A basic introduction to Dynamic Light Scattering (DLS) for particle size analysis

February 1st – 10:30 – 11:30 EST

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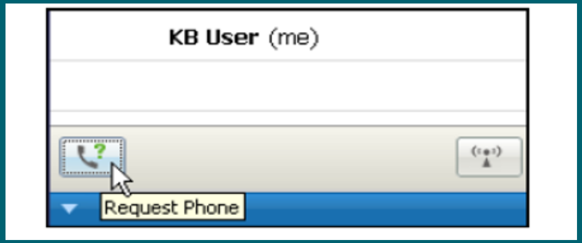

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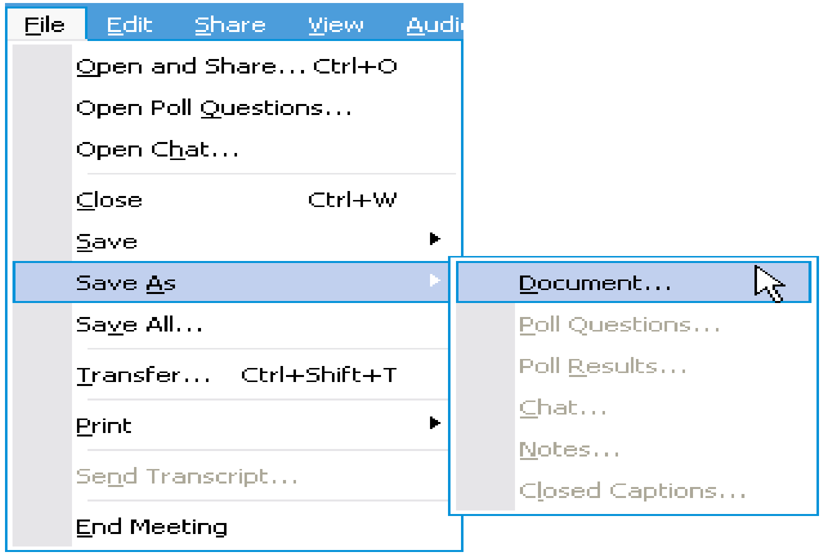

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DYNAMIC LIGHT SCATTERING IN 30 MINUTES

Particle and Molecular Size

Nano

μV

APS

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Agenda

1. Brownian Motion
2. Correlation
3. Analysing the Correlation Function

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1. **Brownian Motion**
2. Correlation
3. Analysing the Correlation Function

Dynamic Light Scattering and Brownian Motion

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- Non-invasive technique for measuring the size of particles and molecules in suspension
- Brownian motion is the **random** movement of particles due to the bombardment by the solvent molecules that surround them
- DLS measures the speed of particles undergoing Brownian motion
  - *Small particles diffuse rapidly*
  - *Large particles diffuse slowly*

Brownian Motion

- Velocity of the Brownian motion is defined by the translational diffusion coefficient (D)
- The translational diffusion coefficient can be converted into a particle size using the **Stokes-Einstein** equation

$$d_H = \frac{kT}{3 \pi \eta D}$$

Where d_H = hydrodynamic diameter, k = Boltzmann’s constant, T = absolute temperature, η = viscosity and D = diffusion coefficient

