Accurate Number Distribution Measurements for Airborne Micron and Supermicron Particles by Shadowgraphy Method

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May 5, 2017

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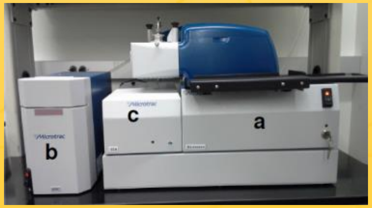
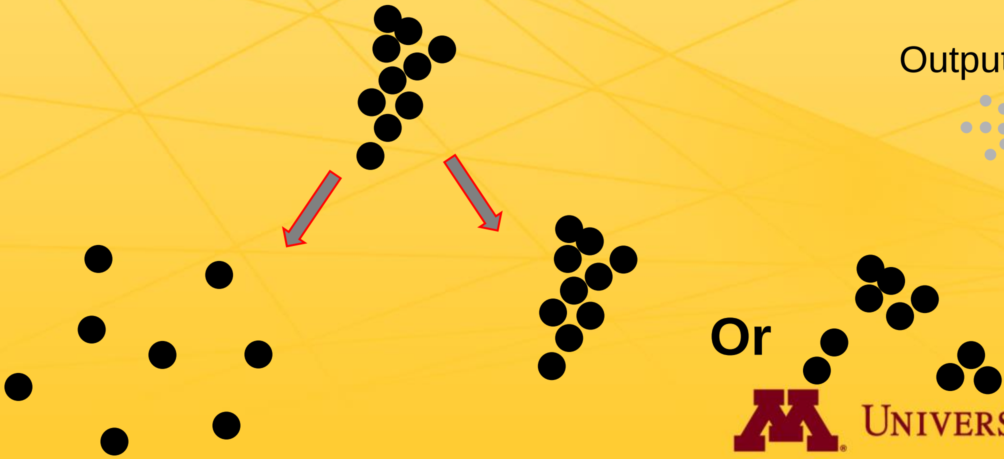
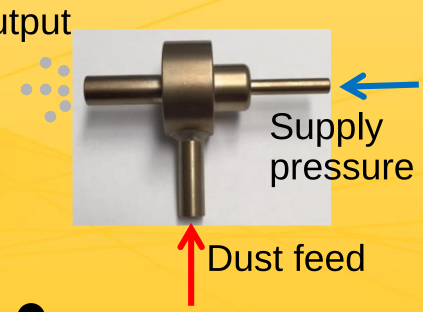
Introduction—dust size distribution measurements

- ISO dust injectors and other commercial dispersers are normally used to re-suspend standard dusts, e.g. ISO A2, for testing the air filters used in internal combustion engines and many industrial applications.
- Characterization of the size distribution and shape of the dust particles from the different dispersers are essential.
- However, it is very difficult to obtain a similar size distribution as that provided by the manufacturer for the re-suspended dusts.
- Powder manufacturer: Wet sample dispersion, adding surfactants, applying ultrasonication, laser diffraction instrument (e.g. Bluewave),
- General powder users: dust injectors, pressurized air, real-time aerosol instrument (e.g. APS, LPC).



Bluewave, Microtrac





ISO Injector

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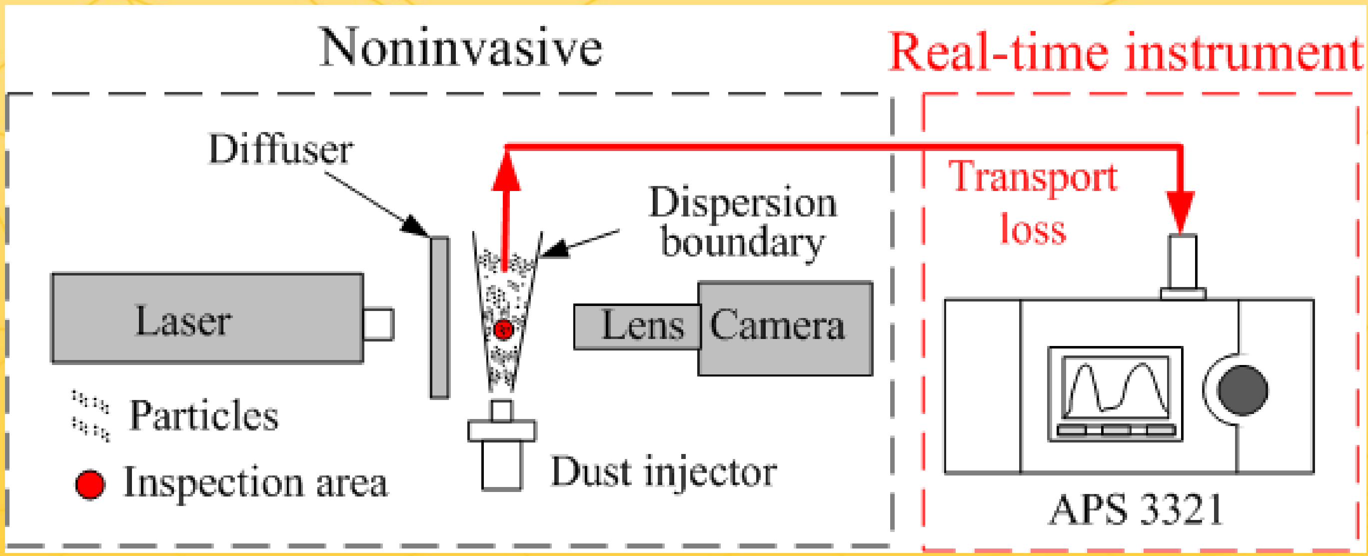
Introduction—noninvasive method

- Droplet measurement: shadowgraph technique has been widely applied to spray droplet studies [1-3].
- Solid particle measurement: Only few studies are found from literature (ice size measurement, dust velocity measurement).
- This study is the very first application of Shadowgraph for the measurement of dusts produced from standard ISO injectors



[1] Inthavong et al. Aerosol Medicine and Pulmonary Drug Delivery. 28, 59–67 (2015).
[2] Kashdan et al. Particle & Particle Systems Characterization. 20, 387–397 (2003).
[3] Lecuona et al. Measurement Science and Technology. 11. 1152–1161 (2000).

Challenge 1

Clear images of captured particles are desirable but not always available.

Challenge 2

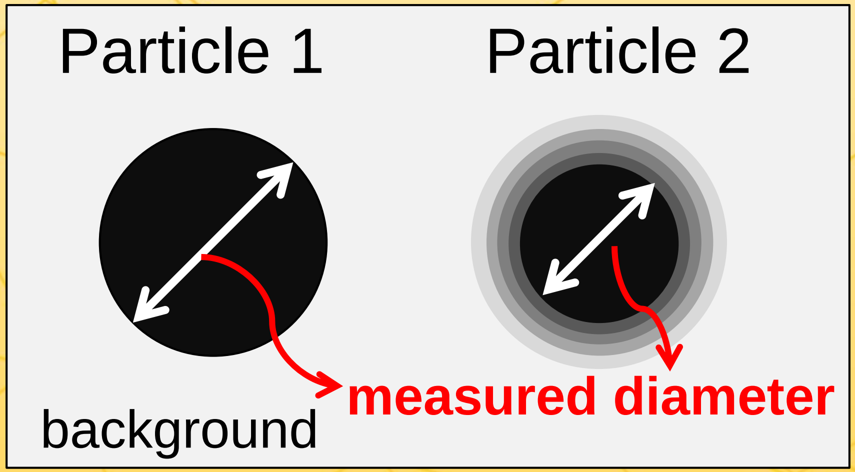
Many calibration methods have been developed for the accurate size measurement, but there is no method for obtaining accurate size distribution.

