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Application News

Fourier Transform Infrared Spectrophotometer IRXross™/IRTracer™-100
Infrared Microscope AIMsight™

Technique for Measuring Microplastics Collected on Various Filters Using a Particle Filter Holder

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User Benefits

- Particle filter holders provide clear microscope images of samples, enabling highly accurate measurements.
- Microplastics can be analyzed accurately and efficiently by using a particle filter holder with a high-speed mapping program and a particle analysis program.

■ Introduction

Since microplastics (MPs) (with diameters ranging from a few µm up to 5 mm) are widely recognized as an environmental problem in marine environments, Japan's Ministry of the Environment has issued guidelines for preparation and measuring MPs in environmental surface waters.[1)]

The guidelines include examples of using a Fourier transform infrared spectrophotometer (FTIR) to identify plastics by the total reflectance measurement method. However, to identify fine MPs, they recommend using instruments like infrared microscopes.

In recent years, an increasing number of studies have analyzed fine MPs smaller than 100 µm. Measuring MPs with an infrared microscope requires collecting samples on a filter. Various types of filters can be used for infrared microscopes, each with unique characteristics. But if a filter cannot be held horizontal during drying or measuring, it can develop wrinkles that might decrease measurement accuracy. Therefore, a particle filter (PF) holder was developed that can be placed directly on an infrared microscope stage while holding the filter horizontally. This article describes using the IRXross Fourier transform infrared spectrophotometer, an AIMsight infrared microscope (Fig. 1), and a PF holder to evaluate the properties of various filters and measure a standard sample of MPs.

Fig. 1 IRXross™ FTIR Spectrophotometer (Left) and AIMsight™ IR Microscope (Right)

■ Filter Selection

Table 1 indicates the characteristics of typical filters used to filter MPs. The larger the outside diameter of a filter, the shorter the filtration time and the less MPs overlap. But it also results in a larger measurement range, which might require more time to search for candidate MP particles on the filter and longer measurement times.

Typically, 13 and 25 mm diameter filters are most commonly used. The filter pore diameter is selected based on the size of the target MPs to be collected. Small pores can collect smaller particles, but they tend to require more time for filtration. (Table 1 indicates reference times for filtering standard MPs dispersed in purified water.) The infrared measurement range will also depend on what the filter is made of. Examples of transmittance data for filters alone are shown in Fig. 2.

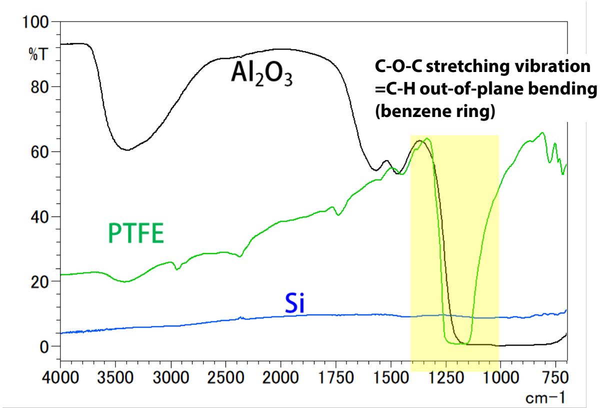


Fig. 2 Transmittance of Respective Filter Types

Due to the absorption of PTFE near 1,200 cm^{-1}, it can overlap with C-O-C stretching vibration peaks contained in the spectra of polyethylene terephthalate and other MPs, making it difficult to clearly recognize the peaks in some cases. Similarly, Al_2O_3 absorbs light in the 1,200 to 700 cm^{-1} range, which can make it difficult to recognize =C-H out-of-plane bending (benzene ring) peaks contained in polystyrene (PS) spectra. On the other hand, using filters made of stainless steel enables transmittance to be measured, depending on the pore diameter. Therefore, filters must be selected based on their characteristics and the material to be analyzed.

