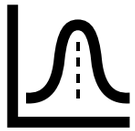
New ASTM Test Method for Determination of Microplastics Particle and Fiber Size, Distribution, Shape and Concentration in Waters with High to Low Suspended Solids Using a Dynamic Image Particle Size and Shape Analyzer

William Lipps
Shimadzu Scientific Instruments, Inc.
August 2022

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



Reference materials



Sample Collection

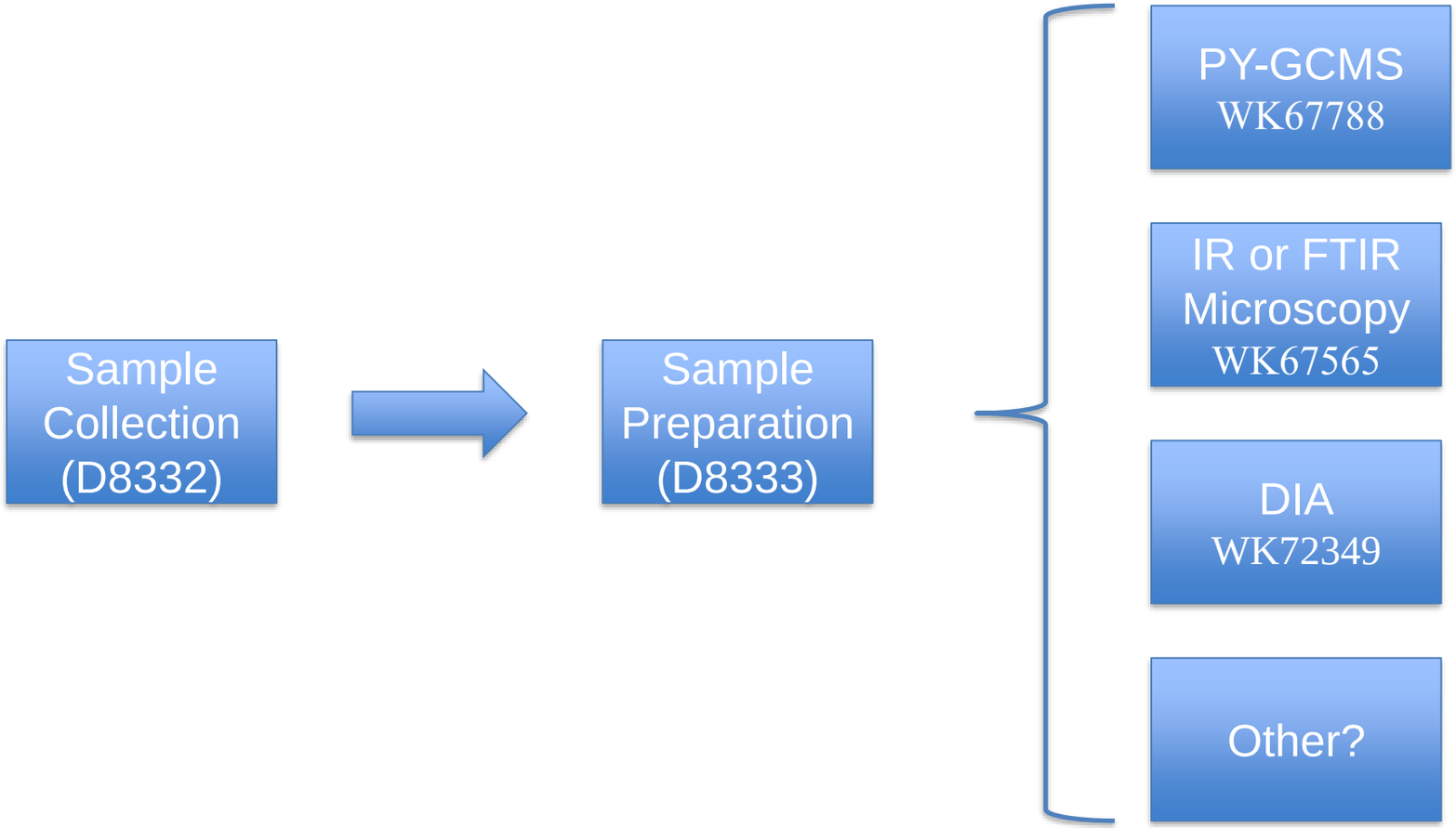


