

Supplementary Information:

A HoLDI mass spectrometry platform for airborne nanoplastic detection

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Glossary of terms

ATR: Attenuated total reflectance

CPE: Chlorinated polyethylene

DSC: Differential scanning calorimetry

EDS: Energy-dispersive spectroscopy

HDPE: High-density polyethylene

HEPA: High efficiency particulate air

HoLDI: Hollow-laser desorption/ionization

HR-TEM: High-resolution transmission electron microscopy

LC: Liquid chromatography

μ FTIR: Micro-Fourier transform infrared spectroscopy

μ Raman: Micro-Raman spectroscopy

LDIR: Laser direct infrared

LDPE: Low-density polyethylene

MALDI-TOF-MS: Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry

MOUDI: Micro-orifice uniform deposit impactor

MS/MS: Tandem mass spectrometry

NTA: Nanoparticle tracking analyzer

OPS: Optical particle sizer

PA: Polyamide

PB: Polybutadiene

PC: Polycarbonate

PDMS: Polydimethylsiloxane

PE: Polyethylene

PEG: Polyethylene glycol

PES: Polyester

PET: Polyethylene terephthalate

PP: Polypropylene

PS: Polystyrene

PUR: Polyurethane

PVA: Polyvinyl alcohol

PVC: Polyvinyl chloride

PVDF: Polyvinylidene fluoride

Pyr-GC-MS: Pyrolysis gas chromatography mass spectrometry

QQQ-ICP-MS/MS: Triple quadrupole inductively coupled plasma tandem mass spectrometry

SMPS: Scanning mobility particle sizer

TD-PTR-MS: Thermal desorption proton-transfer-reaction mass spectrometry

TEM: Transmission electron microscopy

THF: Tetrahydrofuran

TOC: Total organic carbon

UV: Ultraviolet

Table S1 | A comparison of selected environmental nano/microplastic research carried out using various sample collection approaches, preparation methods, and major analytical techniques.

Environmental matrix and sampling site			Sample collection	Sample preparation	Analytical technique	Plastic detected	Ref.
Air	Suspended aerosol	Within and above the planetary boundary layer over urban and rural areas, Spain	Onto stainless-steel filters by a vacuum pump, on an airplane	Not applied	μ -FTIR	PA, PB, PE, PES, PP, PUR, and acrylic; 1.5 particles·m ⁻³ (rural) - 13.9 particles·m ⁻³ (urban)	[1]
		Marine atmosphere, North Atlantic Ocean	Onto polycarbonate filters by a vacuum pump	Not applied	μ -Raman	PDMS, PE, PP, and PS; ~ 0.01 - 0.1 particles·m ⁻³	[2]
	Dry deposition	Domestic indoor dust, Netherlands	Into glass bottles from vacuum cleaner	In-matrix depolymerization and filtration	LC-UV	PET; 1.2 - 305 mg·g ⁻¹	[3]
		Outdoor dust from windowsills and open-air balconies, major cities, China	Into paper bags with aluminium foil lining, using hog bristle brushes	Visual sorting, sieving, depolymerization, extraction, spiking, solid-phase extraction, and re-dissolving	LC-MS/MS	PC (2.0 mg·kg ⁻¹) and PET (212 - 9020 mg·kg ⁻¹)	[4]
	Wet deposition	Rainwater in an urban area, China	Into glass containers	Filtration, ultrasonication, digestion, and evaporation	μ -Raman	PS and PVC; 0.28 % - 0.40 %	[5]
		Fresh and aged snow, urban area, Canada	Into glass jars with a scope	Melting and homogenization	MALDI-TOF-MS	PE (> 100 ng·mL ⁻¹) and PEG (21.9 ng·mL ⁻¹)	[6]
Water	River	Surface water, Rhine River	Via a manta net from vessels	Rinsing, sieving, digestion, density separation, and visual sorting	ATR-FTIR	PES, PP, PS, PVC, and acrylate; 892777 particles·km ⁻²	[7]
		Gallatin River watershed, USA	Into stainless-steel bottles using a grab	Filtration, drying, visual inspection, and hot needle test	μ -FTIR	PA, PES, PET, PP, PVA, PVDF, poly(trimellitamide	[8]

			approach; or wading and using a sampling pole			imide), acrylic, butyl rubber, neoprene, and semi-synthetic cellulose; 1.2 particles·L ⁻¹	
Lake		Surface water, western Lake Superior	Via a manta net and a collection vessel	Sieving, washing, digestion, density separation, filtration, and drying	Pyr-GC-MS and ATR-FTIR	PE, PET, PDMS, PP, PS, PVC, CPE, and dodecyl phthalate resin; 91 - 3538 mg·km ⁻² (0 - 110000 particles·km ⁻²)	[9]
		Surface water, lake Gårdsjön, Sweden	Into glass bottles by hand	Filtration	TD-PTR-MS	PE, PET, PP, and PVC; 563 µg·L ⁻¹	[10]
Ocean		Surface layer seawater, subtropical gyre, western North Atlantic Ocean	Manta nets and glass bottles, on a sailing vessel	Agitation, digestion, filtration, ultrafiltration, freeze-drying, and homogenization	Pyr-GC-MS and µ-FTIR	PE, PET, PP, PS, and PVC; 500000 - 7000000 particles·km ⁻²	[11]
		The southernmost of the Tyrrhenian Sea	By a Bongo net	Visual sorting, washing, and centrifugation	Raman and ATR-FTIR	LDPE and PVC	[12]
Ground-water		Unconfined groundwater aquifer, Bacchus Marsh, Australia	Into glass bottles, by a stainless-steel bailer with a braided polyamide rope	Filtration, digestion, and density separation	LDIR	PA, PC, PE, PET, PMMA, PP, PS, and PVC; 16 - 97 particles·L ⁻¹	[13]
		Karst aquifers, Illinois, USA	Into HDPE bottles and glass bottles	Filtration, drying, and visual inspection	Pyr-GC-MS	PE; 0 - 15.2 particles·L ⁻¹	[14]
Soil and sediment	Industrial soil	Soil near an industrial site, Australia	Not specified	Drying, sieving, pressurized fluid extraction, and evaporation	ATR-FTIR	PE, PS, and PVC; 0.03% - 6.7%	[15]

		Soil of Ahvaz metropolis, Iran	Into glass jars by a stainless-steel shovel	Drying, manual sorting, sieving, digestion, and floatation	μ -Raman	PP, PS, PET, and nylon; 80 - 1220 pieces·kg ⁻¹	[16]
Agricultural field		Agricultural soil, Chilean central valley, Chile	Collected using a metallic soil auger and stored in PP plastic bags	Drying, sieving, and wet extraction (i.e., floatation, stirring, centrifugation, and filtration cycles)	Stereo microscope (visual inspection)	Fibers, films, pellets, and fragments; 0.6 - 10.4 pieces·g ⁻¹	[17]
		Highly contaminated agricultural soil, central France	Not specified	Drying, sieving, water extraction, filtration, and fractionation	Pyr-GC-MS	PE, PS, and PVC	[18]
Sand		Beach sand from El Quetzalito Beach, Guatemala	Into aluminum trays using a stainless-steel spatula	Drying, sieving, and density separation	ATR-FTIR	PE, PP, and PS; 279 pieces·m ⁻² (30 pieces·kg ⁻¹)	[19]
		Beach sand along the South Korean coast, Korea	Scooped and sieved into PE bottles	Sieving, homogenization, extraction, density separation, drying, and digestion	μ -FTIR	PE, PES, PP, PS, polyether urethane, poly(vinyls), poly(lauryl acrylate), and phenoxy resin; 1400 - 62800 pieces·m ⁻²	[20]
Sediment		Upper River Tame catchment, UK	Collected using a stainless-steel scoop	Wet sieving, rinsing, drying, floatation, and visual sorting	ATR-FTIR	PE, PVC, and acrylic; 165 particles·kg ⁻¹	[21]
		St. Lawrence River, Canada	Collected using a petite Ponar grab and a single Peterson grab	Sieving	Stereo microscope (visual inspection) and DSC	PE microbeads; 13832 microbeads·m ⁻²	[22]

Table S2 | 3D-printed matrix-assisted laser desorption/ionization (MALDI) target plates for mass spectrometry analysis of various analytes.