Table 1 Characteristics of Filters Compared in this Article

Filter	Material	Pore Diameter (µm)	Measurement Method*1	Pricing	Comments	Filtering Time (s)*2	Infrared Measurement Range*3
Hydrophilic PTFE	Polytetra-fluoroethylene	0.45	Transmission	Inexpensive	Easily wrinkled	30	4,000 to 1,300 cm^{-1} 1,000 to 700 cm^{-1}
SUS	Stainless steel	8	Transmission /Reflection	Medium	Easy to achieve a flat surface	2	4,000 to 700 cm^{-1}
Au/PC	Gold-coated polycarbonate	0.8	Reflection	Expensive	High reflectance	12	4,000 to 700 cm^{-1}
Al_2O_3	Aluminum oxide	0.2	Transmission	Expensive	Brittle, easy to achieve a flat surface	53	4,000 to 1,200 cm^{-1}

*1: Measurement method recommended by Shimadzu
*2: The reference value was obtained by suction filtering 1 mL of standard MPs dispersed in purified water through a 13 mm diameter filter. The value depends on the concentration and size of particles contained in the solution.
*3: The measurement range was based on the range (700 to 4,000 cm^{-1}) of the infrared microscope.

■ PF Holders

Two types of PF holders are available for either 13 mm diameter or 25 mm diameter filters. Both include a sample base, filter holder, cover, and filter mounting base. The cover has a magnetic latch that makes it easy to place and remove filters. The convex on the front surface of the filter holder is designed to stretch wrinkles apart in filters made of polymer and other flexible filters. In contrast, the reverse side is flat for holding filters made of metal or other hard materials. Fig. 3 shows holders holding the front sides of PTFE filters.

Fig. 3 PF Holders Holding PTFE Filters

The PF holder can hold the broad area of flat surfaces on the filter. Since PTFE filters are prone to wrinkling when dry, which can limit the area where infrared microscope images are in focus, it can be difficult to obtain sharp images over a broad area. If a large area is out of focus, it can be difficult to find particles, decreasing the accuracy of acquired data. However, by using a PF holder when drying PTFE filters, the flatness of the filter surface can be maintained, enabling clear microscope images that are in focus over a large area. That makes it easier to observe particles and enables more accurate measurements. Fig. 4 compares tiled images with one point in focus that were obtained from a PTFE filter dried with and without using a PF holder. The images are clearly in focus over a large area and the particles are easier to observe when the PF holder was used.

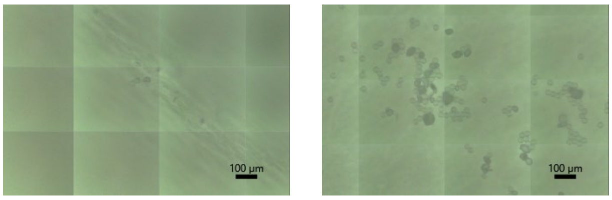


Fig. 4 Images of MPs on PTFE Filters Photographed
with a 15× Reflecting Objective Mirror
Left: Without Using a PF Holder; Right: Using a PF Holder

■ Example of Measuring MPs

Polystyrene (PS), polyethylene (PE), and polypropylene (PP) particles that fell through a 100 µm mesh were dispersed in purified water. They were then collected by suction filtering through PTFE, Al_2O_3, Au/PC, and stainless steel filters (Table 1). After collecting the particles, the filters were dried using a PF holder. Fig. 5 shows the filter holder placed in the infrared microscope. Measurement condition settings are indicated in Table 2. The PTFE and Al_2O_3 filters were measured by the transmission method, whereas the stainless steel and Au/PC filters were measured by the reflection method. For the high-speed mapping program in this measurement, a peak detection range of 3,400 to 2,400 cm^{-1} was specified for detecting the signal from hydrocarbons (C-H). If there are peaks in that range, the range will be scanned the specified number of times. A tiled image of each filter is shown in Fig. 6. The areas marked with a yellow box were measured. For more information about the high-speed mapping program, refer to Application News No. 01-01001-EN.

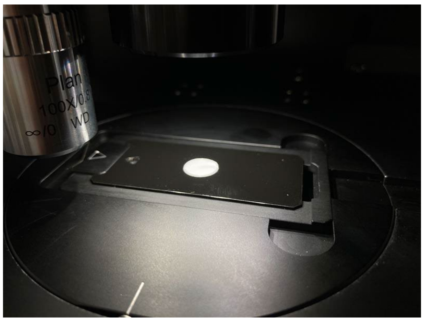


Fig. 5 PF Holder Placed in an Infrared Microscope

Table 2 Measurement Conditions

Instruments:	IRXross, AIMsight
Software:	High-speed mapping program and particle analysis program
Resolution:	8 cm^{-1}
Scanning Count:	50
Apodization Function:	SqrTriangle
Aperture Size:	20 µm × 20 µm
Measurement Interval:	20 µm
Mapping Range:	PTFE: 480 × 400 µm Al_2O_3: 360 × 560 µm Au/PC: 780 × 500 µm Stainless steel: 480 × 400 µm
Detector:	T2SL
High-Speed Mapping Conditions	
Noise Level:	PTFE, Al_2O_3, Au/PC: 0.05, Stainless steel: 0.1
Threshold Values:	PTFE, Au/PC: 0.5, Al_2O_3: 0.1, Stainless steel: 0.2
Excluded Ranges:	4,000 to 3,400 cm^{-1} and 2,400 to 700 cm^{-1}