Sample Preparation

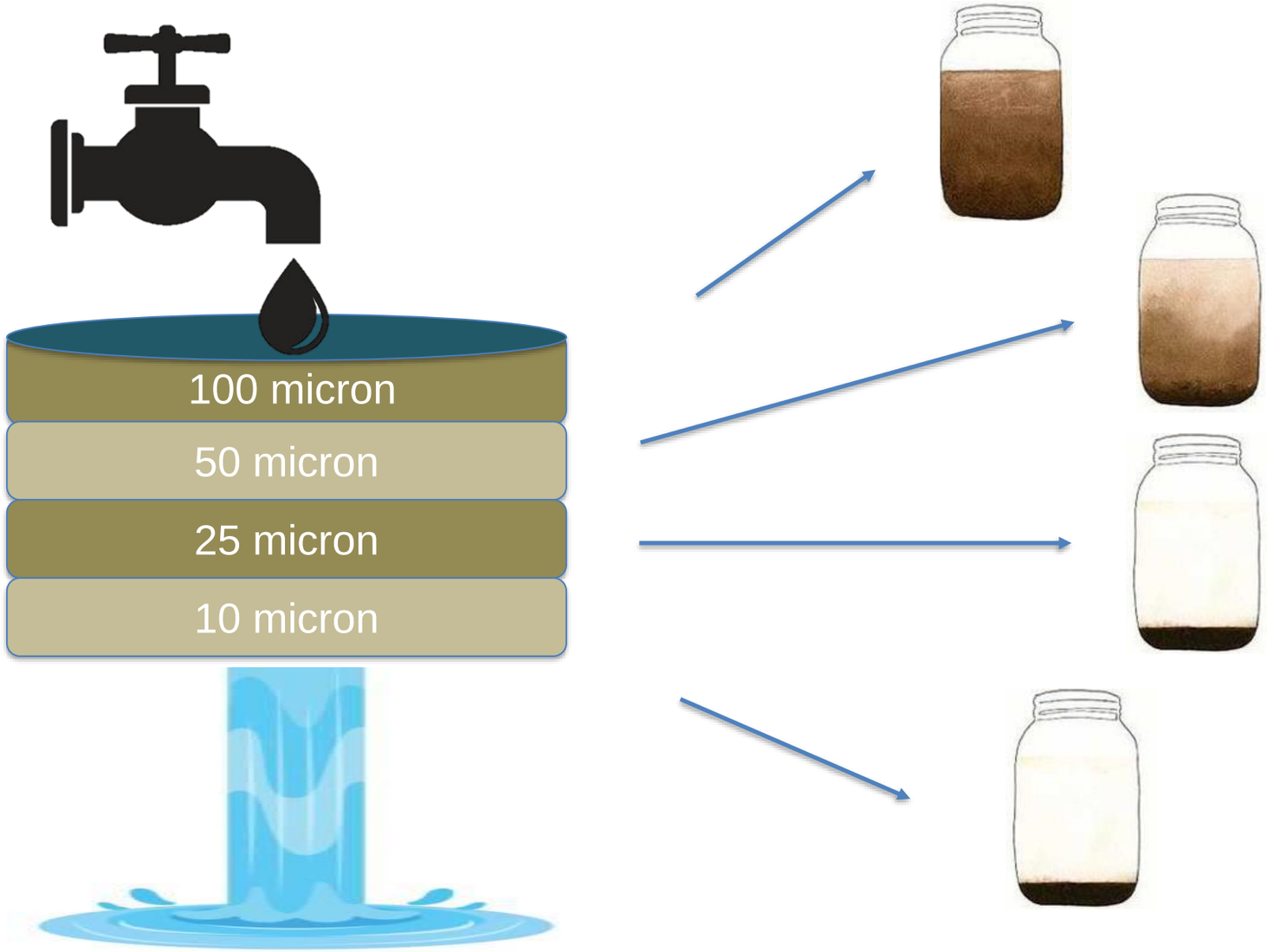


Analysis

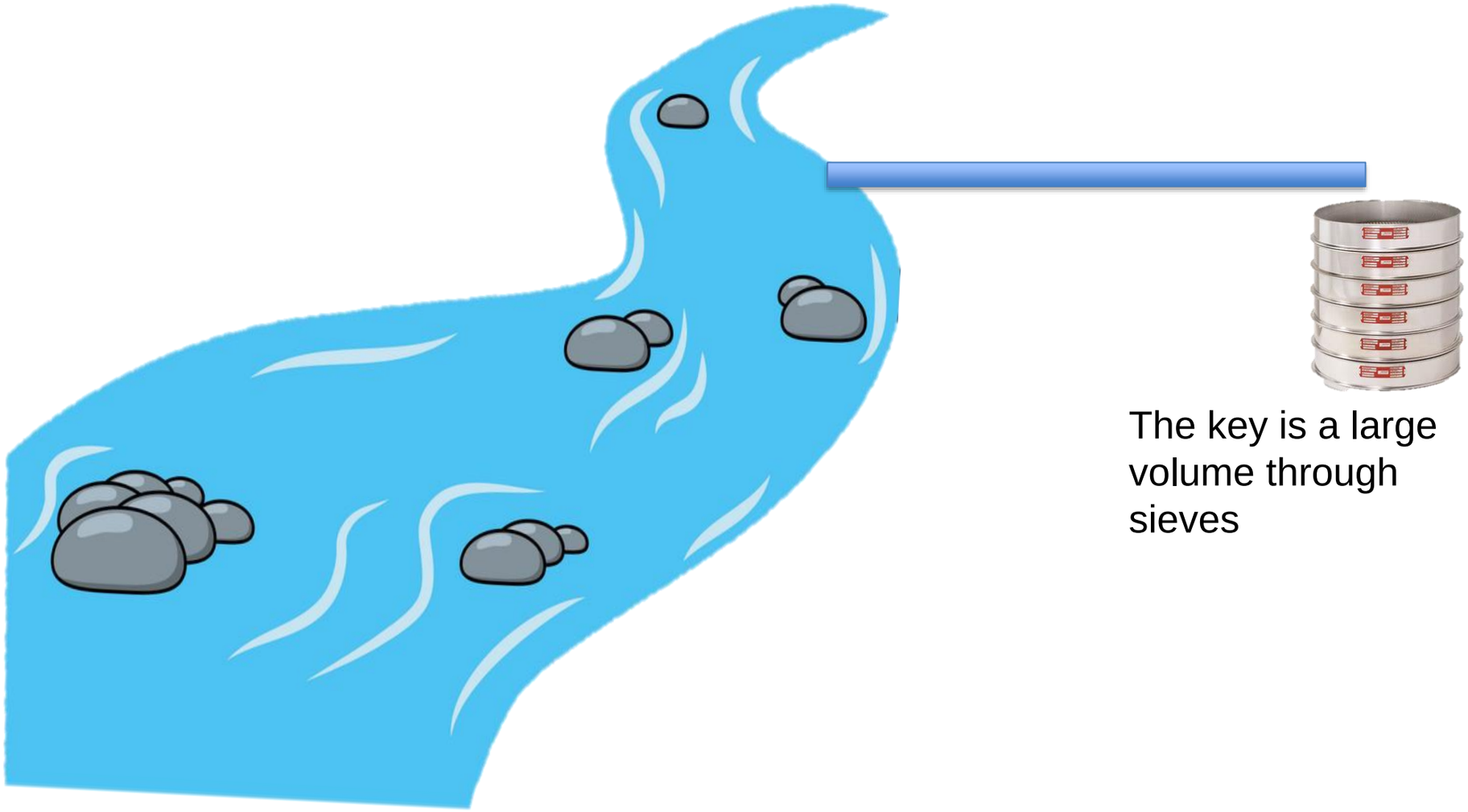
The methods (in development) reference and rely on the existing practices



Sample collection by D8332



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



The key is a large volume through sieves

Sample preparation by D8333



- H_2O_2
- Schweizer 5 minutes
- Ammonium Hydroxide 5 minutes
- Buffer
- Protease, buffer, lipase
- React 20 hours