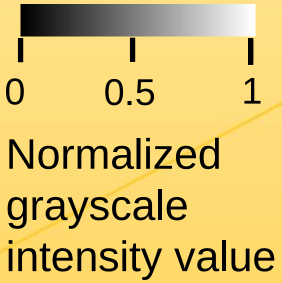
Objectives

- Develop an image post processing algorithm which is suitable for the dust characterizations, i.e. size, shape and concentration.
- Calibrate the current shadowgraphy method using known size and known concentration standard PSL particles.
- Provide correct number distribution for both airborne and liquid-borne particles produced by different generators using shadowgraphy technique and newly developed image analysis method.

Image processing algorithm

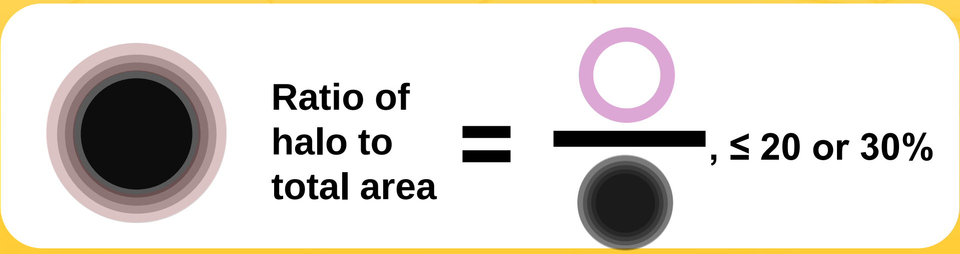
- Commercial image analysis software generally applies a background threshold value subtraction for particle recognition and detection, but more algorithms should be added.





- Degree of focus of particles should be considered for the determination of particle sizes.

- Distinguishing in-focus and unfocused particles automatically—excluding unfocused ones and improving sizing accuracy significantly.



Kashdan et al. (2004)

Kashdan et al. Part. Part. Syst. Charact. 20: 387-397 (2004).

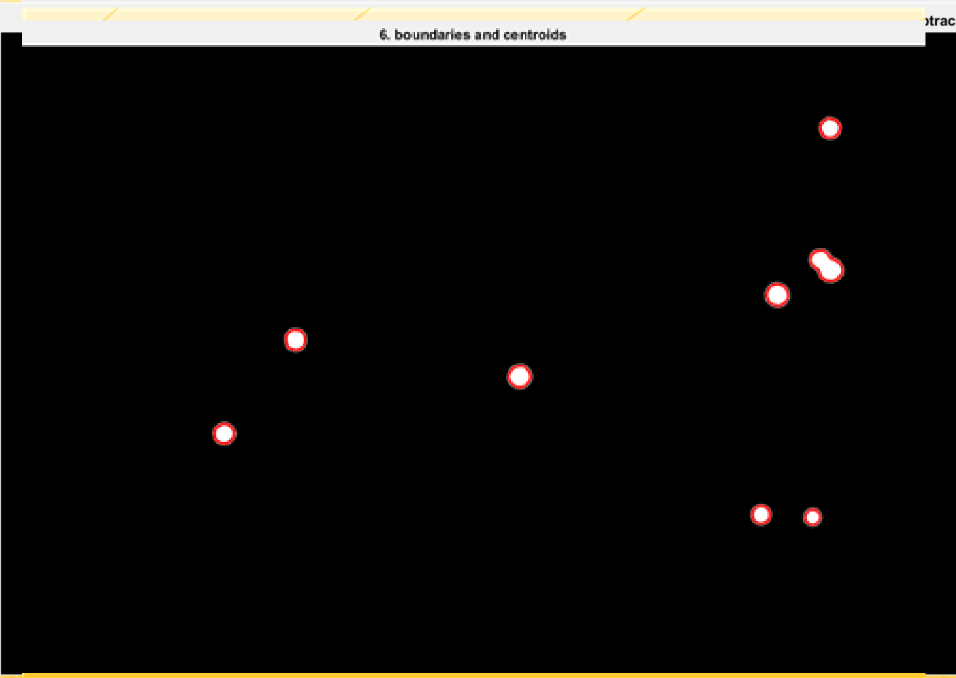
Flow chart of the current image processing procedure

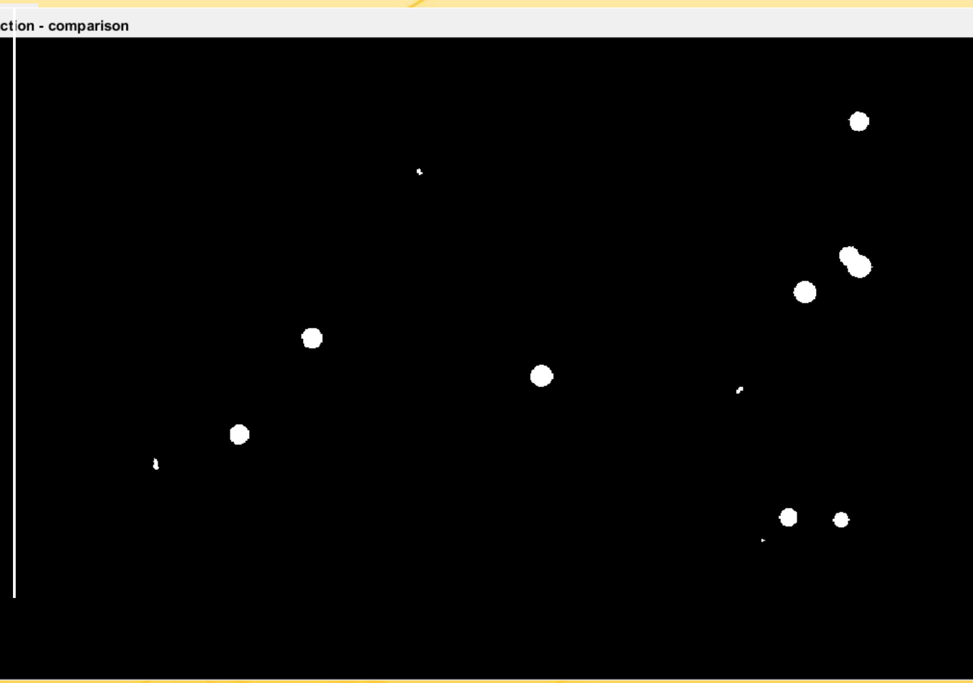
Image binarization	Threshold is chosen by I_I and I_O values
Particle boundary detection	8-connected pixels below the background intensity will be detected
Classification 1	1. Particles having pixels less than 8 are excluded 2. Dusts on the optical system are excluded by comparing background images 3. Particles touching the boundary are excluded
Classification 2	In-focus particles will be classified
Particle size determination	Individual particle size will be extracted using calibration data
Particle velocity calculation	The velocity of individual particle will be calculated by tracking the moving distance of each particle (not shown)

Image Analysis

Raw image

Selected images





1. Gray scale conversion and detect particles above certain value of threshold value (same as the previous and current code)

