Detection of Microplastics in Consumer Products with Single-Particle ICP-MS

Francisco Laborda[1], Celia Trujillo[1], Ryszard Lobinski[2], Mollie Cyr[3]
[1]University of Zaragoza, Zaragoza, Spain; [2]Université de Pau et des Pays de L'Adour, Pau, France; [3]PerkinElmer Inc., Shelton, CT, USA

1 Introduction

Detection of Microplastics
- Microscopy, ATR-FT-IR
- Detect & identify composition of microplastics
- Limited sample throughput

Increasing Sample Throughput with SP-ICP-MS
- Minimal sample preparation
  - Just dilute the sample, if necessary
- High sample throughput
  - Ability to detect thousands of particles per minute
- Particle size & number concentration information

Microplastic Detection with SP-ICP-MS Challenges
- - Must be able to reliably detect carbon
  - High background due to CO_2 in the Ar used to generate the plasma & presence as impurity in reagents
- - High ionization potential (11.3 eV)

Overcoming the Challenges
- Shorten measurement times by using microsecond dwell times
  - Lowers the background, but overall signal from particles is unaffected
- Monitor C13 (1.1% abundance)
  - Much lower background that C12

Goal

Use SP-ICP-MS as a Rapid Screening Tool for Microplastic Detection

2 Analyses

NexION ICP-MS
- Rapid data acquisition
- Short dwell times

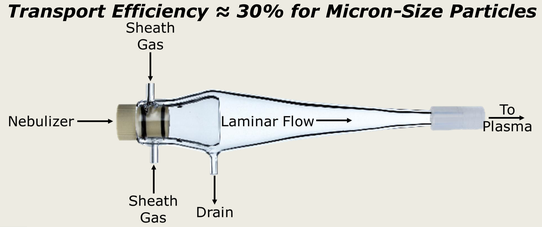
Syngistix Nano Application Module
- Rapidly measures particles
- Provides particle size & concentration information
- Real-time histograms of data

3 Transport of Microplastics to the Plasma

Conventional spray chambers are best for small particles/aerosols
- Larger particles/droplets are filtered out
- Micron-size particles will not reach the plasma

Asperon Spray Chamber Designed for Optimal Transport of Micron-sized Particle to the Plasma
- Linear-pass spray chamber
- Sheath gas flows tangential to chamber walls
- Laminar flow reduces particle-contact with walls

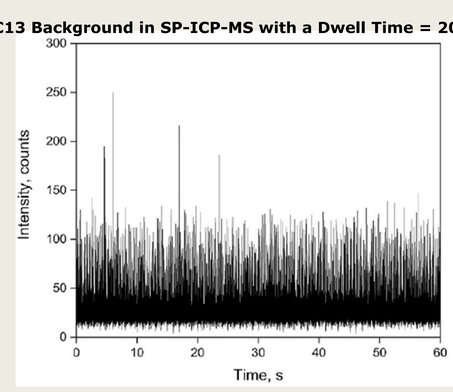
Transport Efficiency ≈ 30% for Micron-Size Particles



4 Reduction of the Carbon Background

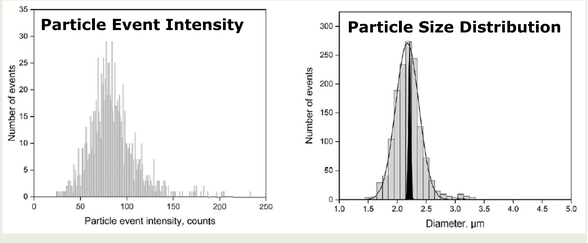
Microsecond Dwell Times
- 100-200 µs provided best signal-to-noise ratio -
  - Best accuracy
  - Lowest detection limits
- C13 background reduced to < 50 counts

