Ministry of the Environment
FY2023 Environmental Technology Verification Program

Environmental Measurement Technology Field

Verification Report

March 2024

Verification Organization	:	**Saitama-ken Environmental Analysis & Research Association**
Name of Technology Subject to Verification	:	**Microplastic automatic preparation device**
Verification Applicant	:	**Shimadzu Corporation**
Verification Number	:	１４０-２３０５

— Contents —

Summary

Technology Subject to Verification	Microplastic automatic preparation device
Verification Applicant Address	Shimadzu Corporation 1, Nishinokyo Kuwabara-cho, Nakagyo-ku, Kyoto 604-8511, Japan
Verification Organization Address	Saitama-ken Environmental Analysis & Research Association 1450-11 Kamiko-chou, Omiya-ku, Saitama
Verification Period	August 29 to December 28, 2023
Purpose of Technology	The purpose of the technology subject to verification is to automate the sample preparation processes (oxidation and density separation) described in Guidelines for River and Lake Microplastics Monitoring Methods* and thereby reduce human error and ensure good sample preparation reproducibility.

* Guidelines for River and Lake Microplastics Monitoring Methods, March 2023, Water Environment Management Division, Environmental Management Bureau, Ministry of the Environment

1. Overview of the Technology Subject to Verification

1.1 Principles, Technology Objectives (Impact on Environmental Conservation), and Characteristic Feature

The technology subject to verification is an automatic sample preparation device that performs “oxidation” and “density separation” automatically in a single reaction vessel (figure below). Preparing samples automatically with this technology ensures that sample preparation occurs with good reproducibility and empirical results are not affected by the skill level of the worker. This technology will improve the accuracy of empirical data used in studies into the sources and distribution of microplastics (MPs) and environmental conservation measures in MPs Pollution, and can be expected to have a positive impact on the development of future measures concerning MPs.

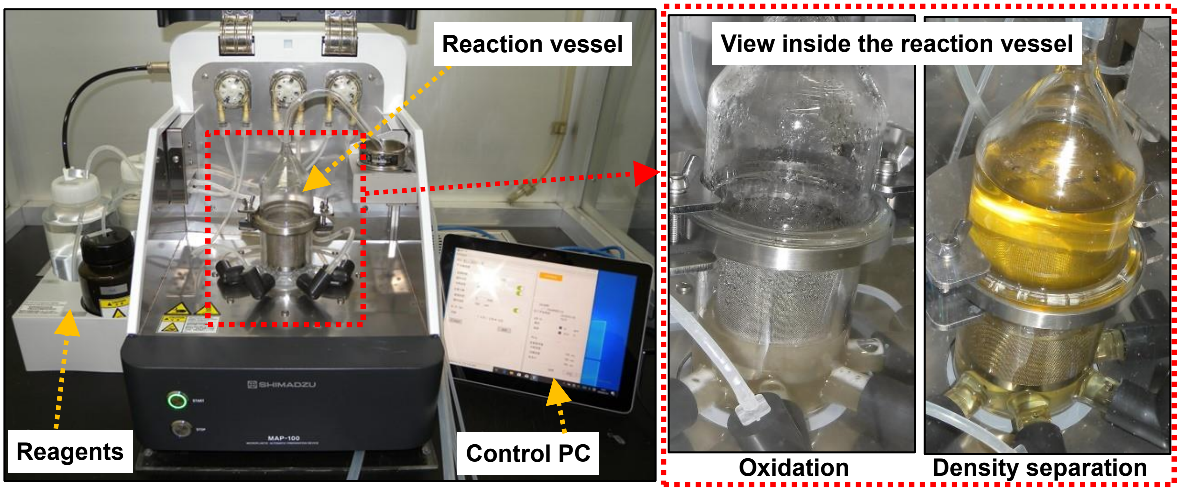


Figure: Device Subject to Verification (left) and View Inside the Reaction Vessel (right)

1.2 Equipment Specifications and Scope of Application (see page 12 of Main Text for details)

Size (mm)	300 (W) × 550 (D) × 400 (H) (not including protruding parts)
Weight	Approx. 25 kg
Power Supply	100 V AC ± 10 %, 50/60 Hz, 850 VA
Ambient Temperature	Room temperature 15 to 30 °C
Particles Extracted by Device	Size: 0.3 to 5.0 mm (major diameter: length of longest part) Density: 1.5 g/cm^3 or below
Particle Extraction Method	Extraction via overflow process (Solution is pumped out of the top of the reaction vessel and candidate MP particles floating in the top part of the solution due to density separation are carried out of the reaction vessel together with the solution)
Reagents Used	• Washing, etc.: pure water • Decomposition: Hydrogen peroxide solution (conc. 30 % (w/w) or less) • Separation: Sodium iodide solution (conc. 5.3 mol/L)

2. Overview of Verification

2.1 Purpose of Verification

To evaluate the performance of the device subject to verification by verifying reproducibility and accuracy when the device is used to recover MPs from samples. This evaluation was performed using a addition and recovery test that added commercially available reference plastic particles (size: 500 to 600 μm) to environmental samples,*1 used the device to automatically process the added samples, then calculated MPs recovery (%) based on the number of added reference plastic particles recovered that were recovered (see page 15 of Main Text for details).

*1: River water samples after filtration through a 0.1 mm mesh net, removal of contaminants 5 mm or larger, and drying at 60 °C.

2.2 Performance Test and Quantitative Result (Verification Test and Performance Parameter Subject to Verification)

Addition and recovery test was performed using river water samples.

Verification Test	Performance (Parameter) Subject to Verification
Addition and Recovery Test	At least 80 % recovery*2 of added reference plastic particles by automatic preparation device.

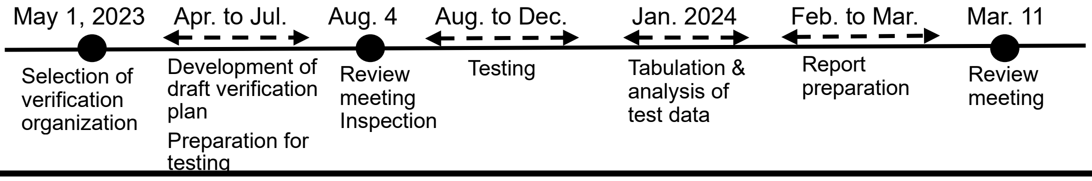
*2: Reference particle recovery is the proportion (%) of particles added to the environmental sample collected from the river recovered by the device and was calculated using the equation below.

$$\text{Reference particle recovery (\%)} = \frac{\text{Number of added reference plastic particles recoverd by the device}}{\text{Amount of reference plastic particles added added to environmental sample}} \times 100$$

2.3 Testing Site

Testing Site	Saitama-ken Environmental Analysis & Research Association (Main Building) Test Laboratory
Address	1450-11 Kamiko-chou, Omiya-ku, Saitama

2.4 Verification Period (Schedule)



3. Verification Results and Discussion

3.1 Monitored Items (see pages 20 to 23 of Main Text for details)

(1) Sample collection conditions

The indoor environment conditions in the laboratory during automatic sample preparation (August 29 to December 1, 2023) included temperatures between 22.1 and 23.8 °C and humidities between 20.0 and 68.0 %. These environmental conditions had no impact on the operation of the device.
The on-site (river) conditions during MP sample collection are shown in **Table 1**. Sample collection was performed on days when the rivers were not muddy due to rainfall or other factors. Samples were collected in 3 points and 3 samples were collected at each point (9 samples in total).

Table 1 MP Sample Collection Conditions

River Name	River A			River B			River C		
Sampling points	**Point (1)**			**Point (2)**			**Point (3)**		
Date Collected	September 19, 2023			September 28, 2023			September 28, 2023		
Weather	Clear			Cloudy			Clear		
Temperature (°C)	30.5			27.0			30.0		
Water Velocity (m/s)	0.26			0.20			0.45		
Water Depth (cm)	45			48			32		
Sample Number	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
Sample Name	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Filtering Time (min)	10	12	8	15	15	15	10	15	15
Filtered Volume (m^3)	8.4	9.3	6.8	7.3	10.5	8.5	13.0	18.0	15.0

(2) Water quality and other conditions

The turbidity, suspended solids, and total organic carbon in the rivers at the 3 sampling points ranged from 2 to 5 mg-kaolin equivalent/L, 3 to 10 mg/L, and 1.2 to 2.9 mg/L, respectively. The bottom of the river was visible in all sampling points. The weights of the environmental samples after being filtration and drying at 60 °C ranged from 3.67 to 10.2 g.

3.2 Verification Test and Reference Test (see pages 24 to 31 of Main Text)

The results of the verification test are shown in **Table 2**. During oxidation of environmental samples (2)-1 and (2)-3, a device feature that replaces the decomposition reagent in the reaction vessel was used to ensure complete decomposition of the organic matter in the samples. The recovery of reference plastic particles (1.00 g/cm3 and 1.35 g/cm3) was 92 to 100 % (coefficient of variation: 4.1 %) and 96 to 100 % (coefficient of variation: 2.0 %), respectively, which satisfied the performance that was subject to verification (at least 80 % recovery)

Table 2 Verification Test (Added Recovery Test with Device Subject to Verification) Results

Sampling Points		Point (1)			Point (2)			Point (3)		
Sample Name		(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Specific gravity 1.00	Number Recovered	23	25	23	25	23	23	24	25	23
	Reference Particle Recovery (%)	**92**	**100**	**92**	**100**	**92**	**92**	**96**	**100**	**92**
Specific Gravity 1.35	Number Recovered	25	25	25	25	24	24	25	24	25
	Reference Particle Recovery (%)	**100**	**100**	**100**	**100**	**96**	**96**	**100**	**96**	**100**

The results of the reference test are shown in **Table 3**. The recovery of environmental MPs (1 mm or larger) from river water samples collected 3 times in 3 points was 82 to 100 % (coefficient of variance: 5.6 %). The most common type of MPs recovered were polypropylene and polyethylene, which accounted for at least 70 % of the MPs in each sample.

Table 3 Reference Test (Environmental MP Recovery Test with Device Subject to Verification) Results

Sampling Points	Point (1)			Point (2)			Point (3)		
Sample Name	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Number Recovered	11	25	24	30	49	44	47	103	78
Number Not Recovered	0	2	3	4	6	7	10	16	12
Environmental MPs Recovery (%)*1	**100**	**93**	**89**	**88**	**89**	**86**	**82**	**87**	**87**

*1: Environmental MPs recovery is the proportion (%) of the total number of environmental MPs (1 mm or larger) originating from the river water sample that were recovered by the device (where the total number of environmental MPs in a sample was the sum of the number of MPs recovered by the device and the number of MPs not recovered by the device) and was calculated using the equation below.

$$\text{Environmental MPs recovery (\%)} = \frac{\text{Number of environmental MPs recovered by the device}}{\text{Number of environmental MPs recovered by the device} + \text{Number of environmental MPs not recovered by the device}} \times 100$$

3.3 Findings (see page 35 of Main Text for details)

Item	Findings
Technology overall	The characteristic feature of the device subject to verification is that it performs all sample preparation processes (oxidation and density separation) automatically in a single reaction vessel. Because the device is not impacted by the skill level of the operator, the device reduces variability in MPs recovery. In a spiked recovery test using plastic reference particles added to river water samples, the recovery of reference particles by the device was at least 80 %, which satisfied the performance subject to verification. The recovery of environmental MPs (1 mm or larger) by the device was also at least 80 %. This device can be expected to improve the accuracy of empirical data in studies and research into MPs, can be expected to have a positive impact on the development of future environmental conservation measures in MPs Pollution.
Miscellaneous	When hydrogen peroxide solution has lost its activity during the oxidation process and a substantial amount of organic matter remains in the device subject to verification, the remaining organic matter can be decomposed by using the “decomposition reagent replacement feature” of the device (a feature that replaces the decomposition reagent in the reaction vessel). Using this feature, the recovery of environmental MPs was at least 80 % even from environmental samples containing a large amount (approx. 10 g) of algae and other organic matter, which demonstrated the efficacy of this feature. Testing also demonstrated that recovery was at least 80 % when around 120 MPs were present in the environmental sample.

4. Reference Information

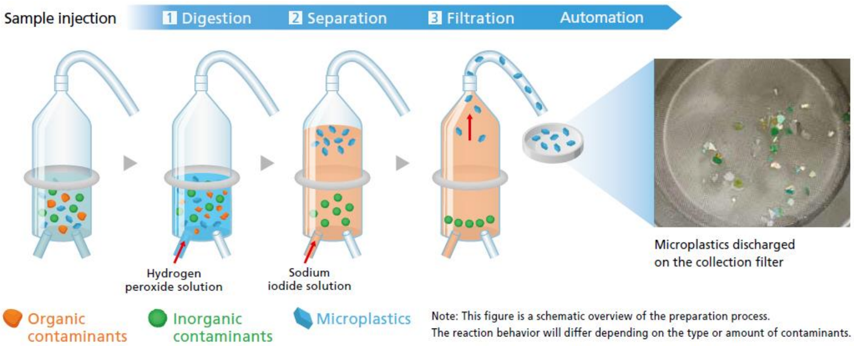
Note: All the information displayed on this page was submitted by the verification applicant of their own volition to publicize the technology. The Ministry of the Environment and the verifying organization assume no responsibility for its contents.

4.1 Product Data

Item		**Section Completed by the Verification Applicant or Developer**			
Product Name/Model Number		MAP-100 Microplastic Automatic Preparation Device			
Name of Manufacturing (Distributor) Company		Shimadzu Corporation			
Contact Information	**Phone/Fax**	(81)75-823-1635			
	Website Address	https://www.shimadzu.co.jp/			
	E-mail	ikezawa@shimadzu.co.jp			
Installation Requirements		Indoors at room temperatures between 15 °C and 30 °C with minimal variation in temperature			
Required Maintenance		Periodic inspection and replacement of maintenance parts			
Weather Resistance and Product Lifespan, etc.		Design standard use period: 10 years			
Cost		**Item**	**Unit Price**	**Quantity**	**Total**
Cost Estimate (Conditions: case-by-case quote)		**Initial costs**			
		Main unit	JPY 2,000,000/set	Complete set	JPY 2,000,000
		Installation cost	Quoted case-by-case		
		Other	Quoted case-by-case		
		Maintenance costs			
		Frequency of maintenance and parts replacement varies based on the circumstances of use. Contact the manufacturer for further details.			

4.2 Other Information Provided by the Manufacturer

Automatic Preparation Processes
1. Decomposition of organic matter
2. Separation from inorganic matter
3. Extraction and recovery



Note: This figure is a schematic overview of the preparation process.
The reaction behavior will differ depending on the type or amount of contaminants.

MAP-100 Microplastic Automatic Preparation Device

Labor savings	• Automating the complicated sample preparation process substantially reduces the number of man-hours required.
Reproducibility	• Automation ensures that sample preparation is highly reproducible, which improves the accuracy of comparisons between data.
Safety	• Simplifies the handling of reagents by operators.

Main Text

1. Program Overview

1.1 Objectives

The purpose of the Environmental Technology Verification Program (hereinafter referred to as the “Verification Program”) is to promote the widespread adoption of environmental technologies, have a positive impact on environmental conservation, and contribute to the growth of environmental industries, including the development of small and medium-sized enterprises. The Verification Program seeks to achieve this by establishing a method/system of verification for advanced environmental technologies already in circulation that assigns third parties to objectively verify the impact of these technologies on environmental conservation, their secondary environmental effects, and other aspects of their performance considered important for the environment (hereinafter referred to as their “impact on environmental conservation.”). Through this program of verification, the Verification Program seeks to provide users of environmental technologies with a simple means of comparing and reviewing the impact of these technologies on environmental conservation and make appropriate choices when purchasing or procuring these technologies.

The Verification Program operates under an international unified framework and in conformance with the international standard ISO 14034:2016 “Environmental management — Environmental technology verification (ETV).”

1.2 Definition of Verification

“Verification” as used by the Verification Program refers to the action of a third party who is neither a developer nor user of the environmental technology that shows the impact of the environmental technology on environmental conservation based on objective data obtained through testing. Environmental technology is defined as advanced technology or advanced measuring technology related to the environment that has an ameliorative impact or conserving impact on the environment. “Verification” differs from “certification,” which establishes a set of determining criteria and assesses conformance with those criteria.

1.3 Outline of the Verification Report

This report was prepared in accordance with “Appendix 4: Matters to be included in the verification report and report synopsis” and “Appendix 5: Verification report preparation guidelines Ver. 3.2” of Environmental Technology Verification Program Implementation Guidelines [November 16, 2023, Office of Environmental Research and Technology, General Policy Division, Minister’s Secretariat, Ministry of the Environment] (hereinafter referred to as the “Verification Guidelines”).

In this verification, the below-described performance of the “Microplastic Automatic Preparation Device,” which was selected as the technology subject to verification according to the Verification Guidelines, was verified objectively.

Furthermore, the impact of the technology subject to verification on environmental conservation was examined by technology verification review meetings comprised of experts based on the results of the verification. This report compiles the results of this verification.

- Device performance in conditions of environmental use based on the scope of the technical specifications set forth by the verification application
- The energy and materials required and amount of waste generated in using the device
- The environmental conditions that allow proper operation of the device
- The labor required to use and upkeep the device

2. Verification System and Division of Responsibilities among Verification Participants

The organizations participating in the verification and the implementation structure of the verification are shown in **Fig. 2-1**. The verification participants and the division of responsibilities are shown in **Table 2-1**.

