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Application News

iSpect™ DIA-10 Dynamic Particle Image Analysis System
IRTracer™-100/IRXross™ Fourier Transform Infrared Spectrophotometer
AIMsight™ Infrared Microscope

Comprehensive Approach for Successful Microplastics Analysis

William Lipps[1], Yusuke Mizuno[1], Hiroki Maeda[2], Kazumi Kawahara[2], Kazuki Sobue[2]
1 Shimadzu Scientific Instruments, 2 Shimadzu Corporation

User Benefits

- Rapid measurement of total number of particles, size distribution, and shape in particles < 100 µm by Dynamic Particle Image Analysis System.
- Qualitative and quantitative microplastics analysis for particles > about a few hundred µm by Benchtop FTIR System.
- Qualitative and quantitative microplastics analysis for particles < about a few hundred µm by Infrared Microscope System.

■ Introduction

Generally, microplastics are evaluated by observing their appearance, measuring their number and size, as well as qualifying the materials.

Regarding the evaluation of the total number of particles, particle size distribution, shape, etc., we actively participated in data acquisition for standardization [1)], and ASTM D8489 (hereinafter referred to as D8489) was published in 2023 [2)]. D8489 focuses on smaller particles between the range of 5 - 100 µm, and provides information on particle size, size distribution, concentration, and shape.

On the other hand, infrared spectroscopy is a powerful method for evaluating the analysis of materials, and ASTM International is currently studying [1)] for standardization. ASTM WK87463, which is currently under investigation, is expected to be a standard for evaluating microplastics by FTIR and LDIR (Laser Direct Infrared) for particles of 20 µm to 5 mm.

In this article, we will introduce microplastics evaluation using iSpect DIA-10, a dynamic particle image analysis system in line with D8489, and microplastics material evaluation using FTIR.

■ Materials and Methods

As mentioned above, we conducted data acquisition for the standardization of D8489 and summarized the results in White Paper [3)]. In this document, only analyzed reference materials of plastic beads with a known diameter and concentration.

In this Application News, we include analysis of real plastic particles after simulating sampling and preparation by ASTM Practices D8332 and D8333. Here, we examine particles in the 5 - 100 µm range by the Shimadzu iSpect DIA-10 Dynamic Particle Image Analysis System (Fig. 1), and use the Shimadzu IRTracer-100 Fourier Transform Infrared (FTIR) spectrophotometer system with QATR™ 10 single reflection type attenuated total reflection attachment integral with sample compartment (Fig. 2) to identify some of the larger particles. Additionally, we analyzed the entire < a few hundred µm fraction by infrared microscope system using the Shimadzu IRXross FTIR spectrophotometer and AIMsight infrared microscope (Fig. 3).

Fig. 1 iSpect™DIA-10

Fig. 2 Benchtop FTIR System
(IRTracer™-100 and QATR™ 10)

Fig. 3 Infrared Microscope System
(IRXross™ and AIMsight™)

Microplastic particles were prepared according to procedure A of ASTM D8402 [4)]. Sheets of polypropylene (PP), pieces of polyethylene (PE), and a food container of polystyrene (PS) were obtained and manually shredded into fragments using a grater as shown in Fig. 4. Next, the plastic particles were mixed, and about 50 mg of the combined plastic was added to 1 mL methanol to create a synthetic D8333 [5)] mixture, then sieved through 212 and 100 µm.

Fig. 4 Preparation of PP Microplastic Particles

■ Evaluation of Microplastic using DIA-10

The <100 µm fraction of particles <100 µm was analyzed by the Shimadzu iSpect DIA-10 Dynamic Particle Image Analysis System for determination of size distribution, shape, and counting of particles between 5 - 100 µm. A 250 µL aliquot of the < 100 µm fraction was added to 2250 µL of 50% methanol and 50% glycerin according to D8489. Seven 150 µL replicates were introduced, captured, and analyzed.

