Effortless Emulsion Evaluation for Everyone

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Outline

- What is an emulsion?
- Measuring size->laser diffraction
- Measuring size->dynamic light scattering
- Measuring charge->zeta potential

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What is an emulsion

- "Mixture" of two immiscible liquids such as oil in water.

Also need an emulsifier!

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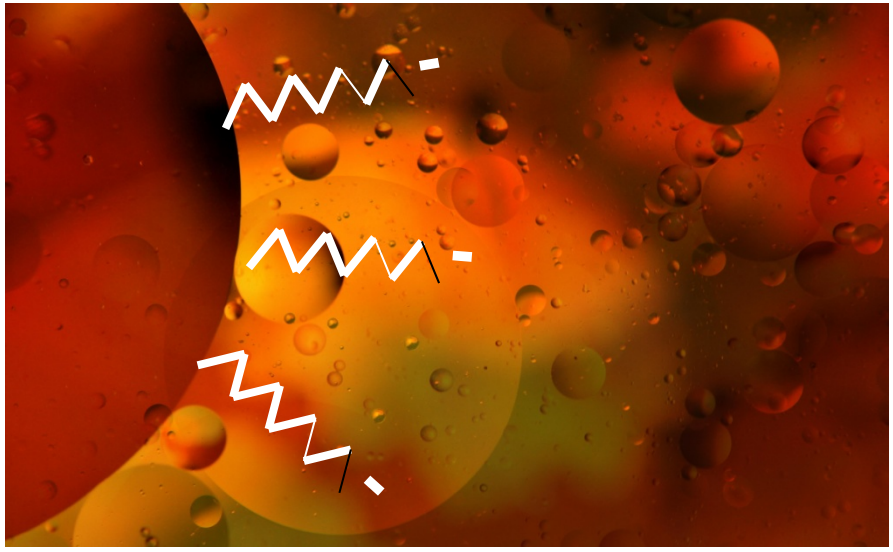
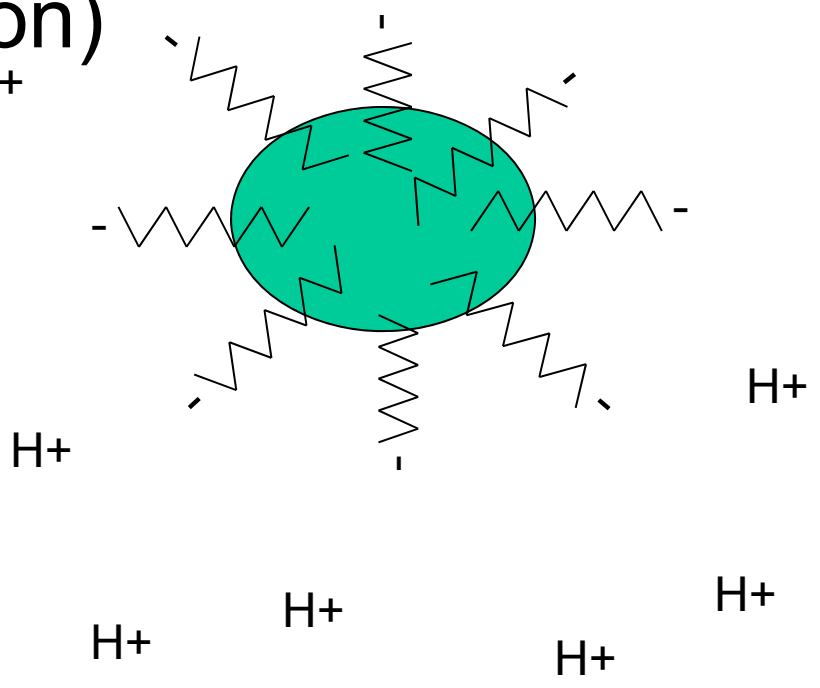
Emulsifier?

- Droplets are not stable due to large surface area.
- Use an emulsifier to reduce surface energy (surface tension)





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Examples of Emulsions

- Food
  - Salad dressings
  - Mayonnaise (oil in water, high particle conc.)
  - Butter (water in oil)
  - Flavoring

- Pharma/Personal Care
  - Lotions/Creams
  - Drug delivery

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Industrial Emulsions

- Drilling Fluid
  - MI SWACO Schlumberger has a reversible oil-water emulsion!

https://www.slb.com/~/media/Files/resources/oilfield_review/ors04/aut04/06_reversible_drilling.pdf

- Cutting Fluid
  - Water for heat removal/environmental friendliness
  - Oil for lubrication

- Waste
  - Waste that includes oil droplets that are emulsified cannot be treated by simple flotation. The emulsion should be broken.

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Emulsion polymers

- You often hear of emulsion polymers. When prepared, they are suspensions of polymer particles in a liquid.
- One step in their preparation is the preparation of an oil (monomer) in water emulsion.

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What do we care about?

- Droplet Size
  - Affects mouth feel and flavor for food
  - Affects kinetics of chemistry (e.g., flavor or drug release)
  - Affects suspension viscosity
  - Affects suspension appearance
  - Affects suspension stability
- Surface Charge
  - Affects suspension stability

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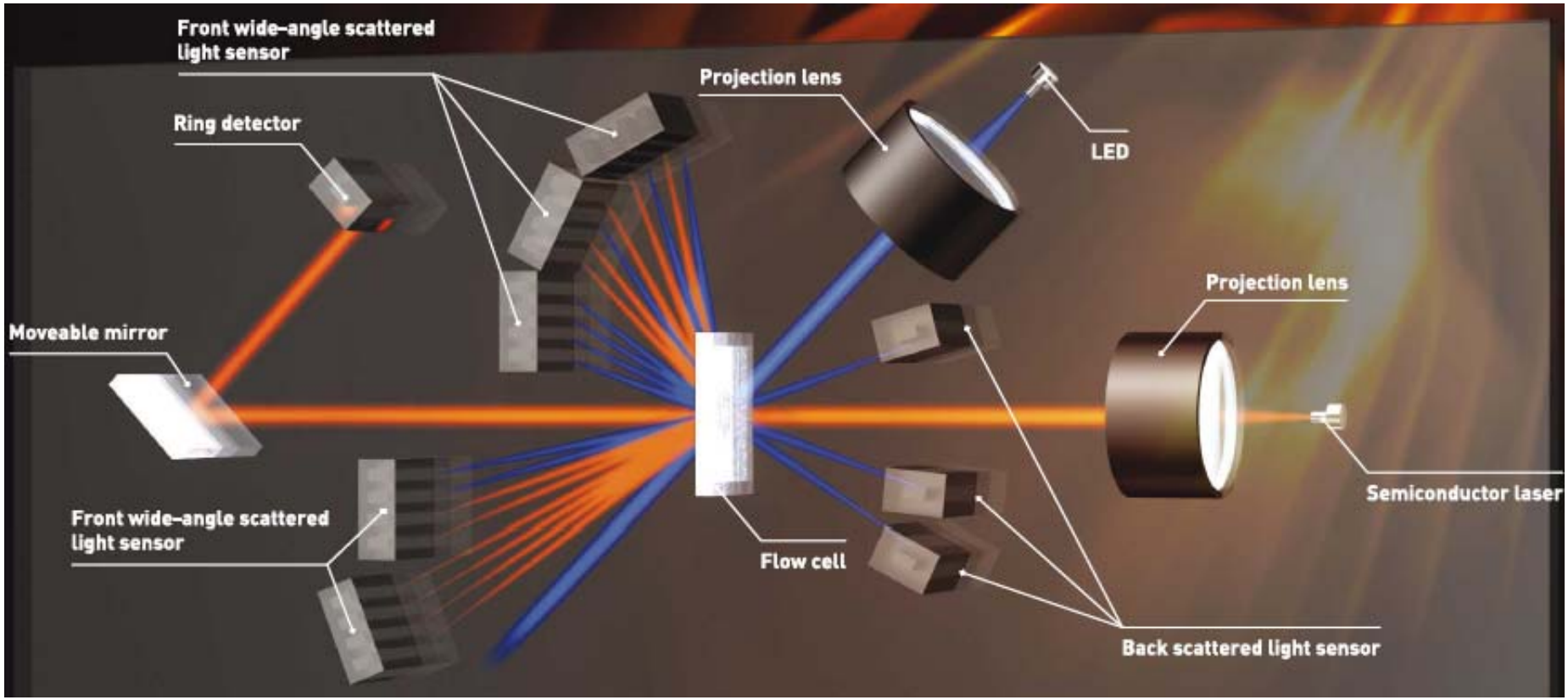
The Measurement: LA-950 Optics



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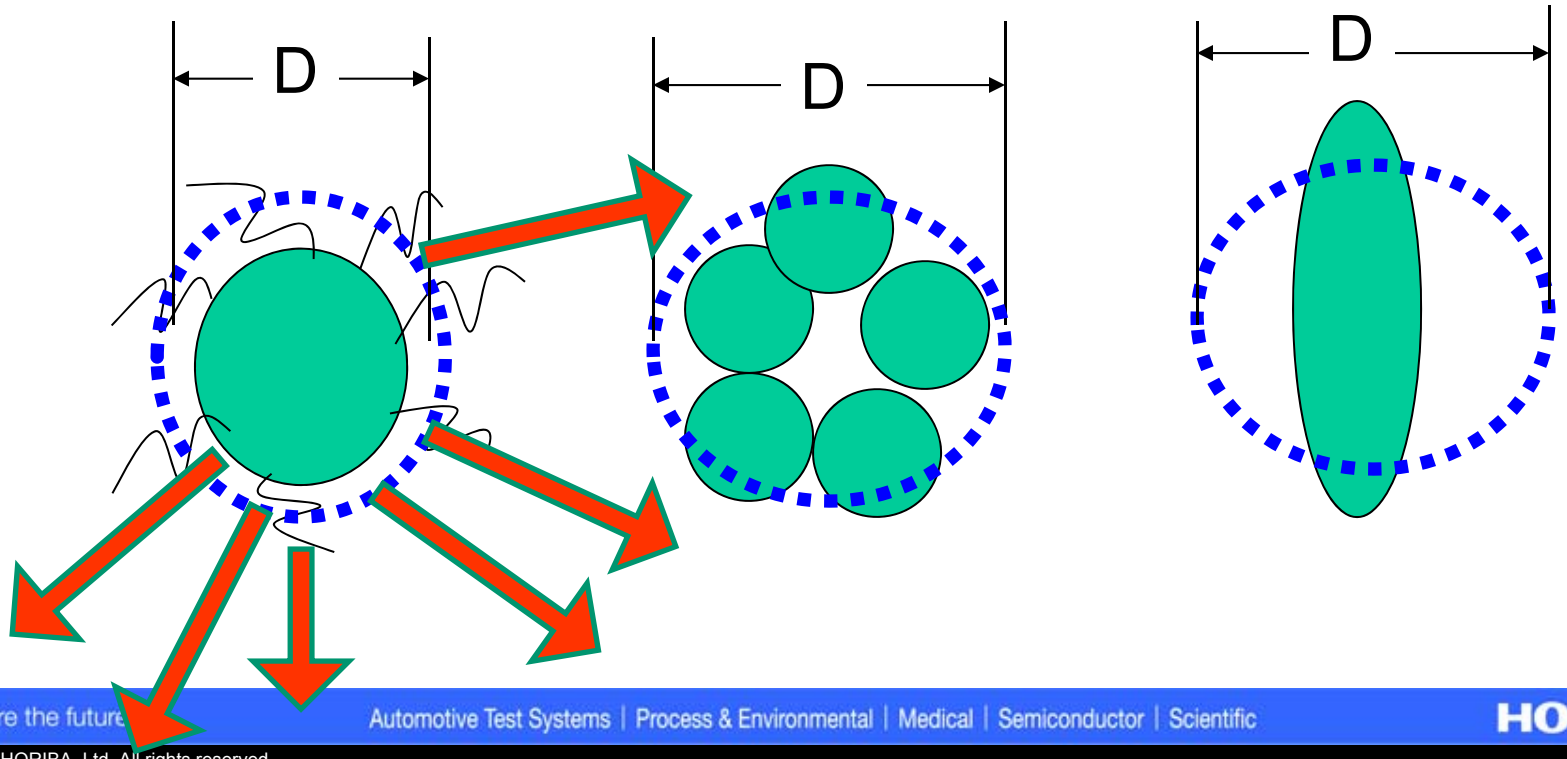
What is Measured?

