Microplastics measurement interlaboratory calibration study

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Background

- SB 1422 (fall 2018) requires microplastic monitoring in drinking water starting in 2021
  - SB 1263 requires statewide management strategy for microplastics in coastal waters

- This is a significant challenge
  - Achieving mandates requires adoption of state-approved measurement methods
  - Methods must be scientifically sound, and technologically and logistically feasible

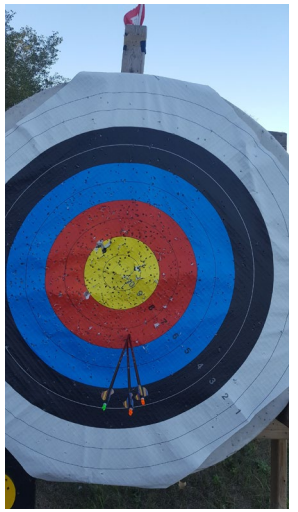
Scientific needs

- Standard Operating Procedures
  - Everyone uses their own, so hard to compare results
- Understanding method performance
  - How to select among methods
  - How to interpret results
- Accreditation for laboratories

SCCWRP intercalibration study foundation

- Measure known **blind samples** processed by participating labs
  - Using **standard methods** for several candidate methods
  - Quantify **accuracy**: differences from knowns as function of parameters
  - Quantify **precision**: repeatability
- Quantify technical method **capabilities** and **limitations**
  - From same laboratory
  - From experienced laboratories
  - From labs with different levels of experience
- Quantify **feasibility** by tracking resources needed
  - Personnel time to implement
  - Cost of expendable supplies
  - Capital costs for equipment

accuracy

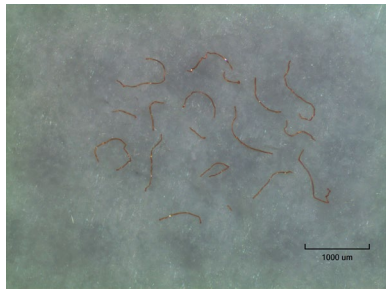

precision

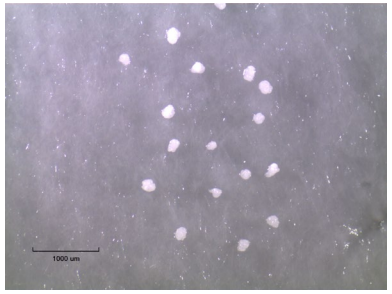
Five major methods used

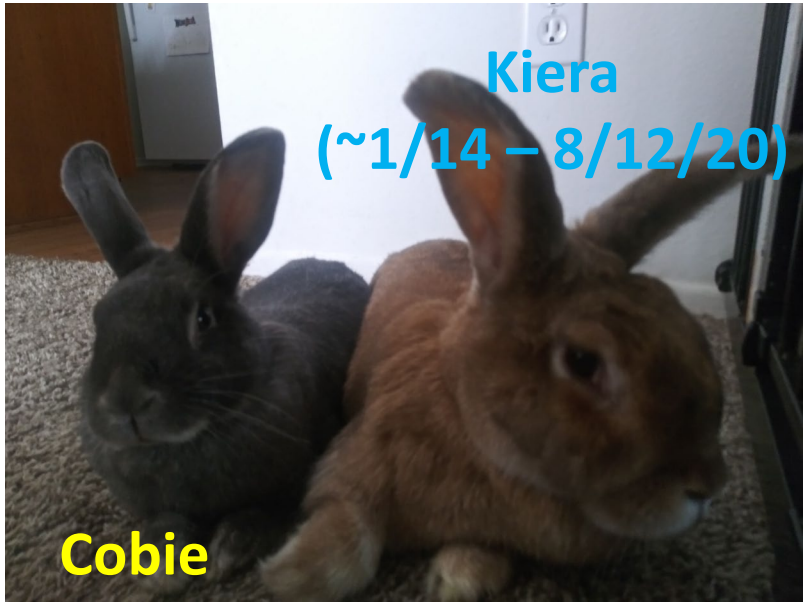
- SCCWRP workshop in April 2019 invited experts to select candidate methods, and draft SOPs
  - Visual microscopy
  - Visual microscopy with fluorescence staining (Nile Red)
  - Fourier-transform infrared spectroscopy (FTIR)
  - Raman spectroscopy
  - Pyrolysis gas chromatography-mass spectrometry (pyro)