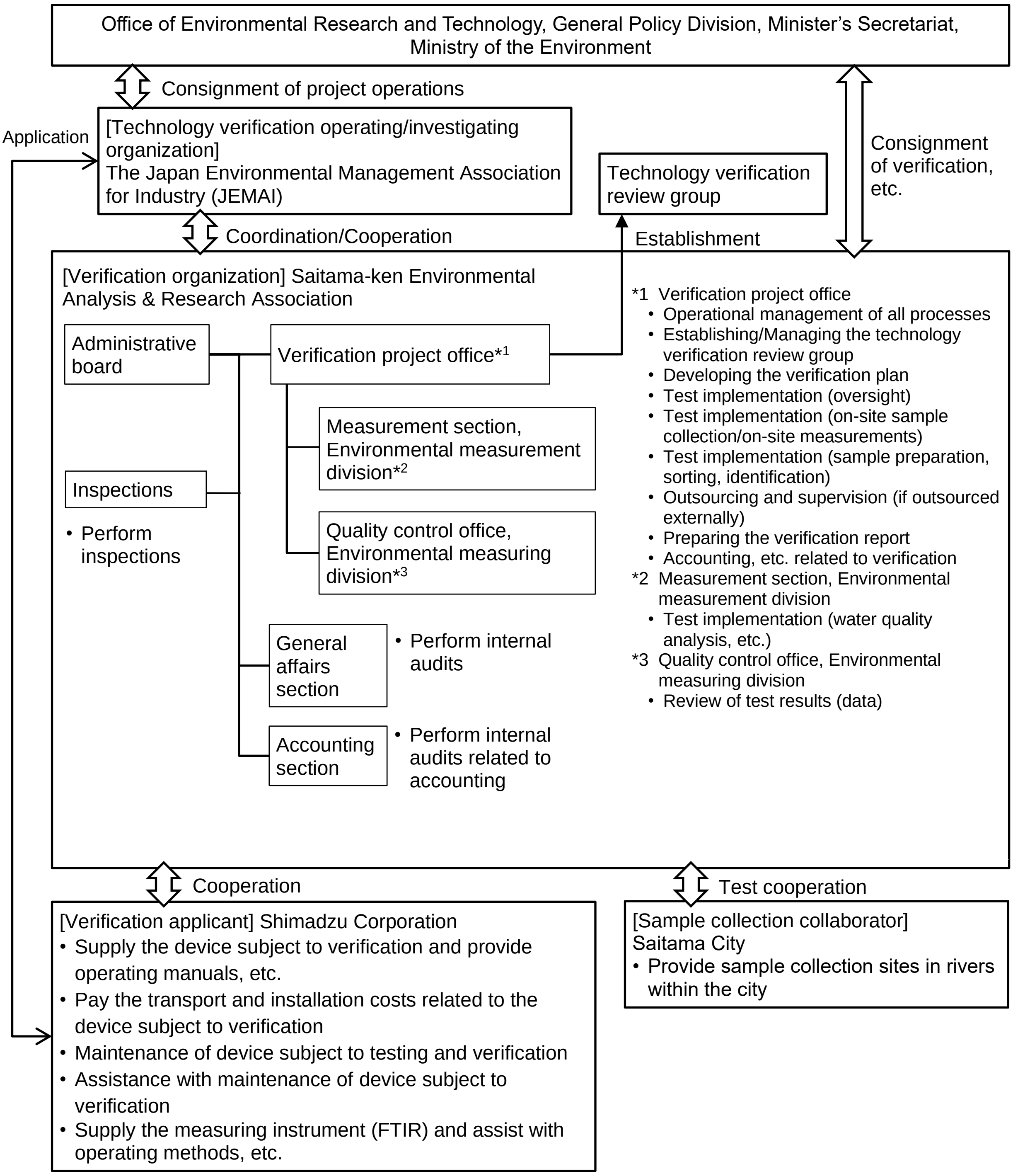


Fig. 2-1 Organizations Participating in Verification and the Implementation Structure

Table 2-1 Verification Participants and Division of Responsibilities

Group	Party Participating in Verification		Responsibilities	Individual Participants
Verification Organization	Saitama-ken Environmental Analysis & Research Association	Oversight and project management	Operational management of all Verification Program processes	Verification project office Tomohiko Yamagishi Kazuyuki Nagahama Toshihiko Otsuka Naohiro Kishida
			Establishing/Managing the technology verification review group	
			Developing the verification plan	
			Test implementation (oversight)	
			Preparing the verification report	
			Outsourcing and supervision (if outsourced externally)	
		Sample collection	Test implementation (on-site sample collection/on-site measurements)	
		Measurement	Test implementation (sample preparation, sorting, identification)	
		Analysis	Test implementation (water quality analysis, etc.)	Manager, Environmental measurement division Hiroshi Takahashi
			Management of test data and information	
		Review of data	Review of test results (data)	Head of quality control office, Environmental measuring division Katsunori Santo
		Internal audits	Perform internal audits	ISO specialist, General affairs section Satoshi Ogura
		Accounting	Accounting, etc. related to verification	Verification project office Naohiro Kishida
		Accounting audits	Perform internal audits related to accounting	Chief financial officer, Accounting division Susumu Asakawa
		Inspection	Perform inspections related to verification	Director in charge of inspections Yuji Noguchi
Verification Applicant	Shimadzu Corporation		Provide existing data and information on testing organization	Environment Business Unit, Analytical & Measuring Instruments Division Yoshio Ikezawa
			Pay testing costs	
			Supply device subject to verification and provide operating manuals, etc.	
			Pay transport, installation, and removal costs for device subject to verification	
			Pay costs required for maintenance of device subject to verification	
			Assist with maintenance of device subject to verification as required	
			Supply a Fourier transform infrared spectrophotometer (FTIR) and assist with operation, etc.	
Sample Collection Collaborator	Saitama City		Provide sample collection sites in rivers within the city	Environment action section, Ecology department, Environment bureau, Saitama City

3. Overview and Specifications of Technology Subject to Verification

3.1 Principles of Technology Subject to Verification

(1) Principles and effectiveness

The technology subject to verification is an automatic sample preparation device for microplastics (MPs) surveys that minimizes human error and facilitates sample preparation with good reproducibility by automating (1) oxidation and (2) density separation (**Fig. 3-2**), which are the sample preparation processes (**Fig. 3-1**) described in “Guidelines for River and Lake Microplastic Monitoring Methods” (March 2023, Water Environment Management Division, Environmental Management Bureau, Ministry of the Environment) (hereinafter referred to as the “MPs Monitoring Guidelines”).

The sample preparation processes described in the MPs Monitoring Guidelines often result in the skill level of the operator affecting the empirical data. This technology can be expected to improve the accuracy of research into the sources and distribution of MPs and the empirical data used to make environmental conservation measures in MPs Pollution, and thereby can be expected to have a positive impact on the development of future measures concerning MPs.

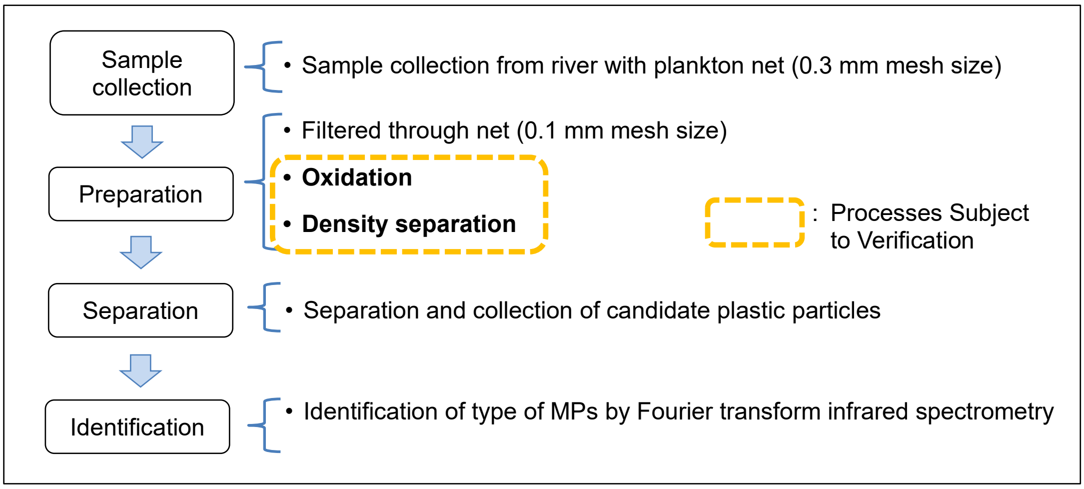


Fig. 3-1 Survey Procedure in “Guidelines for River Microplastic Monitoring Methods”

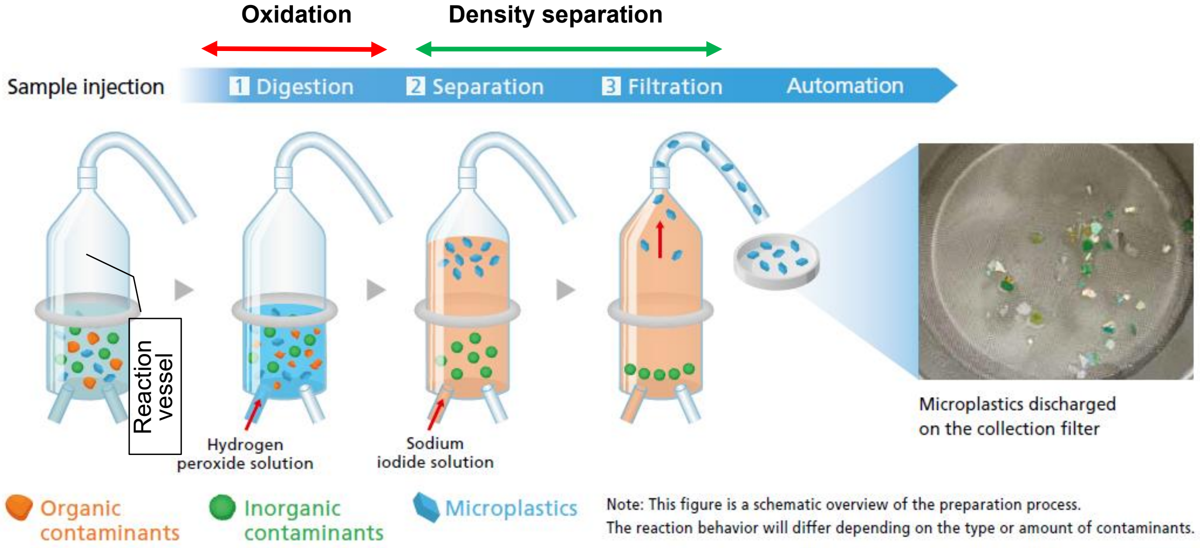


Fig. 3-2 Automated Sample Preparation Performed in Reaction Vessel

(2) Sample preparation procedure using device subject to verification

Table 3-1 shows the sample preparation procedure described in the MPs Monitoring Guidelines and when using the device subject to verification. The device subject to verification performs all sample preparation processes inside a single reaction vessel (**Fig. 3-2**). The photographs shown are excerpted from reference (1).

Table 3-1 Sample Preparation Procedure in MPs Monitoring Guidelines and for Device Subject to Verification

Process	Name of Operation		MPs Monitoring Guidelines		Device Subject to Verification
Sample preparation	Oxidation		[Outline] Removal of organic matter from sample by oxidation		
			 Samples standing in a water bath	[Operations] • Remove moisture from filtered sample using a drier (60 °C) • Add 30 % hydrogen peroxide solution and leave in water bath (55 °C) for 3 days • Filter with a net and recover the filtered residue	[Automated operations] ➡*1 Recover solid matter from filtered sample with strainer and place in the reaction vessel > Add 30 % hydrogen peroxide solution to the reaction vessel and process for 72 hours while heating and stirring > Recover solid materials with a strainer
	Density separation	Separation	[Outline] Separation of plastics from the sample		
			 Sample left to stand	[Operations] • Add 5.3 mol/L sodium iodide solution to the filtered residue and leave to stand for around 3 hours	[Automated operations] > Add 5.3 mol/L sodium iodide solution to the reaction vessel, stir, then leave to stand for 3 hours
		Recovery	[Outline] Recovery of separated plastics		
			 Recovering upper part of aqueous solution	[Operations] • Dispose of the lower part and recover the upper part of the aqueous solution • Filter with a net and recover the filtered residue	[Automated operations] > Create an overflow from the top of the reaction vessel to extract the candidate MP particles in the upper part of the reaction vessel solution > Recover the solid material in the overflow solution with a recovery filter
Remarks: Reagents must be prepared separately					

*1: The only manual task

[References]
(1) “Guidelines for River and Lake Microplastics Monitoring Methods,
March 2023, Water Environment Management Division,
Environmental Management Bureau, Ministry of the Environment”

3.2 Configuration and Characteristic Feature of Technology Subject to Verification

The configuration of the device subject to verification is shown in **Fig. 3-3** and photographs of the device are shown in **Fig. 3-4**.

The device subject to verification is comprised of a reaction vessel, hot plate stirrer, regent bottles (2 pieces), wash water bottle (1 piece), hydrogen peroxide solution/pure water supply pump, sodium iodide solution/pure water supply pump, reagent/wash water switching solenoid valve (2 pieces), waste solution/effluent pump, waste solution switching solenoid valve, waste solution collection bottles (2 pieces), recovery filter, tubing (silicone tubes with fluorine-based plastic connectors), a control PC, and external casing. The only plastics used by the device were in the reagent bottles and the tube and solenoid valve fittings (polypropylene).

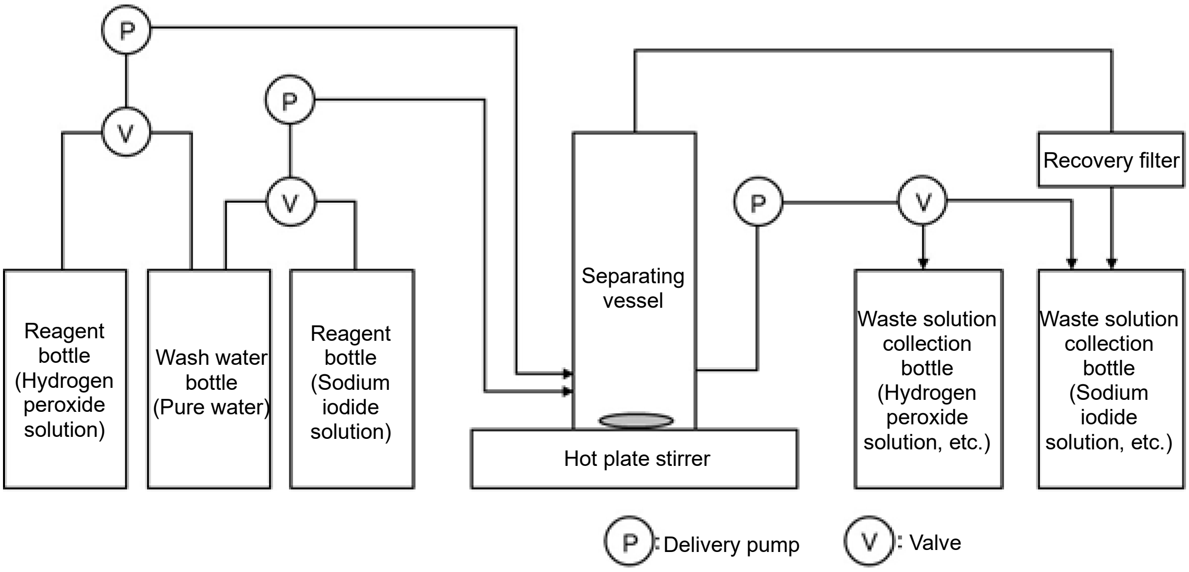


Fig. 3-3 Configuration of Device Subject to Verification

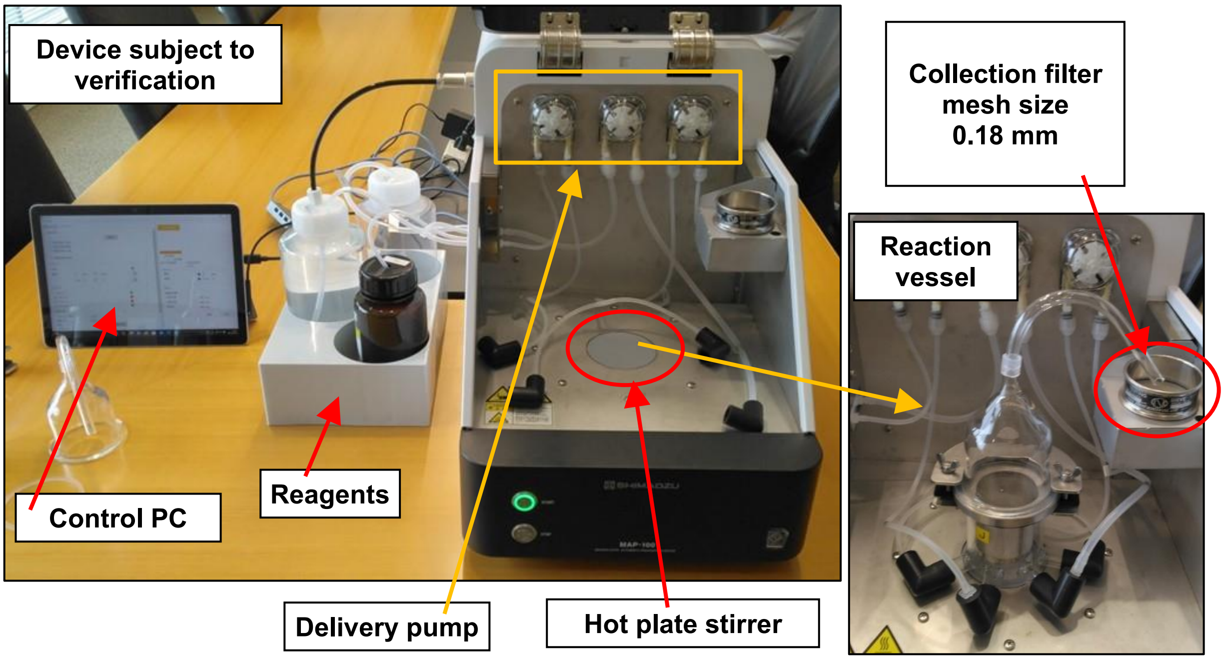


Fig. 3-4 Photographs of Device Subject to Verification

The characteristic feature of this device is that it performs all sample preparation processes automatically in a single reaction vessel (**Table 3-1**). This reduces the labor required to carry out sample preparation, which reduces the number of MPs not recovered during sample preparation and ensures that sample preparation is highly reproducible. By contrast, the (1) oxidation and (2) density separation sample preparation processes described in the MPs Monitoring Guidelines (**Table 3-1**) are performed manually, which includes the handling of glassware and filtering samples through nets. The issue with this approach is that the skill level of the worker may create variability in MPs recovery and affect empirical results.

A photograph of the reaction vessel is shown in **Fig. 3-5**. The reaction vessel is made of glass and has a capacity of approx. 400 mL. The vessel has separate upper and lower sections that are held together with a clamp. During sample preparation, the environmental sample (the river water sample after filtering through a 0.1 mm mesh net and removing contaminants 5 mm and larger) is poured over a strainer installed in the bottom section of the vessel to separate the solid matter from the solution.

During oxidation, hydrogen peroxide solution (100 mL) is introduced from an inlet port in the bottom section of the vessel, and organic matter in the sample is decomposed while heating and stirring the sample on the hot plate stirrer (72 hours). If the hydrogen peroxide solution loses its activity during oxidation and a substantial amount of organic matter remains in the sample, the “decomposition reagent replacement feature” can be used to change the treatment conditions manually during the oxidation process. This feature enhances the decomposition of organic matter by draining the inactive hydrogen peroxide solution from the reaction vessel and adding new hydrogen peroxide solution (100 mL) to the reaction vessel. After oxidation, the decomposition solution is discharged into the hydrogen peroxide waste collection bottle from a discharge port in the bottom section of the vessel.

During density separation, sodium iodide solution is added from an inlet port in the bottom section of the vessel, stirred, left to stand for 3 hours, then candidate MP particles isolated in the upper part of the sodium iodide solution are extracted by creating an overflow from the nozzle in the upper section of the vessel and recovering the MP particles carried out of the vessel with a recovery filter. After density separation, the solution remaining in the vessel is discharged into the sodium iodide waste collection bottle from a discharge port in the bottom section of the vessel.

