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that must be overcome. The ability to measure these real-world environments is needed to better assess the risks associated with micro-plastics. To that end, the current study focused on developing a methodology for sampling and characterizing airborne micro-plastics. Particulate sampling was carried out at a municipal materials recovery facility near a conveyor belt containing sorted plastic materials to collect airborne environmental particles on filters. Nucleopore filters were mounted on Teflon support rings, coated with 100 nm aluminum to reduce the background signal for micro-Raman spectroscopy, and marked with a fiducial pattern using a laser engraver. The fiducial pattern was crucial in identifying samples, relocating particles, and efficiently enabling orthogonal measurements on the same samples. Optimum sampling conditions of 2 h at 25 L/min were determined using light microscopy to evaluate the particle loadings. The filters were then cut into slices which were attached to sections of thin beryllium-copper sheeting for easy transfer of the filter between microscopy platforms. Scanning electron microscopy was used to identify carbon-rich particles. Light microscopy was used to identify colored particles which were also carbon-rich which were then analyzed using micro-Raman spectroscopy to identify specific polymers.

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Keywords (separated by '-') Sampling - Environmental - Micro-plastics - Microscopy - Micro-Raman spectroscopy

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## 2 Sampling of micro-plastics at a municipal recycling facility

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### 6 Abstract

**AQ1** Detecting, separating, and characterizing airborne micro-plastics from other airborne particulates is currently challenging due to the various instrumental constraints and related sample preparation hurdles that must be overcome. The ability to measure these real-world environments is needed to better assess the risks associated with micro-plastics. To that end, the current study focused on developing a methodology for sampling and characterizing airborne micro-plastics. Particulate sampling was carried out at a municipal materials recovery facility near a conveyor belt containing sorted plastic materials to collect airborne environmental particles on filters. Nucleopore filters were mounted on Teflon support rings, coated with 100 nm aluminum to reduce the background signal for micro-Raman spectroscopy, and marked with a fiducial pattern using a laser engraver. The fiducial pattern was crucial in identifying samples, relocating particles, and efficiently enabling orthogonal measurements on the same samples. Optimum sampling conditions of 2 h at 25 L/min were determined using light microscopy to evaluate the particle loadings. The filters were then cut into slices which were attached to sections of thin beryllium-copper sheeting for easy transfer of the filter between microscopy platforms. Scanning electron microscopy was used to identify carbon-rich particles. Light microscopy was used to identify colored particles which were also carbon-rich which were then analyzed using micro-Raman spectroscopy to identify specific polymers.

20 **Keywords** Sampling · Environmental · Micro-plastics · Microscopy · Micro-Raman spectroscopy

### 21 Introduction

22 Environmental plastic pollution is a growing problem. It's  
23 been estimated that there have been 8300 million metric tons  
24 of plastic produced as of 2017 and production is expected  
25 to reach 12 billion metric tons by 2050 [1]. Plastics start  
26 out as primary plastics made up of large objects such as  
27 plastic bottles or bags and range in size down to nanome-  
28 ter-sized particles in personal care products. They undergo  
29 photochemical and mechanical weathering [2, 3] to pro-  
30 duce smaller particles or secondary plastics. Micro-plastics  
31 (MPs) [3] are defined as being smaller than 5 mm whereas  
32 nanoplastics (NPs) are generally considered to range from  
33 1 to 100 nm [4]. Plastic particles can be found in almost  
34 every environment [5], from soils [6], oceans [7, 8], ocean  
35 sediments [9], freshwater systems [10], the atmosphere [11],  
36 to the Arctic sea ice [12] and Antarctic waters [13]. The

37 persistent presence of plastic particles in the global food  
38 supply [14] and the environment affects marine organisms  
39 [15], livestock animals [16], and humans [17, 18]. Their  
40 potential toxic effects on human and animal health remain  
41 a subject of intense investigation and research is in its early  
42 state [19, 20].