Blind samples

- Several types of polymers
  - Polystyrene, polyethylene, PVC, PET
- Four size fractions
  - 1-1000 um
  - 1-20 um, 20-212 um, 212-500 um, >500 um
- Several morphologies
  - Pellets, fragments, spheres, fibers
- False positive materials
  - Look like synthetic polymers, but aren't
  - Examples: sand, shell fragments, natural fibrous material (cotton, cellulose, bunny fur)







Matrices for blind samples

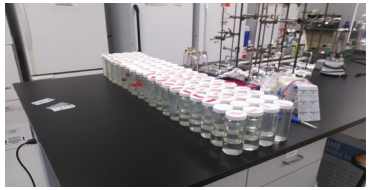
- Clean water matrix
  - Proxy for drinking water
  - Lab work and analysis complete
- Dirty water matrix
  - Proxy for surface water
- Sediment matrix
- Fish tissue matrix

Data submission at end of May

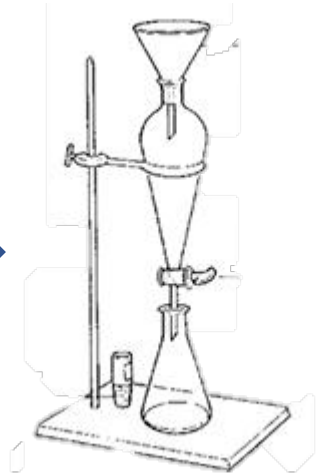
Participating labs

- 40 participating laboratories in 6 countries
- Mix of academic, government (federal, state/provincial, county, municipal), and private-sector labs (industry and consulting)
- Highly experienced labs to novice organizations
- 3-22 laboratories per method