Hydrodynamic Size

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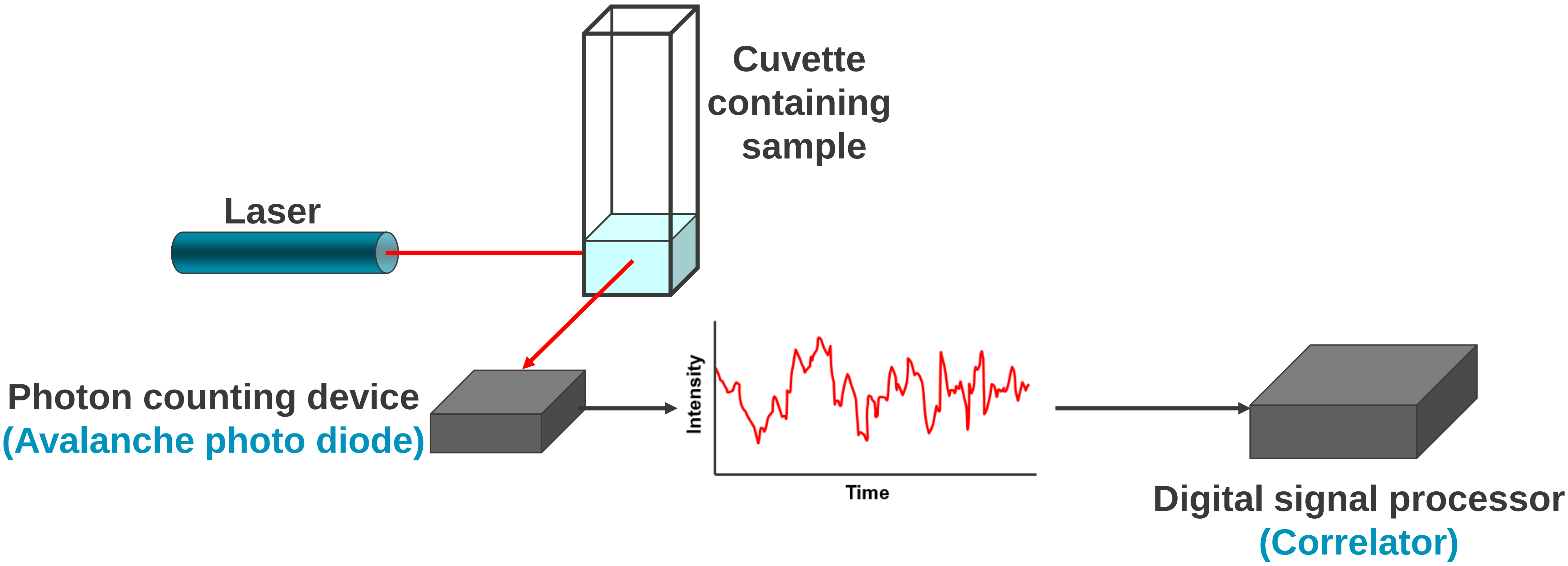
- Definition of Hydrodynamic Diameter (D_H):

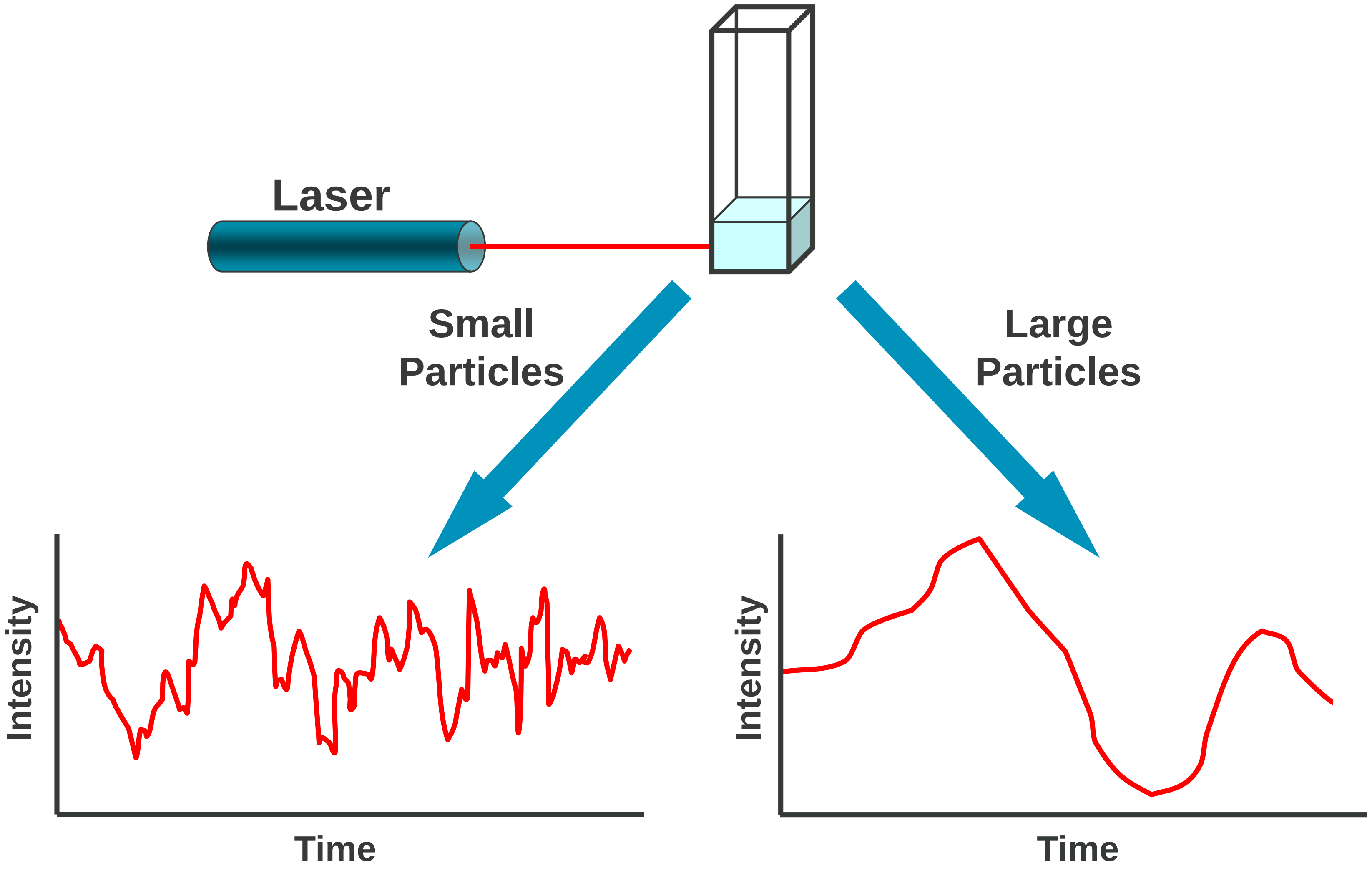
The diameter of a hard sphere that diffuses at the same speed as the particle or molecule being measured

