Microplastics measurement intercalibration study

Charles S. Wong[1], Scott Coffin[2], and Steve Weisberg[1]

[1]Southern California Coastal Water Research Project Authority

[2]California State Water Resources Control Board

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

Challenges

- Developing standard methods is often a **very long** process
  - Which method(s) to standardize?
  - What are procedures for standardization, including data management?
  - Perform inter-laboratory studies to quantify method performance
  - Refine methods to reduce variability and repeat

- Not clear yet what to measure (size, shape, polymer type, etc.)

- Little consensus yet in measuring microplastics
  - Legislative requirement is a way to meet significant need for consensus
  - Difficult to provide big-picture assessment otherwise
  - But challenging to do in (original) 2-year timeframe!

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

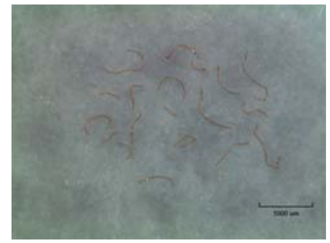

accuracy

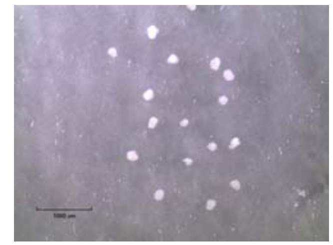