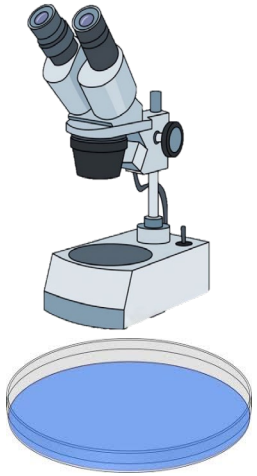
General flow of lab work



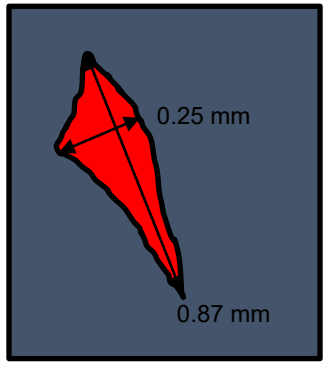
Blind Samples



Particle Extraction



Particle Identification & Categorization

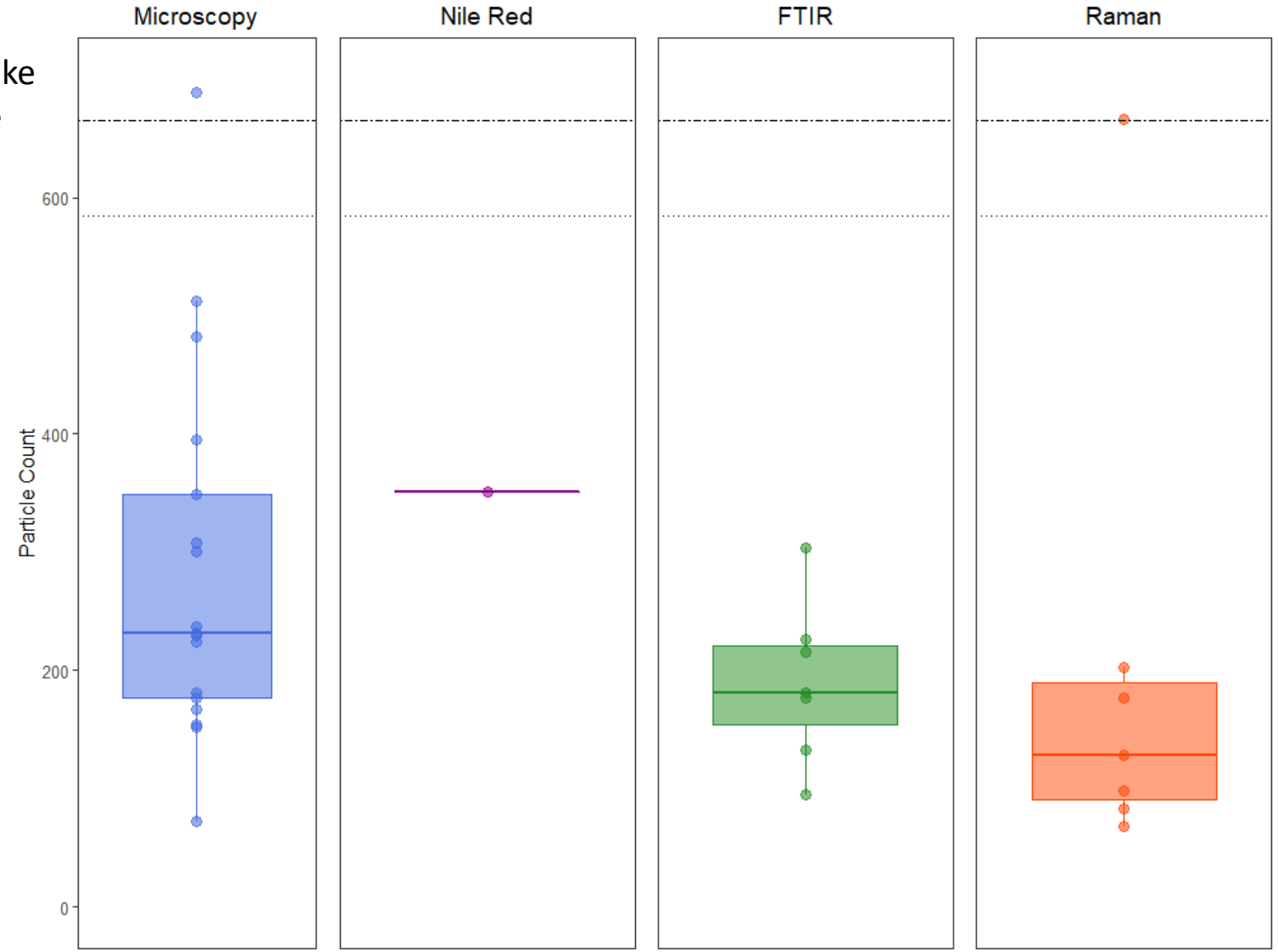


