operations, says Jaeger, is to control and avoid background contamination. They do their analysis in a clean room with an airlock access setup. They keep the room under constant positive pressure to keep contaminants out. Their analysts clean the equipment every evening and let everything settle down overnight. To avoid introducing extraneous polymer fibers, all personnel wear cotton laboratory coats. They decontaminate all glassware in a furnace and use plastic-free water to wash everything. In addition, they run monthly air blanks to check for background contamination. To perform these checks, they prepare samples by placing slides in locations inside and outside the lab and letting them collect deposits for 24 hours. Furthermore, they routinely carry out matrix blank tests and if the results are too high, they will redo the analysis instead of doing blank subtraction, following the guidelines from the California Water Board. The reason they limit their analyses to the nine most common polymers is that those are the ones for which they have reference materials and are in their spectral library. Jaeger explains that they only report polymers that they have checked against two independent reference materials. Currently, the lab only reports on native polymers, but they expect to be able to handle weathered polymers soon.

Recovery tests, proficiency, and accreditation

From the early days, Jaeger's team has performed recovery tests for each method they have developed. They initially used PS beads for the tests. Still, they encountered stability problems with them, so they switched to co-spheric PE beads in surfactant solution, with a bimodal size distribution of 250–300 µm and 75–90 µm. Their control chart shows recoveries from about 80–120% between the two standard deviation warning limits (Figure 4). In addition, they have participated in different proficiency tests organized by the European Joint Research Centre (JRC), EUROqCHARM, Wageningen Evaluating Programmes for Analytical Laboratories - Quality Assurance of Information for Marine Environmental Monitoring in Europe (WEPAL-QUASIMEME), and the Southern California Coastal Water Research Project (SCCWRP). Their accreditation process started 12 months ago, and they aim to be the first laboratory in

On-Demand event

Challenges in microplastics analysis: from routine laboratory testing to pushing the boundary on particle sizes

Australia accredited to ISO17025 standards. In addition, they recently applied for accreditation to the National Association of Testing Authorities (NATA) for their Potable Water Method. One of their biggest challenges has been finding reliable and knowledgeable technical assessors. Nevertheless, Jaeger expects a very successful outcome for their applications.

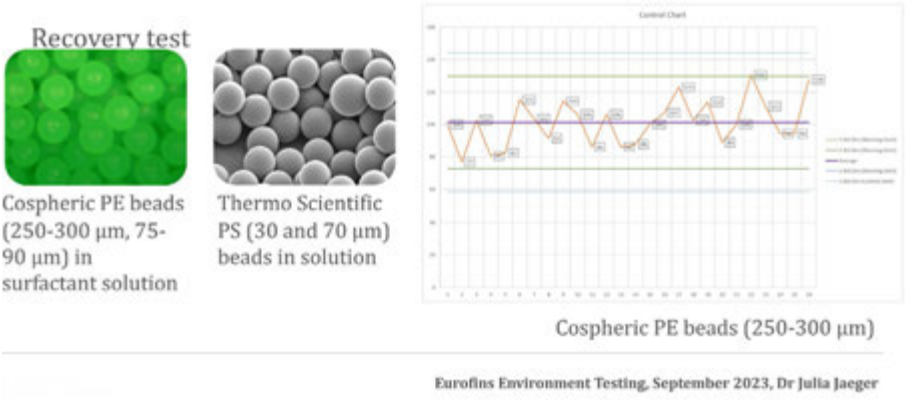


Figure 4. Quality assurance and quality control procedures.

Conclusions

LDIR is an effective technique for identifying and quantifying microplastic particles ranging from 20–500 µm. At sizes below 10 µm, the accuracy of the particle size measurement drops to below 20% if using the automated particle analysis workflow within Clarity software. The chemical identification can range from 65–95%. Manual operation of the instrument to probe several sites within the same particle determination help improve the hit quality and using manual visible imaging for the size determination also improves the particle size accuracy results. However, it would be challenging to use this manual approach for routine analysis of particles smaller than 20 µm. On the other hand, at sizes above 20 µm, scientists have successfully established a commercial laboratory for microplastics analysis. With stringent QA/QC procedures to control background contamination and routine recovery tests, they deliver reliable and accurate results from a wide variety of environmental and natural samples. In fact, they recently have been granted accreditation to ISO17025 and NATA.

Dr. Julia Jaeger
Technical Specialist
Eurofins Environmental Testing – Australia & New Zealand

Understanding the Regulatory Environment Around Microplastics Analysis

By Dr. Anja Sokolowski and
Dr. Andreas Kerstan

Many microplastics experts feel that having standards and regulations in place, such as those in development by ISO and other organizations, is very important because of the potential environmental and human health impacts of microplastics. Such rules and guidelines will help contract and state laboratories contribute to the monitoring of microplastics in drinking water, bottled water, personal care products, and more. Indeed, standardized methodology is critical if regulations are proposed. Along with the work of ISO, other organizations are developing standards. ASTM in the United States has also developed some standard methods for microplastics (e.g., D8332- 20, "Standard Practice for Collection of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers"). These standards must clearly define what must be analyzed for microplastics: the amount, polymer type, particle size, particle shape, particle number, and statistical distribution as well as the mass or the mass content, depending on the analysis.

To date, there are few, if any, regulations in place regarding microplastics around the world. Those that are in place primarily focus on the use of microparticles (microbeads) that are intentionally added to rinse-off personal care products such as the Microbead-Free Waters Act, signed into law by President Obama in 2015 and European Commission Regulation (EU) 2023/2055 which, starting from October 17, 2023, restricts synthetic polymer microparticles intentionally added to products.

Other regulations, such as the US California State legislation SB1422 (2018) mandates the monitoring of drinking water for a period of 4 years, following the development of standardized methodology. The analysis period of this mandate is due to commence in 2024. Likewise, in early 2024 the European Parliament and Council published the supplementing Directive (EU) 2020/2184 which established a methodology to measure microplastics in water intended for human. This was done with the view to including certain microplastics on the "watch list" also referred to in that directive which, if implemented, would also mandate testing.

As the focus on microplastics grows and there is increasing attention paid to mandated monitoring and/or regulations, the establishment of standardized methodology is clearly essential.

On-Demand event

Standardized ISO methodologies for the assessment of microplastics: an update on key developments

Overview of ISO and CEN

The ISO is a global network of national standardization bodies. ISO has 167 members (one member per county), with members representing the organization in their own countries. ISO has 810 technical committees and subcommittees that are responsible for the development of standards. To date, the organization has published more than 24,600 international standards covering all aspects of technology management and manufacturing. ISO often works in collaboration with the Comité Européen de Normalisation (CEN), an association that brings together the national standardization bodies of 34 European countries. CEN has 317 technical committees and 18,411 living documents (as of December 2022). Through the Vienna Agreement, ISO and CEN have agreed on technical cooperation between the groups to avoid duplicative work and structures. For instance, to support efficient standardization, the drafting of work is carried out in only one organization. Standardization projects are developed simultaneously through parallel voting procedures, and they are adopted as both ISO and CEN standards at the same time. A fundamental challenge for ISO is to avoid duplication when it comes to overarching horizontal topics. An example of efforts to avoid such double standardization was the creation of a joint committee looking at standards for plastics (including microplastics) in waters and related matrices. In 2019, ISO/TC 61/Subcommittee 14 on Environmental aspects started working on ISO/NP 24542, Methods for analyzing microplastics in water with very low contents of suspended solid. But at the same time, ISO/TC 147/ Subcommittee 2 on Physical, chemical and biochemical methods started a project on ISO/NP 24606-1, Water quality – Analysis of microplastics in drinking water and groundwater – Part 1: Method using vibrational spectroscopy.