Fig. 5 and 6 show a particle size distribution and scattergram, respectively, with particle size distribution of one of seven replicates of the < 100 µm fraction. Fig. 7 shows a thumbnail display of particle images.

The results indicate that particles are present in the size range of 5-100 µm for the < 100 µm fraction, and it is observed that the number of particles increases as they become smaller. Furthermore, from the scattergram and thumbnail display, it is apparent that the shredding process employed in this experiment has resulted in particles with a variety of aspect ratios.

Table 1 lists the concentration and repeatability data of all seven replicates differentiated by size. The results indicate that in the fractions of larger than 25 µm, the RSD decreases as the size of particles increases because of the small number of detected particles. However, when considering the concentration across the whole size range, the RSD is found to be within 10%.

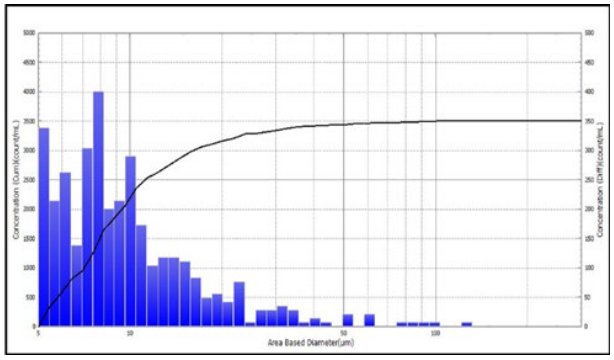


Fig. 5 Particle Size Distribution

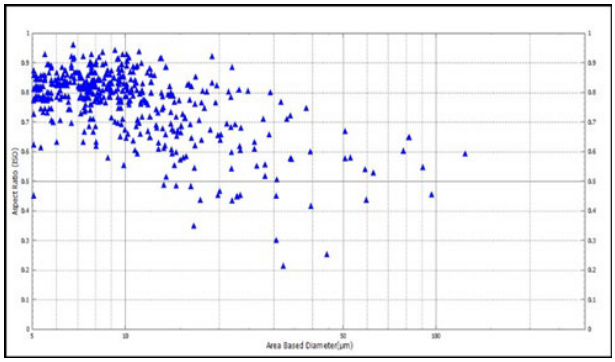


Fig. 6 Scattergram of Area Based Diameter (x-axis) and Aspect Ratio (y-axis)