43 Separating micro- and nanoplastic particles (MNPs) from  
44 other constituents in complex environmental samples has  
45 proven to be difficult due to limitations in the sampling and  
46 characterization of such low masses and small sizes [21].  
47 While there are a variety of methods to analyze MNP parti-  
48 cles [22], these generally involve extraction of the particles  
49 from water or soil onto a suitable substrate for analysis. Dif-  
50 ferent types of filter materials can be used to extract MNPs  
51 from samples prior to analysis, including nylon, nitrocellu-  
52 lose, glass fiber, polycarbonate, and stainless steel, all with  
53 distinct advantages and disadvantages [23]. More recently,  
54 silicon membrane filters were developed and used as suit-  
55 able substrates to filter and extract MNPs while showing  
56 comparability within different analytical methods [24]. This  
57 study aims to bypass extraction methods by collecting and  
58 analyzing MNPs on the same substrate. The decision to use

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59 polycarbonate (PC) filters as the substrate for this work was  
 60 based on past experience with these filters. PC filters are  
 61 commonly used in air monitoring and microscopy tech-  
 62 niques due to their smooth and transparent surface, precise  
 63 pore diameters, and durability. However, a major problem  
 64 with the direct employment of PC filters is their high fluo-  
 65 rescence background [25]. The background can be reduced  
 66 with a layer of aluminum (Al) deposited on the surface [26].

67 The development of metrology for airborne MNPs relies  
 68 on effective sampling and sample preparation.

69 This work presents MNP sampling and sample prepara-  
 70 tion methods that include coating commercially avail-  
 71 able polycarbonate filters with a layer of Al and engraving  
 72 fiducial patterns on the coated filter. This novel approach  
 73 achieves (1) reduced Raman background issues because of  
 74 the Al coating [26], as well as (2) improved conductivity for  
 75 scanning electron microscopy (SEM), and (3) the fiducial  
 76 markers permit characterization of the same sample across  
 77 multiple microscopy techniques. Carbon-rich particles were  
 78 identified using SEM with energy dispersive X-ray analysis  
 79 (SEM–EDX), identified as colored using light microscopy,  
 80 and analyzed by micro-Raman spectroscopy to identify the  
 81 polymer type. A common approach for chemical identifi-  
 82 cation was tested to examine the applicability of airborne  
 83 samples collected in a real-world indoor environment, spec-  
 84 ifically at a local municipal recycling facility (MRF). This  
 85 location was chosen since it is an indoor environment where  
 86 recycled materials, including plastics, are mechanically  
 87 sorted and handled via abrupt movements on conveyer belts  
 88 and rotary drums. Due to the location, we expect a higher  
 89 abundance of MNPs than in a more diluted outdoor environ-  
 90 ment; however, any discussion about particle concentration  
 91 at the MRF is beyond the scope of this work. The scope of  
 92 this work encompasses method development for sampling  
 93 and sample preparation of airborne MNPs and not ambient  
 94 particle concentration monitoring of the MRF indoor air.

