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Application News

Fourier Transform Infrared Spectrophotometer
Infrared Microscope

High-Speed Measurement of Microplastics Smaller than 100 µm Collected on a Filter and Efficient Analysis

—Using a High-Speed Mapping Program and Particle Analysis Program—

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User Benefits

- It enables high-speed measurements of microplastics (MPs) smaller than 100 µm that are collected on a filter.
- Particle analysis program can identify the types of MPs and easily analyze color-coded by type and counting the number of MPs.
- In addition to calculating the size of MPs (minor axis, major axis, Feret diameter, and area), it can also estimate the volumes and masses of particles.

■ Introduction

Microplastics (MPs) with diameters ranging from a few µm to 5 mm are widely recognized as an environmental problem in marine environments, and reports of MPs smaller than 100 µm in drinking water are attracting particular attention.[1)] To confirm the number and shape of MPs smaller than 100 µm and qualitatively analyze them, samples are usually collected on a filter and observed and measured using an infrared or Raman microscope. For that process, mapping measurements of a specified area is simpler than searching for candidate MPs on the filter and performing point measurements, and it reduces the risk of overlooking particles. However, it is more time-consuming, and it also requires analyzing the MPs after they are measured to identify the candidates, determine their sizes, aggregate the data, and so on. Therefore, a High-Speed mapping program and particle analysis program were developed to reduce the time required for these measurement and data analysis steps.

In this example, High-Speed mapping was used to measure a standard MPs sample, and the particle analysis program was used to analyze the distribution of MPs within the sample.

Fig. 1 IRTracer™-100 + AIMsight™ System

■ Measurement Sample and Particle Filter Holder

Fig. 1 shows the external appearance of the system. A reference material (a tablet) from Chiron AS was prepared as the measurement sample. The tablet was dispersed in filtered purified water and collected as a sample on an Si filter (10 mm square, with 5 µm diameter pores) [*1]. The Si filter was installed in the particle filter (PF) holder (Fig. 2), which was secured with the cover, and then it was placed in the infrared microscope (Fig. 3). (The PF holder is designed to smooth out wrinkles or other irregularities in round filters.)

*1 Purchased from the Korea Institute of Analytical Science and Technology (KIAST).

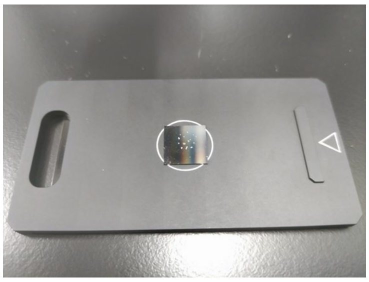


Fig. 2 Si Filter Installed in the PF Holder

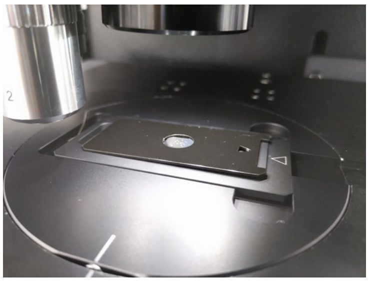


Fig. 3 PF Holder Placed on the Dedicated Stage and Loaded in the Infrared Microscope

■ Measurement Conditions and High-Speed Mapping Program

Measurement conditions are listed in Table 1. Mapping measurements are especially useful for measuring samples with many of target objects in the field of view. Normally, they take longer for larger measurement areas or larger numbers of measurement points, but that can be shortened with the High-Speed mapping program.

The program detects peaks based on data from the first scan at each measurement point and the specified noise level and threshold value settings. If no peak is confirmed, the program moves to the next measurement point. (If a peak is confirmed, the same number of measurements is performed as the number of scans specified.)

In this example, a peak detection range of 3,200 to 2,800 cm^{-1} was specified for detecting the signal from hydrocarbons (C-H), and the data from peaks in that range were scanned the specified number of times.

Table 1 Measurement Conditions

Instruments:	IRTracer-100, AIMsight PF Holder (13 mm dia.)
Software:	High-Speed mapping program and particle analysis program
Optical System:	Transmission (imaging acquired from reflection)
Resolution	8 cm^{-1}
Accumulation:	30
Apodization Function:	SqrTriangle
Aperture Size:	20 µm × 20 µm
Step Size:	20 µm
Mapping Range	1,700 × 2,100 µm (1.7 × 2.1 mm)
Detector:	T2SL
High-Speed Mapping Conditions	
Noise Level:	0.02
Threshold Value:	0.4
Excluded Ranges:	4,000 to 3,200 cm^{-1} and 2,800 to 700 cm^{-1}

■ Measurement Results

The tiled image of the measurement range in Fig. 4 shows the large number of candidate measurement points. The 1.7 × 2.1 mm region in the red box was measured. The High-Speed mapping program shortened the measurement time to about one-eighth of the time required for regular mapping measurements.