Description	Configuration adopted	Printing material	Analyte	Ref.
96 sample spots, with a 10 μ L reservoir	MBT Biotarget 96 (Bruker Daltonics)	Conductive acrylonitrile butadiene styrene with carbon particles	Proteins and peptides	[23]
96 sample spots, matrix-free	MSP 96 Target (Bruker Daltonics)	Graphene-doped photocurable resin	Low-mass environmental pollutants and biological molecules	[24]
Hollow configurations, in combination with substrates	Prespotted AnchorChip 96 and MSP 96 NALDI Target (Bruker Daltonics)	Electrically conductive polylactic acid, can be replaced by other materials	Environmental nano- and microplastics	This work

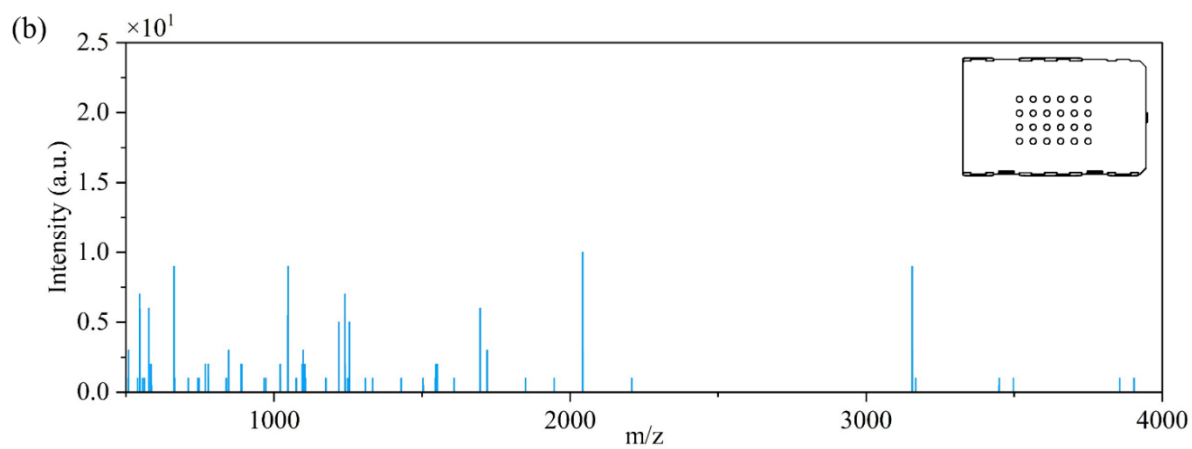
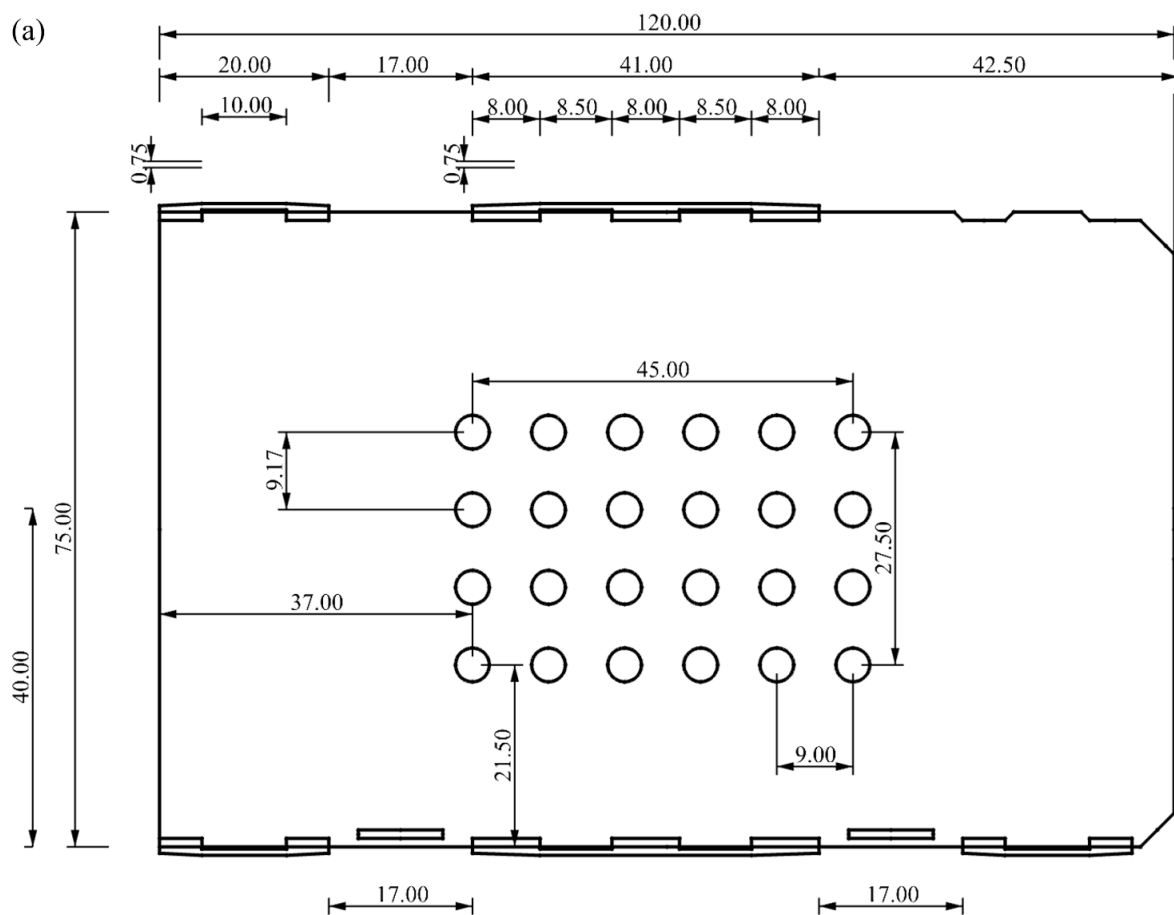
Table S3 | Conductivity measurements of different MALDI plates, including the 3D-printed HoLDI plate developed in this work, Bruker Daltonics's Prespotted AnchorChip 96 target made from conductive polymer, and Bruker Daltonics's stainless-steel MSP 96 NALDI target. Conductivity is calculated using Equation (1) in Methods, with sheet resistance measured via a four-point probe.

	HoLDI Target Plate	Prespotted AnchorChip 96 Target (Bruker Daltonics)	MSP 96 NALDI Target (Bruker Daltonics)
Sheet resistance ($\Omega \cdot \text{sq}^{-1}$)	2.27×10^2	4.93×10^1	2.97×10^0
Thickness (m)	8.50×10^{-4}	1.42×10^{-3}	8.50×10^{-4}
Conductivity ($\text{S} \cdot \text{m}^{-1}$)	5.40×10^0	1.80×10^1	4.13×10^2

Table S4 | Metal analysis of nano-sized airborne particles collected in (a) an indoor public space and (b) an outdoor environment. Concentrations are derived from reverse calculations based on Equation (4) in the Methods section, representing the mass of metals in the total volume of air passed through each stage of the impactor. The values are reported in ppb, with standard deviations in parentheses. Measurements < 0.000 ppb by ICP-MS prior to reverse calculations are denoted as “ND” (not detected).