Since the two sets of activities were quite similar, ISO put the activities on hold and decided to merge the groups. This resulted in the creation of the Joint ISO/TC 147/SC 2 – ISO/TC 61/ Subcommittee 14 Working Group: Plastics (including microplastics) in waters and related matrices. The scope of this group is the standardization of methods for the characterization and quantification of plastics including microplastics and related polymers in water (e.g., bottled water, drinking water, groundwater, seawater, rainwater, wastewater, and more) within the intersection of the scopes of ISO/TC 61 and ISO/TC 147.

Thus far, this joint group has started to work on four projects:

- ISO/CD 16094-1, Water quality – Analysis of microplastics in water – Part 1: General and sampling for waters with low content of suspended solids including drinking water (currently in the committee draft stage, with a projected publication date of May 31, 2024)
- ISO/CD 16094-2, Water quality – Analysis of plastics in water – Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water (currently approved for registration as a draft international standard, with a projected publication date of May 31, 2024)
- SO/CD 16094-3, Water quality – Analysis of plastics in water – Part 3: Thermo-analytical methods for waters with low content of suspended solids including drinking water (currently approved for registration as a draft international standard, with a projected publication date of May 31, 2024)
- ISO/PWI 16094-4, Water quality – Analysis of plastics in water – Part 4: Sample preparation for monitoring of microplastics for waters with low content of suspended solids including drinking water (currently proposed for a new project)

A challenge for this joint working group will be the alignment of this four-part water quality standard as well as harmonization with other existing standards such as ISO 24187 (ISO/TC 61 "Plastics"). This point will be explored later in the paper.

Overview of standardization projects on microplastic at the european and international level

ISO and CEN technical committees are involved in several microplastic standardization projects across areas like food products, textiles, water quality, soil quality, and more. Such standards are either published or in development and include those listed in Table 1. These projects are at various stages of development, which is typically a six-stage process. The stages include:

1. Proposal of a new project, with active participation from at least 5 members
2. Preparation of a working draft and registration as a committee draft
3. Committee review, where a draft is circulated in TC/SC and members of TC/SC provide comments
4. Enquiry, where a Draft International Standard (DIS) is circulated to all ISO members. The national standards bodies (NSBs) have 12 weeks to vote and comment on the draft, and the DIS is open for public comments
5. Approvals
6. Publication

Table 1. ISO and CEN technical committees involved in microplastic standardization.

Technical committee	Standards published or in development
ISO/TC 38 Textiles and CEN/TC 248 Textiles and textile products	EN ISO 4484-1:2023, *Textiles and textile products— Microplastics from textile sources—Part 1: Determination of material loss from fabrics during washing*
	ISO/DIS 4484-2, *Textiles and textile products— Microplastics from textile sources—Part 2: Qualitative and quantitative evaluation of microplastics* (under development)
	ISO 4484-3: 2023, *Textiles and textile products— Microplastics from textile sources—Part 3: Measurement of collected material mass released from textile end products by domestic washing method*
DIN Standardization Committee on Food	DIN TS 10068:2022, *Food—Determination of microplastics—Analytical methods*
CEN/TC 249 Plastics	EN 17615:2022, *Plastics—Environmental Aspects— Vocabulary*
CEN/TC 249 Plastics and ISO/TC 61 Plastics/SC 14 Environmental aspects	EN ISO 17422:2018, *Plastics—Environmental aspects— General guidelines for their inclusion in standards*
	EN ISO TR 21960:2020, *Plastics—Environmental aspects— State of knowledge and methodologies*
	ISO/FDIS 24187, *Principles for the analysis of microplastics present in the environment* (final stages of development)
ISO/TC 38 Textiles and CEN/TC 248 Textiles and textile products	ISO/FDIS 5157, *Textiles—Environmental aspects— Vocabulary* (under development)
ISO/TC 147 Water quality and CEN/TC230 Water analysis	ISO/CD 5667-27, *Water quality—Sampling—Part 27: Sampling for microplastic particles and fibres in water* (under development, committee draft approved for registration as a DIS)
	ISO 16094 — *Series for the analysis of plastic in water* (under development)
CEN/TC 444 Test methods for environmental characterization of solid matrices	CEN/TS, *Sludge, treated biowaste, soil and waste— Sampling, pre-treatment and analysis of microplastics* (project planned)

Special considerations in microplastics analysis

Labs have some important considerations with respect to microplastics analytical processes: sample preparation. There are numerous potential matrices (e.g., ocean water, fresh water, animal organs, drinking water, sewage sludge, fertilizer, soils, and more) for microplastics analysis and the sample preparation can be quite challenging for some. Protocols are needed for sampling, preparation, and detection. Documentation for the whole procedure will likely come in the years after the initial standards are published.

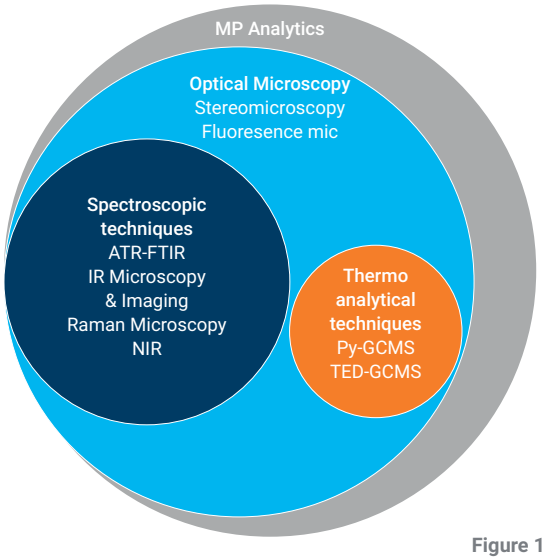
Systems and solutions are becoming increasingly automated to help address some of these challenges. Automation is a "must-have" given how many samples are being analyzed and the potential for tens of thousands of particles to be present within them.

Detection methods

Figure 1 shows how the various detection methods may be useful in microplastics analysis. There is some division among optical microscopy, spectroscopic techniques, and thermal techniques, with each offering unique benefits. For instance, fluorescence microscopy and optical microscopy might be able to provide information about the particle shape and number, but discerning the chemical ID and mass is more difficult with these techniques. Meanwhile, FTIR imaging can provide a lot of information, but determining mass is a challenge because 2D techniques such as this can only estimate the heights and densities of particles. Measuring mass content would require a second method be used as well. Thus, spectroscopic techniques are thermal techniques complementary, and both are needed to have the full analytical picture of microplastics.

Automation

Systems and solutions are becoming increasingly automated to help address some of these challenges. Automation is a "must-have" given how many samples are being analyzed and the potential for tens of thousands of particles to be present within them.



Analytical technique	Shape Info	Chemical ID	MP_{Num}	MP_{Mass}
Optical microscopy	🟢	🔴	🟢	🔴
Fluoresence microscopy	🟢	🔴	🟢	🔴
ATR-FTIR	N/A	🟢	N/A	🟢 *
µFT-IR-Imaging/ LDIR	🟢	🟢	🟢	🟡
NIR, Hy-Spec-Imaging	🟢	🟢	🟢	🔴
µRaman	🟢	🟢	🟢	🔴
Pyr-GC-MS	🔴	🟢	🔴	🟢
TED-GC-MS	🔴	🟢	🔴	🟢

* Using a balance to weigh particles

Figure 1. The world of detection methods.

ISO/FDIS 24187 highlights and considerations

As noted previously, ISO is in the final stages of development for ISO/FDIS 24187, *Principles for the analysis of microplastics present in the environment*. These are the first guidelines to set out principles to be followed in the analysis of microplastics in various environmental matrices.

