Microplastics Analysis: A Simplified Workflow for Comprehensive Characterization in the Environment

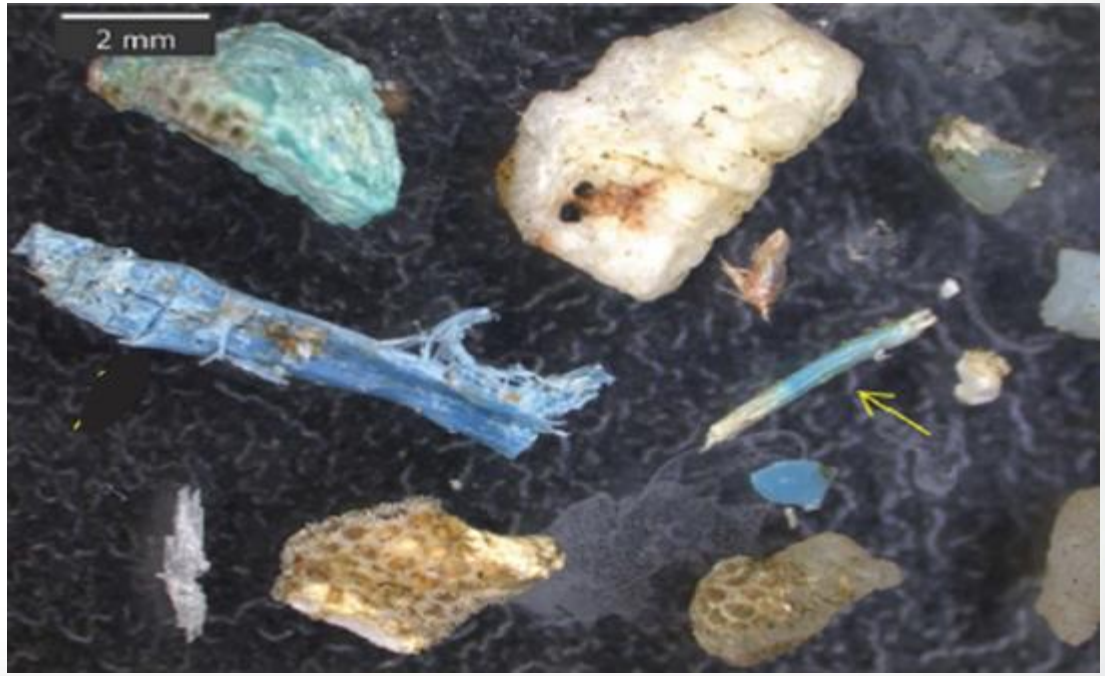
- **Anna-Marie Cook, EPA Region 9 STL, ORD/OSP (Ret)**
- **Harry L. Allen, EPA Region 9 On-Scene-Coordinator**
- **Bill Robberson, P.E. – Ocean P3 Systems (EPA Contractor)**

Microplastics Analysis: A Simplified Workflow

Microplastics Analysis: A Simplified Workflow for Comprehensive Characterization in the Environment

Lead: US EPA Region 9

Partners: California Department of Public Health – EHL
Amergent Techs
Pima County Water Resources
University of Arizona
Ocean P3 Systems
ASTM D19 Committee
Agilent
Frontier Labs
Tohoku University
PerkinElmer
Shimadzu



Microplastics Analysis: A Simplified Workflow

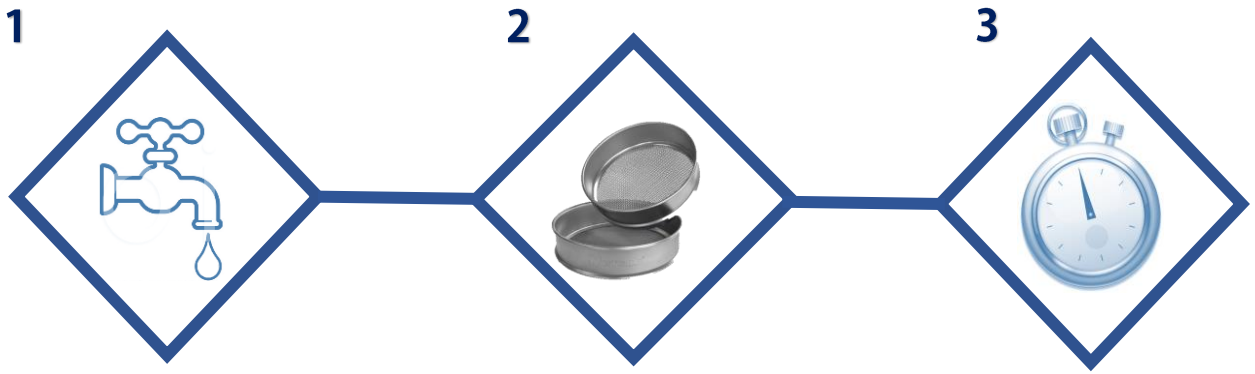
Challenges:

1. Sample Reproducibility
2. Sample Collection - QA/QC & Reproducibility
3. Sample Preparation for Py-GC/MS, IR & Raman Analysis
4. Reference Sample Development for Calibration/Proficiency
5. Sample Analysis both Count-based and Mass-based
6. Data Quality Objectives and Data Interpretation

The development of reliable, reproducible and high-quality methods for microplastics quantification and characterization is fundamental and of paramount importance for understanding microplastics risks.