## Materials and methods

95

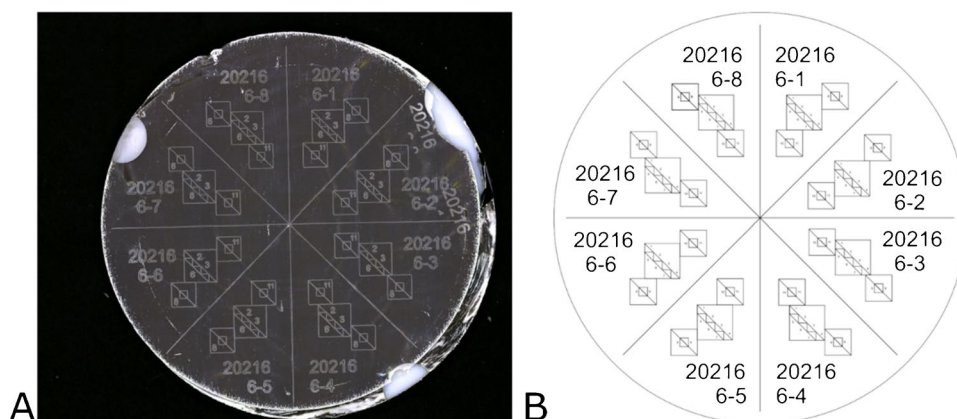
### Filter preparation

AQ2 6

97 To prepare substrates ideal for both MP sampling and Raman  
 98 characterization, Whatman (Marlborough, MA) 47-mm  
 99 polycarbonate (Nucleopore) filters, with 0.4  $\mu\text{m}$  diameter  
 100 pores, were glued to a Teflon support ring using a thin layer  
 101 of toluene-diluted contact cement (DAP Products, Balti-  
 102 more, MD). (See Supplemental information (SI) for more  
 103 details.) Subsequently, the filters were coated with 100 nm of  
 104 aluminum using an electron beam evaporator (Denton Vac-  
 105 uum Infinity 22, Denton Vacuum LLC, Moorestown, NJ) to  
 106 reduce the high Raman background signal from the polycar-  
 107 bonate filter [26]. All handling of the filters was performed  
 108 with stainless steel tweezers. Finally, a fiducial pattern and  
 109 sample identifiers were created using a laser engraver (Epi-  
 110 log, Golden, CO) to locate individual particles on filters,  
 111 catalog the samples, and enable sample transfer between dif-  
 112 ferent spectroscopy and microscopy-based techniques (see  
 113 SI for more details). Additionally, the engraving prepared  
 114 for the eventual segmenting of the filter into 8 slices which  
 115 will be discussed further in the next section. The total pro-  
 116 cess to create a set of 15 filters took less than 6 h. Figure 1  
 117 shows an example of the completed filter prior to sampling,  
 118 which includes the date that the filter was engraved as well  
 119 as other identifiers and markers, as well as the design of  
 120 the fiducial markers. The filters were stored until ready for  
 121 use in separate 100 mm polystyrene Petri dishes. Controls  
 122 were run using the same analytical equipment described in  
 123 the following sections with results suggesting that no cross  
 124 contamination occurred. Just before the filters were taken to  
 125 the MRF for sampling, they were weighed. See SI for more  
 126 details on the weighing procedure.

127 To have “control” particles with known composition  
 128 for Raman micro-spectroscopy, pure granular or pelleted  
 129 polyethylene terephthalate (PET), polyethylene (PE),

**Fig. 1** Light microscope image of filter with fiducials (A) after coating and an image of fiducials showing triangles used in particle location (B). The light areas around the edges of the filter in A are where tabs were placed to hold the filter in the coater and the Teflon ring shows through



high-density polypropylene (HDPE), and polystyrene (PS) were cryomilled to micrometer and submicrometer sizes. They were dispersed (see SI for more details) on an aluminum-coated filter mounted on a glass slide for analysis.

### Sampling protocol

Air sampling was conducted at MRF on a mezzanine level above a conveyer belt and rotary drum where recyclable glass, metals, and plastics first enter the MRF stream and where trash is manually removed.

For the sampling, a continuous-duty HI-Q Environmental model PSU-2 air sampler (HI-Q Environmental, Inc., San Diego, CA) with a maximum flow of 50 LPM was used. Before sampling started, the sampler's flow rotometer was calibrated at 15 L/min and 45 L/min using a Magnehelic differential pressure gauge. The sampler's inlet consisted of an open-faced filter holder for the Al-coated, polycarbonate membrane filter. Several inches above the inlet was a cover plate to keep excessively large particles (e.g., > 1 mm in size) from landing on the filter. The inlet extended above the railing of the mezzanine, providing a direct line of sight between the sampling inlet and the conveyer belt, about 3 m (10 feet) from the sampler. Since there was little to no size segregation, except for that provided by the cover plate, particles were collected over a large size range.

After the filter was mounted, the air sampler was turned on manually and the flow rate was adjusted. Typical air sampling conditions included one or 2 h at around 30 LPM, reduced from an initial sampling of 6 h, which produced filters that were too loaded for individual particle analysis. Table S2 in the SI shows the sampling rates and durations. For shorter sampling intervals, the sampler was timed to operate when the conveyor belt was running. Prior to removing the filter, the sampler was turned on briefly, to ensure that the flow rate had not changed. The flow rate was evaluated using the gauge on the sampler. There was no noticeable difference in flow rates with or without a filter before and after sampling, so it was concluded that the filter had no effect on the flow rate.