The document discusses the requirements for all analytical steps:

- Avoid plastics during sample preparation
- Laminar flow box
- Sterilization
- Control and blank measurements

It also covers particle size classification and mass content (using thermal techniques) versus particle number, ID, and shape (using spectroscopic techniques). The document discusses the types of results that various detection techniques can generate (Figure 2). The guideline also covers sampling and sample preparation steps (e.g., drying, milling, removing organic matter). Processing such as through a data library and machine learning, reference materials, and the need for interlaboratory studies are also discussed. Regarding the latter, the guidelines suggest goals for this work could be:

- Verification of sizing
- Specificity and scope of the method
- Possible recovery rate percentage
- Exploration of false positive and false negative results
- Blank measurement standards done daily or before each measurement
- Blank subtraction
- Interferences
- Recovery rate

Table 3 – Type of results that can be generated with different detection methods

Characteristics	Spectroscopic					Thermoanalytical				Chemical
	µ Raman	(FT)IR (µ, FPA)	µ ATR-(FT)IR	ATR-(FT)IR	NIR	Py-GC-MS	Mod. Py-GC-MS	TED-GC-MS	DSC	ICP-MS
Type of polymer	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Only PE, PP	Only for plastics with inorganic contents
Detectable additives	Pigments	No	No	No	No	Yes	No	No	No	No
Particle surface (chemical)	Yes	No	No	Yes	Yes	No	No	No	No	No
State of degradation[a]	Surface Oxidation	No	Surface Oxidation	Surface Oxidation	No	Oxidation	No	No	Mol. weight	No
Particle number, particle size, particle shape, morphology[b]	Yes	Yes	Yes	Yes	No	No	No	No	No	No
Mass balances	No	No	No	No	No	No	Yes	Yes	Yes	Yes

a Any physical or chemical change in polymer as a result of environmental factors, such as light, heat, moisture, chemical conditions or biological activity.
b Here only information obtained by the method itself and not by additional investigation is considered.

Point wise measurements vs hyperspectral imaging

Table 6 – Comparison of spatial analysis approaches

Characteristics	Point-wise	Hyperspectral imaging
Time dependence on number of particles	Yes	No
Applicable to agglomerated particles	No	Yes
Dataset size	small	large

Figure 2. ISO 24817: a first guideline.

ISO/FDIS 16094-2 vibrational spectroscopy highlights and considerations

Another ISO document that is already well into the development and approval stages is ISO/CD 16094-2, Water quality – Analysis of plastics in water – Part 2: *Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water*. As noted previously, this document is currently approved for registration as a draft international standard, with a projected publication date of May 31, 2024. It also has some potential implications for analytical labs.

It focuses on:

- Characteristics of techniques
- Principles IR versus Raman
- Interferences, with a focus on suitable sample preparation
- Laboratory environment
- Minimum requirements for IR/Raman equipment

IR spectra acquisition/particle ID, which include those listed in Figure 3. The document lists criteria for the spectral libraries. The libraries that often come with the IR or Raman software are often generic and only have the raw polymer spectra. To optimize these libraries and obtain analytical spectra that fully match the spectral database, it is possible to expand the database with internally acquired spectra. ISO/CD 16094-2 also includes the criteria for reporting such as listing the sample analysis detection technique, filtered sample volume, filter's pore size, total number of microplastics in the test sample, and more.

Agilent's QCL/LDIR 8700 will be part of ISO 16094-2 (Figure 4).

IR Spectra Acquisition/Particle ID

- Transmission, transflectance, reflectance or ATR (automated?)
- Infrared spectral acquisition will be typically performed from 700 cm^{-1} to 4000 cm^{-1}. **For some instrument working with a smaller spectral range, the laboratory should refer to Annex G.**
- Accurate BG subtraction
- HQI conformity
- Control blanks
- Search Algorithms: Pearson recommended, **but not mandatory**
- Open library: Spectra can be added!
- Size Verification (standard Microbeads)/ID Verification
- Minimum HQI/Size limits must be clearly defined
- Clear Reporting
- Challenges: Pigments/natural/synthetic polymers

Interferences: Pigments Raman

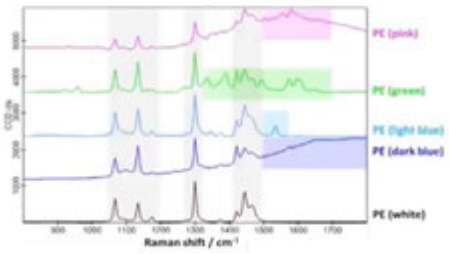


Fig 3: Examples of Raman spectra of coloured PE particles.

Naturally occurring PA vs Nylon

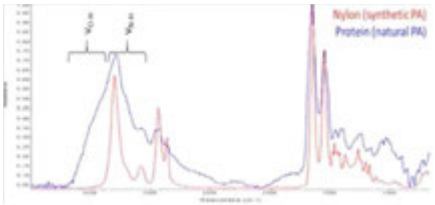


Fig 4: Examples of similarity of natural and synthetic polyamides (proteins and Nylon) IR spectra.

Figure 3. ISO/CD 16094-2 vibrational spectroscopy.

- Novel technique
- Differences from classical FTIR and Raman
- Rapid scanning of Spectra
- Particle by Particle approach
- High Analysis Speed with promising results: 4-5s per particle
- Minimum particle sizes already reported- ca. 5-6 µm (in automation)
- High confidence and complete data down to 10 µm
- 975- 1800 cm^{-1} sufficient for Microplastics analysis (point of discussion)
- Substrates: Gold/silver filter or low e slides
- Search algorithm derivative based- no mandatory algorithm up to now
- More and more references

- Ca. 40 publications up to now since launch
- Proof of Principle for Several matrices
- Several Collaborations and Partnerships in place
- Participation to several Round Robin Tests/Ringtests

Machine Learning

Drinking Water/Air

Body Fluids/Organs

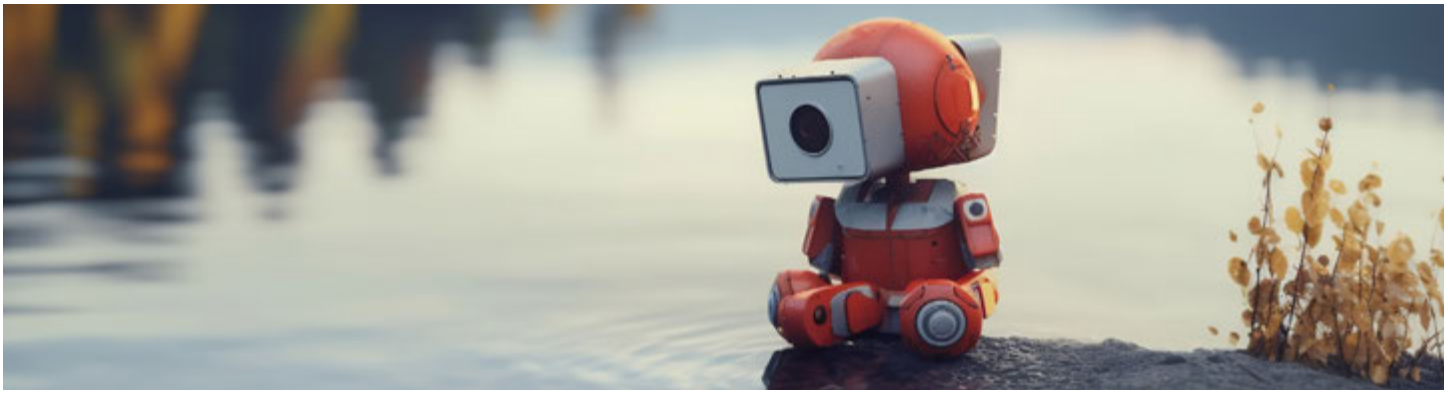
Figure 4. QCL/LDIR 8700 will be part of ISO 16094-2.

Conclusions

Understanding Regulations FTIR/IR (e.g., QCL based) and Raman are useful techniques for the determination and characterization of microplastics.
The degree of automation for these systems is well advanced and continues to improve. Standards in this space are also developing quickly. ISO is in the final stag es of development for ISO/FDIS 24187, Principles for the analysis of microplastics present in the environment. These are the first guidelines to set out principles to be followed in the analysis of microplastics in various environmental matrices. A second guideline, ISO/CD 16094-2, Water Analysis Techniques Sample Prep quality – Analysis of plastics in water – Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water, is slated to be released next year.