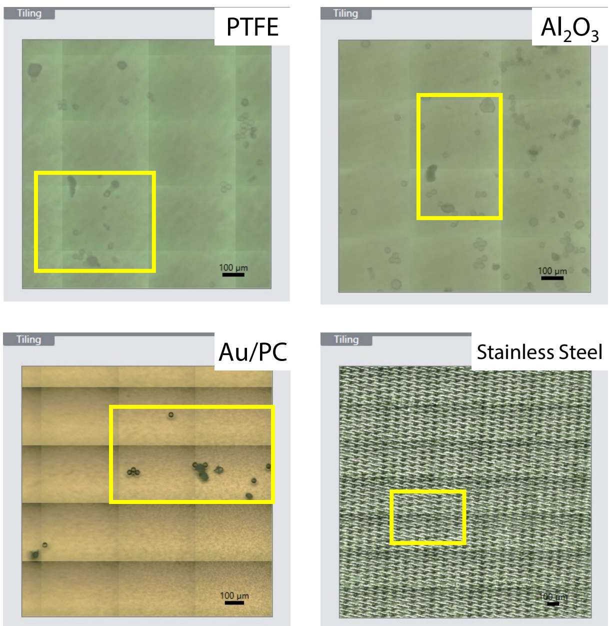


Fig. 6 Tiled Images of Each Filter

Examples of infrared spectra obtained using the high-speed mapping program are shown in Figs. 7 and 8. Fig. 7 shows that each type of plastic was more than adequately confirmed in infrared spectra, when collected on a PTFE filter. However, absorption by the filter in the 1,200 cm^{-1} region prevented a signal from being obtained, which decreased search scores. In addition, PS particles on the Au/PC generated interference fringes, which also decreased search scores.

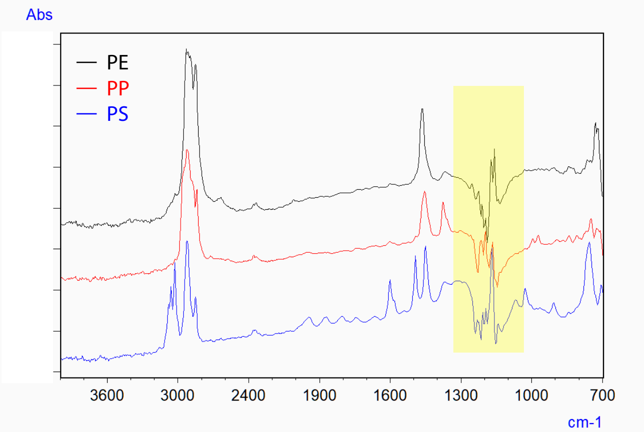


Fig. 7 Infrared Spectra of PE, PP, and PS Particles on a PTFE Filter

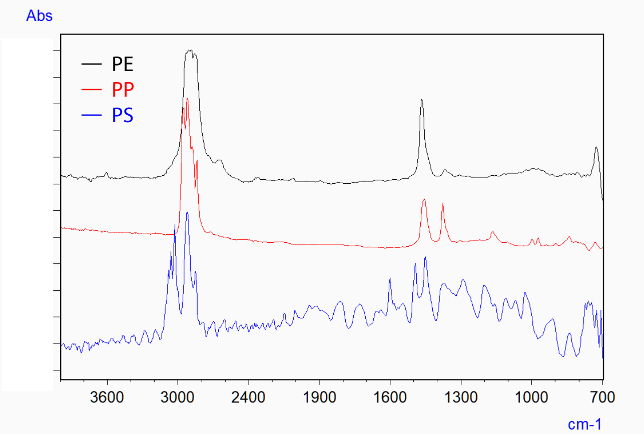


Fig. 8 Infrared Spectra of PE, PP, and PS Particles on an Au/PC Filter

The particle analysis program was used to color-code results for each plastic type (Fig. 9). For example, particle analysis results for a sample on a stainless steel filter are shown in Fig. 10. The particle analysis program can calculate not only particle count, major axis diameter, and Feret diameter values but also area, volume, and mass*4 values. For more information about the particle data analysis program, refer to Application News No. 01-00994-EN. In this example, the results indicate that 26 PS particles, 21 PP particles, and 1 PE particle were detected, and that the most common particle major axis diameter range was 20 to 40 µm.