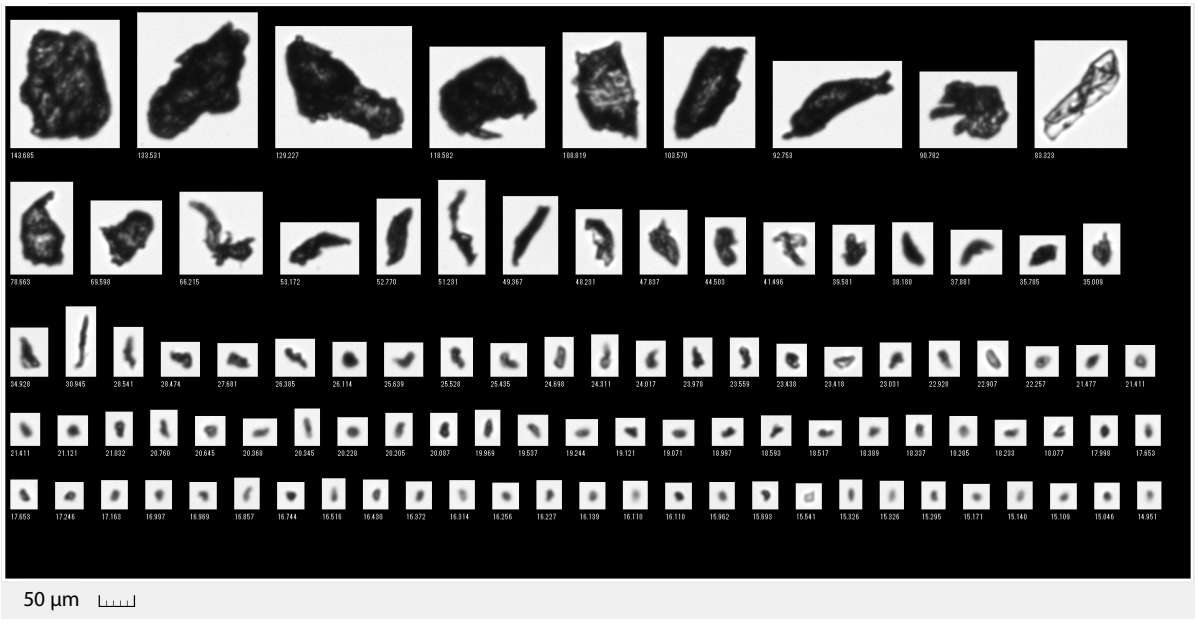


Fig. 7 Thumbnail Display of Particle Images and Ordered by Area Based Diameter (µm)

Table 1 Concentration and Repeatability Data of All Seven Replicates Differentiated by Area Based Diameter

Replicate number	Concentration (count/mL)*					
	All	5-10 µm	10-25 µm	25-50 µm	50-100 µm	> 100 µm
1	3253	1623	1250	214	83	83
2	3667	2051	1140	283	69	124
3	3764	2037	1271	290	90	76
4	3640	2141	1140	228	76	55
5	3419	1837	1167	235	111	69
6	3508	2051	1105	242	76	35
7	3101	1761	1050	186	62	41
Average	3479	1929	1160	240	81	69
S.D.	238.4	190.1	77.9	36.7	15.8	30.1
RSD	6.85%	9.86%	6.72%	15.31%	19.54%	43.59%
* Concentration is calculated from Number of Particles, Number of Frames and flowcell volume.						

■ Evaluation of Microplastic using FTIR

We selected a few of the larger particles that did not pass through the 100 µm sieve for qualitative analysis using the Shimadzu FTIR and attenuated total reflectance attachment. The IRTracer-100 and QATR 10 system are configured according to the conditions in Table 2, and three examples of photographs, maximum length, and infrared spectra are shown in Fig. 8.

The presence of particles from each mixed material was confirmed through qualitative analysis.

Table 2 FTIR Measurement Conditions

Instruments	: IRTracer-100, QATR 10
Resolution	: 4 cm^{-1}
Number of Scan	: 30
Apodization function	: SqrTriangle
Detector	: DLATGS

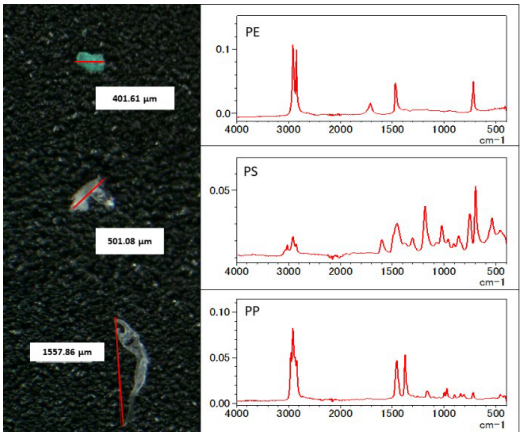


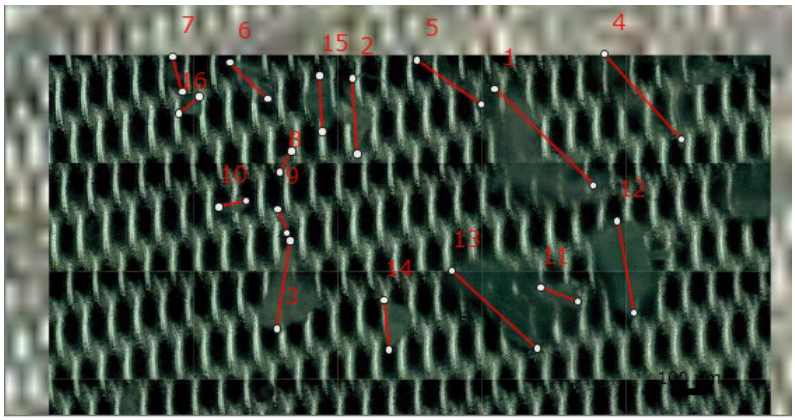
Fig. 8 Three Examples of Photographs, Maximum Length, and Infrared Spectra

