

New ASTM Test Method for Determination of Microplastics Particle and Fiber Size, Distribution, Shape and Concentration in Waters with High to Low Suspended Solids Using a Dynamic Image Particle Size and Shape Analyzer

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### ASTM D19 has developed and is developing several guides, practices, and methods for microplastics in water



# The methods (in development) reference and rely on the existing practices



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### Sample collection by D8332



# D8332 Sampling can be adapted to river sampling, storm water, or even a boat



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### Sample preparation by D8333

• $H_2O_2$
Ammonium Hydroxide 5 minutes
• Buffer
Protease, buffer, lipase React 20 hours
Final sample = plastics suspended in methanol

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## Further prep may be needed after D8333, depending on the method

![](_page_6_Picture_2.jpeg)

![](_page_6_Picture_3.jpeg)

## Once final sample is prepared it is ready for measurement

![](_page_7_Picture_2.jpeg)

## Smaller particles are harder and more time consuming for an analyst to count in IR method

![](_page_8_Picture_2.jpeg)

High Contrast

![](_page_8_Picture_4.jpeg)

### **IR** Objective

### Why have a DIA method? An instrument rather than a person counts particles.

Can you count 1500 particles per slide?									
Number of Frames 4800	Number of Particles	1506 Concentration(count/mL)	1560.133						
		Median Value(µm)	50.906						
		<sup>1</sup> Modal Value(μm)	51.082						
0.9		۵.۹ Mean Value(µm)	49.612						
0.8		<sup>0.8</sup> Standard Deviation	7.166						
0.7		0.7 CV	0.144						
0.6		0.6 Value of Cumulative	Value of Cumulative %(µm)						
tt 0.5		0.000%	0.000						
		0.000%	0.000						
		0.000%	0.000						
		0.3 0.000%	0.000						
0.2		0.2 0.000%	0.000						
0.1		0.1 0.000%	0.000						
	50 10	0.000%	0.000						
Area	0.000%	0.000							

# Preparing a sample for the DIA method, after D8333

![](_page_10_Figure_2.jpeg)

## Method optimization – Standard materials used

	μm	Particles/mL
(sigmaaldrich_72986)Micro Particle, PS based,	10.000	1.82E+08
(sigmaaldrich_74491)Micro Particle, PS based,	20.000	2.27E+07
(sigmaaldrich_74161)Micro Particle, Polymethacrylate based,	50.000	1.25E+06
(sigmaaldrich_59336)Micro Particle, PS based, monodispersed,	100.000	3.64E+04

### **Measuring 20-micron beads**

![](_page_12_Figure_2.jpeg)

•The D8333 final solvent is methanol, however methanol does not work •Water works better, but requires an exchange •High viscosity glycerin works and methanolic sample can be added

### **Measuring 100-micron beads**

![](_page_13_Figure_2.jpeg)

- Methanol or water do not work
- High viscosity glycerin works and methanolic sample can be added

## Experiments to determine the best ratio of glycerin and methanol

![](_page_14_Figure_2.jpeg)

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### 50% glycerin selected; slightly low bias assumed due to sub sampling

![](_page_15_Figure_2.jpeg)

![](_page_15_Picture_3.jpeg)

+MeOH 70wt%

Fig. Sedimentation of PSL 100µm in each solvent

![](_page_15_Picture_6.jpeg)

## Determination of linearity detected a contamination problem 100- micron

![](_page_16_Figure_2.jpeg)

![](_page_16_Figure_3.jpeg)

## Determination of linearity detected a contamination problem 50 micron

![](_page_17_Figure_2.jpeg)

# Blank evaluation showed some particles but not enough to affect linearity

![](_page_18_Figure_2.jpeg)

seven separate blanks were analyzed, according to the method, to find an average of 5.8 particles with a standard deviation of 4.6

MDL ~ 15 particles /mL

## The shape of the smaller particles was not spherical

![](_page_19_Figure_2.jpeg)

We assume the smaller particles are contamination in the standards and not introduced by our process or solvents  $\rightarrow$  low blank

### To measure <u>standard</u> beads correctly, we reject the smaller particles in the count

![](_page_20_Figure_2.jpeg)

### Preliminary precision statement

Particle Size, μΜ	Theoretical Concentration, counts/mL	n	Found Concentration, counts/mL	Standard Deviation	% RSD
100	1700	4	1700	166	9.8
	170	4	168	24.9	14.8
	34	7	34	8.5	25.3
	17	4	24	9.6	39.8
50	1460	4	1460	78.1	5.3
	146	4	152	10.5	6.9
	29	7	43	13	30.3
	15	4	30	2.5	8.4
20	2240	4	2240	104.7	4.7
	224	4	223	12.6	5.7
	45	7	42	8.5	20
	22	4	26	1.6	6
10	1960	4	1960	28.5	1.4
	196	4	197	36.6	18.6
	39	7	31	2.4	7.6
	20	4	20	6.3	31.1

### Next steps for draft method

![](_page_22_Figure_2.jpeg)

- Work item tied to IR and PY-GCMS work items (remove this dependency?)
- Passed Subcommittee balloting except for bullet item above (1 negative)
- Test non-spherical particles
- Test real matrices prepared by D8333
- Reballot for successful sub
- Ballot at main with single laboratory repeatability statement

### Questions?

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