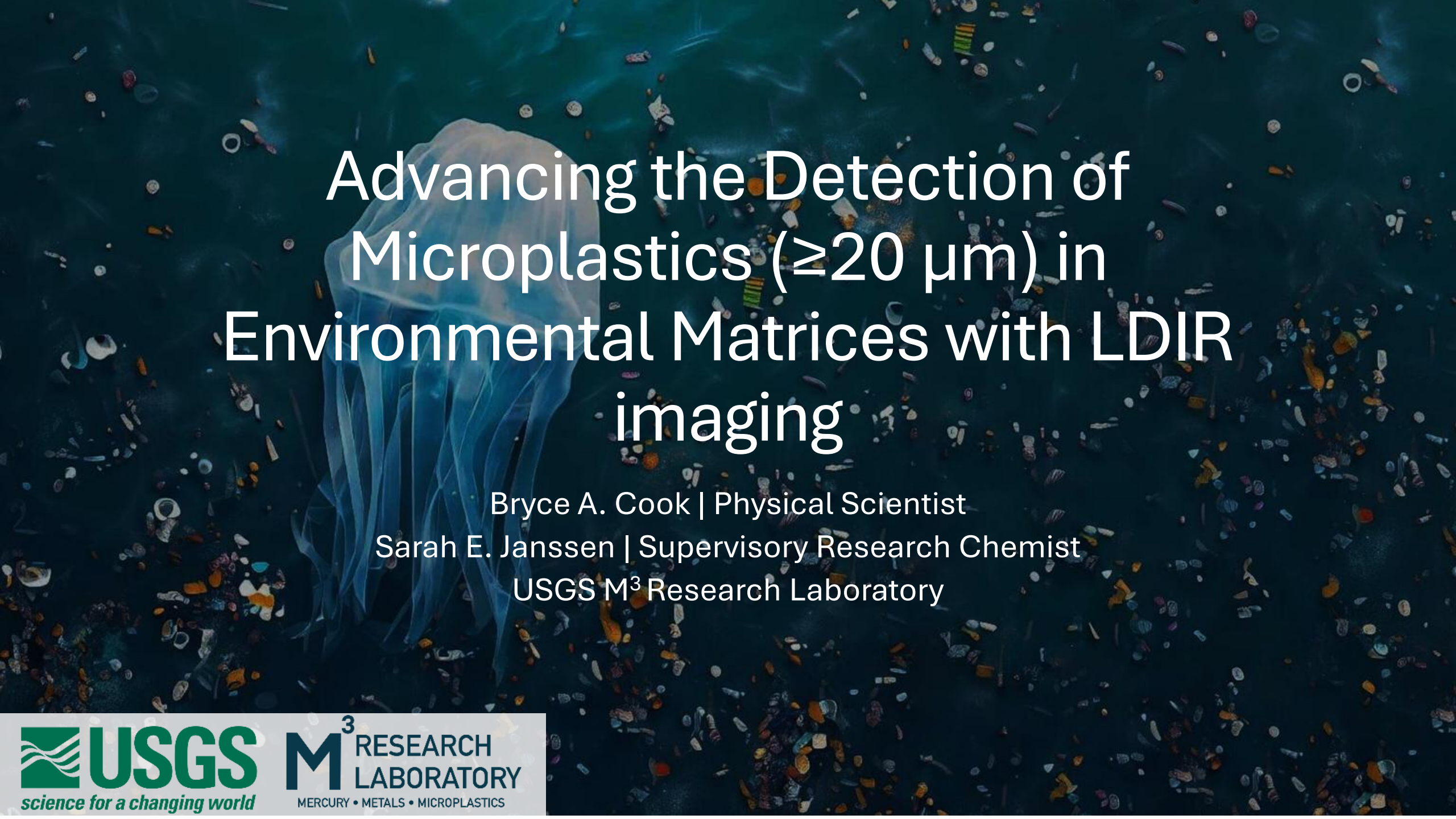


Advancing the Detection of Microplastics (≥20 µm) in Environmental Matrices with LDIR imaging

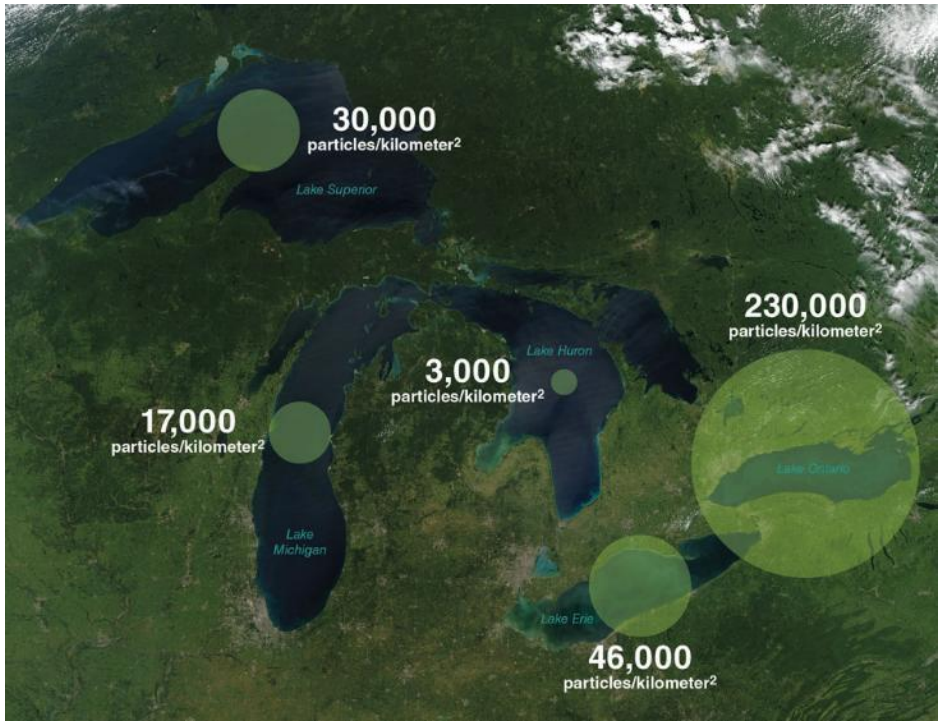
Bryce A. Cook | Physical Scientist
Sarah E. Janssen | Supervisory Research Chemist
USGS M^3 Research Laboratory

USGS science for a changing world

M^3 RESEARCH LABORATORY
MERCURY • METALS • MICROPLASTICS

Plastics Pollution in the Midwest

22 million pounds of plastic waste enter the Great Lakes every year



Adapted from Eriksen et al. 2013 and Liu et al. 2020

Microplastics can potentially:

- Be more bioavailable
- Adsorb more toxic chemicals

Sources include:



Moeck et al. 2022

Microplastics Research at the USGS



Recently the USGS has created a strategic vision document to elucidate knowledge gaps for microplastics fate and transport

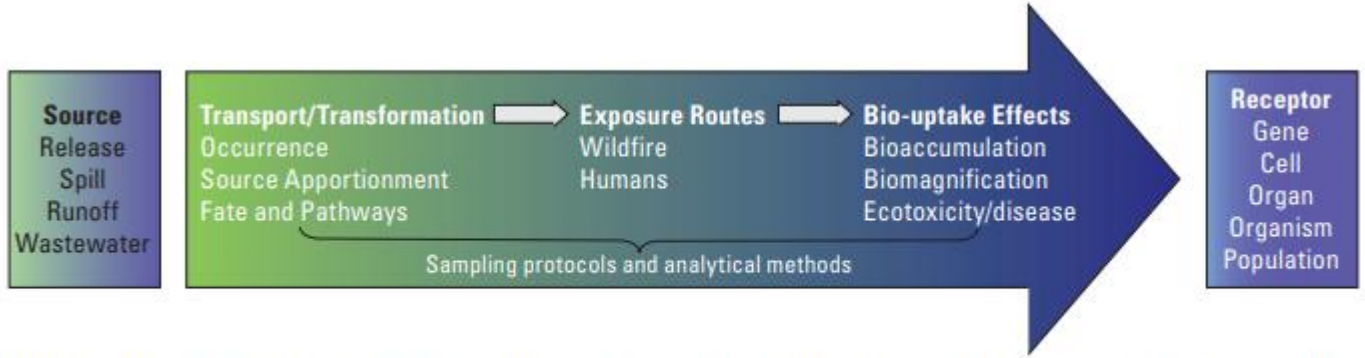


Figure 2. Flow chart showing the source-to-receptor framework for the study of microplastics in the environment. Critical points in this figure include: (1) source, (2) mode of transport/transformation, exposure routes, and bio-uptake effects, and (3) receptors. Examples are given for each critical point.

One key gap is sample collection and analysis!

Current Goals for M^3 Lab

01

Develop a high throughput method for determining MP concentrations in:

- Waters
- Sediments/Soils
- Biota

02

Develop reliable sampling methods for microplastic sample collection

03

Develop robust quality assurance and control metrics to assess laboratory performance

Available Analytical Instrumentation

Agilent LDIR

- Ideal for analyzing MP particles >20 µm relatively fast
- Allows for complete on-filter analysis

Raman confocal microscope

- Ideal for analyzing nano-plastics and microplastics <20 µm
- Much more intensive and time-consuming, full-filter analyses are not practical

Fluorescent microscope

- Allows for detection of microplastics stained with fluorescent dye.
- No information about plastic type
- Significant positive interference from stained organic matter

Water Sample Collection

- Environmental water samples for MP analysis can be collected by a variety of methods:
  - Pumping water through stacked sieves
  - Preferred: collection of particles on stainless steel mesh filters by pumping
- M3-designed sampler:
  - Has be retrofitted for boat sampling (EPA Lake Explorer II), groundwater monitoring well sampling, and wadable water
  - This field season the device was used in:
    - Milwaukee – Menomonee River
    - Ponds/Rivers in Indiana Dunes NP, Voyageurs NP, Isle Royale NP, St Croix Scenic Riverway, Mississippi River recreation Area
    - 11 Lake Superior tributaries across 3 states
    - Zion National Park
    - Groundwater monitoring and supply wells in California, Florida, Illinois, and Long Island
    - Streams/Reservoirs in Washington and New Mexico