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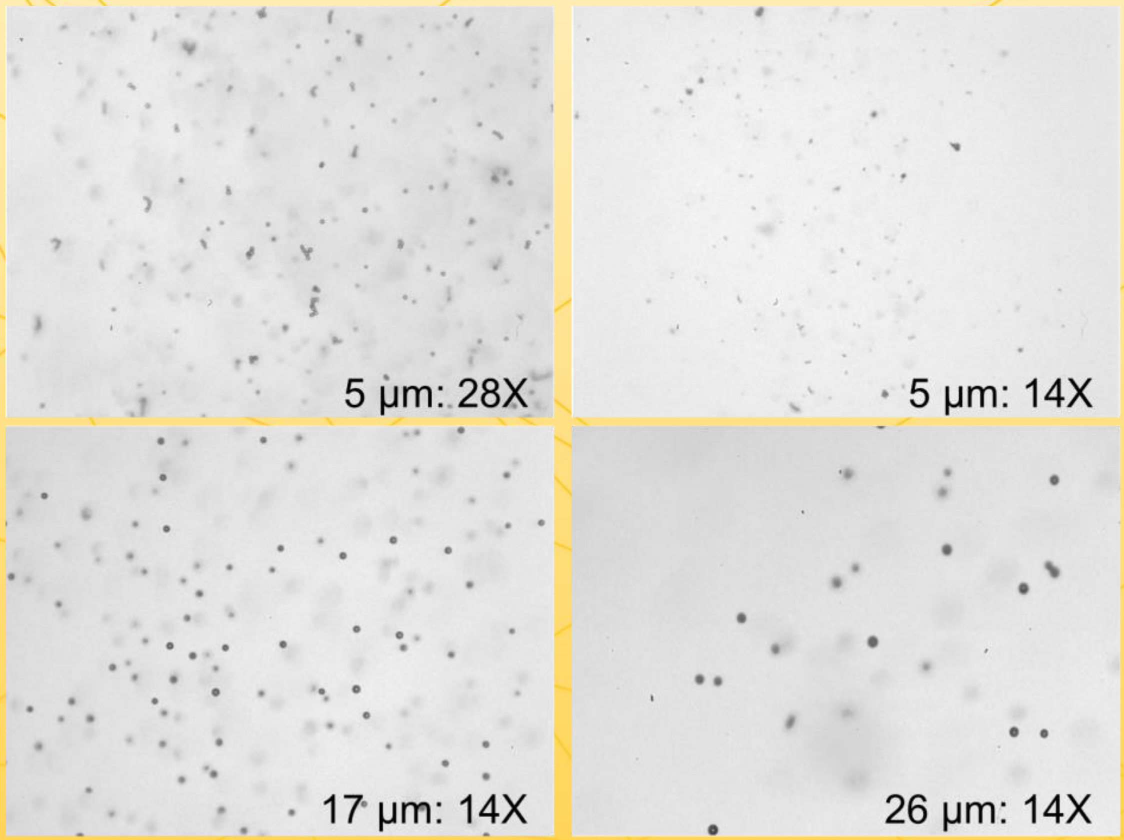
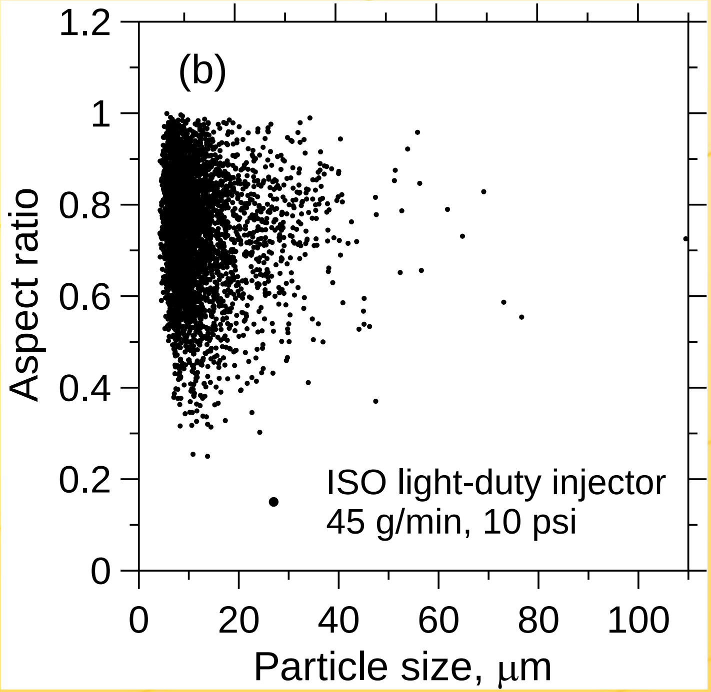
Accurate particle sizing and aspect ratio measuring





Aspect ratio distribution of A2 dusts

Magnification	Manufacturer	Measured diameter
14X	5 ± 0.5 µm	5.1 ± 0.4 µm
28X	5 ± 0.5 µm	4.9 ± 0.3 µm
14X	17 ± 2.7 µm	16.4 ± 1.5 µm
7X	26 ± 3.9 µm	26.1 ± 1.8 µm
14X	26 ± 3.9 µm	26.0 ± 2.7 µm

Kang et al. (2017)