C13 Background in SP-ICP-MS with a Dwell Time = 200 µs



5 Detection of Polystyrene Microparticle Standards

2.2 µm Polystyrene Spheres



Evaluating Accuracy for Different Size Spheres

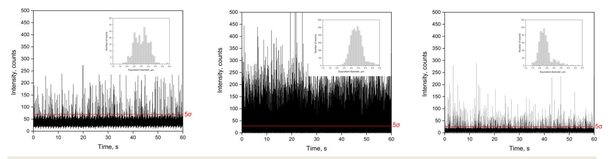
Particle Standard	Certified Diameter (µm)	Measured Diameter (µm)	% Bias
1	1.04 ± 0.03	1.69 ± 0.07	140
2	1.98 ± 0.01	1.83 ± 0.01	93
3	2.02 ± 0.02	1.93 ± 0.01	96
4	2.22 ± 0.01	2.10 ± 0.02	94
5	3.03 ± 0.09	2.74 ± 0.01	91
6	4.00 ± 0.04	3.56 ± 0.06	91
7	5.00 ± 0.04	4.55 ± 0.05	92

Accurate Size Measurements from 2- 5 µm

6 Detection of Microplastics in Exfoliants

Samples & Sample Preparation
- Plastic microparticles are added to exfoliants as abrasives
- 3 consumer exfoliants were purchased
- Samples were mixed with water, sonicated, & filtered through 10 µm filters
- Samples were diluted so the particle concentration was < ≈ 250,000 particles/mL
- Dilutions were done iteratively after the samples were run & the particle concentrations were determined
- At higher concentrations, the chances of multiple particles being ionized simultaneously increase
- Inaccurate results if more than one particle is ionized at a time

Time Scans & Size Distribution Plots of 3 Different Exfoliants



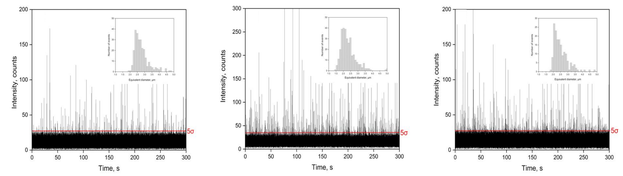
Carbon-Containing Particle Results from Exfoliants

Sample	Mean Diameter (µm)	Particle Concentration (g^{-1})
Blank	---	---
Exfoliant 1	2.89	3.6×10^{7}
Exfoliant 2	3.08	3.1×10^{11}
Exfoliant 3	2.11	4.4×10^{10}

Summary
- Size distribution & number of particles vary between the samples
- Exfoliant 1 has the broadest size distribution
- Exfoliant 2 contains the most particles
- Exfoliant 3 has the smallest particle sizes

7 Detection of Microplastics in Plastic Tea Bags

Samples & Sample Preparation
- 3 brands of tea in plastic tea bags were purchased
- The contents of each tea bag were removed
- The tea bags were then washed & dried
- Each tea bag was placed in 100°C water for 5 minutes to replicate the tea brewing process, then removed
- The water was cooled & analyzed by SP-ICP-MS



Carbon-Containing Particle Results from Plastic Tea Bags

Sample	Mean Diameter (µm)	Particle Concentration (g^{-1})
Blank	---	---
Tea Bag 1	2.70	2.2×10^{4}
Tea Bag 2	2.18	5.5×10^{4}
Tea Bag 3	2.26	1.9×10^{4}

Summary
- All samples contain about the same size & number of carbon-containing particles
- Particles most likely originate from leach leaching and/or the break down of tea bags
- Significantly fewer particles than from the exfoliants

8 Summary

SP-ICP-MS can be used as a complementary screening technique for microplastic detection. Since SP-ICP-MS cannot identify the composition of the carbon-containing particle, other techniques (such as microscopy & ATR-FT-IR) must be used to characterize & identify the composition of the particles.

Nevertheless, because of its analytical speed & low C13 background, SP-ICP-MS can rapidly detect & count large numbers of microplastic particles down to 2 µm. Used in conjunction with techniques which can identify the composition of microplastics, SP-ICP-MS can be a valuable tool in determining the presence of microplastics in consumer & environmental samples.

PerkinElmer, Inc., 940 Winter Street, Waltham, MA USA (800) 762-4000 or (+1) 203 925-4602 www.perkinelmer.com