STANDARDIZING METHODS AND ACCEDITATION FOR MEASURING MICROPLASTICS IN CALIFORNIA

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BACKGROUND

- **California passed legislation that requires microplastics measurements in drinking water beginning in 2021**
  - They also passed legislation to develop a microplastics ocean litter strategy, which will require ambient water sampling
- **Achieving those mandates requires adoption of state-approved measurement methods**
- **We also need to develop an accreditation process for laboratories implementing those methods**
  - California requires use of accredited labs for generation of any data being used for regulatory decisions

WE HAVE A CHALLENGE

- **Developing standard methods is often a decade long process**
  - Decide which method to standardize on
  - Agree on and codify detailed procedures for that method, including data management requirements
  - Perform inter-laboratory studies to quantify method performance
  - Refine methods to reduce their variability and repeat steps above

- **The microplastics world has many classes of methods in active use**
  - Light microscopy with staining
  - Fourier transformed infrared spectroscopy
  - Raman spectroscopy
  - Pyrolysis gas chromatography
  - Others in earlier stages of development

- **We haven’t even agreed on our targets to measure**
  - Size
  - Shape
  - Plastic types (e.g. polyethylene vs. polystyrene)

CHALLENGE ACCEPTED

- **The legislative requirements are onerous, but they have the potential to be a good thing**
  - They give us impetus to move the field forward quickly
- **The scientific community is ready**
  - At every scientific meeting we talk about the need to standardize methods
  - The present diversity of approaches and differing quality assurance protocols limits our ability to integrate results and provide a big-picture assessment
- **California provides us a forum for making a decision**
  - Groups like the American Society for Testing and Materials are also a great option, but California is providing a first set of directed application needs
  - They also create a timeline demand

STEPS WE ARE TAKING

- **California has developed a draft definition of microplastics**
  - Gives us the target we are aiming to measure
  - That definition is presently out for technical review
- **Held a workshop last April to achieve agreement on what methods the State should consider**
  - Used the workshop to develop first-draft standard operating procedures for each method
- **We are about to implement a method evaluation study**
  - Study will quantify performance for the methods identified at the workshop
- **Describing that study is the focus of this talk**

STUDY FOUNDATION

- **Develop standard operating procedures for several candidate methods**
- **Create known blind samples that are processed by multiple groups**
- **Quantify bias as the difference from the known sample**
- **Quantify method repeatability in several ways**
  - Repeatability by the same researcher
  - Repeatability across experienced laboratories
  - Repeatability across labs with different levels of experience
- **Quantify cost by tracking resources expended**
  - People time to implement
  - Cost of expendable supplies

MATRICES AND METHODS BEING STUDIED

- **Five identification methods**
  - Stereoscope
  - Stereoscope with Nile red staining
  - Fourier-transform infrared spectroscopy (FTIR)
  - Raman spectroscopy
  - Pyrolysis gas chromatography

- **Four matrices/extraction methods**
  - Clean water
  - Dirty water (mimicking water collected towing a trawl net through ocean water)
  - Sediment
  - Fish tissue

- **At least three laboratories processing three replicates for each method/matrix**
  - All laboratories follow the same SOP
  - 32 laboratories from seven countries participating

NATURE OF THE BLIND SAMPLES

- **Four types of plastic (PET, PVC, PS and PE)**
- **Multiple sizes for each plastic type**
  - 1-10 microns
  - 10-100 microns
  - 100-300 microns
  - 300-1000 microns
- **Three shapes**
  - Pellets, spheres and fibers
- **Samples also include false positive materials**
  - Cotton and plant material as examples

STUDY AUGMENTATIONS

- **The core study design is based on consensus-developed SOPs**
  - However there are many permutations used at various labs
- **The core study provides a great leveraging opportunity to evaluate how those permutations affect results**
  - Because we have so many participants, results can be compared both within and among labs
- **The study plan calls out multiple augmentations**
  - Some are for extraction procedures
  - Some are for measurement approaches
  - One alternative even involves new approaches to instrument automation

STUDY AUGMENTATION EXAMPLES

- **Extraction augmentations**
  - Altering concentration of KOH used in the fish tissue extraction
  - Comparing digestion using nitric acid:hydrogen peroxide instead of KOH
  - Evaluating effectiveness of different digestion salts in the sediment extraction
  - Comparing recovery rates from water with and without sieving

- **Measurement augmentations**
  - Importance of filter type (gold-coated polycarbonate, polycarbonate, silicon, Teflon)
  - Evaluating the relationship of time and accuracy using alternative subsampling procedures
  - Effect of microplastic and false positive material density

STUDY SCHEDULE

- **Study plan and SOPs were developed last fall**
  - Held training session for less experienced labs in November
- **Preparing the blind samples now**
  - Shipping starts this month and will be completed in March
- **Laboratories to complete sample processing by July 2020**
- **Collaborative workshop to interpret results will be held in August 2020**
  - Will be paired with a workshop on effects of microplastics
  - Workshop will be a forum for experts to recommend methods to California
- **Final Report: October 2020**

DEVELOPING AN ACCREDITATION PROCESS

- **California needs an accreditation process before use**
- **The method evaluation study helps with three important pieces**
  - Agreement on which method(s) to use
  - Standard operating procedure for those methods
  - An understanding of method performance capabilities
- **However, there are some additional pieces needed for accreditation**
  - Developing an auditing checklist
  - Training the laboratory auditors for what to look for
  - Developing performance evaluation samples

TRAINING THE AUDITORS

- **Starts with a checklist**
  - Should be easy to create since we will have an SOP
  - Training will also be easy since we have method developers involved
- **However, there are some interesting nuances associated with microplastics that differ from typical chemical methods**
  - Contamination from normal laboratory activities can be severe
- **Our lab invested $250K in a new air-handling system**
  - We had >1000 fibers deposited per week on background filters before the air-system upgrades
  - We now have positive-pressure HEPA-filtered system with double doors
  - We also needed to create clothing guidelines
- **How much of that will be required for accreditation?**
  - At a minimum, accreditation should require routine background measurements

PERFORMANCE EVALUATION SAMPLES

- **Creating blind samples with small particles is challenging**
  - Counting out small particles is painful
  - Static electricity is problematic when you are working with particles <10 microns
  - Emulsifiers are needed to avoid clumping in the samples
- **We need to agree on how challenging the samples should be**
  - What size classes?
  - How many types of plastic?
  - Add false positive particles?
- **Most importantly, there are no commercial vendors at this time**

IT WILL TAKE A VILLAGE

- **We need the scientists to agree on methods**
  - We need the laboratories to adopt those methods
- **ELAP needs to be an enabler during this break-in period**
- **Commercial sector needs to develop standard reference materials**
- **Regulatory staff need to realize that this is a developing science**
  - Collaboration in reacting to findings
- **The community seems to be coming together**
  - The cooperation we are receiving on the method evaluation study is impressive