Pictures & Measurements

Chemical ID Confirmation

The big (initial) picture for clean water matrix

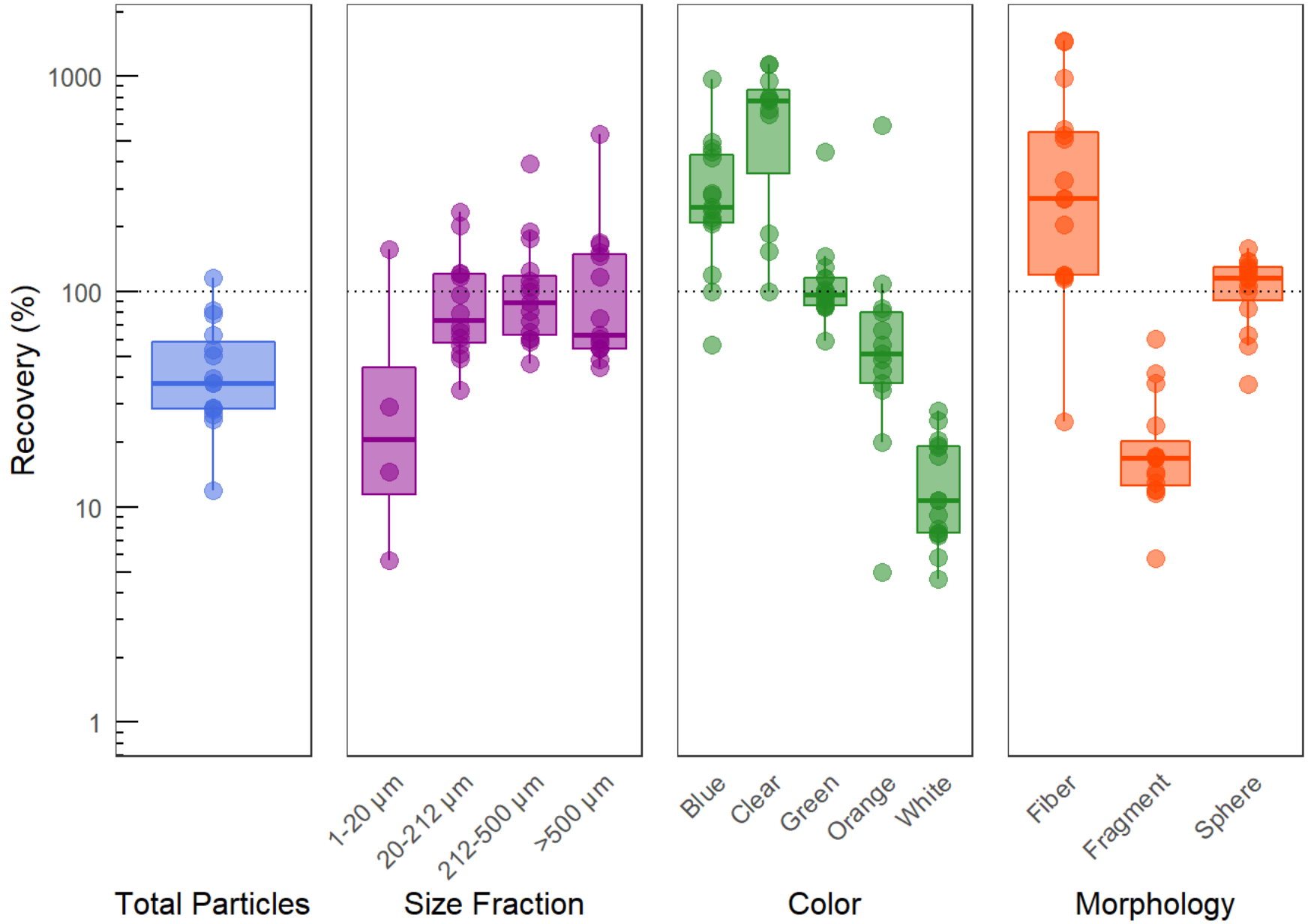
Total particle spike (plastic + false positives)



