A New Spin on Particle Analysis: Introducing Centrifugal Sedimentation

partica Centrifuge

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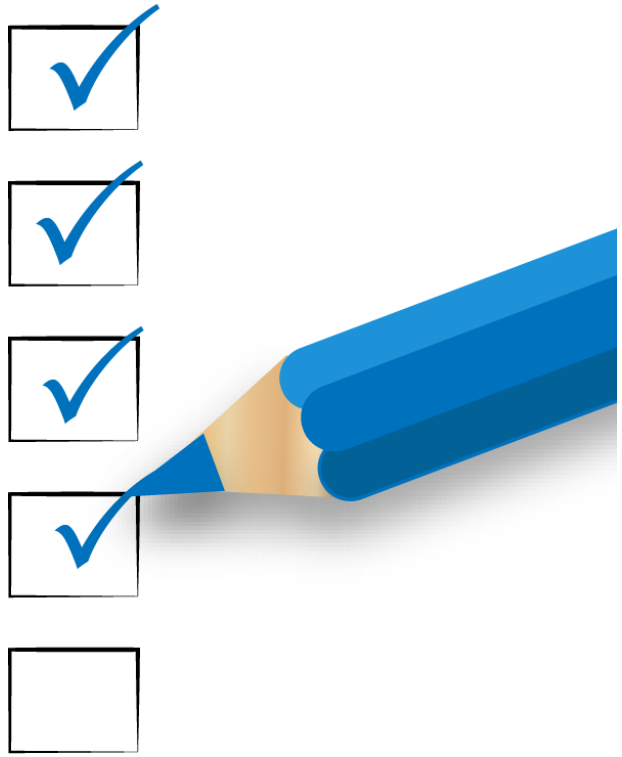
Stokes' Law Requirements

- **Particles must be in a Laminar Flow condition**
- **Can never measure very large particles due to Turbulent Flow**
- **Particles must be able to "block" the radiation source**
- **Particles must be adequately separated to avoid hindered settling**
- **Settling velocity must exceed the Brownian Motion**



Determining particle size from settling velocity

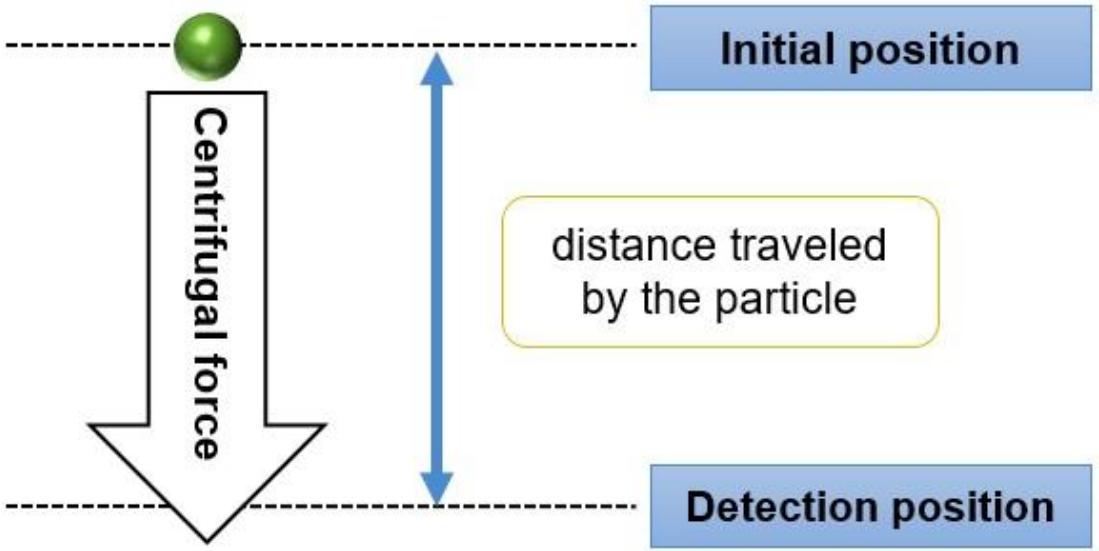
Stokes' Law

Settling velocity | Particle density | Particle size

$$V = \frac{1}{18} \times \frac{(\rho - \rho_0)g}{\eta_0} \times D^2$$

acceleration

Solvent Viscosity | Solvent density



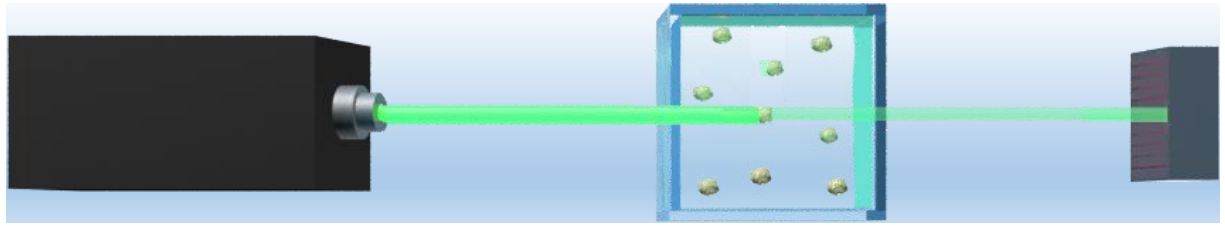
Stokes Law – Sizing by sedimentation speed

$$D(t) = \sqrt{\left(\frac{18\eta_0 \ln\left(\frac{x_2}{x_1}\right)/1000}{(\rho - \rho_0)\left(\frac{1}{3}\alpha^2 t^3 + \omega_0 \alpha t^2 + \frac{1}{3}{\omega_0}^2 t\right)}\right)} \quad (0 \le t \le t')$$

$$D(t) = \sqrt{\left(\frac{18\eta_0 \ln\left(\frac{x_2}{x_1}\right)/1000}{(\rho - \rho_0)\left(\frac{1}{3}\alpha^2 t'^3 + \omega_0 \alpha t'^2 + \frac{1}{3}{\omega_0}^2 t' + {\omega_{max}}^2 (t - t')\right)}\right)} \quad (t' \le t)$$