- Laser diffraction gives the diameter of a sphere that <u>scatters</u> the same way as your sample.



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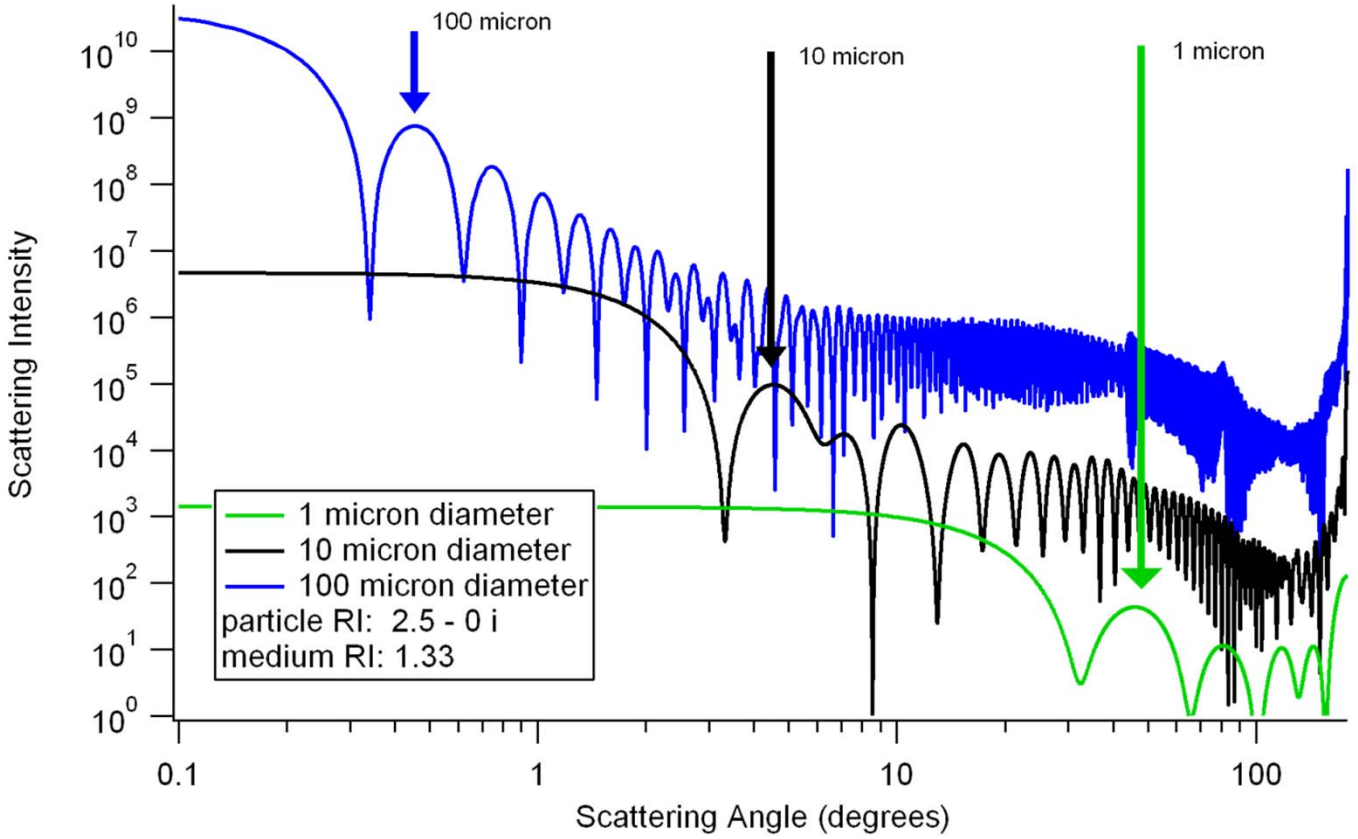
Mie: Effect of Size



As diameter increases, intensity (per particle) increases and location of first peak shifts to smaller angle.

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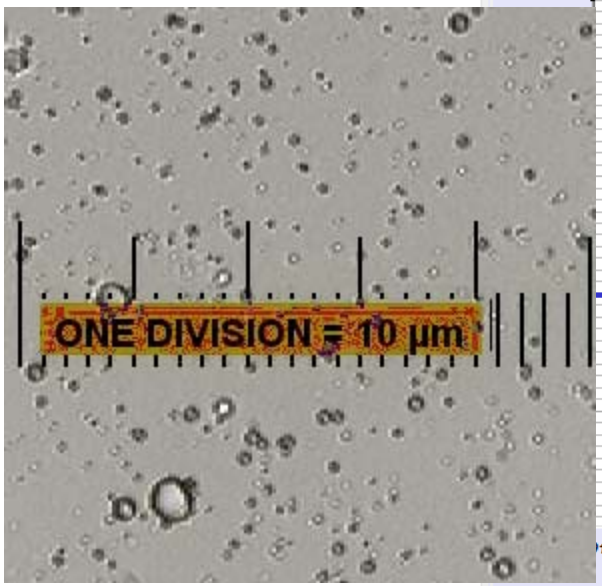
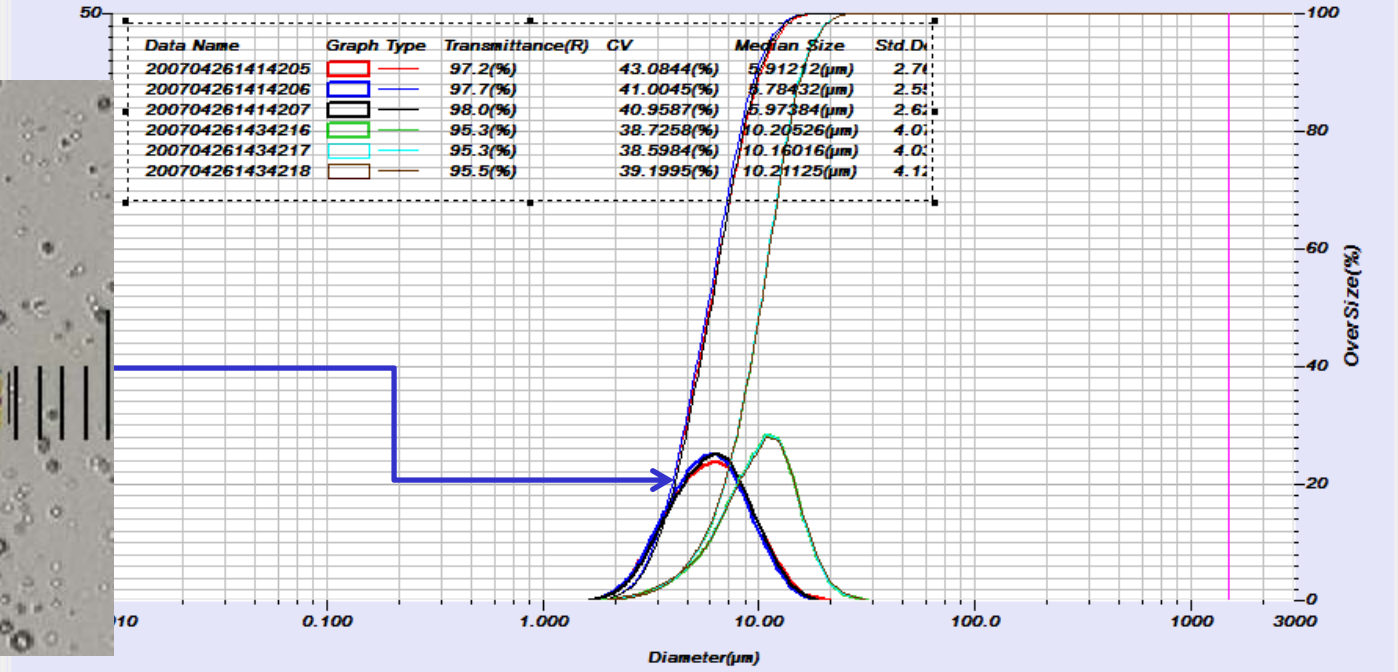
Water in oil emulsion

- Oil industry



Distribution Graph | Data Table | Result Data

S.P.Area	: 11385(cm²/cm³)	Transmittance(R)	: 97.7(%)
Mean Size	: 6.22020(µm)	Transmittance(B)	: 91.9(%)
Variance	: 6.5054(µm²)	Sample Data Acquisition Times (LD)	: 5000
Median Size	: 5.78432(µm)	Sample Data Acquisition Times (LED)	: 5000
Mode Size	: 6.2572(µm)	Refractive Index (R)	: OIL-WATER[OIL-WATER(1.330 - 0.000i),ISOPAR-V(1.480)]
Chi Square	: 16.673883	Refractive Index (B)	: OIL-WATER[OIL-WATER(1.330 - 0.000i),ISOPAR-V(1.480)]
R Parameter	: 1.2143E-1	Iteration Number	: 15
		Measurement Time	: Thursday, April 26, 2007 5:14:25 PM
		Circulation Speed	: 5
		Ultra Sonic	: OFF