- Dependent on
  - Ionic strength
  - Surface structure
  - Shape

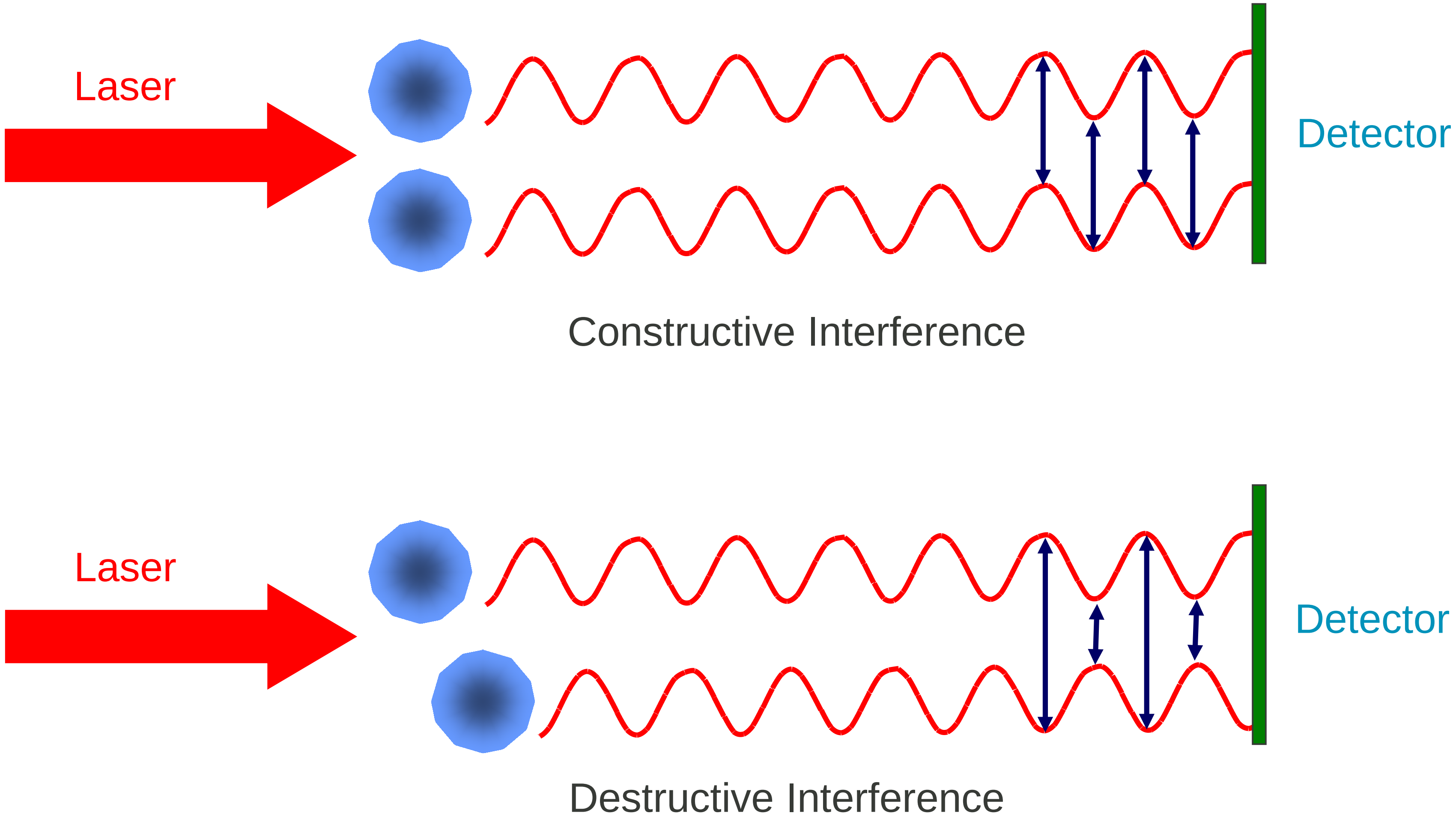
DLS Instrument Components



Intensity Fluctuations and Brownian Motion



Constructive and Destructive Interference



Agenda

1. Brownian Motion
2. **Correlation**
3. Analysing the Correlation Function

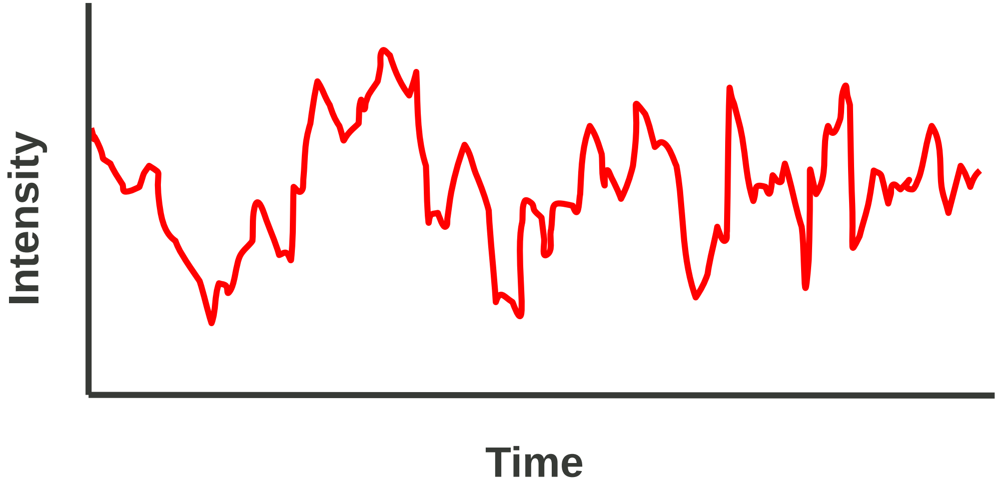