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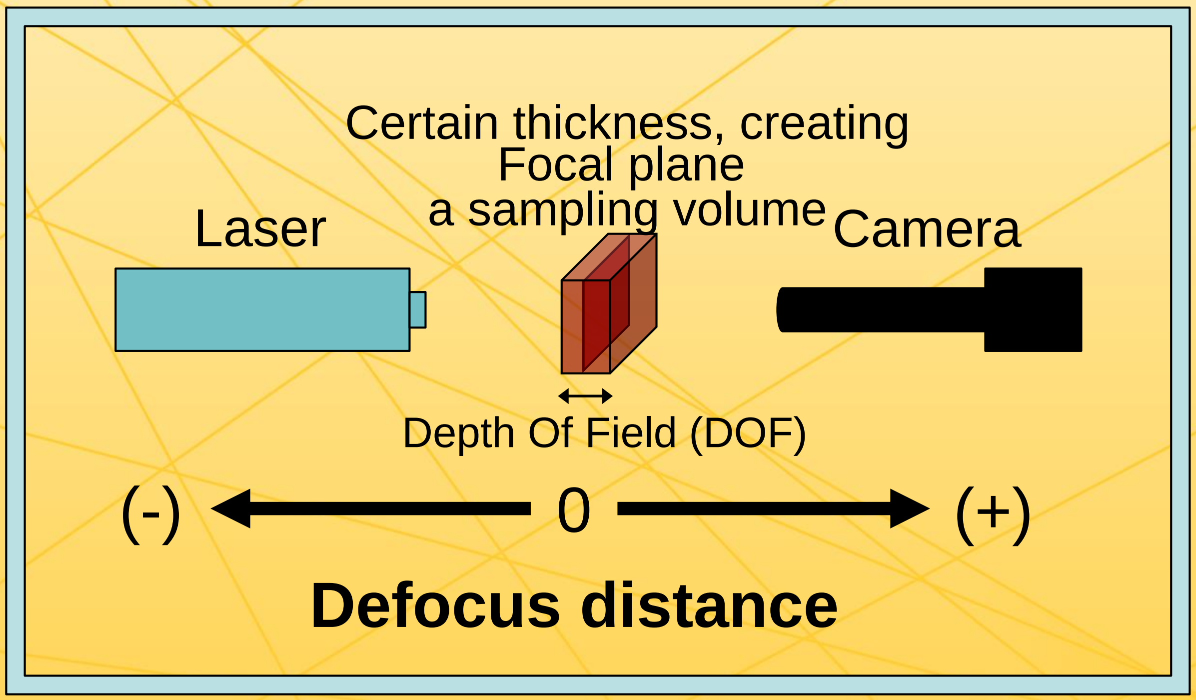
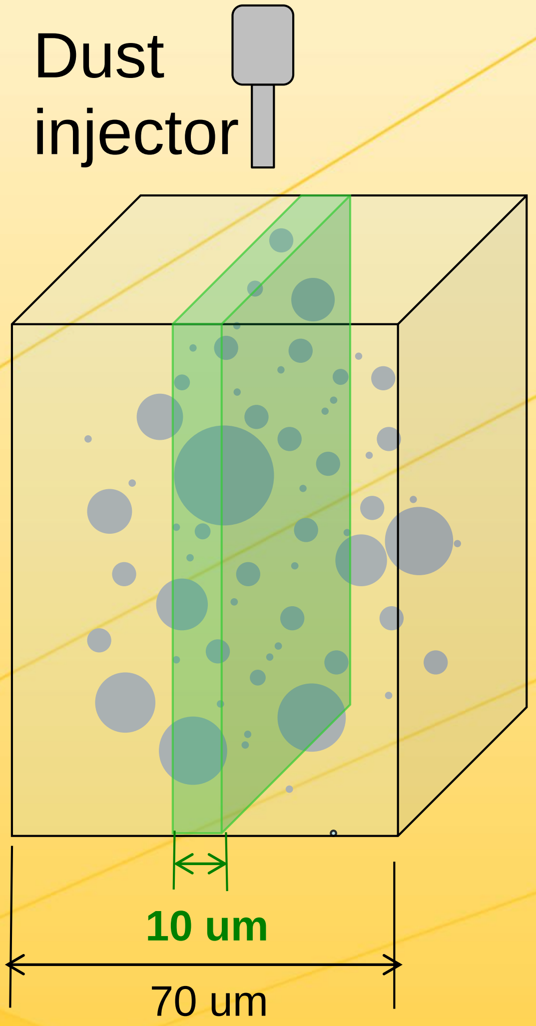
Method for measuring particle concentration





- Particle sizes are in polydisperse, a chosen DOF is usually too big for small particles, leading to the underestimation of small particles' concentration.
- DOF was found to be varied as a function of particle size and it was also influenced by both the threshold level and optical setup [4,5].

[4] Kim et al. Atomization and Sprays. 4: 65–78 (1994)
[5] Kashdan et al. Particle & Particle Systems Characterization. 20: 387–397 (2003)

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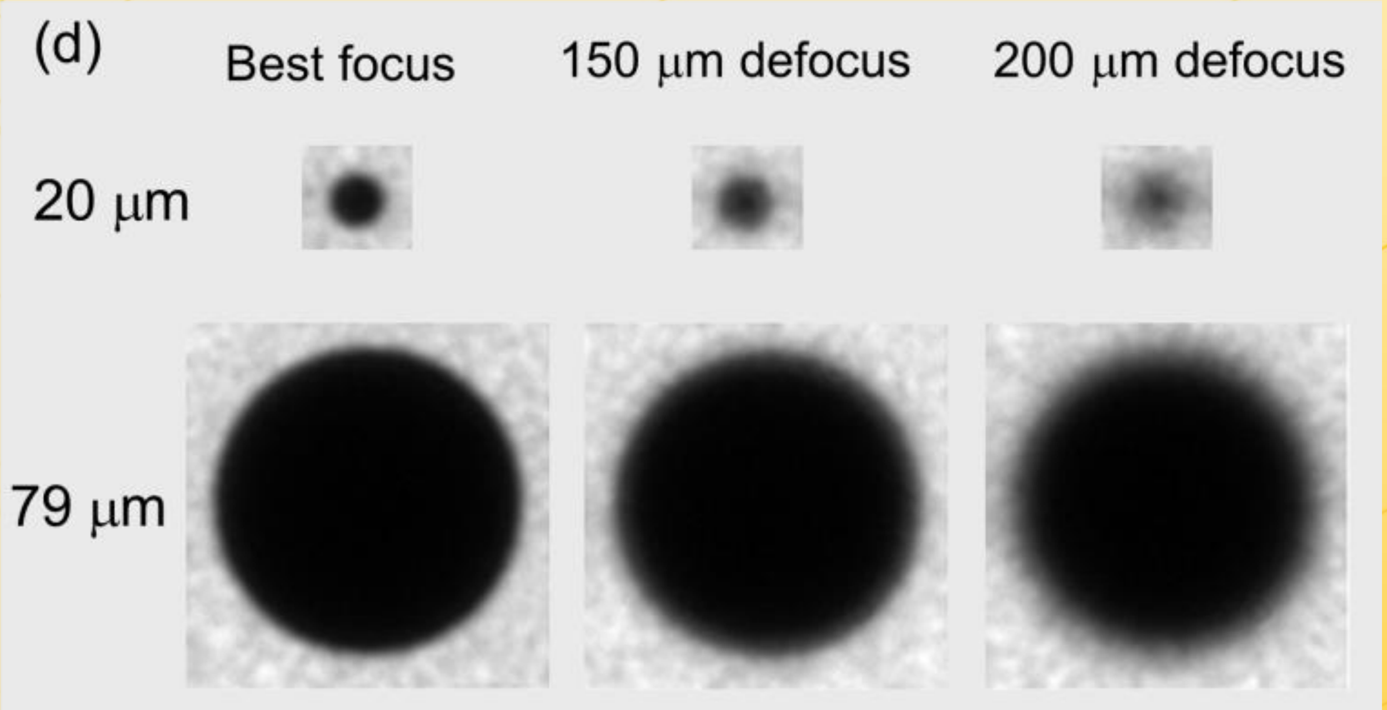
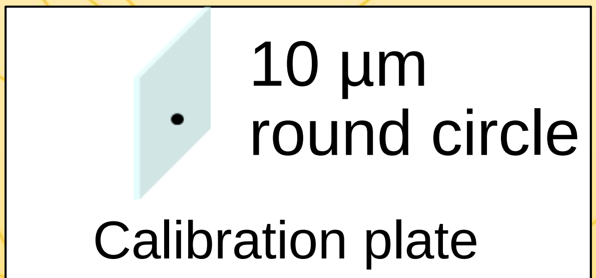
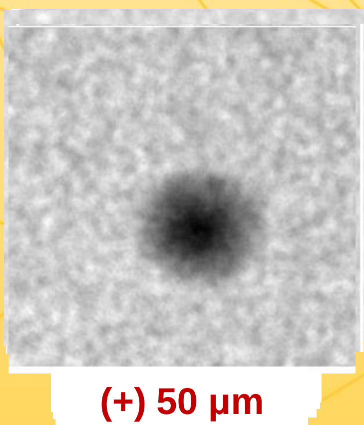
Effects of particle sizes on DOF (depth of field)-concentration issue