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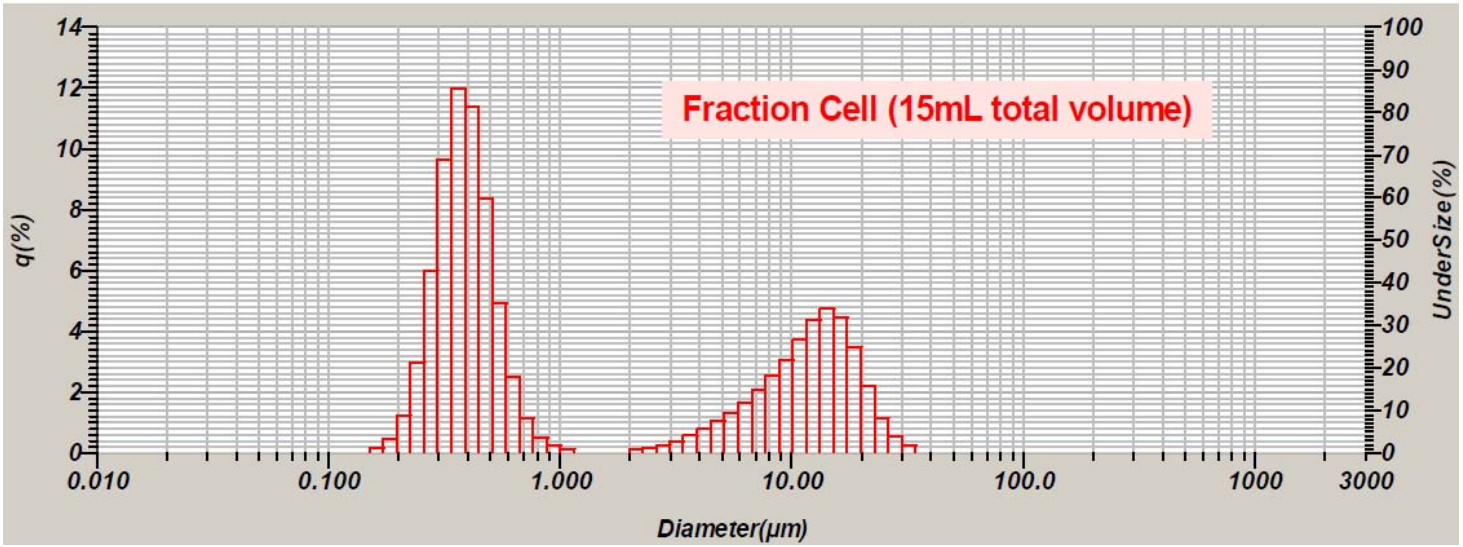
Pharma Emulsion

- Bimodal distribution, another water in oil emulsion.
- Used fraction cell for small sample volume.



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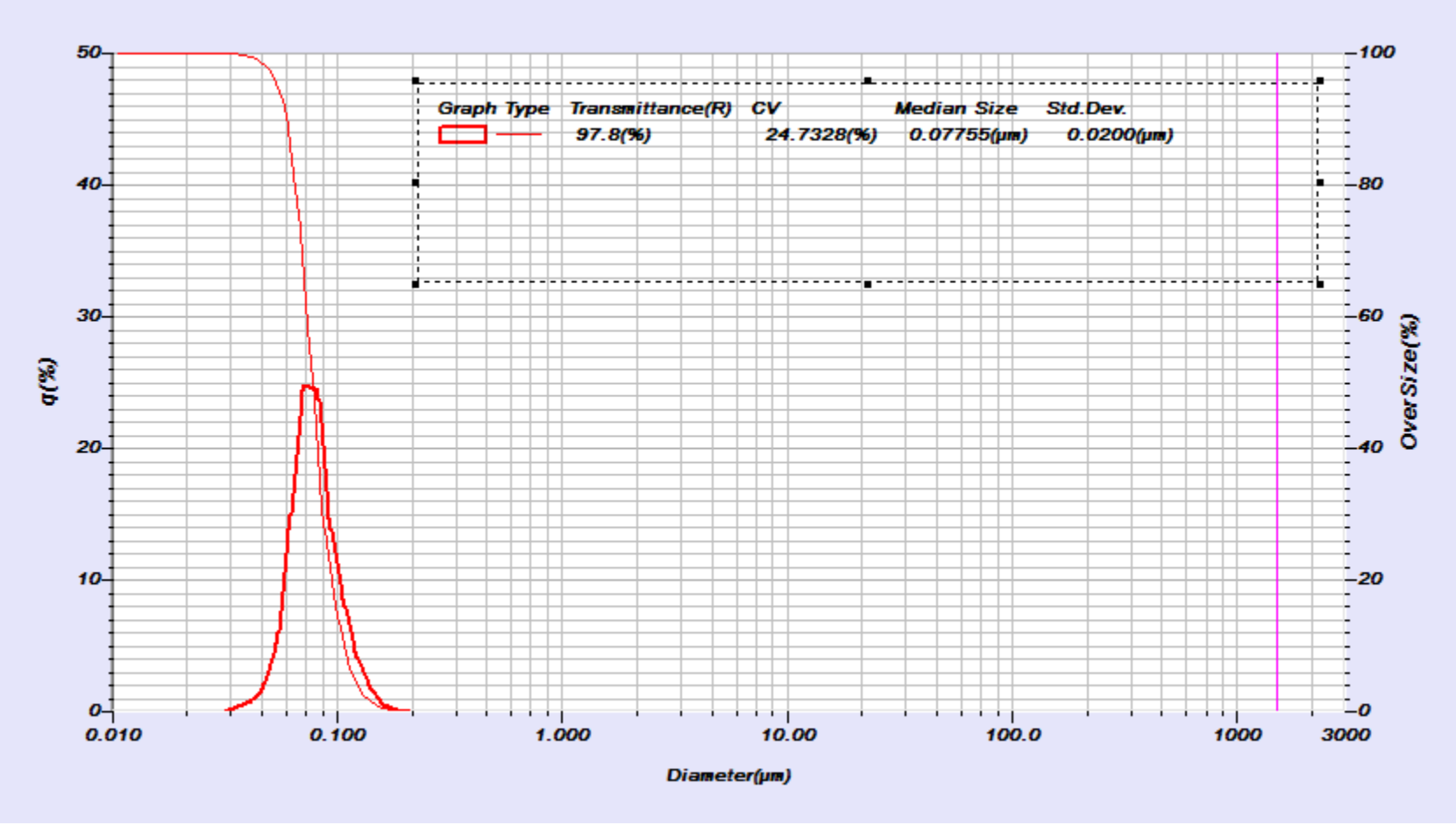
Lotion (oil in water)



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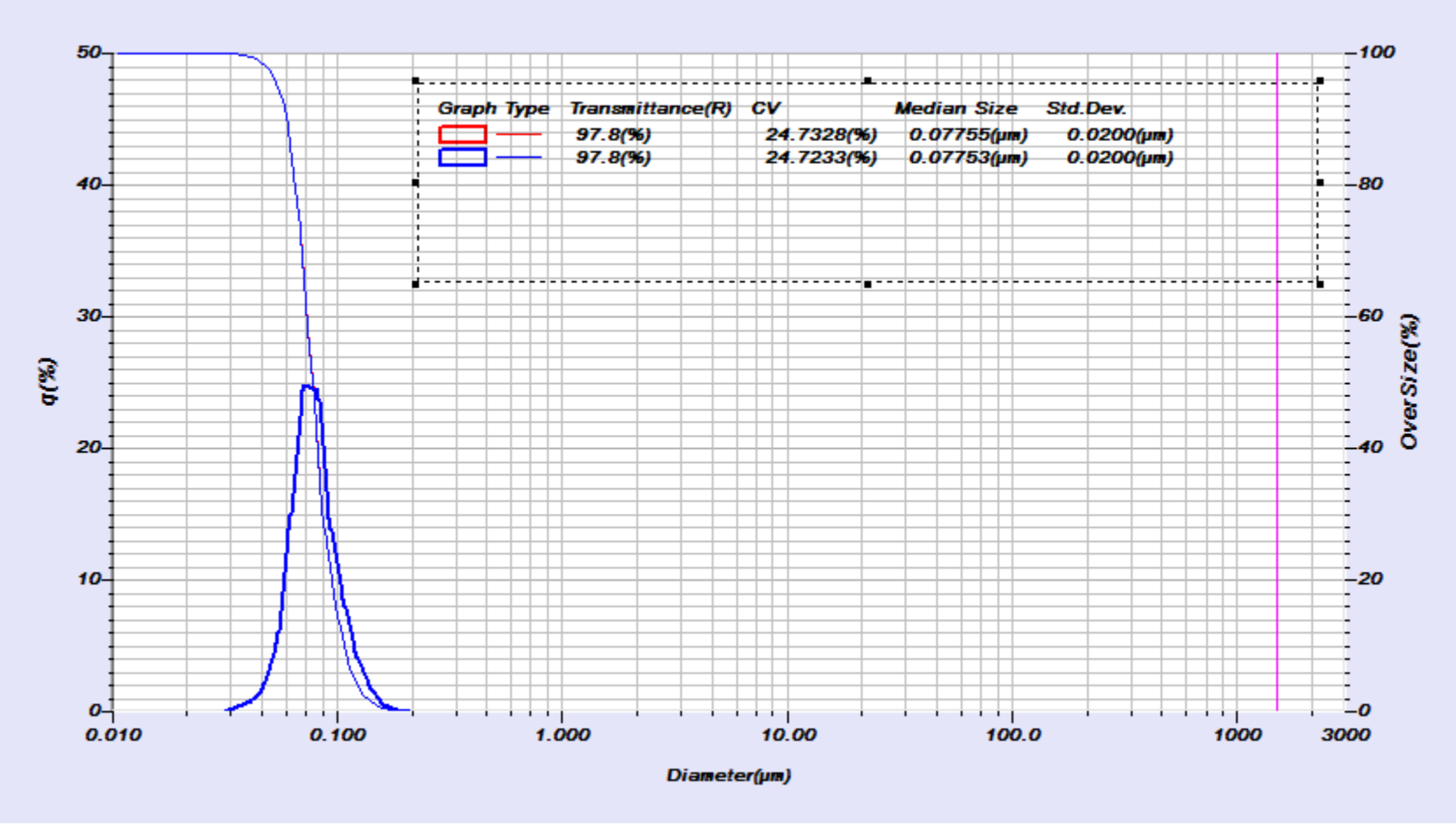
Lotion (oil in water)-overlay