(a)	Stage 6 – 1.0 μm	Stage 7 – 0.56 μm	Stage 8 – 0.31 μm	Stage 9 – 0.18 μm
Na	5.78×10^{-7} (6.42×10^{-8})	5.89×10^{-7} (1.89×10^{-8})	5.56×10^{-7} (2.45×10^{-8})	5.78×10^{-7} (2.49×10^{-8})
Al	ND	ND	ND	ND
K	ND	ND	ND	ND
V	2.34×10^{-6} (2.34×10^{-9})	2.36×10^{-6} (7.06×10^{-9})	2.34×10^{-6} (2.34×10^{-9})	2.34×10^{-6} (2.34×10^{-9})
Cr	ND	ND	ND	ND
Mn	ND	ND	ND	ND
Fe	ND	ND	ND	ND
Ni	1.96×10^{-5} (4.10×10^{-7})	1.15×10^{-5} (2.64×10^{-7})	1.25×10^{-5} (8.72×10^{-8})	1.18×10^{-5} (3.78×10^{-7})
Zn	ND	ND	ND	ND
As	5.78×10^{-7} (5.20×10^{-9})	4.67×10^{-7} (3.13×10^{-8})	4.33×10^{-7} (1.74×10^{-8})	4.33×10^{-7} (1.47×10^{-8})
Se	1.89×10^{-7} (6.34×10^{-8})	2.11×10^{-7} (9.36×10^{-8})	1.89×10^{-7} (5.25×10^{-8})	2.00×10^{-7} (1.15×10^{-7})
Ba	ND	ND	2.22×10^{-7} (5.34×10^{-8})	ND

(b)	Stage 6 (1.0 μm)	Stage 7 (0.56 μm)	Stage 8 (0.31 μm)	Stage 9 (0.18 μm)
Na	2.41×10^{-7} (1.62×10^{-8})	2.70×10^{-7} (1.43×10^{-8})	2.67×10^{-7} (1.70×10^{-8})	3.11×10^{-7} (1.58×10^{-8})
Al	1.76×10^{-6} (1.05×10^{-6})	3.62×10^{-6} (5.67×10^{-7})	2.65×10^{-6} (3.10×10^{-7})	ND
K	ND	ND	7.41×10^{-9} (9.36×10^{-10})	3.70×10^{-9} (1.18×10^{-9})
V	8.04×10^{-7} (1.61×10^{-9})	8.15×10^{-7} (1.63×10^{-9})	1.11×10^{-6} (1.11×10^{-8})	8.44×10^{-7} (5.06×10^{-9})
Cr	2.22×10^{-8} (1.14×10^{-8})	1.11×10^{-7} (2.91×10^{-8})	4.17×10^{-6} (5.83×10^{-8})	1.25×10^{-6} (1.78×10^{-7})
Mn	1.39×10^{-5} (2.64×10^{-7})	ND	ND	ND
Fe	ND	ND	8.15×10^{-8} (1.16×10^{-8})	ND
Ni	4.26×10^{-6} (2.38×10^{-7})	4.58×10^{-6} (5.04×10^{-8})	5.12×10^{-6} (7.68×10^{-8})	4.36×10^{-6} (9.60×10^{-8})
Zn	6.69×10^{-6} (2.74×10^{-7})	1.98×10^{-6} (1.09×10^{-7})	2.16×10^{-5} (3.02×10^{-7})	1.44×10^{-7} (1.33×10^{-7})
As	ND	ND	ND	ND
Se	1.33×10^{-7} (4.74×10^{-8})	3.70×10^{-8} (2.35×10^{-8})	1.41×10^{-7} (9.84×10^{-9})	5.56×10^{-8} (3.00×10^{-8})
Ba	3.85×10^{-7} (1.15×10^{-8})	3.41×10^{-7} (6.00×10^{-8})	ND	3.74×10^{-7} (1.54×10^{-8})



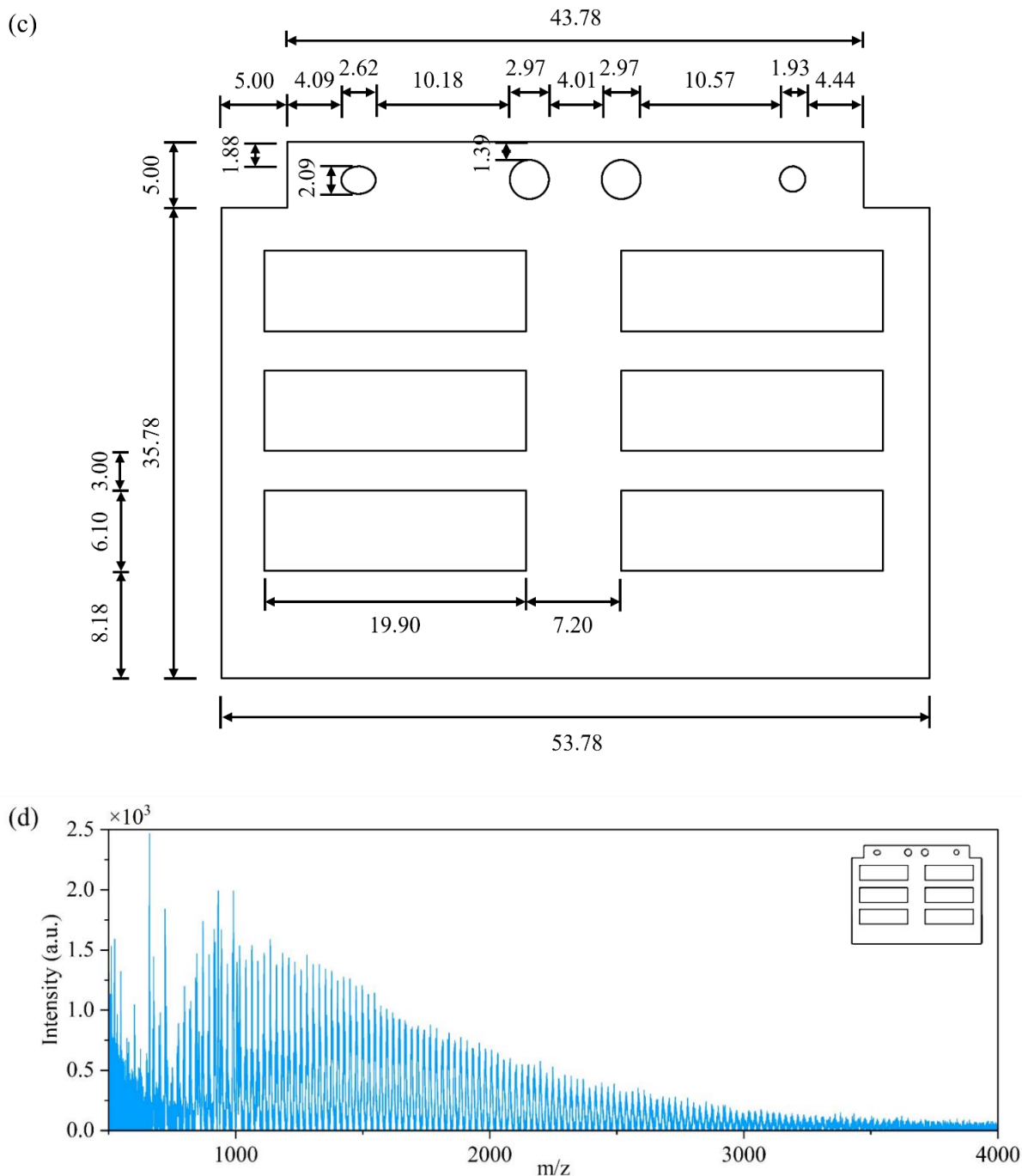


Figure S1 | Configurations of different 3D-printed HoLDI targets and corresponding mass spectra of the same aerosol sample acquired in pre-testing. (a) The first version prototype features an array of circular structures, resulting in weak intensity signals (magnitude = 10^1) as shown in (b). The size and shape of the plate are based on the Prespotted AnchorChip 96 target from Bruker Daltonics. (c) The second version includes larger rectangular windows instead of round hollows, producing improved signals (magnitude = 10^3) as presented in (d). The shape and size of the second plate are based on MSP 96 NALDI Target from Bruker Daltonics. Dimensions in (a) and (c) are labeled in millimeters (mm).