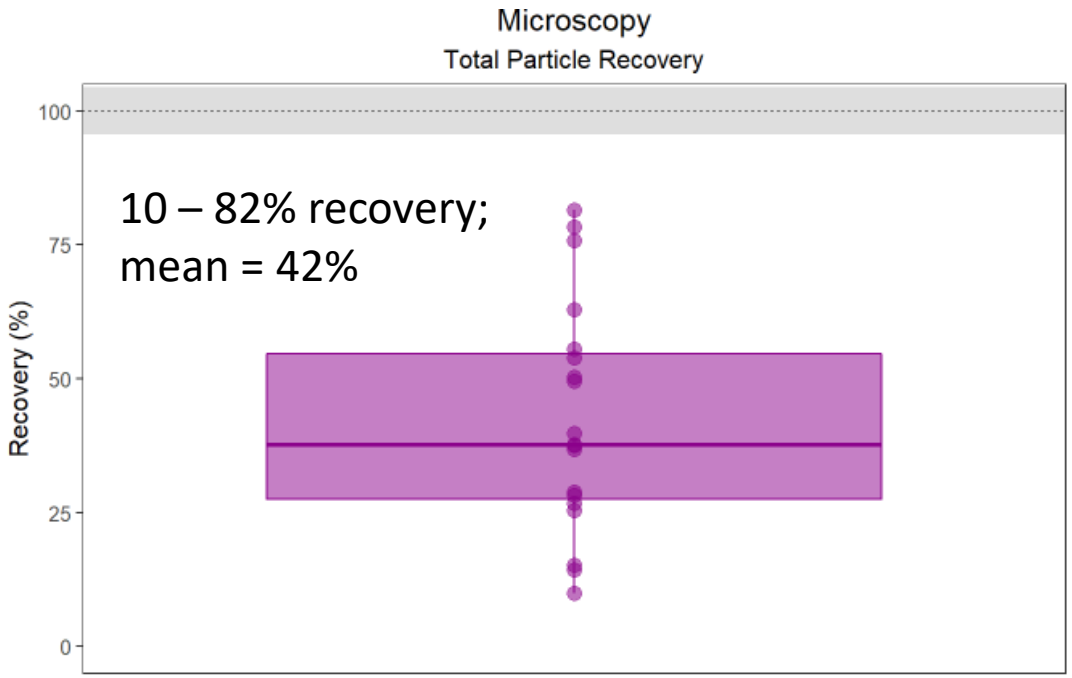
Total plastic particle spike

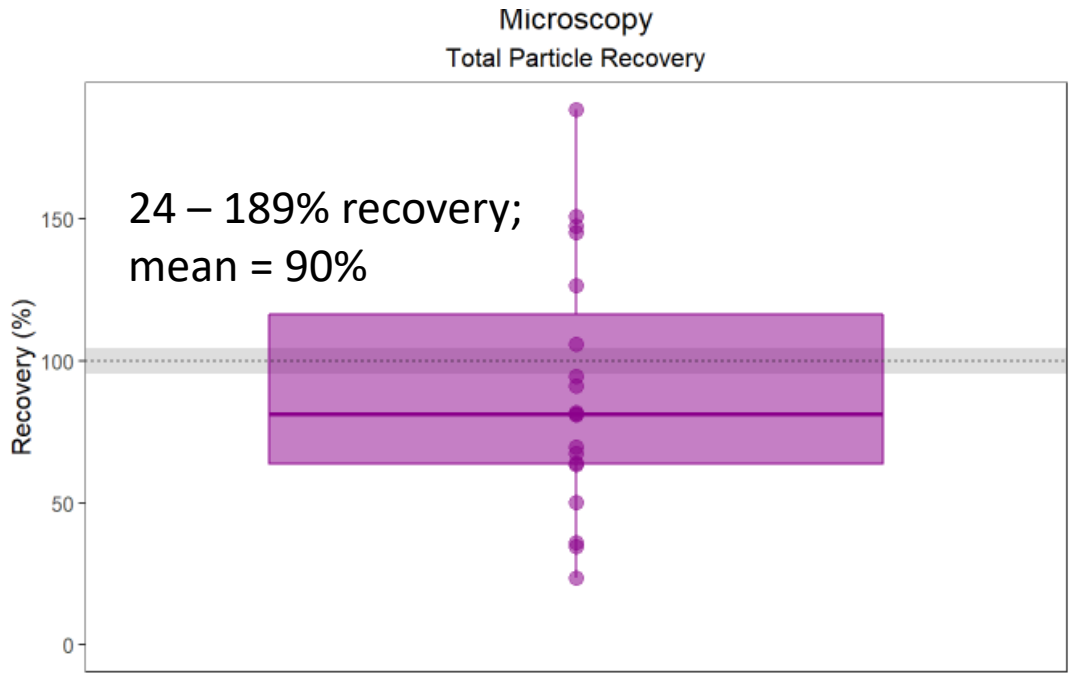
Initial performance at a glance

- It’s not poor accuracy & precision across the board!



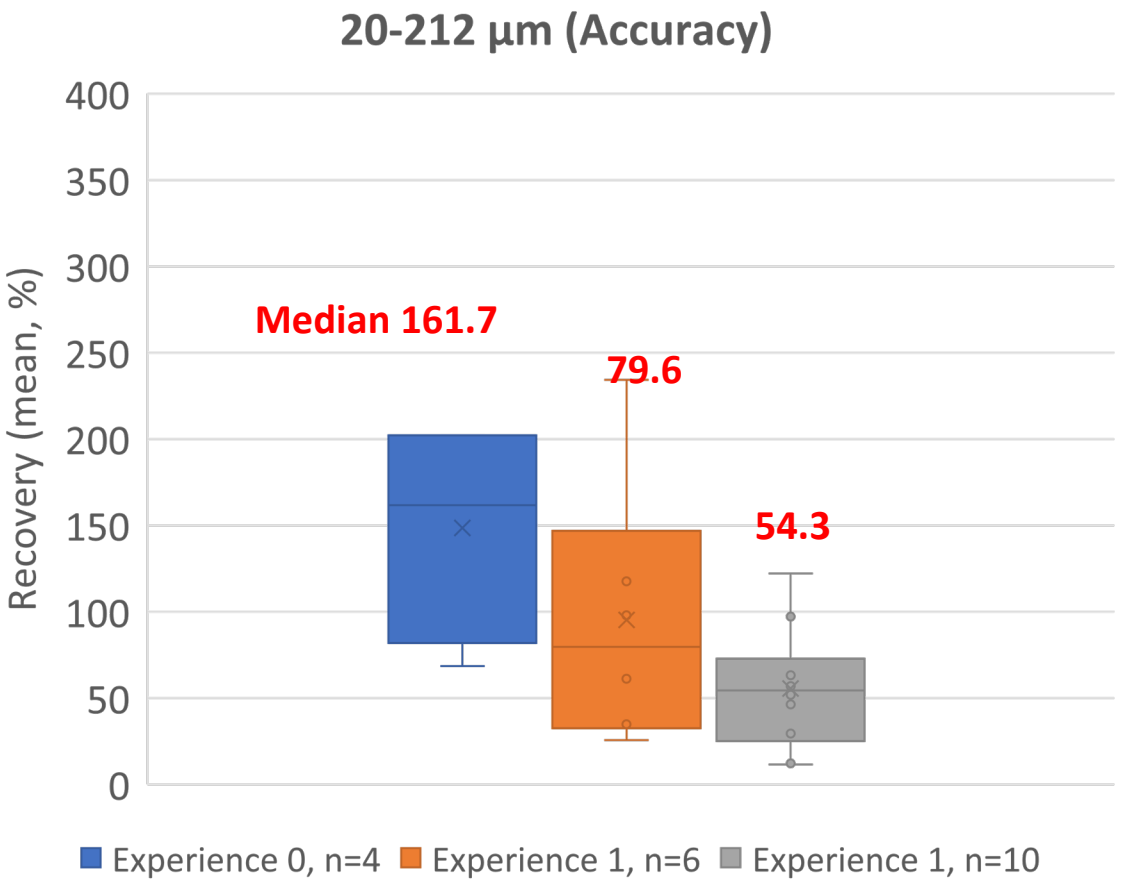
Recovery much more accurate for size fractions >20µm