Finally, we analyzed two < a few hundred µm fraction aliquots using the Shimadzu IRXross FTIR and the AIMsight infrared microscope. The IRXross and AIMsight system are configured according to the conditions in Table 3. For this test, 200 µL of the D8333 mixture of microplastics in methanol was transferred onto a 25 mm diameter 15 µm stainless steel mesh screen and filtered. Through two rounds of this filtration process, we obtained two filters that captured particles (designated as sample 1 and sample 2). Three individual measurement areas of 1.232 mm^2 were selected per aliquot from each of two filters, which had a filter area of 490.625 mm^2 each. Based on the measurement results, the measurement areas and the filter area, the number of particles retained on each filter was estimated to contain about 3000 – 4000 particles.

Table 3 Infrared Microscope Measurement Conditions

Instruments	:	IRXross, AIMsight
Resolution	:	8 cm^{-1}
Number of Scan	:	5
Apodization function	:	SqrTriangle
Aperture size	:	50 µm × 50 µm
Measurement interval	:	50 µm
Mapping range	:	850 µm × 1,450 µm
Detector	:	T2SL

Fig. 9 shows the AIMsight infrared microscope view of a measurement area of one aliquot with particle length as determined by the Shimadzu AMsolution software and the chemical identification of each particle as determined by the FTIR spectrum. Fig. 10 shows mapping images identifying the chemical identity of the individual plastic particles found. Areas where large numerical values were obtained for the plastic component are shown in red, while areas with small values are shown in blue. Fig. 11 shows the spectra of the three plastics.



No.	Length(µm)	Material
1	382	PP
2	209	PP
3	245	PP
4	319	PS
5	217	PS
6	145	PS
7	100	PS
8	67	PS
9	70	PS
10	78	PS
11	110	PS
12	257	PE
13	319	PE
14	138	PE
15	156	PE
16	74	PE

Fig. 9 Unit Area and Length (µm) of Each Particle with ID as Determined by FTIR

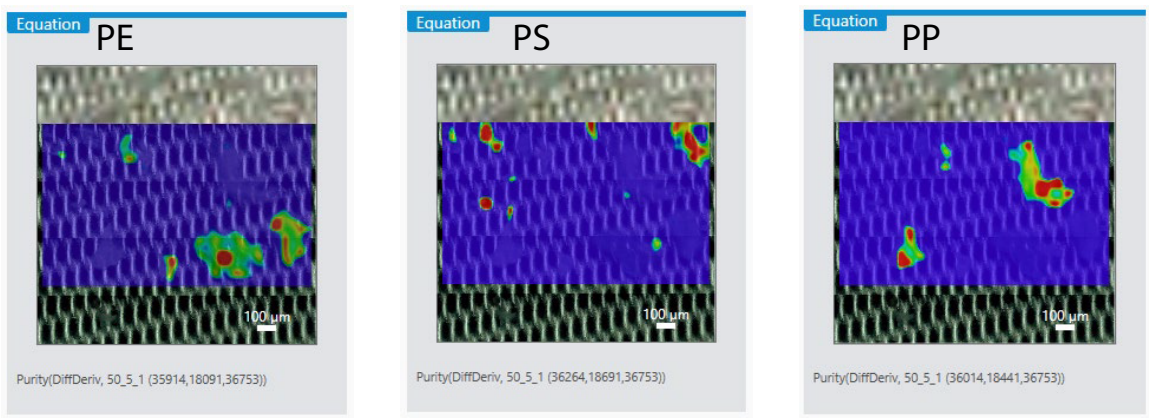


Fig. 10 Mapping Images Identifying Each Plastic in the Unit Area

The plastics in each measurement area were tabulated by chemical identity and count, averaged, and then calculated using the following equation to estimate the total plastic particles in the aliquot filtered.