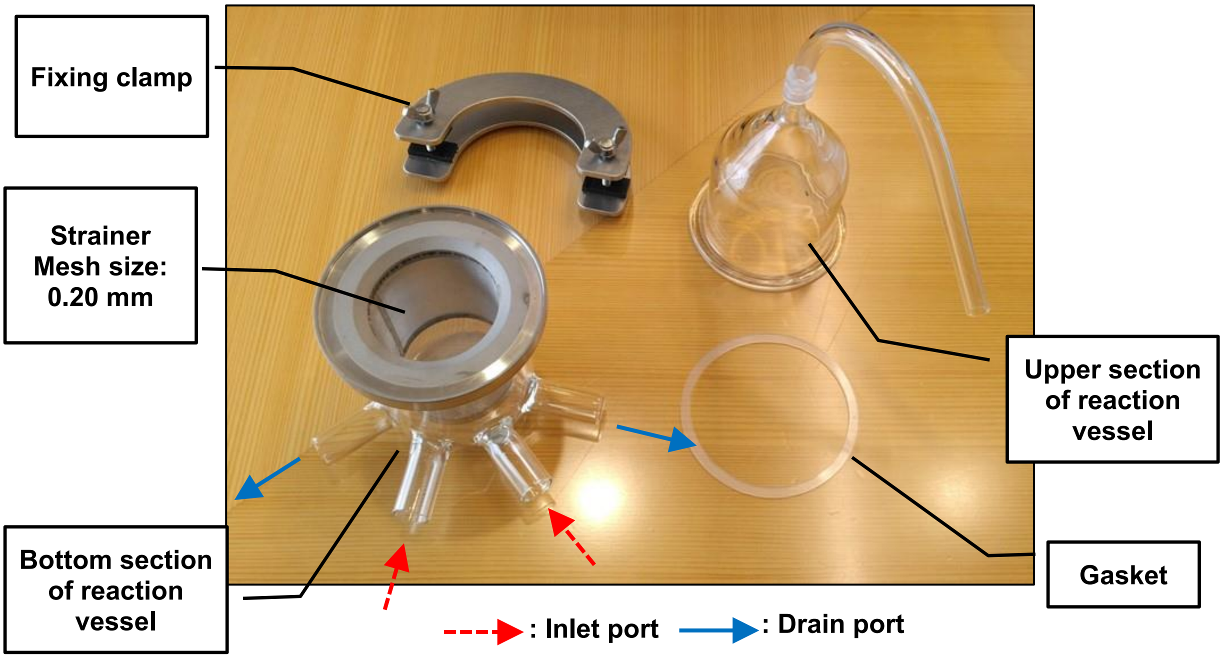


Fig. 3-5 Photograph of Reaction Vessel Components

The reagents used in sample preparation are added to the reaction vessel automatically and the waste liquids created during sample preparation are also discharged and collected automatically in waste solution bottles by the device, thereby avoiding worker exposure to harmful materials during sample preparation and reducing risk from the perspective of occupational health and safety.

3.3 Specifications of the Technology Subject to Verification

(1) Specifications

The specifications of the device subject to verification are shown in **Table 3-2**.

Table 3-2 Specifications of Device Subject to Verification

Size (mm)	300 (W) × 550 (D) × 400 (H) (not including protruding parts)
Weight	Approx. 25 kg
Power Supply	100 V AC ± 10 %, 50/60 Hz, 850 VA
Configurable Temperature Range[*1]	30 to 100 °C (above room temperature only)
Ambient Temperature	Room temperature 15 to 30 °C
Compatible Samples	Samples collected from the surface water layer of rivers, oceans, lakes, etc.
Size of Particles Extracted	Extracts MPs 0.3 to 5.0 mm (major diameter) in size.
Density of Particles Extracted	MPs with a density of 1.5 g/cm^3 or less.
Particle Extraction Method	Extraction via overflow process (Solution is pumped out of the top of the reaction vessel and candidate MP particles floating in the top part of the solution due to density separation are carried out of the reaction vessel together with the solution)
Reagents Used	• Washing, etc.: Pure water • Decomposition: Hydrogen peroxide solution (conc. 30 % (w/w) or less) • Separation: Sodium iodide solution (conc. 5.3 mol/L)

*1: The temperature range of the hot plate stirrer configured by the software . The temperature in the reaction vessel may differ from the temperate set using the software (the device also features the ability to adjust temperature while only operating the hot plate stirrer).

(2) Consumable parts, consumable materials, and power consumption

The consumable parts, etc. are as shown **Table 3-3**.

Table 3-3 Electricity Consumed and Parts Consumed during Single Pretreatment (82 hours)

Consumable		Units	Amount Consumed
Power consumption		kWh	69.7
Chemical and reagent consumption	Pure water	mL	1,000
	30 % hydrogen peroxide solution	mL	100 to 200[*2]
	5.3 mol/L sodium iodide solution	mL	700[*3]

*2: Normally uses 100 mL, but an additional 100 mL is used when the sample contains a large amount of organic matter (200 mL total)

*3: Includes the solution with entrained MPs extracted from top nozzle during overflow process (150 mL used per overflow: overflow can be performed 3 times).

(3) Collected materials, waste materials, and handling of same

The recovered materials and waste materials produced during the sample preparation process and their handling are shown in **Table 3-4**.

Table 3-4 Collected Materials, Waste Materials, and Handling Instructions

Material	Handling Instructions
Waste hydrogen peroxide solution	Dispose of appropriately in accordance with laws and regulations.
Waste sodium iodide solution	

3.4 Operational Tasks and User Skills Required to Operate and Maintain the Device Subject to Verification

The recommendations for daily and periodic management of the device are as shown in **Table 3-5**. Also, users of the device must have received education and training sufficient to understand the handling and disposal, etc. of chemical substances based on regulations on chemical substances in the Industrial Safety and Health Act.

Table 3-5 Maintenance

Type of Maintenance	Person Responsible	Work Tasks	Frequency
Daily Inspection	User	• Confirm there are no leaks from piping, etc. • Confirm no reagents are adhered to the device or around it	Daily
Periodic Inspection	User or developer of the technology	• Replacement of device tubing • Replacement of air filter on air intake	Once per year

3.5 Important Notes on Device Operation and Precautions

Safety Precautions

- Wear safety glasses and gloves for personal protection from the corrosive reagents used.
- Exercise caution as the internal parts of the device become hot during processing (also indicated on label).
- Only use the specified reagents.
- Use separate waste collection bottles for each type of waste solution (do not mix waste solutions).
- More safety precautions are provided in the operating manual and all safety precautions must be observed.

4. Verification (Test) Method

The device subject to verification reduces the number of unrecovered MPs and ensures consistent empirical measurements by automating sample preparation and performing sample preparation within a single reaction vessel. To confirm the efficacy of the sample preparation performed by this device, the performance of the device was evaluated by performing a (1) addition and recovery test of commercially available reference plastic particles in river water samples and (2) testing the recovery of environmental MPs present in the river water samples used in (2).

4.1 Complete Verification Schedule

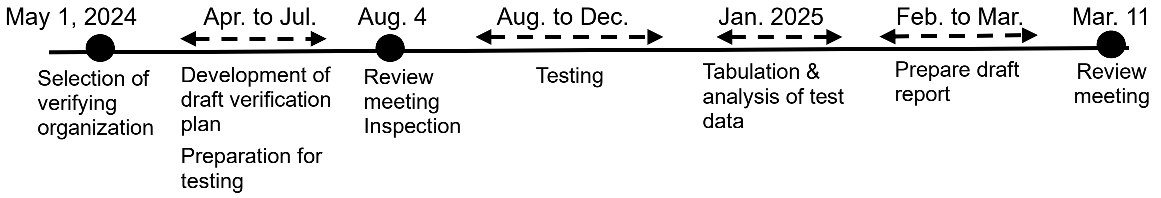


Fig. 4-1 Complete Verification Schedule

4.2 Testing Site Information

Information on the testing site is shown in **Table 4-1**.

Table 4-1 Testing Site Information

Name	Saitama-ken Environmental Analysis & Research Association (Main Building) Test Laboratory
Address	1450-11 Kamiko-chou, Omiya-ku, Saitama
Testing	Sample preparation of river water samples and testing up to the point of MP characterization; water quality analysis
Main Facilities	Chemistry laboratory, fume hoods, pure water system, effluent treatment system, electronic balance, turbidity meter, drier, automatic sample preparation device, stereoscopic microscope (with digital camera), Fourier transform infrared spectrometer (FTIR)

4.3 Monitored Items

The items shown in **Table 4-2** were monitored. Monitoring was performed on conditions and parameters that were anticipated to have an impact on the results.

Table 4-2 Monitored Items

Monitored Item	Details
Environmental conditions in the test laboratory	Temperature, humidity
Conditions during sample collection	Weather data, water velocity, water depth, filtering time, filtered volume
Water quality of river water sample	Appearance, odor, turbidity, suspended solids, total organic carbon
Condition of environmental sample*	Appearance, weight (after 60 °C drying process)

* River water sample collected in plankton net after filtration through a 0.1 mm mesh net, removal of contaminants 5 mm or larger, and drying (in a drier at no higher than 60 °C to prevent deterioration of plastics) to remove water without drying completely (drying to a wet sample that resists gravity when tilted on a flat surface).

4.4 Verification Test, Verified Performance, and Reference Test

The verification test, verified performance, and reference test used are as shown in **Table 4-3** and **Table 4-4**. The addition and recovery test with river water samples that was employed as the verification test was performed based on “Supplementation: The Standard Specification of Automatic Sample Preparations in Guidelines for River Microplastics Monitoring Methods” of “Guidelines for River and Lake Microplastics Monitoring Methods, March 2023, Water Environment Management Division, Environmental Management Bureau, Ministry of the Environment” (see Supplementation page 40). A reference test was also performed alongside the verification test. The reference test was a recovery test of MPs present in river water samples. Note that a procedure blank test was performed before these tests to investigate MPs contamination originating from the automatic sample preparation device and confirm no contamination was introduced by the device.

Table 4-3 Verification Test and Verified Performance

Verification Test	Performance Subject to Verification
Addition and recovery test	At least 80 % recovery*[1] of added reference plastic particles by automatic preparation device.

*1: Reference particle recovery

$$\frac{\text{Number of added reference plastic particles recovered by the device}}{\text{Amount of reference plastic particles added to environmental sample}} \times 100 = \text{Reference particle recovery (\%)}$$

Table 4-4 Reference Test

Reference Test	Overview
Environmental MPs recovery test	A performance test based on the recovery (%)*[2] of natural MPs (1 mm or larger) from the river water samples by the automatic sample preparation device

*2: Environmental MPs recovery

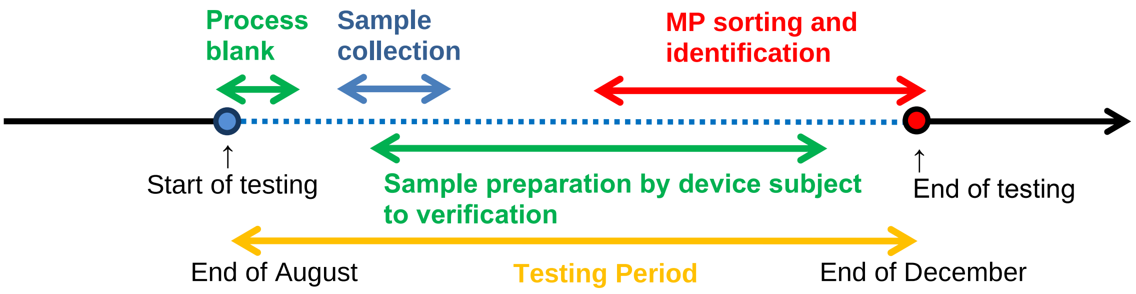
$$\frac{\text{Number of environmental MPs recovered by the device}}{\text{Number of environmental MPs recovered by the device + Number of environmental MPs not recovered by the device}^{*3}} \times 100 = \text{Environmental MPs recovery (\%)}$$

*3: Number of environmental MPs not recovered by the device:
Number of environmental MPs remaining inside reaction vessel after sample preparation
(Number of environmental MPs not recovered by the recovery filter during the overflow extraction process)

4.5 Verification Method

(1) Test schedule

Samples were collected and testing was performed according to the schedule shown in **Fig. 4-2**.



* The weather and other on-site information were recorded during sample collection.

Fig. 4-2 Test Schedule

(2) Details of testing

Sample collection and testing are described in **Table 4-5**. All testing shown in the table was performed in accordance with the MPs Monitoring Guidelines except for the process blank test and sample preparation.

Table 4-5 Stages of Testing

Stage of Testing	Details	Testing Period
Process blank test	MPs contamination originating from the device subject to verification was investigated.	August 29
Sample collection	Samples were collected for recovery testing and for water quality analysis.	September 19 to 28
Sample preparation	The device subject to verification was used to perform automatic sample preparation with environmental samples collected from river water.	September 25 to December 1
MP sorting and identification	Candidate MP particles were sorted using a stereoscopic microscope and identified by FTIR.	Late November to Late December

(3) Point of sampling points

Samples were collected at the 3 points in Saitama City (Saitama Prefecture) shown in **Table 4-6** that are also subject to constant water quality monitoring.

Table 4-6 Point of sampling points

River Name	Sampling Points	Land use in upper river area
River A	Point (1)	Agricultural land, multipurpose fields (green space), residential area
River B	Point (2)	Residential area, industrial plant, logistic warehouse, etc.
River C	Point (3)	Park (green space), residential area

(4) Sampling methods of river water samples

Samples were collected by the methods shown in **Table 4-7**.

Table 4-7 Sampling methods of river water samples

Sample	Sampling Method
For recovery testing	• The sample collector entered the river, submerged a plankton net (mouth diameter: 30 cm, mesh size: 0.3 mm) in the river, and held the plankton net in the surface river water (0 to 30 cm under the water surface) until 6.8 to 18 m^3 of water*1 filtered through the net. After holding the net for the required length of time, the sample collector raised the net out of the water, washed it with water,*2 gathered the sample into the cod end assembly at the end of the net, and transferred the sample into a glass container. • Samples were collected at 3 points and 3 samples were collected at each point. A total of 9 samples were used in testing.
For water quality analysis	• The sample collector entered the river, collected water from the surface river water (from 0 to 30 cm under the water surface), and transferred it to a plastic container. • Samples were collected at the same 3 points where samples were collected for recovery testing, and 3 samples were collected at each point. The 3 samples from each point were combined into a single sample, giving 3 samples in total for analysis.

*1: River water velocity was measured using a flowmeter attached to the plankton net and the water volume was calculated based on the flow velocity and filtering time.

*2: River water filtered through a 0.1 mm mesh net

The plankton net and flowmeter used to collect the MPs samples are shown in **Fig. 4-3**.

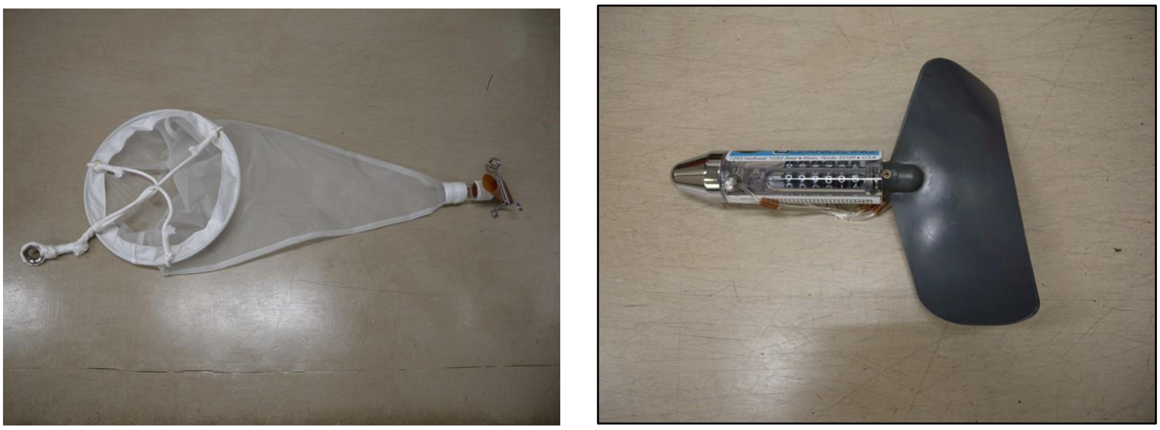


Fig. 4-3 Plankton Net (left) and Flowmeter with Low-Velocity Rotor (right)

(5) Recovery test methods

Recovery tests with river water samples were used to evaluate the performance of the device subject to verification in terms of sample preparation reproducibility. The recovery test methods are shown in **Table 4-8**.

Table 4-8 Recovery Test Methods

<table>
<tr><th>Test</th><th>Method</th></tr>
<tr><td>Addition and Recovery Test</td><td>
<p>Commercially available reference plastic particles were added to environmental samples (9 samples in total),*1 the samples were processed using the automatic sample preparation device, a stereoscopic microscope was used to count the number of plastic reference particles recovered by the device, and this number was used to calculate recovery.</p>
<p>[Plastic reference particles added to samples]</p>
<table>
<tr><th>Model No.</th><th>Material</th><th>Specific Gravity g/cm^3</th><th>Size µm</th><th>Shape</th><th>Color</th></tr>
<tr><td>PNKPMS-1.00</td><td>Polyethylene</td><td>1.00</td><td>500-600</td><td>Spherical</td><td>Pink</td></tr>
<tr><td>WPMS-1.35</td><td>Polyethylene (containing titanium dioxide)</td><td>1.35</td><td>500-600</td><td>Spherical</td><td>White</td></tr>
</table>
<p>[Settings configured for automatic preparation device]</p>
<table>
<tr><th>Process</th><th>Parameter</th><th>Configured Setting</th></tr>
<tr><td rowspan="5">Oxidation</td><td>Reagent for oxidation</td><td>Aqueous hydrogen peroxide solution: conc. 30 %</td></tr>
<tr><td>Amount used</td><td>100 mL*2</td></tr>
<tr><td>Oxidation time</td><td>3 days (72 hours*4)</td></tr>
<tr><td>Heating temperature</td><td>Solution temperature: approx. 55 °C</td></tr>
<tr><td>Stirring speed</td><td>200 rpm</td></tr>
<tr><td rowspan="4">Density separation</td><td>Reagent for density separation</td><td>Aqueous sodium iodide solution: conc. 5.3 mol/L</td></tr>
<tr><td>Amount used</td><td>150 mL
(per overflow during extraction)</td></tr>
<tr><td>Stirring speed</td><td>500 rpm</td></tr>
<tr><td>Density separation standing time</td><td>3 hours</td></tr>
<tr><td>Recovery</td><td>No. of overflows</td><td>3</td></tr>
</table>
<p>*1: Amount of reference particles added: 25 particles of 1.00 g/cm^3 specific gravity and 25 particles of 1.35 g/cm^3 specific gravity (50 particles total)
*2: 200 mL total was used during the oxidization of environmental samples (2)-1 and (2)-3 due to insufficient decomposition of organic matter and use of the decomposition reagent replacement feature.*3
*3: A feature that discharges hydrogen peroxide solution (100 mL) from the reaction vessel and adds new hydrogen peroxide solution (100 mL) to the reaction vessel.
*4: The oxidation time for sample (2)-1 was 44 hours (see Reference Materials page 46)</p>
</td></tr>
<tr><td>Environmental MP recovery test</td><td>
<p>The environmental MPs (MPs originally present in the environmental sample) recovered and remaining in the reaction vessel during addition and recovery Test*5 were identified and counted using a stereoscopic microscope and the results used to calculate recovery.</p>
<p>*5: Plastic materials were also identified by FTIR.</p>
</td></tr>
</table>

(6) Analytical methods

The monitored items were analyzed by the methods shown below in **Table 4-9**.

Table 4-9 Analytical Methods

Parameter	Analytical Method
Turbidity	JIS K 0101 9.4 Integrating-sphere turbidity
Suspended solids (SS)	Environment Agency Notification No. 59 of 1971, Table 9: Superposition Method
Total organic carbon (TOC)	Ministry of Health, Labour and Welfare Announcement No. 261 of 2003 Total Organic Carbon Analyzer Measurement Method

(7) Operation and maintenance

During testing, the verification organization operated and maintained the device subject to verification according to the operating manuals.