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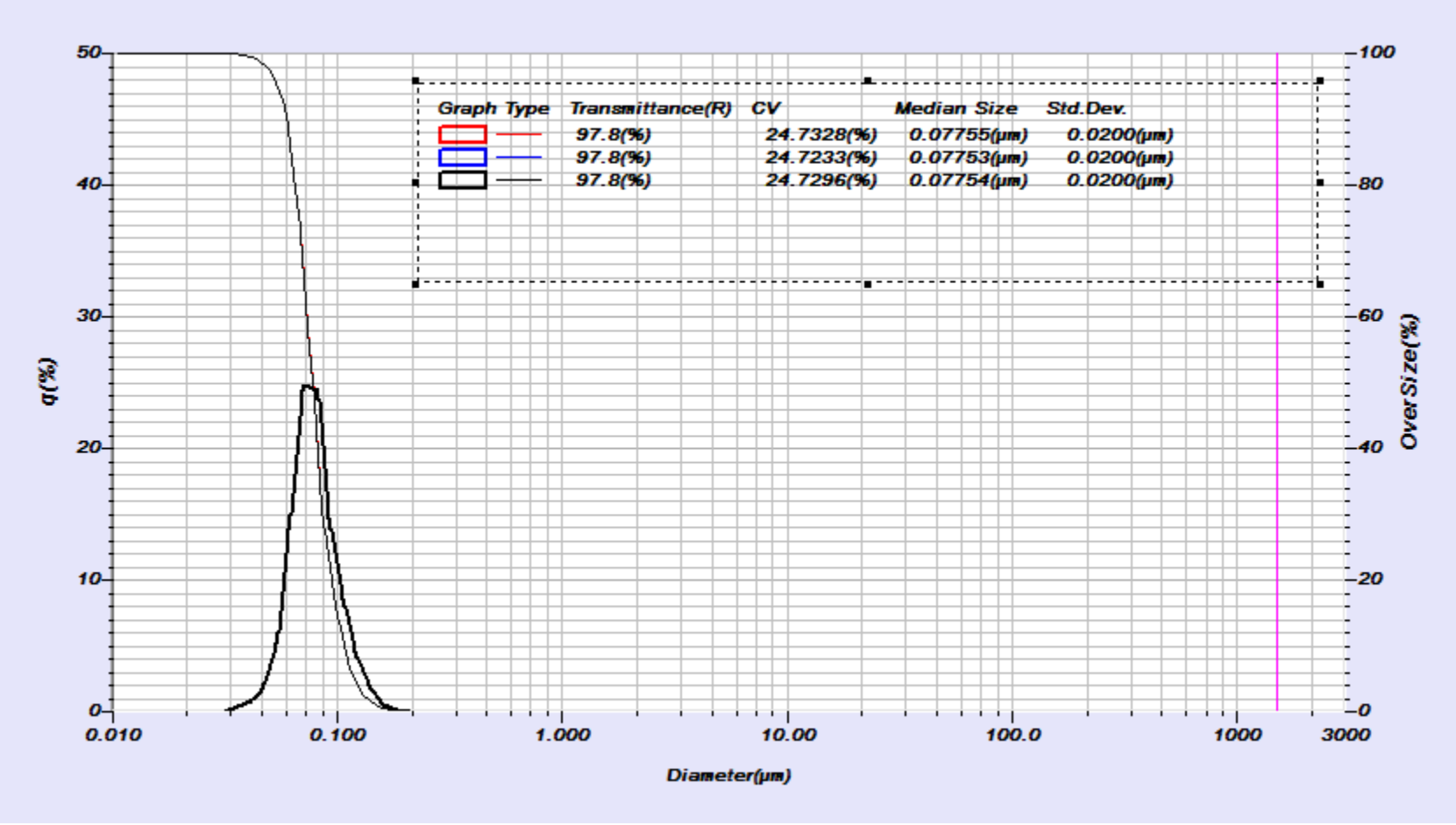
Lotion (oil in water)-3rd repeat



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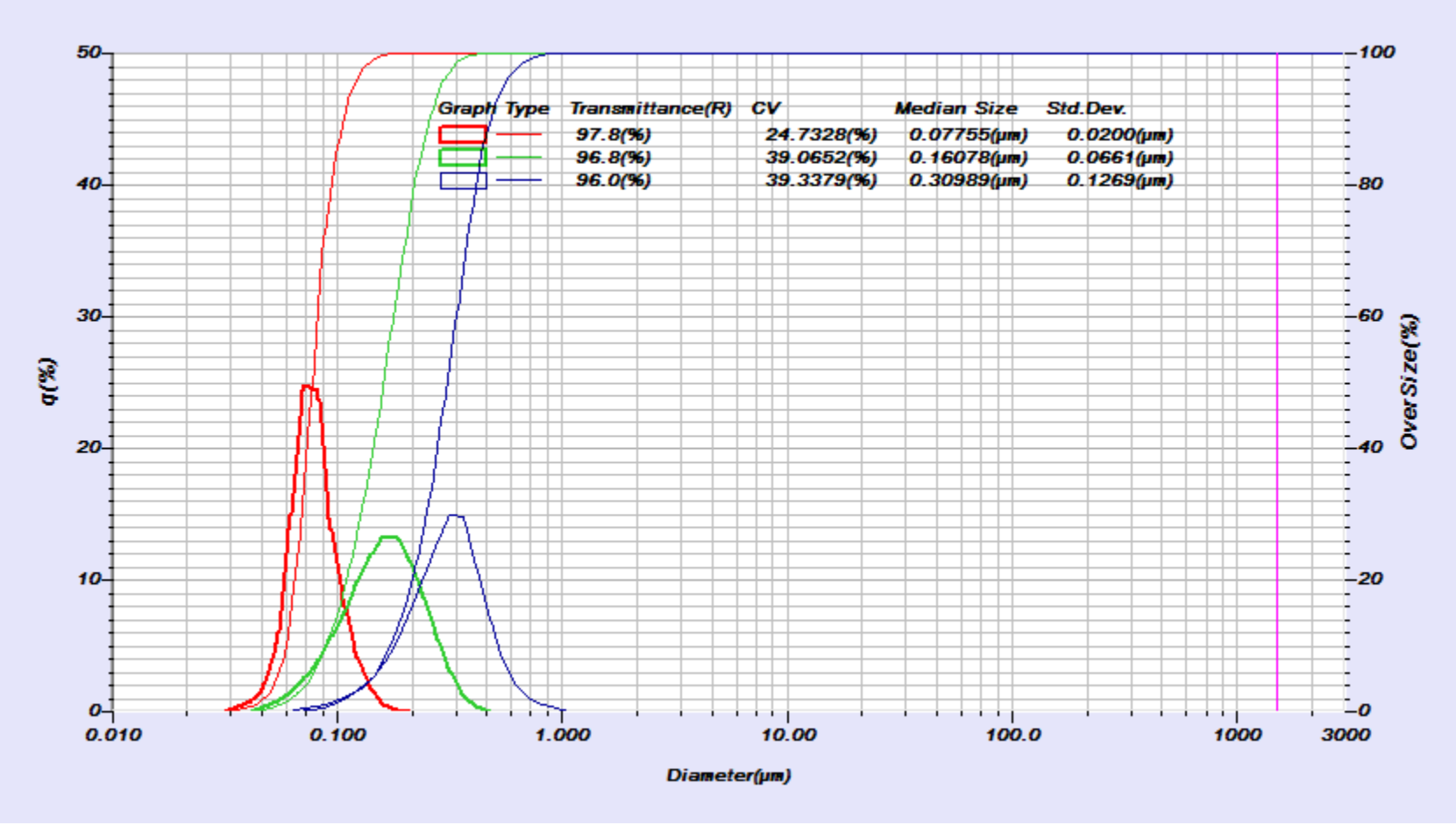
A variety of formulas



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Pop Quiz

Laser Diffraction can be used to measure:

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Pop Quiz

Laser Diffraction can be used to measure:

Any emulsion, but check chemical compatibility.

Instruments with chemically resistant parts are easy, but somewhat more expensive to build. Check this point when using (or purchasing) an instrument.

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Outline

- What is an emulsion?
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- Measuring size->dynamic light scattering
- Measuring charge->zeta potential

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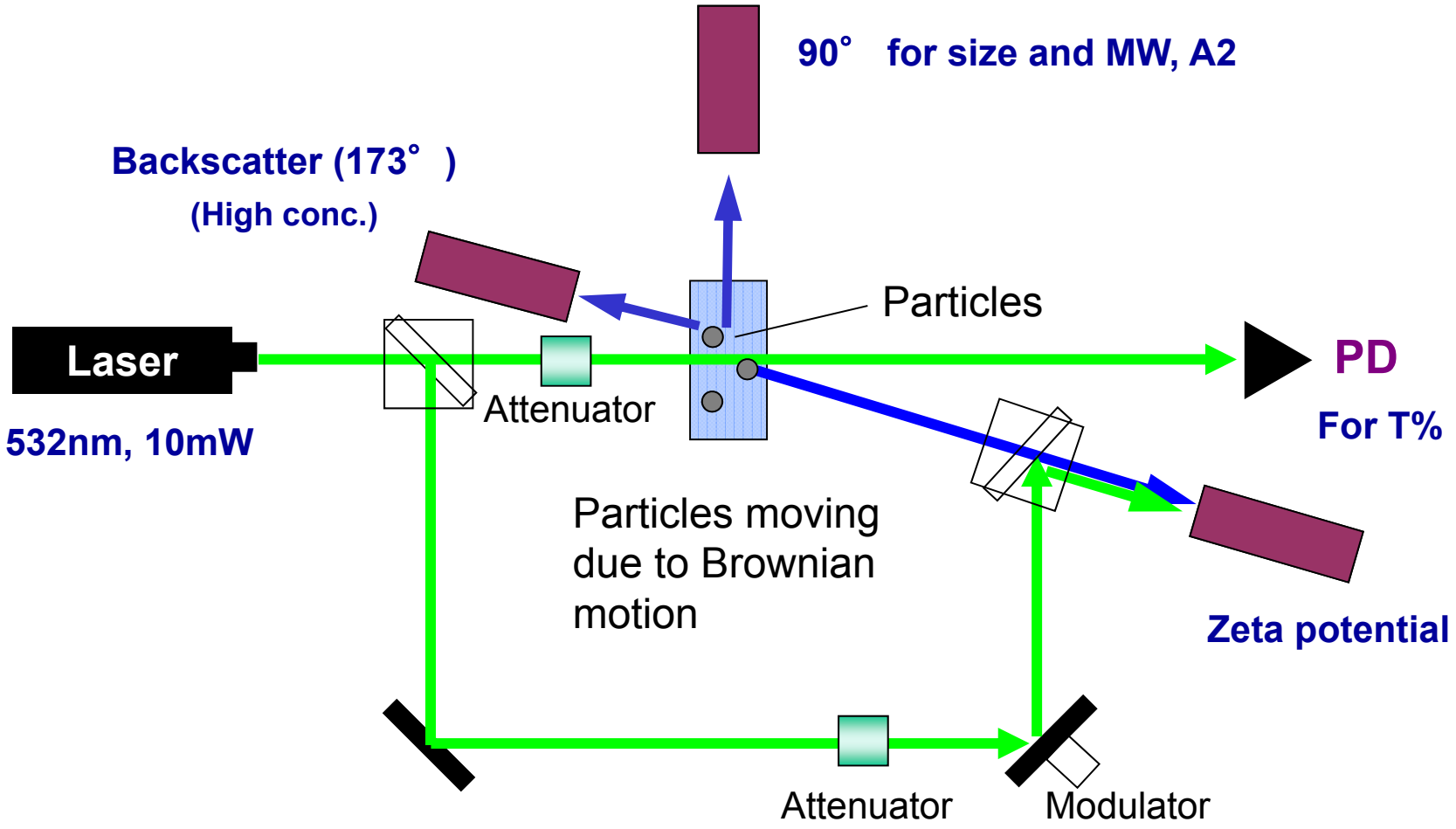
DLS Optics