### Preparation for analysis

To calculate particle mass loading, samples were returned to the lab and weighed again using the same balance that was used to weigh the samples prior to collection. The filters were too large to fit into the field of view of the imaging instruments; therefore, they were cut into "pie" slices by rocking a scalpel blade along the etched lines between the slices (Fig. 1). Slices from the loaded filters were attached with small dabs of silver paint to a 10 mm × 10 mm section of a 0.15 mm thick beryllium copper (BeCu) sheet. The thin 10 mm × 10 mm sections allowed the sample slices on

the BeCu sheet to be easily mounted on a glass slide for light microscopy and micro-Raman spectroscopy, and on aluminum stubs for SEM.

Prior to light microscopy and SEM, some filter slices were coated with  $\geq 20$  nm of carbon from a Cressington Carbon Coater (Cressington Scientific Instruments, Watford, UK) to minimize the charging of particles by the electron beam in SEM. Carbon coating also helped to provide a visual contrast of the particles in light microscopy.

### Imaging

Light microscopy was used to qualitatively inspect the population of particles by color, size, and shape in addition to visual inspection of filter loading to determine optimal sampling conditions (i.e., flow rate and sampling times). Images were acquired using a semi-motorized Olympus BX53 upright microscope in bright-field reflected mode. Image stitching and focus stacking were performed with Olympus cellSens Dimension 1.17 software. Lower magnification ( $\times 10$  objective) stitched images were taken of the filter slices and the large squares shown in Fig. 1A and B. Higher magnification ( $\times 50$  objective) stitched images with extended focal imaging were taken of each individual triangle inside the large square (Fig. 1C). Qualitative assessments using the light microscope are focused on the areas of the individual triangles in the fiducial pattern. Figure 2 shows how the image can be zoomed in to show an individual triangle and the particles in the triangle.

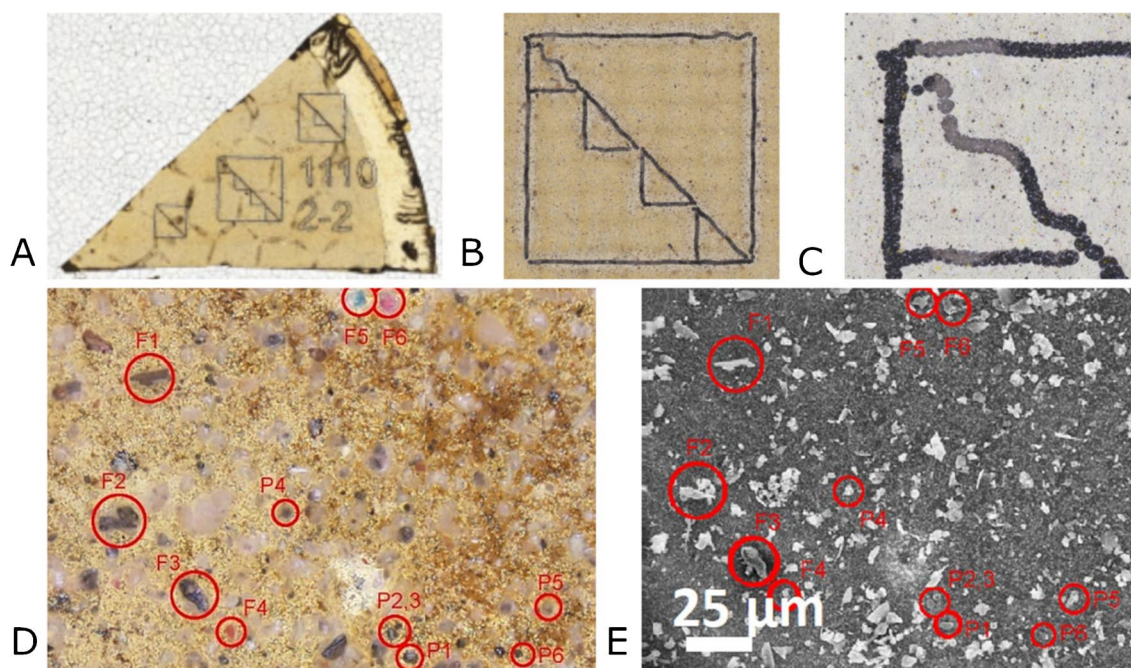
A coated filter that had not been taken to the MRF but was handled in the same manner as the sampled filters was imaged using the light microscope to check for PS particles on the filter.