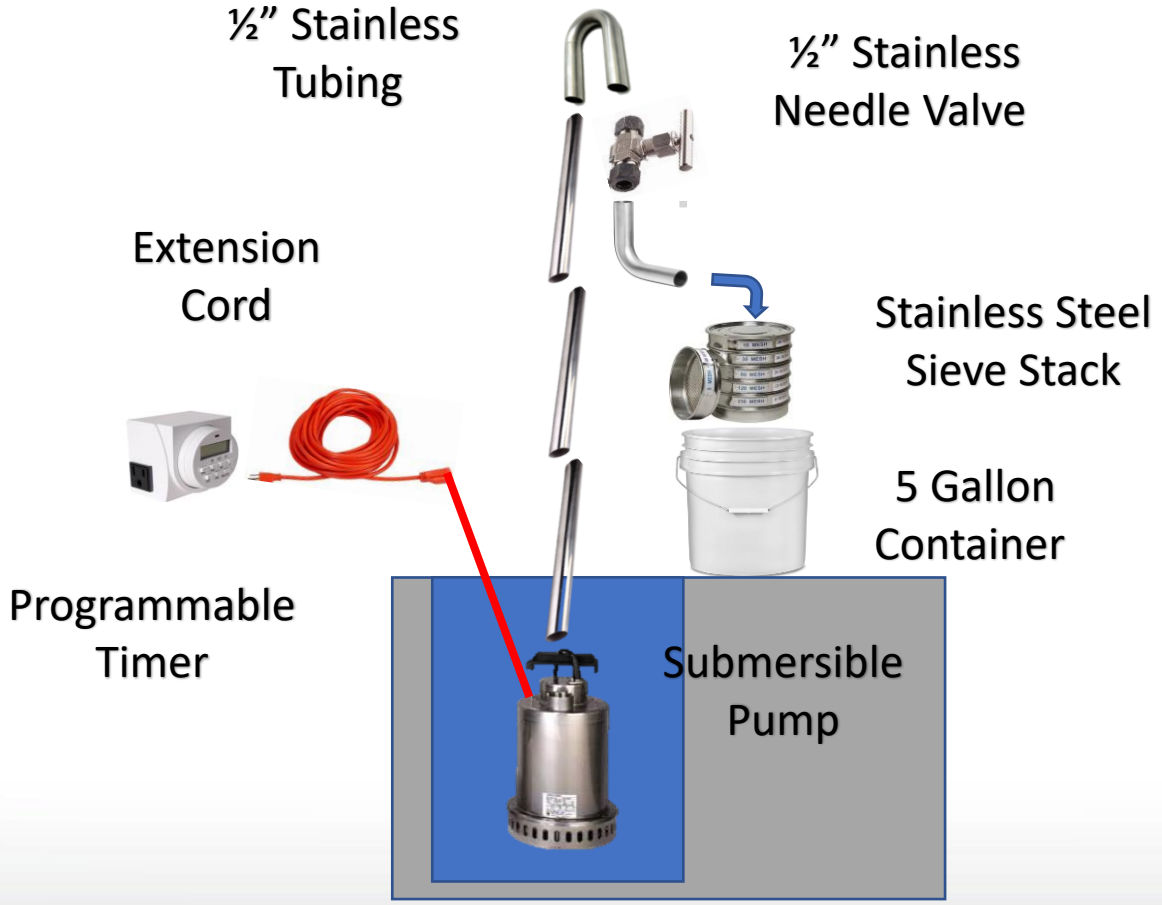
Sample Collection for Microplastics

Sample Collection



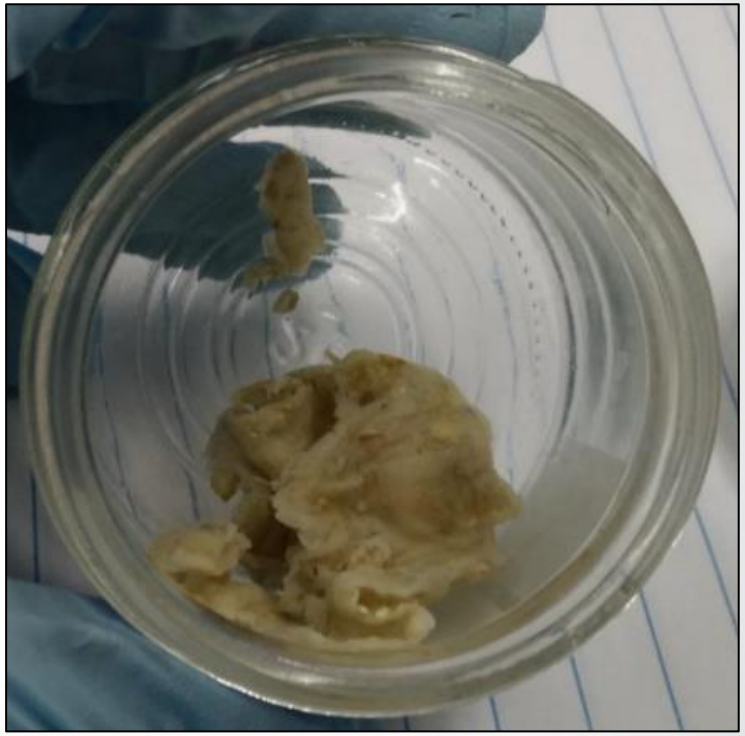
- Straightforward
- Simple to follow
- Reproducable

Wastewater Sampling Apparatus



Sample Retrieval

Sieved material rinsed and collected in sample container



Wastewater Influent (Sewage)