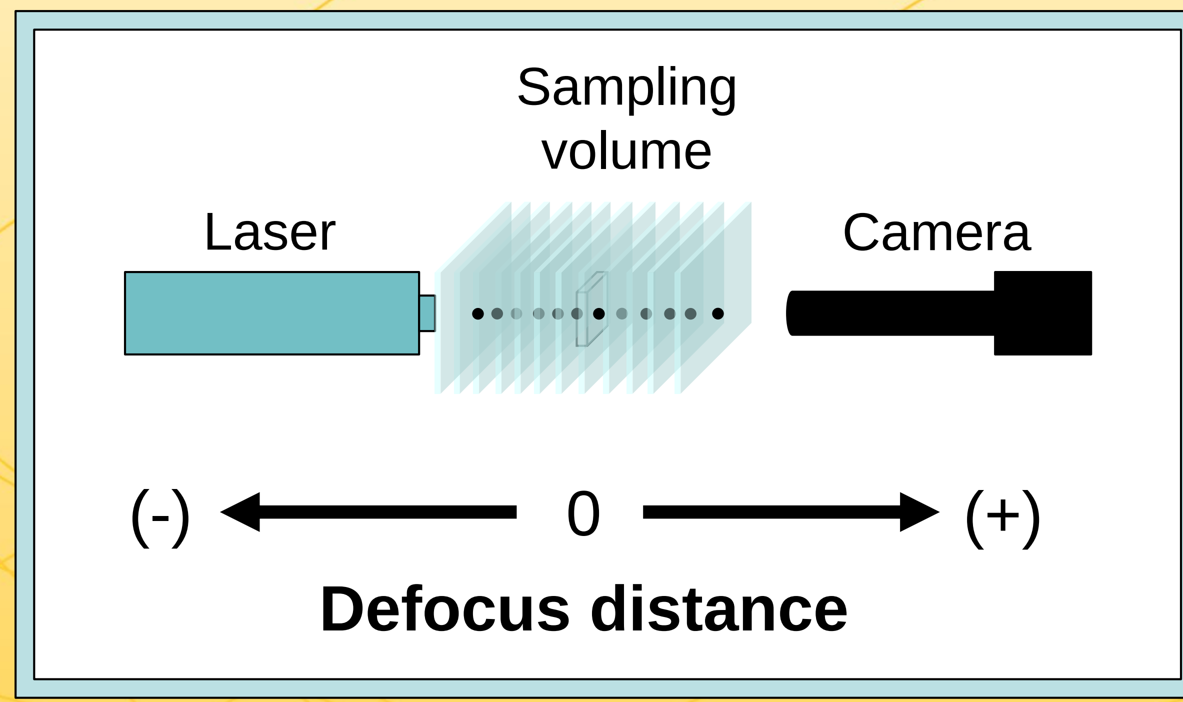
- The smaller particles were found to be defocused faster.
- DOFs should be investigated for each size of particles for the correction of particle size distribution.

Measurement of DOF for different sizes particles using calibration plate





Defocus distance



- Calibration circles with 10, 50, and 100 µm were measured at different defocus distances.
- Halo (blur area on the edge of the particle) width increases with increasing defocus distance.

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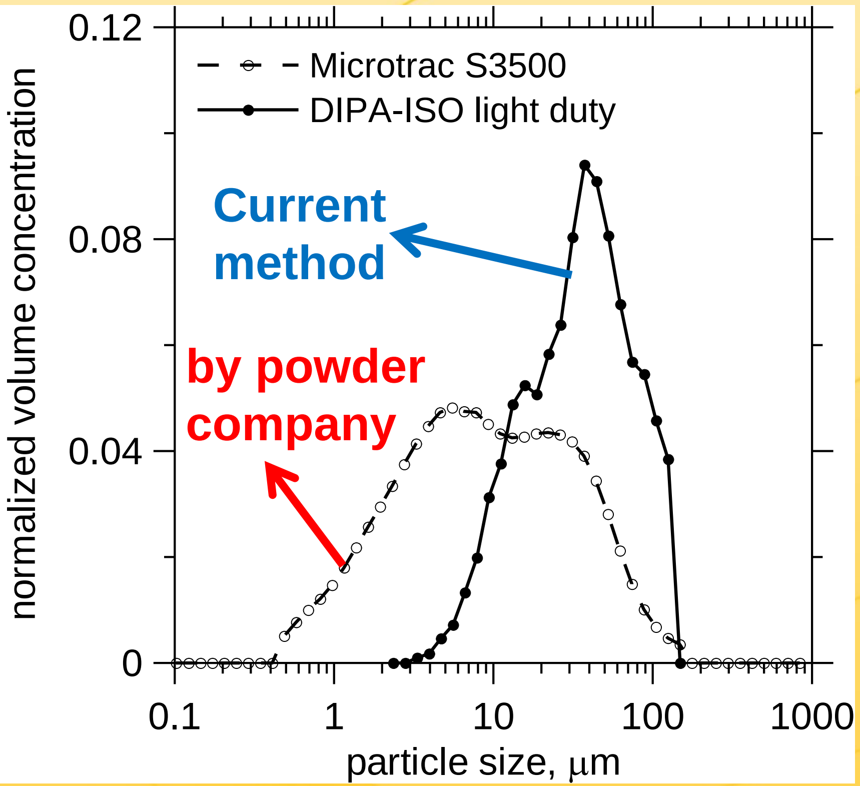
Kang et al. Separation and Purification, under review (2017)

Size distribution of ISO A2 dusts

$$Conc.(dx) = \frac{observed\ number \times weighting\ \ factor}{Sampling\ volume\ \ for\ 30\ \mu m\ \ particle}$$

- Using a fixed **sampling volume:** (Inspection area x DOF) of 30 μm particle.
- There is a significant difference of size distribution between PDIA method and that by the manufacturer done in liquid media.



Kang, Lai, Chen, Pui (2017)

Calibration of size distribution using known size and known concentration standard PSL particles

- Measurement conditions

a) Types of particle used

Type of particles	case	Particle size in mixture (μm)
PSL	1	1.6, 4, 10, 20, 50
	2	1.6, 10, 50
	3	4, 20, 50
	4	1.6, 4, 10
	5	4, 6, 10, 15
	6	15, 20, 40, 50
A2 fine dust	7	
A2 fine dust (customized)	8	0 - 3
A2 fine dust (customized)	9	0 - 5
Aluminum particles	10	40 - 70

b) Magnification levels

Mag. Level	μm/pixel	num. of pictures	time taken
28x	0.18	500 up to 3000	5 min up to 30 min
19.6x	0.25		
14x	0.35	500 up to 1500	5 min up to 15 min
9.8x	0.50		
5.7x	0.88		
3.9x	1.25		
2.3x	2.15		

c) Prepared PSL concentrations

size	High	Med	Low
1.6	2.00E+07	2.00E+06	2.00E+05
4	3.00E+06	3.00E+05	3.00E+04
10	5.00E+05	5.00E+04	5.00E+03
20	1.00E+05	1.00E+04	1.00E+03
50	3.00E+04	3.00E+03	3.00E+02