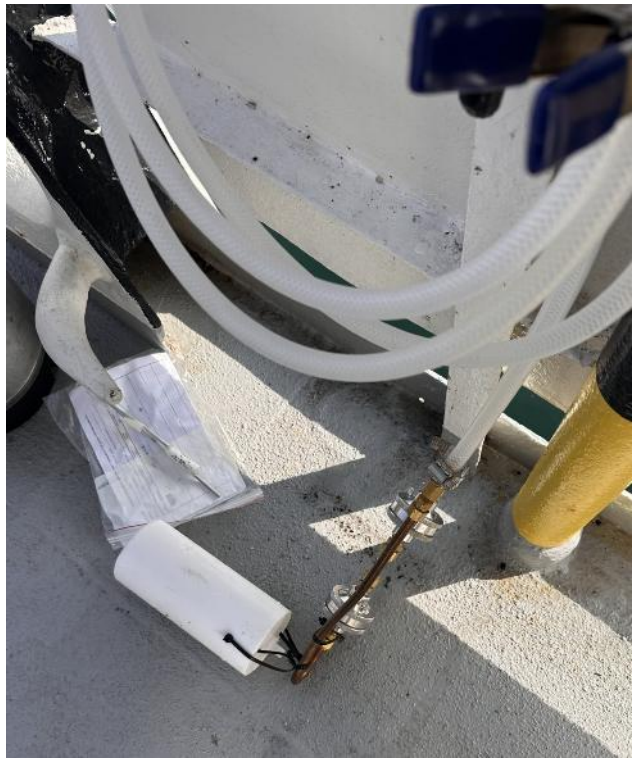




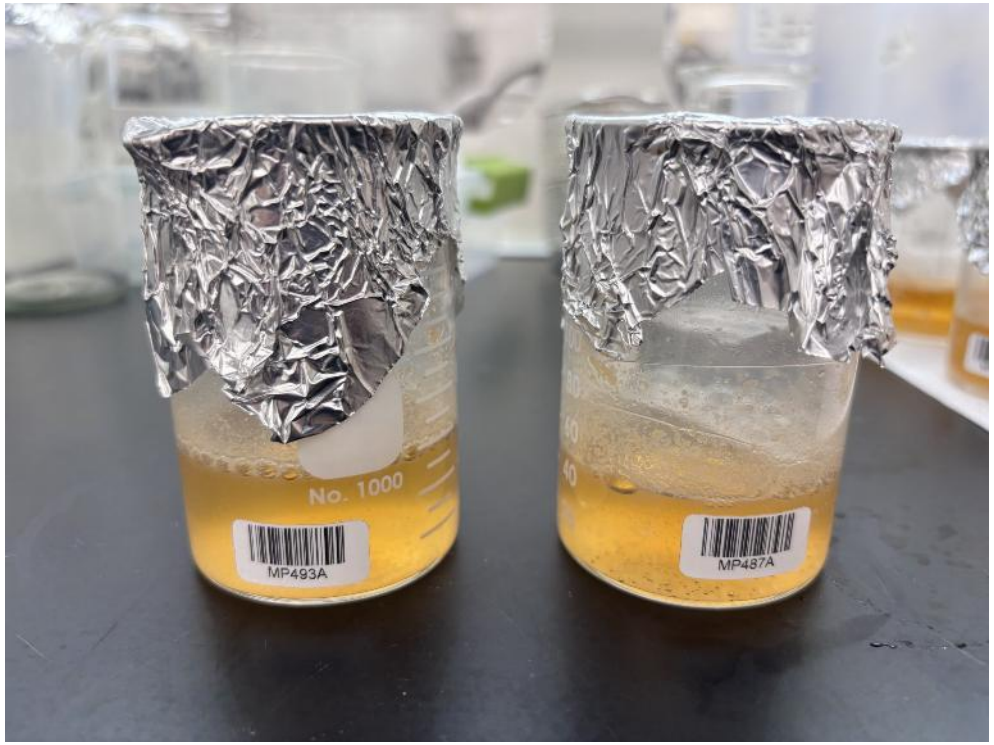
Masterflex peristaltic pump (covered to protect from the elements)

Carboy to collect pumped water. Need to record total volume pumped through device

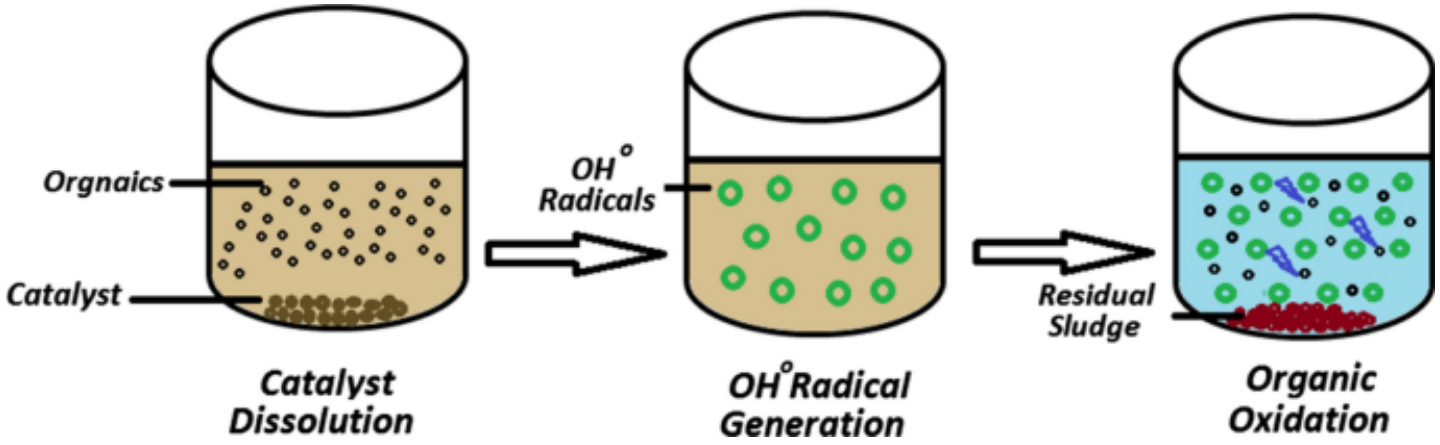




Sampling Device + manifold + 25-foot-long sample line. This is dropped into the water over the edge of the boat, and we pump from the boat to pull water through the device