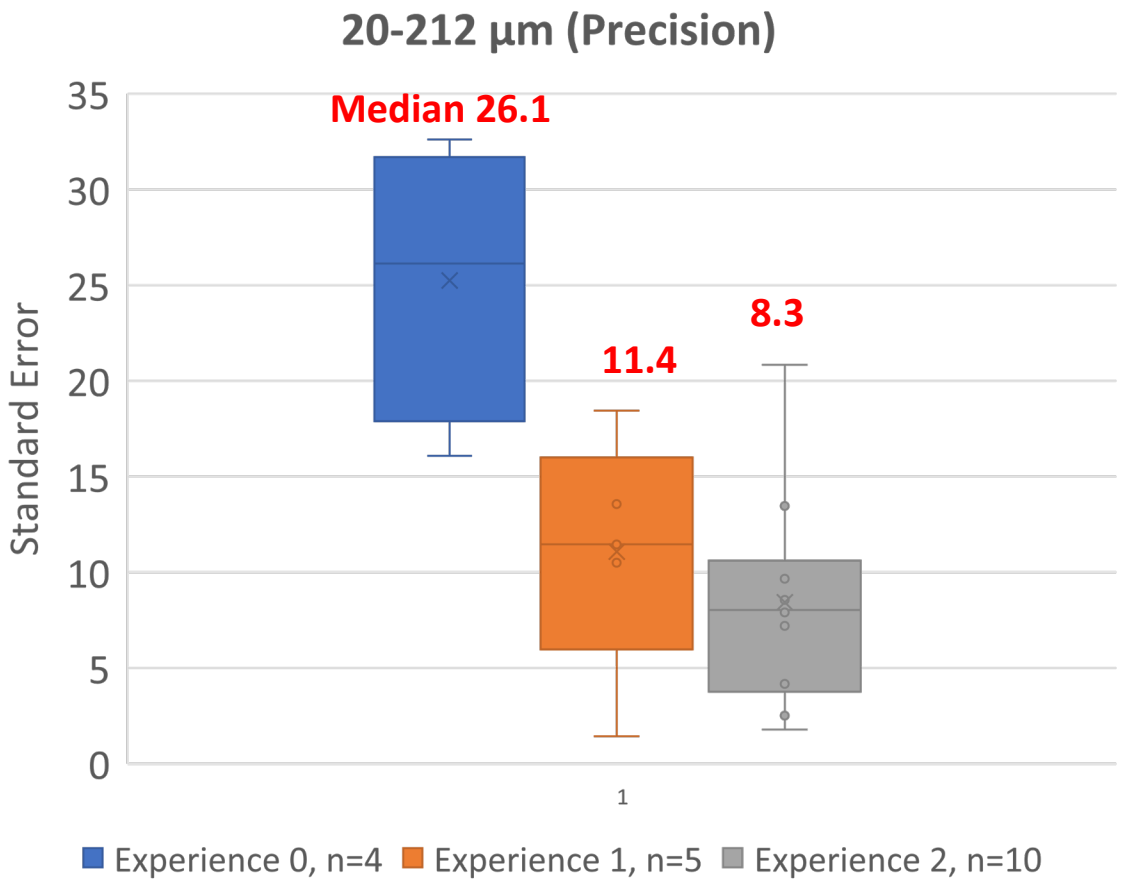




Recovery with (*left*) and without (*right*) inclusion of 1-20µm size fraction

Experience matters across the board!