PSL solution preparation

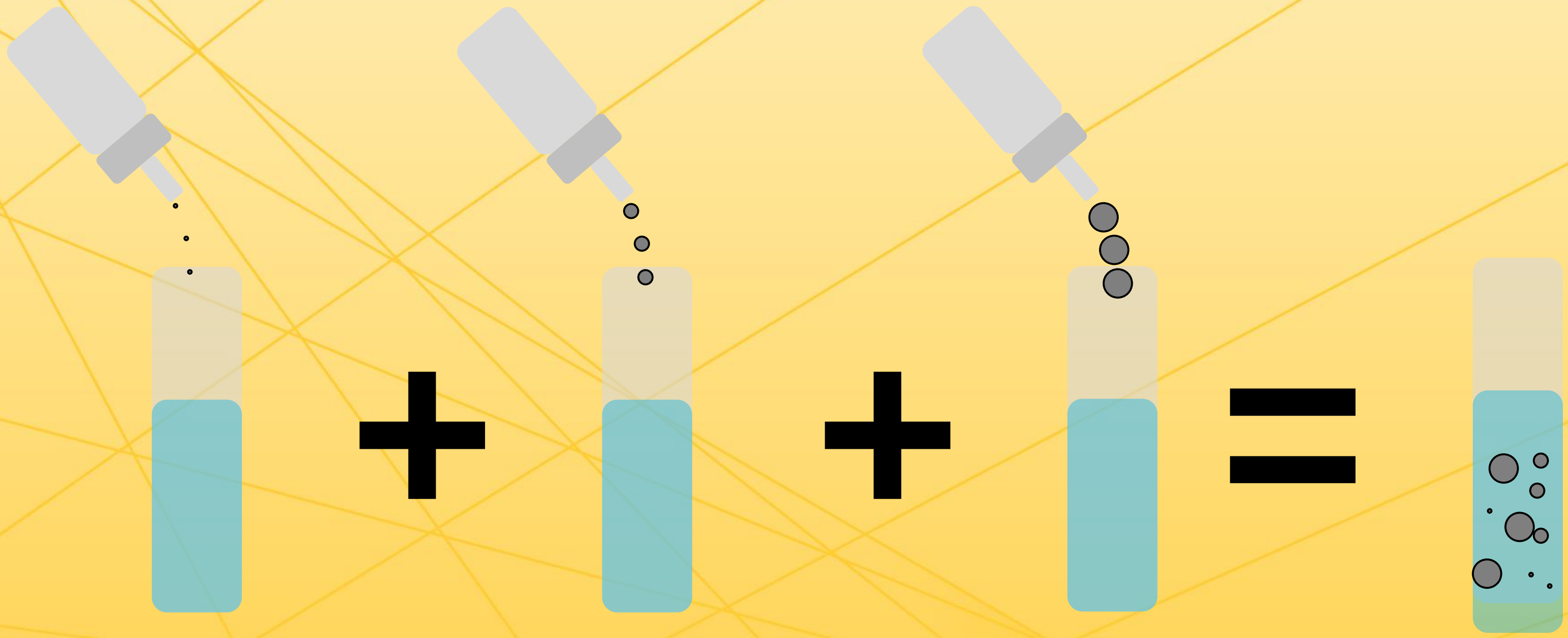


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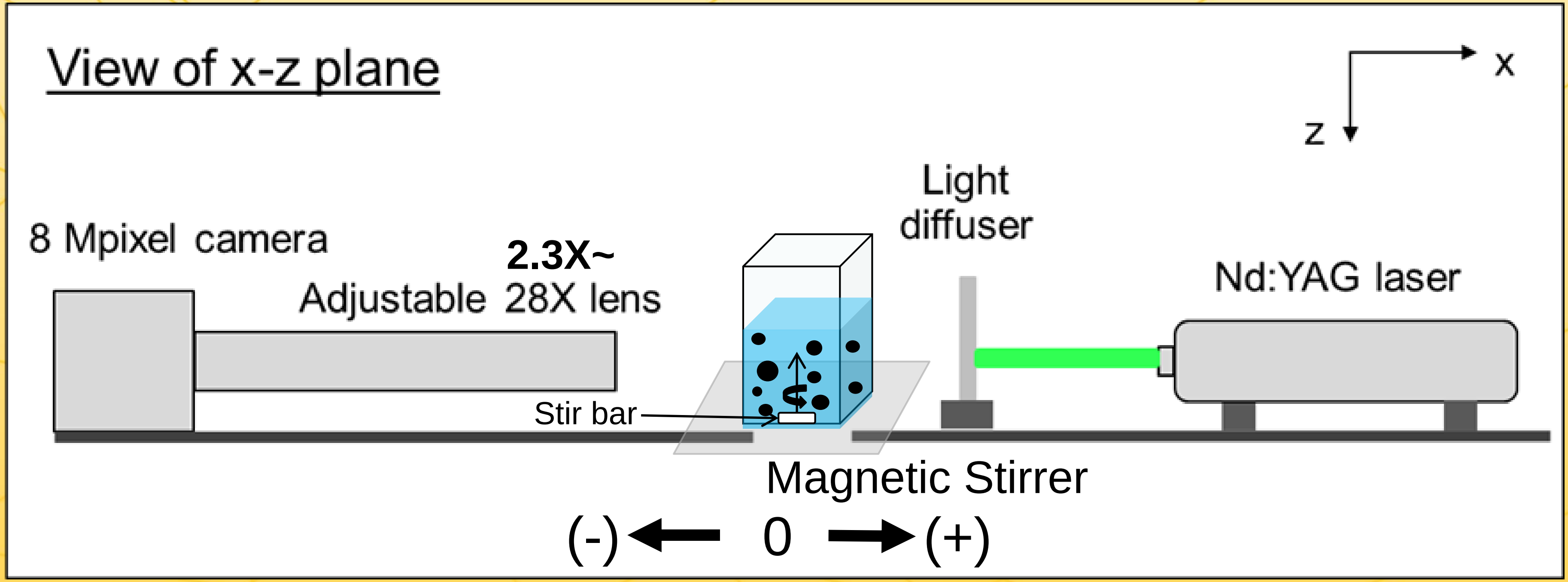
Image captures for the concentration calibration



- Mixture of standard PSL particle suspension was contained by the transparent cuvette.
- Stir bar and magnetic stir plate was applied to enhance the mixing and dispersity of the suspension.
- Translation slide was moved in and out to confirm the measured DOF values for different size particles from the calibration plate.