Correlation in Dynamic Light Scattering

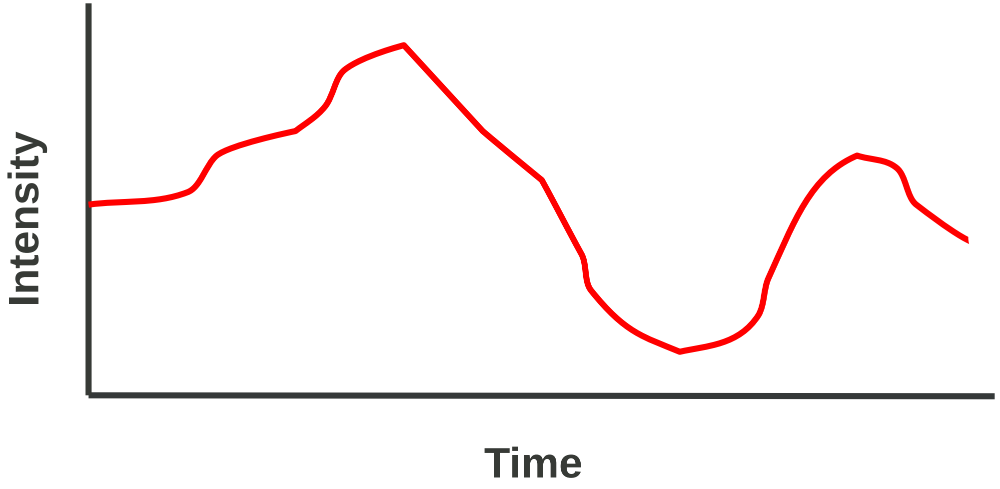
- Technique for extracting the time dependence of a signal in the presence of “noise”
- Time analysis carried out with a correlator
- Constructs the time autocorrelation function G(τ) of the scattered intensity according to

$$G(\tau) = \left\langle \frac{I(t_0) * I(t_0 + \tau)}{I(t_\infty)^2} \right\rangle$$