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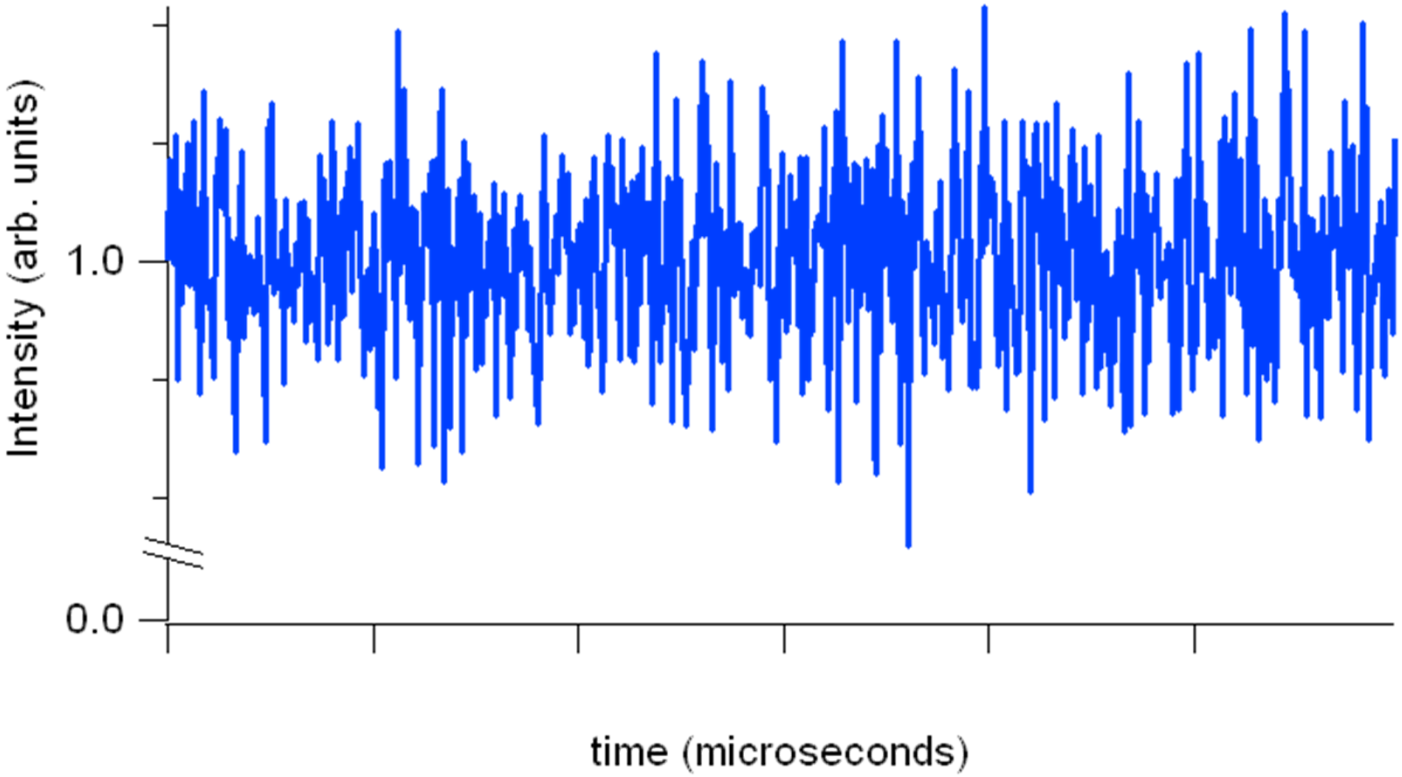
DLS signal

- Random motion of particles leads to random fluctuations in signal (due to changing constructive/destructive interference of scattered light.



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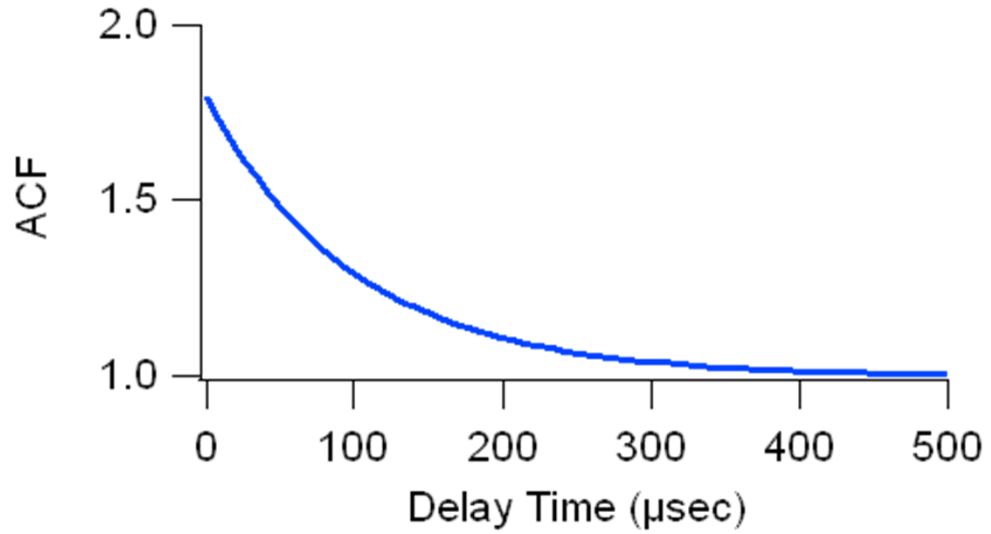
Correlation Function

- Random fluctuations are interpreted in terms of the autocorrelation function (ACF).

$$C(\tau) = \frac{\int_0^T I(t)I(t+\tau)dt}{\langle I(t)I(t)\rangle}$$



$$C(\tau) = 1 + \beta \exp(-2\Gamma\tau)$$

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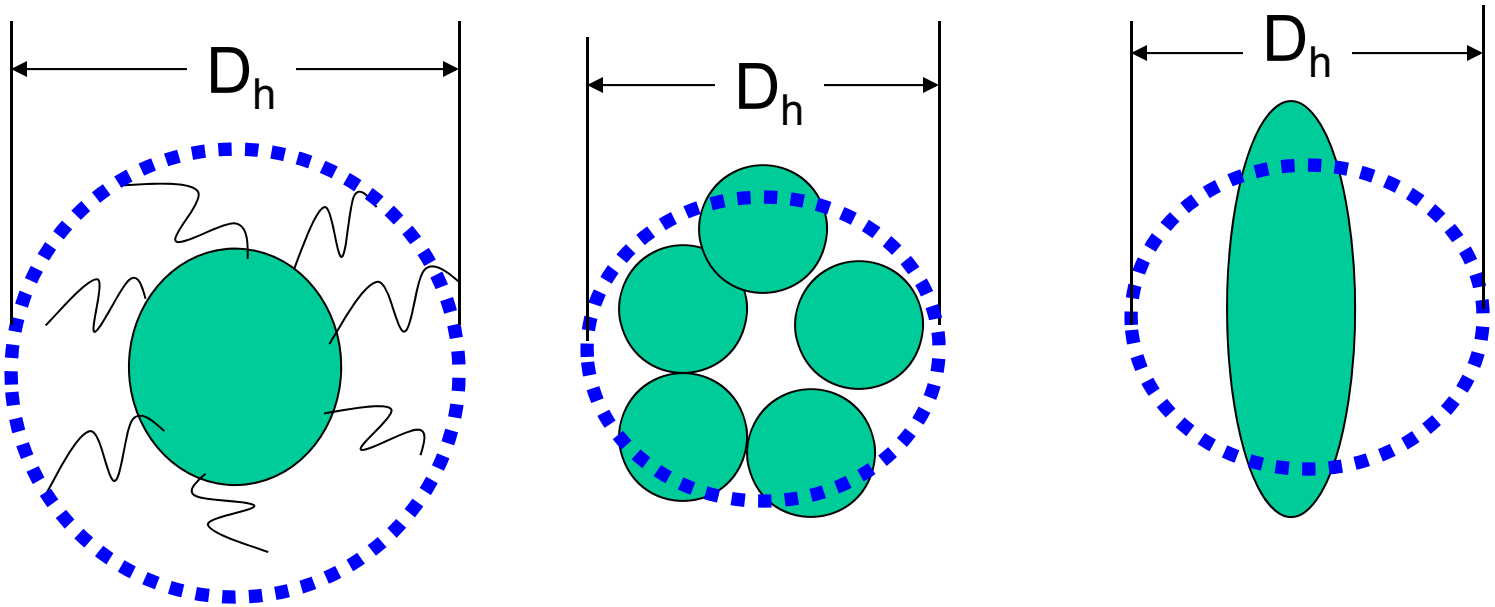
What is Measured?

- DLS gives the diameter of a sphere that moves (diffuses) the same way as your sample.



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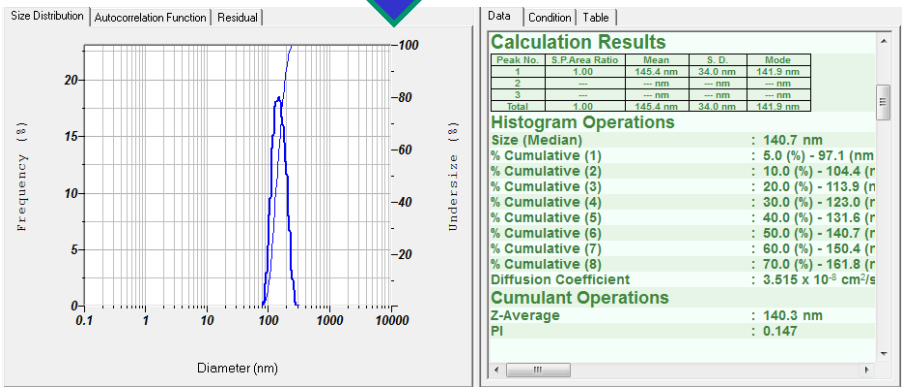
Making the Measurement