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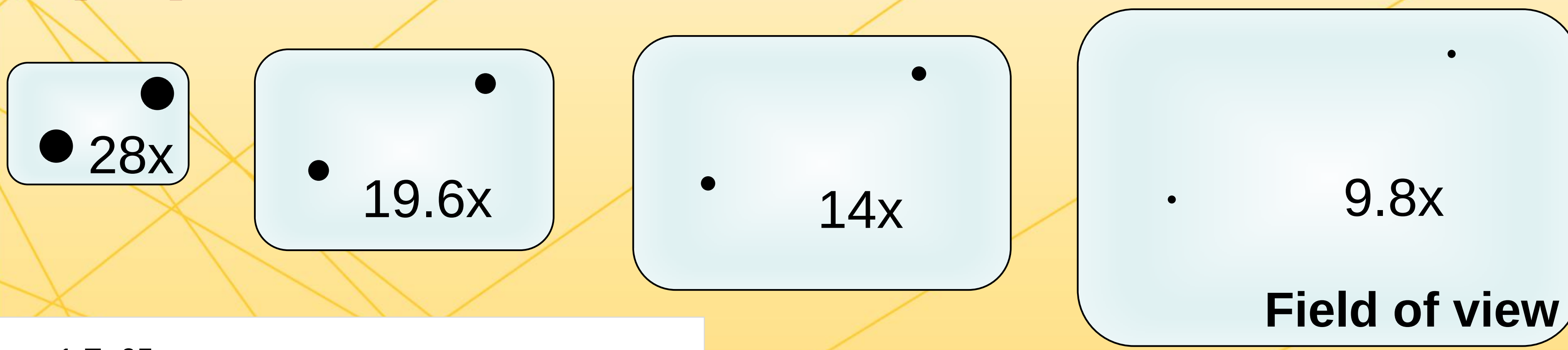
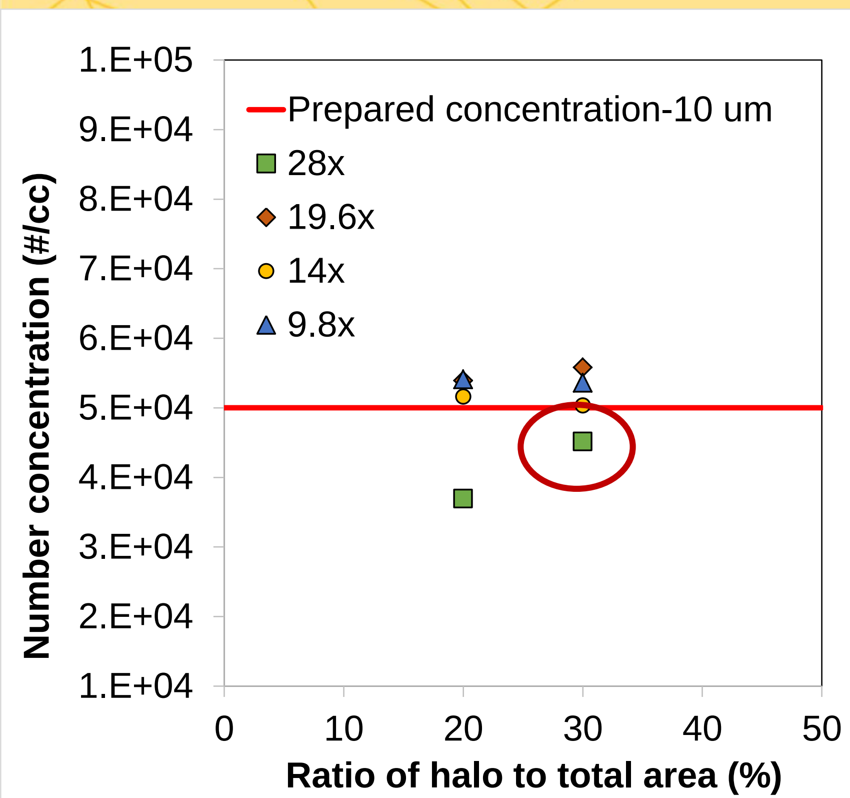
Concentration measured by DPIA for 10 μm PSL: prepared sample concentration = 50000 #/cc





$$Conc.\,(dx) = \frac{observed\ number}{Sampling\ volume\ \ for\ 10\ \mu m\ \ particle}$$

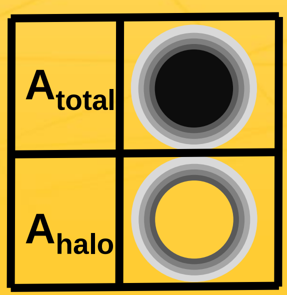
W= 0.5796 mm

H= 0.4354 mm

DOF= 0.07 mm

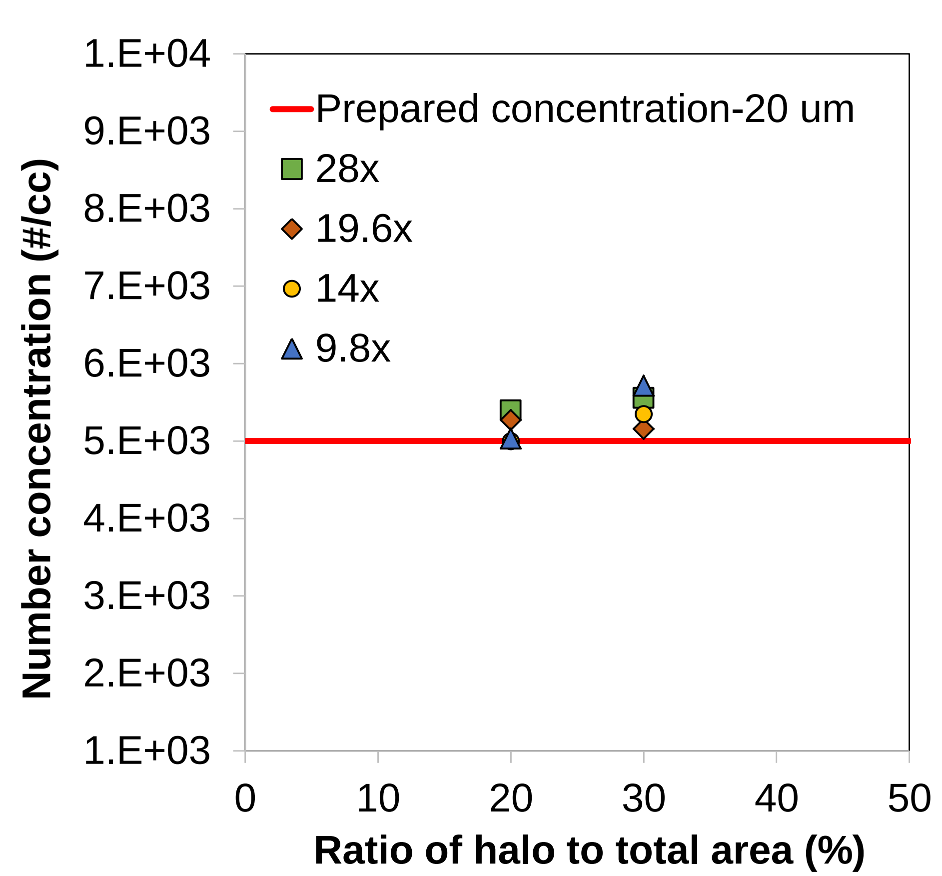
Sampling volume=1.767×10^{-5} cm^3

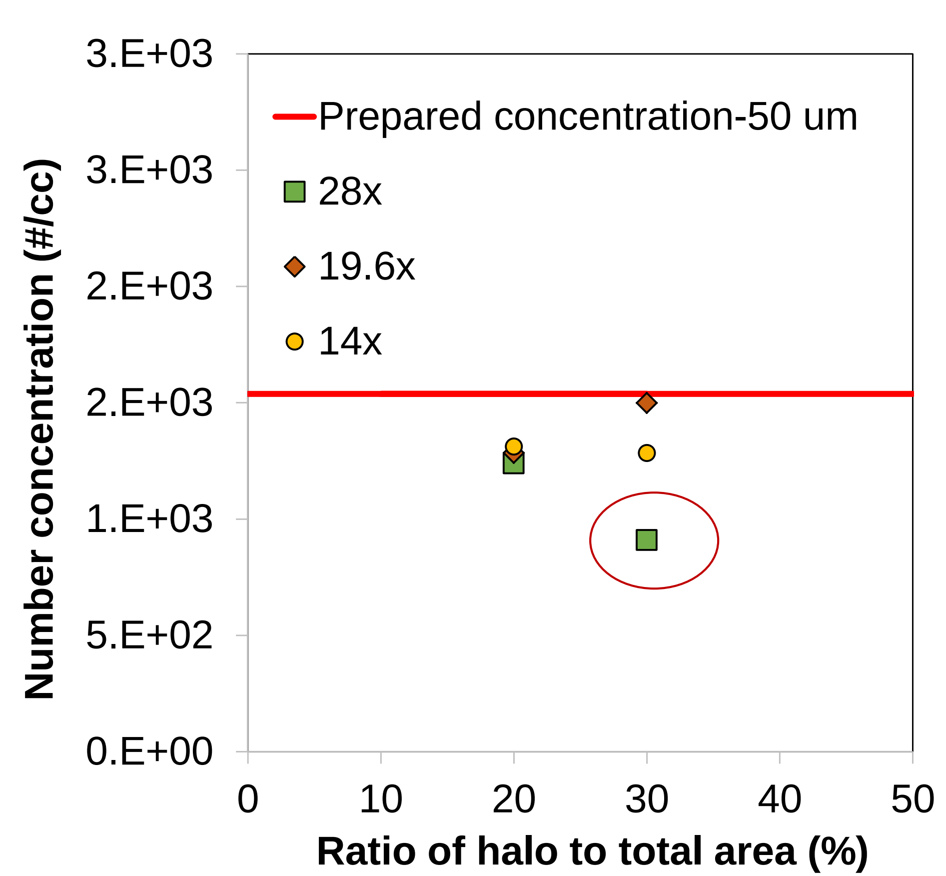
Average particle number on each image: 0.797