Identifying the spectra of particle peaks confirmed the presence of polyethylene (PE), polyethylene terephthalate (PET), polystyrene (PS), and protein (Fig. 5). Typically, peaks are identified by searching for matching spectra one at a time. Here, however, the MPs distribution was confirmed using the particle analysis program indicated below.

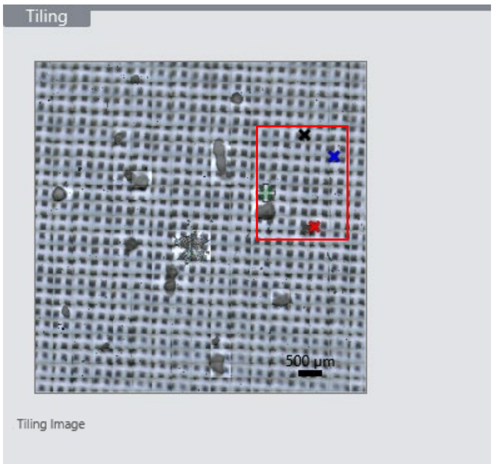


Fig. 4 Tiled Image and Measurement Range (Red Box) for the Measurement Range

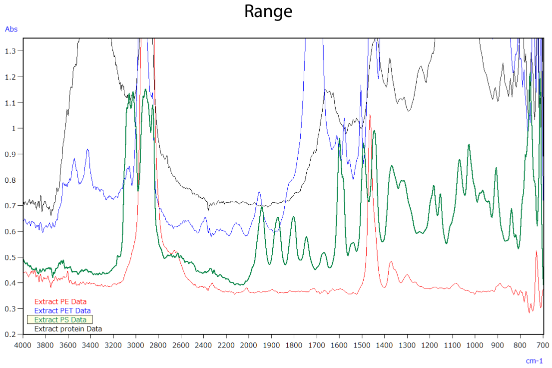


Fig. 5 Examples of Infrared Spectra

■ Data Analysis from the Particle Analysis Program

The measured data were analyzed using the particle analysis program. A screenshot of the particle analysis tab page is shown in Fig. 6.

The PE, PET, and PS components included in the standard sample were listed for analysis in the upper right of the particle analysis program. (In this case, the data indicated in Fig. 5 were registered.) Only data for the components with the corresponding selected checkbox were analyzed (including a template for analyzing the 15 representative types of plastic components). The threshold values used for the algorithm and similarity score can be specified in the data analysis condition settings.

After clicking the data analysis button, the components that exceeded the threshold value in the tiled image on the upper left were color-coded with the specified colors.

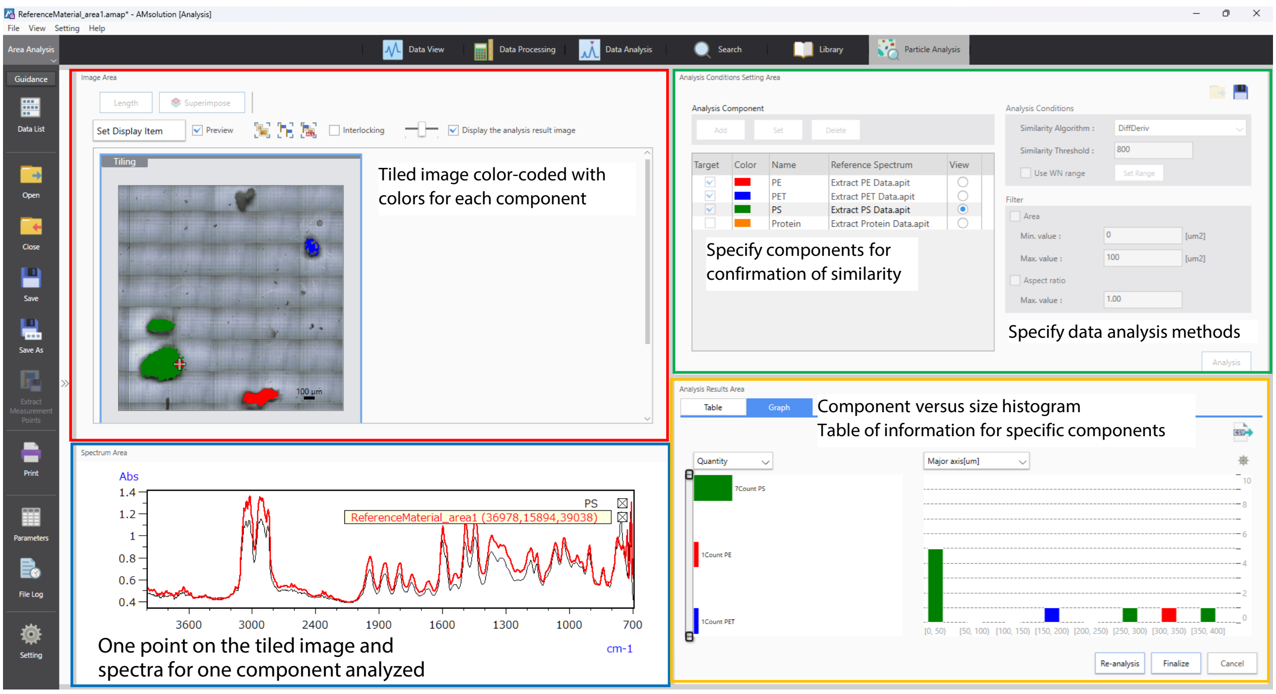
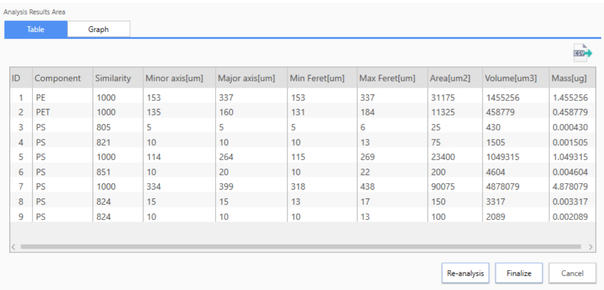