1. Fill sample cell.
2. Insert cell into instrument.
3. Click start.
4. Wait ~2 minutes.
5. Review results.



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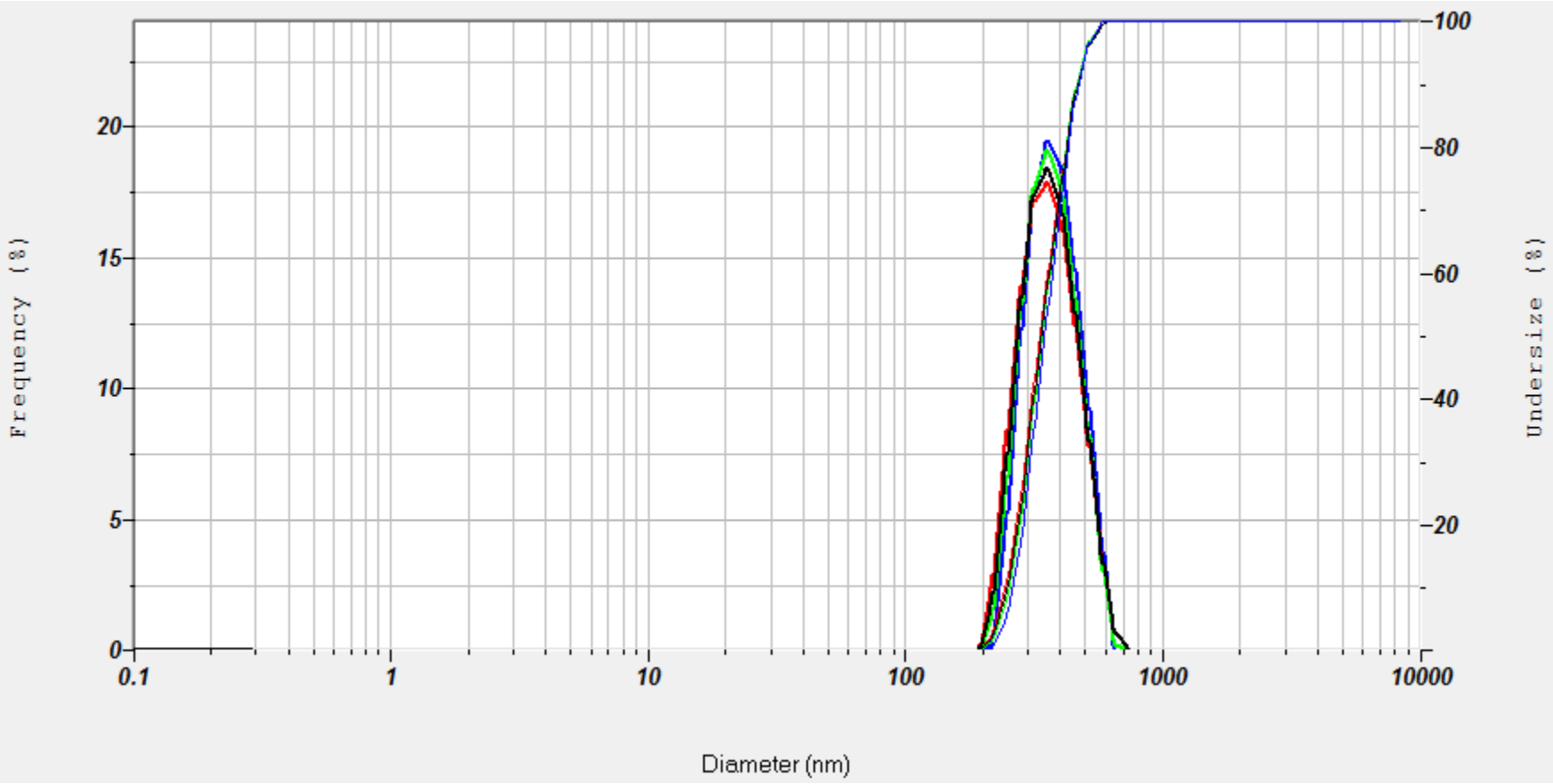
Repeats of flavor emulsion



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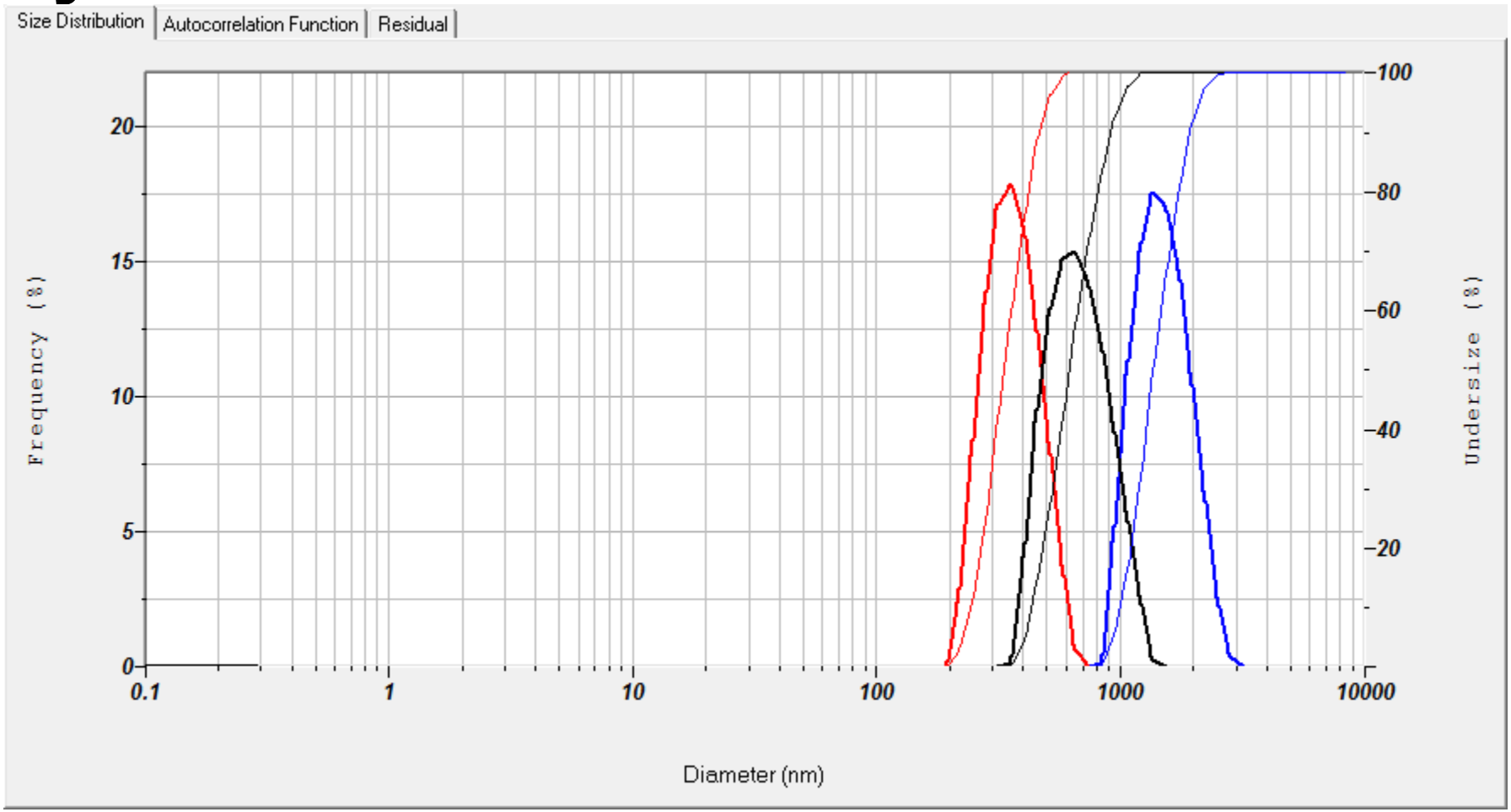
Compare 3 flavor emulsions by DLS



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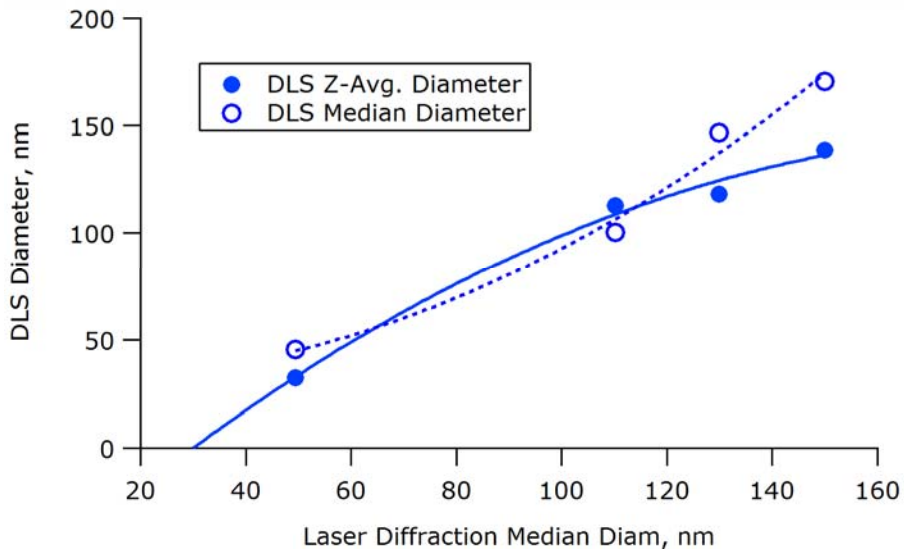
DLS vs Diffraction

- Flavor Emulsions, comparing two techniques



- Measuring slightly different things so results differ.
- Which to use?
  - What other sizes do you want to measure?
  - Stick with Z-average for DLS results
  - Choose one technique and stick with it.

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Outline

- What is an emulsion?
- Measuring size->laser diffraction
- Measuring size->dynamic light scattering
- Measuring charge->zeta potential

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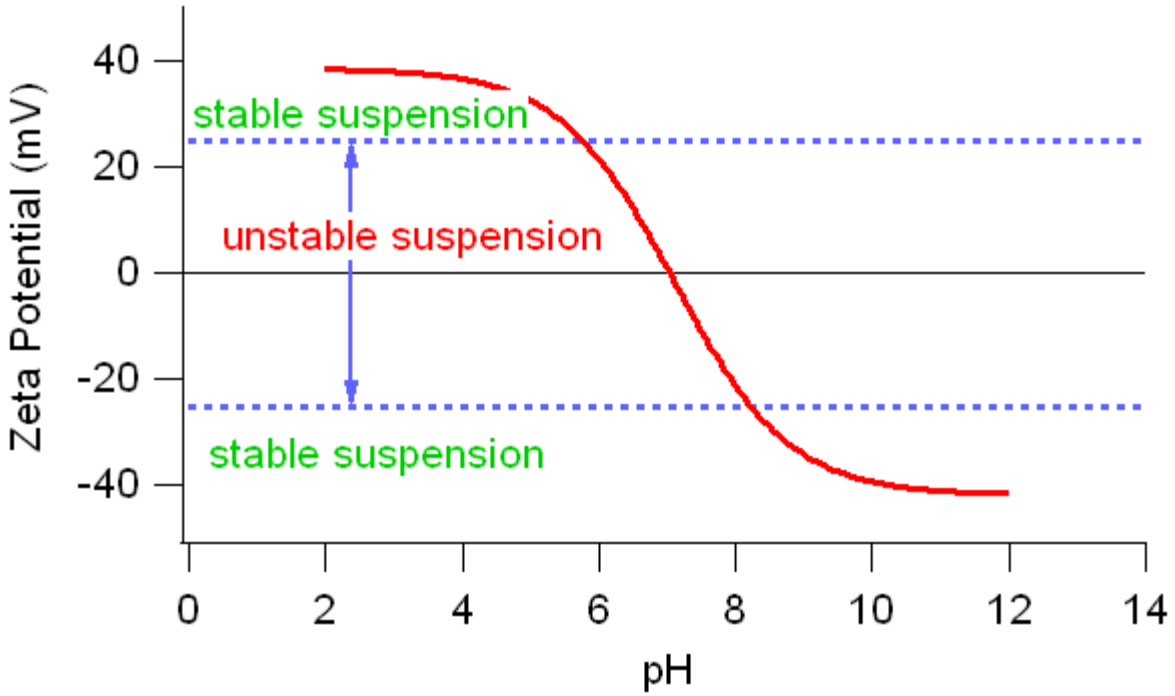
Why Zeta Potential?