precision

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

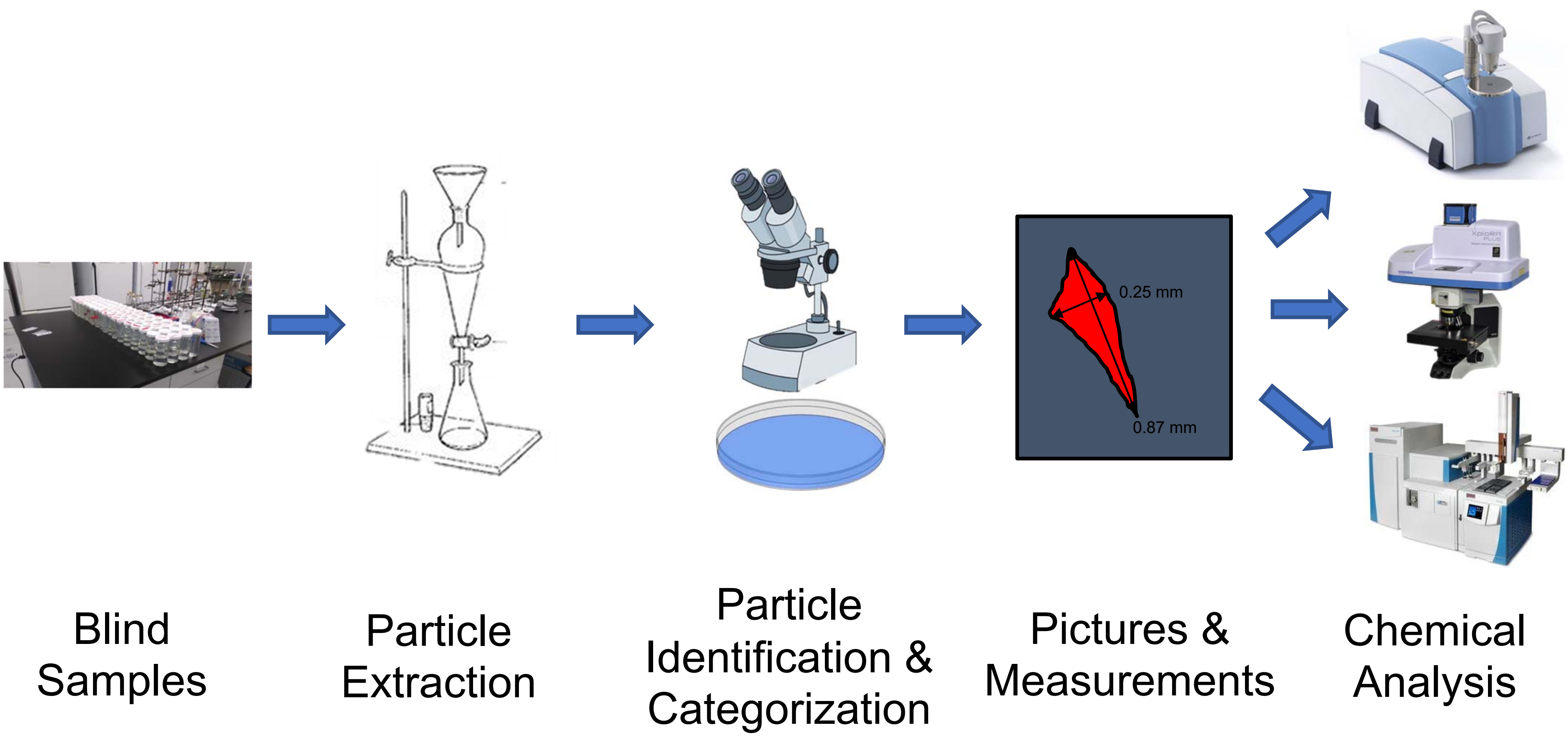
- Fish tissue matrix

Data submitted end of May
data analysis and interpretation
in progress

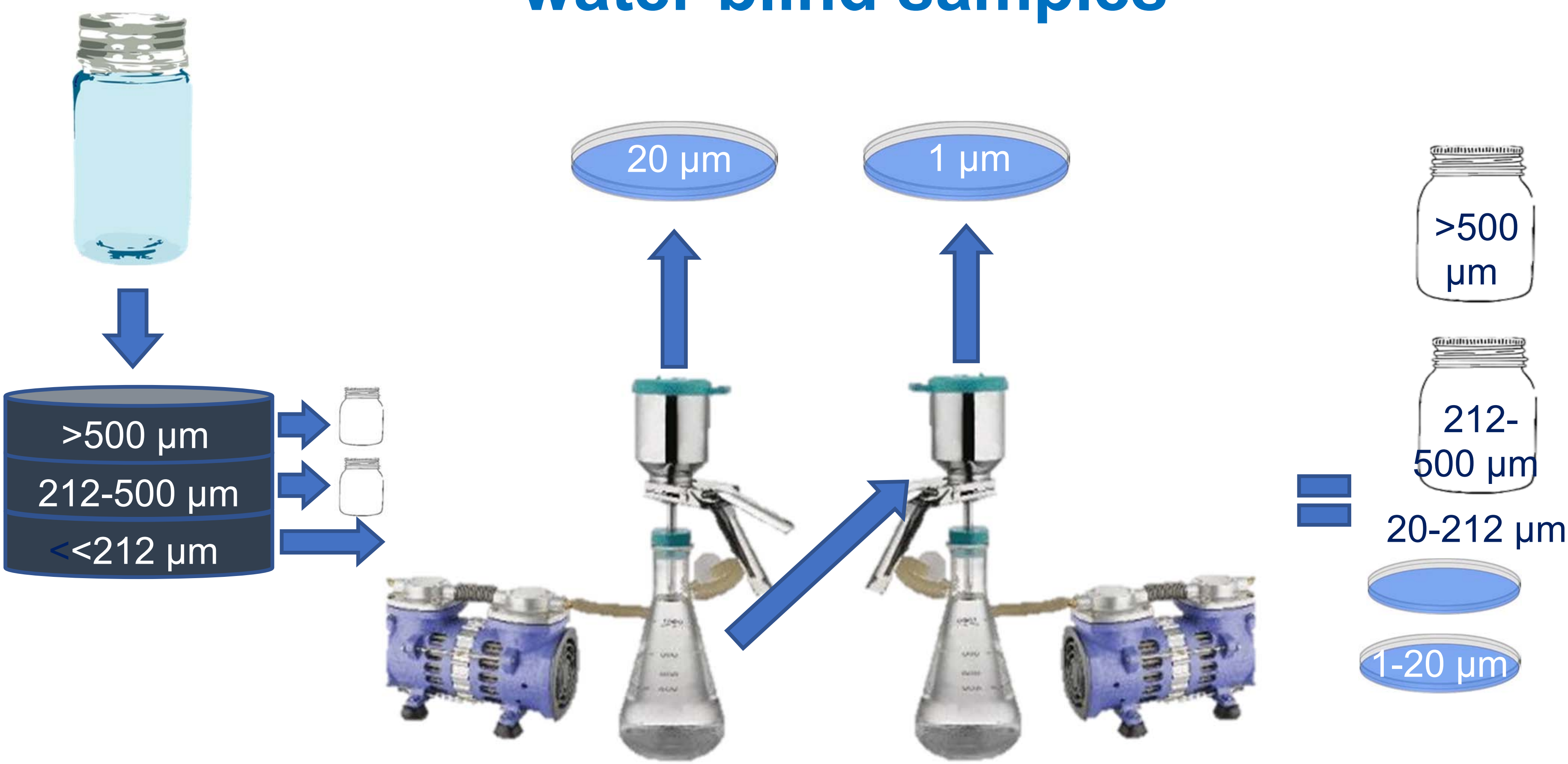
Participating labs

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

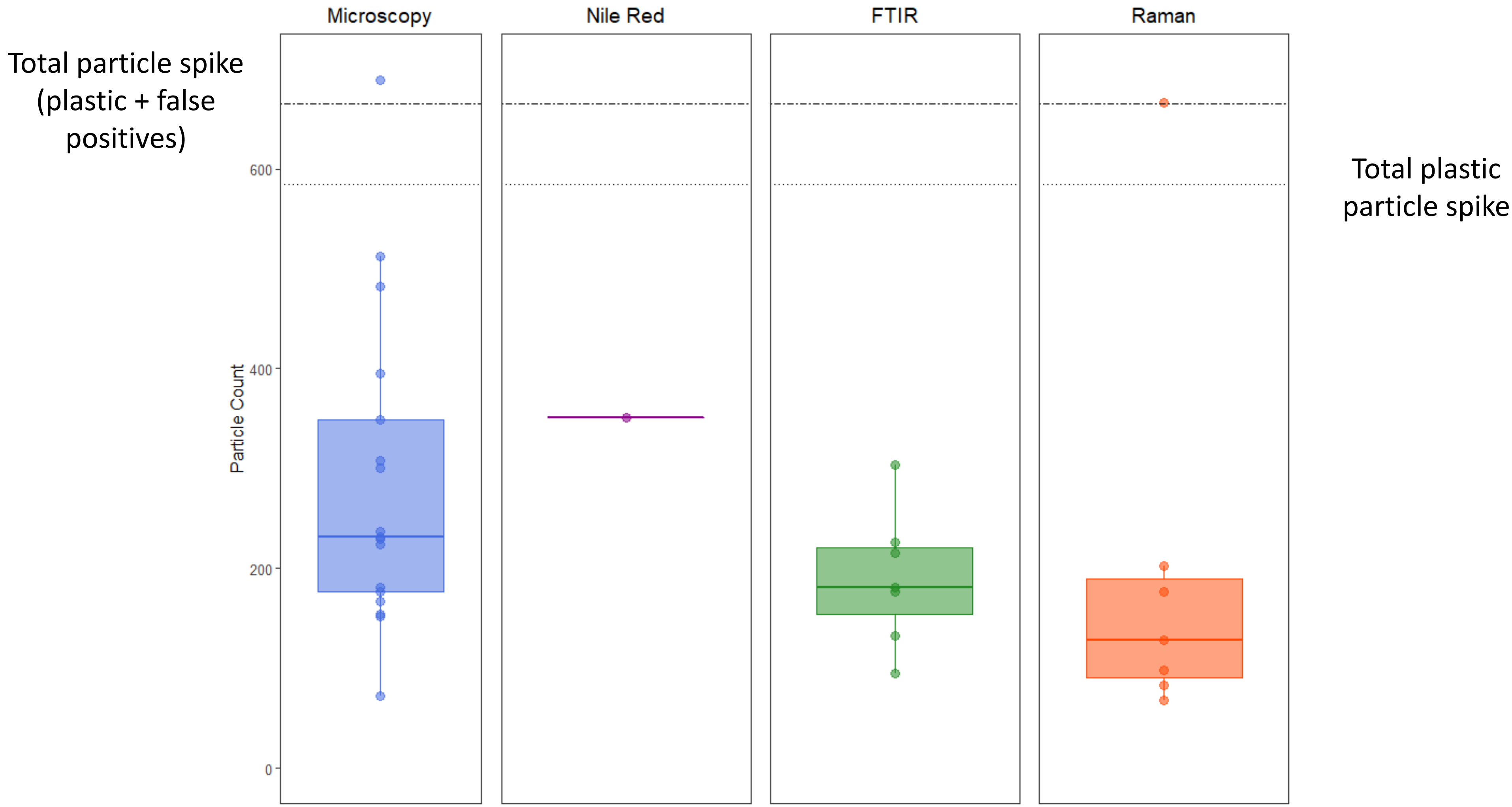