(a)

Increasing signal intensity ↑

Substrate	Ion-pairing agent	Matrix	Order of treatment
PTFE	No salt	No matrix	No treatment
PVDF	AgNO ₃	ZnO	Treatment after sampling
Glass	AgTFA	CHCA	Treatment before sampling
	NaTFA	DHB	
	NaI	Dithranol	

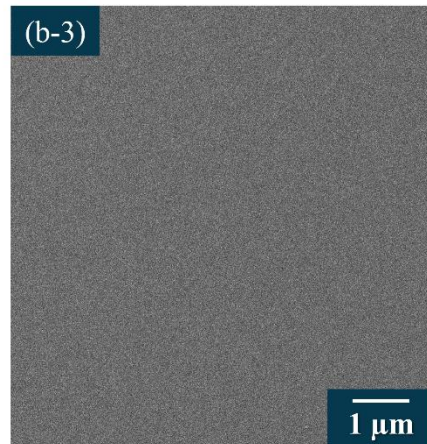
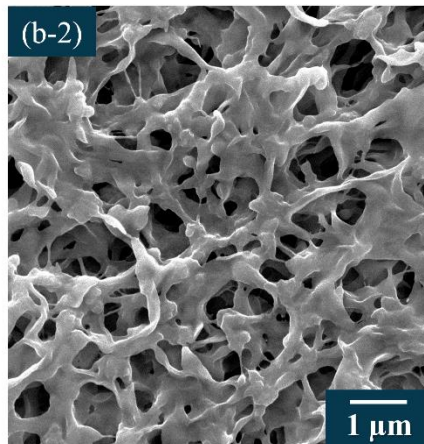
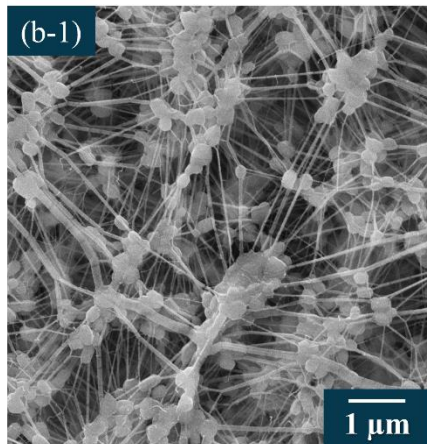


Figure S2 | Distinct analysis efficiencies for the use of different substrates, ion-pairing agents, matrices, and treatment processes. Analysis efficiencies are expressed as relative signal intensities. (a) The efficiencies in descending order for: i) Substrates, with polytetrafluoroethylene (PTFE) membrane > polyvinylidene fluoride (PVDF) membrane > glass; ii) Ion-pairing agents, with no salt > silver nitrate > silver trifluoroacetate (AgTFA) > sodium trifluoroacetate (NaTFA) > sodium iodide; iii) Matrices, with no matrix > zinc oxide nanoparticles > α -cyano-4-hydroxycinnamic acid (CHCA) > 2,3-dihydroxybenzoic acid (DHB) > dithranol; and iv) Treatment processes, with no treatment > applying matrices and salts on top of collected aerosol samples > depositing matrices and salts on blank substrates prior to sampling. (b-1) – (b-3) The surface structures of the PTFE membrane, PVDF membrane, and glass chip, respectively, acquired using scanning electron microscopy (SEM) with 10000× magnification.

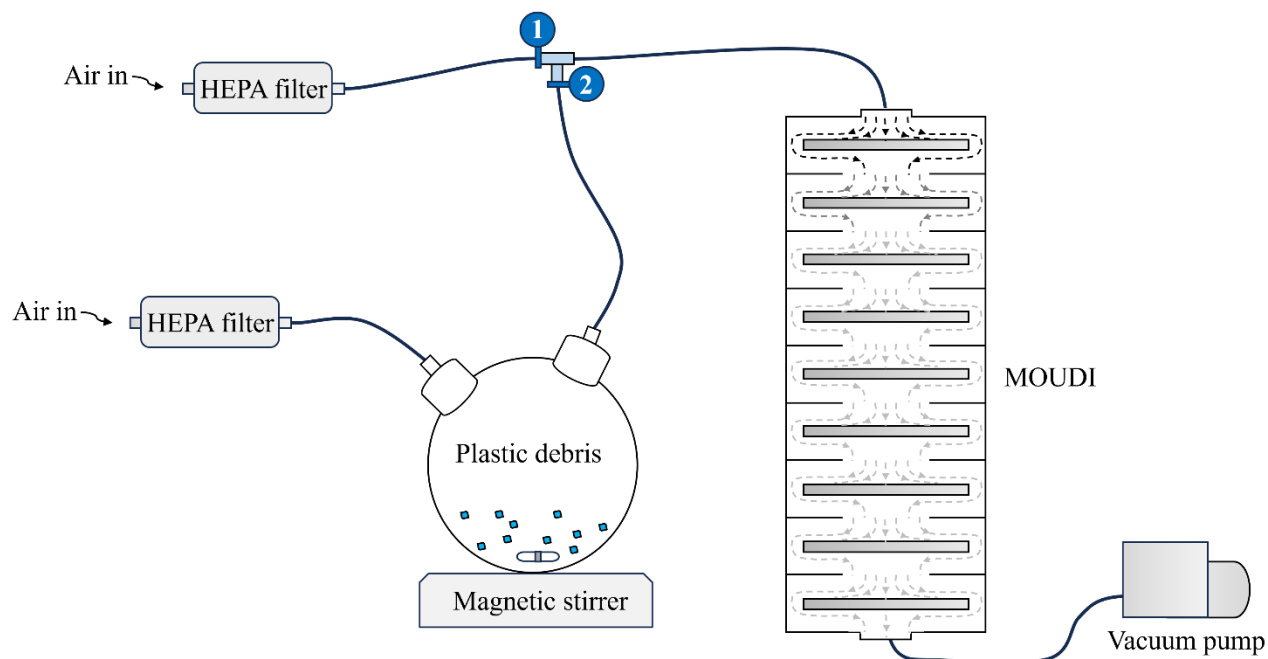


Figure S3 | The aerosol sampling setup for experiment blanks and positive controls. Collected air samples are analyzed using MALDI mass spectrometry to demonstrate the feasibility of the HoLDI target for airborne nano/microplastic detection. Air is passed through a micro-orifice uniform deposit impactor (MOUDI) using a vacuum pump and collected on substrates clamped onto MOUDI stages. For blanks, only valve 1 is open, allowing air in a cleanroom to be pumped into the sampling system, passed through a high efficiency particulate air (HEPA) filter unit, and collected on substrates by MOUDI. For spiked experiments, both valve 1 and valve 2 are open, allowing filtered cleanroom air to aerosolize micro/nanoplastic particles in a glass chamber and deposit airborne micro/nanoplastics onto substrates in MOUDI. Magnetic stirring aids in the generation and aerosolization of nano- and microplastic particles. The figure is not drawn to scale.