- Good way of evaluating electrostatic stabilization of suspensions
- Can use to predict interactions



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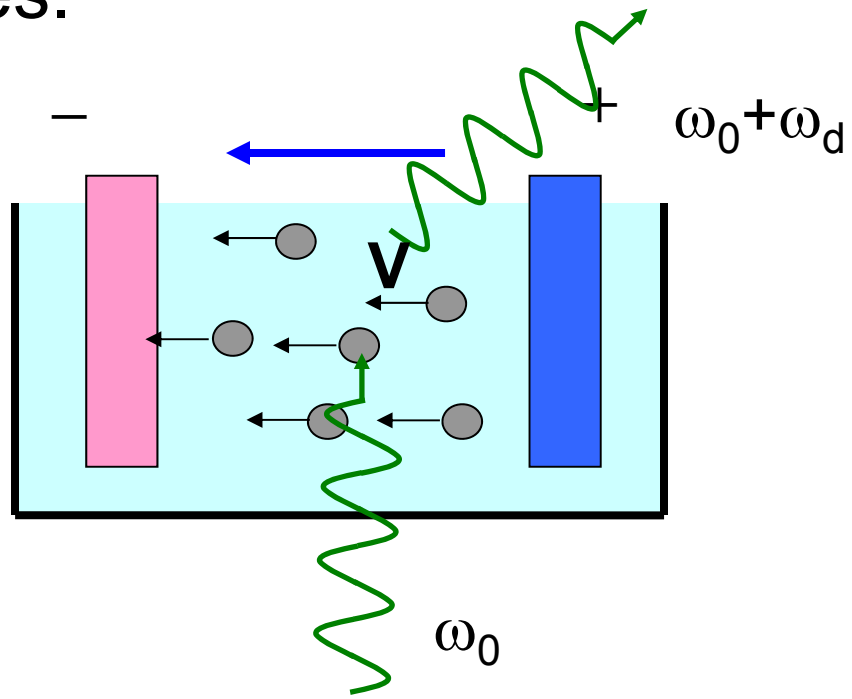
How to determine zeta potential

- Apply an electric field and probe response of particles to applied field.
- You need to see Doppler shift in scattered light due to particle motion with respect to fixed electrodes.



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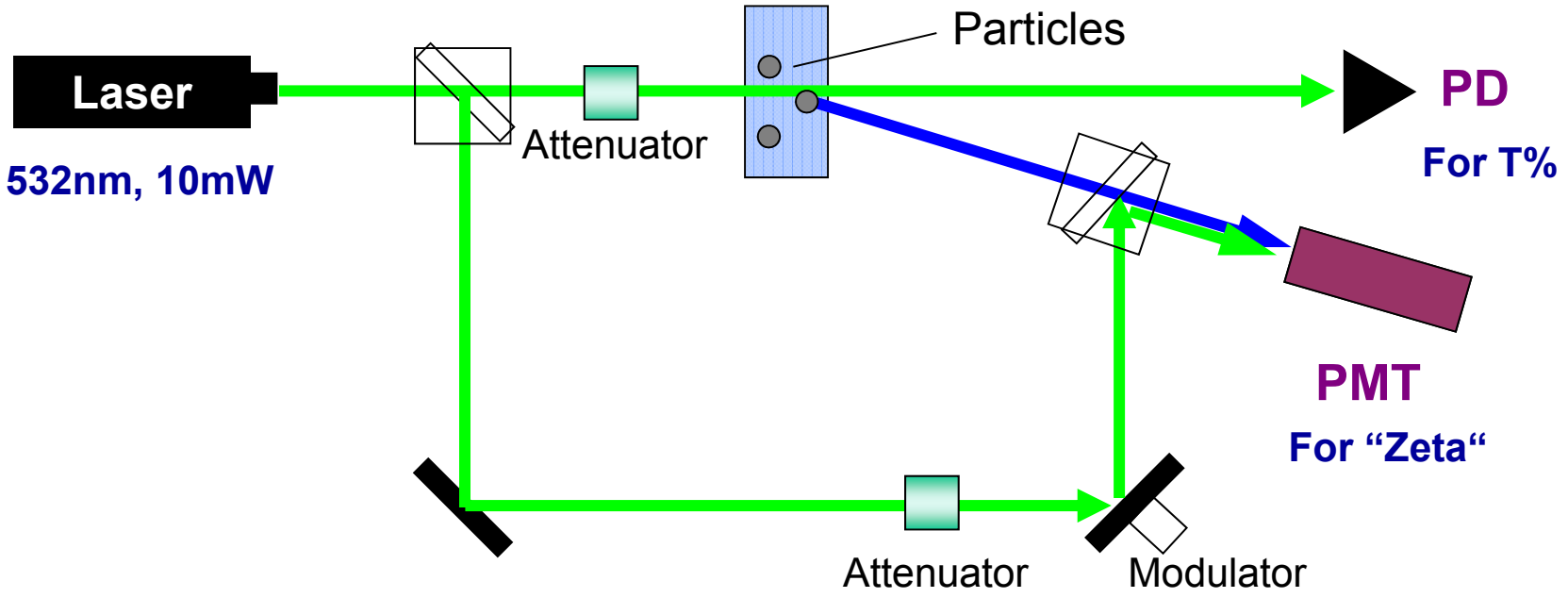
Optical System

Use optical mixing to extract motion of particles relative to electrodes.



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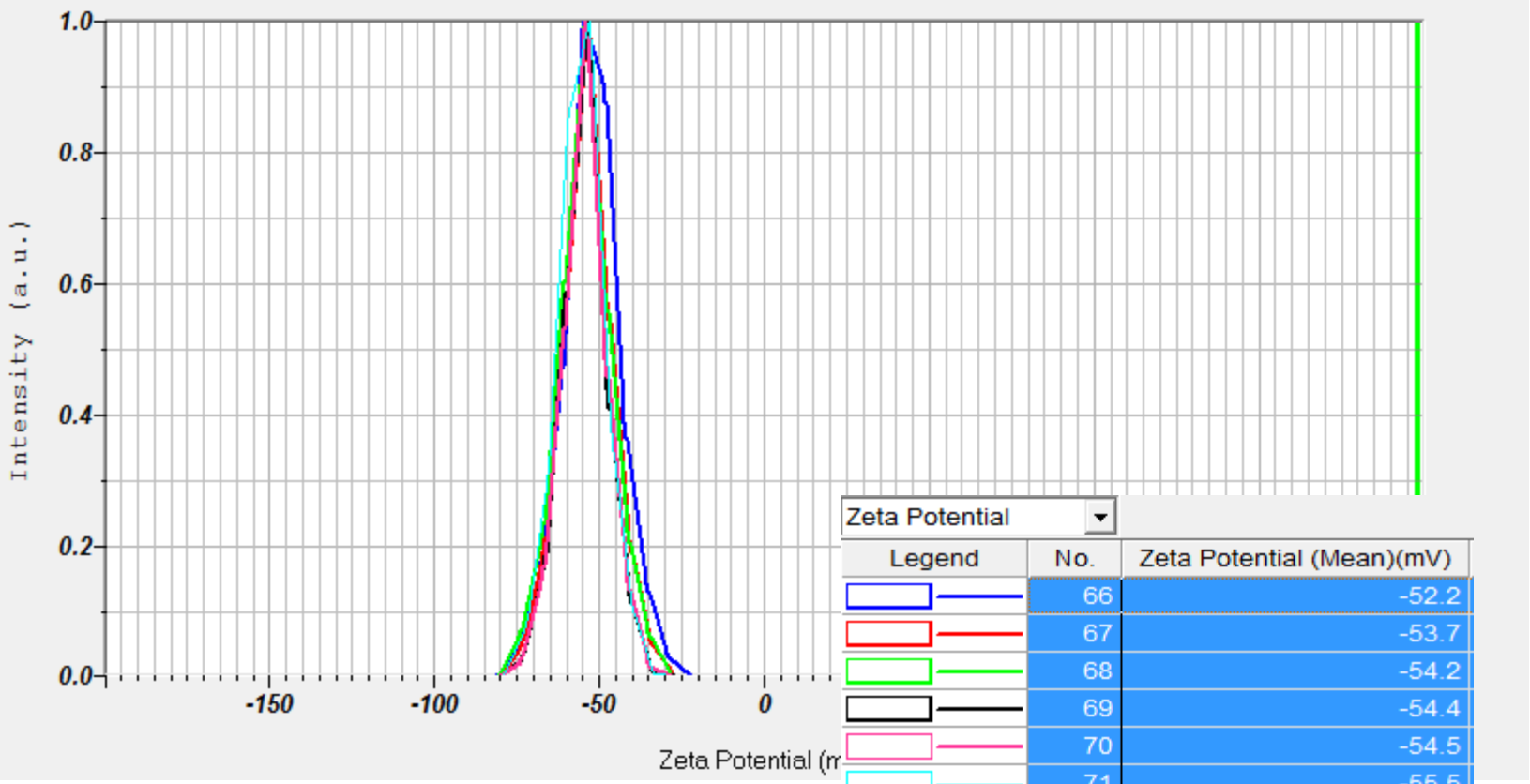
Zeta Potential of Flavor



Zeta Potential

Legend	No.	Zeta Potential (Mean)(mV)
	66	-52.2
	67	-53.7
	68	-54.2
	69	-54.4
	70	-54.5
	71	-55.5
Average		-54.1
S. D.		1.1

Good repeatability!