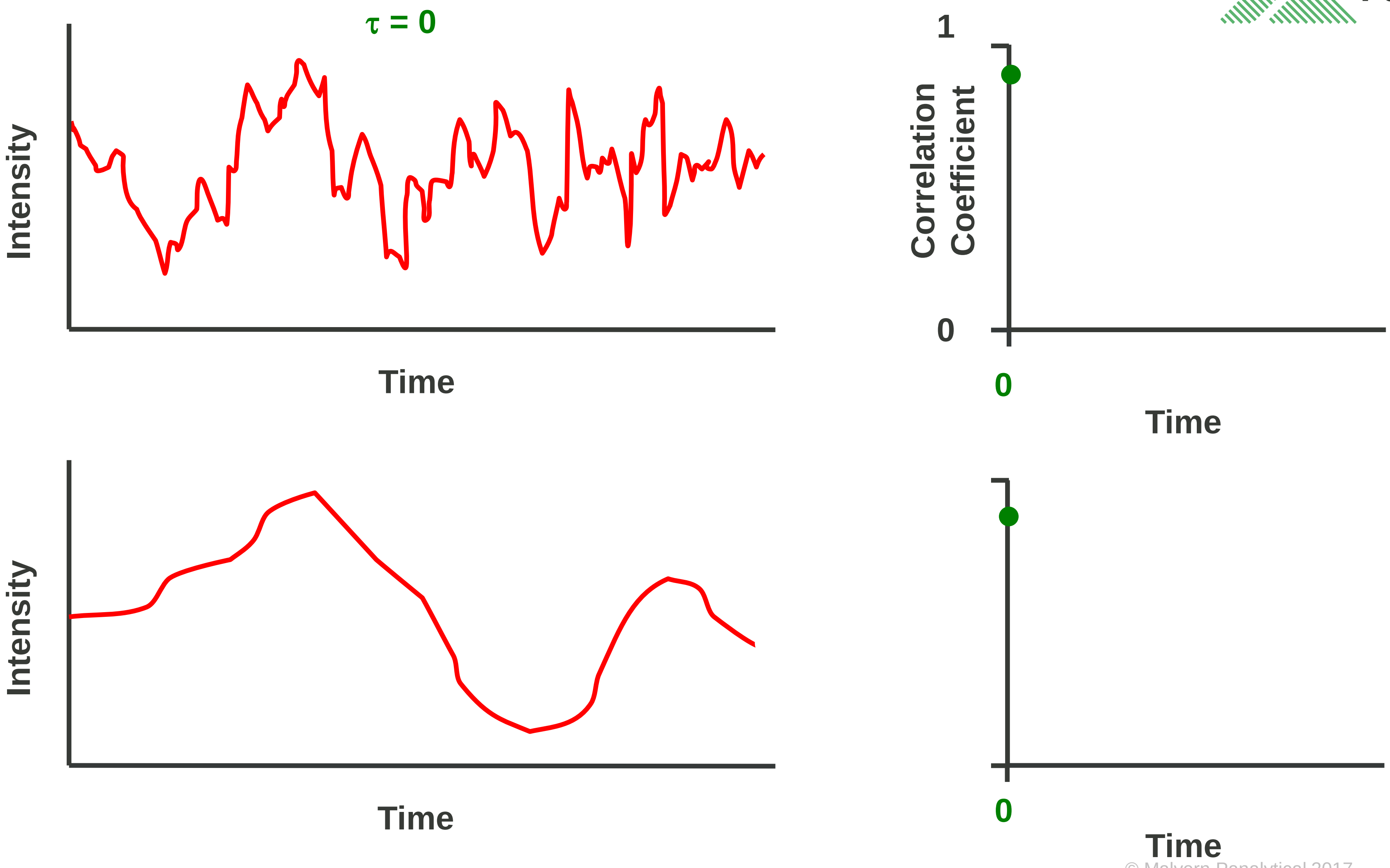
where I = intensity, t is the time and τ = the delay time