Final sample = plastics suspended in methanol

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





Once final sample is prepared it is ready for measurement



Pyrolysis GCMS
WK67788



Dynamic Imaging
WK72349



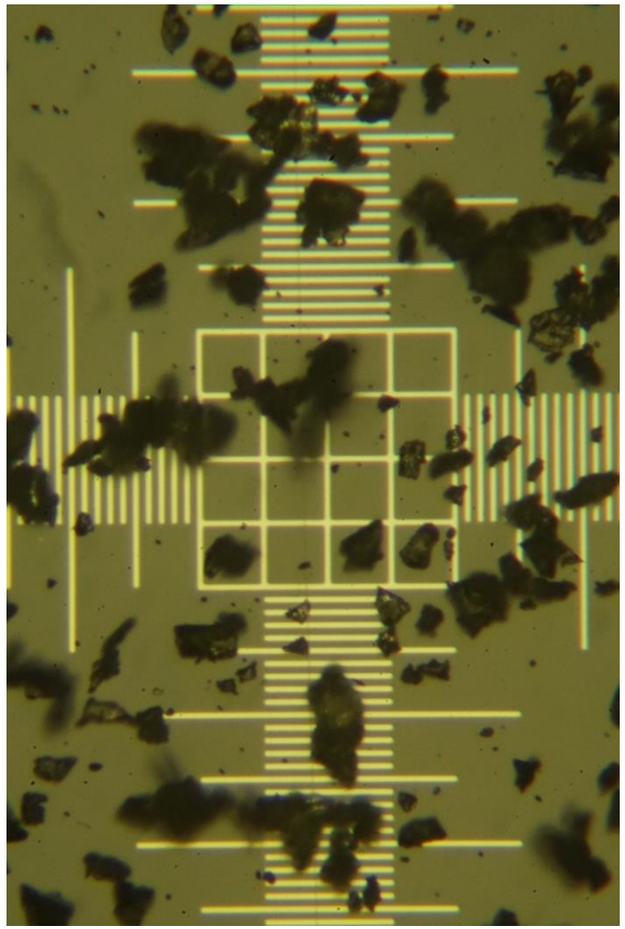


FTIR or Raman Microscopy
WK67565

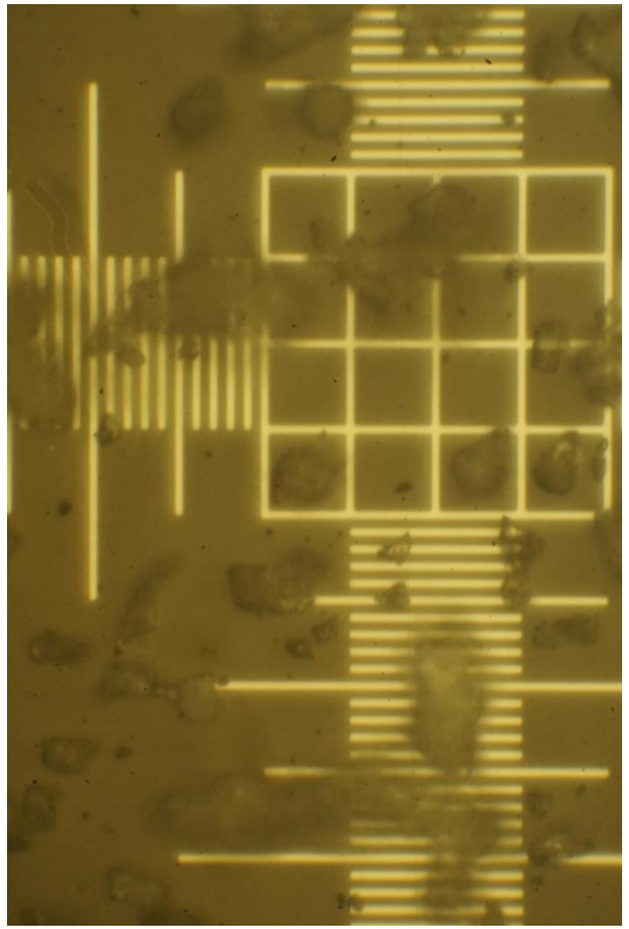


Other?

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



High Contrast

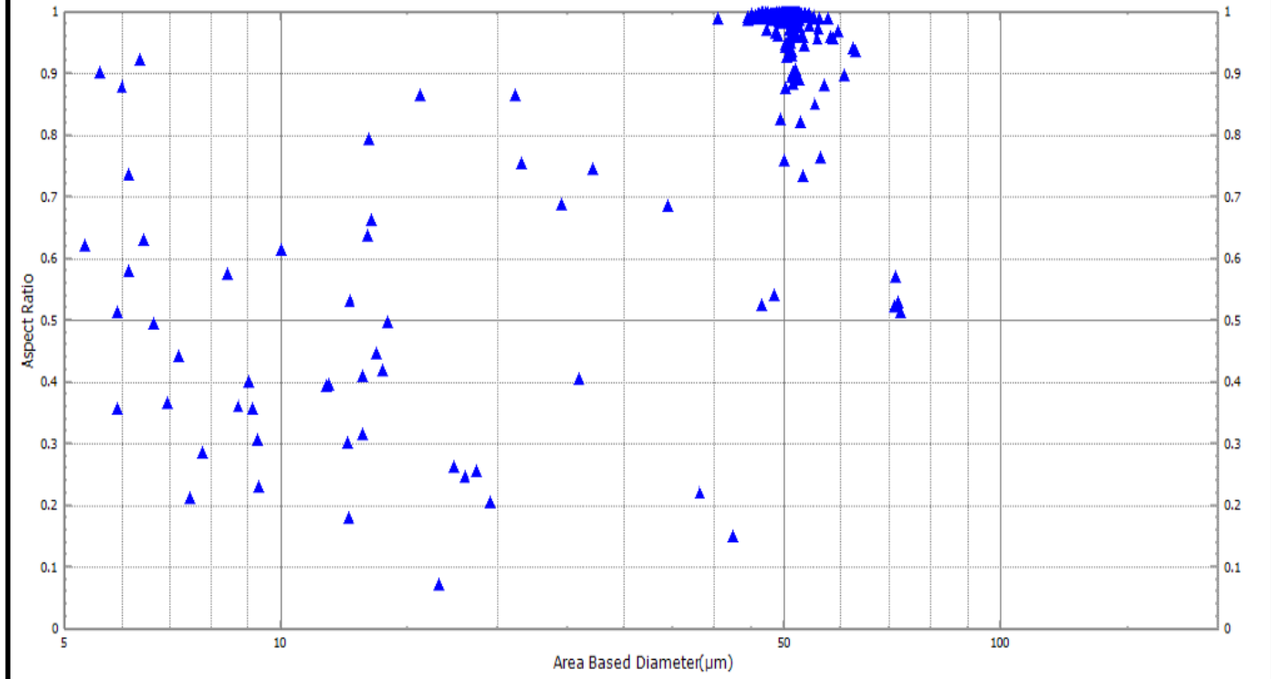


IR Objective

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
Modal Value(µm)	51.082
Mean Value(µm)	49.612
Standard Deviation	7.166
CV	0.144

Value of Cumulative %(µm)	
0.000%	0.000
0.000%	0.000
0.000%	0.000
0.000%	0.000
0.000%	0.000
0.000%	0.000
0.000%	0.000
0.000%	0.000