Symbol	Definition
$D(t)$	Particle size distribution (area based)
η_0	Dispersion medium viscosity
x_2	Distance from center of rotor to detection point
x_1	Distance from center of rotor to liquid surface
ρ_0	Sample density
ρ_0	dispersion medium density
α	angular acceleration
t	sedimentation time
ω_0	angular velocity at t = 0 [rad/s]
ω_{max}	angular velocity at 18,000 rpm

Beer's Law relates absorbance to concentration



$$Abs(t) = -logT$$

$$Abs(t) = -log\left[\frac{I_s(t)}{I_r(t)}\right]$$

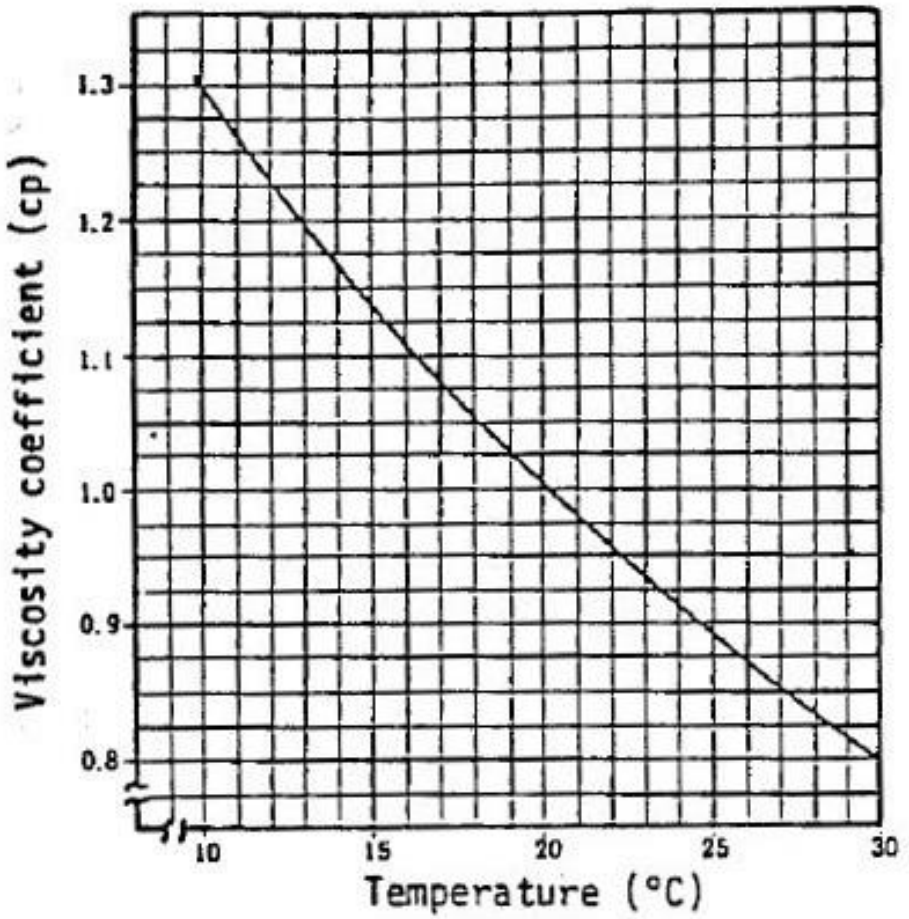
$$Abs(t) = \varepsilon bl$$

Symbol	Definition
Abs(t)	Absorbance
T	Transmittance
I_s(t)	Intensity of light transmitted through sample cell
I_r(t)	Intensity of light transmitted through reference cell
ε	Molar absorption coefficient
b	Molar concentration
l	Optical path length

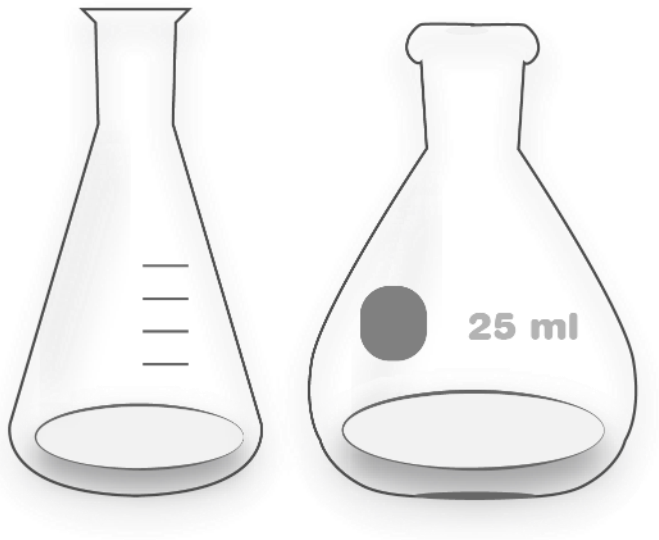
Stokes' Law Challenges

- Materials of different density in the same sample
- Small materials exhibiting Brownian Motion
- Various sources of hindered settling
- Slow measurement for gravity settling
- Cell movement in the case of accelerated measurements
- Non-absorbing particles in the case of X-ray detection methods
- Need for temperature control for the measurement