Digesting Lake Ontario Lake Trout

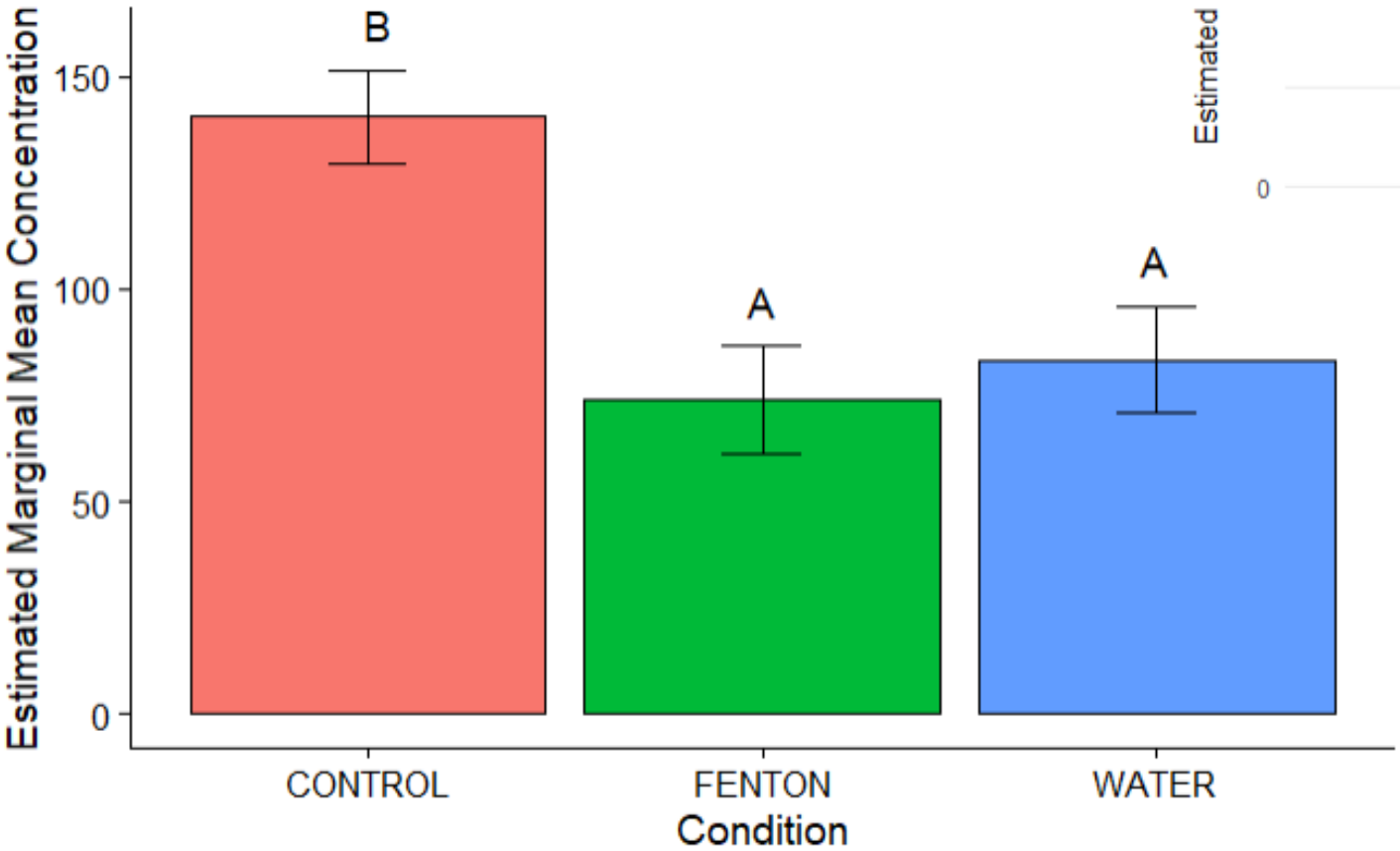


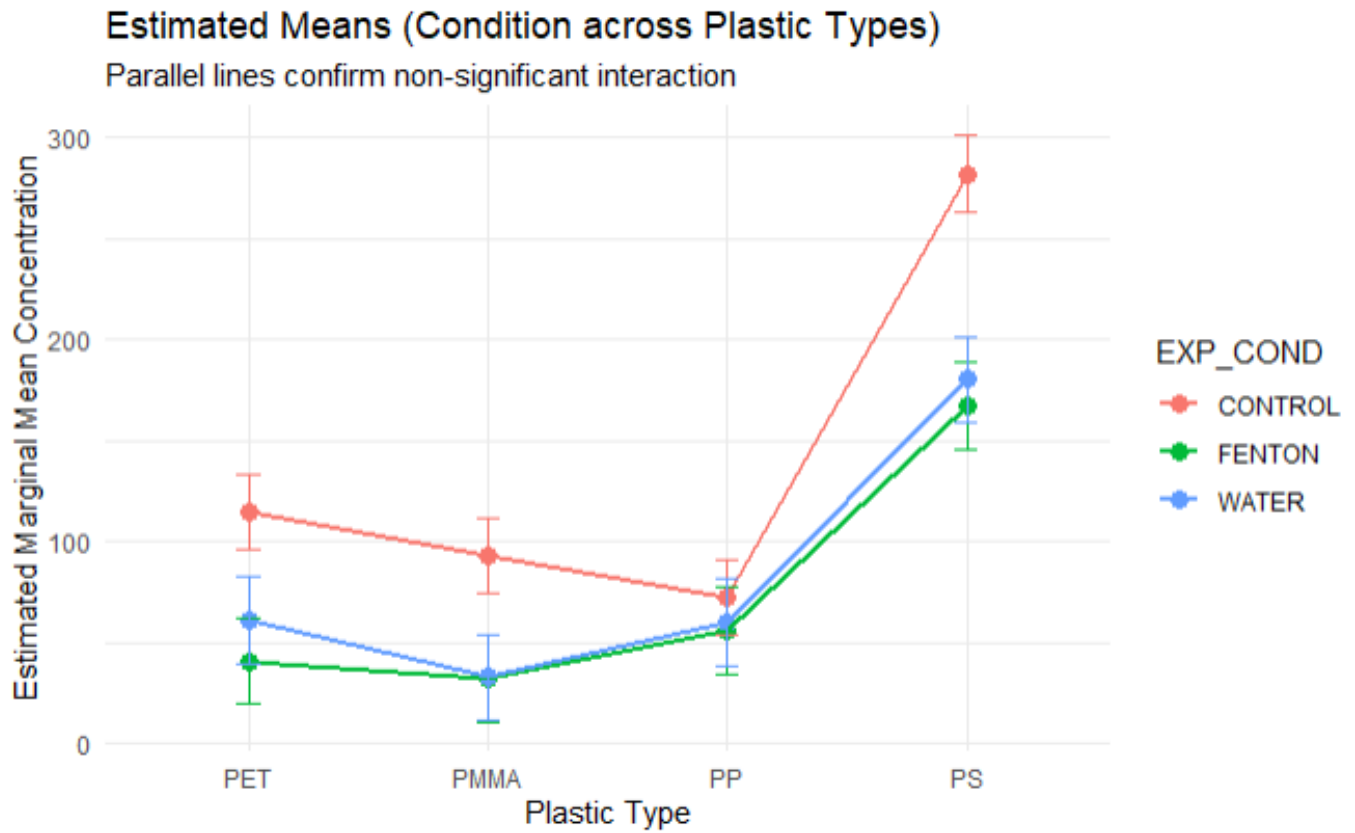
In-Lab Sample Processing

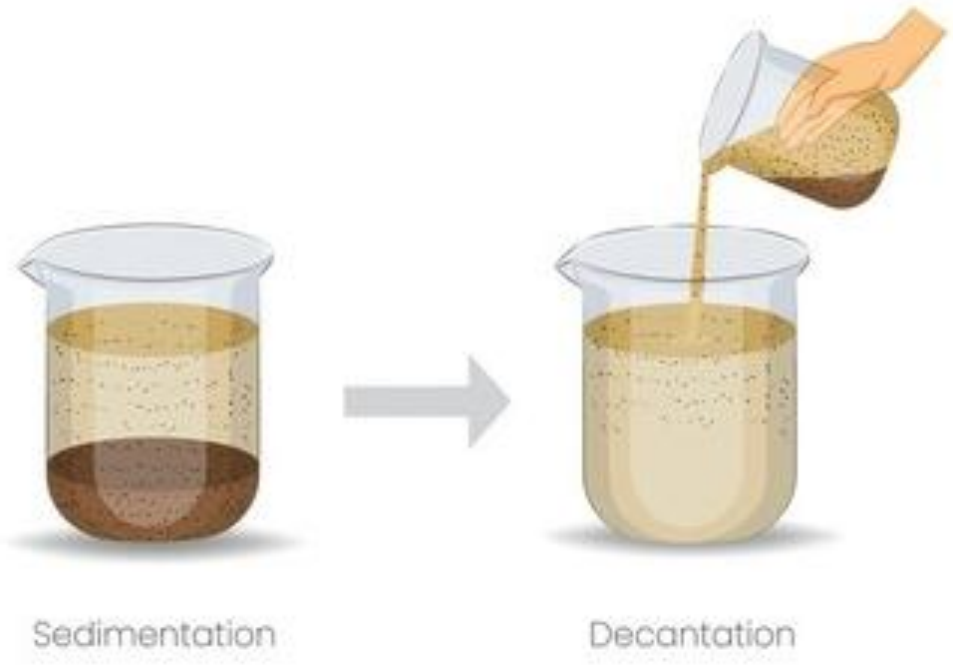
- **Step 1: Organic Matter Digestion**
  - **Catalyzed wet peroxide oxidation (WPO) shown in literature to be effective and minimizes damage to MPs** (Masura, et al., NOAA Technical Memorandum, 2015)
  - **I tested the effect of Fenton's on MPs**
  - Biota rich in tissue and fat benefit from an alkaline treatment post-Fenton's

Fenton's reagent has the same effect as water on microplastics.

The takeaway is that glassware transfer is likely the largest source of recovery loss.





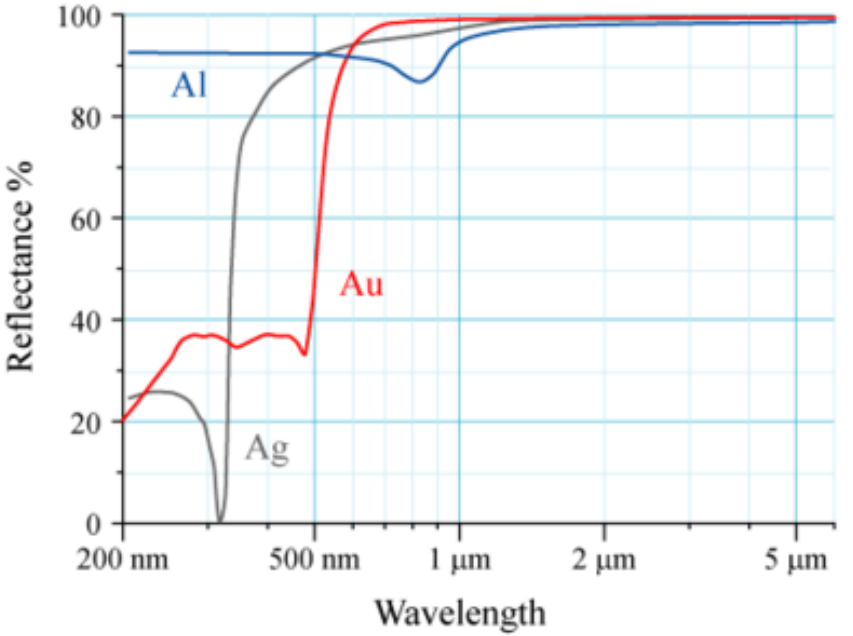


In-Lab Sample Processing

- Step 1: Organic Matter Digestion
  - Catalyzed WPO shown in literature to be effective and minimizes damage to MPs
- **Step 2: Density Separation of non-plastic particles**
  - **This step is optional depending on the sample matrix**
  - **Removes inorganic and many organic interferents**

In-Lab Sample Processing

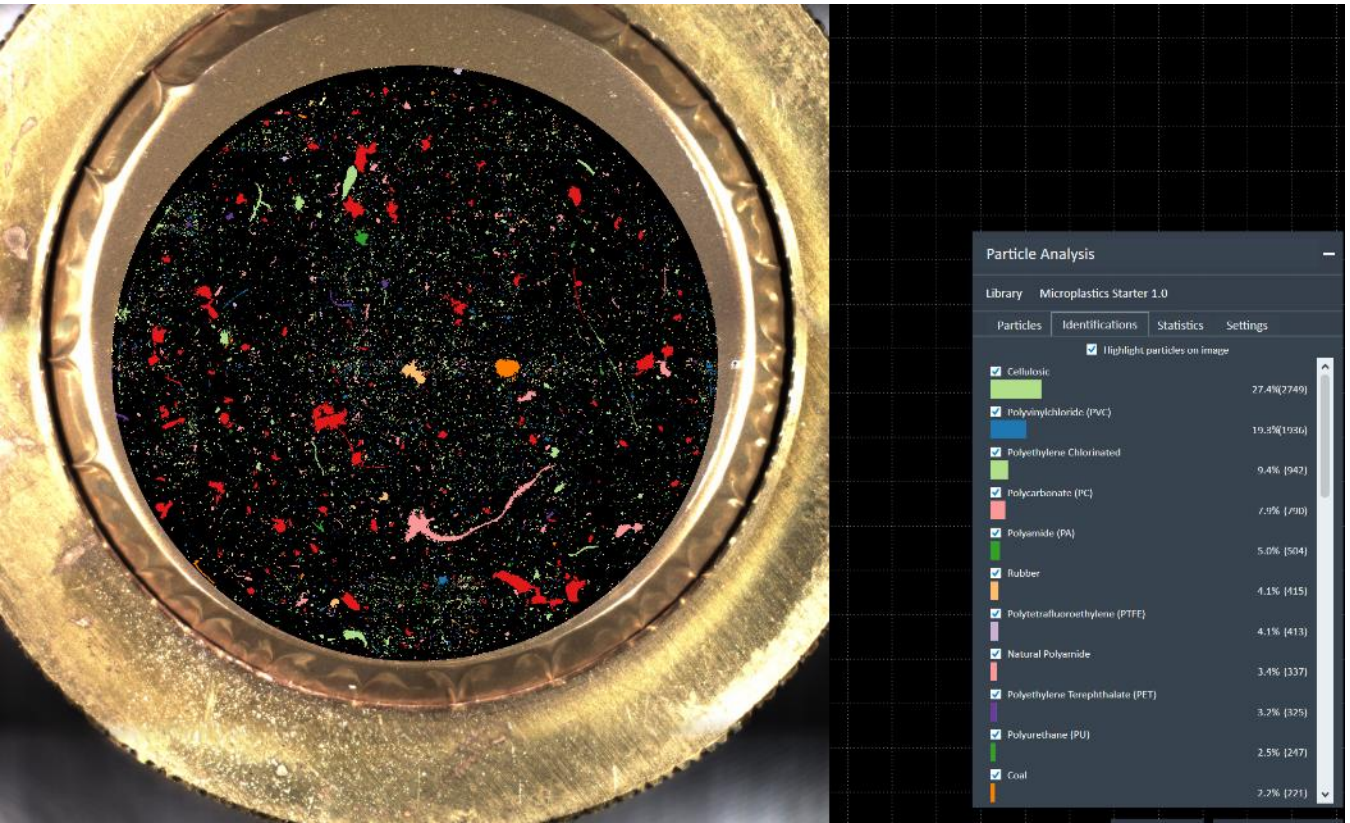
- Step 1: Organic Matter Digestion
  - Catalyzed WPO shown in literature to be effective and minimizes damage to MPs
  - Step 2: Density Separation of non-plastic particles
  - This step is optional depending on the sample matrix
- **Step 3: filtration onto gold-coated LDIR sensors or regenerable anodic aluminum oxide sensors for Raman spectroscopy**
  - **Most samples need to be filtered onto 2 gold sensors to avid overloading the sensor with particles**