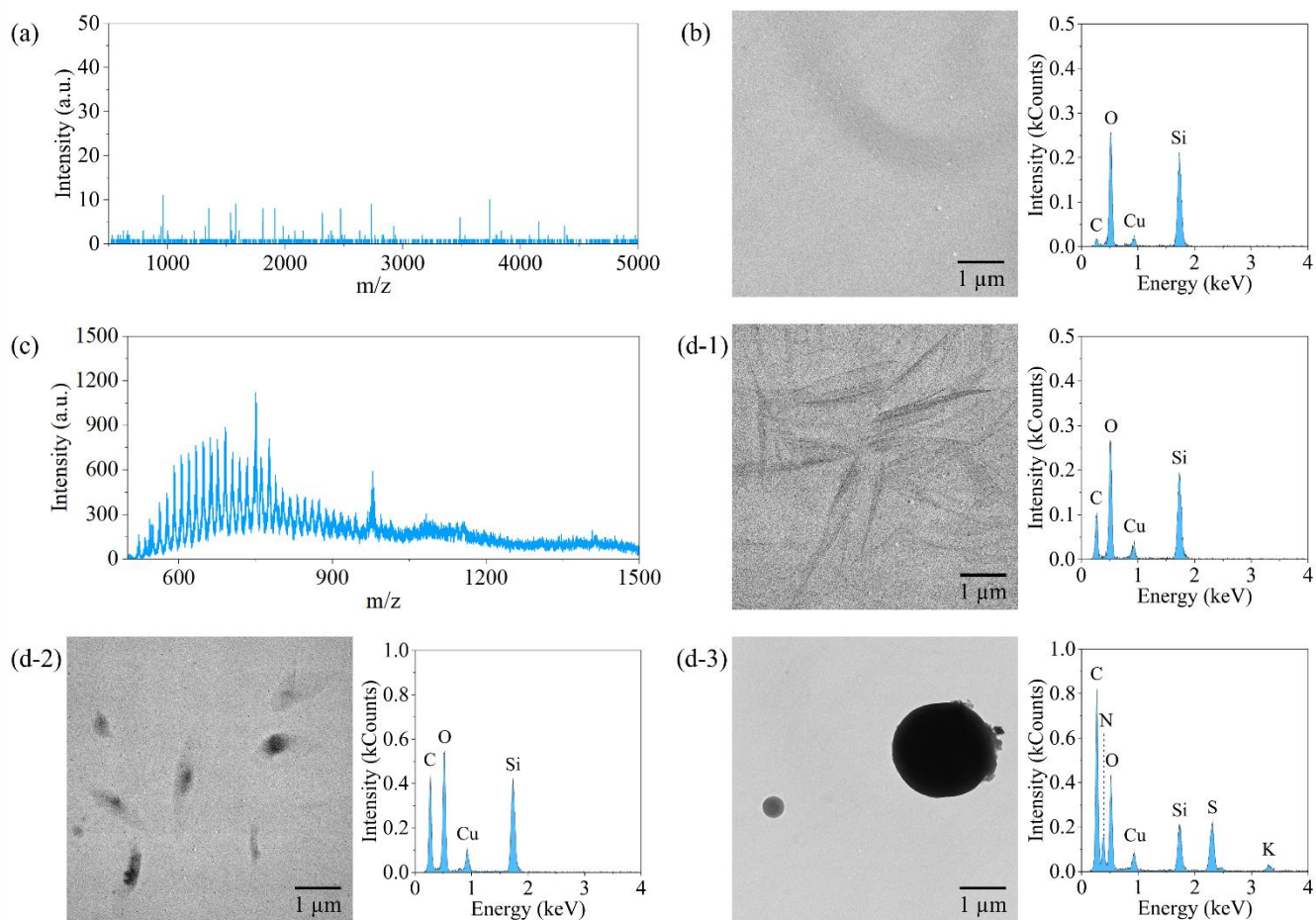
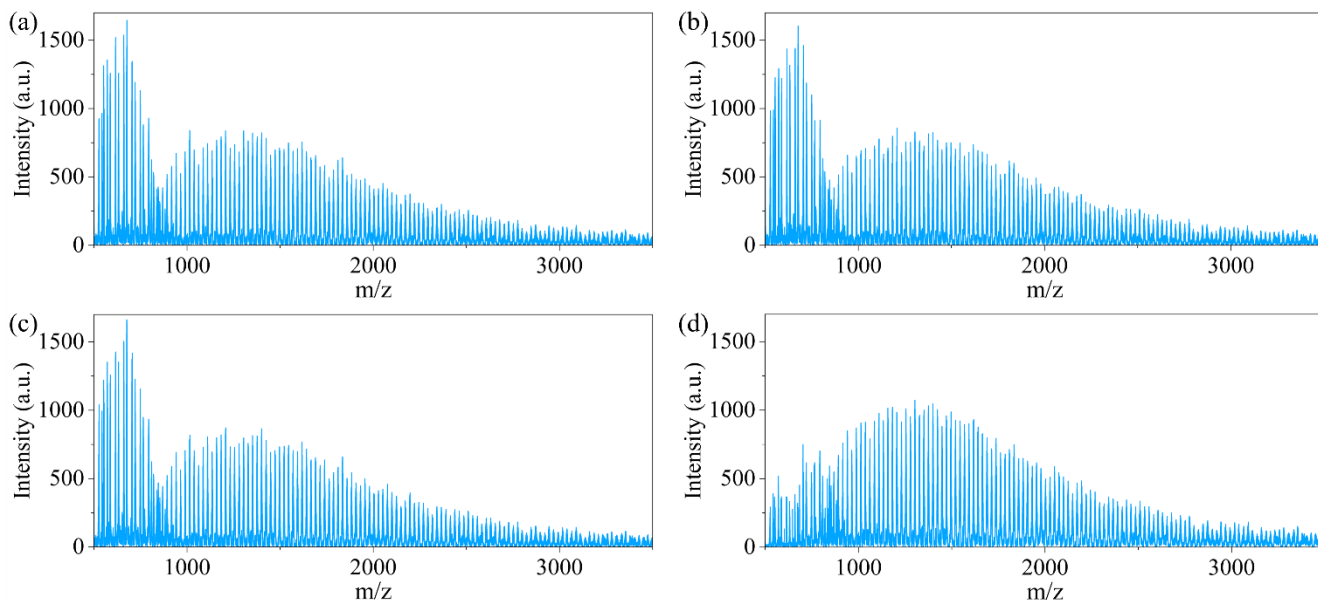