Advanced temperature control is essential for accurate measurements



Centrifuge Sedimentation Operational Procedures

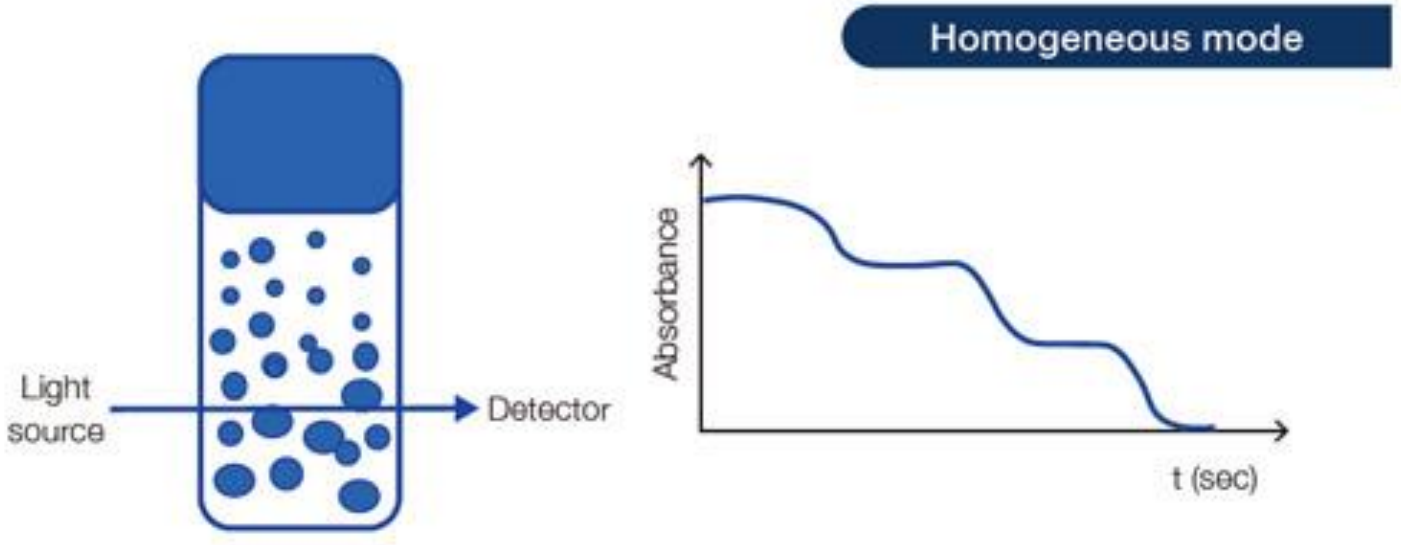


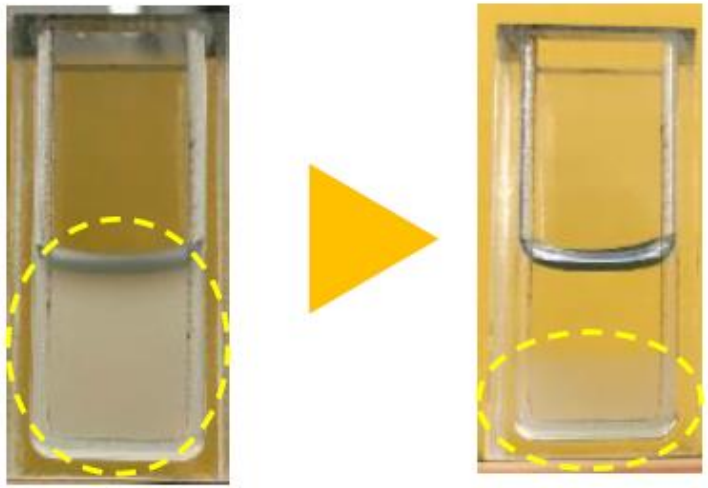
- **It is a first principle measurement method**
  - *Measure time and light intensity*
- **Simple data is required to be input**
  - *Liquid Density, Liquid Viscosity, and Particle Density*
- **Data is often easy to find**
  - *CRC Handbook or other reference source*
- **Parameters can also be measured experimentally**
  - *Graduated flask, viscometer, and a pycnometer required*
- **Following data input calculations are all automated**
  - *Many display options are available*

CN-300 Operational Improvements

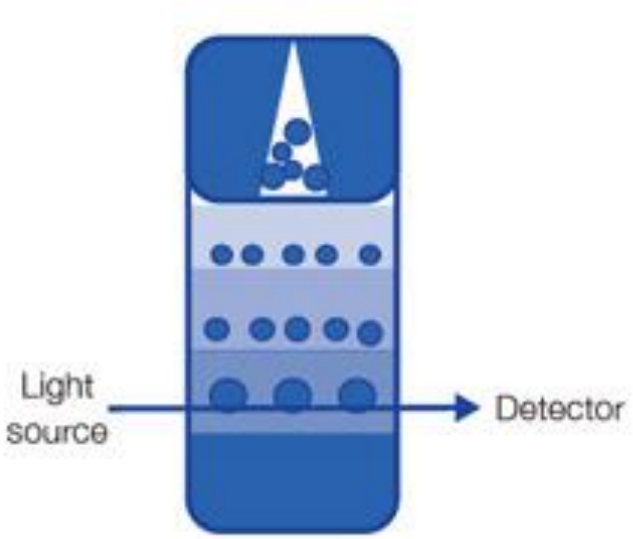
- **Cooling to stabilize the fluid temperature**
- **Higher speeds to measure smaller particles**
- **Gradient Centrifugal speeds for measuring broad distributions**
- **Combined Homogeneous and Line Start measurements**
- **Quiet design for low noise, even at high speeds**
- **Versatile data display capabilities**