4.6 Environmental Impact, Operation, and Maintenance

Matters related to environmental impact, operation, and maintenance of the device subject to verification are shown in **Table 4-10**.

Table 4-10 Environmental Impact, Operation, and Maintenance

Category	Parameter	Details, Measurement Method, etc.
Environmental impact	Electricity usage	The amount of electricity consumed by the device subject to verification during sample preparation was calculated.
	Noise*	The noise produced during automatic sample preparation was measured 3 times (during operation of the solution discharge pump, during operation of the sodium iodide solution supply pump, and during operation of the hydrogen peroxide solution supply pump).
Operation and maintenance	Quality of sample water that can be processed by the device subject to verification	The relationship between recovery and turbidity, SS, and TOC was assessed.
	Skills required to operate and maintain the device subject to verification	The working time associated with each procedure (number of people and days) and the difficulty and expertise required by maintenance were recorded.
	Reliability of device subject to verification	The causes of problems that occurred were investigated.
	Resumption of operation after trouble	The ease of resuming device operation after a problem and outstanding issues were assessed.
	Assessment of operation and maintenance manuals	The operation and maintenance manuals were assessed for readability, understandability, and outstanding issues.

* The sound produced by the pumps used by the device subject to verification when in operation.

5. Test Results and Discussion

5.1 Monitored Items

5.1.1 Environmental Conditions in the Test Laboratory

The device subject to verification was located inside a fume hood in the test laboratory with the room temperature set to 23 °C. The environmental conditions in the test laboratory during the period when the device was used to perform automatic sample preparation are shown in **Table 5-1**.

Over the period when automatic sample preparation was being performed, the room temperature was between 22.1 and 22.8 °C and the humidity between 20.0 and 68.0 %. The room temperature, which is an installation requirement for the device, remained stable at around 23 °C and had no notable impact on device operation.

Table 5-1 Environmental Conditions in the Test Laboratory

Period of Automatic Sample Preparation	Room Temp. (°C)	Humidity (%)
August 29 to December 1, 2023	22.1 to 23.8	20.0 to 68.0

5.1.2 Conditions During Sample Collection

(1) Weather data

The weather conditions in the vicinity of the sample collection sites are shown in **Fig. 5-1** (see Reference Materials page 47 for details). The sample collection days were selected (September 19 and 28, 2023) to ensure samples were collected where the rivers at the sampling points were not turbid due to rainfall or other factors.

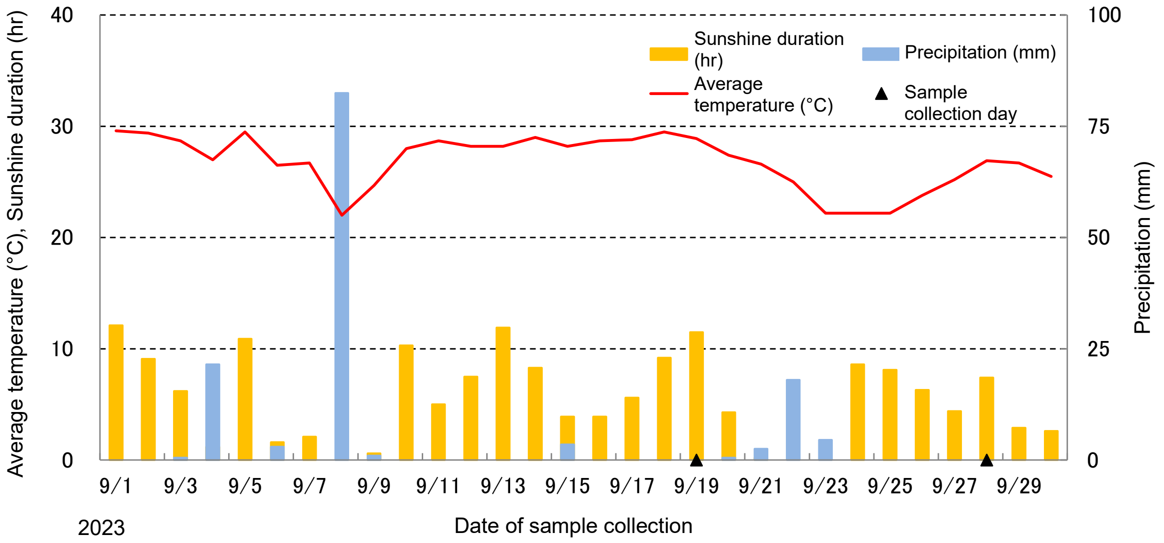


Fig. 5-1 Weather Conditions in Vicinity of Sample Collection Sites (Saitama Weather Station)

(2) Water velocity, water depth, filtering time, filtered volume, etc.

The on-site conditions during sample collection, including water velocity, water depth, filtering time, and filtered volume are shown in **Table 5-2**.

The water velocity and water depth at the 3 sampling points were between 0.20 and 0.45 m/s and 32 to 48 cm, respectively, and samples were collected with the sample collector standing in the river. Samples were collected with a plankton net by fully submerging the net opening close to the surface of the river and avoiding contaminating the sample with particles from the river bed. The filtering times were between 8 and 15 minutes and the filtered volumes were between 6.8 and 18.0 m^3. Filtering times were calculated based on the water velocity during collection and the required volume of filtered water (at least 10 m^3) and adjusted based on the rotation speed of the rotor attached to the flowmeter and the degree of clogging by suspended solids inside the net.

Table 5-2 Conditions during Sample Collection

River	River A			River B			River C		
Sampling Points	Point (1)			Point (2)			Point (3)		
Date Collected	September 19, 2023			September 28, 2023			September 28, 2023		
Collection Start Time	10:40			9:50			11:40		
Weather	Clear			Cloudy			Clear		
Current Direction	Normal flow			Normal flow			Normal flow		
Temperature (°C)	30.5			27.0			30.0		
Water Temperature (°C)	25.4			26.5			24.3		
Water Velocity (m/s)	0.26			0.20			0.45		
Water Depth (cm)	45			48			32		
Transparency (cm)	74			70			≥100		
Sample Number	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
Sample Name	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(2) -2	(3) -3
Filtering Time (min)	10	12	8	15	15	15	10	15	15
Filtered Volume (m^3)	8.4	9.3	6.8	7.3	10.5	8.5	13.0	18.0	15.0

5.1.3 Water Quality of River Water Samples

The results of the water quality analysis of river water samples, which were collected during MP sample collection, are shown in **Table 5-3**.
Samples were collected with the sample collector standing in the river. The turbidity, suspended solids, and total organic carbon at the 3 sampling points ranged from 2 to 5 mg-kaolin equivalent/L, 3 to 10 mg/L, and 1.2 to 2.9 mg/L, respectively. The river bed was also visible at each of the 3 points. The rivers at each sampling point flowed through residential areas in Saitama City and were therefore slightly murky with a foul odor.

Table 5-3 Water Quality of River Water Samples

River	River A	River B	River C
Sampling Points	Point (1)	Point (2)	Point (3)
Appearance	Pale yellow turbidity	Pale grayish-yellow turbidity	Pale turbidity
Odor	Mild foul odor	Mild foul odor	Mild foul odor
Turbidity (mg-kaolin equivalent/L)	3	5	2
Suspended Solids (mg/L)	6	10	3
Total Organic Carbon (mg/L)	1.5	2.9	1.2

5.1.4 Condition of Environmental Samples

The data on the condition of the environmental samples[*1] are shown in **Table 5-4**.

*1: River water samples collected in plankton net after undergoing subsequent filtration through a 0.1 mm mesh net, removal of contaminants 5 mm or larger, and drying (in a drier heated to no higher than 60 °C to prevent deterioration of plastics) to remove water without drying the sample completely (producing a wet sample that does not flow when tilted on a flat surface).

Table 5-4 Condition of Environmental Samples

Sampling Point	Sample Name	Appearance	Sample Weight (g)	After Filtration	After Drying
Point (1)	(1)-1	Blackish brown	4.38		
	(1)-2	Blackish brown	3.67		
	(1)-3	Blackish brown	6.72		
Point (2)	(2)-1	Blackish brown	10.2		
	(2)-2	Blackish brown	9.88		
	(2)-3	Blackish brown	8.51		
Point (3)	(3)-1	Blackish brown	5.27		
	(3)-2	Blackish brown	9.36		
	(3)-3	Blackish brown	6.70		

As described in **Table 5-4**, all environmental samples were blackish brown in appearance and the weights of the environmental samples (after drying at 60 °C) from Point (1) were 3.67 to 6.72 g, from Point (2) were 8.51 to 10.2 g, and from Point (3) were 5.27 to 9.36 g.
As the volume of filtered water differed between each environmental sample, environmental sample concentrations (g/m^3) were calculated based on the ratio of environmental sample weight (g) to volume of water filtered (m^3) (**Table 5-5**).

Table 5-5 Concentration of Environmental Samples (g/m^3)

Sampling Point	Point (1)			Point (2)			Point (3)		
Sample Name	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Concentration (g/m^3)	0.52	0.40	0.98	1.4	0.94	1.0	0.41	0.52	0.45

As shown in **Table 5-5**, the environmental sample concentrations were 0.40 to 0.98 g/m^3 at Point (1), 0.94 to 1.4 g/m^3 at Point (2), and 0.41 to 0.52 g/m^3 at Point (3). The relatively high concentration environmental samples collected in Point (2) ((2)-1 and (2)-3) contained numerous fragments of filamentous algae (spirogyra, etc.) (see Reference Materials page 40).
After automatic sample preparation was complete, material that precipitated during the density separation process remained as a residue on the strainer (see Reference Materials page 42). The main component of this residue was sand. Other than sand, the residue contained fibrous plant-derived material not fully decomposed by the hydrogen peroxide solution, fibers, and other material. The weight of the residue on the strainer after sample preparation was determined by drying the residue in a dryer at 105 °C for 2 hours and weighing what remained (**Table 5-6**).

Table 5-6 Weight of Residue on Strainer

Sampling Point	Point (1)			Point (2)			Point (3)		
Sample Name	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Residue Weight (g)	0.13	0.01	0.24	0.42	0.03	0.48	0.04	0.09	0.03

As shown in **Table 5-6**, samples (2)-1 and (2)-3 had particularly high residue weights (0.42 and 0.48 g respectively, after drying at 105 °C) and the residue from these two samples also contained more sand than other samples. Multiple large carp fish were observed swimming upstream of this sampling point which could have stirred up mud from the riverbed during sample collection.

5.2 Verification Test

The results of the verification test, which was the addition and recovery test, are shown in **Table 5-7**. The recovery of added reference plastic particles was 92 to 100 %, showing the device satisfied the performance under verification (at least 80 % recovery) with all samples. Examining the recovery results by specific gravity of added reference plastic particle reveals that 100 % recovery of 1.00 g/cm^3 particles was achieved in 33 % of samples (3 of 9 samples) and 100 % recovery of 1.35 g/cm^3 particles was achieved in 67 % of samples (6 of 9 samples), indicating that particles with a higher specific gravity were more readily recovered. Unrecovered reference plastic particles were found together with other residue adhered to the upper section of the reaction vessel and to the inner surface of the nozzle.

Table 5-7 Addition and Recovery Test Results

Sample Name	Sample Weight (g)	Reference Plastic Particle Specific Gravity (g/cm^3)	Number Added	Number Recovered	Number Not Recovered	Recovery
			(No. of Particles)			(%)
(1)-1	4.38	1.00	25	23	2	**92**
		1.35	25	25	0	**100**
(1)-2	3.67	1.00	25	25	0	**100**
		1.35	25	25	0	**100**
(1)-3	6.72	1.00	25	23	2	**92**
		1.35	25	25	0	**100**
(2)-1*3	10.2	1.00	25	25	0	**100**
		1.35	25	25	0	**100**
(2)-2	9.88	1.00	25	23	2	**92**
		1.35	25	24	1	**96**
(2)-3*3	8.51	1.00	25	23	2	**92**
		1.35	25	24	1	**96**
(3)-1	5.27	1.00	25	24	1	**96**
		1.35	25	25	0	**100**
(3)-2	9.36	1.00	25	25	0	**100**
		1.35	25	24	1	**96**
(3)-3	6.70	1.00	25	23	2	**92**
		1.35	25	25	0	**100**

*3: Decomposition reagent was replaced

For samples (2)-1 and (2)-3,*4 recoveries of 92 to 100 % were achieved by replacing the decomposition reagent during the oxidation process to enhance the decomposition of organic matter and ensure the organic matter in the samples was fully decomposed.

*4: A substantial amount of brown or white floating matter and suspended solids were still present and no reaction with hydrogen peroxide solution (bubbling) was apparent 24 hours into the oxidation process

The reproducibility of recovery was evaluated by calculating the coefficient of variation from the recovery mean and standard deviation. The results of this evaluation are shown in **Table 5-8**.

Table 5-8 Recovery Statistics for Spiked Recovery Test (n = 9)

Specific Gravity (g/cm^3)	Max. Recovery (%)	Min. Recovery (%)	Mean Recovery (%)	Standard Deviation (%)	Coefficient of Variation (%)
1.00	100	92	95.1	3.9	4.1
1.35	100	96	98.7	2.0	2.0

The coefficient of variation, which is a measure of dispersion, was within 5 % for the 1.00 g/cm^3 particles (4.1 %) and the 1.35 g/cm^3 particles (2.0 %). The results also showed that reproducibility was better for the higher density particles.

5.3 Reference Test

5.3.1 Recovery

The results of the reference test, which was an environmental MPs recovery test, are shown in **Table 5-9**.
The recovery of environmental MPs (1 mm or larger) already present in the river water samples was 82 to 100 %.

Table 5-9 Environmental MP (1 mm or larger) Recovery Test Results

Sample Name	Sample Weight	Weight of Residue on Strainer	Number Recovered (A)	Number Not Recovered (B)	Number of Environmental MPs (A) + (B)	Recovery
	(g)	(g)	(No. of Particles)			(%)
(1)-1	4.38	0.13	11	0	11	**100**
(1)-2	3.67	0.01	25	2	27	**93**
(1)-3	6.72	0.24	24	3	27	**89**
(2)-1*5	10.2	0.42	30	4	34	**88**
(2)-2	9.88	0.03	49	6	55	**89**
(2)-3*5	8.51	0.48	44	7	53	**86**
(3)-1	5.27	0.04	47	10	57	**82**
(3)-2	9.36	0.09	103	16	119	**87**
(3)-3	6.70	0.03	78	12	90	**87**

*5: Decomposition reagent was replaced

Similar to the reference plastic particles, most of the unrecovered environmental MPs were adhered to the upper section of the reaction vessel and to the inner surface of the nozzle together with other residue.

The reproducibility of recovery was evaluated by calculating the coefficient of variation from the recovery mean and standard deviation. The results of this evaluation are shown in **Table 5-10**. Although each sample varied in weight and contained different amounts of algae and other organic matter, the coefficient of variation in the environmental MP recovery test was within 10 % at 5.6 %.

Table 5-10 Recovery Statistics for Environmental MPs Recovery Test (n = 9)

Max. Recovery (%)	Min. Recovery (%)	Mean Recovery (%)	Standard Deviation (%)	Coefficient of Variation (%)
100	82	89.0	5.0	5.6

5.3.2 Relationship between Sample Weight and Recovery

Fig. 5-2 shows sample weight plotted against recovery for each sample.

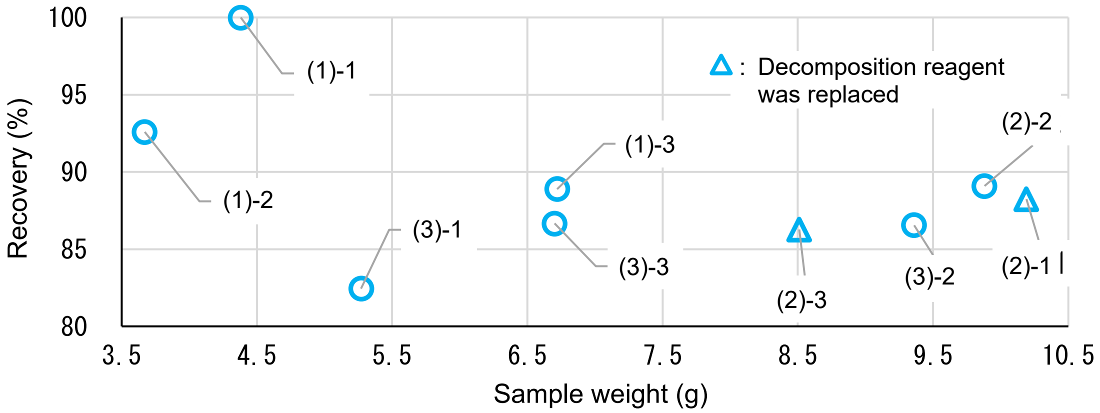


Fig. 5-2 Sample Weight and Recovery

As shown in **Fig. 5-2**, recovery did not exceed 90 % for any of the samples weighing over 4.5 g. Environmental samples (2)-1 and (2)-3 were heavy, weighing 8.51 g and 10.2 g, and contained fragments of filamentous algae. Replacing the hydrogen peroxide solution in the reaction vessel allowed the device to achieve recoveries of 86 % and 89 % from these two heavy samples. This shows that the device subject to verification can achieve recovery of at least 80 % even when processing samples that weigh around 10 g that contain a large amount of algae and other organic matter.

5.3.3 Relationship between Weight of Residue on Strainer and Recovery

Fig. 5-3 shows the weight of the residue on the strainer plotted against recovery for each sample.

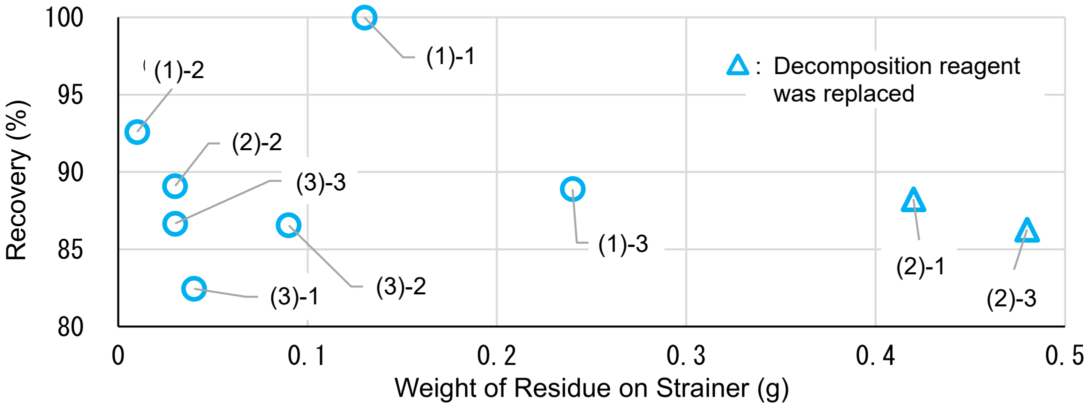


Fig. 5-3 Weight of Residue on Strainer and Recovery

Although the residue collected on the strainer from samples (2)-1 and (2)-3 contained a large amount of sand and was heavy compared to the other samples, **Fig. 5-3** shows the device achieved recoveries of 88 % and 86 % from these samples, respectively. Conversely, the lowest recovery was 82 % from environmental sample (3)-1, which had a low residue weight with minimal sand compared to the other samples. No relationship was revealed between residue weight and recovery, and the test confirmed that recoveries of 80 % and higher can be obtained from samples with residue that contains 0.5 g or less sand.