Total particles = Number of particles in measurement area x filter area / measurement area

For sample 1 and sample 2, the average per measurement area and calculated number of microplastic particles per filter are shown in Table 4.

In this study, various particles with different maximum lengths and aspect ratios were found in all three materials, polypropylene (PP), polyethylene (PE), and polystyrene (PS). However, no clear correlation was observed between the material and the maximum length or shape of the particles. This is believed to be due to the particles being created using the same method. On the other hand, based on the particle counts for each material in sample 1 and sample 2, it is likely that polyethylene (PE) is present in larger quantities compared to polystyrene (PS) and polypropylene (PP). From this, it can be inferred that the detected particles using iSpect DIA-10 are also predominantly composed of polyethylene (PE).

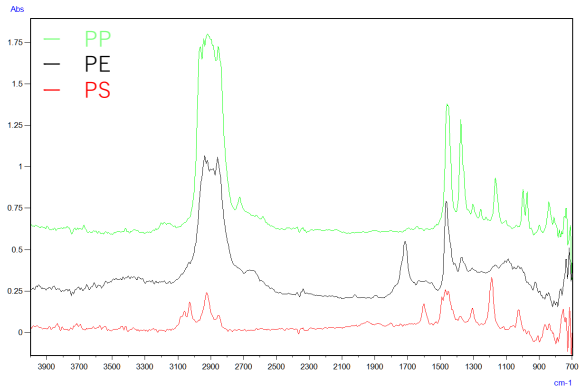


Fig. 11 Color coded FTIR Spectra of the Individual Plastics

Table 4 Average Particles Per Measurement Area and Total Particles per Filter

	Sample 1				Sample 2			
	PE	PS	PP	Total	PE	PS	PP	Total
Average Particles per measurement area	4.3	3.3	2.3	10.7	5.3	1.3	1.7	8.3
Total Particles per filter	1725	1327	929	4246	2110	517	663	3317

■ Conclusion

In this Application News we demonstrated use of the Shimadzu iSpect DIA-10 Dynamic Particle Image Analysis System and followed ASTM D8489 in the analysis of particle shape, size, and size distribution of real plastics between 5 and 100 µm. In addition, we followed ASTM work item WK87463 currently under consideration to measure and identify microplastic particles using microscopy and IR spectroscopy. Here, we used the Shimadzu IRTracer-100 FTIR and QATR 10 to identify some of the larger, > a few hundred µm, particles.

Next, with assistance of the Shimadzu AMsolution length measurement and mapping software, we used the Shimadzu IRXross FTIR and AIMsight infrared microscope to classify, enumerate and identify the < a few hundred µm particles. Using ASTM standards developed for sampling, sample preparation, and analysis for microplastics in water, the portfolio of Shimadzu Instruments enable a very comprehensive characterization of the distribution and composition of microplastics in water.

<References>

1) WK87463, New Test Method for Spectroscopic Identification and Quantification of Microplastic Particles in Water Using Infrared (IR) Spectroscopy
2) ASTM D8489, Test Method for Determination of Microplastics Particle and Fiber Size, Distribution, Shape, and Concentration in Waters with High to Low Suspended Solids Using a Dynamic Image Particle Size and Shape Analyzer
3) Shimadzu Whitepaper, New Standard Determination of Microplastics Particle and Fiber Size, Distribution, Shape and Concentration in Waters with High to Low Suspended Solids Using a Dynamic Image Particle Size and Shape Analyzer, September 2022
4) ASTM D8402, Standard Practice for Development of Microplastic Reference Samples for Calibration and Proficiency Evaluation in All Types of Water Matrices with High to Low Levels of Suspended Solids
5) ASTM D8333, Standard Practice for Preparation of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers Using Raman Spectroscopy, IR Spectroscopy, or Pyrolysis-GC/MS

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