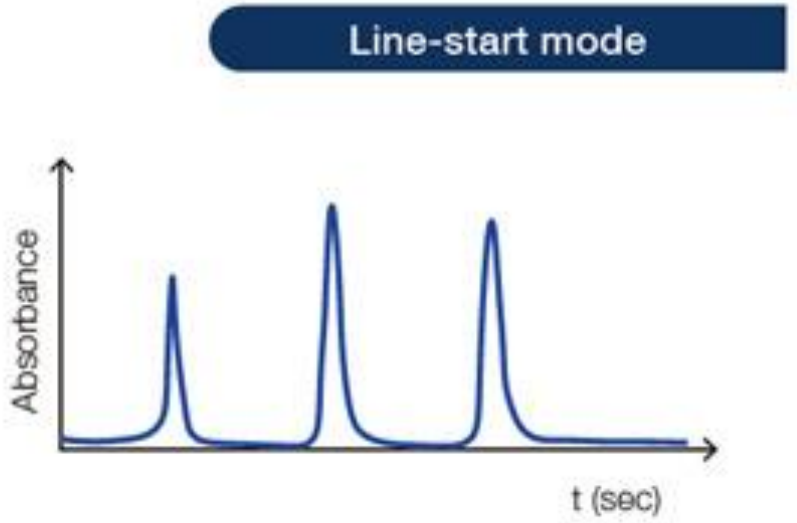
Homogeneous method for low concentration samples