1,000 micron

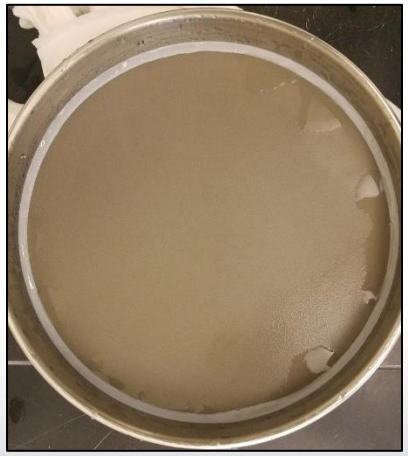
500 micron

300 micron

150 micron

Depicted - 30 gallons of sewage retained on sieves

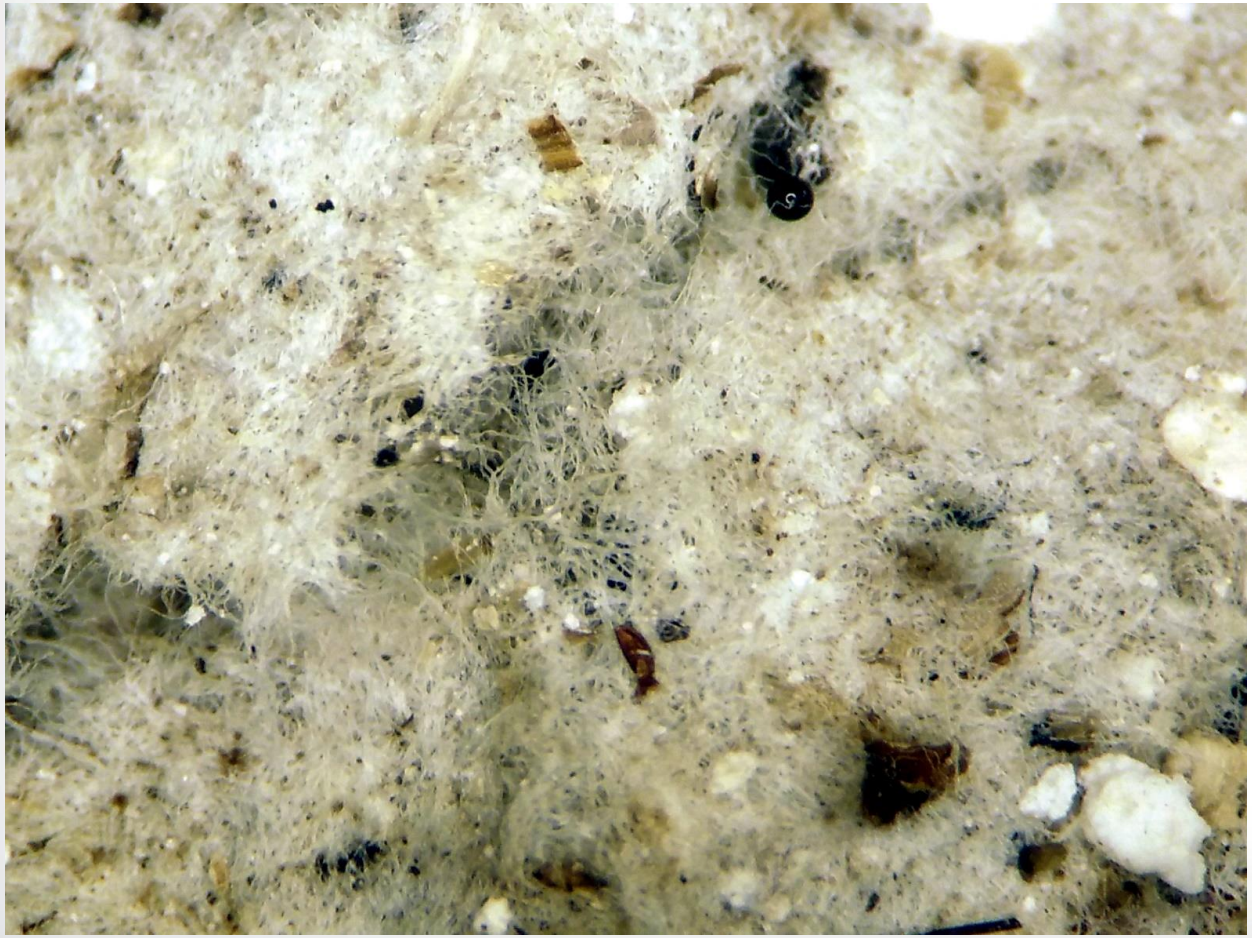
45 micron



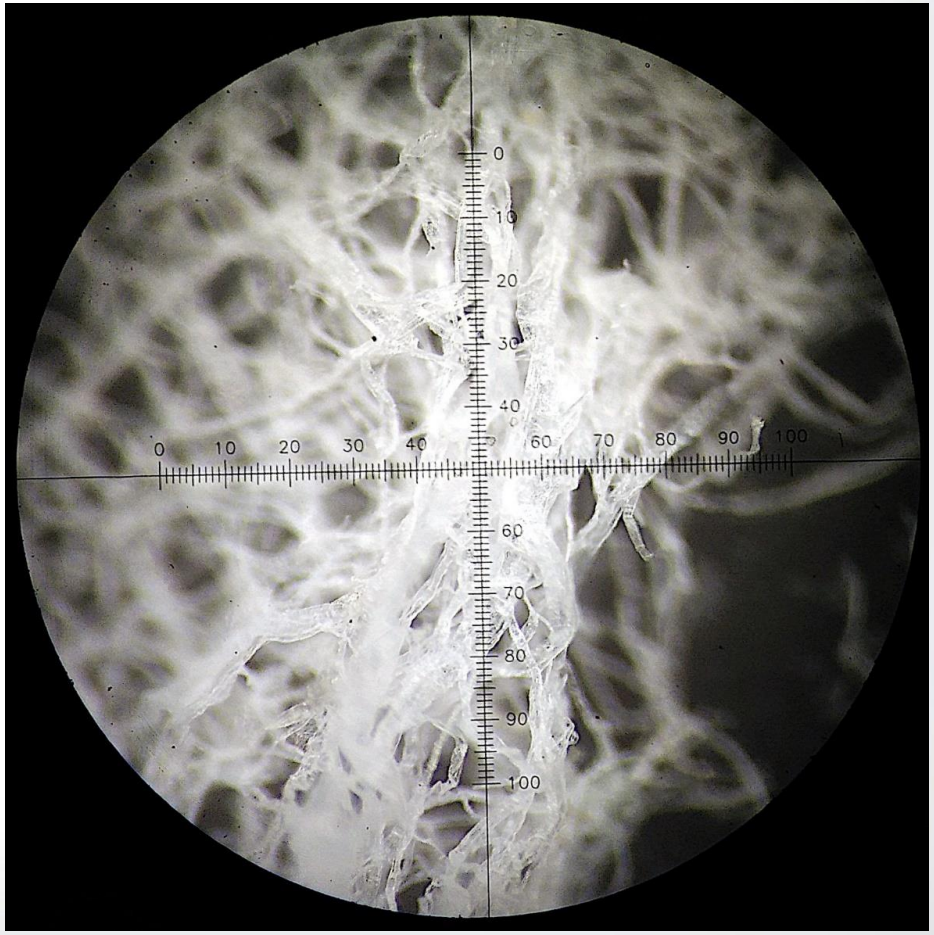
20 micron

What's in there?

20x magnification

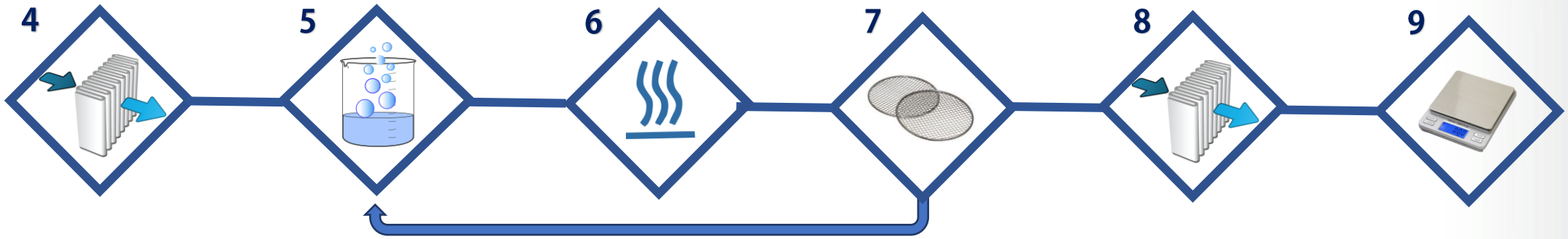


150x magnification



Sample Preparation of Microplastics

Sample Preparation



- Depends on Water Quality and Characteristics
- Needs to be Simple to follow
- Needs to be Reproducable