Dr. Anja Sokolowski
Senior Project Manager
DIN Standards Committee Water Practice

Dr. Andreas Kerstan
Product Specialist
Agilent Technologies



Key Microplastics Analysis Techniques: GC, LDIR

Improve microplastics identification by analyzing particles directly on gold-coated filters with a laser-based IR chemical imaging system.

By Darren Robey, Wesam Alwan, and David Troiani

Challenges of microplastics analysis

Analyzing the presence of microplastics in the environment and food chains is a crucial task, and selecting the right technique depends on the specific information needed. Some of the key options are:

Pyrolysis coupled with GC/MS (Gas Chromatography-Mass Spectrometry)

Purpose: Pyrolysis is a technique that breaks down complex organic materials into simpler fragments by heating them in the absence of oxygen. When coupled with GC/MS, it allows for the identification of volatile compounds released during pyrolysis.

Advantages:

- Provides information about the chemical composition of microplastics.
- Detects volatile organic compounds (VOCs) associated with plastics.
- Useful for identifying additives or contaminants.

Limitations:

- Does not provide detailed morphological information.
- Limited to volatile compounds.
- Requires specialized equipment and expertise.

Spectrophotometer coupled with a microscope

Purpose: Spectrophotometry measures the absorption or emission of light by a sample. When coupled with a microscope, it allows for visual examination and quantification of microplastics.

Advantages:

- Provides information about particle size, shape, and morphology.
- Non-destructive technique.
- Can be used for both qualitative and quantitative analysis.
- Can provide chemical identity.

Limitations:

- Quantification is based only on particle count, not total mass
- Sample preparation can be complex
- Analysis time highly variable and dependent on number of particle and/or area analyzed.

On-Demand event

Microplastics analysis just got easier: analysis direct on-filter

When choosing the right technique some of the critical things to consider may include:

- Objective: Are you interested in overall plastic mass or detailed particle characteristics?
- Sample Type: Is it environmental water, sediment, or food?
- Budget and Resources: Some techniques are more expensive or require specialized equipment.
- Expertise: Ensure you have the necessary expertise to perform the chosen technique.
- Any regulatory requirements

Often, a combination of techniques (e.g., spectroscopy and chemical analysis) provides a more comprehensive understanding of microplastics.

Among the spectroscopic techniques, Fourier transform infrared (FTIR) microscopes (both imaging and nonimaging), Raman microscopes, and laser direct infrared (LDIR) spectroscopy are the commonly used techniques, each with benefits and limitations.

FTIR instruments range from simple (and lower-priced) single point microscopes, through to much more expensive focal plane array (FPA) microscopes that can acquire spectra simultaneously for a number of pixels over a larger area. FTIR is a mature and well-understood technology, and there is significant relevant literature and extensive spectral libraries available. Time of analysis, however, can be prohibitive. Even the largest FPA systems can only image areas of less than 1 mm^2 at a time, and these areas must be mosaiced and the data stitched together. As a result, data acquisition on a typical 13 mm filter area would exceed 3 hours. After acquisition, a vast amount of data must then be separately processed to obtain results. Depending on the area imaged and system used, this can take many hours. LDIR is a technique for IR spectroscopy combining a tunable quantum cascade laser (QCL) as the IR source with rapidly scanning optics. It can be used in two modes: frequency parked with rapid scanning over a large area, or position parked with rapid sweep through the entire available wavelength range with resolution at the diffraction limit. LDIR detects microplastic particles by rapid imaging of the area using IR light rather than visible cameras to determine the location, size, and shape of particles. Spectra can then be obtained from individual particles and compared to the onboard library, with results presented in real time.

In contrast to FTIR and LDIR, Raman imaging uses the Raman effect rather than IR transmission or reflection. Many Raman systems can detect particle sizes down to 1 µm, where the very best IR systems are limited to the diffraction limit of the light (>10 µm). Another benefit is that Raman measurements are less disturbed by water. A key limitation—the Raman effect—is relatively weak, so these systems use higher-powered lasers. Sample damage may occur, or the sample may exhibit fluorescence, drowning out the useful Raman signal. While each can be mitigated using lower powered lasers and/or by attenuating the signal, this comes at the cost of speed.

Some systems do particle-by-particle analysis while others have imaging capability that measures all or part of the filter. Then they then conduct a post-analysis with databases or through machine learning. While databases for machine learning are more expensive, the data gets more and more precise as more entries are created. Figure 1 shows two ways of collecting data through microscope measurement. On the left is the detect-and-identify method. This is used by the LDIR but may also be used in some FTIR and Raman systems where a visible or IR image is used to detect particles.

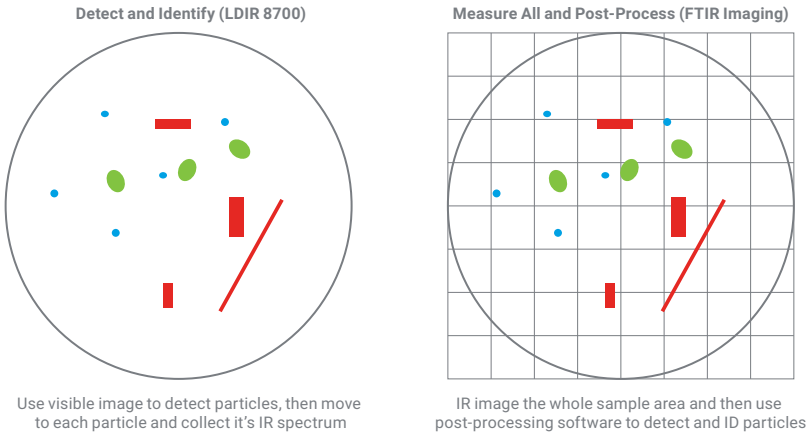


Figure 1. Two approaches to FTIR microscope measurement.

The instrument targets the identified particles and collects a spectrum of each one. Each spectrum is then compared against a spectral library to identify the chemical composition of each particle. Meanwhile, with the measure everything and post-process method on the right, the whole sample area is imaged by either focal plane array or line array detectors and then post-processing software is used to detect and identify particles.

LDIR overcomes some of the key limitations of FTIR systems such as eliminating the need to collect data in empty spaces. This results in significantly faster analysis times and it can also be fully automated. A QCL operates at lower power than lasers used in Raman, hence fluorescence and sample damage pose no risk. An electrically cooled detector eliminates the need for liquid nitrogen, yet it has the highest resolution of any IR system and can detect particles as small as 10 µm. It is, however, relatively new technology. This, coupled with the use of the fingerprint region of the IR spectrum, only means that relevant libraries of data are less developed than other systems.

The traditional approach to analyzing microplastics has been by optical microscopy followed by physical tests, such as the hot needle test, which determines that the particle is plastic if it melts. This is a subjective and not very accurate test and is incapable of determining the chemical identity of the plastic. Wet chemistry methods, in combination with GC/MS and pyrolysis, are useful in determining the total mass of the plastics and require very simple sample preparation. They do not, however, provide any individual particle information and are destructive.

eBook

Achieving accurate microplastics characterization

In contrast, spectroscopic methods, such as Raman and FTIR, allow individual particle characterization. In addition, they are nondestructive and can be highly automated. The drawbacks to these methods are that they require complicated sample preparation processes and are slow. Raman spectroscopy is useful for particles as small as 1 µm and can tolerate the presence of water better than FTIR. Fluorescence and pigment interference, however, can cause strong interferences in Raman spectroscopy. The Raman signal from the pigment is often stronger than that of the plastic itself. Using a lower-powered laser or attenuating the laser signal can mitigate the interference but slows down the analysis. On the other hand, FTIR is generally faster than Raman, and there are extensive libraries available that aid in identifying the specific plastics analyzed. For small particles, it is better to use FTIR in transmission mode, for which particle transparency is necessary. Because the traditional FTIR uses a large, incoherent light source, focusing it on a small particle produces a weak signal, as only a fraction of the light source's energy ends up on the particle. As a result, the minimum particle size that this technique can handle is 10 µm with top-of-the-line instruments, and up to 50 µm with less expensive ones.