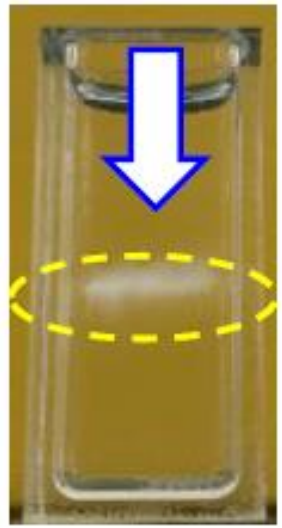




Line-start method for high concentration, high resolution



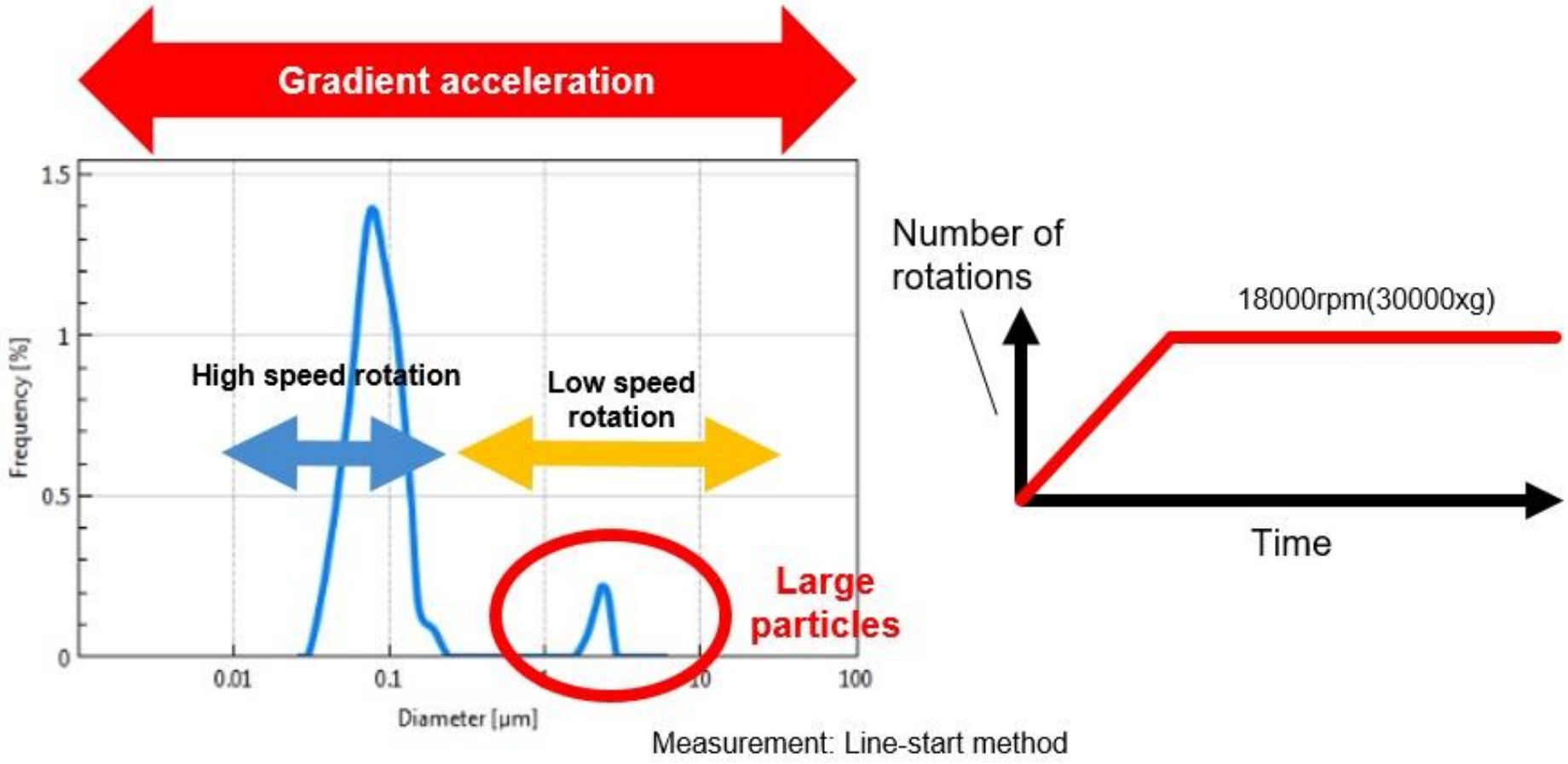




Density gradient fractionator

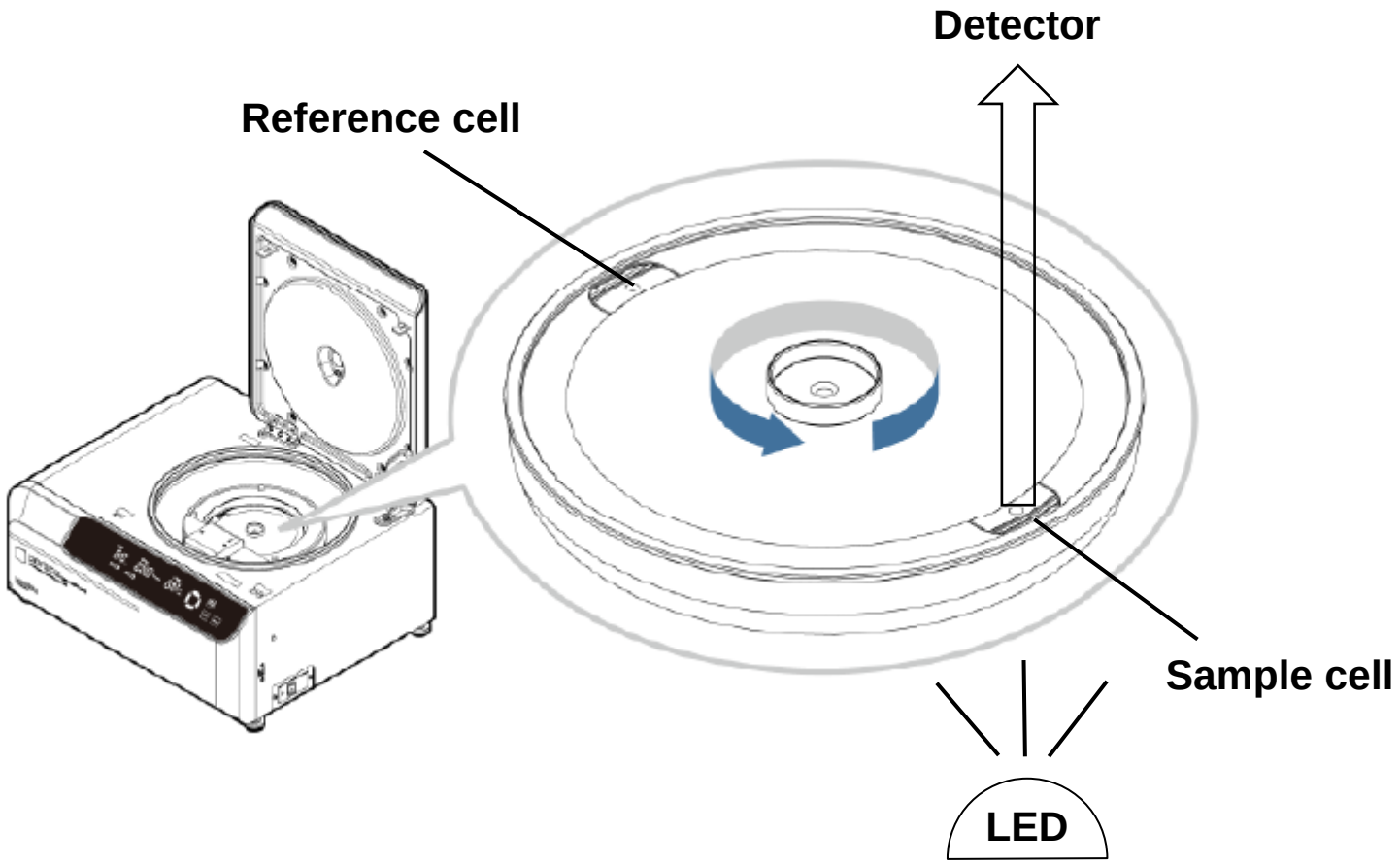
※Option

Gradient acceleration allows for wide range analysis