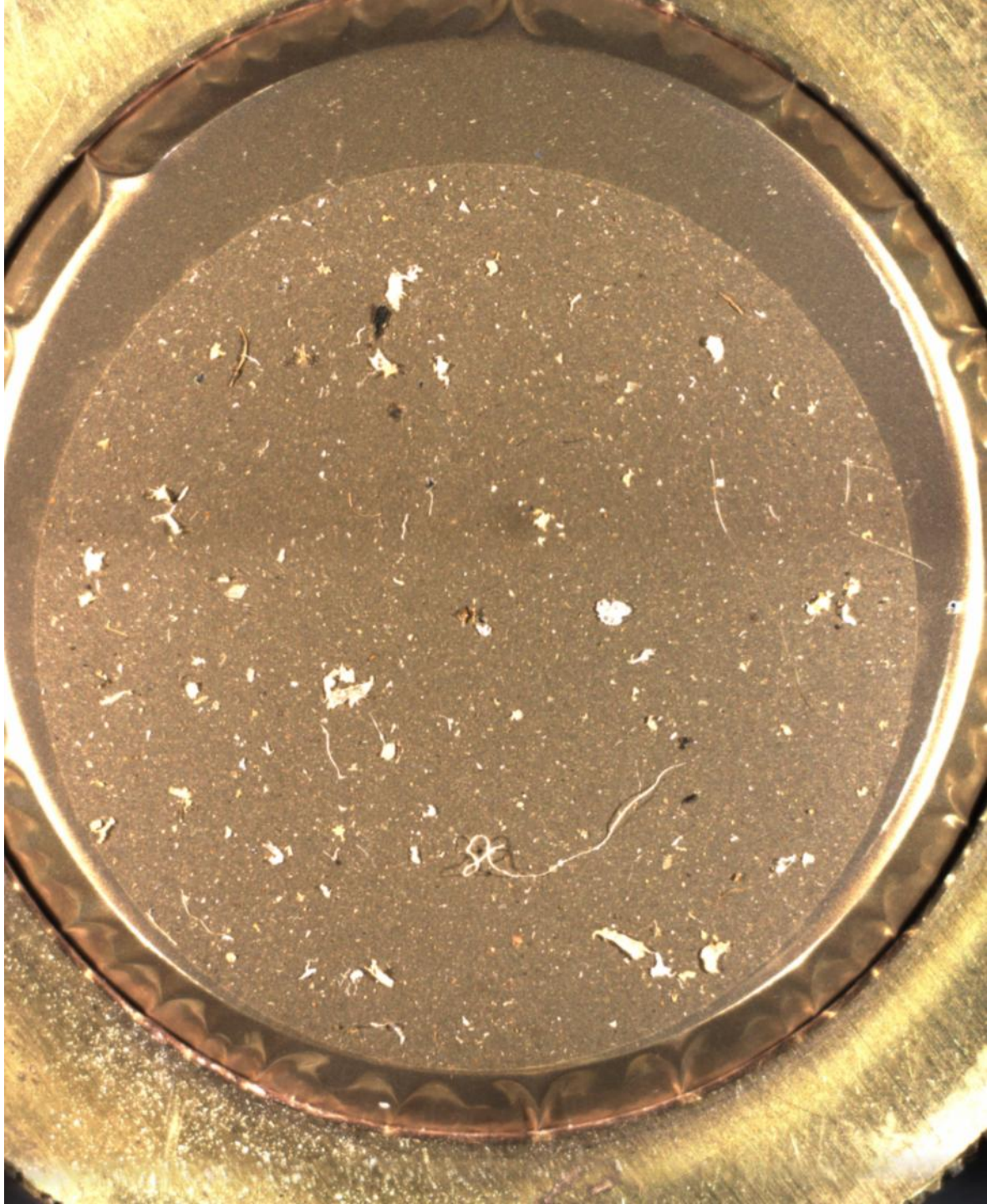




LDIR analysis (>20 µm)

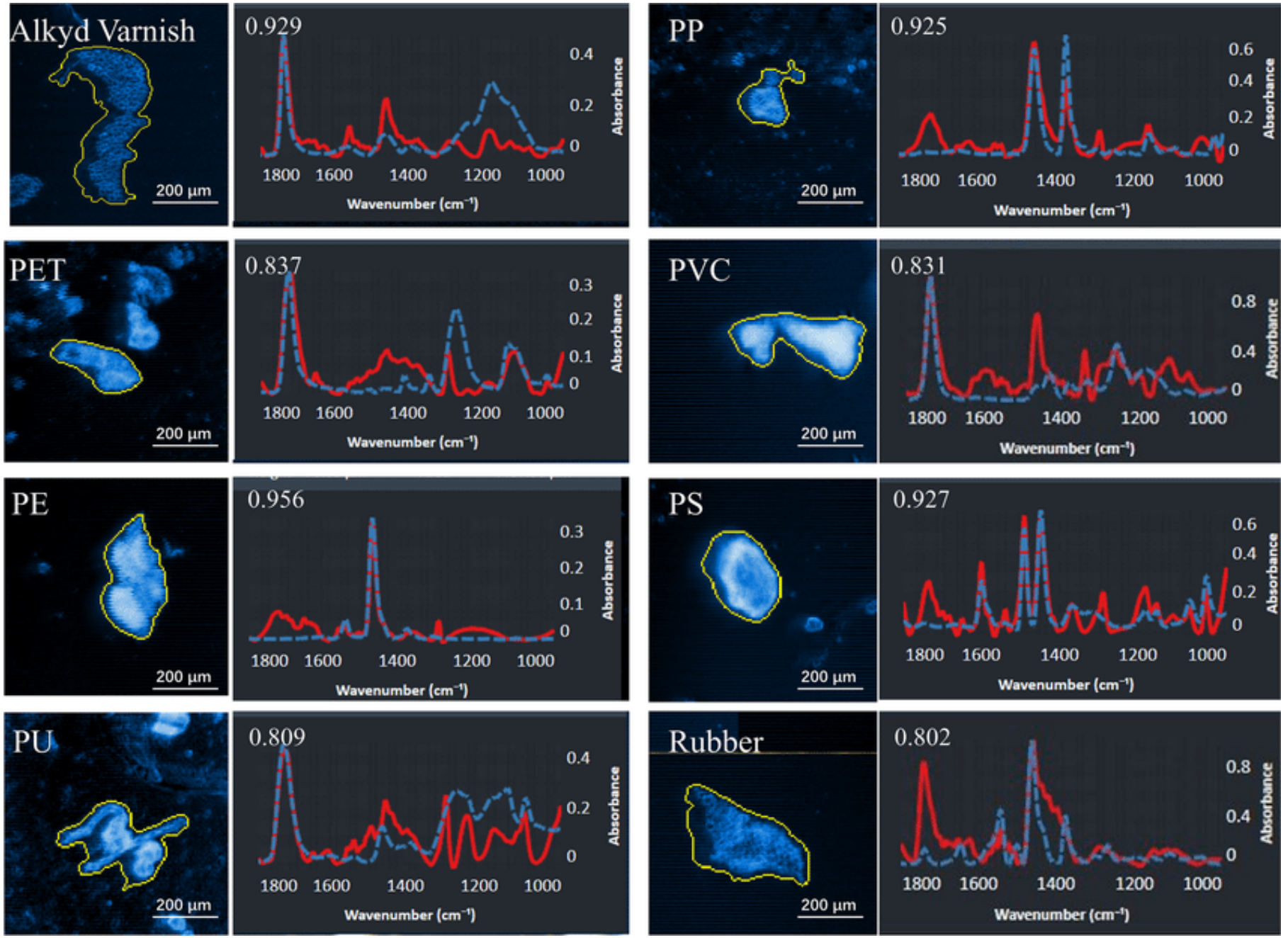
- Laser Direct Infrared Imaging
- Scans the entire filter to count particles
- Uses IR image to identify organic particles.
- Takes rapid IR fingerprint spectra of each particle using QCL in transflectance mode





LDIR analysis (>20 µm)

- Particles are identified based on comparison to IR spectral libraries
- Hit Quality Index is used to calculate how well the two spectra match
- Only spectra with a HQI ≥0.8 are accepted as a positive identification



Bao, et al., Environ. Sci. Pollut. Res. Int., 2022

LDIR analysis (>20 µm)

- After spectral IR identification, the entire filter can be mapped and color-coded based on the most abundant plastics



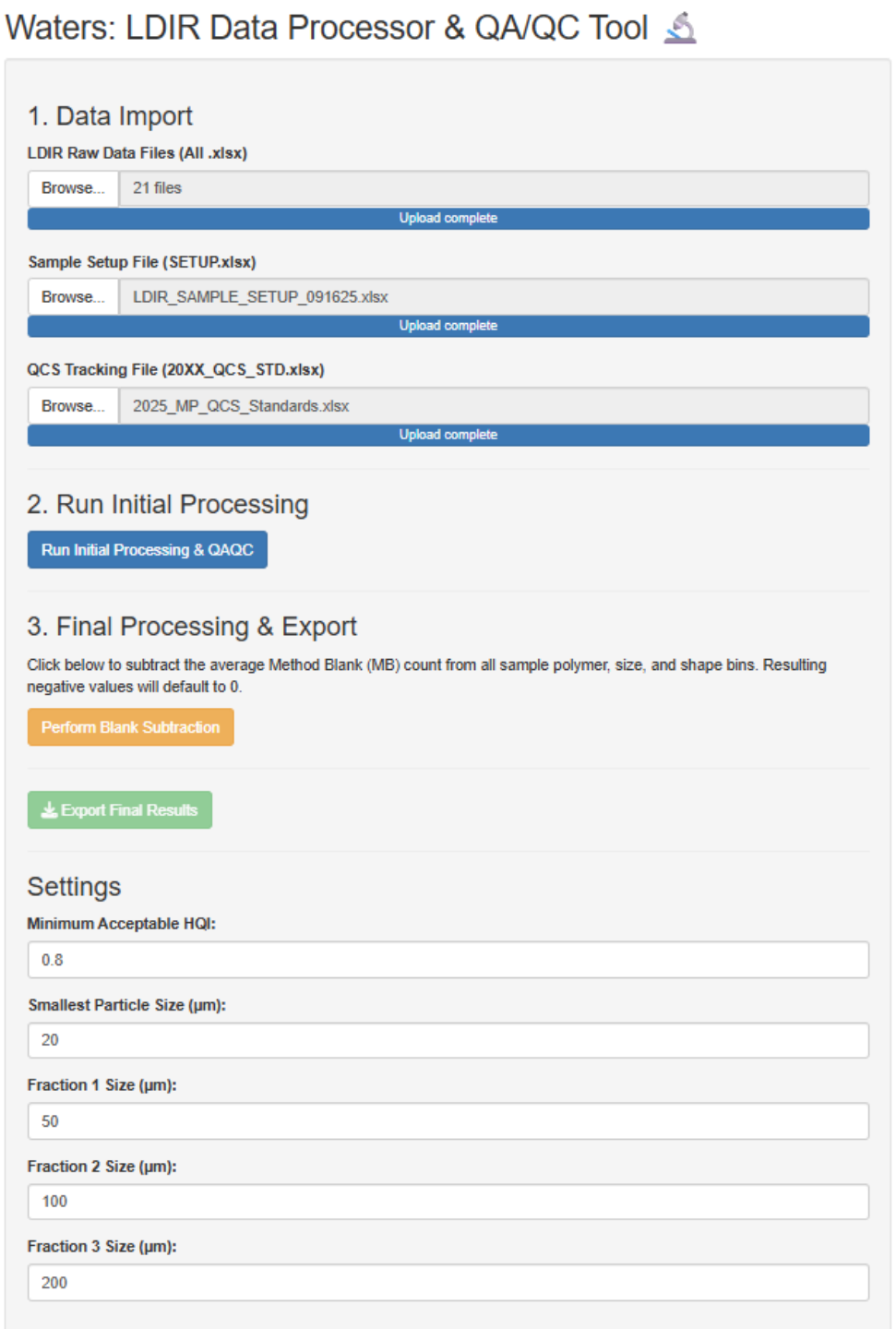
Data Wrangling

- The LDIR spits out a lot of data for each individual particle
- The most important are:
  - Aspect ratio (shape determination)
  - Diameter (size determination)
  - Identification
  - Quality
- Some samples have 30,000+ particles split across multiple gold filters to be wrangled