Preparing a sample for the DIA method, after D8333

Sample from D8333
10 – 100 µm

Add 4750 microliters dilution solvent to 15 ml tube

Add 250 microliters sample

Mix 20 times

Analyze







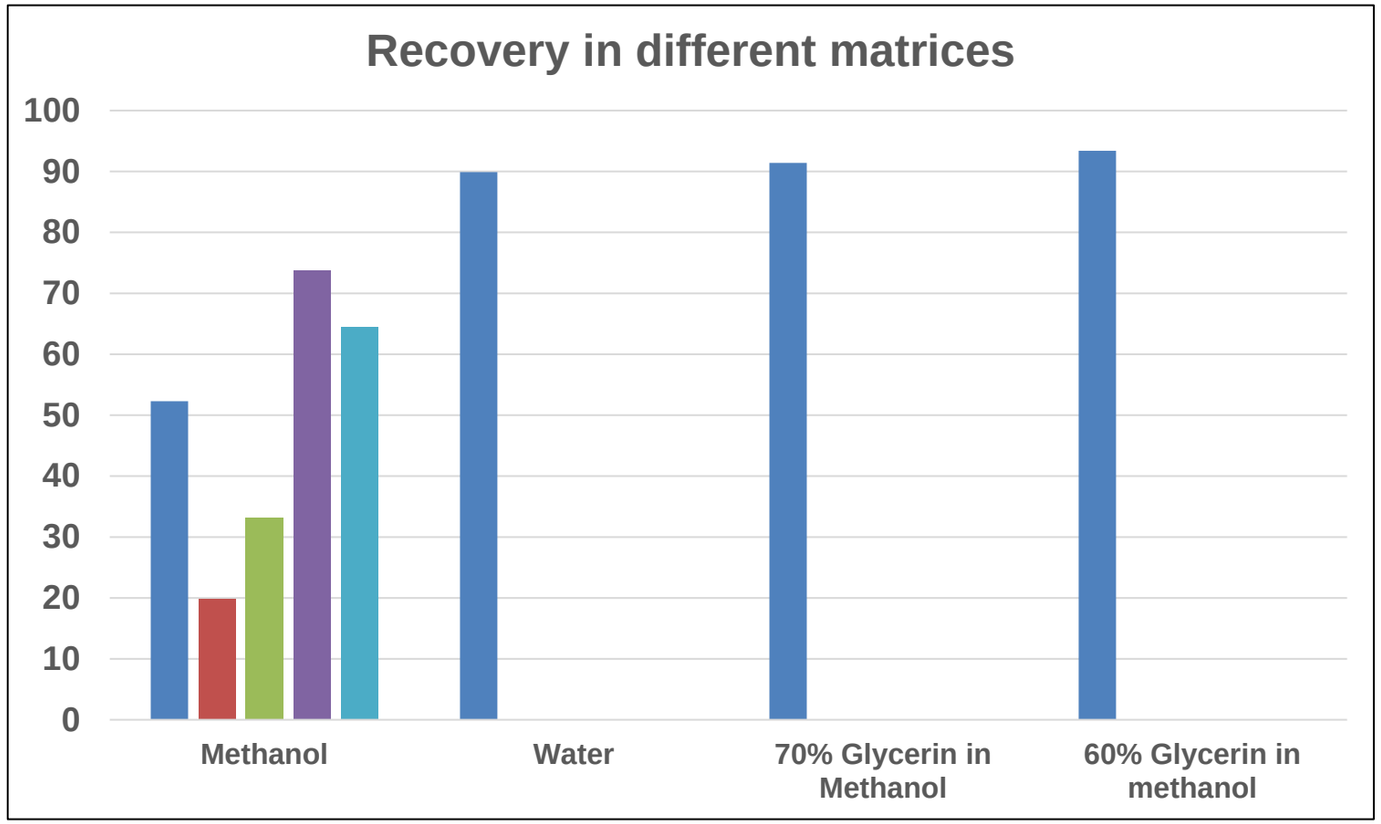




Method optimization – Standard materials used

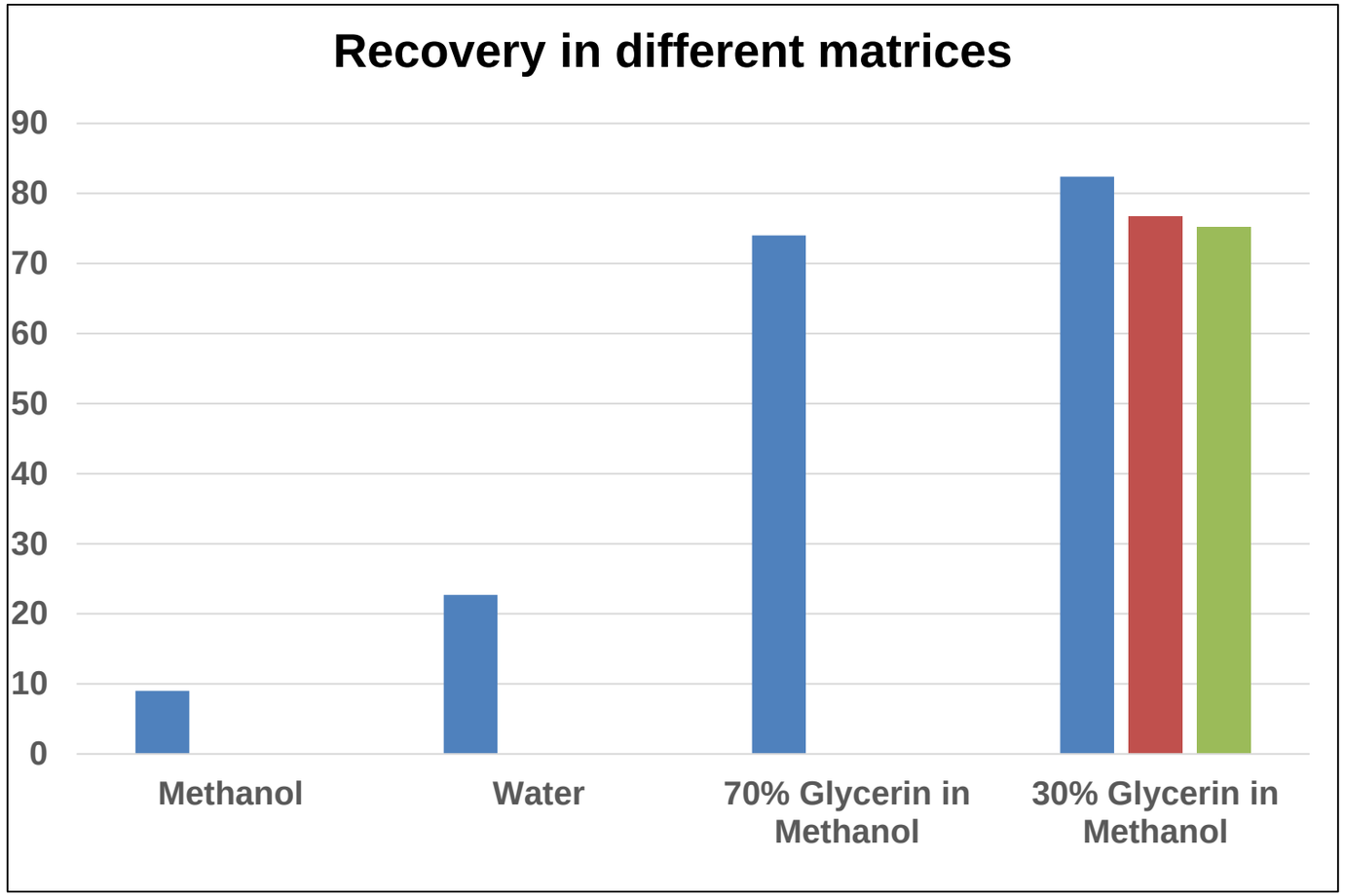
	µm	Particles/mL
(sigmaaldrich_72986)Micro Particle, PS based,	10.000	1.82E+08
(sigmaaldrich_74491)Micro Particle, PS based,	20.000	2.27E+07
(sigmaaldrich_74161)Micro Particle, Polymethacrylate based,	50.000	1.25E+06
(sigmaaldrich_59336)Micro Particle, PS based, monodispersed,	100.000	3.64E+04