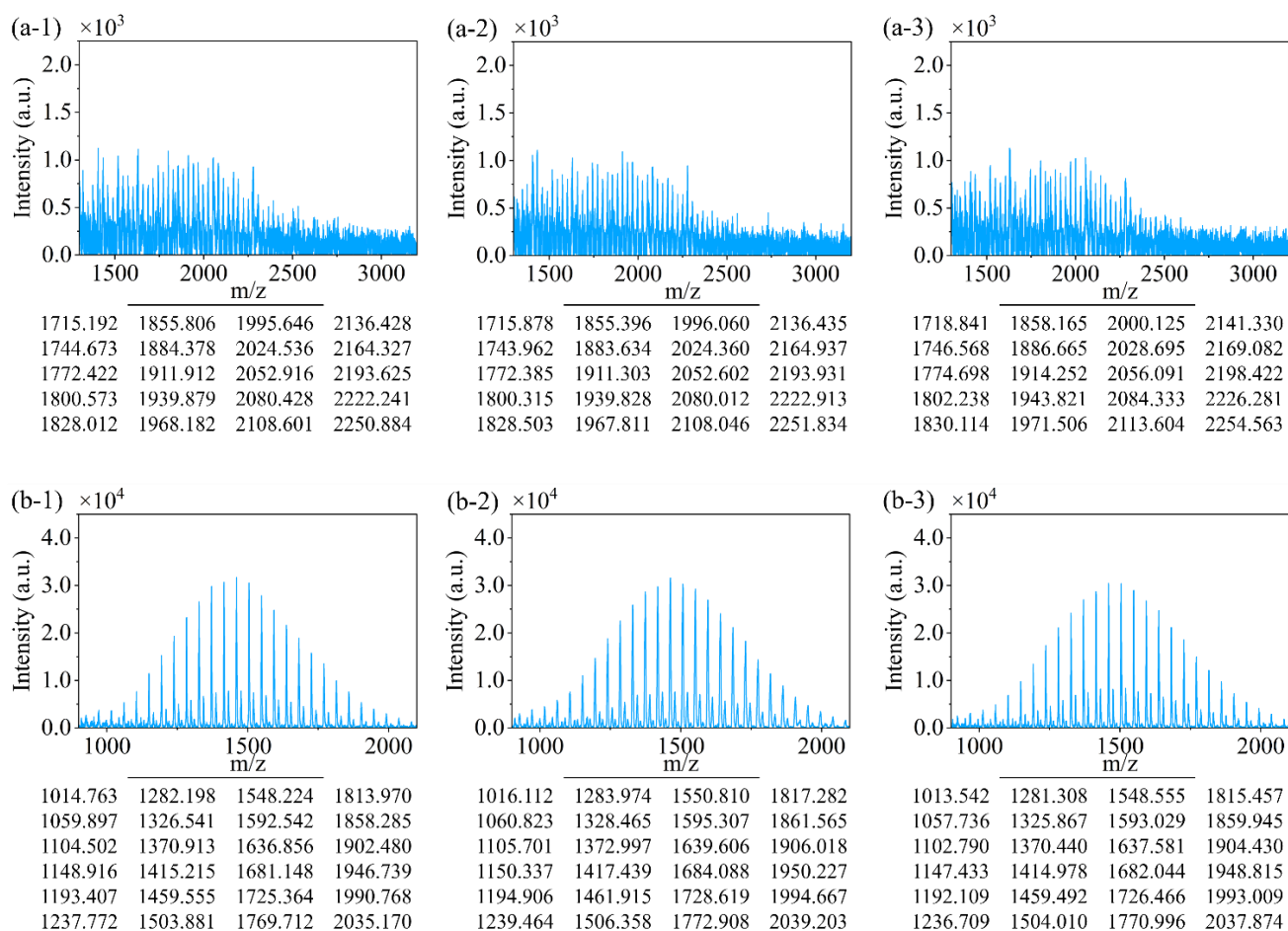
Figure S4 | Mass spectrometry (MS), transmission electron microscopy (TEM), and energy-dispersive X-ray spectroscopy (EDS) results of spiked experiments and blanks. (a) MS and (b) TEM-EDS data of one of the procedural blanks, showing no signals of plastic particles. (c) The mass spectrum of clean air spiked with PE form aerosols collected during positive controls, presenting signals of PE with mass differences of 28 Da. (d-1) – (d-3) TEM images of aerosols generated from PE forms, displaying three types of dominant structures. Elemental compositions of the generated aerosols exhibit increased carbon signal intensities, corresponding to plastic carbon backbones.



(e)	PEG, Na ⁺		PEG, K ⁺		PAHs							
	m/z	Intensity (a.u.)	m/z	Intensity (a.u.)	m/z	Intensity (a.u.)	m/z	Intensity (a.u.)	m/z	Intensity (a.u.)	m/z	Intensity (a.u.)
Replicate (a)	528.222	903.301	544.237	966.493	1182.906	794.639	1352.073	820.720	1520.955	700.399	1690.651	656.640
	572.006	1355.462	588.001	1258.832	1207.015	836.314	1376.986	793.043	1545.149	748.465	1714.009	573.196
	616.497	1518.894	632.799	1257.642	1232.147	771.282	1401.008	822.201	1569.806	688.088	1739.263	583.583
	661.012	1536.796	677.105	1637.339	1255.950	709.526	1425.095	782.847	1593.288	706.016	1763.209	495.974
	705.391	1345.033	721.496	1161.269	1280.609	675.189	1448.760	660.361	1617.872	755.688	1786.434	550.205
	748.978	1131.116	765.860	880.584	1304.609	816.059	1472.628	674.733	1641.672	674.050	1811.019	623.357
	794.884	889.554	809.648	595.125	1328.880	748.184	1496.651	712.076	1665.181	640.194	1835.351	639.657
Replicate (b)	527.887	983.221	543.570	871.370	1183.204	797.675	1353.005	725.008	1521.043	703.617	1690.995	665.416
	572.887	1289.494	588.535	1220.870	1207.320	823.328	1377.040	814.886	1545.848	747.270	1715.325	555.218
	616.416	1435.170	632.253	1316.831	1232.073	676.388	1400.260	823.847	1568.924	642.601	1739.332	554.809
	660.525	1430.602	676.621	1605.107	1255.428	731.619	1425.123	761.661	1594.202	680.381	1763.358	517.710
	705.323	1406.951	720.910	1110.351	1280.259	752.806	1449.679	682.059	1617.731	736.626	1787.596	521.610
	749.122	1039.359	765.983	911.767	1304.446	828.474	1472.920	749.748	1642.207	693.847	1810.680	617.634
	794.746	914.131	809.362	622.506	1328.011	750.106	1497.033	750.103	1665.669	682.075	1835.319	596.800
Replicate (c)	527.999	1023.776	544.282	994.828	1183.216	818.772	1351.741	814.245	1521.848	737.118	1690.134	653.883
	572.088	1352.501	588.292	1257.407	1207.458	857.787	1376.193	812.297	1545.701	730.243	1714.389	595.678
	617.216	1425.999	632.538	1351.433	1231.806	732.512	1400.438	864.008	1569.809	705.702	1738.685	635.452
	661.246	1495.885	677.001	1629.979	1255.083	726.747	1425.524	780.398	1593.962	696.643	1763.477	498.245
	705.680	1416.775	721.680	1225.484	1280.292	756.181	1449.066	692.135	1617.382	767.736	1787.199	544.420
	749.576	1155.190	765.309	937.378	1304.660	793.130	1472.400	705.286	1641.366	715.925	1811.610	584.261
	794.056	934.226	809.788	623.387	1327.873	758.032	1496.226	732.389	1666.885	636.670	1835.404	653.230
Average integrated	1230.259 ± 18.199	1097.569 ± 20.872	695.998 ± 6.375									

intensity (a.u.)			
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Figure S5 | The reproducibility of HoLDI in analyzing real-world aerosol samples and a comparison between HoLDI and MALDI results. (a) – (c) Mass spectra of three separate subsamples of the same real-world aerosol sample obtained from the same filter substrate, analyzed using HoLDI-TOF-MS. Signals corresponding to sodium and potassium adducted polyethylene glycols (PEGs) and polycyclic aromatic hydrocarbons (PAHs) are consistently observed across all three spectra within the same mass range and with comparable signal intensities. (d) The mass spectrum of a subsample of the same real-world aerosol sample analyzed in (a) – (c), also taken from the same filter substrate, acquired using conventional MALDI-TOF-MS. PEG peaks are largely weakened in the MALDI spectrum. Tabulated in (e) are the m/z and signal intensity data for the detected PEGs and PAHs in the three subsamples, along with the calculated average integrated intensity and standard deviation values for the corresponding contaminants.