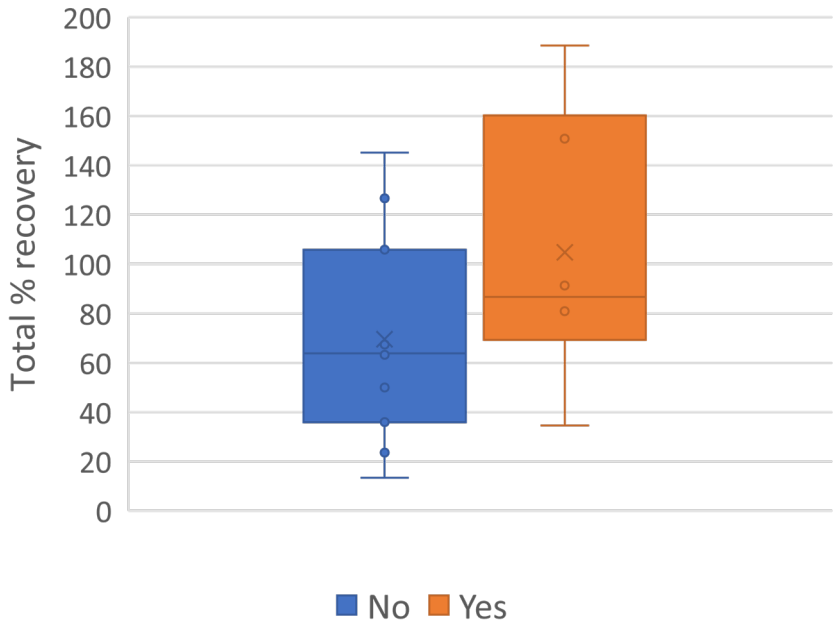
Level 0

Level 1

Level 2

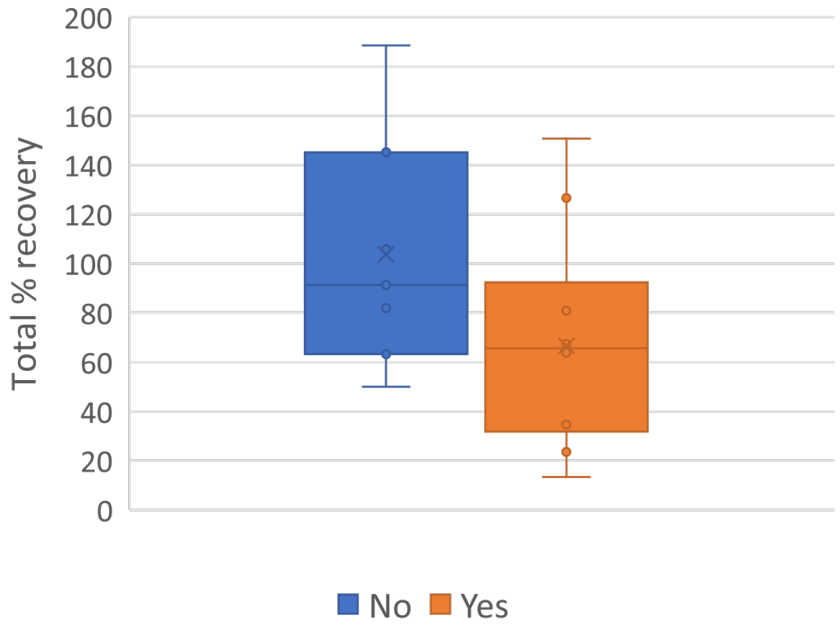
Training at SCCWRP and following the SOP improved recovery

Did you train at SCCWRP?



>20µm size fractions

Did you deviate from the SOP?