Measuring 20-micron beads



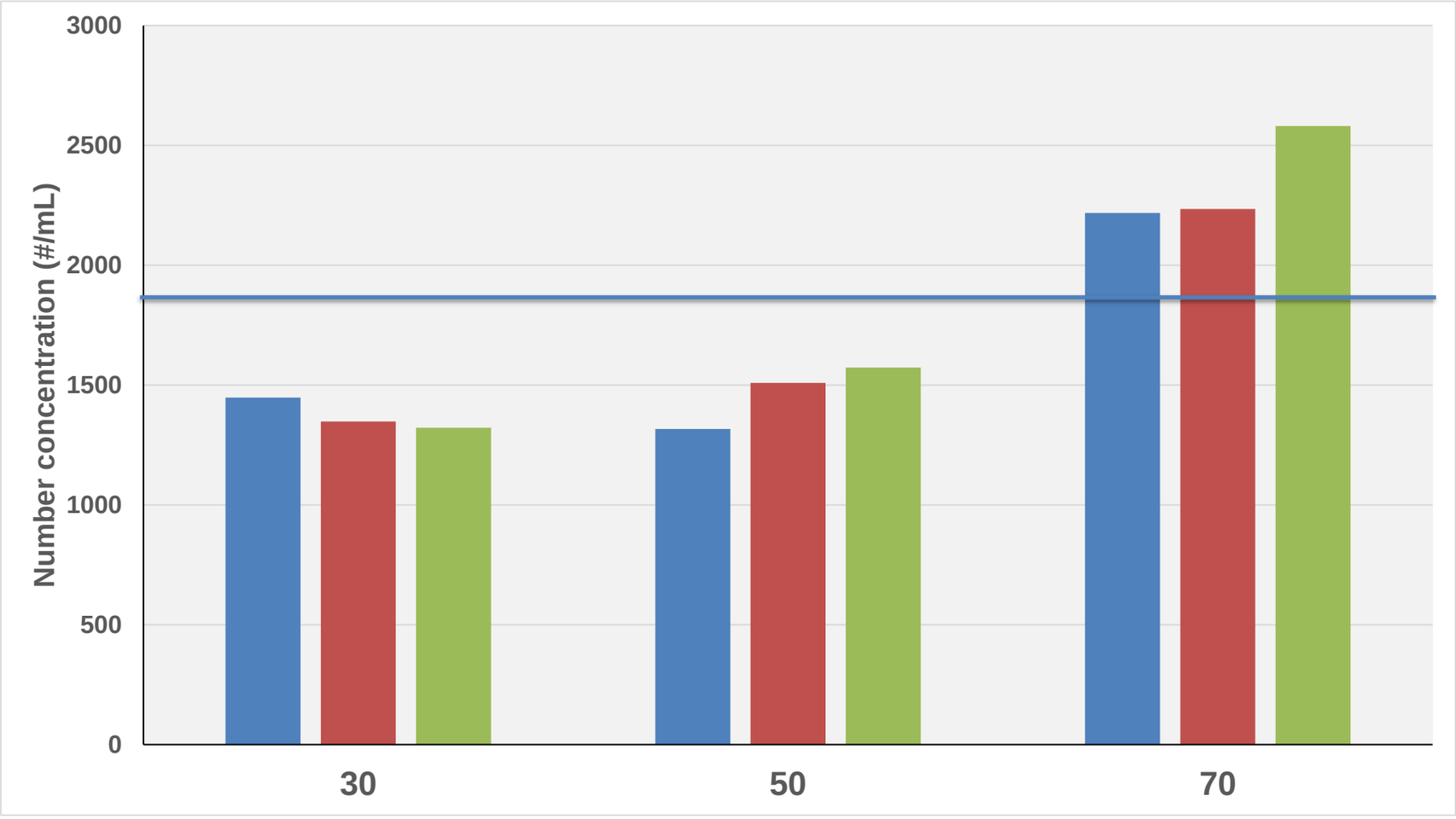
- The D8333 final solvent is methanol, however methanol does not work
- Water works better, but requires an exchange
- High viscosity glycerin works and methanolic sample can be added

Measuring 100-micron beads



- 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



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

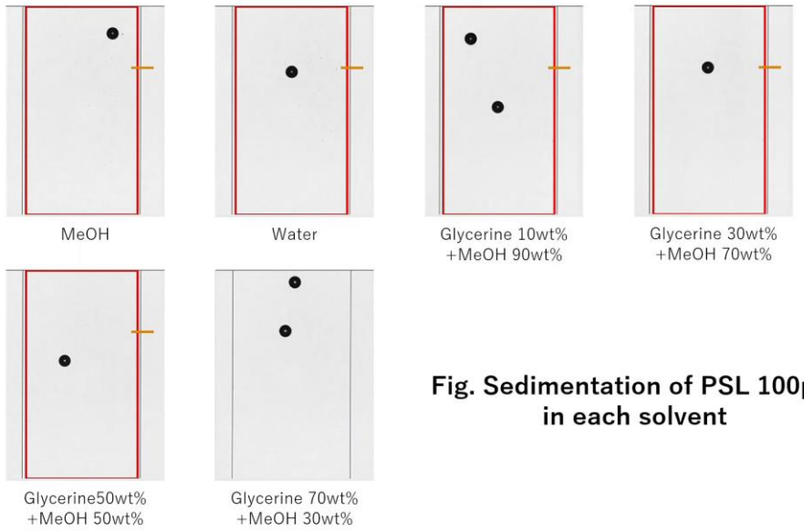
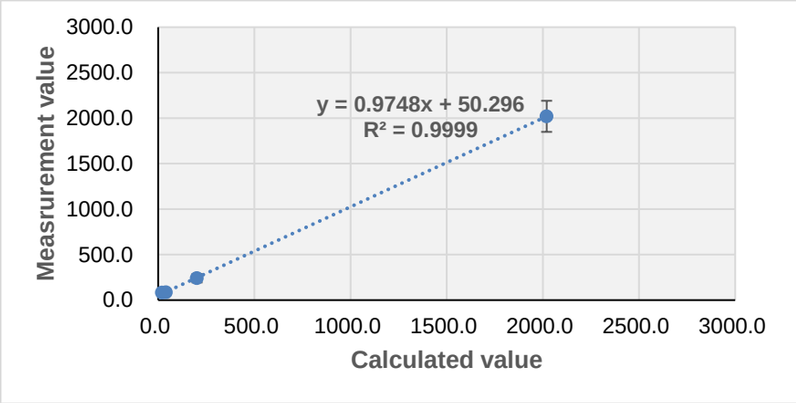
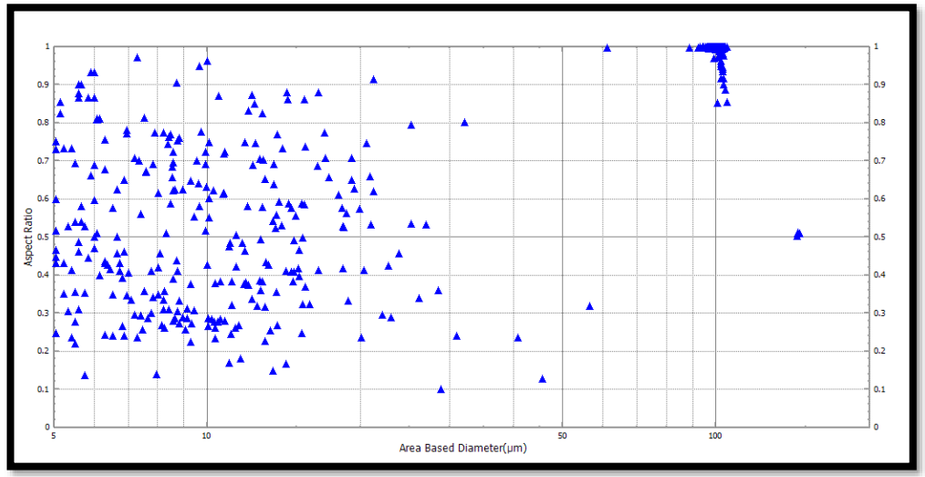