What are the pros and cons of LDIR for microplastics?

Pros:

- Highly automated and fully integrated workflow; no need for external data processing
- Fast, as the quantum cascade laser allows it to scan the area for particle location using IR rather than visual light for particle detection
- Fast, as it can obtain particle spectra much faster than FTIR (1 second compared to 30 seconds)
- Does not suffer any of the Raman associated limitations such as damage and fluorescence
- Can detect the smallest particles of any IR systems
- No liquid nitrogen cooling required

Cons:

- New technology so libraries not yet well developed, but this is improving all the time
- Uses only the fingerprint region of the IR spectrum so may have some challenges differentiating more difficult samples

What are the pros and cons of FTIR micro-spectroscopy for microplastics?

Pros:

- Well understood, mature technology
- Established techniques, methods, and libraries
- Price can be low (single point microscopes)—however, limited functionality does impact sample throughput

Cons:

- Time consuming, even for the most advanced FPA equipped systems
- Can be very costly (FPA systems)
- Requires liquid nitrogen for detector cooling in many cases
- Generates vast quantities of data for post processing
- Even the best has limited resolution (particle sizes >30 µm best case while lower-end instruments may be >50 µm or more)

What are the pros and cons of imaging for microplastics?

Pros:

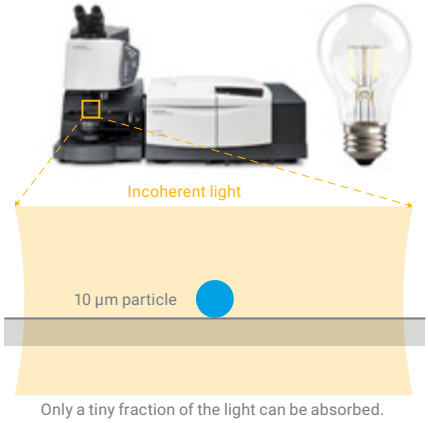
- Particle-specific data acquisition and can use automated workflows to conduct analysis
- Relatively mature technology with a good range of libraries available
- Can detect particles to 1 µm particle size

Cons:

- Fluorescence and sample damage can occur; power must be attenuated (analysis time slowed) to overcome
- If polymer is mixed with pigment, the pigment may have a stronger Raman signal than the polymer, making determination impossible
- While it can be highly automated, fast, safe (no sample damage) and capable of very small particle sizes, combining all of these is difficult
- Simpler systems are quite manual and may require external data processing, while highly automated systems are very expensive

A better alternative: laser IR micro-spectroscopy

To solve these problems, Agilent developed the 8700 LDIR Chemical Imaging System, an instrument that uses its proprietary Quantum Cascade Laser QCL as the infrared light source (Figure 2).



- To measure small things, combine FTIR spectrometer + microscope
- Problem: a large incoherent source cannot be focused onto small microparticle
- Weak signals, slow analysis
- Up to 30 seconds per spectrum typical

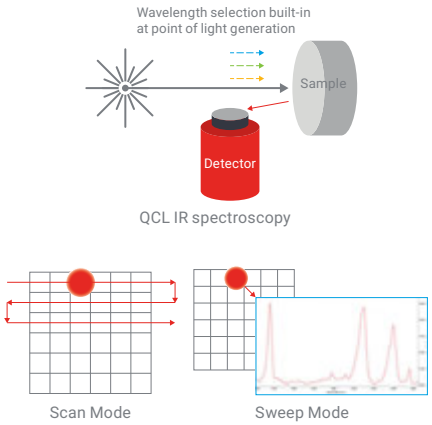
Solution: use a laser!

Laser Direct Infrared Spectroscopy using a Quantum Cascade Laser

- Bright, coherent light source
- Rapidly tunable across the mid-infrared for spectroscopy
- Focus all laser power onto a particle
- Light reflected to the detector (reflection mode)

Figure 2. FTIR micro-spectroscopy.

It incorporates aspects of microscopy and image recognition technology to fully analyze individual particles as small as 10 µm. The laser is tunable across the mid-IR range and can focus all its power onto a particle. In the scan mode, it sweeps a single wavelength across the sample area to generate a visual representation of the particles it contains. In sweep mode, it remains stationary on a single particle, and does a full sweep of the available wavelengths to generate a spectrum of the particle and determines its chemical identity using the extensive library provided with the instrument. The instrument does this for every particle it identified in the previous step automatically, using the included Clarity software (Figure 3). It is important to note that for all sources of samples, including clean water, drinking water, sand, river water, soil, and biota, sample preparation always requires filtering as the last step. There are two methods for preparing samples for analysis in this instrument. The first method uses a low-emissivity (low-e) reflective slide of standard dimensions, which Agilent recommends for samples with very high particle loads, up to several thousands.



Modes of Action

Proprietary Agilent quantum cascade laser (QCL) technology
- Bright, coherent light source. More power, directional: Focus all laser power onto a particle
- Rapidly tunable across the mid-infrared for spectroscopy

Scan Mode
- Single wavelength, scan the sample quickly
- Can be done multiple times for multiple wavelengths at high speed.
- Understand the spatial distribution of known components
- Locate discrete particles

Sweep Mode
- Single Position
- Full Sweep available wavelengths
- Utilize full spectrum for library matching

Figure 3. 8700 LDIR modes of action.

The analyst can spread the particles over the relatively large area to avoid aggregation or agglomeration and improve image recognition of the particles. One of the downsides of this method is that the analyst needs to transfer the particles from the filter to the slide, which can lead to lower recovery of particles or contamination. The second method allows for the instrument to analyze the particles directly on the filter, reducing the potential for contamination or loss of particles. The sample holder accommodates two of the special filters used, which speeds up the analysis. The design of the holder ensures sample flatness, an important requirement for this method to produce reliable results. The key component of this method is the filter itself, which is a polycarbonate membrane coated with a highly IR-reflective, thin gold or aluminum coating with a pore size of 0.8 µm. This type of filter allows for visual and IR imaging of the particles and for their automated identification and statistical data collection. Additionally, it is commercially available (Figure 4).

On-filter analysis

- Less laborious
- Reduce the potential for contamination or particle loss
- Analyzing two samples sequentially – time saving
- Flatness can be achieved easily

Low-e slide analysis

- Characterizing large number of particles in a sample
- Sample needs to be transferred from filter to slide

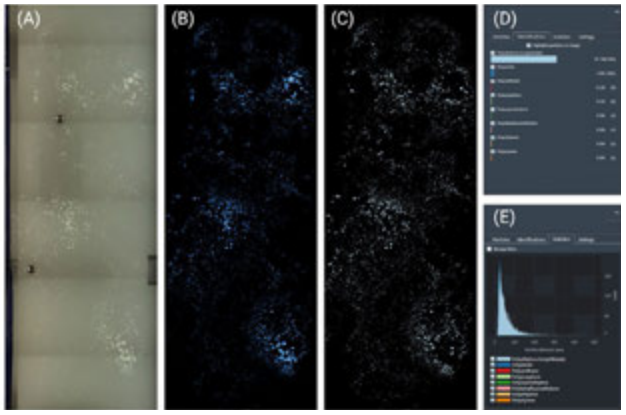
Figure 4. Particle analysis on different substrates.