5.3.4 Relationship between Number of Environmental MPs and Recovery

Fig. 5-4 shows the number of environmental MPs plotted against recovery for each sample.

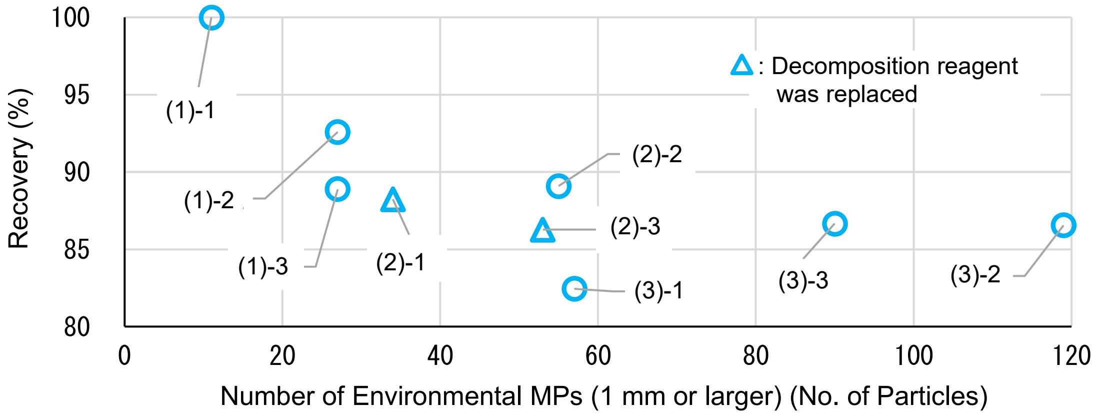


Fig. 5-4 Number of Environmental MPs and Recovery

Fig. 5-4 shows that recovery tends to decrease with increasing number of MPs, but also confirms that the device can achieve recoveries of at least 80 % from samples that contain around 90 to 120 environmental MPs.

5.3.4 Identification of Different Types of MP by FTIR

After the sample preparation process had been completed and candidate MP particles were retrieved from the recovery filter, FTIR was used to identify the types of MPs. The results from this identification analysis are shown by point in **Fig. 5-5**, **5-6**, and **5-7**.

The MPs that were recovered from the river water samples included six different types of plastic: polystyrene (PS), polypropylene (PP), polyethylene (PE), ethylene-vinyl acetate (EVA), acrylonitrile butadiene styrene (ABS), and polyethylene terephthalate (PET). PP and PE in particular were found in every environmental sample and accounted for at least 70 % of all MPs. The next most common type of MP recovered was EVA, which was found in all environmental samples other than (1)-1 and accounted for between 6.4 and 22.4 % of MPs in these samples.

The unrecovered MPs were shown to include three types of plastic, PP, PE, and EVA. A particularly large number of unrecovered environmental MPs (at least 10) were observed from samples collected in Point (3) (**Fig. 5-7**). PP accounted for between 75.0 and 87.5 % of these unrecovered environmental MPs in samples from Point (3), which was like the PP proportion of recovered environmental MPs.

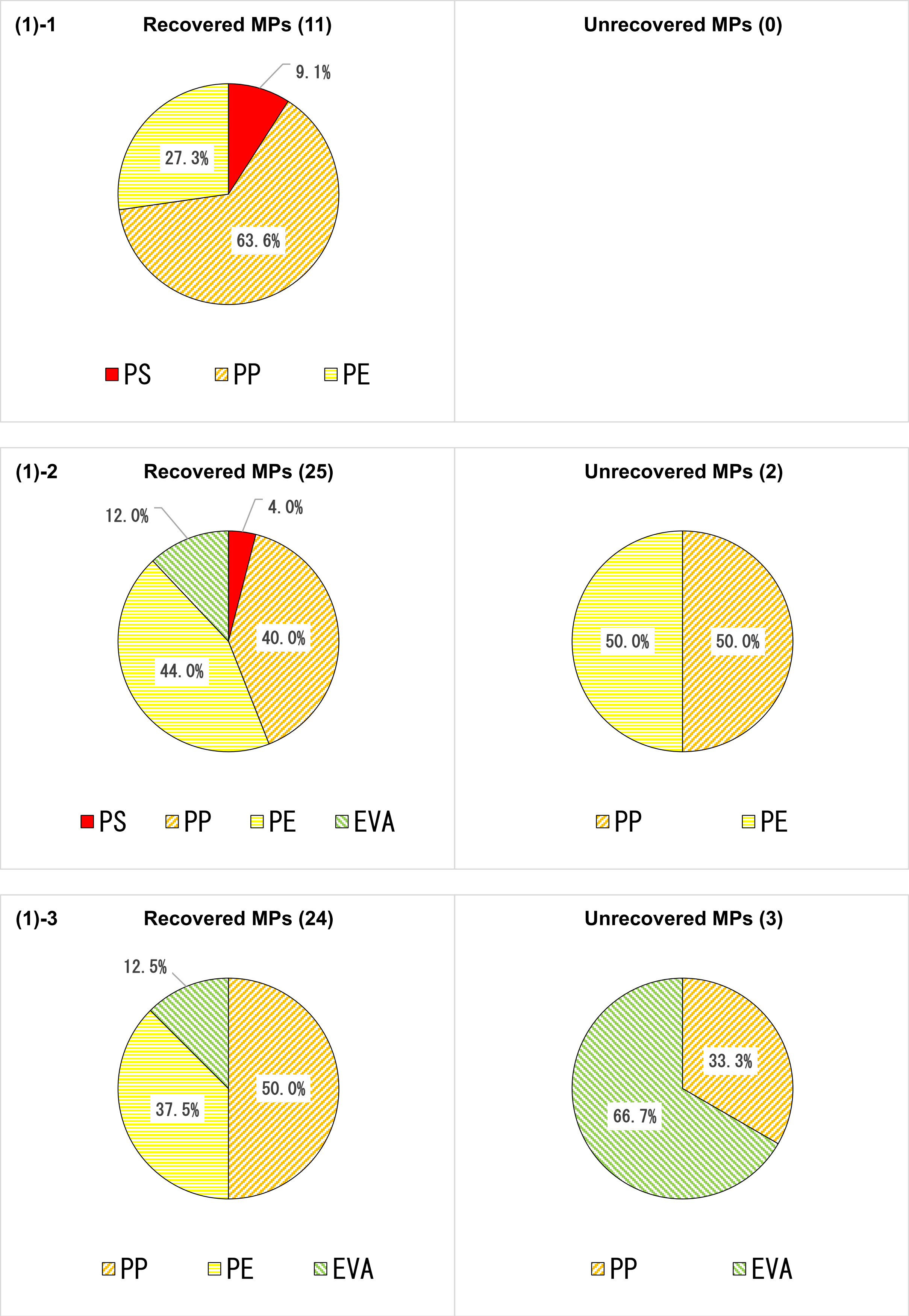


Fig. 5-5 MP Polymers and Proportion of Each Polymer (Point (1))

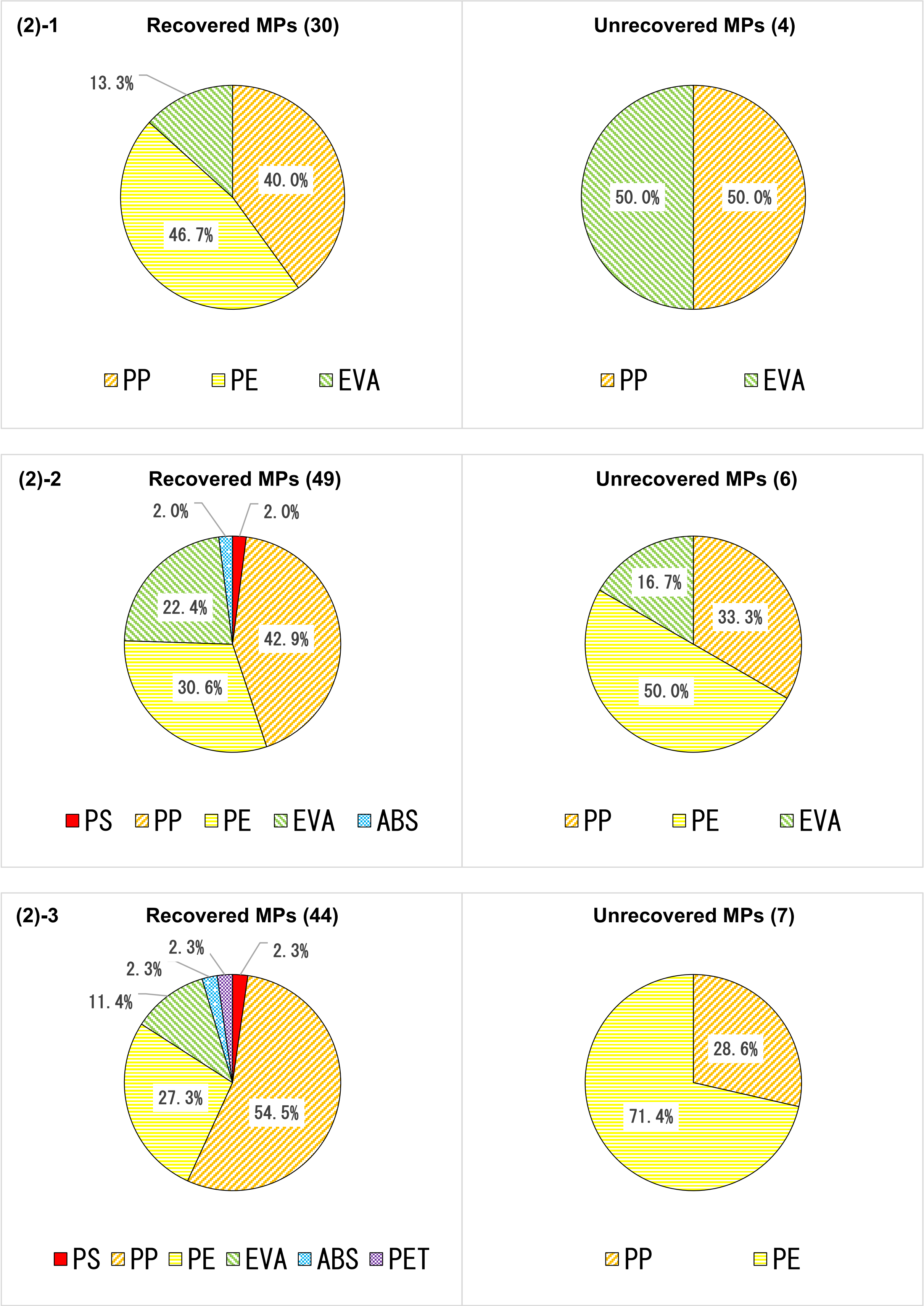
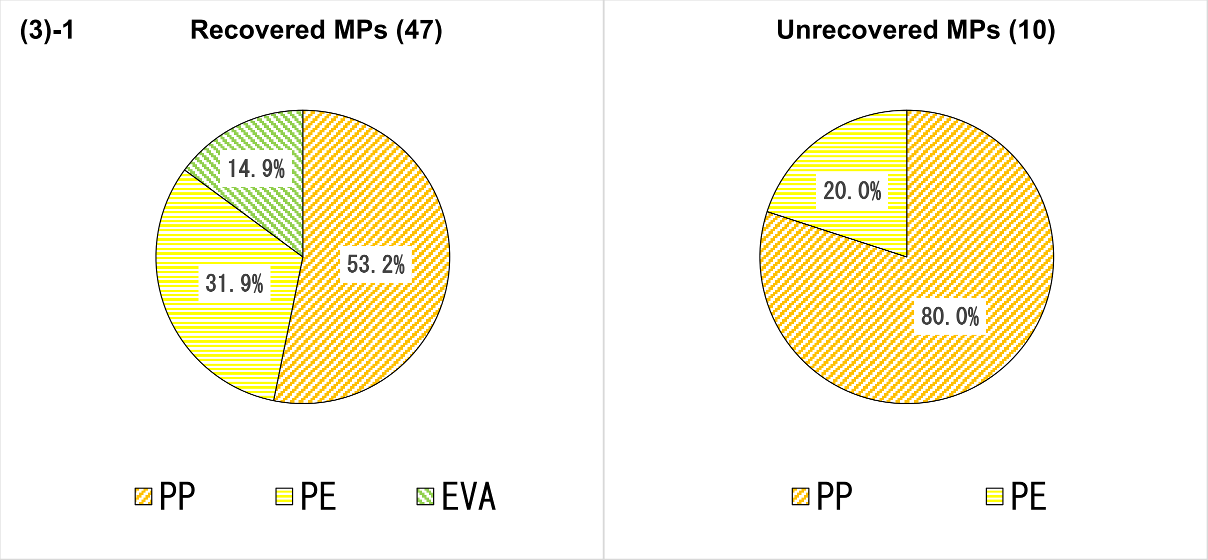
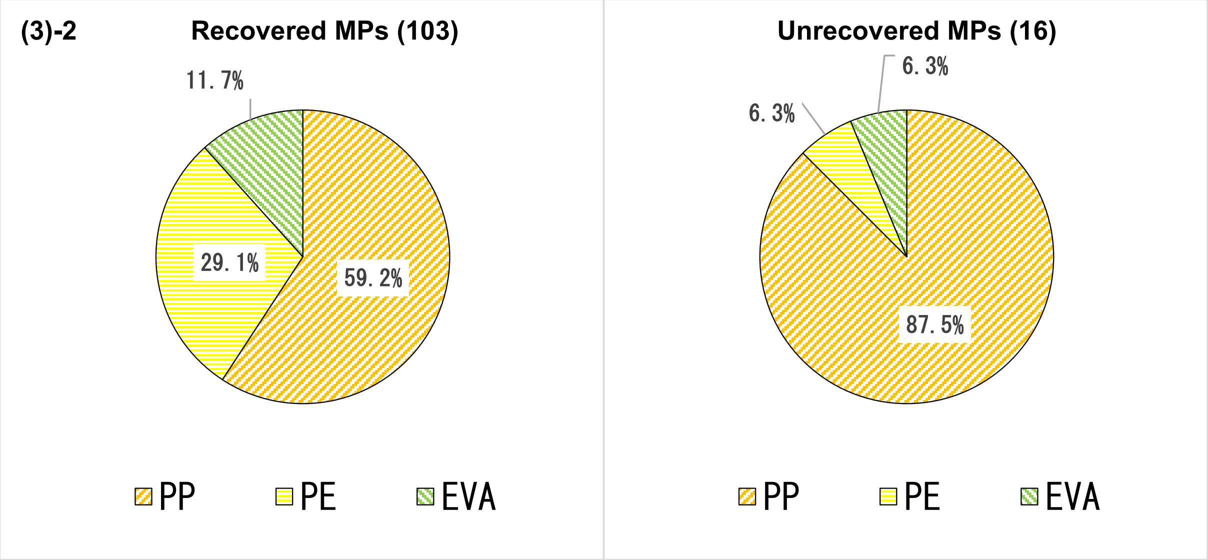


Fig. 5-6 MP Polymers and Proportion of Each Polymer (Point (2))

(3)-1



(3)-2



(3)-3

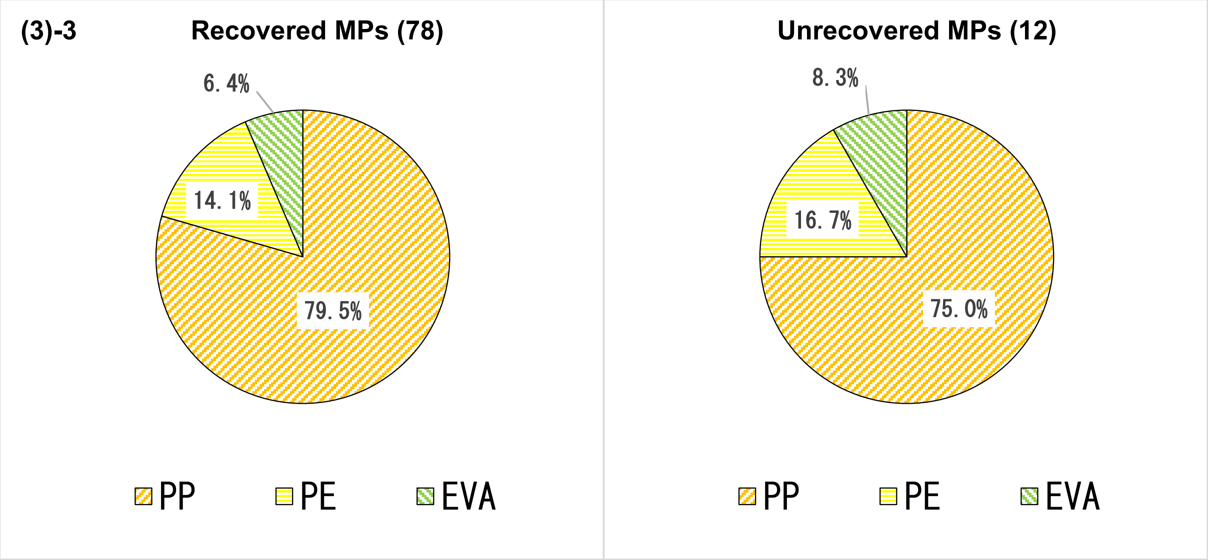


Fig. 5-7 MP Polymers and Proportion of Each Polymer (Point (3))

5.4 Environmental Impact, Operation, and Maintenance of Device

Table 5-11 Environmental Impact, Operation, and Maintenance

Category	Item	Results
Environmental impact	Electricity usage	The amount of power consumed by the device subject to verification during sample preparation (lasting approx. 82 hours) was approx. 4.2 kWh (mean).
	Noise	Noise level measurements are shown in **Fig. 5-8**.
	Waste liquid	The amounts of waste liquid (including wash water) produced under the operating conditions used by the automatic sample preparation device during this verification are as shown below. • Waste hydrogen peroxide solution: approx. 520 mL (When decomposition reagent is replaced： approx. 620 mL) • Waste sodium iodide solution: approx. 770 mL
Operation and maintenance	Quality of sample water that can be processed by the device subject to verification	Water quality analysis results are shown in **Fig. 5-9**.
	Skills required to operate and maintain the device subject to verification	Users of the device must have received education and training sufficient to understand the handling and disposal, etc. of chemical substances based on regulations on chemical substances in the Industrial Safety and Health Act. The labor required to commence automatic sample preparation is approx. 15 minutes of labor by a single worker (preparing reagents, transferring sample to the reaction vessel, assembly, etc.) and the device is also simple to operate from the control screen.
	Reliability of device subject to verification	• When there was an air gap in the wash water bottle at the start of automatic sample preparation, “Not Ready” was displayed on the control screen and sample preparation did not start. • A buzzer sounded when the cover of the main unit was opened while the automatic sample preparation device was running.
	Resumption of operation after trouble	• When the wash water bottle was filled with pure water, “Ready” was displayed on the control screen and sample preparation started. • When the main unit cover was closed and the “stop” button on the front of the main unit was pressed, the buzzer sound ceased and device operation was restarted from the control screen.
	Decomposition reagent replacement feature	• This feature is a manual feature and not automatic, but starting the feature only requires around 1 minute of simple work. • The decomposition reagent was replaced once during oxidation for samples containing a large amount of algae and other organic matter (approx. 10 g) to enhance decomposition and ensure that the organic matter was completely decomposed. This replacement of the decomposition reagent resulted in recoveries of 80 % or higher. • Even for samples weighing more than 10 g, this feature can probably be used multiple times to enhance the decomposition of organic matter and achieve a recovery of 80 % or higher.
	Assessment of operation and maintenance manuals	The manual included device drawings and images of the control screen that ensured the manual was easy to read and device operation and maintenance were easy to understand.