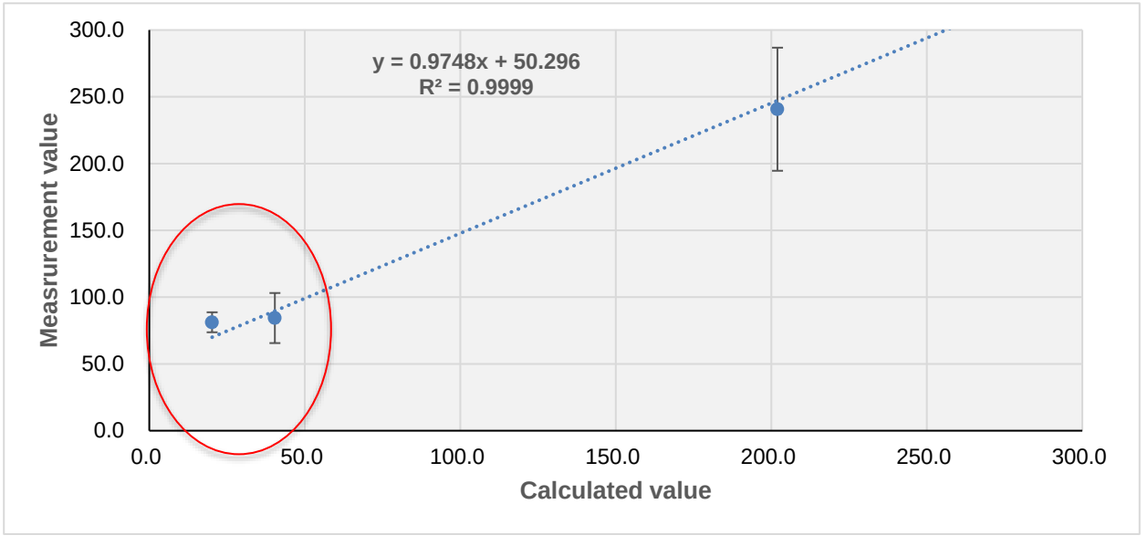
Fig. Sedimentation of PSL 100μm in each solvent



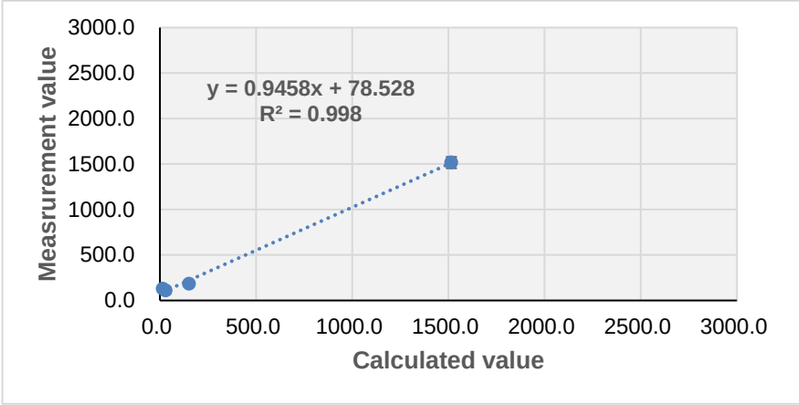
Determination of linearity detected a contamination problem 100- micron