*4 Mass and volume are calculated based on the following theoretical formula, which is indicated as equation (1) in the literature:

$$\log_{10}(M) = b \cdot \log_{10}(S) + a$$

This theoretical formula applies only to microplastics. Shimadzu cannot guarantee the validity of the mass results.
Tomoya Kataoka, Yota Iga, Rifqi Ahmad Baihaqi, et al. Geometric relationship between the projected surface area and mass of a plastic particle. Water Research. 2024;61:122061

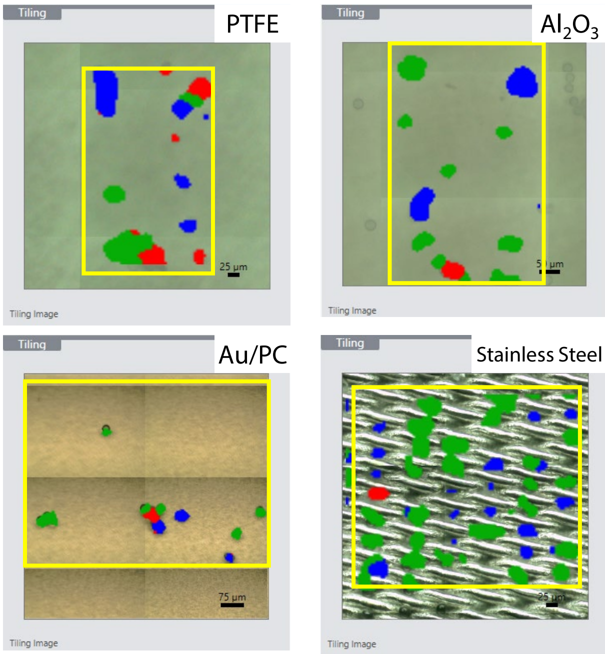
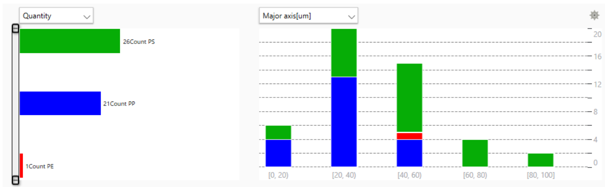


Fig. 9 Results from Analyzing Particles on Each Filter



ID	Component	Similarity	Minor axis[um]	Major axis[um]	Min Feret[um]	Max Feret[um]	Area[um2]	Volume[um3]	Mass[ug]
18	PP	723	10	10	10	13	100	2321	0.002089
19	PP	815	25	25	25	31	550	16210	0.014589
20	PP	772	10	15	10	17	150	3686	0.003317
21	PP	987	40	40	39	45	1125	36650	0.032985
22	PE	985	25	45	25	46	925	28072	0.026388
23	PS	928	65	75	65	99	2975	93410	0.099948
24	PS	840	10	30	10	31	250	5550	0.005938
25	PS	861	10	30	10	30	250	5550	0.005938
26	PS	930	61	172	61	172	5700	196026	0.209748
27	PS	918	35	40	34	46	1200	33180	0.035503
28	PS	913	55	85	55	91	3275	104221	0.111517

Fig. 10 Results from Analyzing Particles on a Stainless Steel Filter

■ Conclusion

This article describes using a PF holder to collect fine microplastic particles on various types of filters, and it provides a summary of the characteristics of each filter. Since there are differences in the wavenumber ranges and data acquisition methods for each filter type, users need to understand the individual characteristics of a filter when using it. The results also show that by using a PF holder to hold the filter flat, fine microplastic particles can be conveniently measured with an infrared microscope to obtain clear images and accurate measurements. Furthermore, a high-speed mapping program and particle analysis program can be used to accurately and efficiently analyze microplastics.

References

1) Ministry of the Environment, FY 2020 Comprehensive Assessment of the Current Status, Biological Impacts, and Other Considerations Regarding Marine Debris
https://www.env.go.jp/content/900543555.pdf
(Sep. 8, 2025)

Related Movie

1) Perform Microplastics Analysis More Quickly and Accurately
https://youtu.be/nBl3uOtRhNE?si=az2nTTKIVQLdZcKR

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