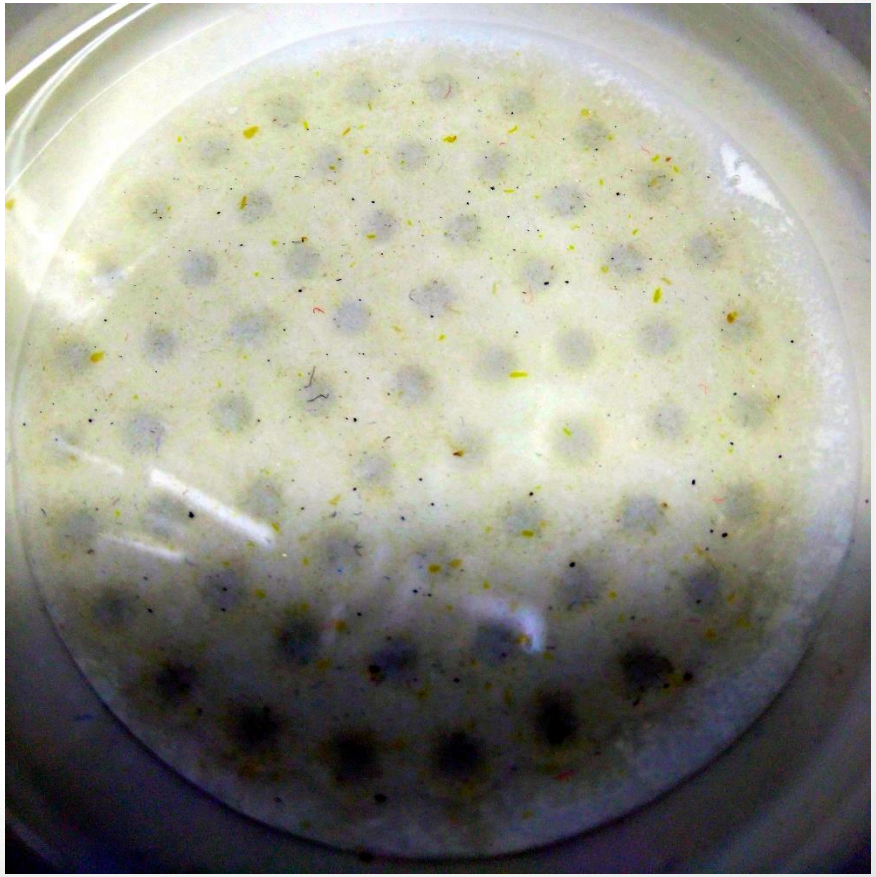
Sample Preparation

30 gallons of sewage after sieving, rinsing, and drying

Filtration Improvement

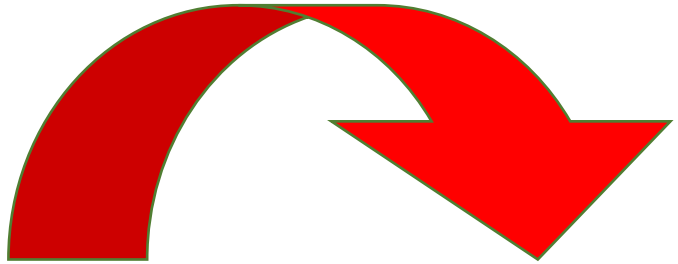
From this

To this



Analysis Preparation

From this