5.4.1 Noise Levels

Fig. 5-8 shows noise level measurements taken during sample preparation. During pump operation, the noise level was approx. 3 dB higher than the noise level prior to starting the device (noise from the fume hood). The pumps in the device operate continuously for approx. 4 minutes at a time and for just 1 hour in total over the course of a single sample preparation lasting approx. 82 hours, showing the noise produced by the device was only produced for a negligible period of time.

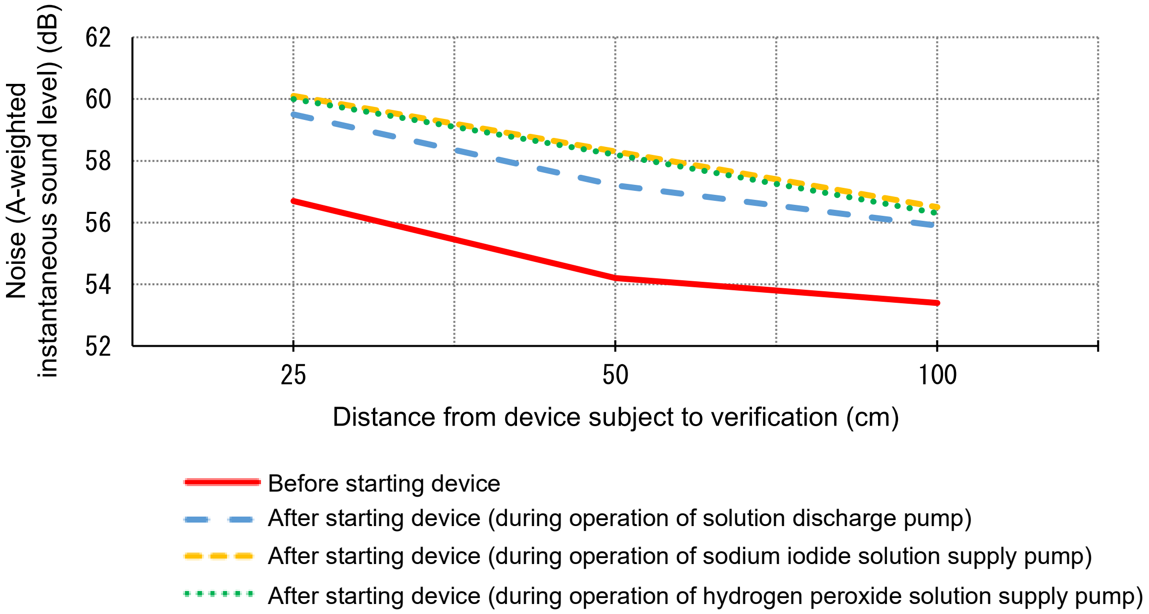


Fig. 5-8 Noise Level Measurements
(Noise Level and Distance from Device Subject to Verification)

5.4.2 Sample Water Quality Processable by Device Subject to Verification

Fig. 5-9 shows water quality parameters (turbidity, SS, and TOC) plotted against recovery of environmental MPs (1 mm and larger).
There was no clear association between any of the water quality parameters and recovery, though the results do confirm that the device satisfies the performance subject to verification (80 % or higher recovery) when the sample water was high in turbidity (5 mg-kaolin equivalent/L), SS (10 mg/L), and TOC (2.9 mg/L) and the decomposition reagent is replaced during the oxidation process.

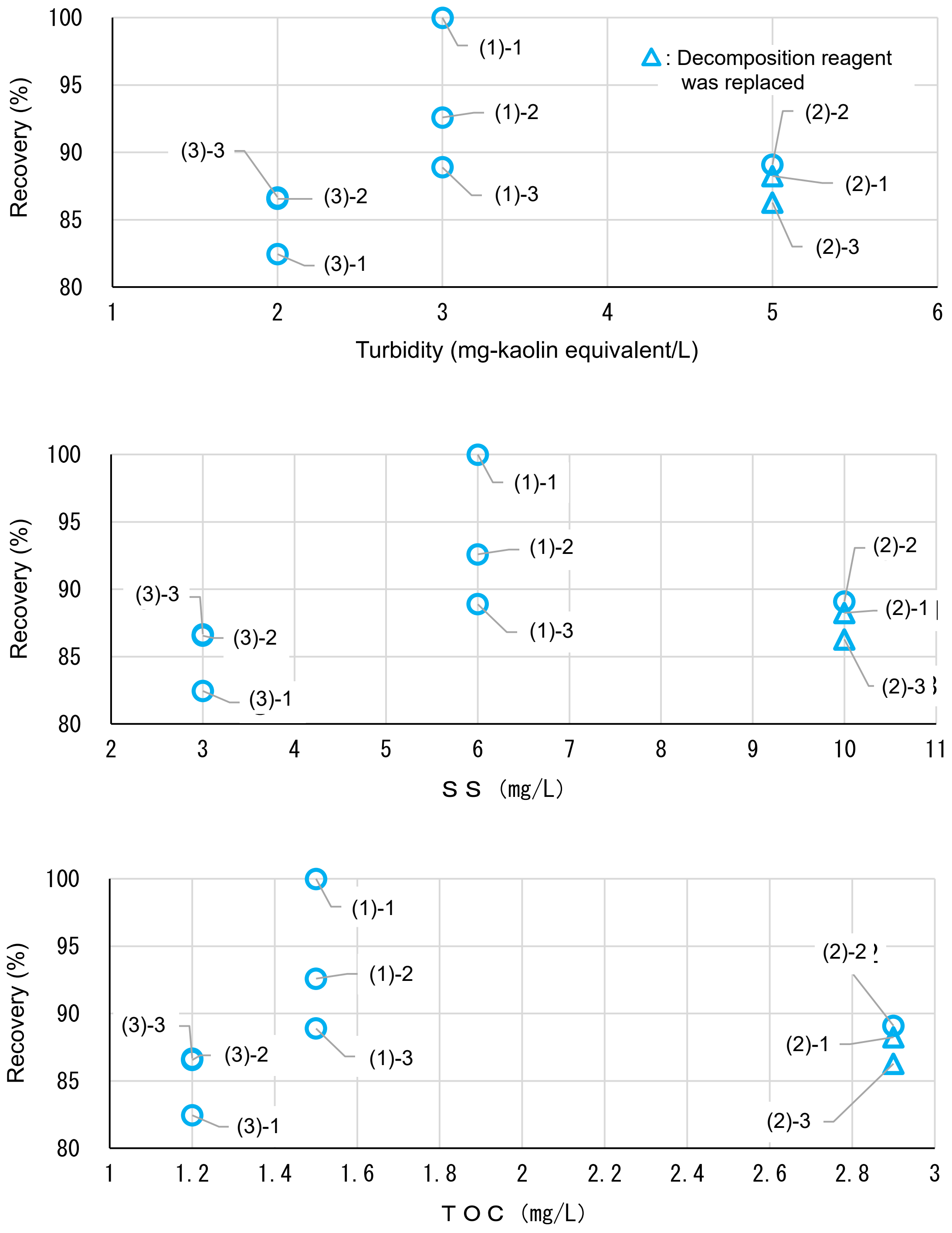


Fig. 5-9 Relationship between Water Quality Parameters (Turbidity, SS, and TOC) and Recovery of Environmental MPs

6. Findings (Summary of Verification Results)

In conclusion, the characteristic feature of the technology subject to verification as viewed from the perspective of the verification results is summarized below.

(1) Technology overall

The characteristic feature of the device subject to verification is that it performs all sample preparation processes (oxidation and density separation) automatically in a single reaction vessel. Because the device is not impacted by the skill level of the operator, the device reduces variability in MPs recovery. In an addition and recovery test using reference plastic particles (specific gravity: 1.00 and 1.35 g/cm^3) added to river water samples, the recovery of reference particles was 92 to 100 % (coefficient of variance: within 5 %) from environmental samples weighing 3.67 to 10.2 g (9 samples), which met the performance subject to verification (80 % or higher recovery). The recovery of environmental MPs (1 mm or larger) by the device was also 82 to 100 % (coefficient of variation; 5.6 %), or 80 % or higher.

This device can be expected to improve the accuracy of empirical data in studies and research into MPs and have a positive impact on the development of future MP-related measures. For the above reasons, this device can be considered beneficial for environmental conservation.

(2) Miscellaneous

When hydrogen peroxide solution loses its activity during the oxidation process and a substantial amount of organic matter remains, the device subject to verification has a feature that replaces the decomposition reagent in the reaction vessel during ongoing sample preparation. This feature provides a simple method of introducing new hydrogen peroxide solution into the reaction vessel and enhancing the decomposition of organic matter. When this feature was used, the recovery of environmental MPs was 80 % or higher even from environmental samples that contained a large amount of algae and other organic matter (samples weighing approx. 10 g, or samples obtained by filtering approx. 10 m^3 of river water containing 10 mg/L of suspended solids), which demonstrated the effectiveness of this feature.

Testing also demonstrated that recovery was at least 80 % when around 120 environmental MPs were present in the sample.

Appendix

1. Glossary

(1) Terminology related to verification

Term	Description
Technology subject to verification	The technology that is being verified.
Device subject to verification	Actual equipment or apparatus used during testing and the physical embodiment of the technology subject to verification.
Verification test	Test used to measure the performance and impact of the technology subject to verification.
Reference test	Test that serves as a reference for measuring the performance and impact of the technology subject to verification.
Monitored item	The items used to monitor the test conditions.

(2) Terminology related to water quality

Term	Description
Turbidity	A measure of water clarity
Suspended solids (SS)	Particulate matter 2 mm or smaller in diameter that is floating or suspended in water
Total organic carbon (TOC)	A measure of the amount of organic matter in water and expressed as total carbon

(3) Terminology related to plastics

Term	Description
Microplastics (MPs)	Plastic waste that is 5 mm or smaller in size
Polystyrene (PS)	A polymer of styrene. This plastic is very workable and used to make electrical appliances and as styrofoam to make packing material and food packaging.
Polypropylene (PP)	A polymer of ethylene. This plastic is strong enough to withstand deformation and failure and is used to make automobile parts, home appliances, medical devices, and food containers.
Polyethylene (PE)	A polymer of ethylene. This plastic is very workable and used to make packaging material, containers, pipes, plastic bags, and plastic wrap.
Ethylene-vinyl acetate (EVA)	A copolymer of ethylene and vinyl acetate. This plastic is very durable and used to make protective material, various types of insulating materials, and sign boards.
Acrylonitrile butadiene styrene (ABS)	A copolymer of acrylonitrile (A), butadiene (B), and styrene (S). This plastic is very resistant to impact, heat, and cold, exhibits excellent glossy qualities, and is readily colored with pigments or dyes. Due to these properties, this plastic has a wide range of applications including for daily commodities, automobile parts, and electrical appliances.
Polyethylene terephthalate (PET)	A copolymer of ethylene glycol and terephthalic acid or ethylene glycol and dimethyl terephthalate. This plastic is resistant to pressure and chemicals and is used to make plastic bottles, films, and clothing fibers.

(4) Terminology related to microplastics monitoring

Term	Description
Guidelines for River and Lake Microplastics Monitoring Methods, March 2023, Water Environment Management Division, Environmental Management Bureau, Ministry of the Environment	Users: Local public entities, and researchers, institutions, and businesses working with local public entities assessing the extent of MPs in rivers and lakes. MPs covered by guidelines: MP fragments and fibers under 5 mm in size in rivers and lakes Standard specifications related to automation of sample preparation: These guidelines include supplementary material titled "The Standard Specifications of Automatic Sample Preparations in Guidelines for River Microplastics Monitoring Methods." These standard specifications describe a method for testing the performance of a device that automates oxidation and density separation. The method described is an addition and recovery test performed using commercially available reference plastic particles (two specific gravities: one around 1.00 g/cm^3 and another around 1.35 g/cm^3, each 500 to 600 μm in size) (25 of each reference particle are added: 50 total reference particles added) where the determining criterion is a recovery of 80 % or higher.
30 % hydrogen peroxide solution	Classified as a deleterious substance by the Poisonous and Deleterious Substances Control Act. A dangerous and hazardous substance that requires notification or labeling by name under the Industrial Safety and Health Act. In MPs monitoring, a weakly acidic aqueous solution used during the oxidation process to decompose and remove organic matter.
5.3 mol/L sodium iodide solution	A dangerous and hazardous substance that requires notification or labeling by name under the Industrial Safety and Health Act. In MPs monitoring, an aqueous solution with a specific gravity of 1.5 g/cm^3 used in the density separation process to separate soil particles and other inorganic matter from plastic.
Fourier transform infrared spectrophotometer (FTIR)	An instrument that irradiates a sample under analysis with infrared rays (wavenumbers: 4000 to 500 cm^{-1}) and measures the intensity of transmitted or reflected light. Infrared rays are absorbed by molecules as vibrational or rotational energy and samples produce unique infrared absorbance spectra based on the structure of the component molecules, which allows for the identification of different types of plastic.

2. Information on Quality Control-Related Matters

2.1 Quality Control of Data

Analysis was performed based on standard operating procedures prepared in accordance with JIS and other standards and accuracy control was implemented through data management and review as shown in **Appendix Table 1-1**. Other matters were conducted in accordance with JIS and other standards and mean values were used when multiple measurements could be taken.
In light of the above, it was confirmed that data was subject to proper quality control.

Appendix Table 1-1 Method Used to Check the Reliability of Measurements

Parameter	Method of Accuracy Control
Turbidity Suspended solids Total organic carbon	Duplicate measurements were performed on some samples.

* The balance used for weighing underwent periodic external calibration.

2.2 Audit of Quality Control System

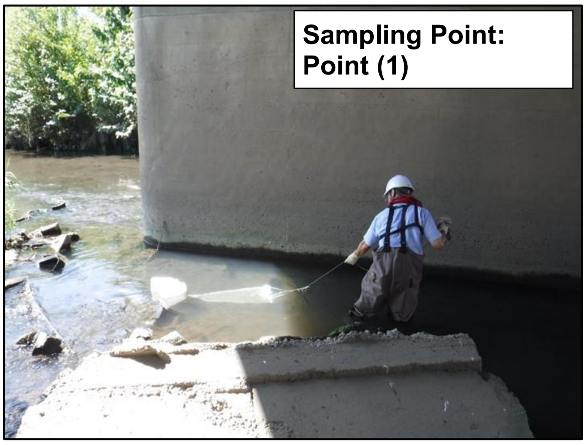
A quality audit was carried out on the data obtained during this verification to check the verification was being conducted appropriately. This quality audit was an internal audit conducted once during the verification period by a department independent of the verification and in accordance with the quality management system established by the verifying organization.
The internal audit confirmed that the verification was in compliance with the requirements of the quality control system according to the manual and was properly implemented and maintained.
Summary information about the internal audit is provided in **Appendix Table 1-2**.

Appendix Table 1-2 Summary of Internal Audit

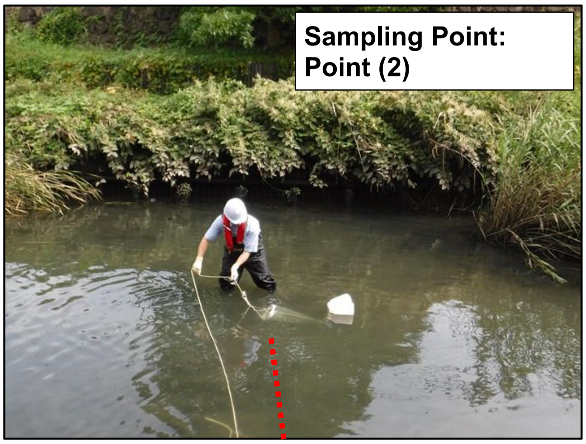
Date of Internal Audit	March 18, 2024 (Monday)
Internal Auditor	ISO specialist, General affairs section, Administrative headquarters
Audited Department	All departments involved in verification
Outcome of Internal Audit	The verification was in compliance with the requirements of the quality control system and was properly implemented and maintained.

Reference Materials

1. Collecting MP Samples



Sampling Point: Point (1)



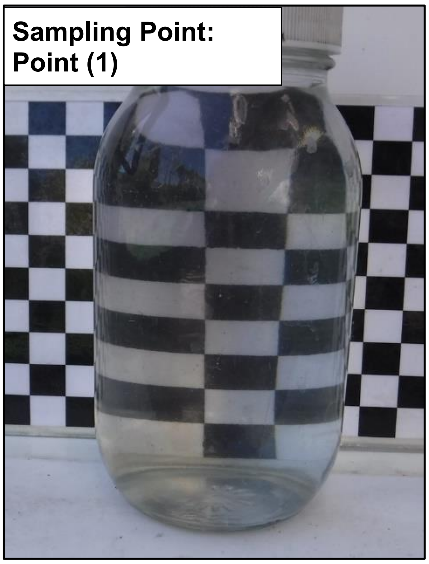
Sampling Point: Point (2)

Sampling Point: Point (3)

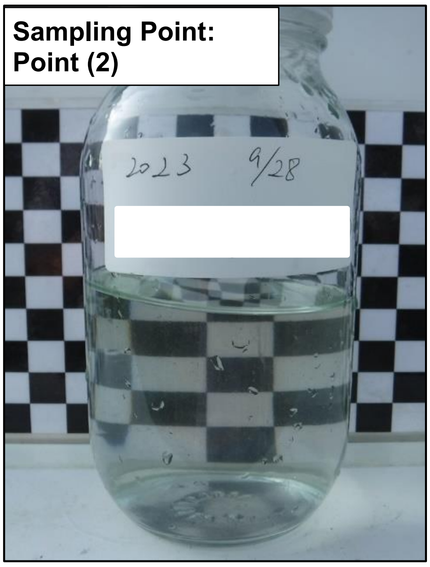
Inside the net after sample collection

2. Images of Collected Samples

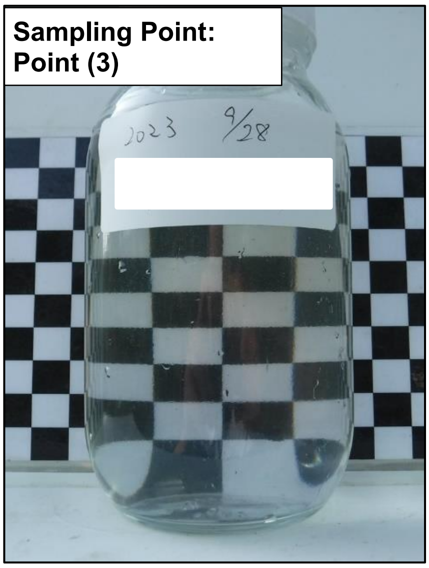
(1) Samples for water quality analysis



Sampling Point: Point (1)