General flow of lab work



SOP for processing simulated clean water blind samples

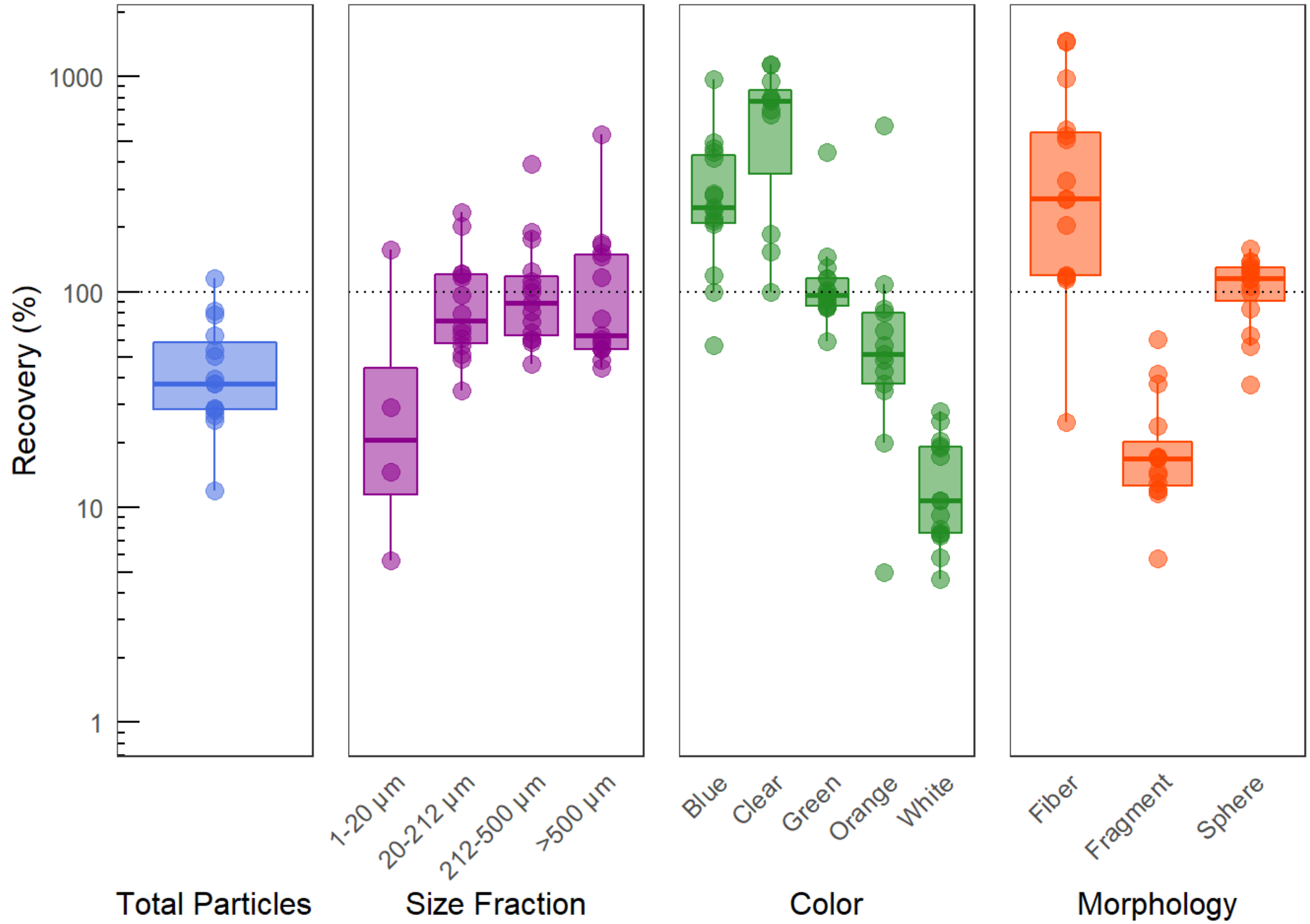


The big (initial) picture for clean water matrix

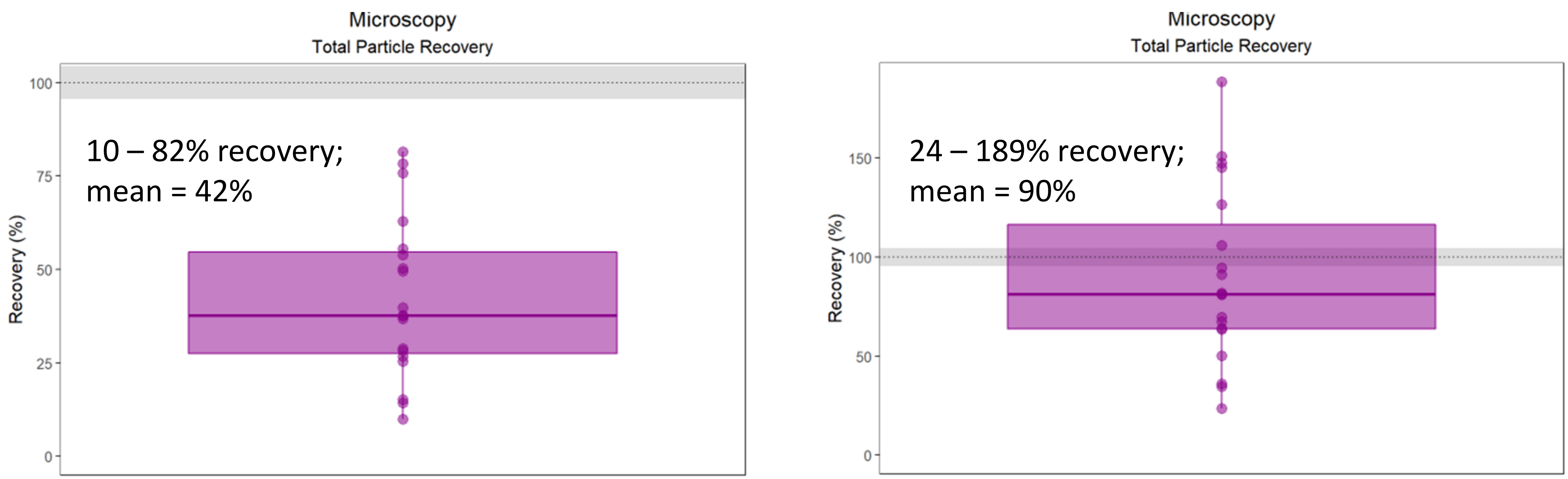


Initial performance at a glance

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

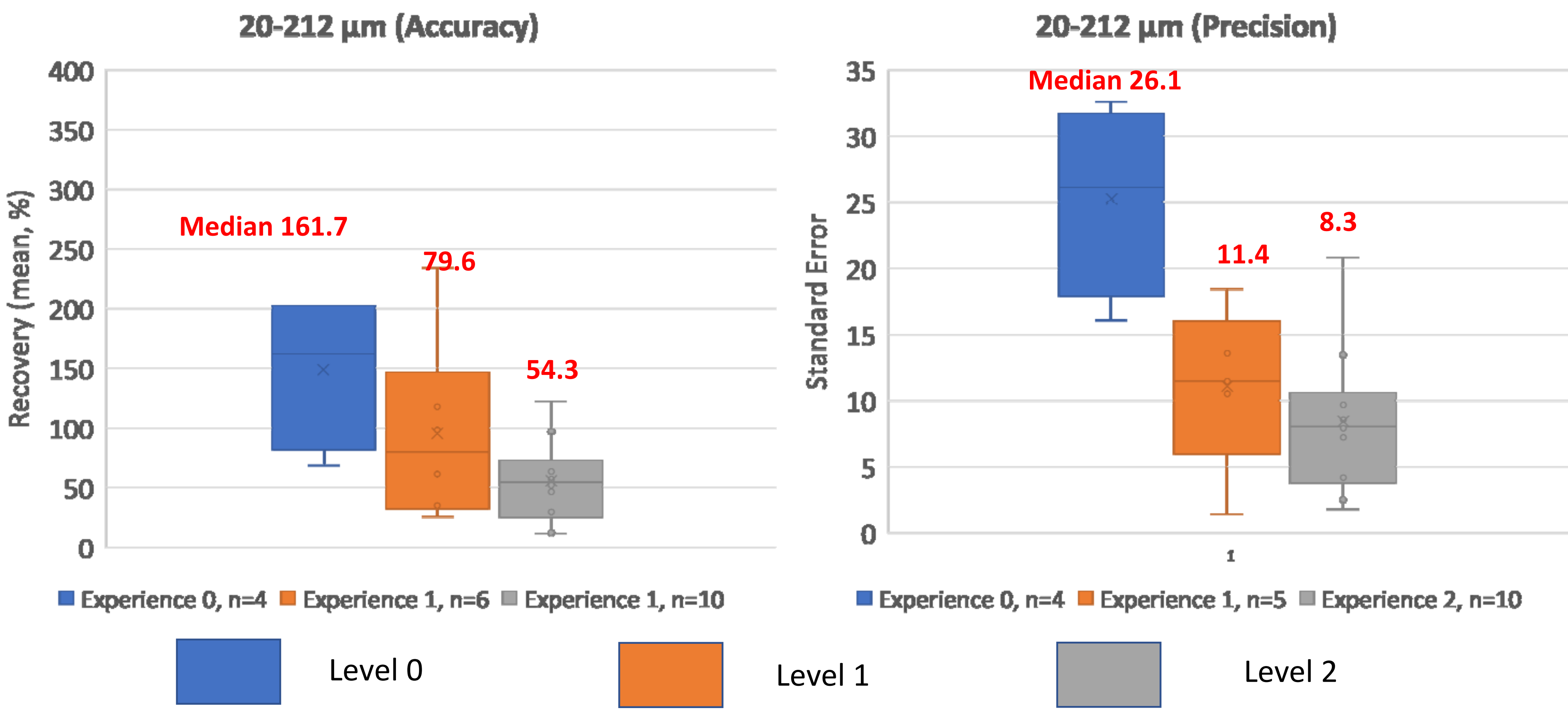