#	Id	Width (µm)	Height (µm)	Diameter (	Aspect Ratio	Area (µm²)	Perimeter (µm)	Eccentricity	Circularity	Solidity	Identification	Notes	Match Type	Quality	Is Valid
1	A1	178	510	202.4804	0.348778798	32200	2790.78208	0.875523259	0.051953389	0.463977	Polyethylene Terephthalate (PET)		Auto	0.819332213	true
2	A2	106	259	103.1866	0.408516598	8362.5	911.3351291	0.642655259	0.126528912	0.446298	Polyethylene Terephthalate (PET)		Auto	0.881201336	true
3	A3	105	110	73.45306	0.946387008	4237.5	720.6244522	0.658323114	0.102541953	0.439689	Polyurethane (PU)		Auto	0.687877284	true
4	A4	149	56	56.98035	2.651821614	2550	571.1269784	0.554836085	0.098239172	0.417178	Polyethylene Terephthalate (PET)		Auto	0.704391523	true
5	A5	97	54	43.15221	1.801886974	1462.5	338.4924215	0.292498608	0.160401284	0.438202	Cellulosic		Auto	0.649268829	true
6	A6	25	30	28.49018	0.833333333	637.5	101.2132031	0.688615265	0.782016137	0.944444	Natural Polyamide		Auto	0.779911276	true
7	A7	25	30	28.49018	0.833333333	637.5	101.2132031	0.784098093	0.782016137	0.962264	Natural Polyamide		Auto	0.756096276	true
8	A8	50	43	28.49018	1.161290408	637.5	191.92388	0.370866539	0.217486359	0.459459	Cellulose Acetate		Auto	0.688730352	true
9	A9	35	45	27.63953	0.777777778	600	174.8528123	0.672941927	0.246612946	0.489796	Polyvinylchloride (PVC)		Auto	0.710195212	true
10	A10	55	30	27.3501	1.833333333	587.5	201.92388	0.797093216	0.181068277	0.431193	Polyamide (PA)		Auto	0.688803388	true
11	A11	32	49	26.16039	0.642857115	537.5	191.92388	0.78387845	0.183370852	0.477778	Polyvinyl alcohol		Auto	0.663667296	true
12	A12	50	29	26.16039	1.742857219	537.5	176.0660154	0.668419303	0.217889981	0.5	Polyvinylchloride (PVC)		Auto	0.70578565	true
13	A13	31	62	25.54477	0.504347786	512.5	244.3502861	0.758966347	0.107864375	0.369369	Cellulosic		Auto	0.764763638	true
14	A14	76	31	24.59245	2.437500082	475	237.2792184	0.486280477	0.106019089	0.275362	Cellulosic		Auto	0.634983788	true
15	A15	53	30	24.26671	1.74193555	462.5	207.7817446	0.609485225	0.134619144	0.342593	Polymethylmethacrylate (PMMA)		Auto	0.655932408	true
16	A16	40	40	24.26671	1	462.5	207.7817446	0.571812613	0.134619144	0.385417	Polyurethane (PU)		Auto	0.688092751	true
17	A17	52	24	23.60174	2.149999985	437.5	171.92388	0.521857181	0.186001101	0.448718	Rubber		Auto	0.686082773	true
18	A18	15	53	22.91749	0.283018858	412.5	123.6396092	0.946864152	0.339092801	0.673469	Cellulosic		Auto	0.723055248	true
19	A19	25	20	22.91749	1.25	412.5	75.35533845	0.717125698	0.912863342	1	Cellulosic		Auto	0.718742435	true
20	A20	31	22	22.56758	1.400000085	400	96.56854153	0.661395252	0.539012095	0.780488	Cellulosic		Auto	0.663096095	true
21	A21	35	28	22.56758	1.250000126	400	144.8528123	0.609750427	0.239560931	0.5	Polyurethane (PU)		Auto	0.738048877	true
22	A22	17	65	22.21217	0.255319173	387.5	167.7817446	0.827064501	0.172978513	0.449275	Polyvinylchloride (PVC)		Auto	0.679968623	true
23	A23	25	35	21.85097	0.714285714	375	116.5685415	0.587412993	0.346799455	0.6	Coal		Auto	0.629579952	true
24	A24	20	25	21.85097	0.8	375	78.28427076	0.630674079	0.768939394	0.9375	Polyvinylchloride (PVC)		Auto	0.671867323	true
25	A25	20	35	21.4837	0.571428571	362.5	119.4974738	0.697982991	0.319007157	0.630435	Polyurethane (PU)		Auto	0.738255208	true
26	A26	20	25	21.4837	0.8	362.5	95.35533845	0.580199869	0.500988609	0.783784	Polyurethane (PU)		Auto	0.680225841	true
27	A27	37	22	21.4837	1.68750006	362.5	129.4974738	0.335435555	0.271640976	0.568627	Polyvinylchloride (PVC)		Auto	0.665112262	true
28	A28	45	49	21.11004	0.9090909	350	217.2792184	0.476112479	0.093162584	0.256881	Polyvinylchloride (PVC)		Auto	0.676085051	true
29	A29	19	29	21.11004	0.655172384	350	82.42640615	0.780026763	0.647358983	0.848485	Polyvinylchloride (PVC)		Auto	0.656665266	true
30	A30	47	20	21.11004	2.354838845	350	130.7106769	0.443560724	0.257427999	0.538462	Cellulosic		Auto	0.725547893	true
31	A31	16	38	20.72965	0.419354849	337.5	103.6396092	0.789341829	0.394849968	0.710526	Polyvinylchloride (PVC)		Auto	0.69101504	true
32	A32	32	42	20.72965	0.749999944	337.5	176.0660154	0.776141607	0.136814639	0.3375	Rubber		Auto	0.684517112	true
33	A33	35	15	20.34214	2.333333333	325	88.28427076	0.755025833	0.523994293	0.866667	Cellulose chemically modified		Auto	0.711307388	true
34	A34	20	38	20.34214	0.536585396	325	126.5685415	0.751961101	0.254942167	0.541667	Polyurethane (PU)		Auto	0.647785311	true
35	A35	35	15	20.34214	2.333333333	325	98.28427076	0.846784234	0.422790466	0.742857	Polyvinylchloride (PVC)		Auto	0.638249599	true
36	B1	94	98	98.28889	0.959183616	7587.5	345.060963	0.648568296	0.800786804	0.952904	Polyethylene Chlorinated		Auto	0.753355659	true
37	B2	266	83	98.0457	3.216080726	7550	687.6955199	0.464327785	0.200615473	0.45827	Cellulosic		Auto	0.652184207	true
38	B3	85	94	87.85792	0.90476189	6062.5	308.4924215	0.767949125	0.800520792	0.947266	Chitin		Auto	0.727160037	true
39	B4	67	78	70.91753	0.863636274	3950	253.1370831	0.648779752	0.77463197	0.923977	Polyethylene (PE)		Auto	0.945502792	true
40	B5	54	75	60.37092	0.726495671	2862.5	216.0660154	0.846997797	0.77051737	0.930894	Polyvinyl alcohol		Auto	0.698410633	true
41	B6	23	126	45.83498	0.184210527	1650	274.8528123	0.979544565	0.274469087	0.717391	Cellulose chemically modified		Auto	0.714646925	true

User Friendly R-Shiny Application

- Pulls in template files with sample information and raw LDIR files
- Filters samples by Hit Quality
- Reports raw counts by shape, size, and polymer for each sample
- Allows for blank subtraction of raw counts
- Calculates sample concentration in cc/L or cc/g depending on matrix
- Reports QCS and Matrix Spike recoveries
- Exports a cooperator-friendly summary document along with individual sample particle data (filtered for polymers and HQI ≥0.8)



Initial QA/QC Review | QCS Validation Report | Blank Corrected Data | Sample Summary Preview

Raw Counts by Polymer Bin

Microplastics_Count	MB_091625 (cc)	MP030A (cc)	MP031A (cc)
Ethylene Vinyl Acetate (EVA)	1	ND	ND
Polyethylene Chlorinated	2	33	2
Polymethylmethacrylate (PMMA)	5	9	1
Polypropylene (PP)	2	30	22
Polystyrene (PS)	2	4	5
Polytetrafluoroethylene (PTFE)	1	18	8
Polyurethane (PU)	5	18	3
Polyvinylchloride (PVC)	7	25	11
Rubber	2	13	10

Showing 1 to 9 of 9 entries

Raw Counts by Size Bin

Size_Bin	MB_091625 (cc)	MP030A (cc)	MP031A (cc)	MP032A (cc)
20-50µm	20	128	72	48
50-100µm	6	43	22	37
100-200µm	1	19	2	12
>200µm	ND	2	2	3

Showing 1 to 4 of 4 entries

Raw Counts by Shape Bin

Shape_Bin	MB_091625 (cc)	MP030A (cc)	MP031A (cc)	MP032A
Fiber	1	8	3	5
Fibrous Fragment	4	42	19	30
Fragment	15	99	51	47

Fi – lab-generated Fibers

Fr – Lab-generated Fragments

Ms – Standard Microspheres

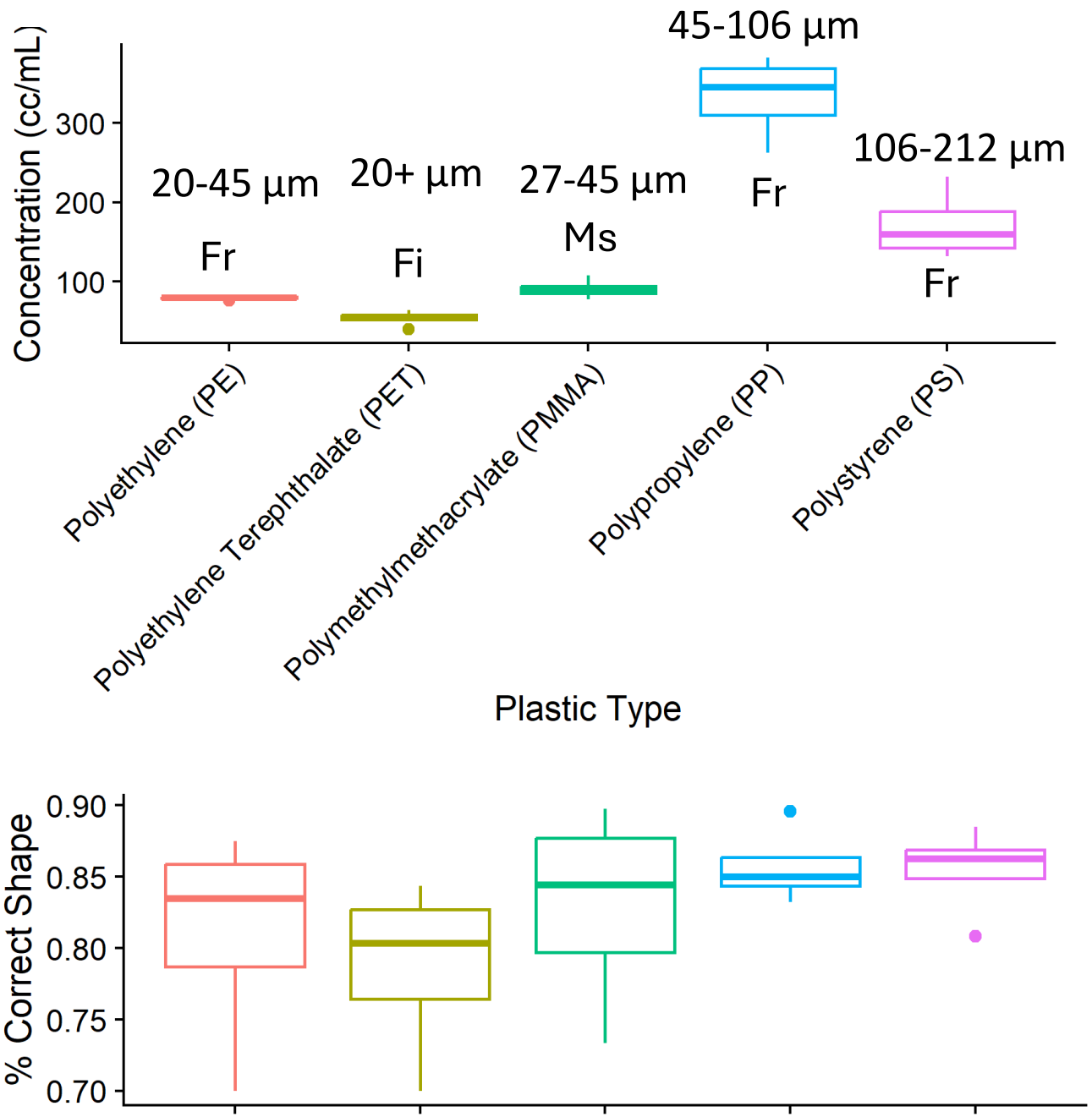
Quality Control

To ensure LDIR analyses are robust, quality control metrics are crucial!