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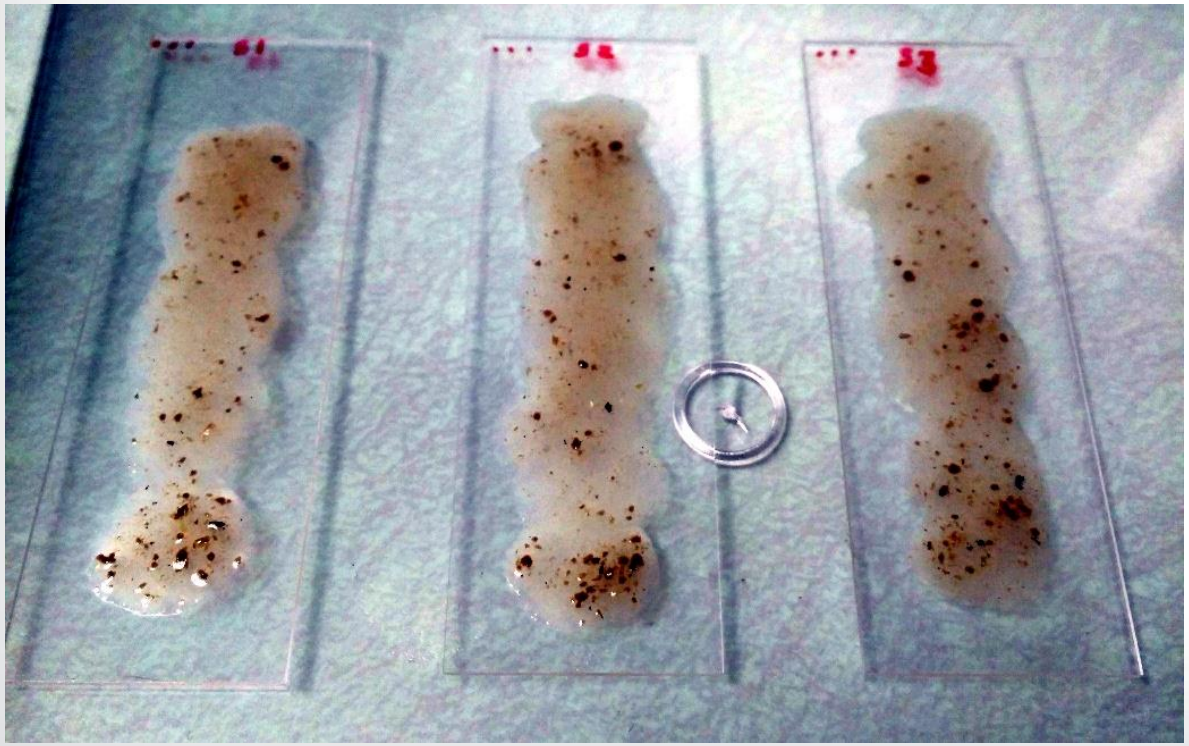
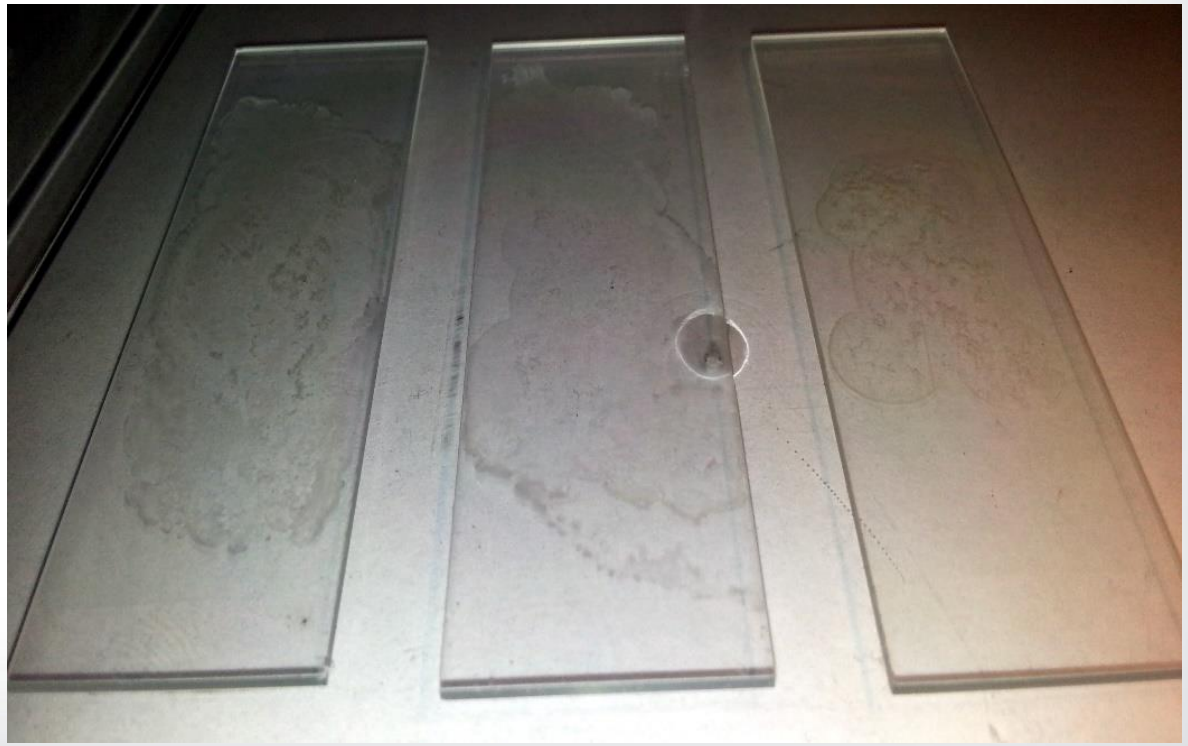


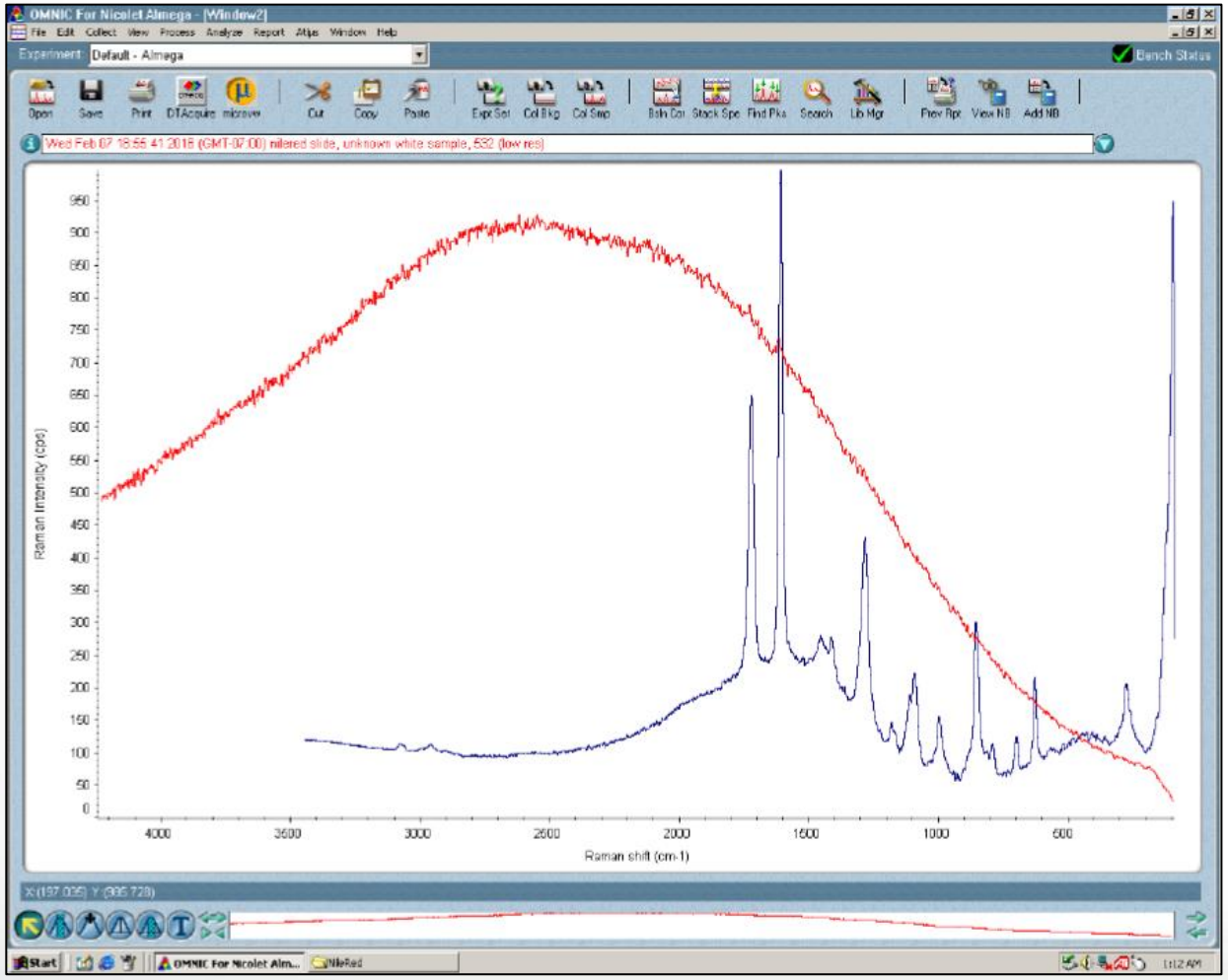


Analysis of Microplastics

Sample Analysis

- Positive identification
- Three analytical methods
  - IR
  - Raman
  - Py-GC/MS