SEM-EDX was conducted to qualitatively identify carbonaceous particles as potential MPs within a fiducial triangle on a filter slice. The carbon coating adds an insignificant signal to the total carbon EDS signal according to modeling [27]. Particles were manually selected for analysis based in part on particle size and morphology using secondary electron (SE) imaging. EDX spectra were collected with an Oxford X-Max detector (Oxford Instrument USA, Concord, MA) on a Nova NanoLab 600 FEI-focused ion-beam SEM instrument (Thermo-Fisher Scientific, Waltham, MA) for 180 s with an electron beam energy of 20 kV and current of 0.62 nA.

### Micro-Raman spectroscopy

Sample particles were analyzed with a Kaiser Optical Systems RXN1 Microprobe. In addition to sample particles, control particles from cryomilling of reagent polymer resins were analyzed.





**Fig. 2** Light microscopy images of a slice of the filter (A), a square fiducial pattern with smaller triangles (B), a triangle (C) corresponding to the upper left triangle seen in panel B, and an image of the same location on a filter using light microscopy (D) and SEM SE

imaging (E) showing the particles of interest (P) and particles having unusual shapes (U) that were also used in particle relocations. Note that the irregular line shown in B and C aids in identifying the relevant triangle

227 Cryomilled “control” particles of PS, PP, and HDPE  
 228 and a bulk sample of polyethylene terephthalate were analyzed with a Kaiser Optical Systems RXN1 Microprobe  
 229 (Endress + Hauser USA, Greenwood, IN, US) fitted with a 785 nm Coherent SureLock diode laser (Coherent, Inc.,  
 230 Santa Clara, CA, US) attached to a Leica (Wetzler, Germany) DM2700P Microscope operating at 17 mW or 70 mW  
 231 (5% or 20% of maximum power at 350 mW, respectively).  
 232 For each sample, the spectrum consisted of 20 accumulations of Raman signals each collected for 5 s or 30 s and  
 233 corrected for dark current.

234 The elevated spectral baseline caused by fluorescence was corrected in Spectragryph-id-on Ver. 1.2.15 software  
 235 (<https://spectroscopy.ninja>) with an adaptive baseline having a coarseness setting of 15 and an offset of 0. Spectra were  
 236 also smoothed in Spectragryph-id-on using the Savitsky-Golay algorithm with an interval of 50 and polynomial order  
 237 of 3.

## 245 Results and discussion

246 A key factor in this work is that it allows for the analysis of individual particles on filters across microscopy platforms  
 247 (light microscopy, SEM, and micro-Raman spectroscopy) using the fiducial pattern with associated triangles to efficiently  
 248 relocate particles between different instruments

251 without the need to transfer the particles between different  
 252 substrates. The focus of the work was colored, carbon-rich  
 253 particles, since they were easy to detect with light micro-  
 254 scopy. Preliminary Raman results on colored carbon-rich air-  
 255 borne particles showed that it is possible to identify specific  
 256 polymers in samples from an MRF.