Recovery much more accurate for size fractions >20µm



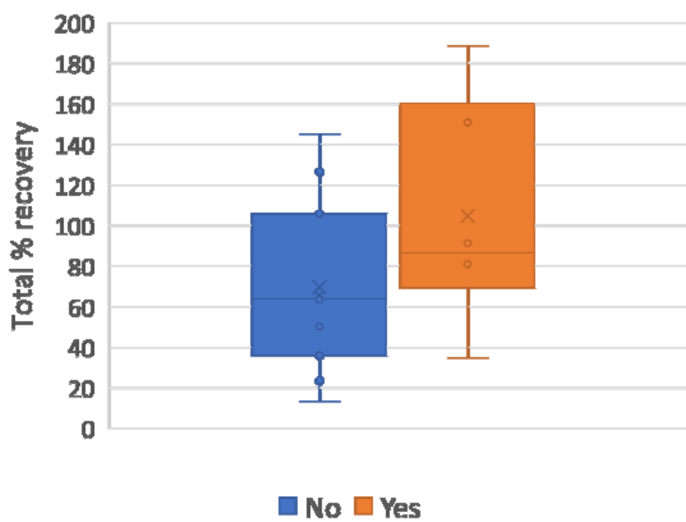
Recovery with (*left*) and without (*right*) inclusion of 1-20µm size fraction

Experience matters across the board!



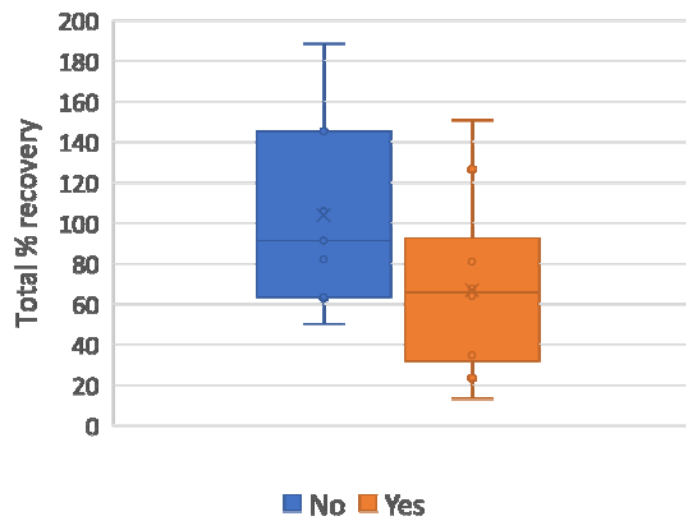
Training at SCCWRP and following the SOP improved recovery

Did you train at SCCWRP?