Correlation

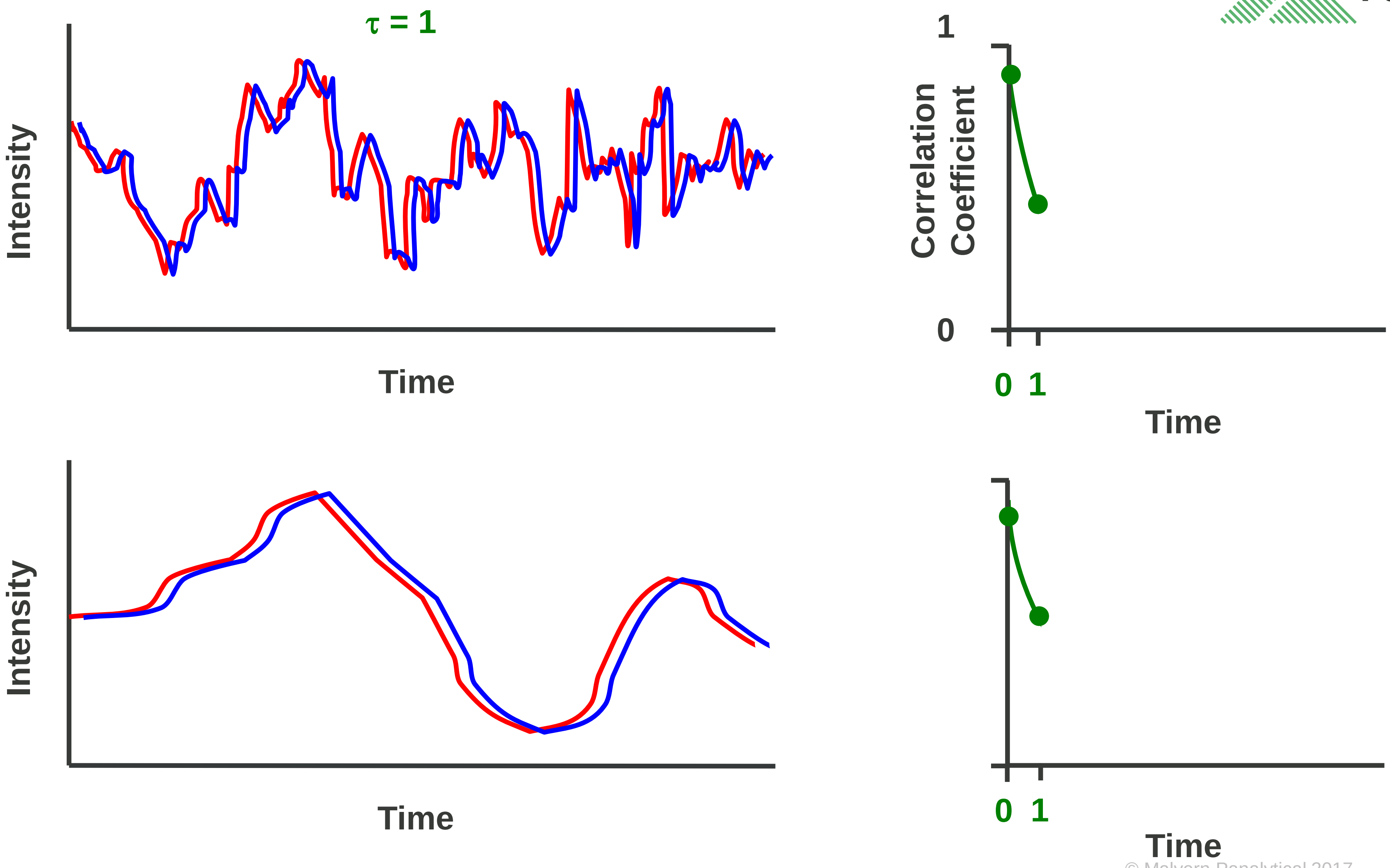




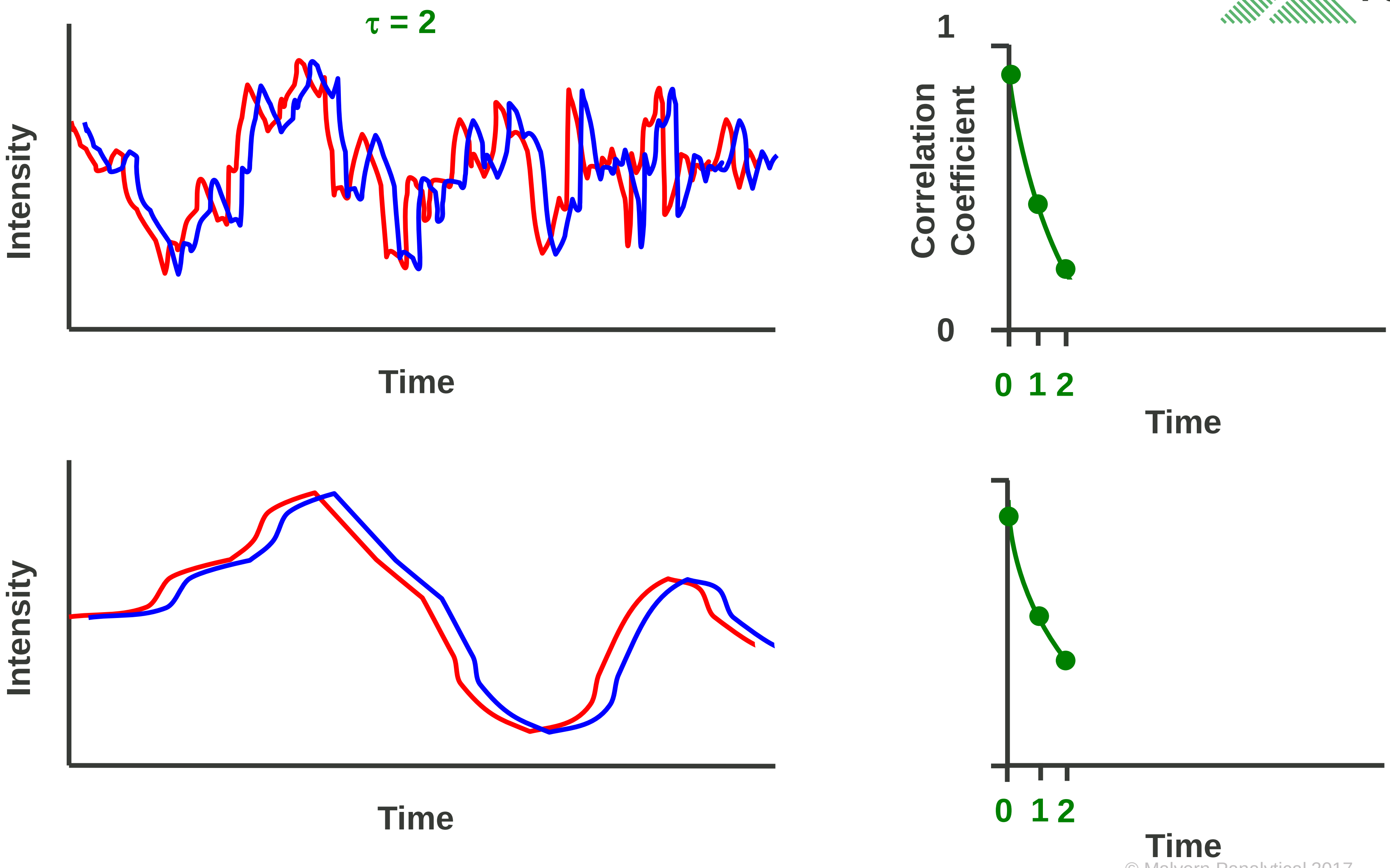