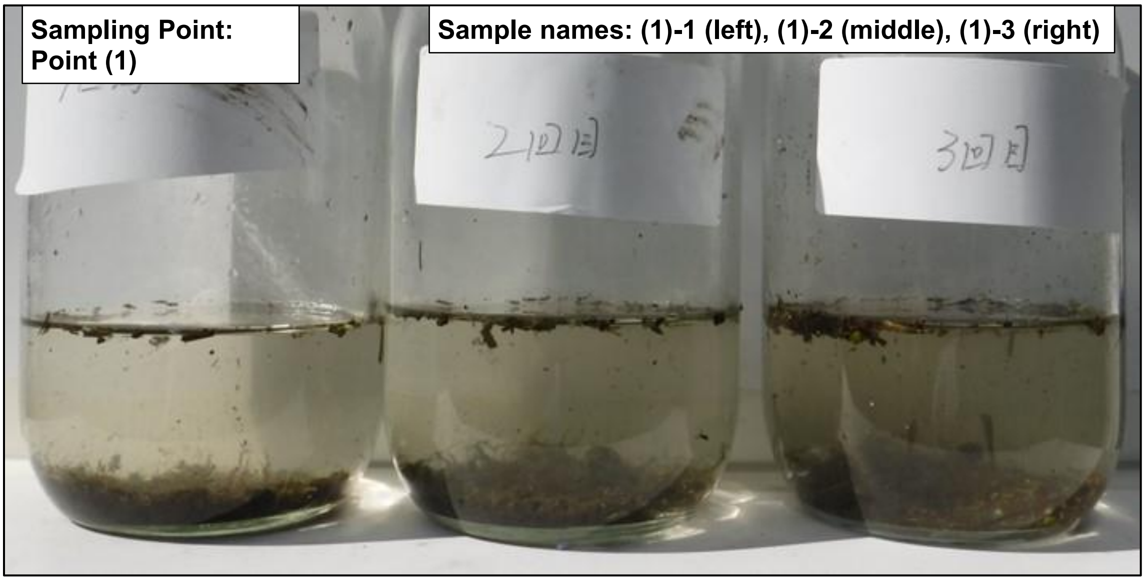


Sampling Point: Point (2)

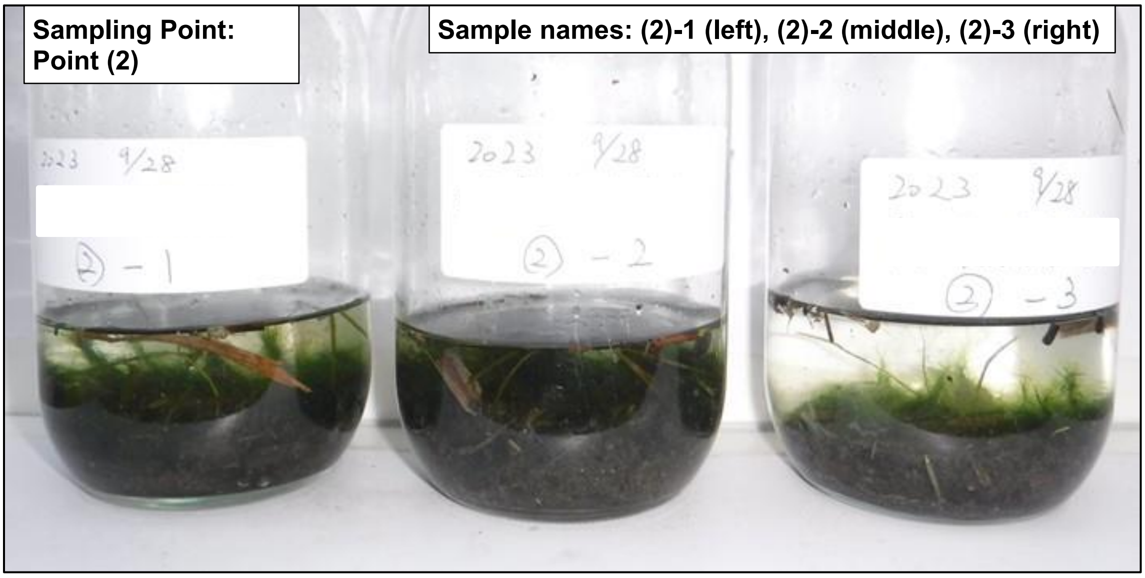


Sampling Point: Point (3)

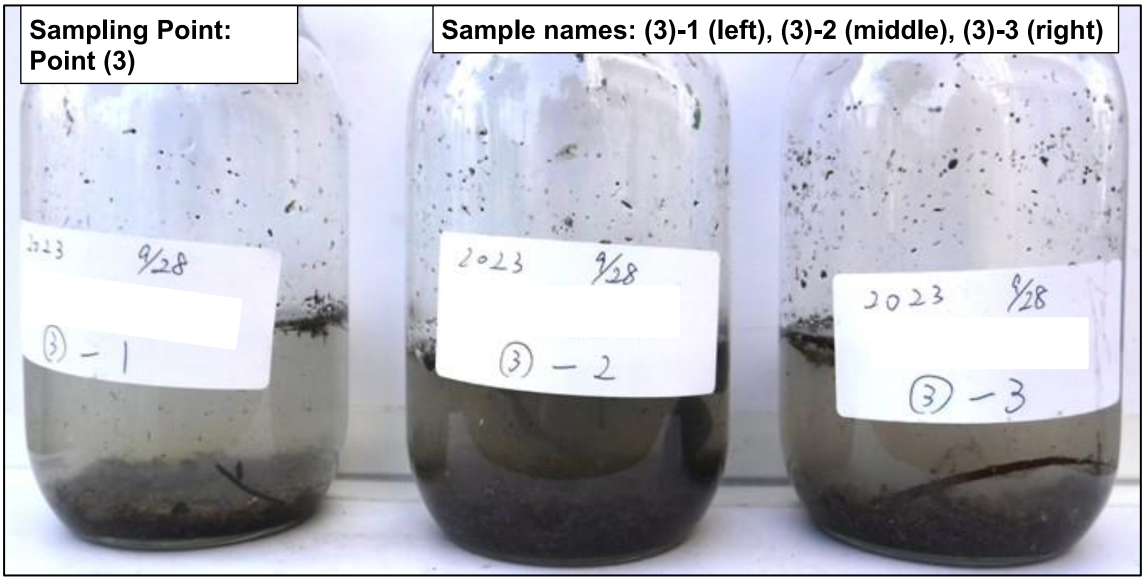
(2) Samples for addition and recovery test


Sampling Point: Point (1)

Sample names: (1)-1 (left), (1)-2 (middle), (1)-3 (right)


Sampling Point: Point (2)

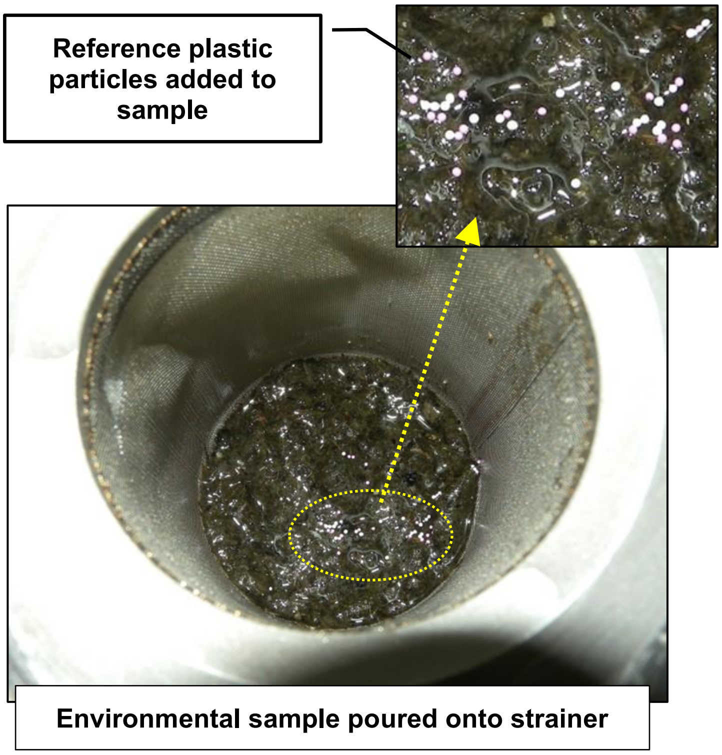
Sample names: (2)-1 (left), (2)-2 (middle), (2)-3 (right)


Sampling Point: Point (3)

Sample names: (3)-1 (left), (3)-2 (middle), (3)-3 (right)

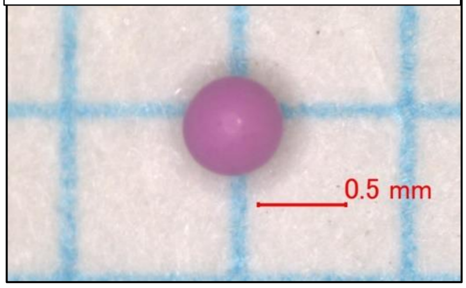
3. Images of Automatic Sample Preparation

(1) Addition of plastic reference particles

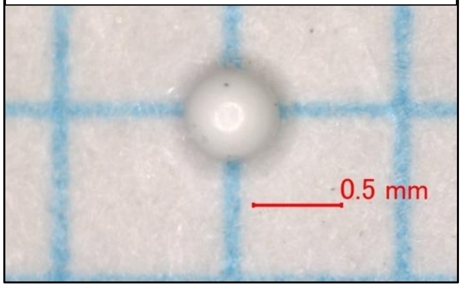

Reference plastic particles added to sample

Environmental sample poured onto strainer

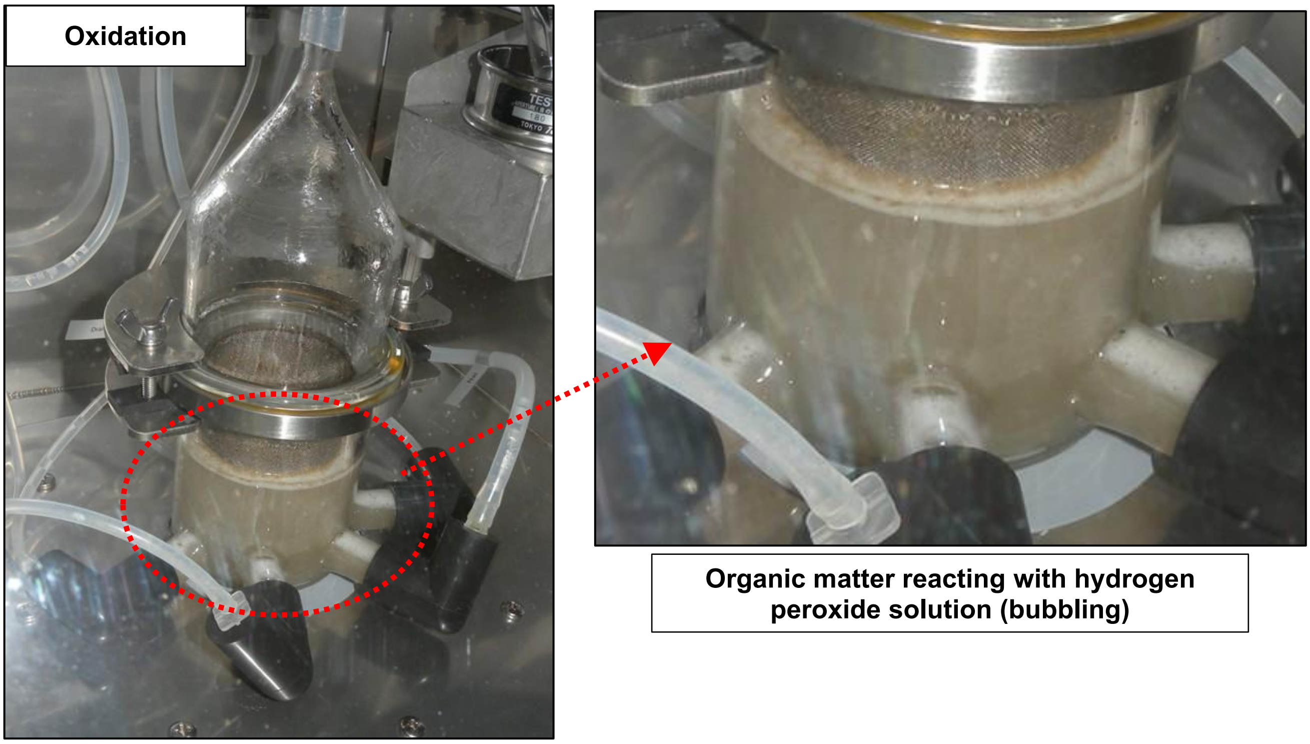
Reference plastic particle: 1.00 g/cm^3



Reference plastic particle: 1.35 g/cm^3

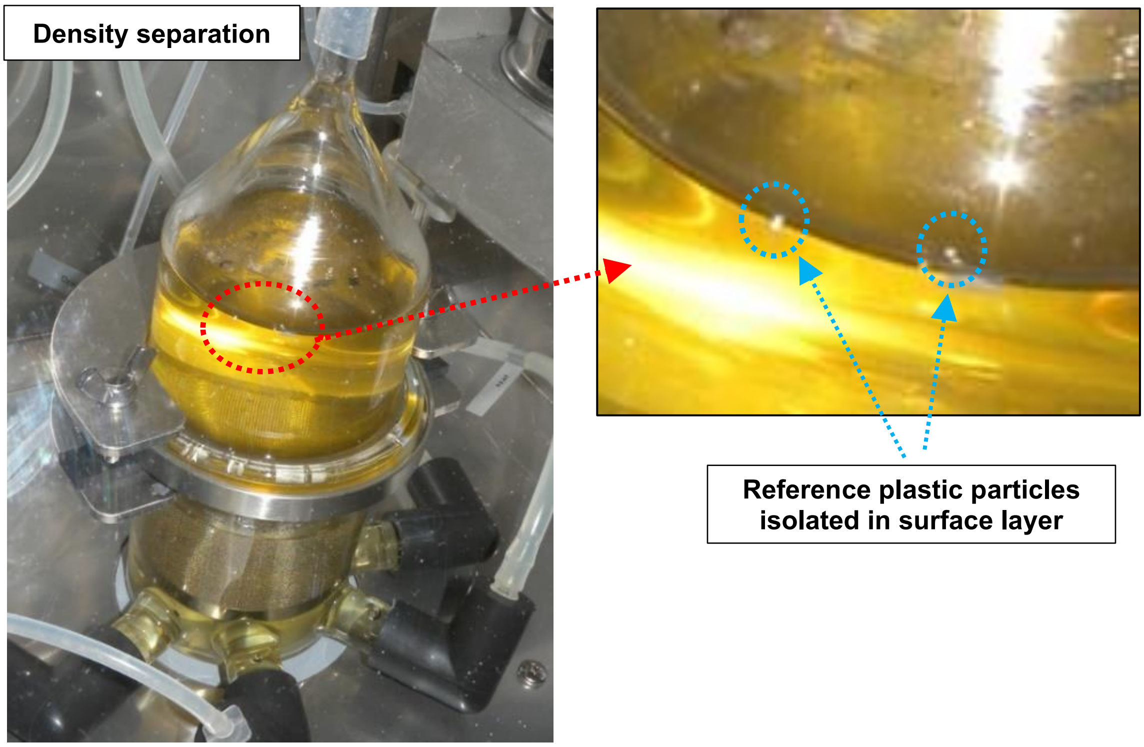


(2) Images of oxidation process


Oxidation

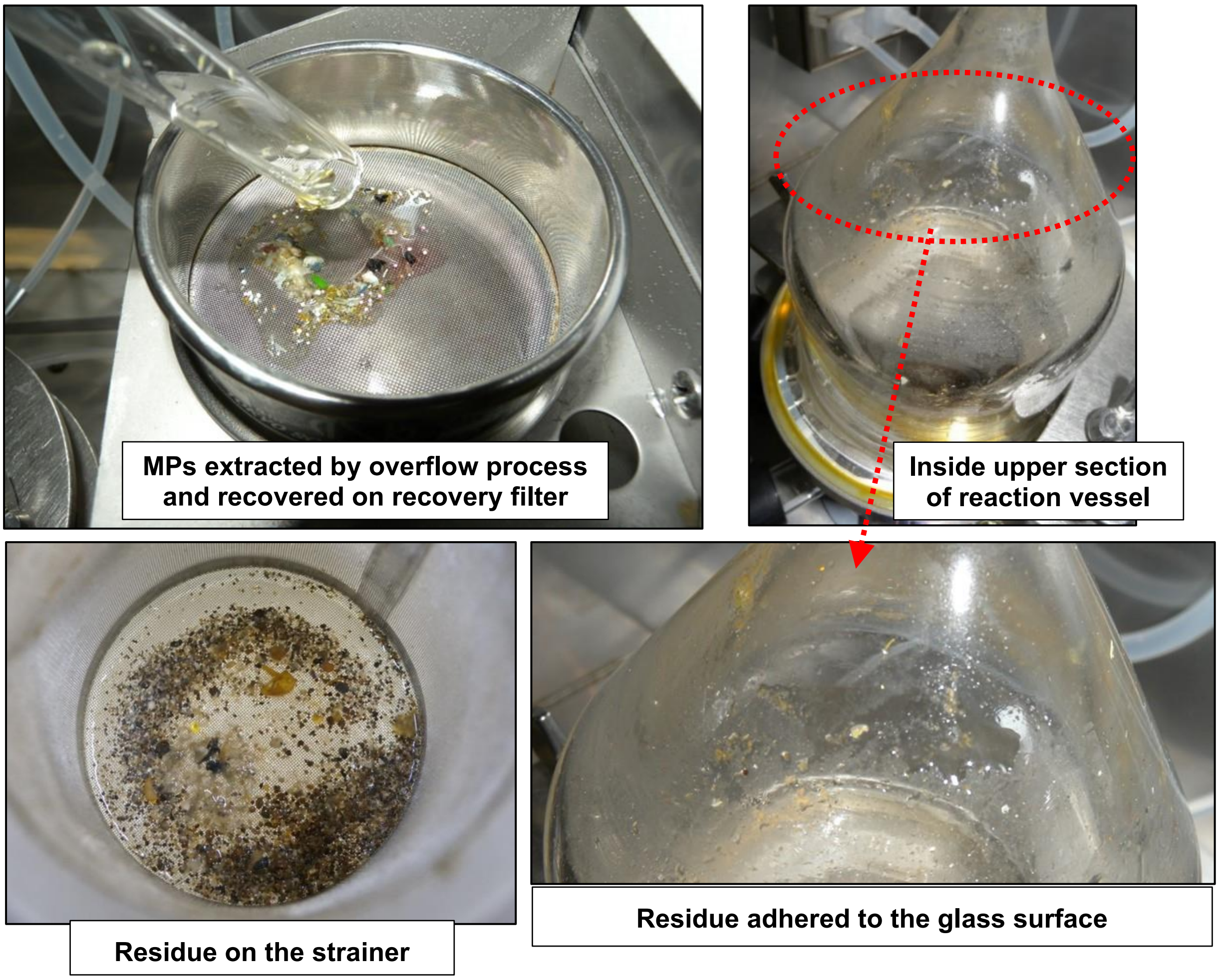
Organic matter reacting with hydrogen peroxide solution (bubbling)