>20µm size fractions

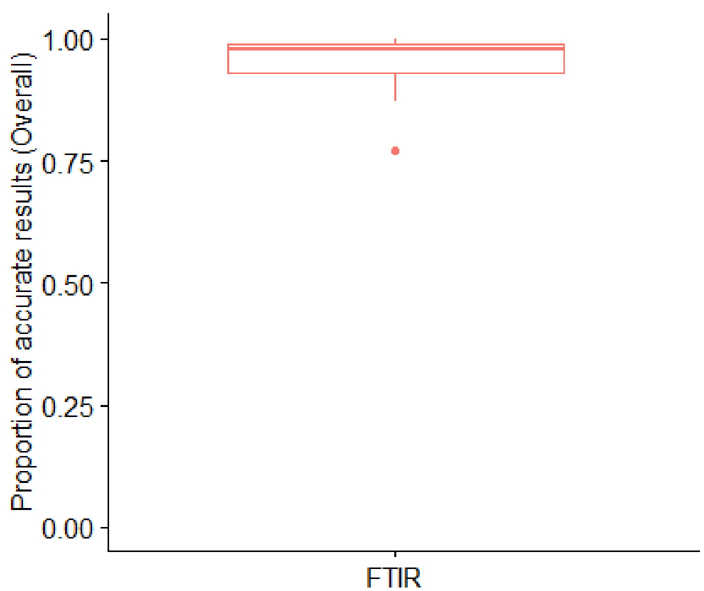
Did you deviate from the SOP?