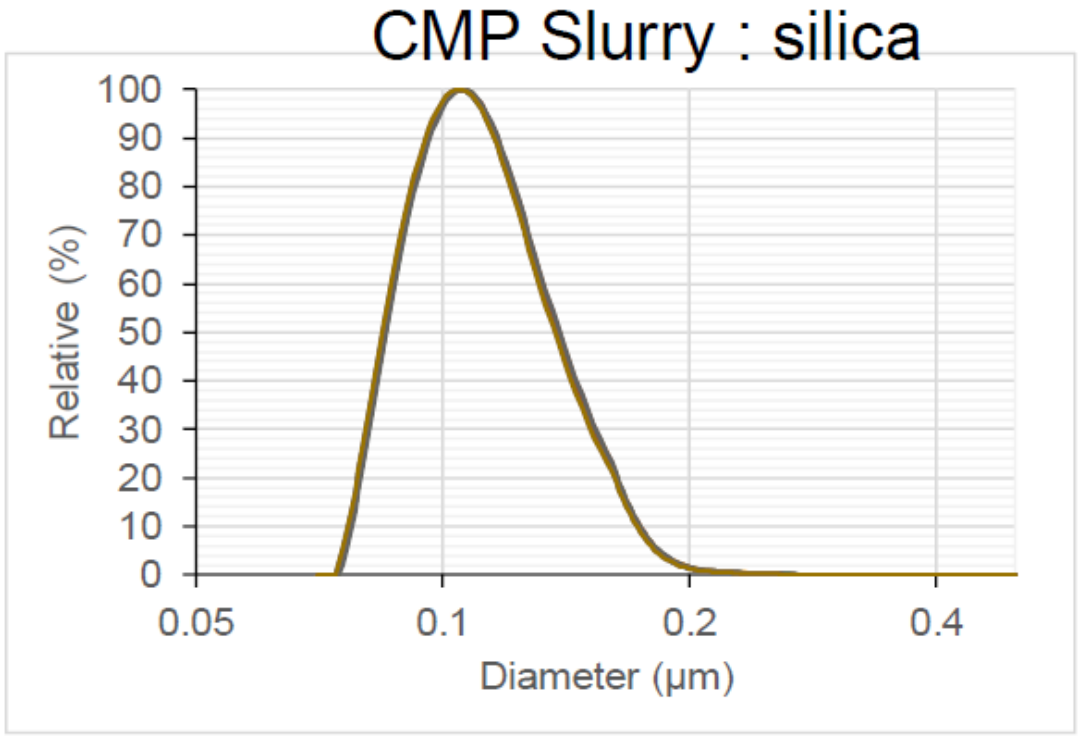
Measurement: Line-start method

Measurement principal diagram



Repeatability of silica

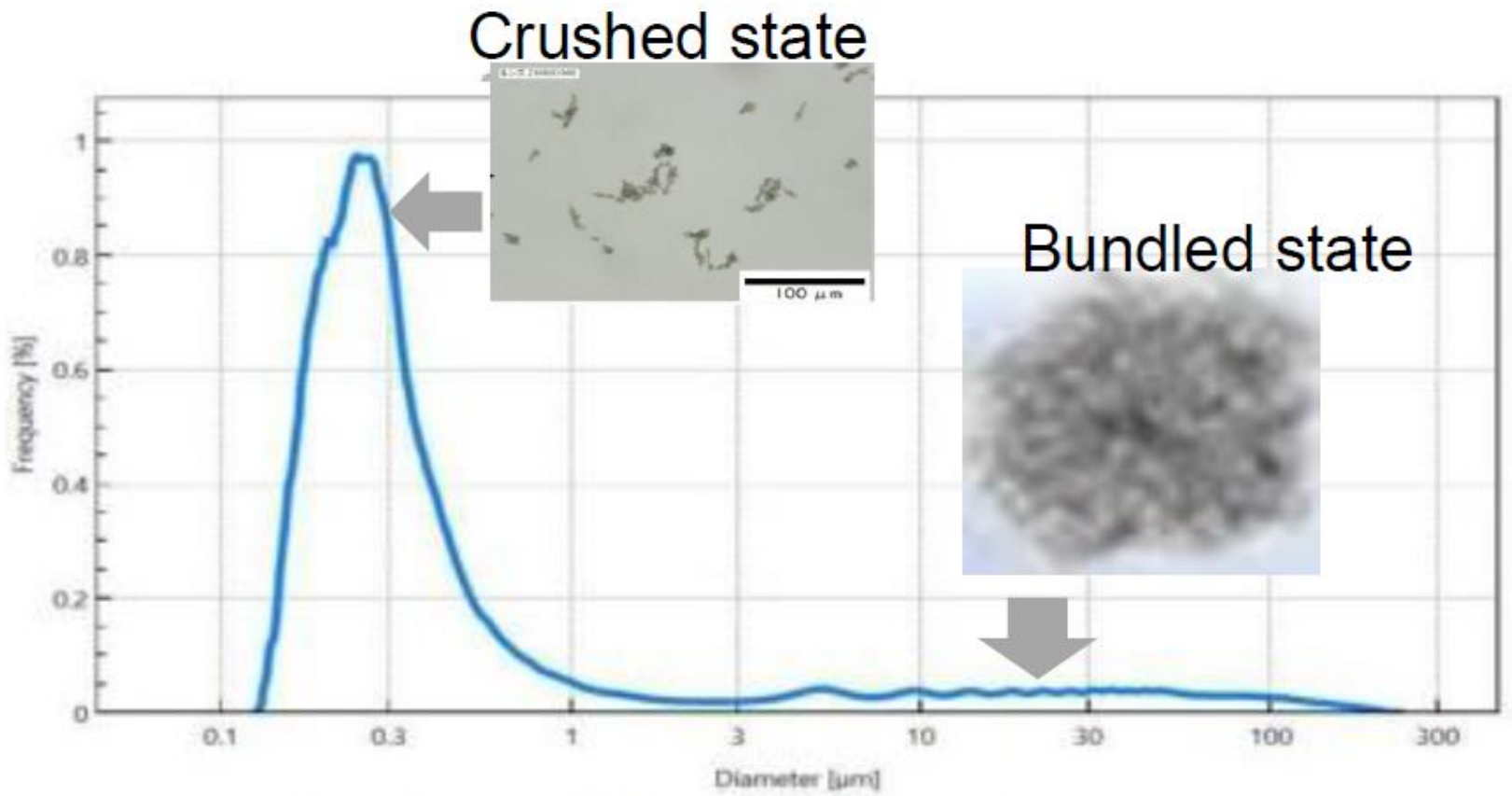
	Average [μm]		Average [μm]
1	0.1125	6	0.1131
2	0.1131	7	0.1123
3	0.1123	8	0.1130
4	0.1129	9	0.1135
5	0.1125	10	0.1119
		Average	0.1127
		CV	0.4 %