Video

LDIR: a fast and accurate solution for microplastic analysis

A practical example: Microparticles from PET bottles

For this analysis, Agilent researchers ground up plastic bottles using clean, standard metallic files. They collected the particles in an ethanol-containing vial and pipetted small aliquots of the solution into 5 ml of absolute ethanol. For the glass slide analysis, they transferred multiple 10 µL aliquots onto the slides. For the on-filter analysis, they vacuum-filtered 5 mL of the solution using the gold-coated filters and transferred the filters to the holder after sufficient drying. The particle analysis workflow involves generating visual and IR images of the particles, which the operator can use to define areas of interest.
The instrument then produces a false color image, highlighting the particles and automatically generating statistical data on them. In the case of the low-e reflective glass slide (Figure 5), the total number of particles detected was close to 8,000, with sizes varying from 10 µm to 486 µm in diameter. The software correctly identified 95.2% of them as PET, 4.6% as polyamide, and small levels of contaminants, such as polyurethane and polypropylene.

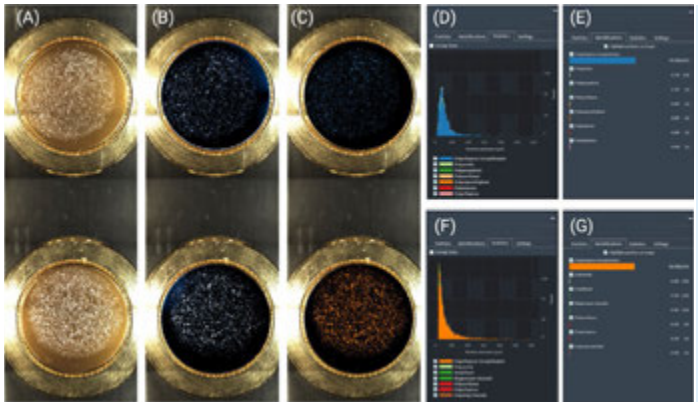


PET 95.2% (7566)

- The number of particles detected on the infrared reflective glass slide totaled **7,949**, spanning a size range of 10 to 486 µm in diameter.
- Out of the detected particles, **95.2%** (7,566) were correctly identified as PET, 4.6% (362) were polyamide, and insignificant numbers of other trace contaminants (polyurethane, polypropylene, and few others).

Figure 5. Method A: particle analysis workflow on low-e reflective glass slide.

For the on-filter analysis, the researchers used the gold-coated membrane filters supported by a small-pore glass frit to carry out the vacuum filtration step to isolate the particles. They used a gentle vacuum pressure of 700 mbar to avoid damaging the filters, completing the filtration in 30 seconds. They mounted the filters onto their circular holders, which allows two of them to be placed on the instrument's stage (Figure 6). The results from this setup are better than the results produced with the low-e glass slides; they correctly identified 99.2% of the particles as PET, and 0.8% as polyamide and polypropylene in one of the holders. On the other holder, the results were similar: 98.4% PET and <1.6% polyamide and others. The number of particles in each holder was around 5,000, the upper limit for the direct-on-filter workflow. With more particles, it would be difficult to have them separated so the imaging software could detect them. In addition, analysts should remove fibers as much as possible, especially long ones, as they make it difficult for the software to separate the particles of interest. Under normal conditions, the analysis takes from 8 to 10 s per particle, as the stage must move to each particle and acquire focus. Particle detection and particle count determination on a whole filter takes no longer than 20 minutes. To determine the effect of particle size on the quality of the spectral matches, the researchers compared the Hit Quality Index [HQI] score obtained by the Clarity software in the automated analysis workflow, where an HQI of 1.0 is an identical library match.



PET 99.2% (4347)

Out of the detected particles, **99.2%** (4,347) were correctly identified as PET, and 0.8% were identified as polyamide, polypropylene, and others.

PET 98.4% (5324)

5411 particles were detected with **98.4%** (5,324) identified as PET and < 1.6% identified as polyamides and others.

Figure 6. Method B: particle analysis workflow on two gold-coated filter samples.

They found that in the size range from 100 µm down to 10 µm, HQI values for the low-e slide method ranged from 0.931 to 0.922, respectively (Figure 7). With the gold-plated filters, the HQI values ranged from 0.941 to 0.935. At particle sizes below 10 µm, it becomes more difficult to obtain good quality spectra and the analysis becomes challenging. Finally, comparing the results from 10 different runs, researchers observed excellent repeatability, with <1% variation in the number of particles detected. The provided library contains data derived not only from clean microplastics, but also from weathered ones, and Agilent continuously updates it with data coming from the microplastics analysis community.

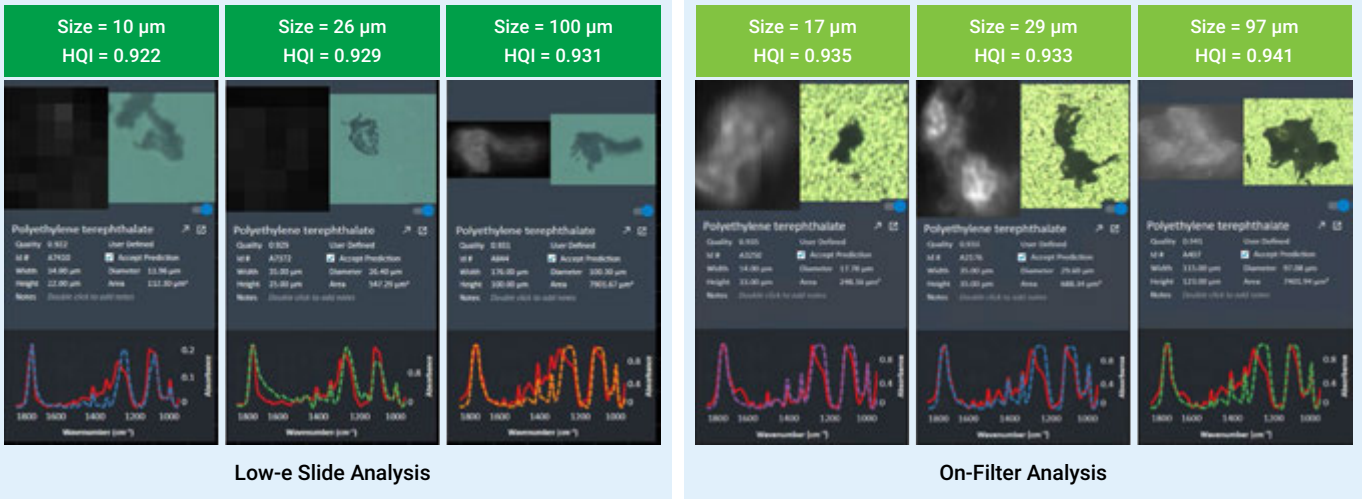


Figure 7. Hit quality and size information of particles analyzed on low-e and gold-coated polyester membrane filters obtained from Agilent Clarity software.

Conclusions

On-filter analysis of microplastics by LDIR imaging greatly improves the speed and repeatability of particle detection, identification, and classification of these ubiquitous materials. Eliminating the need to transfer samples from the filter to a slide greatly reduces the potential for contamination and sample loss. The tunable laser enables not only imaging the particles, but also the swift acquisition of their IR spectra. Using the specifically designed stages, filter holders, imaging software, and IR spectra library, analysts can process high numbers of samples with fast throughput and excellent results.

Darren Robey
Product Manager, IR Imaging
Agilent Technologies

Contributing Authors:
Wesam Alwan and David Troiani
Agilent Technologies

Best Practices for Microplastics Sample Preparation: Focus LDIR Workflow

Microplastics are an emerging issue and the quality of microplastics research is hindered due to their unique physical and chemical characteristics. Thanks to developments from Agilent, scientists can optimize their microplastics workflow to accurately quantify microplastics in various matrices (environmental and consumer products) via the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging system.