Spectroscopic Analysis



IR and Raman spectroscopy are suitable for definitive identification of plastics

Ideal Scenario Desired

From this

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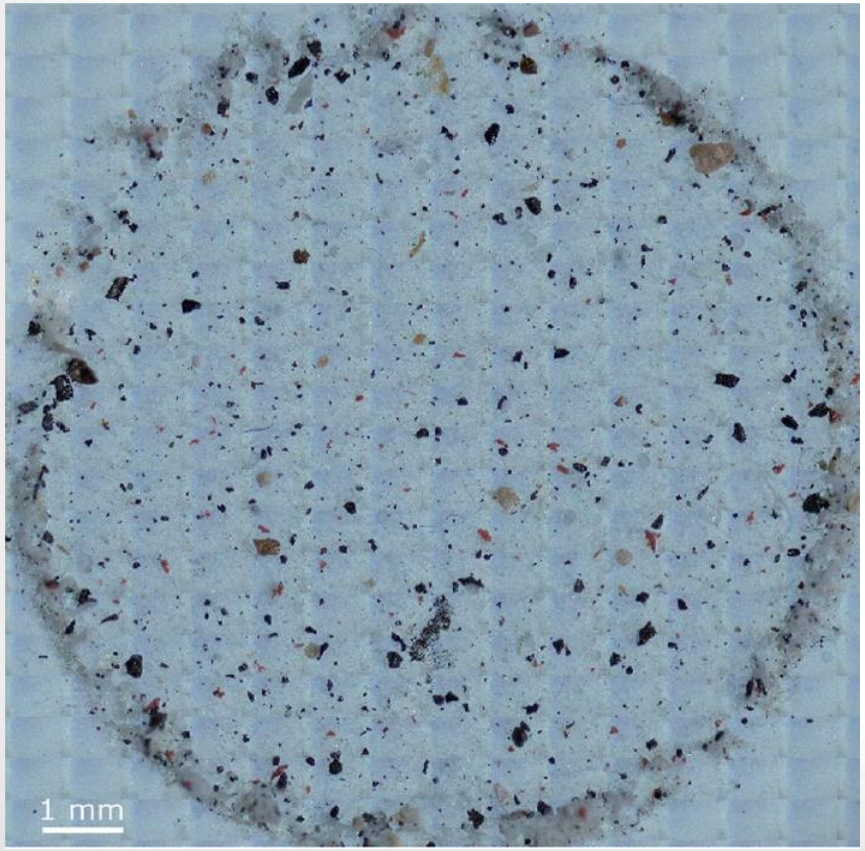
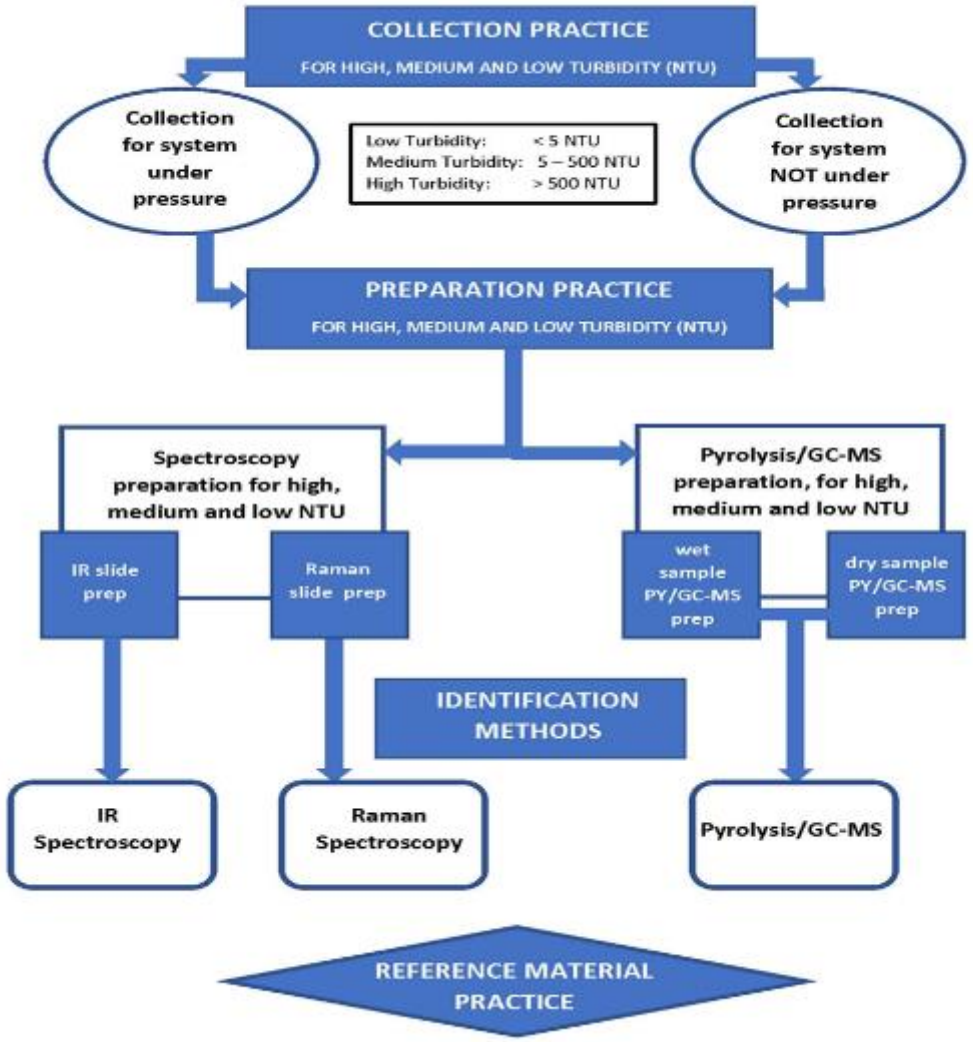