Method : Homogeneous

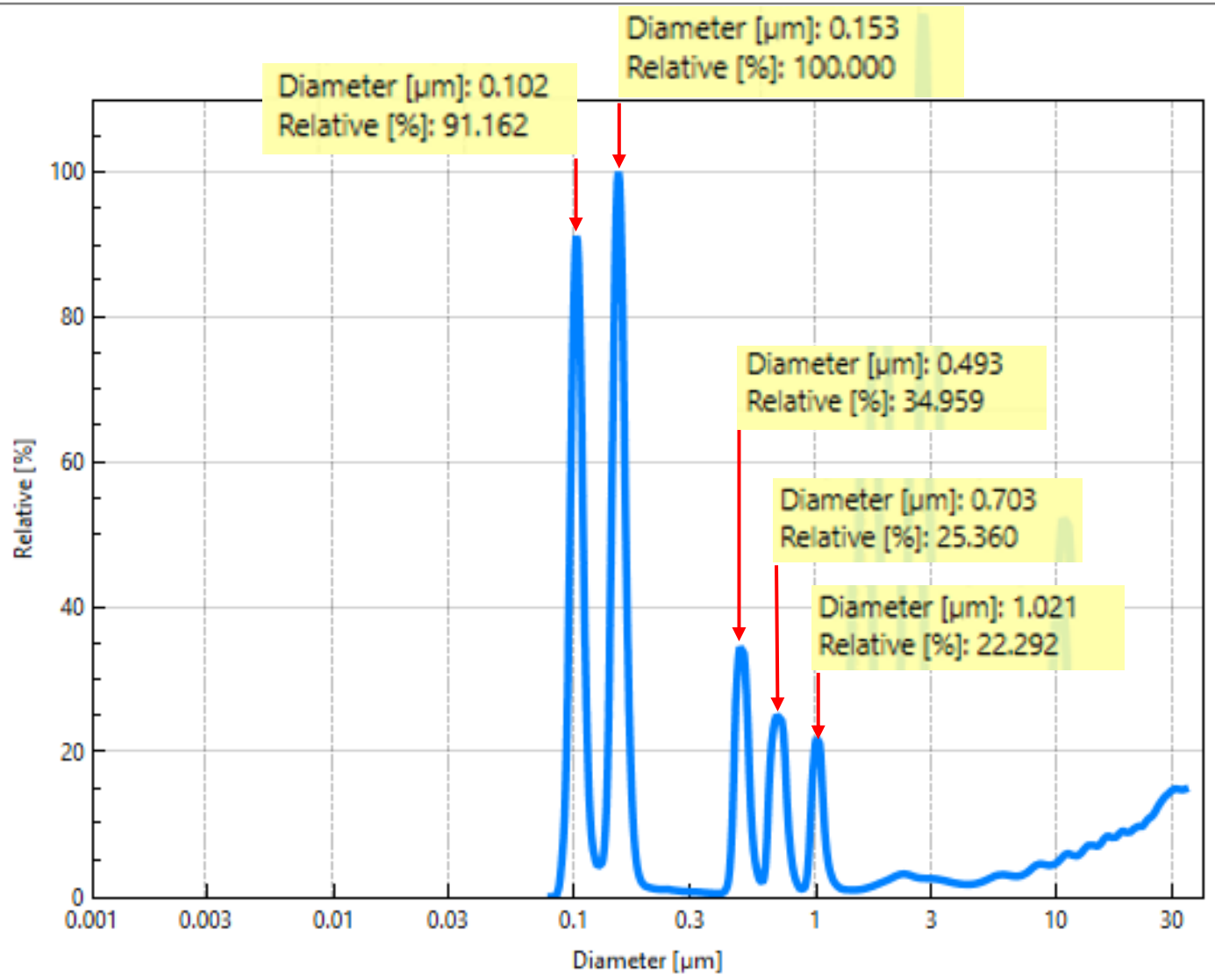
Concentration : 3.5%

Wide size range analysis of carbon nanotubes



Method：Line-start method

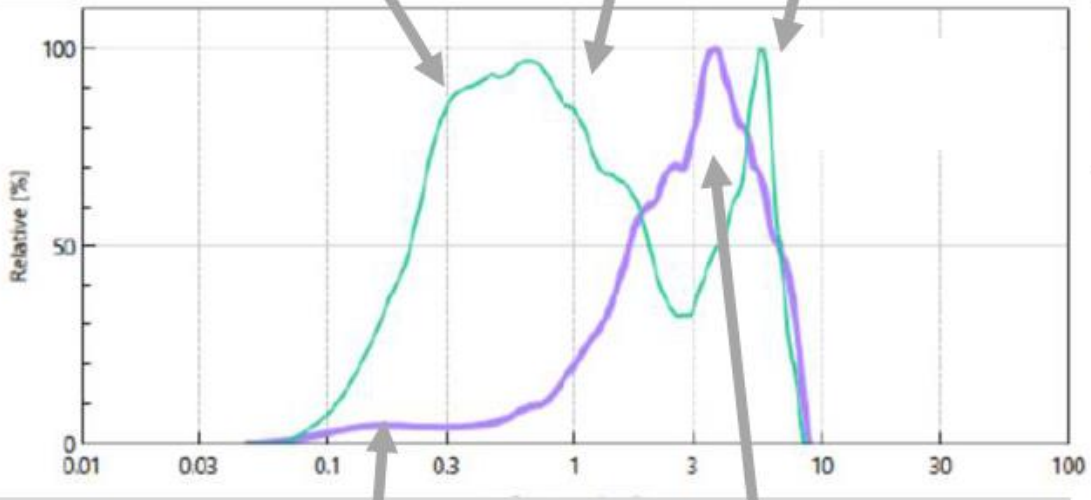
Mixture of 5 polystyrene latex standards