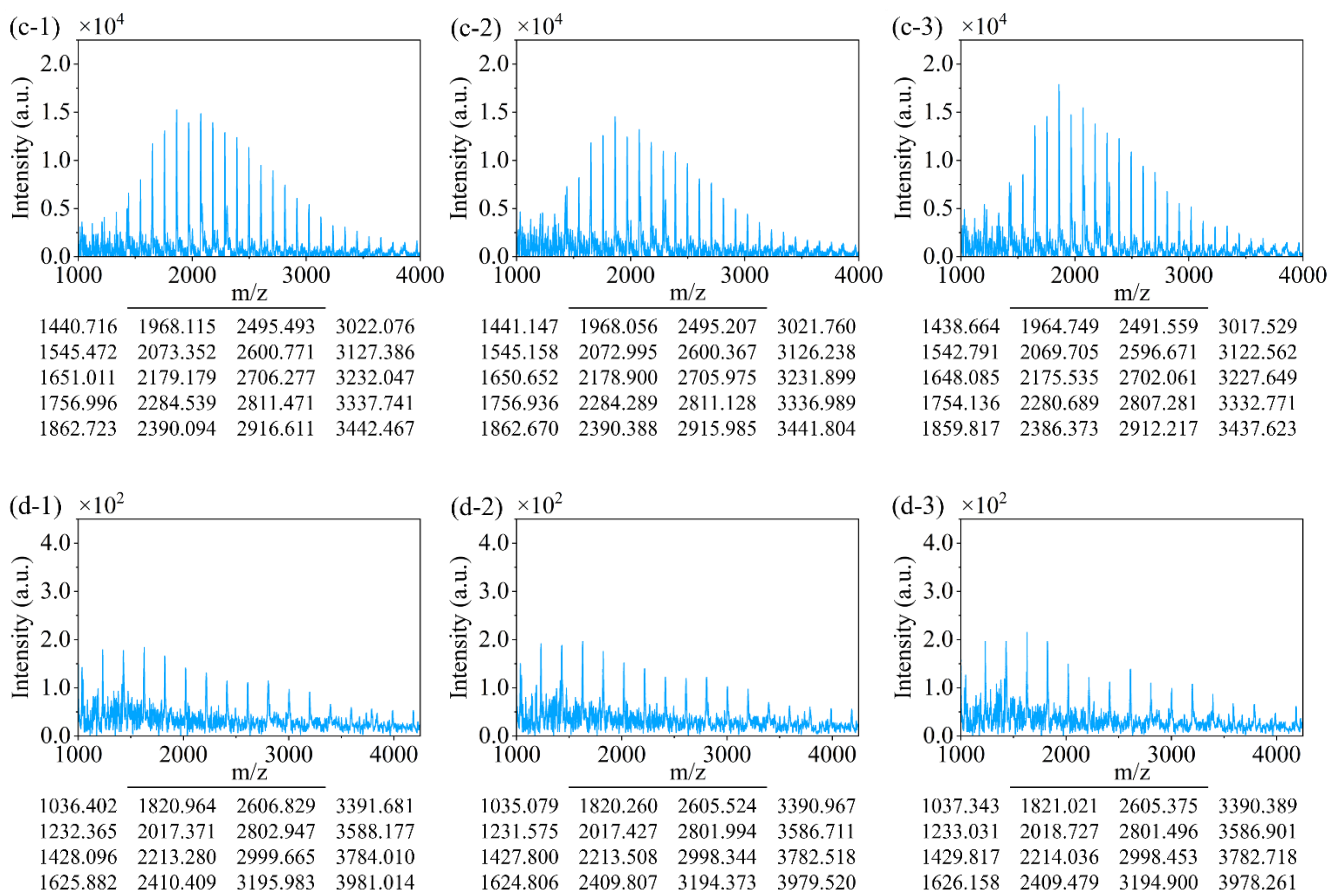
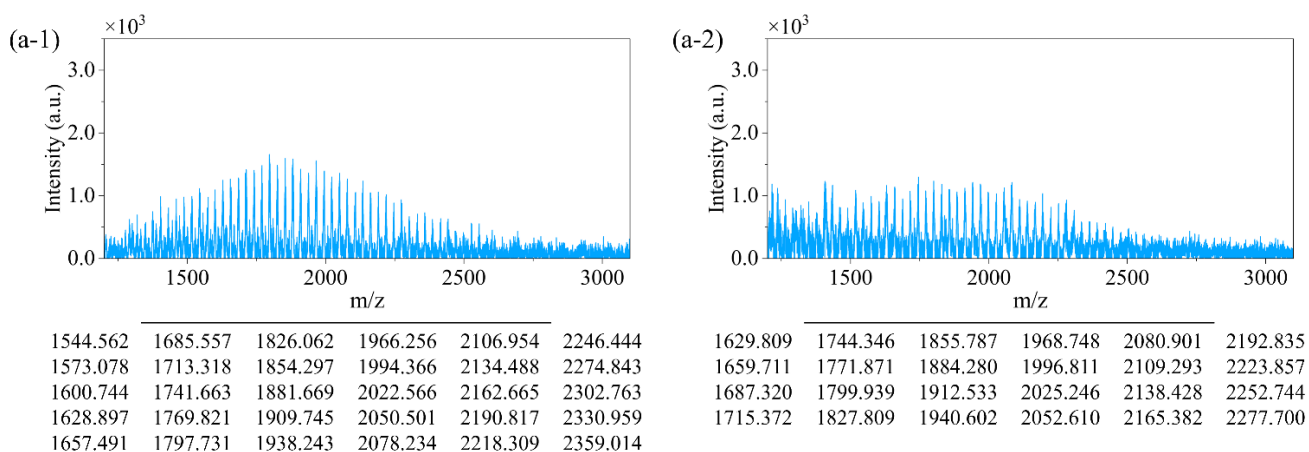


Figure S6 | The reproducibility of HoLDI in analyzing selected nano/microplastic standards spiked into real-world aerosol samples, including (a) polyethylene (PE), (b) polyethylene glycol (PEG), (c) polystyrene (PS), and (d) polyethylene terephthalate (PET).