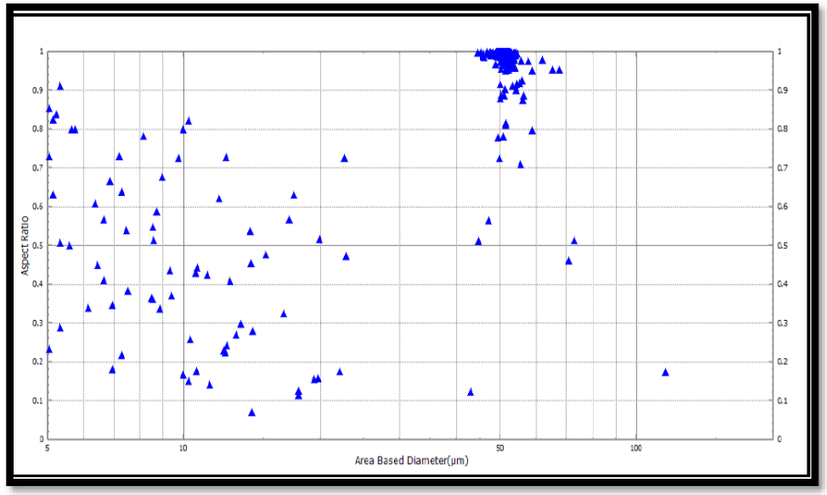


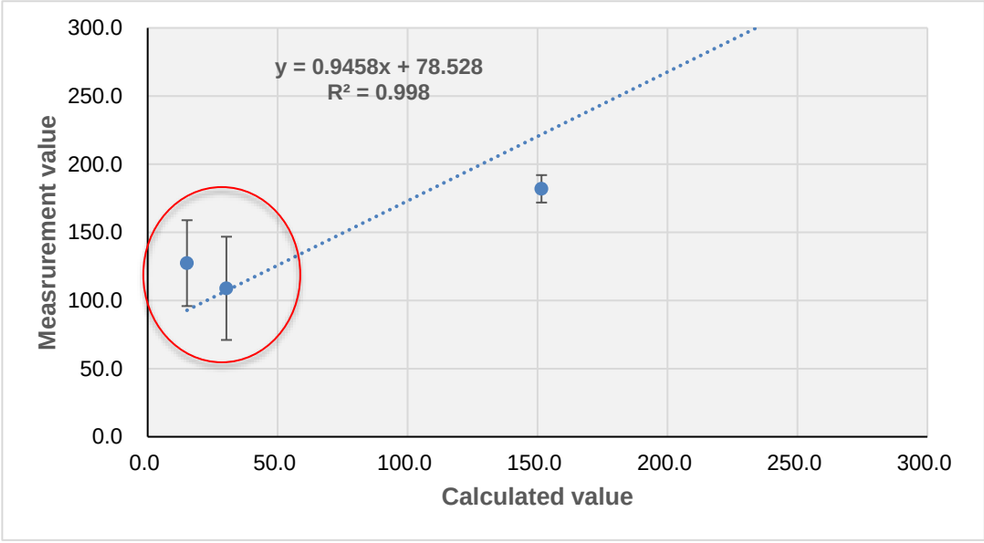




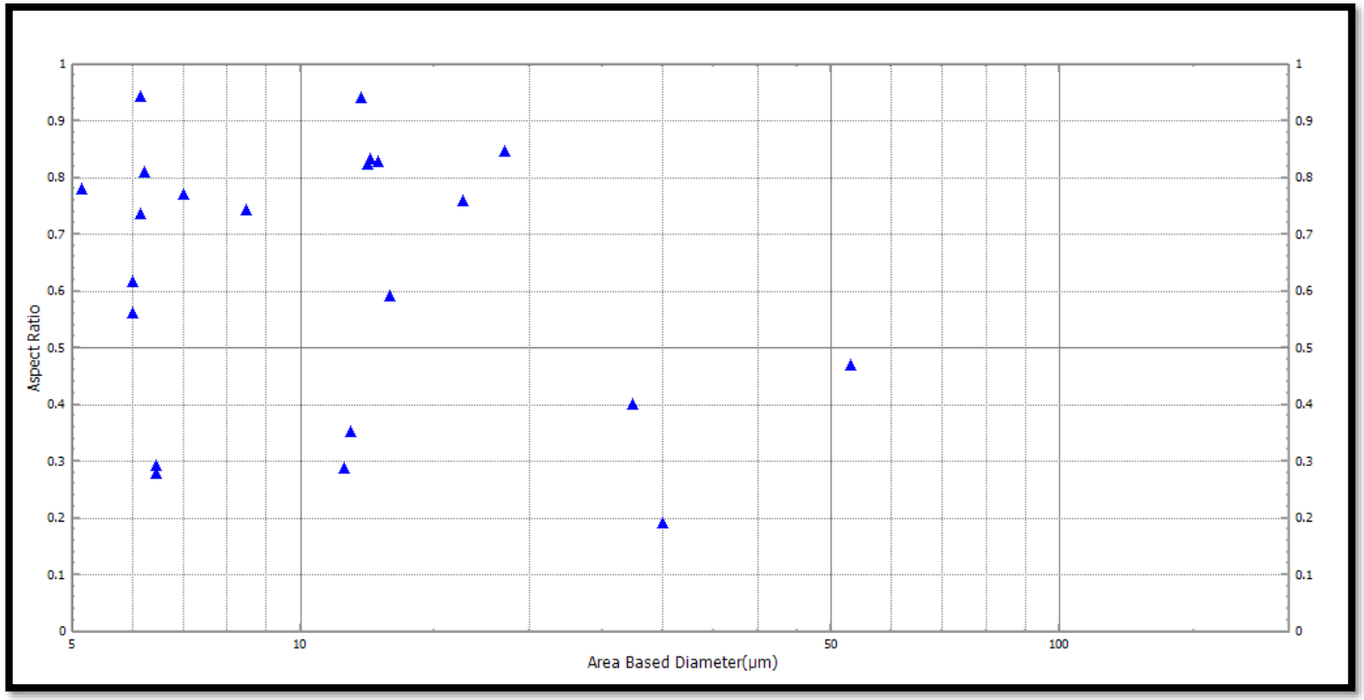
Determination of linearity detected a contamination problem 50 micron

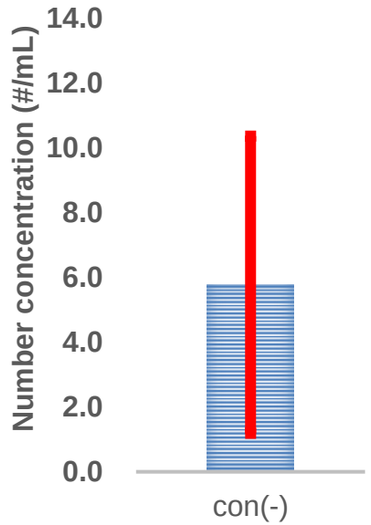






Blank evaluation showed some particles but not enough to affect linearity

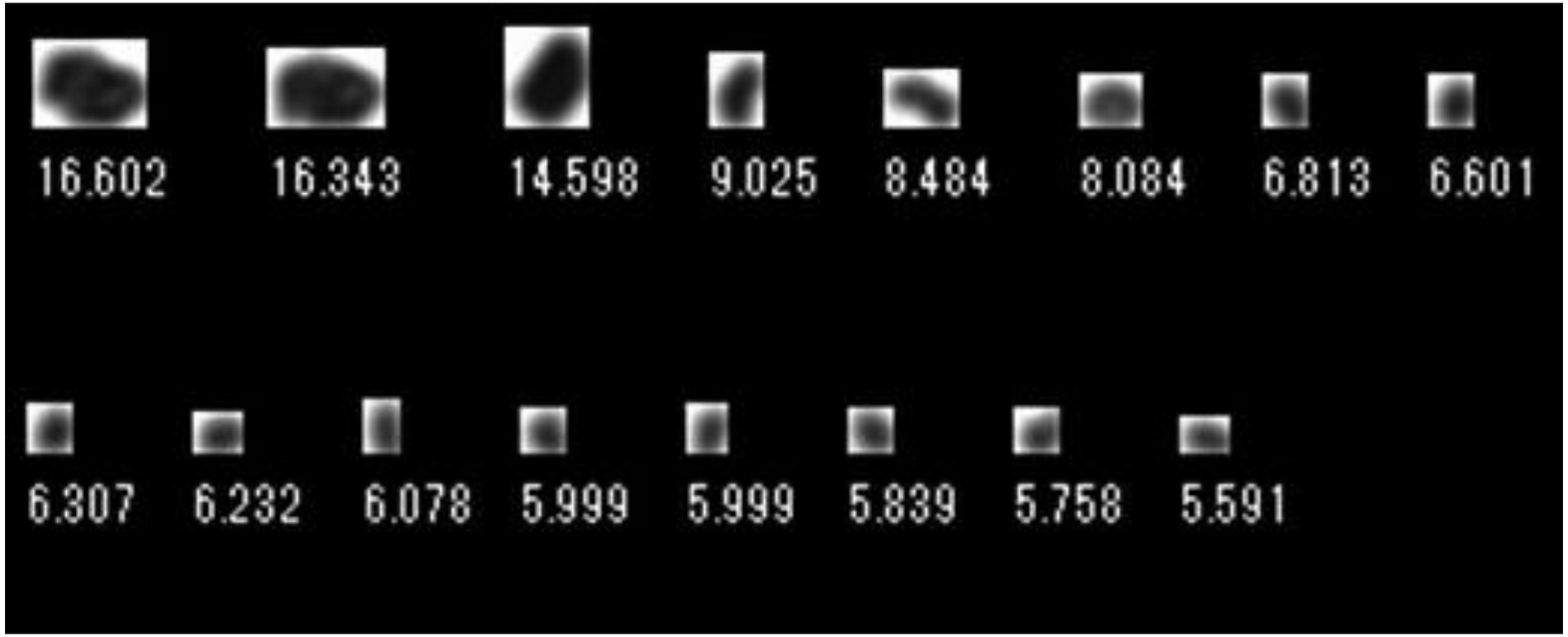




seven separate blanks were analyzed, according to the method, to find an average of 5.8 particles with a standard deviation of 4.6