By Subharthe Samandra and
Dr. Wesam Alwan

For microplastic extractions, the general workflow begins with sample collection, followed by extraction, clean-up, and density separation; from there, scientists can analyze the data on various instruments. However, to evaluate the method, researchers must provide data on their quality controls. Environmental samples contain organic and inorganic matter, so prior to microplastic isolation, the organic matter must be removed (for example, leaves, twigs, hair, cotton fibers, protein, and algae should be isolated via Fenton's Reagent, enzymatic digestion, or another process). Then, the microplastics must be separated from the inorganic matter in the sample, like sand, soil, shells, salt, crystals, and glass. This can be done via sieving, filtration, or density separation. One case study in partnership with the University of Melbourne examined Australians' exposure to microplastics through bottled drinking water. First, the bottled water was filtered through a 5 µm pore-size PC filter paper. The filter paper is then flipped onto a Low-e IR slide to be analyzed via LDIR. The study found that, on average, there were 13 different microplastics present in each liter of bottled water, with an average size of 77 µm. Water bottles sourced internationally had about four times greater contamination than those sourced from Australia (Figure 1). The most probable source for some of these microplastics was fragments of the cap while opening and closing the cap. This study was significant, but there was still an opportunity for improvement. Indeed, the method used— the best available at the time of the test—still lost about 20 percent of the microplastics in the sample due to the process.

Agilent developed a new on-filter analysis method using a gold-coated filter in response. Here, two different bottled water brands through 0.8 µm pore-size gold filter papers. The filter paper was then transferred onto a slide holder for direct analysis (Figure 2).

On-demand event

Sample preparation for microplastics by LDIR: best practice

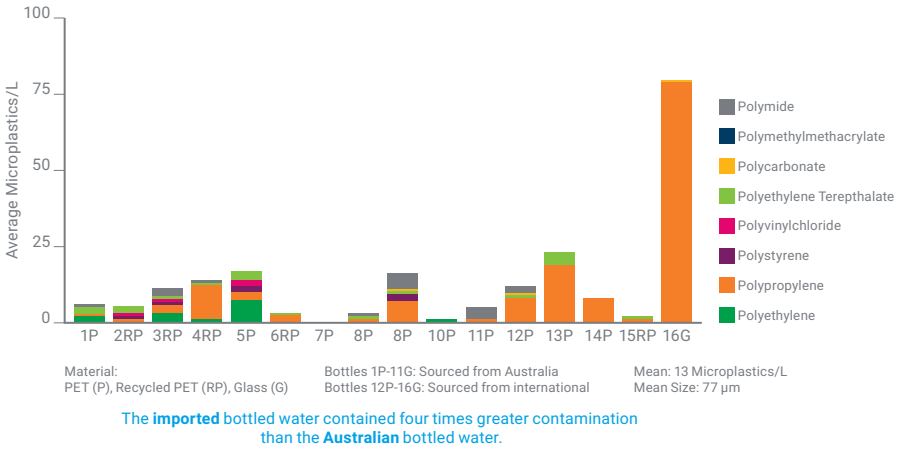


Figure 1. Direct microplastics analysis of bottled drinking water using gold-coated filter.

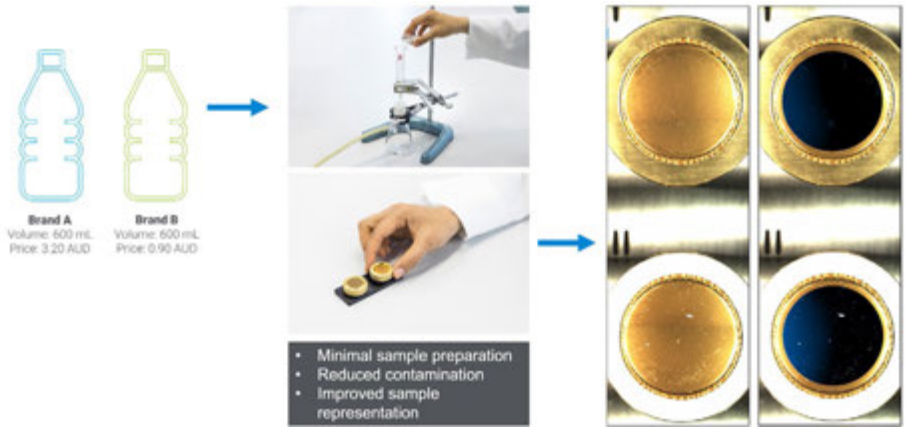


Figure 2. Microplastic contamination of bottled water bought from Australian supermarkets.

Another study examined the microplastic contamination of an unconfined groundwater aquifer in Victoria, Australia. A one-liter sample was first filtered through a 15 µm PC filter paper to remove any fine sediments; then, that filter paper was transferred into a vial that contained hydrogen peroxide and incubated at 60 ºC to remove any organic matter. Finally, any microplastics in the sample were isolated from large inorganic matter via calcium chloride. The calcium chloride solution was filtered through 5 µm PC filter paper and flipped onto an IR reflective slide for analysis on LDIR. The result was 100 percent detection of microplastics (Figure 3), with an average size of 89 µm. The adjacent land was determined to be the source of these microplastics, which found their way into the soil and ultimately filtered into the water. For example, bore 3, adjacent to a meat facility, presented PVC as the main contributor, which is a type of plastic they use in cryovacing. Bores 5 and 6 were adjacent to a plant nursery that used plastic mulching (generally made of PE). Indeed, PE was the main contributor to contamination at both these bores.

Whitepaper

Best practice for on-filter analysis of microplastics using the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System

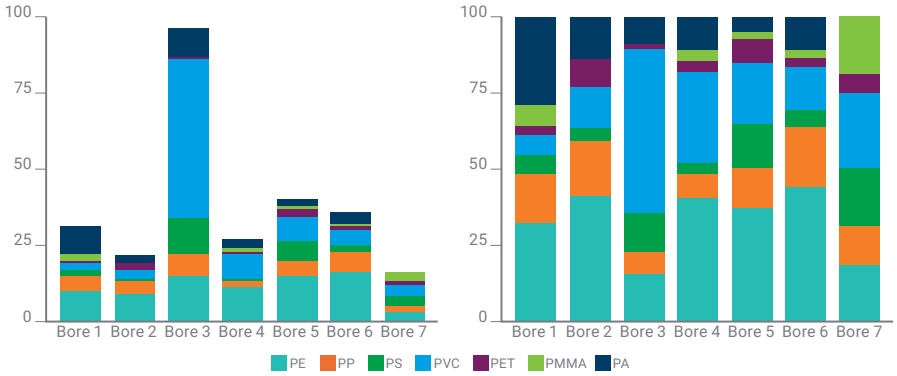


Figure 3. Average number of microplastics in groundwater from each bore (left) percentage contribution of each type of polymer in each bore (right)

Best practices for microplastic characterization

An important step in overcoming this pressing global environmental issue is the advancement of research and addressing the challenges associated with microplastics characterization. Agilent provides the necessary workflow solutions for researchers to further understand this emerging environmental challenge. First, Agilent provides the 8700 LDIR chemical imaging system, which offers a fast and automated solution well-suited for microplastic analysis. The new direct on-filter analysis method promotes easy sample preparation and reduced contamination, increasing the representation of the sample; this is an upgrade from the infrared slide method. To further ensure the accurate identification of microplastics, Agilent launched a new microplastic library (including microplastic and non-microplastic materials to cover all possible contaminations found in samples). Finally, Agilent has created a best practice guide to help labs achieve the best possible harmonized microplastic characterization. The following are some of the most important best practices in the guide.

Lab environment quality control

Before any microplastic testing is performed, Agilent recommends implementing quality control measures in the lab environment.