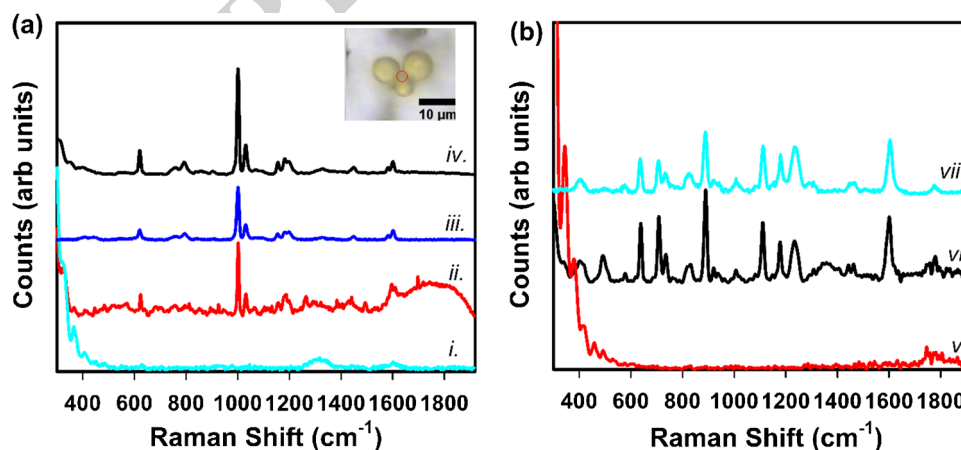
257 The mass of the filter samples as well as the flow rates is  
 258 shown in the SI. The particulate loading of the filters was  
 259 monitored by light microscopy, and it was determined that a  
 260 shorter sampling time was needed. A 6-h sampling produced  
 261 a filter that was too heavily loaded for individual particle  
 262 identification, whereas either a 1-h sampling at 35 LPM or  
 263 a 2-h sampling at 25 LPM had similar loadings. The 2-h  
 264 sampling at the lower flow rate of 15 LPM was preferable  
 265 (Fig. 2). At higher flow rates, the filter surface also became  
 266 irregular due to the honeycomb surface of the filter holder  
 267 screen, which made automated light microscopy and SEM  
 268 more difficult. Given the location of the sampler in the MRF,  
 269 the filters showed much more clear particles, and fewer plas-  
 270 tic particles than expected. Nevertheless, this study was done  
 271 in a MRF where the proportion of plastics in the indoor air is  
 272 expected to be higher than in outdoor air samples. Based on  
 273 SEM-EDS analysis, the majority of the clear particles were  
 274 determined to be glass.

275 Initially, carbon-rich particles were identified as candidate  
 276 MPs by SEM-EDX, then, with the help of light microscopy,  
 277 were relocated and analyzed by micro-Raman to confirm

278 they were plastic particles. Figure 2 shows how the fidu-  
 279 cials were used to identify a location on the filter starting  
 280 from a slice (Fig. 2A) and moving down to a single field  
 281 of view using light microscopy as well as the same field of  
 282 view in SEM (Fig. 2D and E, respectively). Based on the  
 283 morphology of the candidate MPs (e.g., particles of inter-  
 284 est in Fig. 2D and E), we might expect these carbonaceous  
 285 particles to result from the abrasion of bulk plastics (i.e.,  
 286 as secondary MPs). Additional possible sources for carbon  
 287 particles could be the conveyer belt, exhaust from recycling  
 288 trucks, or another source. However, none of the candidate  
 289 particles inspected by micro-Raman produced spectra with  
 290 identifiable polymer peaks. It is noted that the analysis was  
 291 done using a 785 nm laser, and as discussed below, these  
 292 particles may require a higher energy laser to increase the  
 293 intensity of their polymer peak signal. Due to this challenge  
 294 and the high number of carbon-containing particles identi-  
 295 fied using SEM–EDX, light microscopy was ultimately  
 296 used instead to distinguish potential plastic particles based  
 297 on color. The use of the fiducial pattern in the filters made  
 298 it possible to go directly and quickly from light microscopy  
 299 to micro-Raman and skip the time-consuming step of using  
 300 SEM–EDX to identify candidate MPs. SEM–EDX was  
 301 still used to complement the light microscopy and micro-  
 302 Raman analysis since it provides additional information  
 303 about particle populations, especially the inorganic particles.  
 304 Also, SEM–EDX is useful because it is not biased towards  
 305 colored and spherical particles, whereas inspection with  
 306 light microscopy potentially misses white or colorless par-  
 307 ticles. Figure 3a shows micro-Raman spectroscopy and light  
 308 microscopy of 3 yellow spheres as well as the background