$$Conc.\,(10\ \mu m) = \frac{0.797\ \#}{1.767\times10^{-5}\ cm^3} = 45117\ {}^{\#}\!/_{cm^3}$$

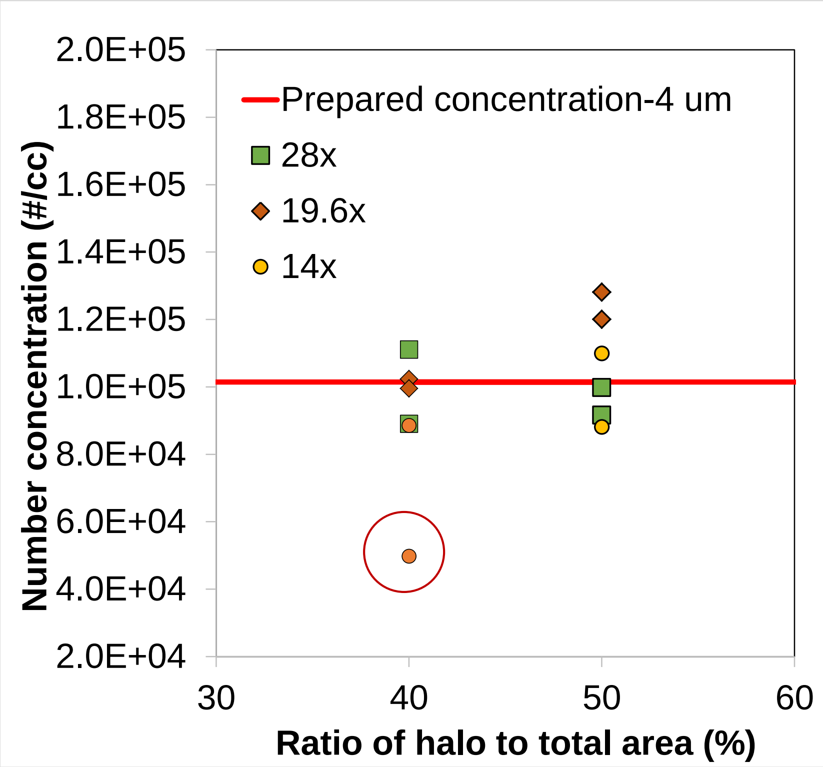
20+50 um PSL Mixture

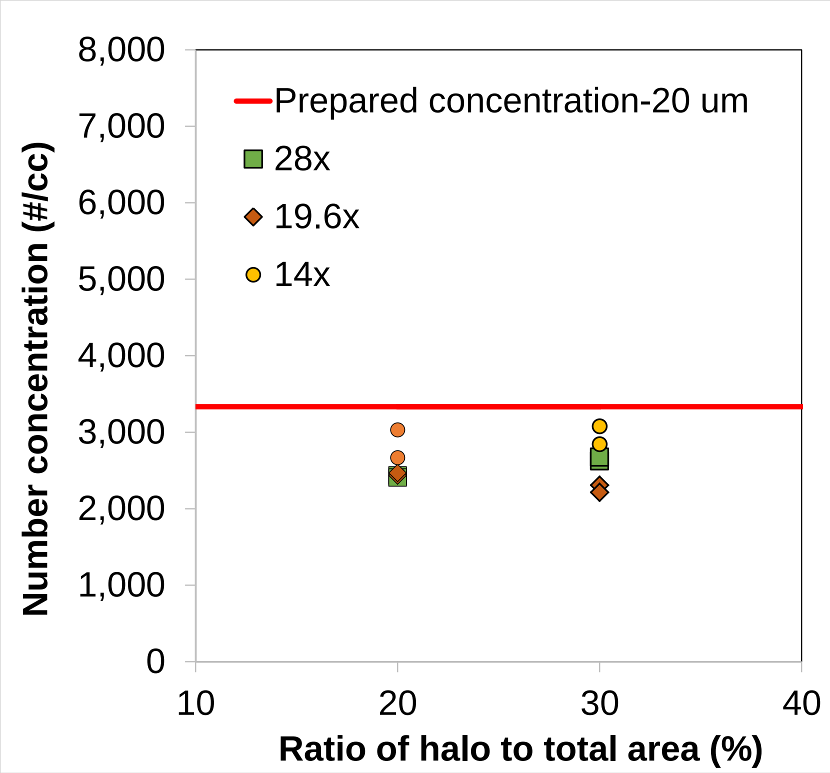




- Very good agreement between prepared and measured concentration for 20 μm PSL particles.
- Under 28x, the capture 50 μm particles were too few, leading to the large difference.

4+20+50 um PSL Mixture





- Very good agreement between prepared and measured concentration for 4 μm PSL particles except the 14x at 40% halo ratio-using too few of pixels to describe the particle.
- Difference between prepared and measured concentration for 20 μm PSL was less than 30%.

Summary

- Shadowgraphy technique, a noninvasive method, is successfully applied to characterize ISO injectors for ISO A2 dusts re-suspensions.
- Size distributions of A2 dusts were quite different between PDIA and that by manufacturer (liquid-borne, laser diffraction), which was mainly due to the different principle of measurements and level of deagglomeration.
- PDIA was further calibrated using known size and known concentration standard PSL particles. It was found the PDIA was able to not only accurately measure particle sizes but also directly measure particle concentration.
- Particles smaller than ~20 μm, a microlens with magnification higher than 14x should be applied, in comparison, lower magnification, < ~14x should be used for particles larger than 50 μm.
- This method should be widely applied for measuring in-situ size distribution of dust particles used to challenge the filters as well as particles in liquid.

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Future work

- 40x magnification (~0.12 µm/pixel) lens may be applied to measure particles below 1 µm, typically for the dusts with sub-5 µm diameter.
- Do the DOF measurements for 0.5-3 µm particles using standard calibration plate and translation slides with 1 µm step.
- Apply the newly developed codes to re-analyze all the previous images, including some field measurements.
- Help CFR companies to evaluate their dust dispersion systems (ISO injectors) for filter tests.