One important element to consider is air quality, which should be monitored to prevent sample contamination; the aim here should be maximum air purity and minimal airborne contaminants. Labs can use fume hoods or air purifiers to achieve this. Second, Agilent recommends examining the reagents used in the lab. Specifically, the water quality used for sample preparation or equipment cleaning. This should be checked by analyzing blanks for any possible baseline microplastic contamination. It is also recommended to check other reagents, like the filtered reagent, for any possible contamination. Third, it is recommended to use thoroughly cleaned glassware (obviously, plastic should be avoided when testing for microplastics), and, once washed, glassware should be covered with aluminum foil to prevent contamination from the lab environment.

Finally, personal protective equipment (PPE) recommendations include lab coats made from natural or particle-free materials. Lint rollers can be used to remove tiny fibers from clothing. Some gloves, like latex gloves, can contain stearates, which should not be used during microplastic analysis. Of course, hand washing is essential, as is avoiding any personal care products that contain microbeads, as they can affect the microplastic analysis results.

Sample filtration and filters handling

Gold-coated filters are recommended for use with LDIR. Since their thickness is roughly 22 µm, they require careful handling and should not be reused. Agilent provides special rounded-edge tweezers, which should be used to transfer the filters onto the filtration stem to avoid any damage to the filter. It is also recommended to wipe the filtration stem surface with ethanol before use to avoid trapping silica particles underneath the filter and compromising its flat nature and ability to detect the number of particles in the sample. Filtration Workflow The first step is to place the filter on the filtration stem. As previously mentioned, these filters are very delicate; as a result, it's recommended to switch to gentle vacuum pressure once the filter is on the stem to keep it in place. The second step is placing the funnel and securing it to the filtration assembly with a clamp. The assembly is now ready to be used. Once the sample filtration is finished, the vacuum should be turned off, and the filter should be left to air dry at room temperature; this takes about two minutes. After removing the filter from the glassware using the special tweezers, cleaning the filter holder with ethanol is recommended to remove any particles present. Then, place the filter on the raised platform and thread the brass retaining ring to secure it. The filter holder is now ready to be inserted into the system for analysis.

Particle analysis workflow settings

While the particle analysis workflow is fully automated and has built-in settings, analysts can adjust settings based on their needs. Here are some alternative settings:

- Auto Scan: Enabling auto scan gives the software full analysis control, including automated sensitivity and minimum and maximum particle sizes in the sample. Analysts can disable this option to input their own parameters.
- Collect Visible Images: High magnification images are used to improve the accuracy of particle size measurements but can be disabled if users want to increase the speed of their analysis. If disabled, particle sizing data is taken from the infrared images generated for each particle.
- Particle Sensitivity Slider: This sensitivity setting can be adjusted to detect smaller and fainter particles in the scanned area if desired.
- Classification Range: This describes how closely the sample spectrum matches the reference library. Users can adjust the classification range criteria based on their reporting requirements. A score of one represents the highest quality results.
- Particle Diameter: Users can change the minimum size detected. The default setting considers particles in the range of 20 to 500 µm.
- Size Classification Ranges: The "bucket" where each particle is placed can be customized according to lab needs.

Data processing and reporting

At the end of the analysis, the software will provide statistical data, including the total number of particles and the total particle count within each size range. Using the identification feature, the software will also identify the type of polymer for each particle detected and provide an infrared and visible image for each particle analyzed. The software also allows users to export the details of each particle detected into a comprehensive Excel spreadsheet. Researchers typically report in one of two ways. The first way is to report only microplastic particles based on a hit quality index according to the lab's specifications. Another way to report is to include all particles detected within the sample, including non-microplastics, based on the selected hit quality index. This can be helpful if a lab wants to explore the ratio of nonmicroplastics to microplastics in a sample or conduct quality control tests.

Conclusions

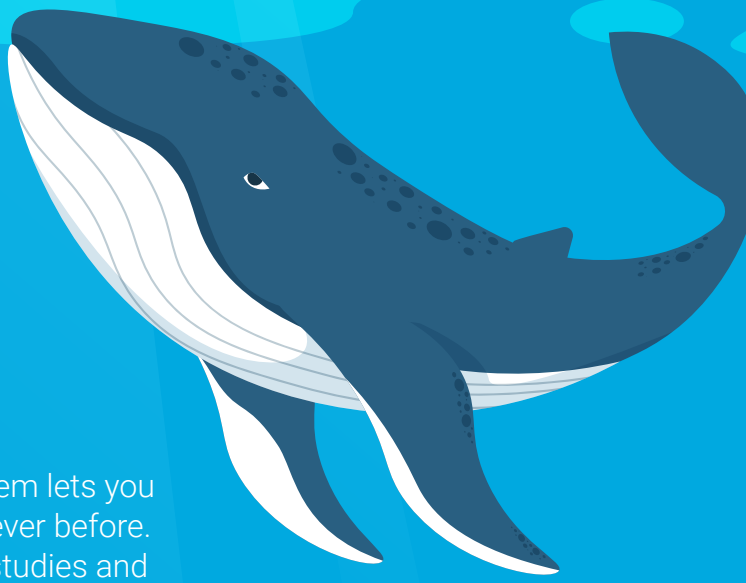
While microplastic characterization still represents a challenge for many labs, LDIR provides a fast and automated workflow that yields high-quality, accurate, and reliable data for microplastics analysis. To achieve accurate and reproducible analysis of microplastics, certain practical aspects should be considered, such as optimizing Analysis Techniques Sample Prep the lab environment, reducing sample contamination, and sample handling. The recent white paper published by Agilent describes the best practices for performing accurate on-filter microplastic analysis using the Agilent 8700 LDIR Chemical Imaging System. Additionally, Clarity software provides extensive information for each particle detected, such as size, ID, and visible and infrared images. For more information, Agilent.com offers many valuable resources for LDIR users, microplastic analysis, and other useful applications.

Subharthe Samandra
Ph.D. Candidate
University of Melbourne
Agilent Technologies

Dr. Wesam Alwan
Applications Scientist
Agilent Technologies

Accurate Microplastics Analysis

in **Minutes, Not Hours**



The automated Agilent 8700 LDIR chemical imaging system lets you obtain high-quality images and spectral data faster than ever before. So, you can perform confident large-scale microplastics studies and monitoring activities.

Find the needle in the haystack


Biota


Drinking water (bottled or tap)

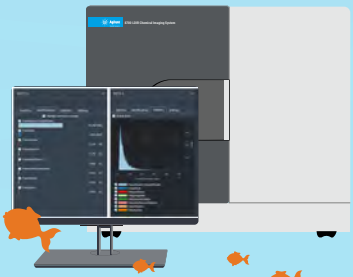

Clean sediment (sand)


Dirty sediment (soil)


Dirty water (river water)

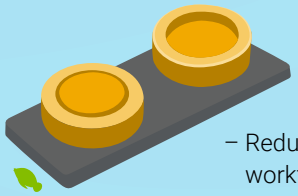

Food

Agilent 8700 LDIR chemical imaging system



- Microplastics analyzed direct on-filter
- Same benefits as mid-infrared spectroscopy
- Fast and simple to use
- Fully automated microplastics workflow

On-filter analysis: A giant leap forward in speed and throughput



- Reduce your sample preparation workflow by two steps
- Save 2–3 hours of water evaporation

Hit “play” and relax

The Agilent 8700 LDIR features a built-in automated particle analysis workflow.

- Two-sample filter holder allows samples to run overnight
- Walk-away operation lets analysts focus on other tasks
- Data generated in real time
- Full results available at end of analysis

Learn more:
www.agilent.com/chem/8700-ldir

Buy online:
www.agilent.com/chem/store

Get answers to your technical questions and access resources in the Agilent Community:
community.agilent.com

U.S. and Canada
1-800-227-9770
agilent_inquiries@agilent.com

Europe
info_agilent@agilent.com

Asia Pacific
inquiry_lsca@agilent.com

DE-002042

Published in the USA, October 22, 2024
5994-7801EN

Agilent
Trusted Answers