Fig. 6 Particle Analysis Program

In the lower right of the particle analysis program, there is a [Table] tab (Fig. 7) and [Graph] tab. Analysis of the data from the measurement range on the Si filter indicates the presence of 1 PE particle, 1 PET particle, and 7 PS particles. In addition, the histogram in Fig. 8 (right side) shows the presence of PS particles with a major axis up to 50 µm long and between 150 and 400 µm. The enlarged tiled image in Fig. 9 shows small PS fragments around the large PS cluster, which indicates consistency between the image and histogram results. The [Table] tab page shown in Fig. 7 shows information about individual components analyzed, including the calculated minor axis, major axis, Feret diameter, and area values. It also shows the estimated volume and mass values calculated for each particle based on the indicated formula*2. Tabular information data analysis results can also be output in CSV file format.

This shows that by using the particle analysis program, summary particle count and size values can be calculated for each type of MPs within the measurement range.



ID	Component	Similarity	Minor axis[um]	Major axis[um]	Min Feret[um]	Max Feret[um]	Area[um2]	Volume[um3]	Mass[ug]
1	PE	1000	153	337	153	337	31175	1455256	1.455256
2	PET	1000	135	160	131	184	11325	458779	0.458779
3	PS	805	5	5	5	6	25	430	0.000430
4	PS	821	10	10	10	13	75	1505	0.001505
5	PS	1000	114	264	115	269	23400	1049315	1.049315
6	PS	851	10	20	10	22	200	4604	0.004604
7	PS	1000	334	399	318	438	90075	4878079	4.878079
8	PS	824	15	15	13	17	150	3317	0.003317
9	PS	824	10	10	10	13	100	2089	0.002089

Fig. 7 Analysis Results Area ([Table] Tab)

Fig. 8 Analysis Results Area ([Graph] Tab)

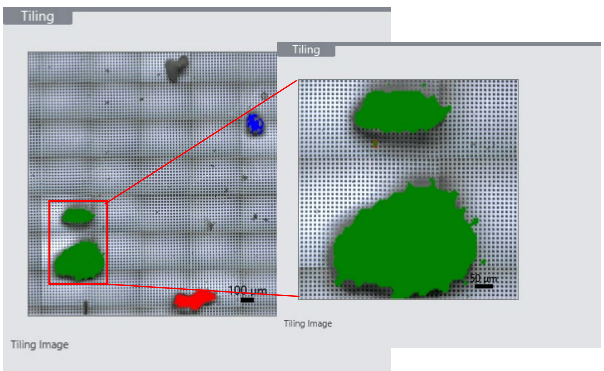


Fig. 9 Color-Coded Tiled and Enlarged Images

■ Conclusion

MPs that were collected on a filter were measured using an infrared microscope and the High-Speed mapping program. Because the High-Speed mapping functionality can measure locations in which only candidate MPs are present, samples can be measured faster than with regular mapping methods.

And with the particle analysis program, candidate MPs can be specified as components to be analyzed, so particle counts and size distributions within measurement areas can be easily determined. This example also showed that the volume and mass of particles can be estimated.

Counting MPs smaller than 100 µm while qualitatively analyzing the particles requires an instrument like an infrared microscope, which enables the High-Speed mapping program or particle analysis program to analyze MPs and MPs data more smoothly.

*2 Mass and volume can be calculated using equation (1) in the paper indicated below.

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

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. The geometric relationship between the projected surface area and the mass of a plastic particle. Water Research. 2024;61:122061.

<References>

1) Ministry of the Environment, “River and Lake Microplastics Investigative Guidelines,” Water Environment Management Division, Environmental Management Bureau, Ministry of the Environment, March 2024 (Refer to 6/10/2025)

<Related Applications>

1. Analyzing Microscopic Contaminants Embedded in Recycled Plastic, Application News No. 01-01001-EN

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01-00994-EN First Edition: Aug. 2025

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