Developed a quality control standard for various MP types, shapes, and size ranges.

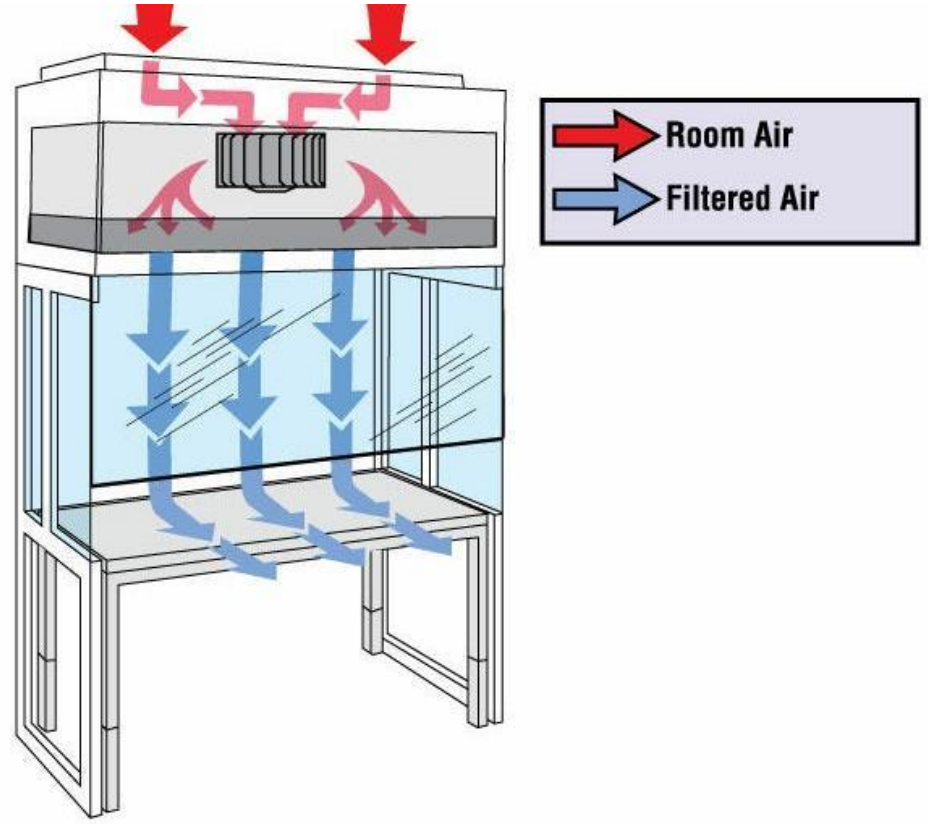
Particles suspended in 95% EtOH to ensure homogenous dispersion.

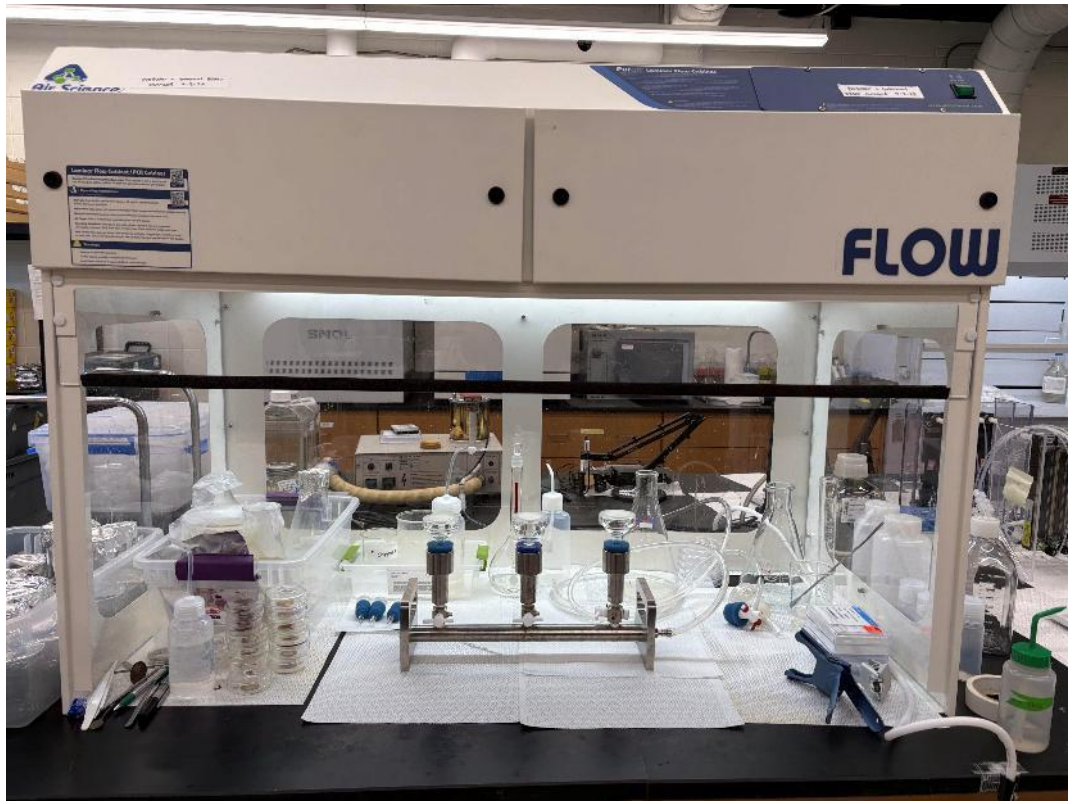
Allows the lab to assess recovery across the entire method



Quality Control

- Minimizing contamination:
  - All MP work performed under laminar flow hoods





Quality Control

- Minimizing contamination:
  - All MP work performed under laminar flow hoods
  - Combust materials in muffle furnace





Quality Control

- Minimizing contamination:
  - All MP work performed under laminar flow hoods
  - Combust materials in muffle furnace
  - Supply 100% cotton lab coats for analysts





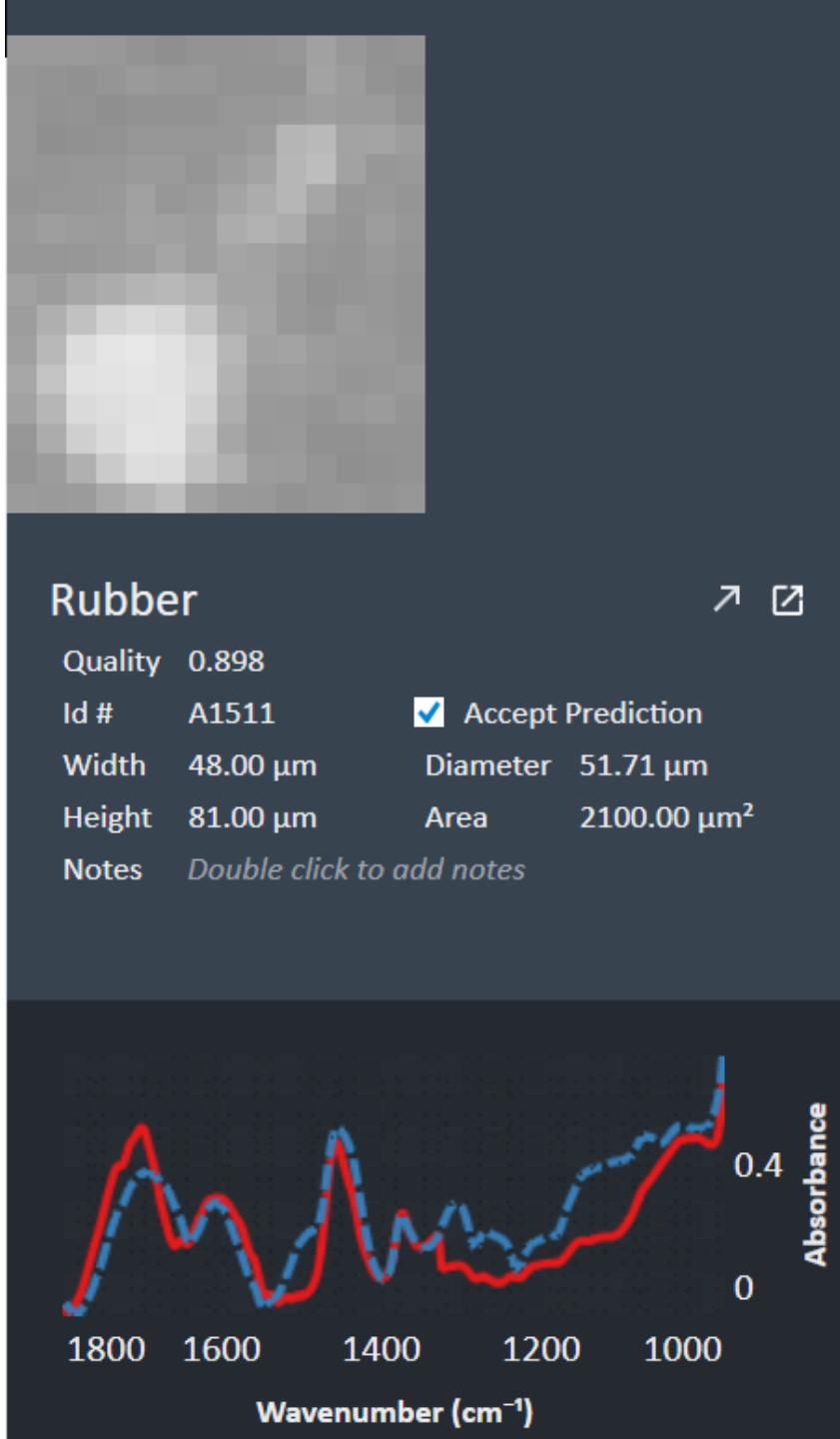
Quality Control

- Minimizing contamination:
  - All MP work performed under laminar flow hoods
  - Combust materials in muffle furnace
  - Supply 100% cotton lab coats for analysts
  - Include **open air blanks** and **method blanks** to assess contamination