309 from the coated filter, a cryomilled PS particle, and a PS  
 310 reference spectra [28]. The matching peak at  $1001\text{ cm}^{-1}$  is  
 311 characteristic for PS. The low background from the filter and  
 312 the collection of Raman spectra from the small particles,  
 313 where both the cryomilled particle and the MRF particles  
 314 are less than  $10\text{ }\mu\text{m}$ , show that identification of MPs in MRF  
 315 samples is possible with further work. The lowering of the  
 316 background from the filter is further illustrated in Fig. 3b,  
 317 which shows an Al-coated filter, uncoated filter, and polycar-  
 318 bonate reference spectra. The source of the spherical MPs is  
 319 currently unknown and is the focus of future research. While  
 320 NP can be collected, characterization is difficult because the  
 321 Raman signal is not strong enough for particles smaller than  
 322 a few micrometers in diameter due to high signal to noise.

323 One of the main benefits of the sampling and filter design  
 324 is minimizing sample preparation that allows direct analysis  
 325 on the filter, and which minimizes processes that can affect  
 326 composition or morphology changes such as when using  
 327 solvents or mechanical forces. The method developed in this  
 328 study has some limitations and needs further improvements,  
 329 despite its obvious benefits. One of the main limitations of  
 330 this method is that the emphasis on light microscopy to iden-  
 331 tify potential plastic particles based on color makes it hard  
 332 to detect white and uncolored particles, especially due to  
 333 the high number of clear or colorless particles on the filters.  
 334 This limitation will be addressed in a future study looking  
 335 at using SEM–EDX identification of carbonaceous particles  
 336 in conjunction with polymer identification using a micro-  
 337 Raman system with multiple wavelengths, particularly one  
 338 at higher energy, which would allow potentially unidenti-  
 339 fied MPs to produce higher intensity signals. Furthermore,



**Fig. 3** a LM and Micro-Raman spectra from three yellow spheres including the filter background, cryomilled PS, and a reference spectrum of PS. Note the overlap of peaks including the characteristic peak at  $1001\text{ wavenumbers}$ . The red circle shows the location of the Raman laser spot. Also note the low background from the filter. Spectrum i shows the coated filter background, ii shows the yellow microspheres, iii shows a PS spectrum from the SLOPP library and

iv shows a cryomilled PS particle reduced by 50%. b shows the spectra from an uncoated filter, coated filter, and polycarbonate reference spectra, all at  $785\text{ nm}$ , to show how the coating reduces the Raman background from the polycarbonate filter. Spectrum v shows the Al-coated filter background, vi shows the uncoated filter background, and vii shows a  $10\text{ }\mu\text{m}$  PS particle from the SLOPP library

340 an automated micro-Raman system would allow for higher  
341 throughput analysis.

342 Another limitation is that the method cannot detect or  
343 characterize plastics in the nanometer range due to the limi-  
344 tations of the micro-Raman instrument. Also, the sampling  
345 procedure lacks size segregation, making it difficult to focus  
346 on a particular size range of particles. Size segregation could  
347 be provided by using a different filter sampler, such as an  
348 impactor. Another benefit of this procedure is that it provides  
349 a complex environmental sample of MPs without a tissue  
350 or water matrix that, with further analysis, could be used  
351 as a proxy for controlled environmental studies. In addi-  
352 tion, sampling at a site such as a materials recovery facility  
353 provides a type of sample where the concentration of plas-  
354 tics is expected to be high and, therefore, advantageous for  
355 studying environmental plastics. Lastly, this method enables  
356 the easy and time-saving relocation of individual particles  
357 between different microscopy techniques.

358 **Supplementary Information** The online version contains supplemen-  
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364 **Author contribution** Abigail Lindstrom: writing original draft and  
365 editing of manuscript, conceptualization, data collection. Diana Ortiz  
366 Montalvo: editing manuscript, conceptualization, data collection and  
367 analysis. Joseph Conny: generation of idea, editing of manuscript, con-  
368 ceptualization, data collection and analysis.

## 364 **Declarations**

370 **Ethics approval** Declarations on human or animal rights are not appro-  
371 priate for this work.

372 **Competing interests** The authors declare no competing interests.

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