ASTM D19-06 Microplastics Subcommittee

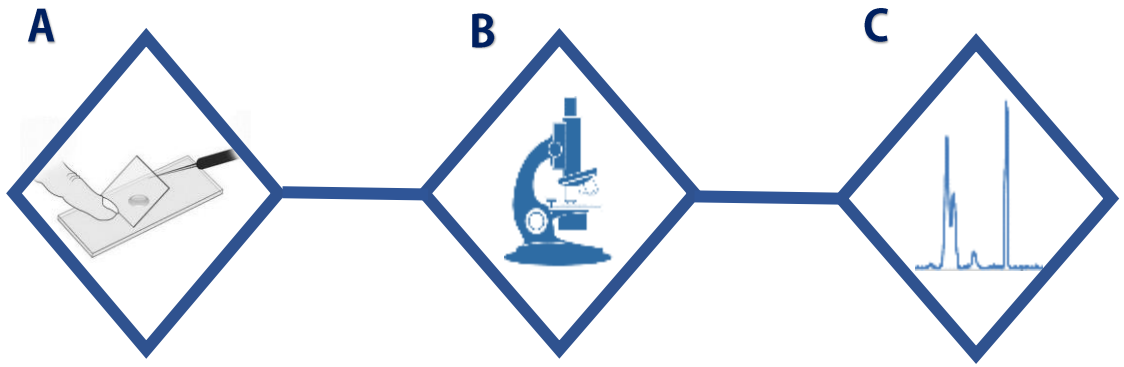
Microplastics Analysis: A Simplified Workflow



"You can't manage what you can't measure!"

Standardized identification methods and solid baseline measurements are needed to assist international, national, regional and local scientists and agencies address concerns associated with the prevalence and risks of microplastics in the environment.

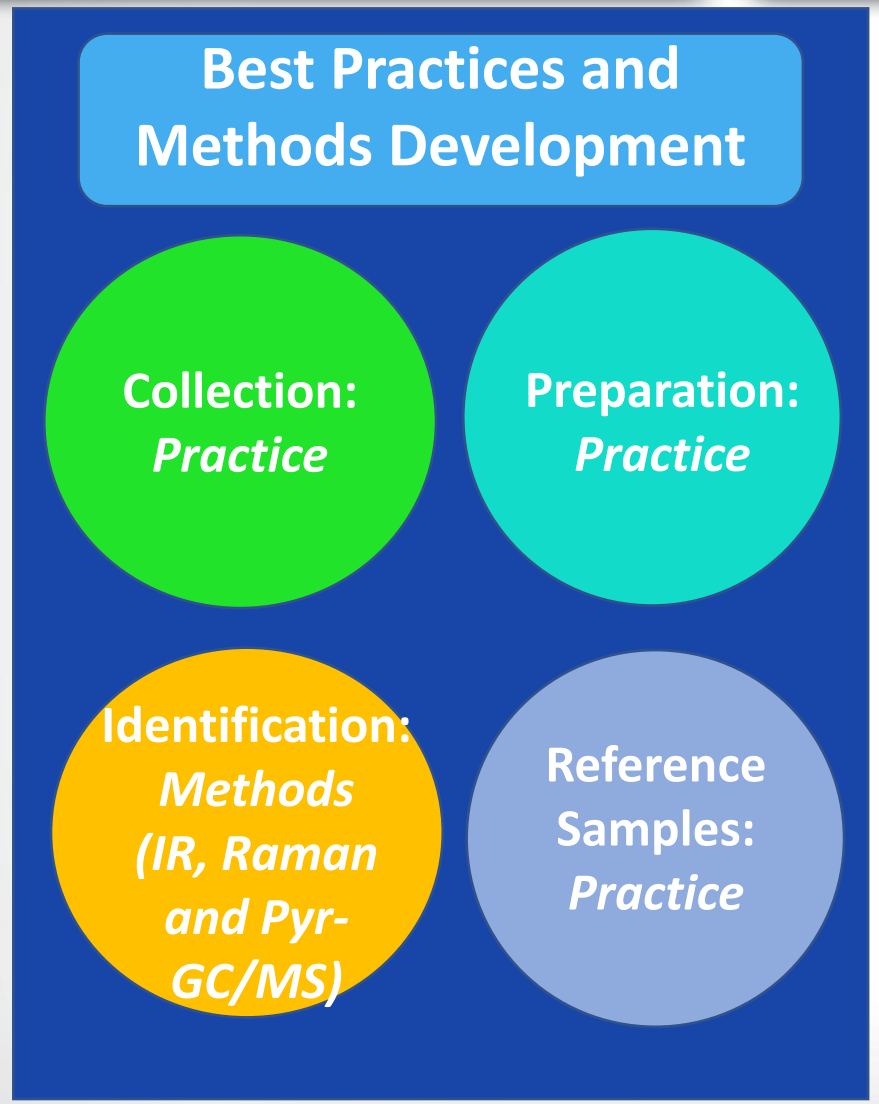
Analysis/Identification of Microplastics



1) Particle count per unit volume (IR and Raman)
   - Identify targets
   - Isolate individual particles
   - Provide positive identification polymer types
   - Quantification of particle size and count

2) Particle mass per unit volume (Py-GC/MS)
   - Provide positive identification polymer types
   - Provide quantification of mass

Microplastics Standards Development

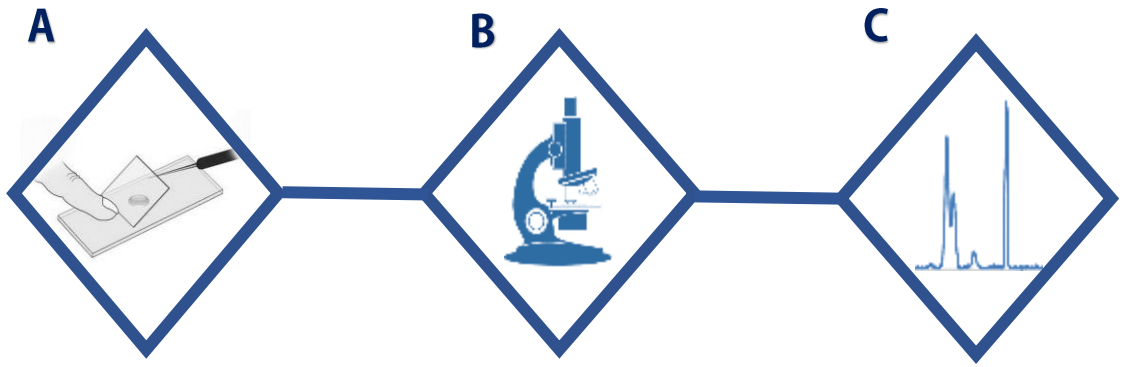


Aug 2021 Status Update

Work Items: <u>All Water Matrices</u> High - Low TSS

- D8332 Collection Practice
- D8333 Preparation Practice
- WK70831 Ref Sample Dev Practice (Post-Ballot)
- WK67788 Py-GC/MS Test Method (Post-Ballot)
- WK67565 IR and Raman Test Method (Data Ex)
- WK72349 Dynamic Image Size and Shape Test Method (Draft 2021)