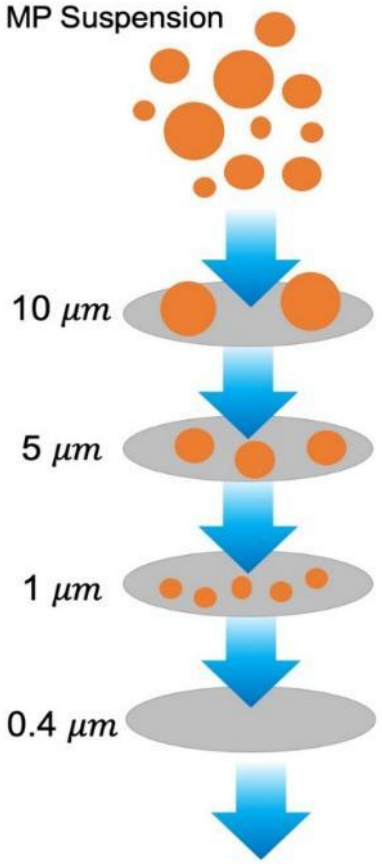


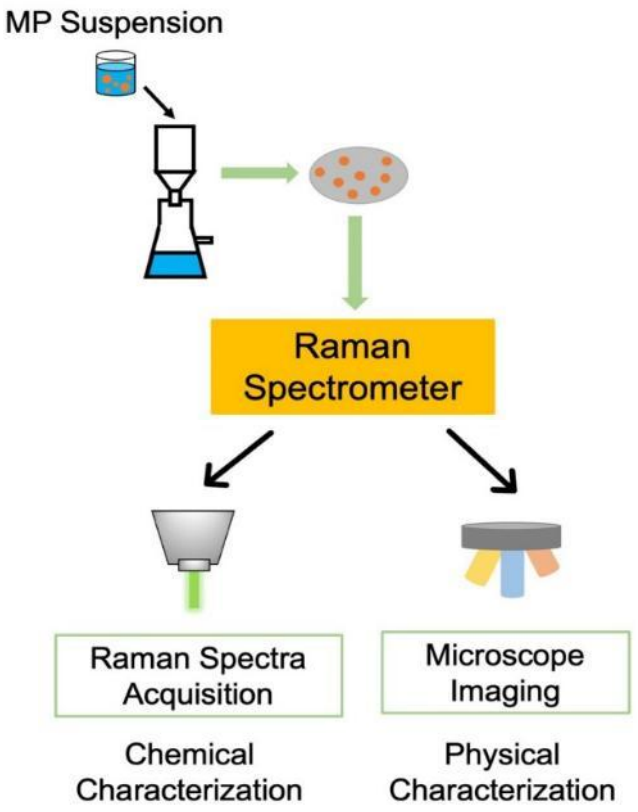
Status update:

- Experiment in progress (leading to eventual methods publication) assessing the previous methods' performance on various matrices:
  - Biota: walleye fillet, lake trout, dolphin liver, dragonfly larvae
  - Waters: high TSS water, low TSS water
  - Sediments/Soils: high OM sediment, low OM sediment
- Matrix spike/recovery assessment
- Stepwise QCS loss experiment
- LDIR reproducibility
- LDIR optimization – can we speed up LDIR analyses by analyzing smaller fractions of the total filter and extrapolating?
- Working to verify LDIR applicability for TRWPs
  - Difficult due to tires being proprietary mixtures of many ingredients
  - Further hindered by presence of carbon black which absorbs strongly in the IR region leading to noisy spectra
  - Strategic Environmental Research and Development Program proposal for TWRPs in urban areas selected for funding

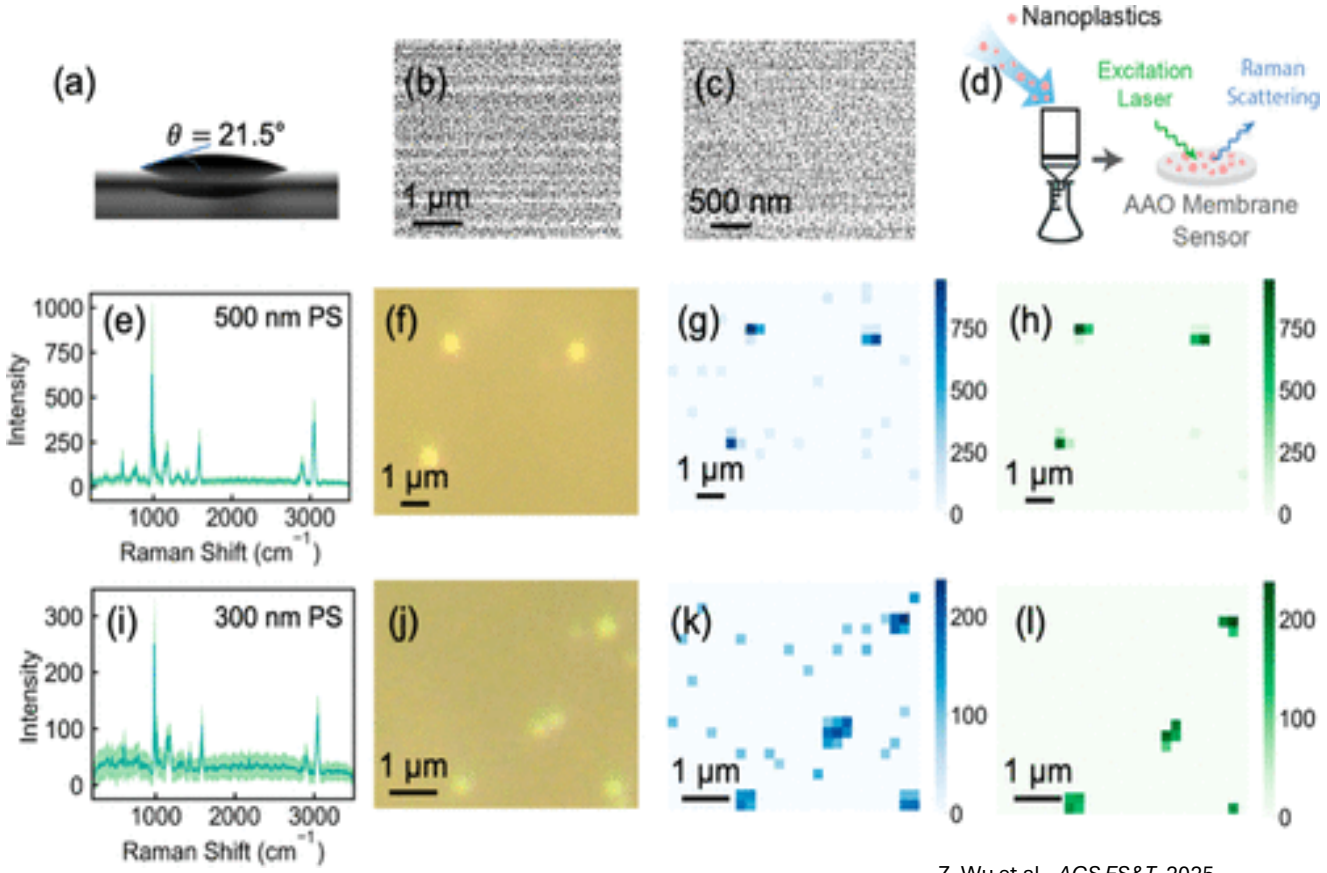


Raman Method Development (<20 µm)





Z. Wu et al., *ACS ES&T Water*, 2023



Z. Wu et al., *ACS ES&T*, 2025

Putting it all together

Current projects in progress at the M3 lab

Exploring Tributary MP Sources

Project Objective:

What are the concentrations and characteristics (e.g. size distribution, polymer type, shape etc.) of microplastics (MPs) in the surface water, water column, and surficial sediment in representative U.S. Lake Superior tributaries across MN, WI and MI?

Develop a standardized sampling approach that can be cross linked with other USGS, NPS, and state-led (MI EGLE) studies