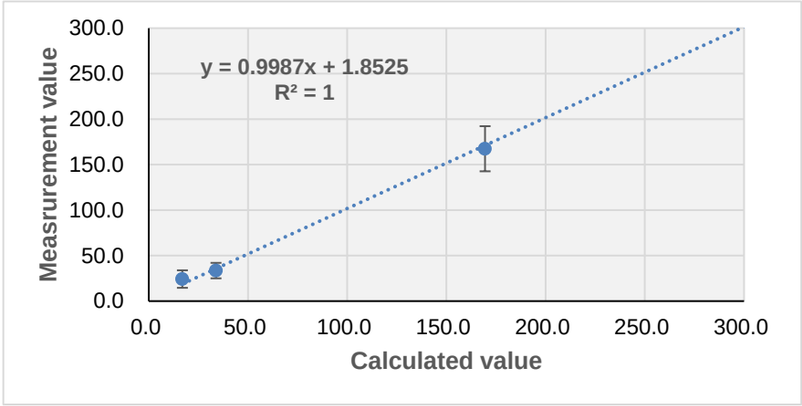
MDL ~ 15 particles /mL

The shape of the smaller particles was not spherical

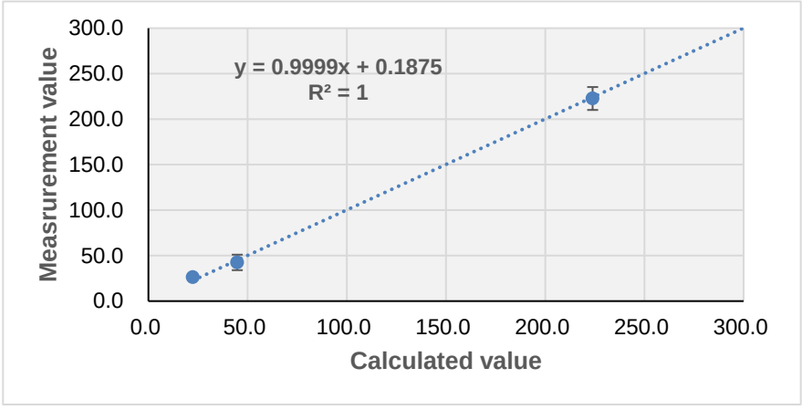


We assume the smaller particles are contamination in the standards and not introduced by our process or solvents → low blank

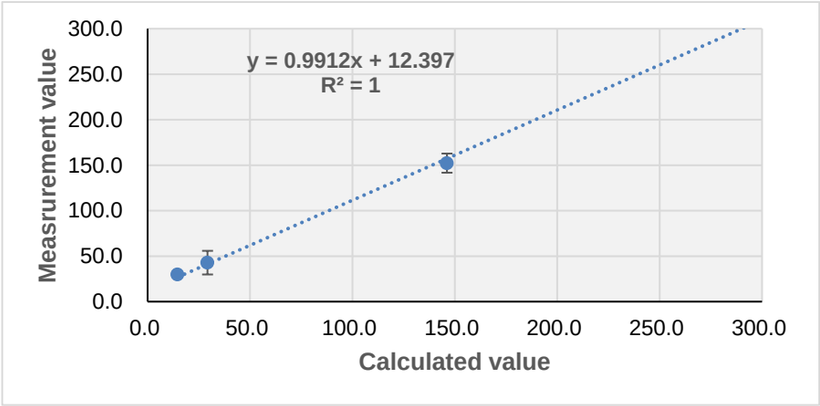
To measure standard beads correctly, we reject the smaller particles in the count



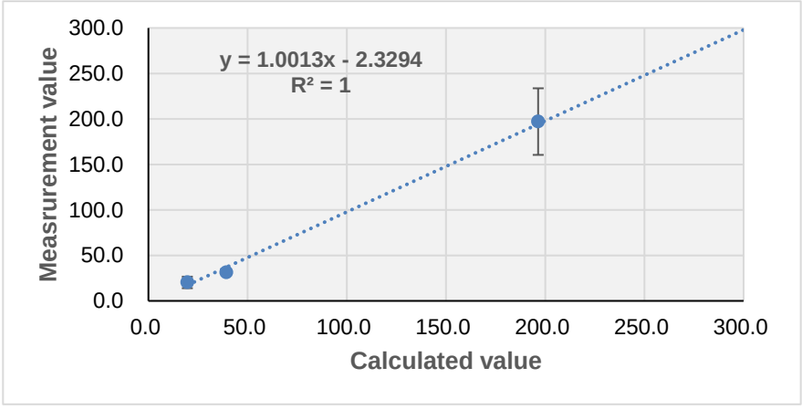
100-microns



20-microns



50-microns

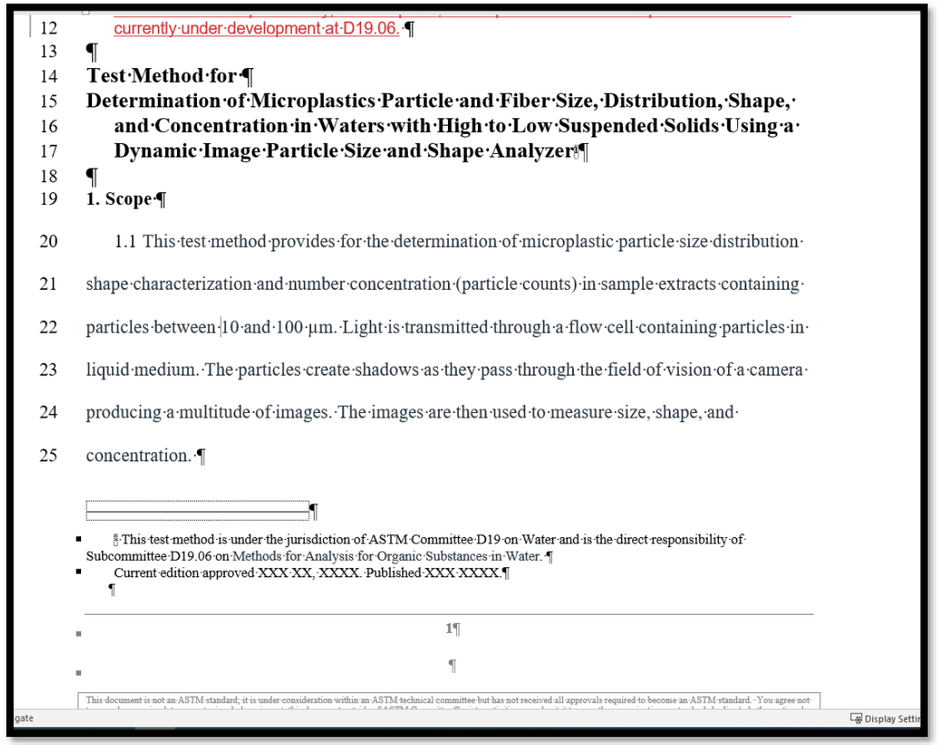


10-microns

Preliminary precision statement

Particle Size, µM	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



currently under development at D19.06. ¶

¶

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 ¶

¶

1. Scope ¶

1.1 This test method provides for the determination of microplastic particle size distribution shape characterization and number concentration (particle counts) in sample extracts containing particles between 10 and 100 µm. Light is transmitted through a flow cell containing particles in liquid medium. The particles create shadows as they pass through the field of vision of a camera producing a multitude of images. The images are then used to measure size, shape, and concentration. ¶

¶

This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water. ¶

Current edition approved XXX XX, XXXX. Published XXX XXXX. ¶

¶

1 ¶

¶

This document is not an ASTM standard; it is under consideration within an ASTM technical committee but has not received all approvals required to become an ASTM standard. You agree not

- 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?

wclipps@shimadzu.com