(3) Images of density separation



Reference plastic particles isolated in surface layer

(4) Images of recovery


MPs extracted by overflow process and recovered on recovery filter

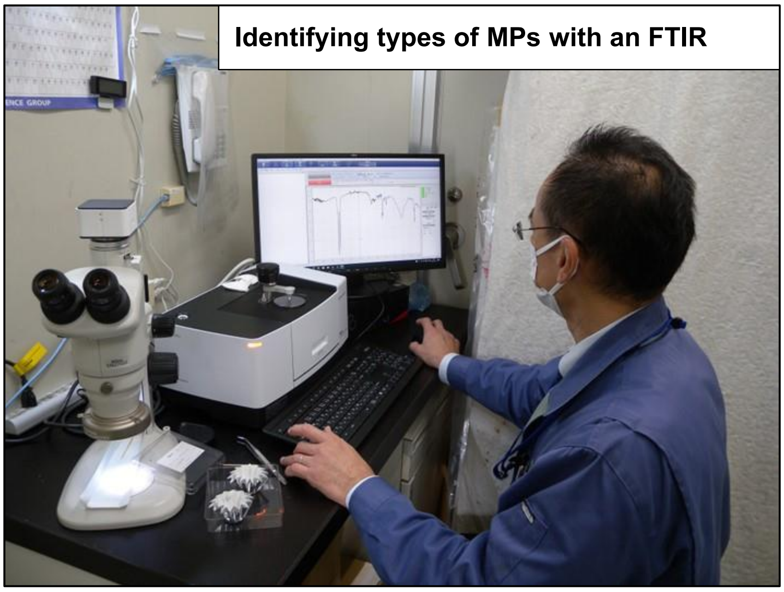
Inside upper section of reaction vessel

Residue on the strainer

Residue adhered to the glass surface

4. Images of MP Sorting and Identification

Sorting candidate MPs with a stereoscopic microscope

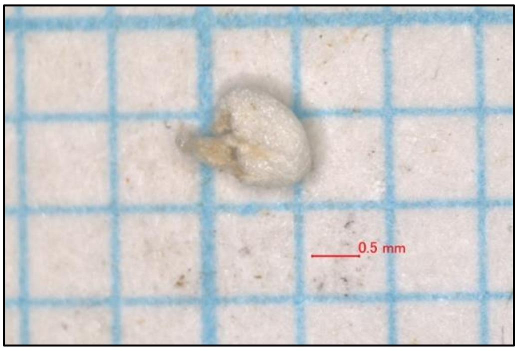


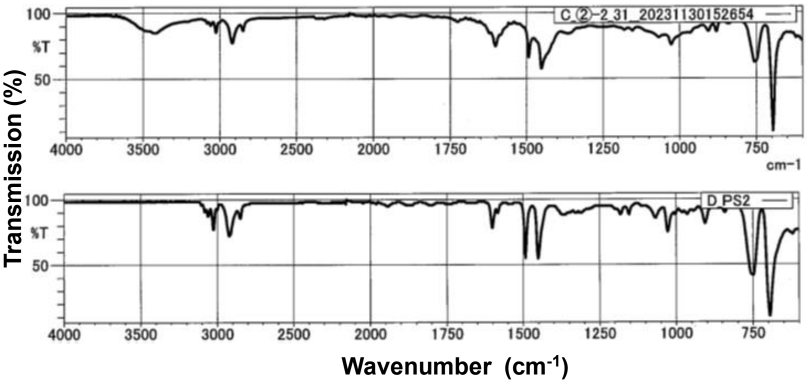
Identifying types of MPs with an FTIR

5. Sorted MPs and Identified Types of MPs

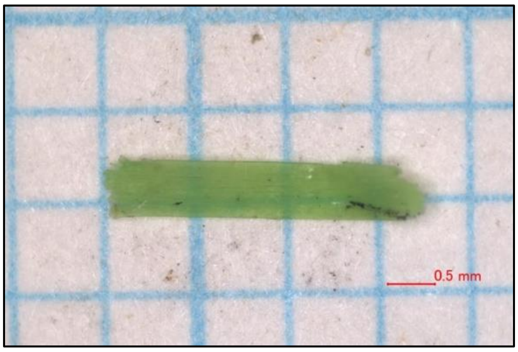
Stereomicroscopic photographs (left) and IR spectra (sample spectrum: top right, library spectrum: bottom right) of example MPs.

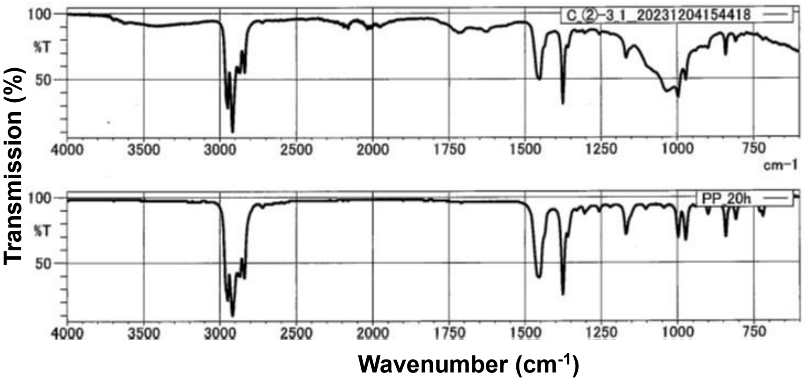
(1) Polystyrene (PS) Shape: Expanded



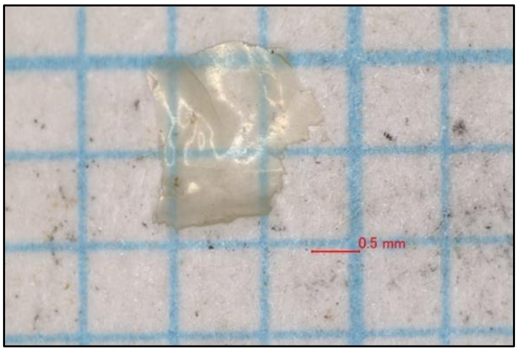


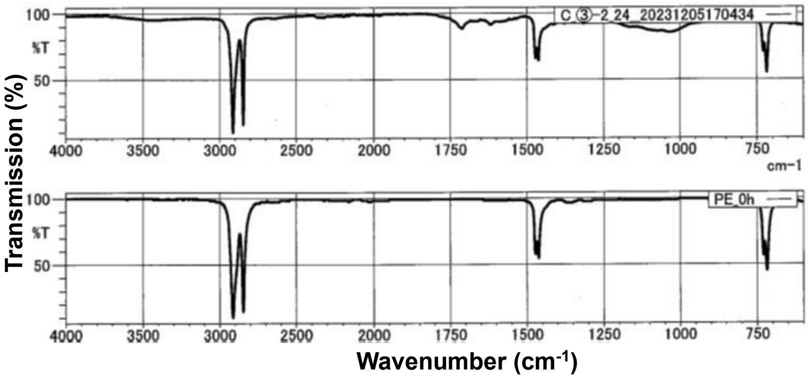
(2) Polypropylene (PP) Shape: Fragment



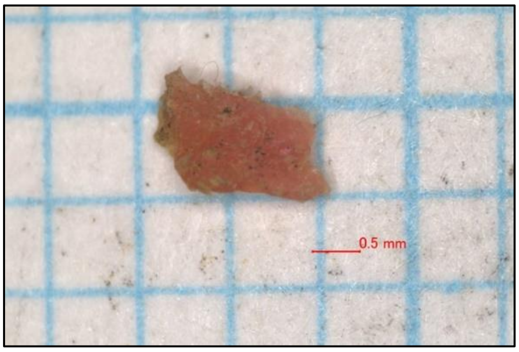


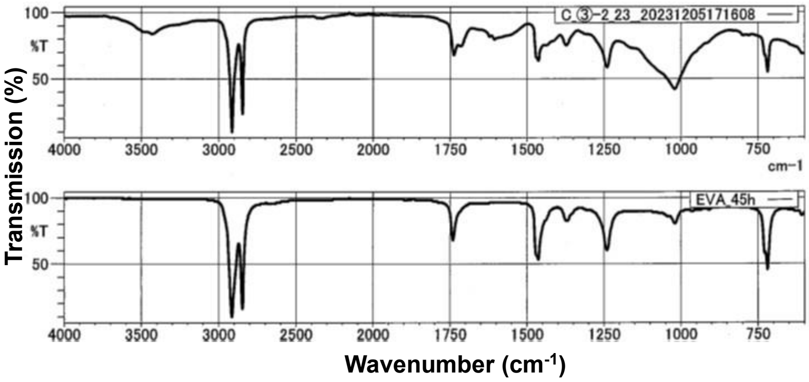
(3) Polyethylene (PE) Shape: Sheet



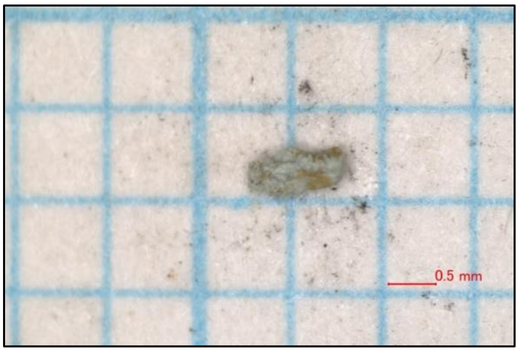


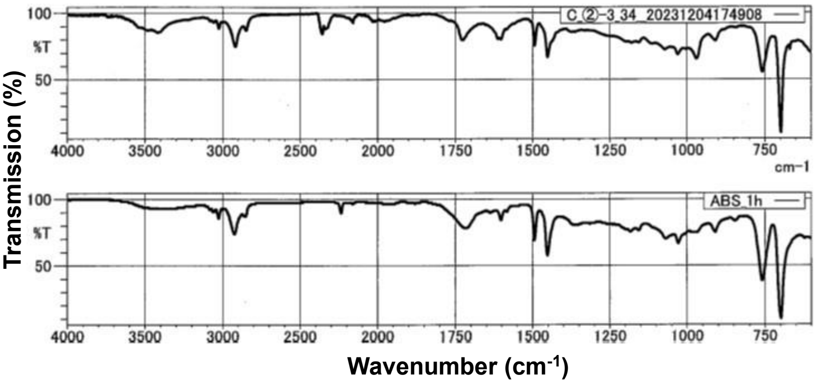
(4) Ethylene-vinyl acetate (EVA) Shape: Fragment



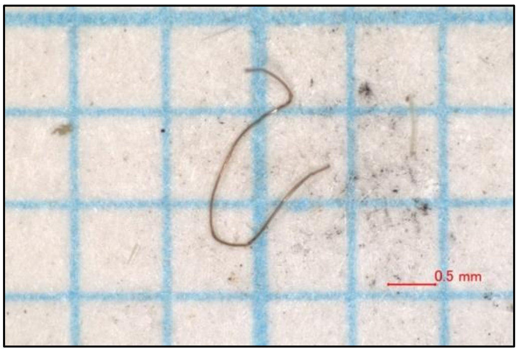


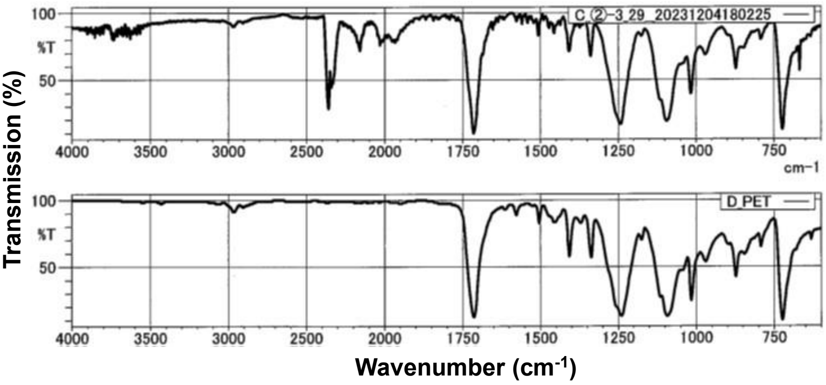
(5) Acrylonitrile butadiene styrene (ABS) Shape: Expanded





(6) Polyethylene terephthalate (PET) Shape: Fiber





6. Measuring Noise Levels

Measuring noise levels with an ordinary sound meter

7. Operating Conditions of Automatic Sample Preparation Device

Sampling Point		-	Point (1)			Point (2)			Point (3)		
Sample Name		Process blank	(1)-1	(1)-2	(1)-3	(2)-1	(2)-2	(2)-3	(3)-1	(3)-2	(3)-3
Start of Operation	**Start Date**	Aug. 29	Sep. 25	Oct. 2	Oct. 23	Oct. 16	Nov. 6	Nov. 13	Oct. 12	Nov. 27	Nov. 20
	Room Temp. (°C)	23.5	23.3	22.7	22.7	22.1	22.9	23.0	22.6	22.5	23.3
	Humidity (%)	68.0	58.0	41.0	34.0	46.0	67.0	21.0	46.0	31.0	26.0
End of Operation	**End Date**	Sep. 1	Sep. 29	Oct. 5	Oct. 26	Oct. 18	Nov. 9	Nov. 17	Oct. 16	Dec. 1	Nov. 23
	Room Temp. (°C)	23.1	23.1	22.8	22.5	23.0	22.5	22.7	23.8	23.0	22.4
	Humidity (%)	62.0	57.5	62.0	35.0	50.0	41.0	42.0	43.0	20.0	36.0
Operating Time (hr)		82	82	82	82	54	82	82	82	82	82
Oxidation Time (hr)		72	72	72	72	44*	72	72	72	72	72
Replacement of Decomposition Reagent		-	-	-	-	Implemented	-	Implemented	-	-	-
Power Consumption (kWh)		4.61	4.41	4.45	4.45	2.72	4.38	4.41	4.47	4.43	4.47

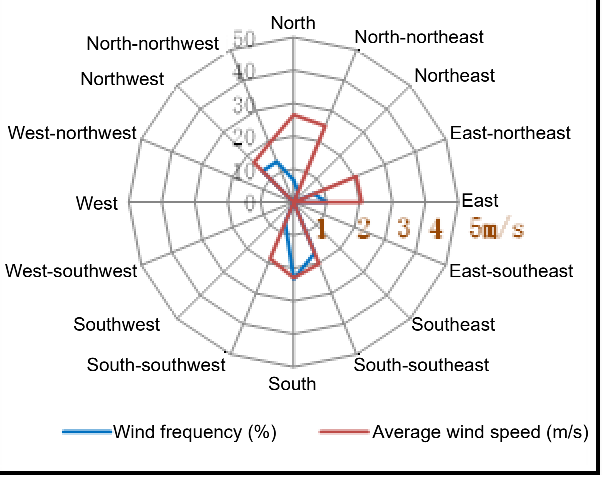
* Because the device (MAP-100) was controlled with an old version of the control software (Ver.1.0.0), the remaining processing time entered in the decomposition reagent replacement function (44 hours) was mistakenly used as the total oxidation time. The applicant has installed the latest version of the software (Ver.1.0.1) and normal operation has been confirmed since the latest version was installed.

8. Detailed Weather Conditions

Weather data observed at the AMeDAS※ station near sampling points data and wind rose
(▨: Sampling dates) AMeDAS station in Saitama city (Saitama prefecture)

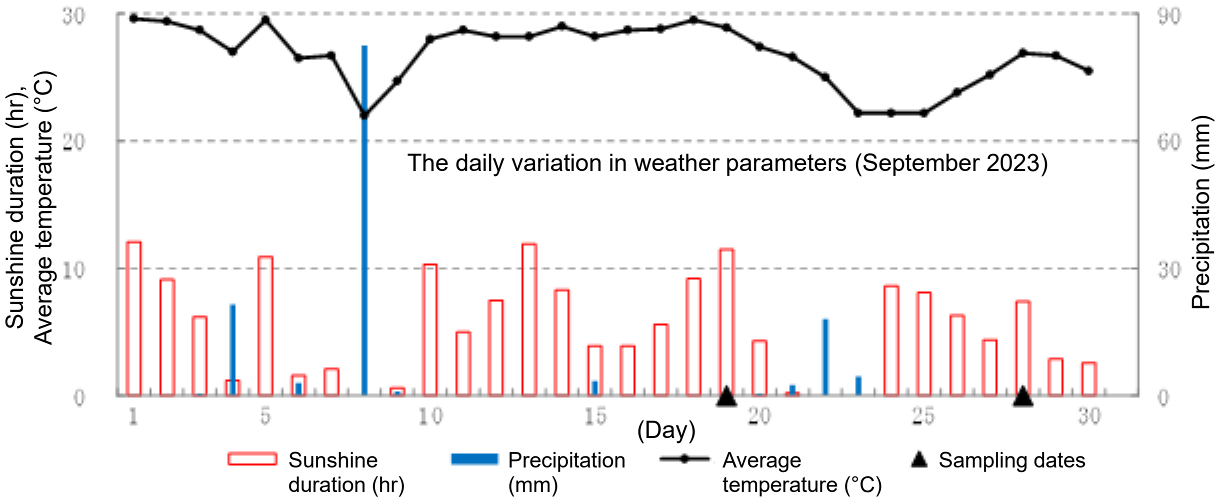
Day	September 2023							
	Precipitation (mm)	Average temperature (°C)	Sunshine duration (hr)	Average wind speed (m/s)	Most frequent wind direction	Wind direction	Appearance frequency of wind direction (%)	Average wind speed (m/s)
1	0.0	29.6	12.1	2.9	South	North	6.7	2.7
2	0.0	29.4	9.1	1.9	South-southwest	North-northeast	3.3	2.5
3	0.5	28.7	6.2	2.5	East-northeast	Northeast	0.0	0.0
4	21.5	27.0	1.2	2.4	North	East-northeast	6.7	2.1
5	0.0	29.5	10.9	2.3	South	East	10.0	2.1
6	3.0	26.5	1.6	1.6	North-northwest	East-southeast	0.0	0.0
7	0.0	26.7	2.1	2.3	East	Southeast	0.0	0.0
8	82.5	22.0	0.0	2.8	North-northwest	South-southeast	16.7	2.0
9	1.0	24.7	0.6	1.2	North-northwest	South	23.3	2.3
10	0.0	28.0	10.3	2.2	South	South-southwest	6.7	1.9
11	0.0	28.7	5.0	2.2	South-southeast	Southwest	0.0	0.0
12	0.0	28.2	7.5	1.8	South-southwest	West-southwest	0.0	0.0
13	0.0	28.2	11.9	1.7	South-southeast	West	0.0	0.0
14	0.0	29.0	8.3	1.8	South	West-northwest	0.0	0.0
15	3.5	28.2	3.9	1.6	Northwest	Northwest	13.3	1.7
16	0.0	28.7	3.9	1.6	South	North-northwest	13.3	1.9
17	0.0	28.8	5.6	2.2	South-southeast			
18	0.0	29.5	9.2	2.2	South			
19	0.0	28.9	11.5	1.9	South-southeast			
20	0.5	27.4	4.3	1.6	Northwest			
21	2.5	26.6	0.2	3.1	South			
22	18.0	25.0	0.0	2.2	East			
23	4.5	22.2	0.0	2.5	North-northeast			
24	0.0	22.2	8.6	2.9	North			
25	0.0	22.2	8.1	2.1	North-northwest			
26	0.0	23.8	6.3	1.6	Northwest			
27	0.0	25.2	4.4	2.0	Northwest			
28	0.0	26.9	7.4	1.6	East-northeast			
29	0.0	26.7	2.9	2.1	South-southeast			
30	0.0	25.5	2.6	1.7	East			

Wind rose (September 2023)



※Automated Meteorological Data Acquisition System

Sunshine duration: duration of direct solar radiation of 120 W/m^2 or higher
(intensity of direct radiation at which objects cast a noticeable shadow)



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