>20µm size fractions

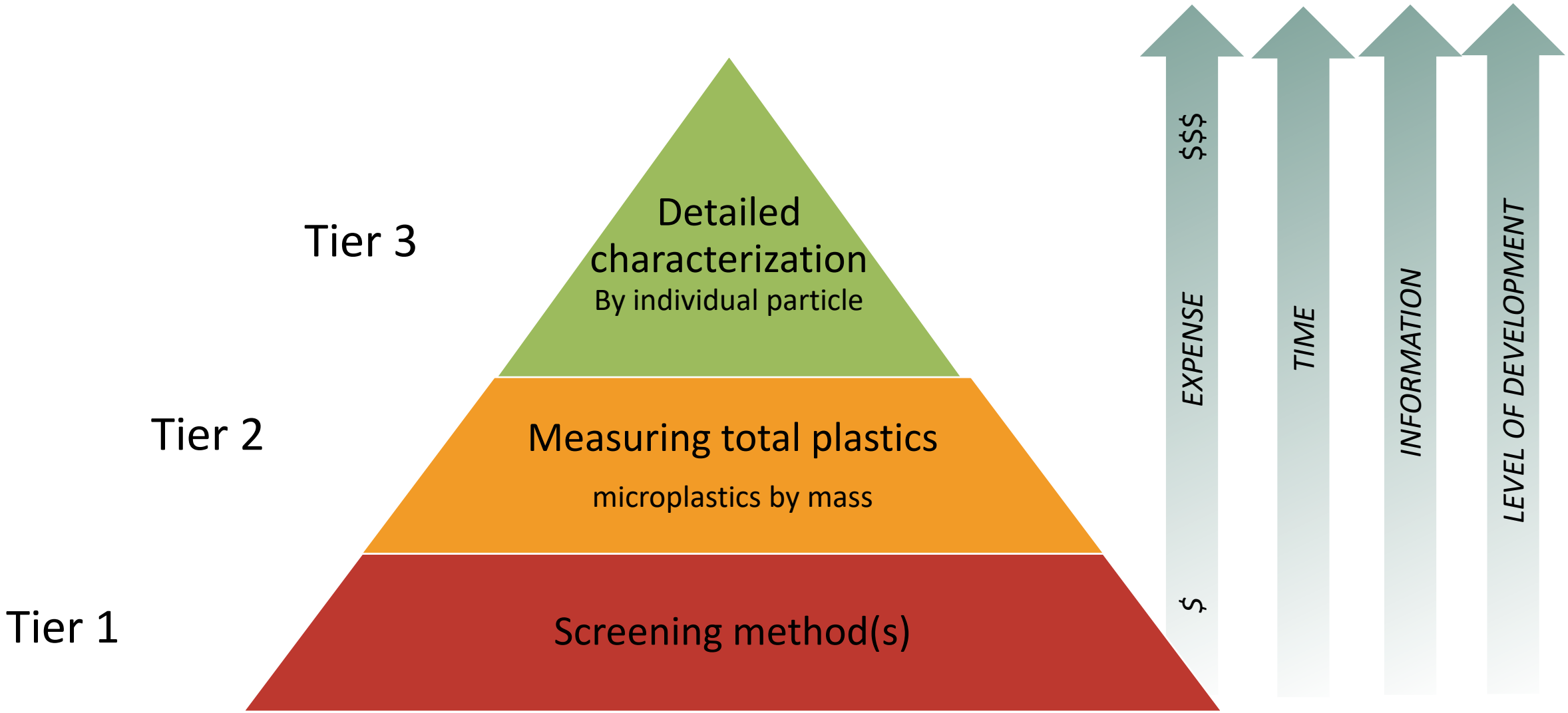
Products from SCCWRP intercalibration study

- Performance characteristics for measurement methods
- SOPs for methods
  - Now refined by participating labs to achieve consensus
- Accreditation needs for labs doing monitoring work
  - We understand performance characteristics
  - We know what a good lab can achieve
  - We work with ELAP to develop this

How long?

Mean time/sample (hr)	Microscopy	FTIR	Raman	Pyro-GC/MS
Filtration	15	15	15	15
Counting	27	27	27	N/A
Size measurements	9	9	9	N/A
Polymer identification	N/A	10	18	5
Total (hours)	**51**	**61**	**69**	**26**

Potential Tiered Monitoring Framework



Based on Cousins et al. (2020). *Environmental Science: Processes and Impacts*

Tier 2 methods

- We are currently evaluating several potential Tier 2 methods
  - Gets total mass of plastic in sample
  - Loses particle-specific information

- Pyrolysis GC/MS
  - Doable in 5 hours vs. 10-18 for particle-specific spectroscopy
  - 5 labs in intercal study now evaluating

- Bulk FTIR and touch Raman
  - Chemical ID in only a fraction of time for particle-specific spectroscopy
  - Limited to larger particles only (>200 um)
  - Working with instrument vendor partners (Horiba, Thermo) to finalize results

Tier 1 screening methods

- SCCWRP and participating labs have identified about a dozen potential Tier 1 screening methods
  - Are levels above or below a threshold number?

- Evaluating these methods for possible tiered monitoring framework is a future research direction

What's next?

- Disseminate results from clean water matrix
  - Special Issue of journal *Chemosphere* dedicated to this (August)
  - Presentation by Dr. Scott Coffin to State Water Board (August)
- Data analysis and interpretation for other matrices (this fall)
- ELAP accreditation development (this fall)