- **100nm +/- 4nm**
- **152nm +/- 5nm**
- **496nm +/- 8nm**
- **702nm +/- 6nm**
- **1.036um +/- .012um**

Measuring mixed high concentration battery materials

after mixing, before kneading

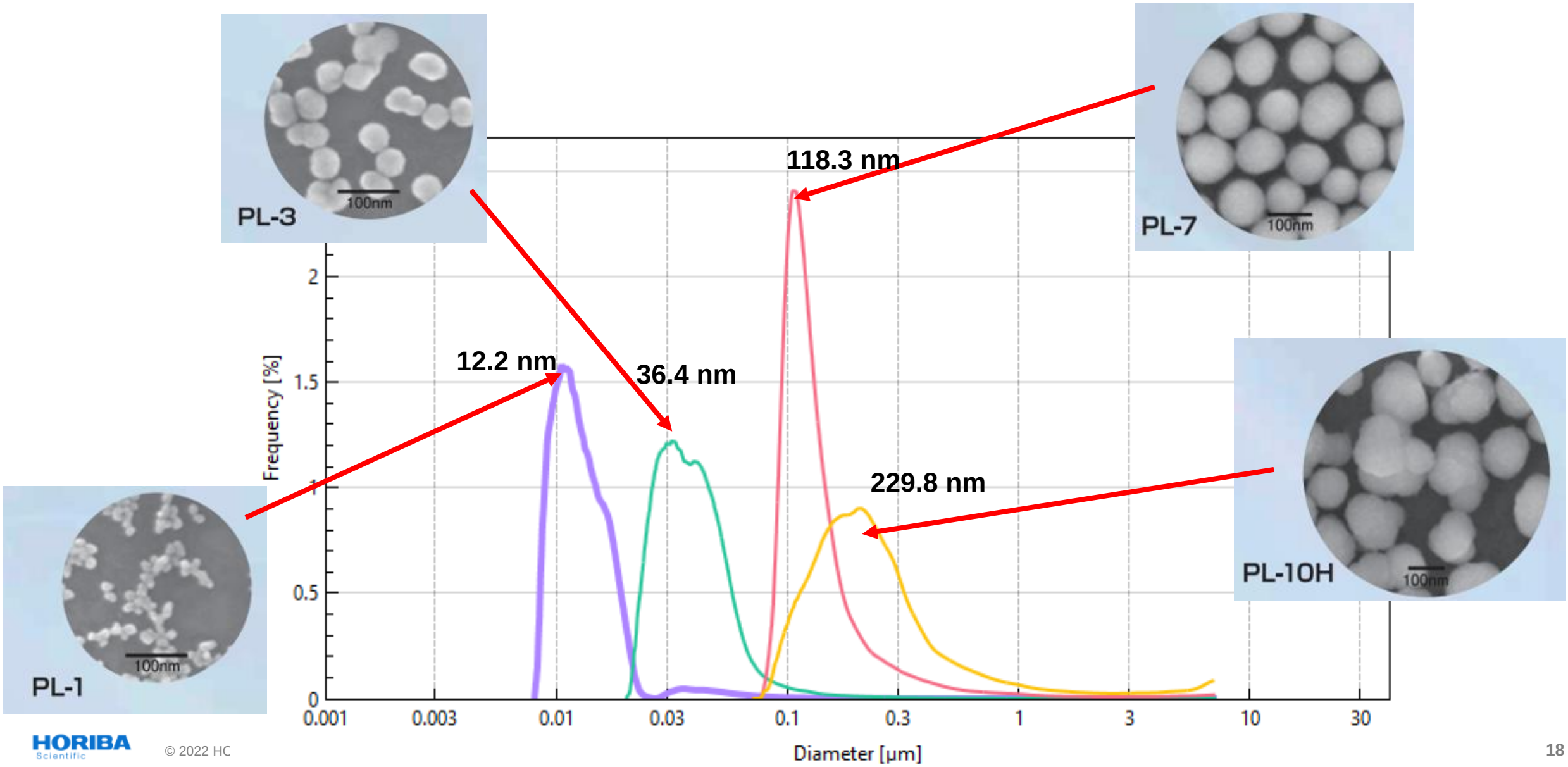


after mixing, after kneading

— after mixing, before kneading
— after mixing, after kneading

● Carbon ~ 100 nm
● NCM ~ a few um

Fuso ultra high purity colloidal silica



CN-300 Fuso Summary

CN-300 Results	Unit	PL-1	PL-3	PL-7	PL-10H
Median Diameter	nm	12.2	36.4	118.3	229.8

項 目 Items		単 位 Unit	PL-1	PL-3	PL-7	PL-10H
一般物性 General properties	外 観 Appearance	—	Slightly opaque	Slightly opaque	Milky	Milky
	比 重 (20/4℃) Specific gravity	—	1.07	1.12	1.14	1.14
	pH	—	7.3	7.3	7.3	7.3
	シ リ カ 濃 度 Silica content	%	12	20	23	23
粒 子 径 Particle size	一 次 粒 子 径 Primary size (D1)	nm	15	35	75	90
	二 次 粒 子 径 Secondary size (D2)	nm	40	70	125	220
	会 合 度 Aggregate ratio	—	2.7	2.0	1.7	2.4

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Joy and Fun