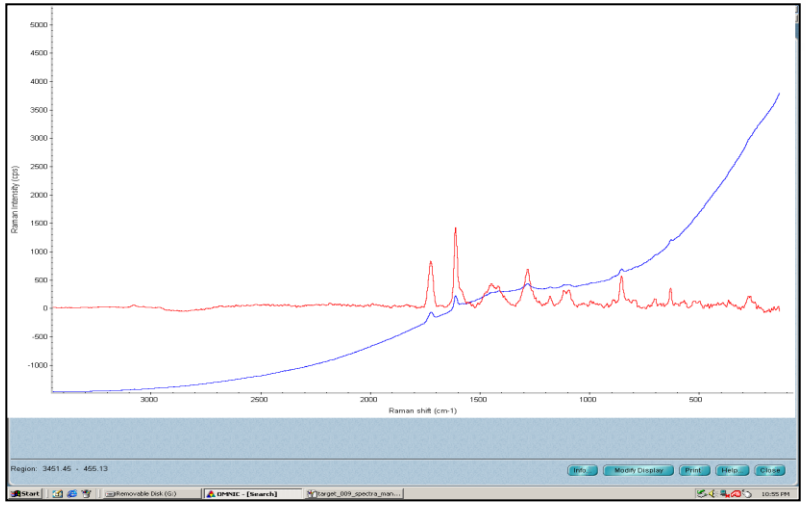
Analysis/Identification of Microplastics

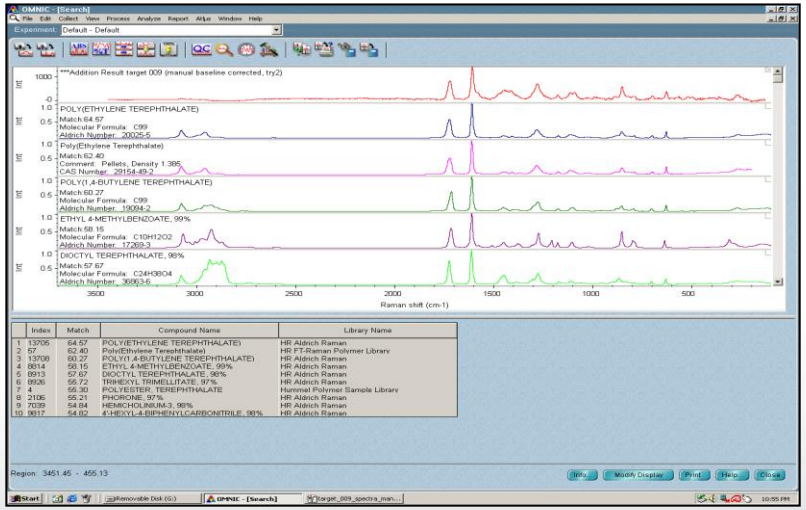


IR Particle Count Analysis Methodology

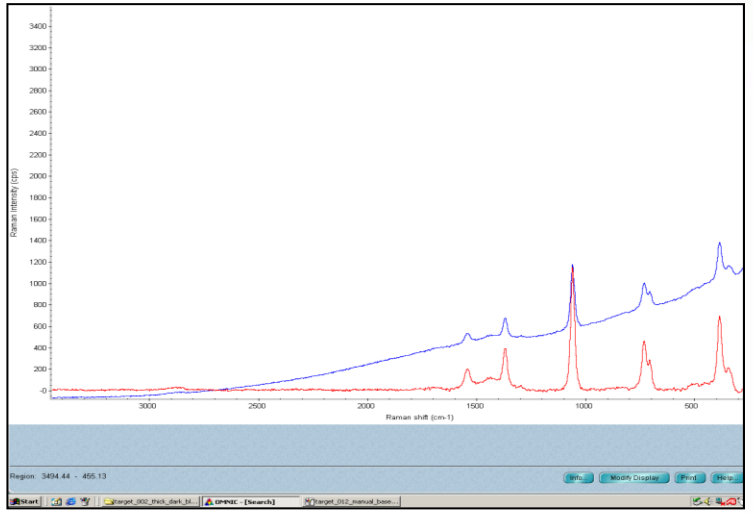
- Single Lab Repeatability Exercise (Not ILS) – Five labs, MP Ref Samples, Slides of MP-Spiked WW Aliquot & MQ, Seven Repeats @: % error/RSD
- MP particles, 50–500 um / PE, PS, PET
- 1 MP wastewater reference sample and 1 MP clean water sample to each of the 5 labs to be assessed, along with instructions to each lab:

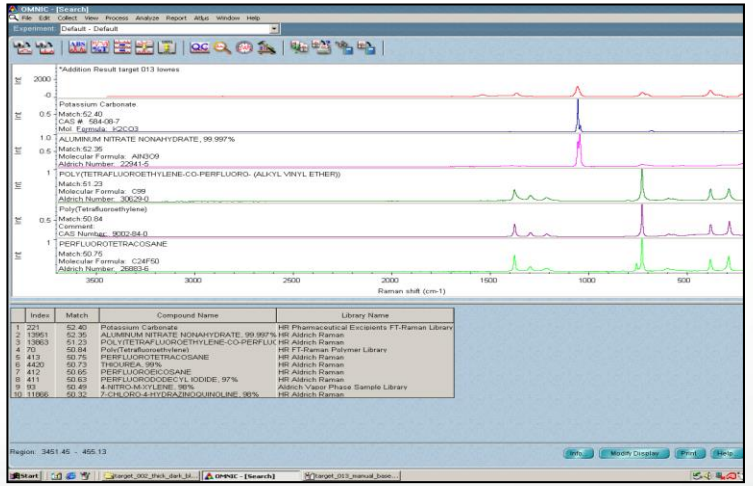
Polyethylene





PTFE


Raman intensity (cps)
Raman shift (cm-1)


Potassium Carbonate
ALUMINUM NITRATE NONAHYDRATE, 99.997%
POLY(TETRAFLUOROETHYLENE-CO-PERFLUORO- (ALKYL VINYL ETHER))
Poly(Tetrafluoroethylene)
PERFLUOROTETRACOSANE
Raman shift (cm-1)