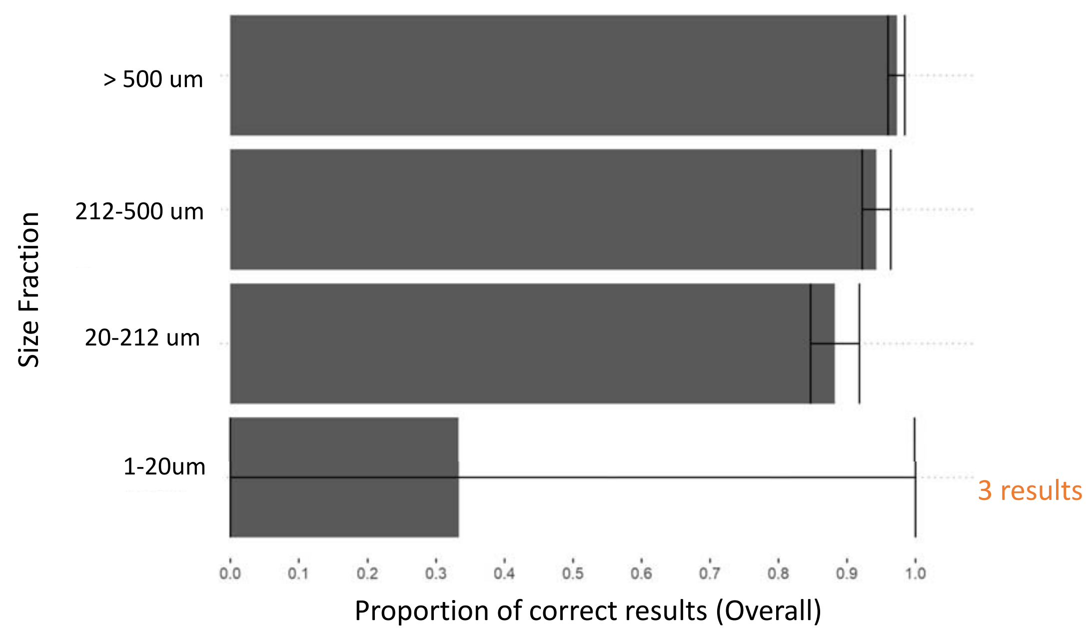


>20µm size fractions

Accuracy of FTIR spectroscopy for chemical ID

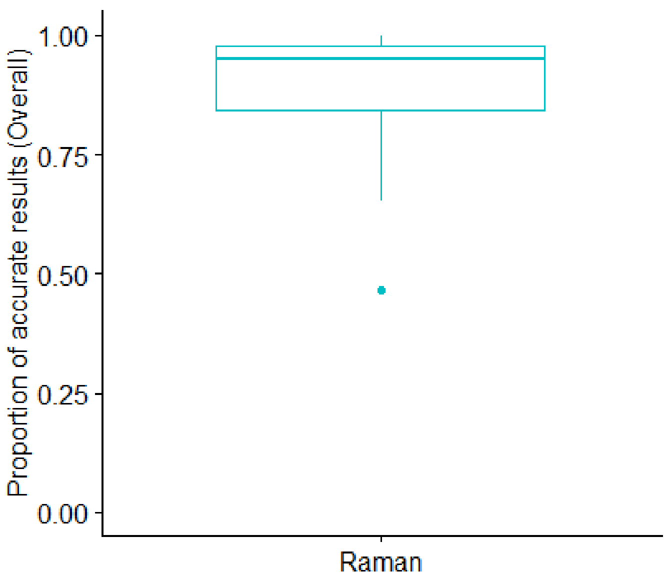
- High overall accuracy (plastic and natural combined), 95%
- Highly accurate ID of all spiked polymer types (>90%)
- Novice labs can get accurate results
- Accurate ID down to 20 um

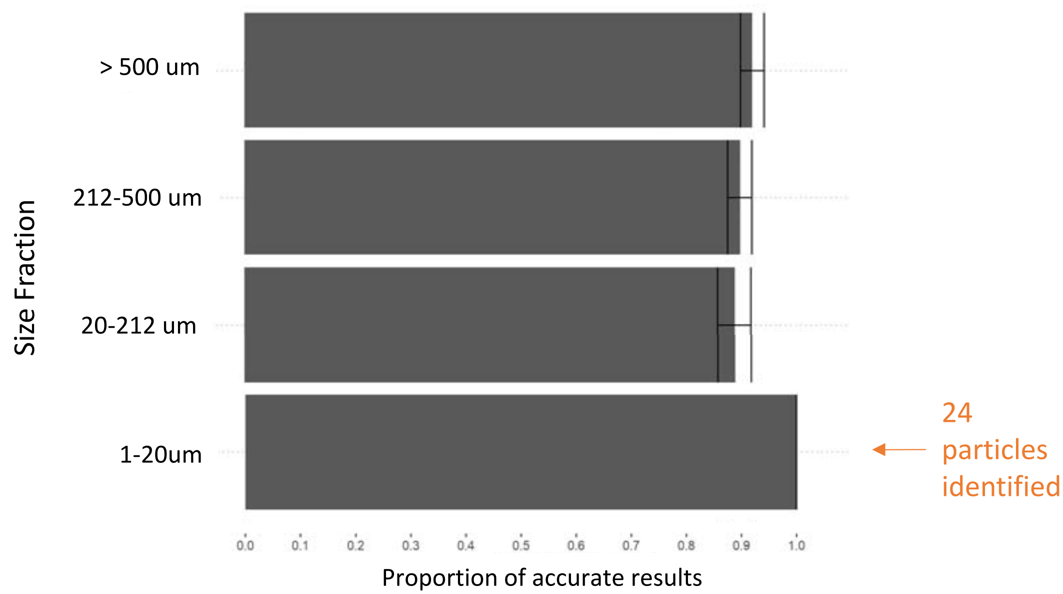




Accuracy of Raman spectroscopy for chemical ID

- Overall accuracy (results for plastic and false positives combined): 86%
- Highly accurate for identifying plastic: 91%
- Novice labs can get accurate results
- High accuracy among all size fractions 84-100%





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

Other matrices

- Drinking water is only one aspect of intercalibration study
  - Parallel Core Study and Augmentations on simulated surface “dirty” water, sediment, fish tissue

- Helps address SB 1422 for statewide strategy to manage microplastics in coastal waters

- Data submitted by participants at end of May

Accreditation

- Need to ensure that labs can acceptably process samples for microplastics
  - Utilities
  - Contract labs
- Part of process is working with California Environmental Laboratory Accreditation Program (ELAP)
  - Recommendations for certifying labs to analyze microplastics in drinking water
  - Inspections, recordkeeping, Performance Evaluation Samples
  - Training of ELAP staff on how these steps in lab certification pertain to microplastics

What's next?

- Disseminate results from clean water matrix
  - Special Issue of journal *Chemosphere* dedicated to this
  - Presentation by Dr. Scott Coffin to State Water Board (September)

- Data analysis and interpretation for other matrices (mid-August)
  - Manuscript in *Chemosphere* Special Issue

- ELAP accreditation development (this fall)

Thank you!

- For more information on the SCCWRP microplastics measurement workshop in 2019:
  - https://www.sccwrp.org/about/research-areas/additional-research-areas/trash-pollution/measuring-microplastics-workshop/