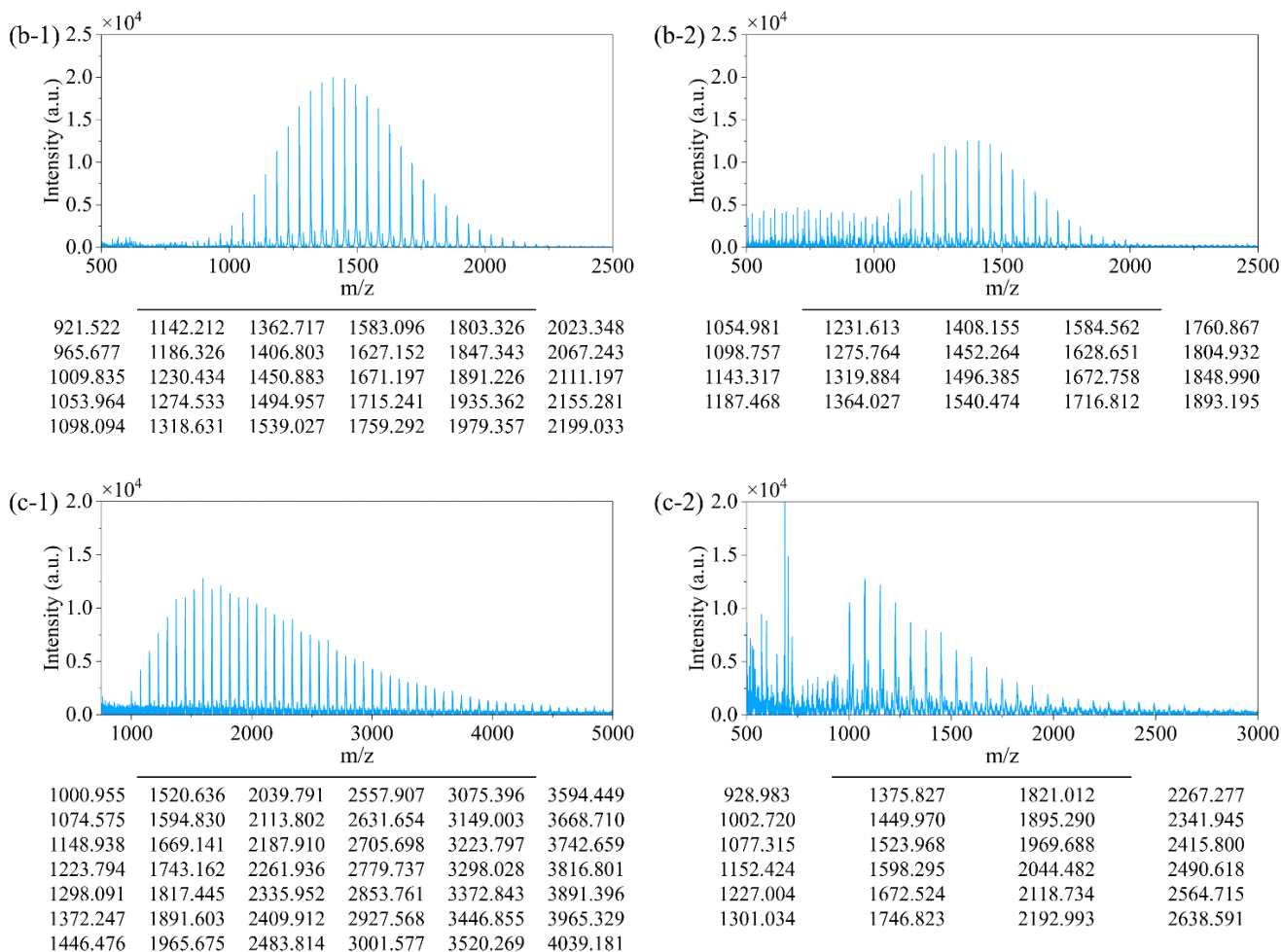


Figure S7 | Examination of potential m/z shifts in plastic signals in the presence of complex airborne particles. (a-1), (b-1), and (c-1) present the mass spectra of polyethylene (PE), polyethylene glycol (PEG), and polydimethylsiloxane (PDMS) standards analyzed using conventional MALDI with a stainless-steel target plate. (a-2), (b-2), and (c-2) present the mass spectra of PE, PEG, and PDMS standards spiked onto substrates containing real-world aerosol samples, analyzed using HoLDI. Characteristic polymer peaks are tabulated below the mass spectra. Only the m/z values of the dominant peak series are listed.

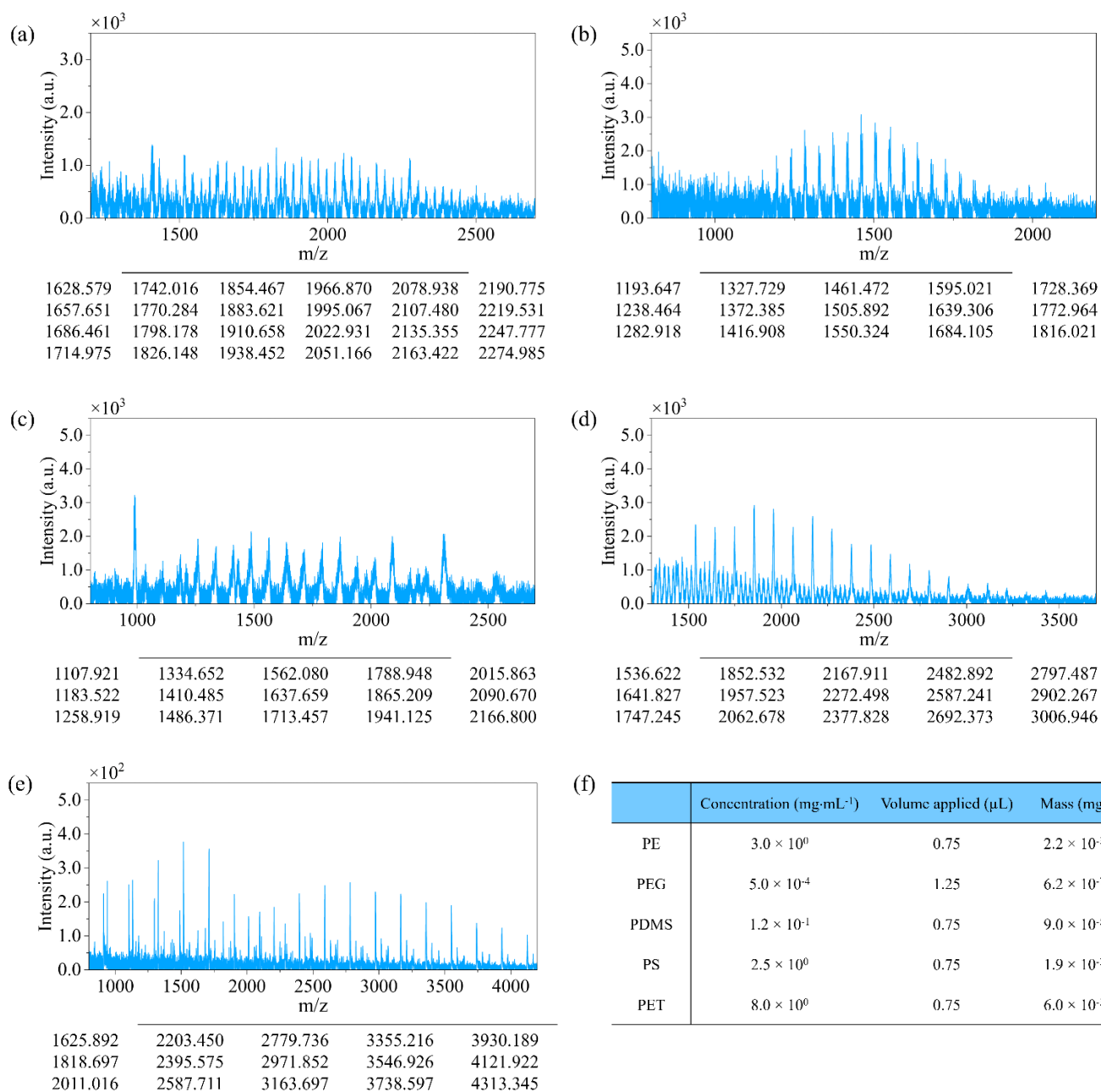


Figure S8 | Limits of detection of HoLDI-TOF-MS for commonly used plastics in the presence of real-world aerosol samples. The mass spectra of the lowest detectable concentration for the plastics (a) polyethylene (PE), (b) polyethylene glycol (PEG), (c) polydimethylsiloxane (PDMS), (d) polystyrene (PS), and (e) polyethylene terephthalate (PET), along with their corresponding characteristic peaks. The limits of detection of studied plastics are summarized in (f).

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