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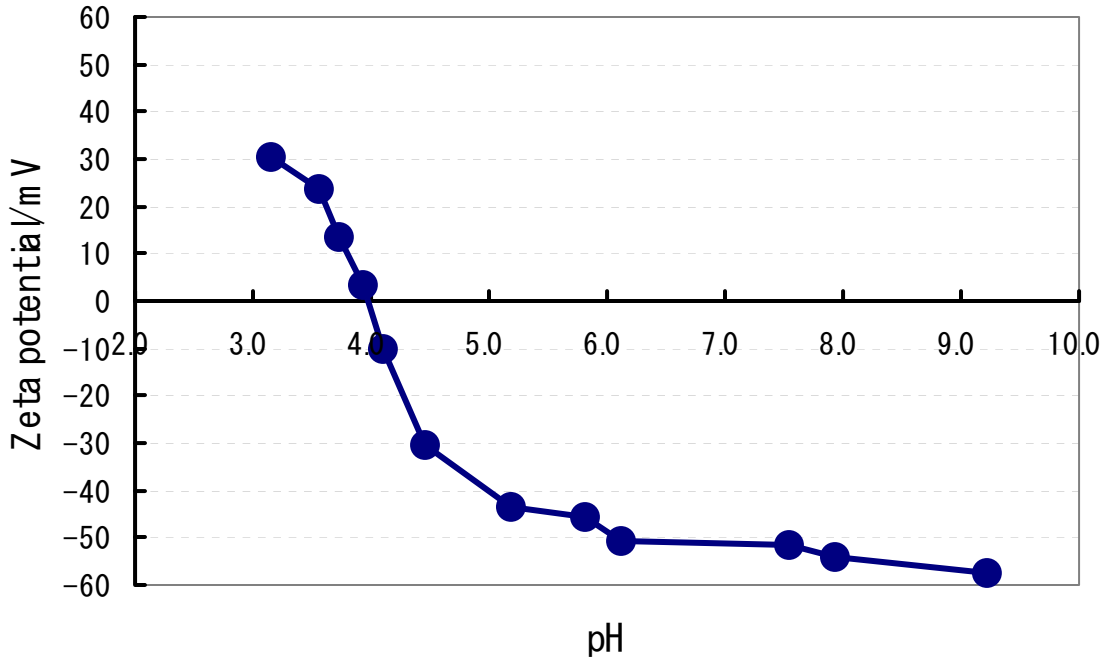
Effect of Liquid

●Iso Electric Point of Coffee Mate

	Results
Iso-electric point	pH 4.0



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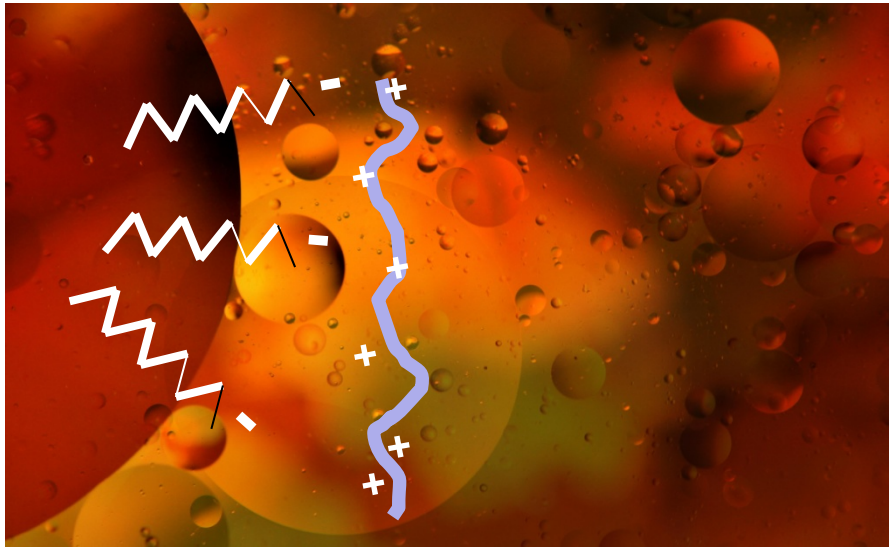
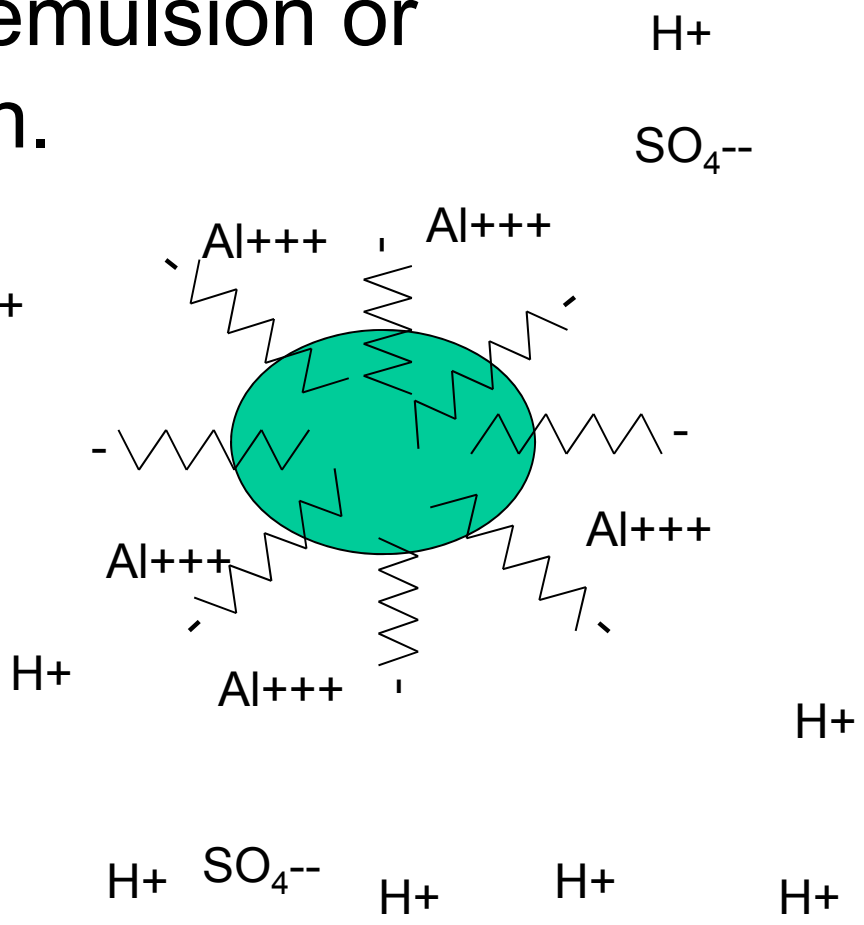
Coagulent

- Use polyions to reduce effect of emulsifier and break emulsion or accelerate flocculation.





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PreTest

- When adding coagulant to flocculate waste: …

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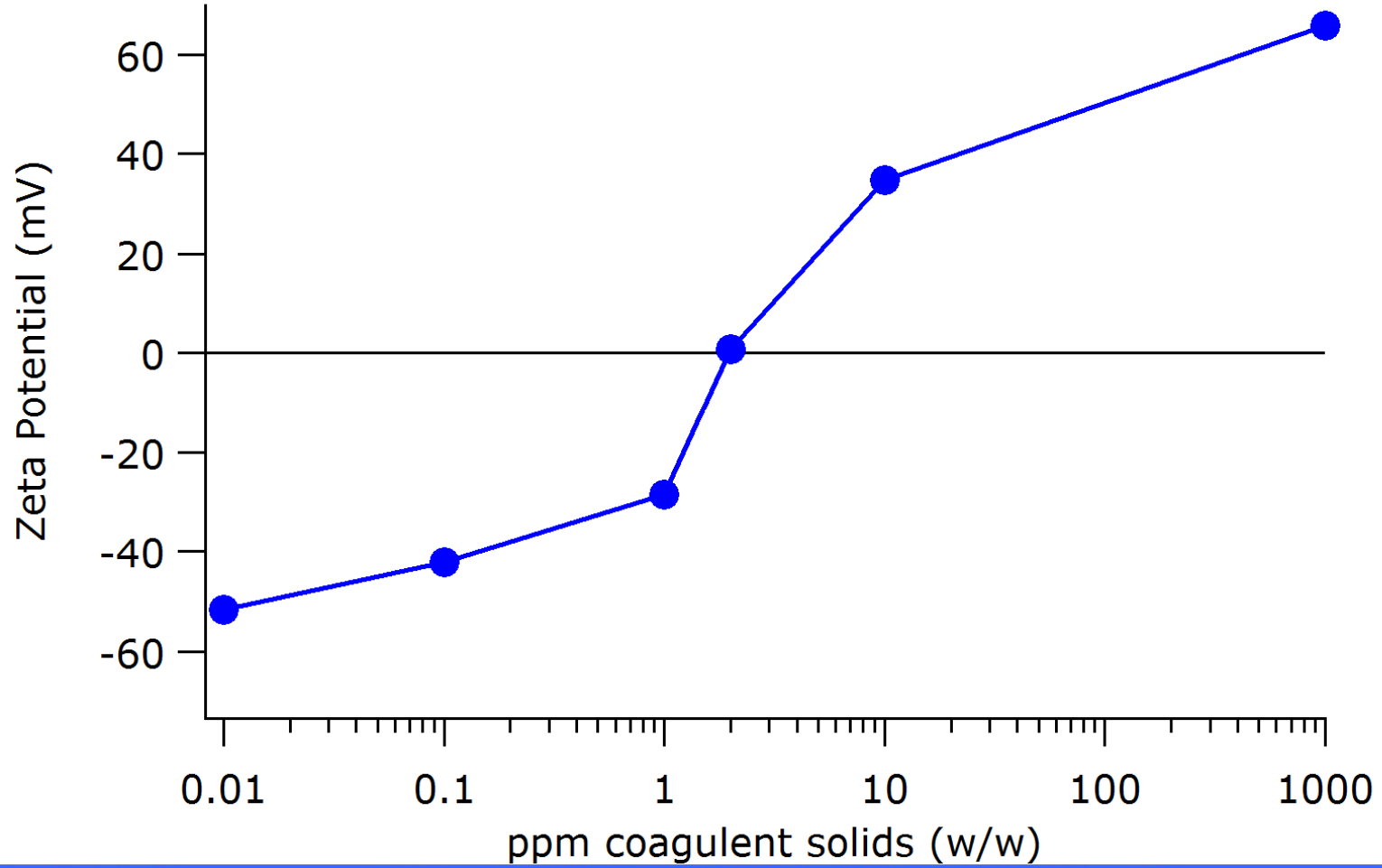
Wastewater

- Add proprietary coagulent to break emulsion



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Dilution

- Sometimes samples need to be diluted. To check if the concentration is correct, do a concentration study.

Dilution DI:sample	Comments	Z-average size, nm (mean of 6 Repeats)
750:1	5 minute runs	670
1000:1	5 minute runs	657
1333:1	5 minute runs	668

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Q&A

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Thank-you

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Thank you

HORIBA Scientific

Thank you

ありがとうございました

Cảm ơn

Dziękuję

ขอบคุณครับ

谢谢

Gracias

Grazie

اشكُرْ

Σας ευχαριστούμε

धन्यवाद

நன்றி

Tacka

Danke

Merci

Obrigado

감사합니다

Большое спасибо

Omoshiro Okashiku

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