Baptism River, MN


St. Louis River, MN


Bad River, WI

Sampling Sites

Sampling Plan

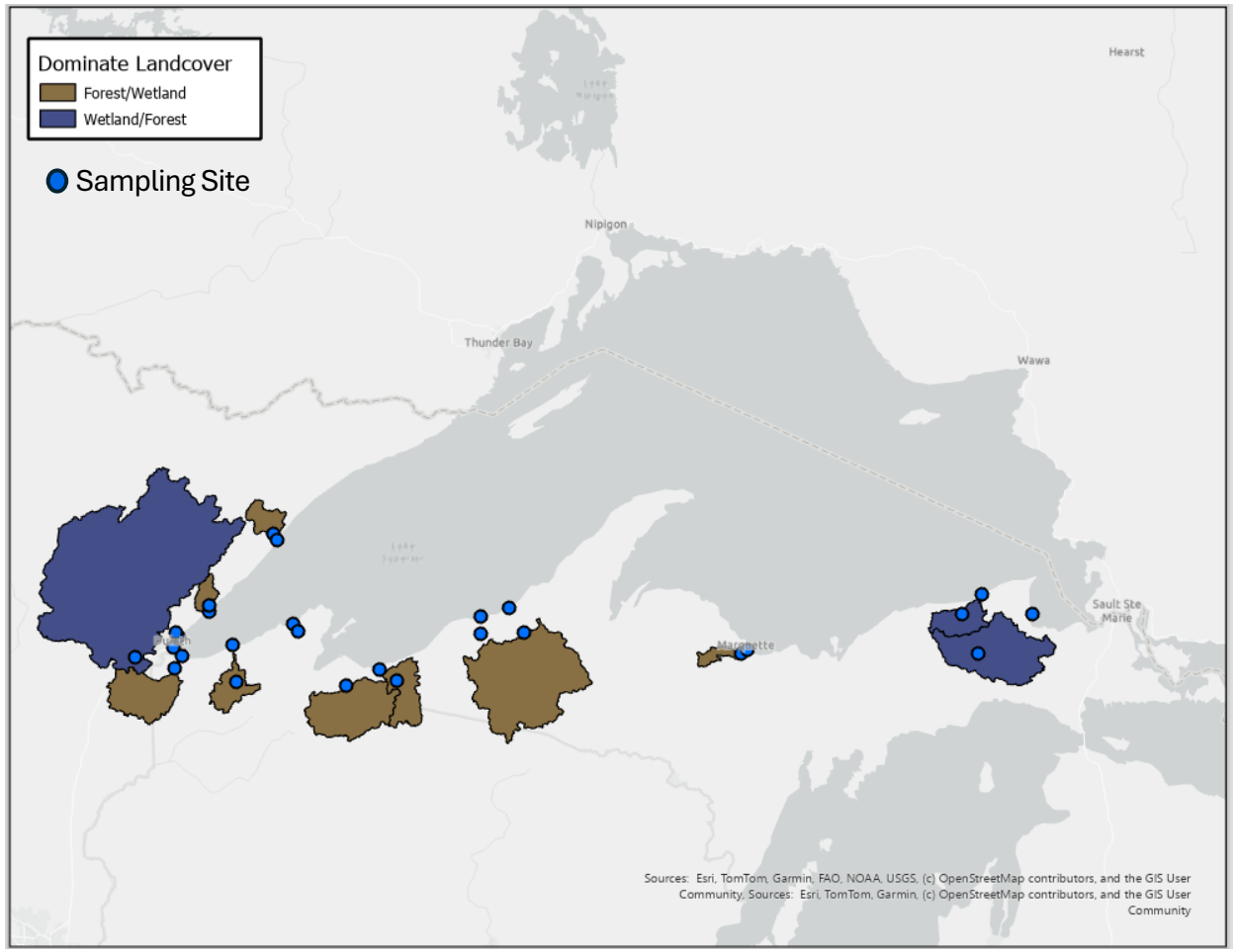
14 tributaries across MN, WI, MI

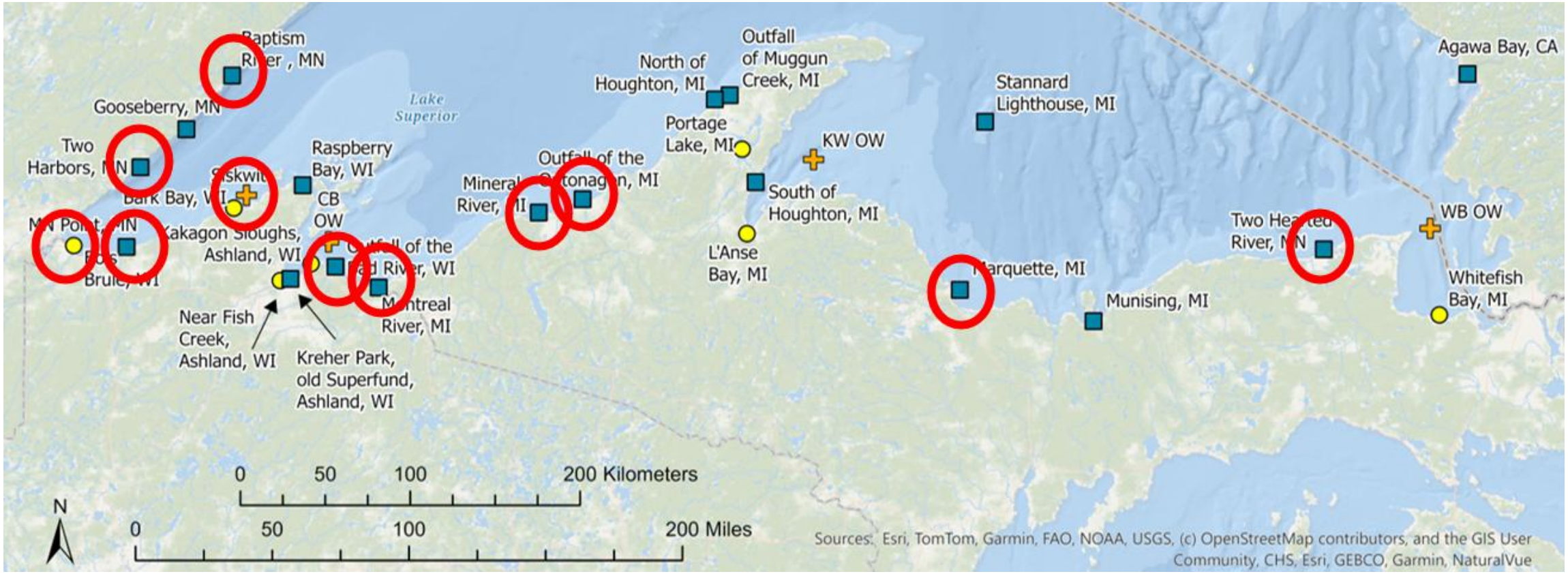
2 sites per tributary (upstream and mouth)

Surface water and sediment collections

Tributaries of Interest

Baptism River
Knife River
Tischer Creek
St. Louis River
Bad River
Nemadji River
Bois Brule River
Siskiwit River
Montreal River
Mineral River
Ontonagon River
Carp River
Two-Hearted River
Tahquamenon River



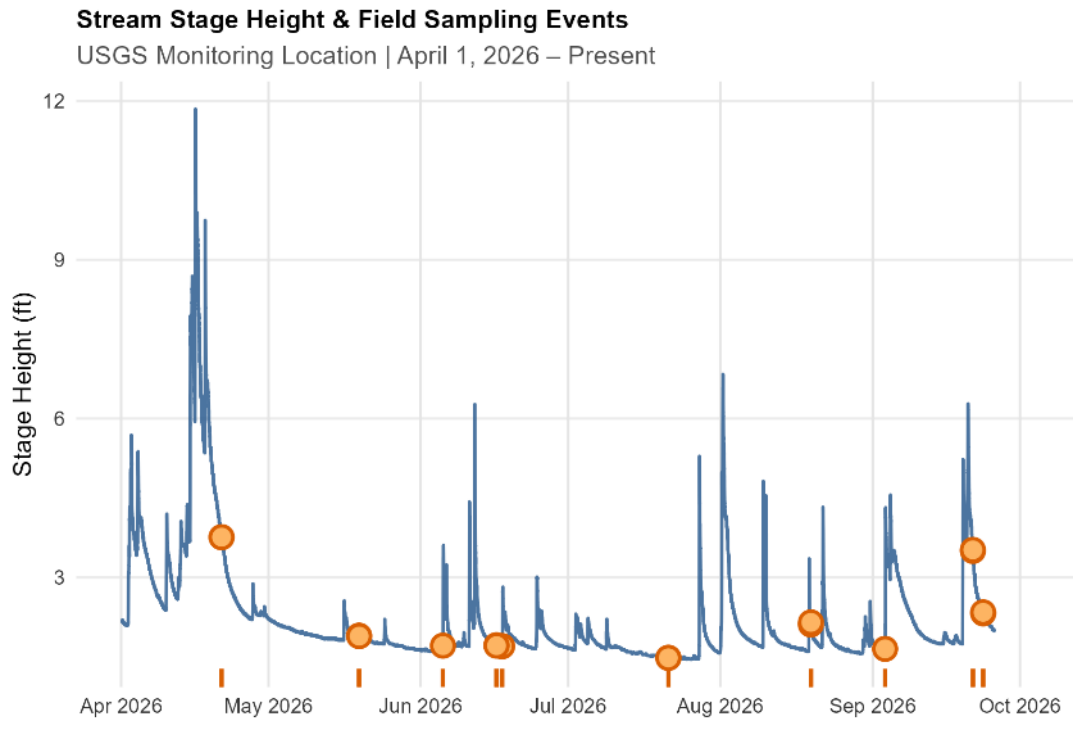


One-time sampling event on the LEII vessel. Sites selected to pair with upstream and downstream measurements at tributaries

MP Loads to Lake Michigan from a Milwaukee Tributary

- Partner project with the Milwaukee Metropolitan Sewerage District to determine MP loading to Lake Michigan
- Menominee River
- Monthly sampling + sampling of hydrologic events throughout the year
- Started April 2026, 3-year project





MPs Across Rural-Urban National Parks Gradient

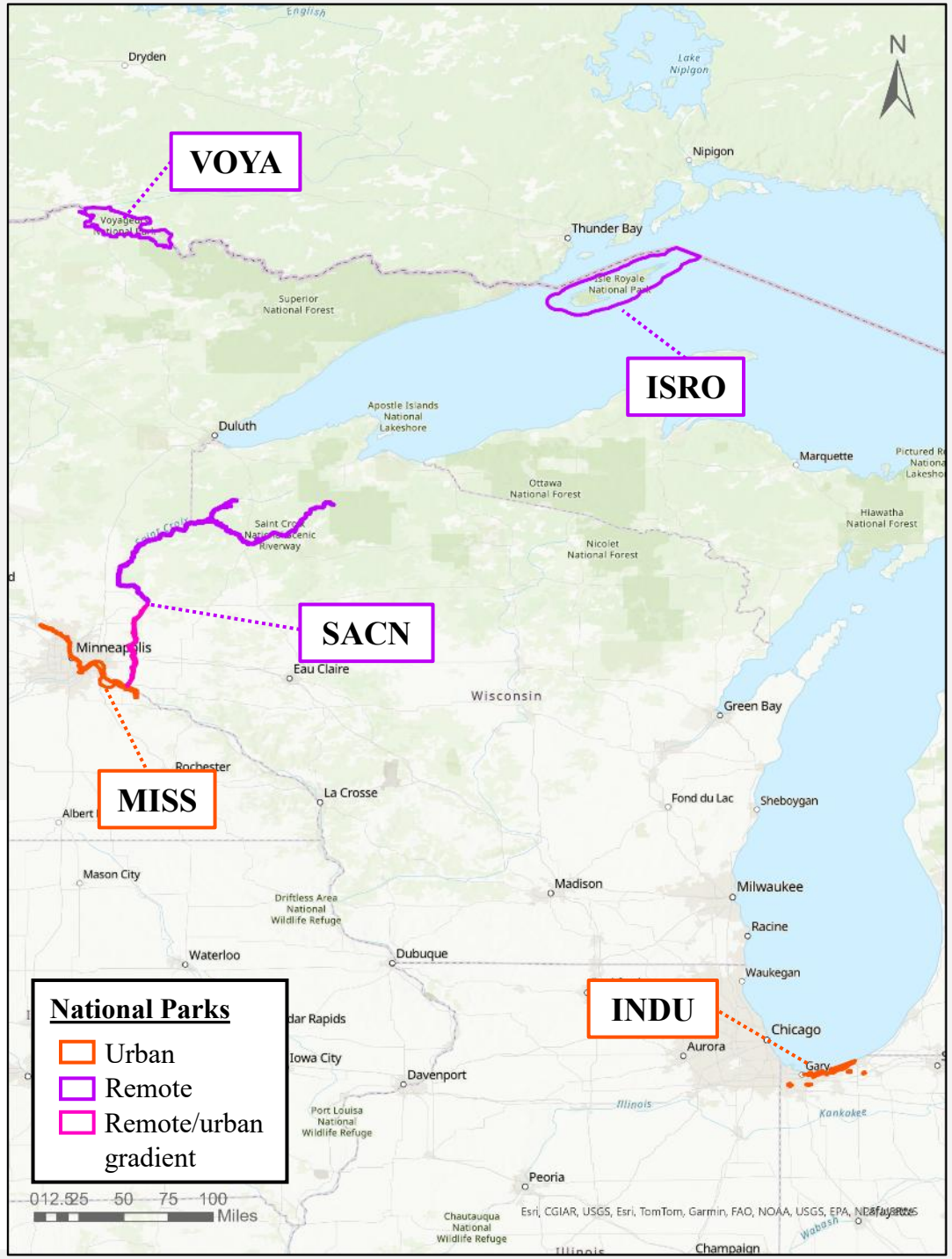
- Collected dragonflies and surface water samples for MP determinations in 5 Great Lakes region national parks
- Work done in conjunction with ongoing Dragonfly Mercury Project

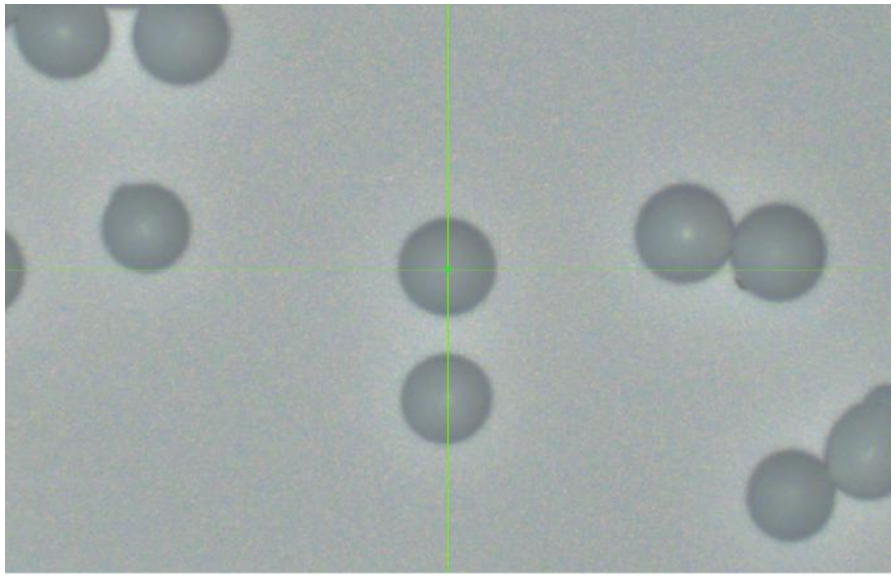
Aeshnidae



Gomphidae







Conclusions



- LDIR technology allows for whole sample microplastics analysis down to 20 µm and gives important information about sizes, shapes, and types of plastics present.
- Future work with Raman spectroscopy will fill in the gap for nanoplastics and small microplastics
- Integration of MP sampling into national monitoring networks could provide invaluable information about the distribution and transport of MPs in groundwater across the country
- Future and ongoing projects hope to start filling in knowledge gaps surrounding fate and transport of MPs

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