Correlation



Correlation



Correlation



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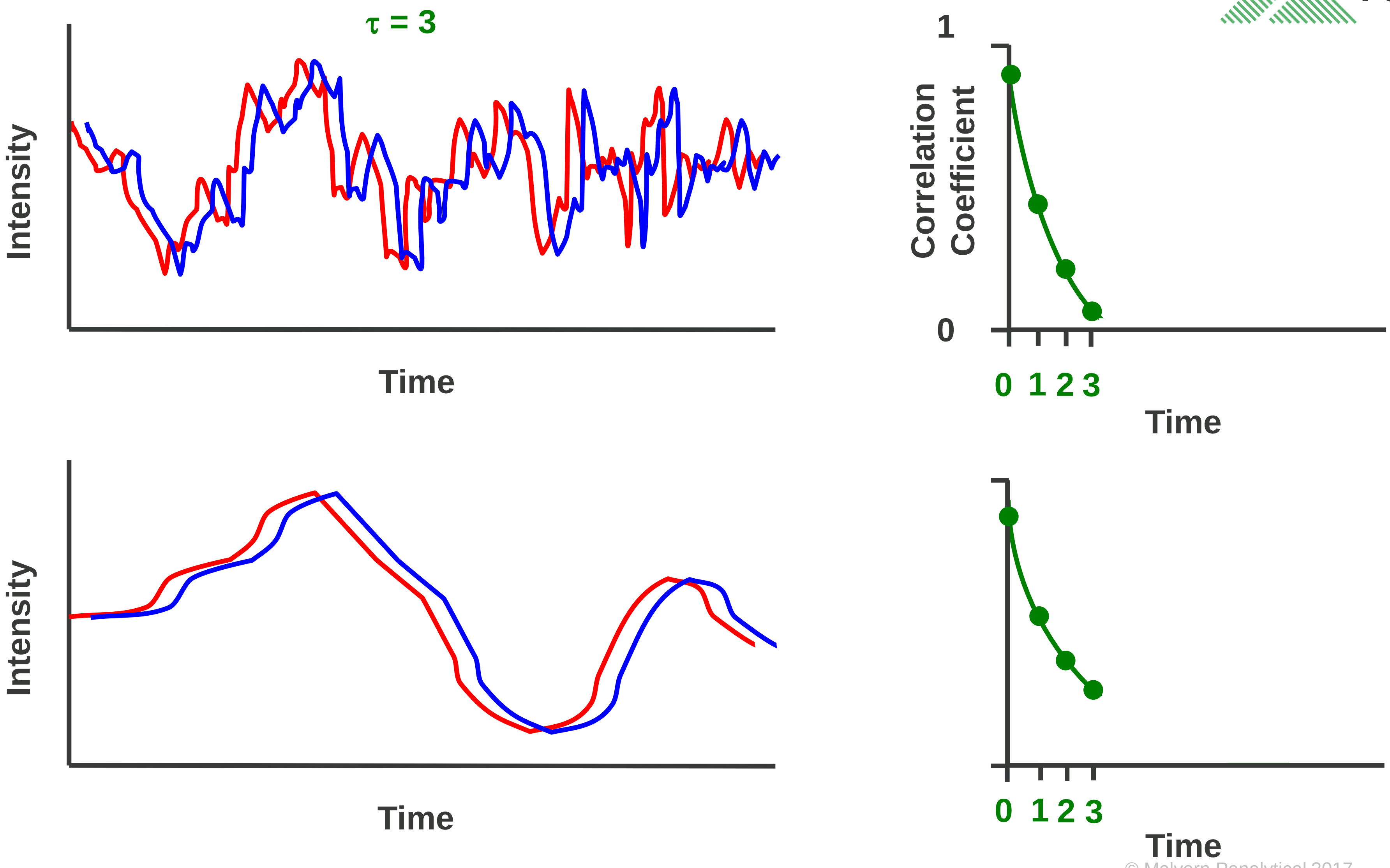
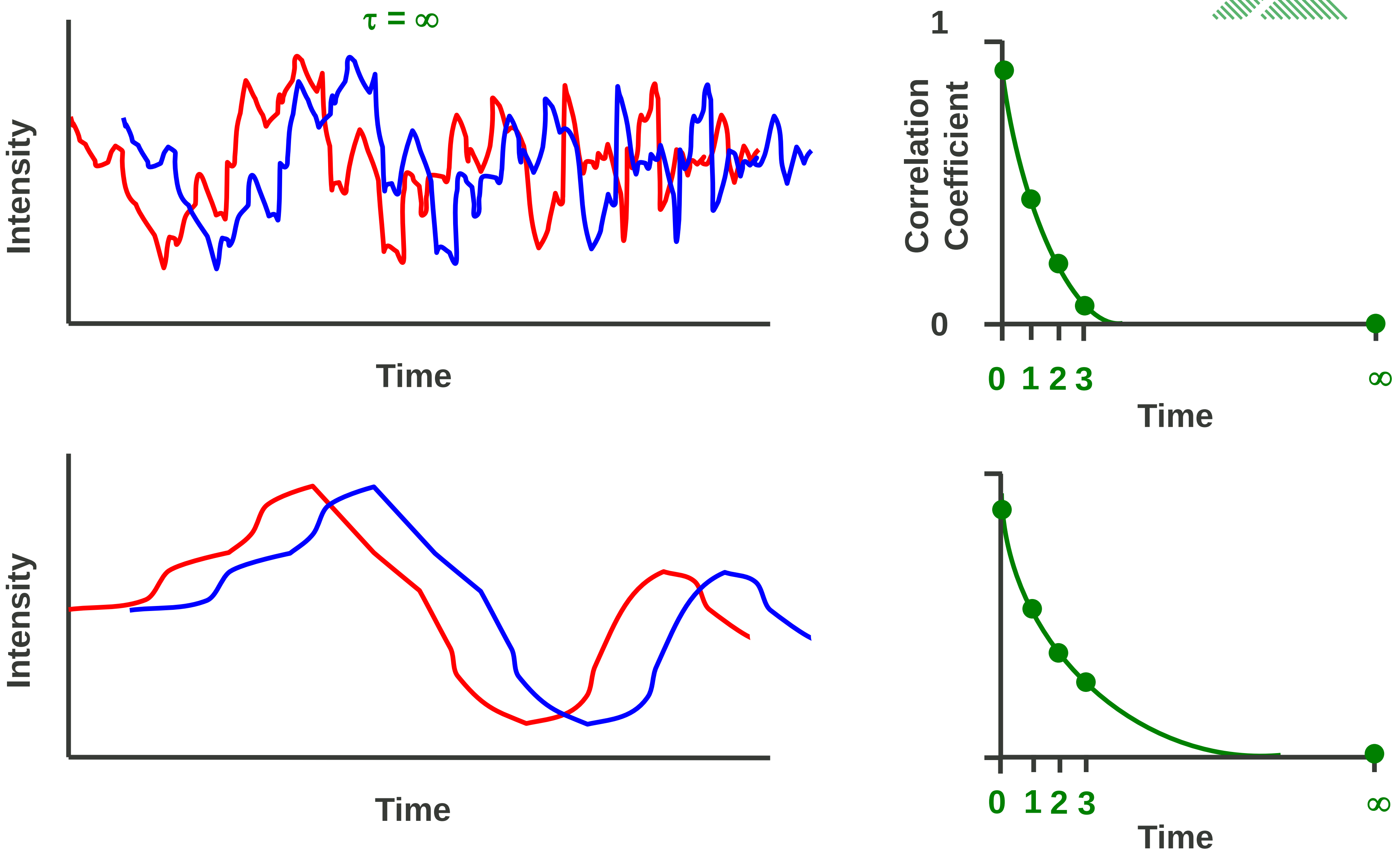
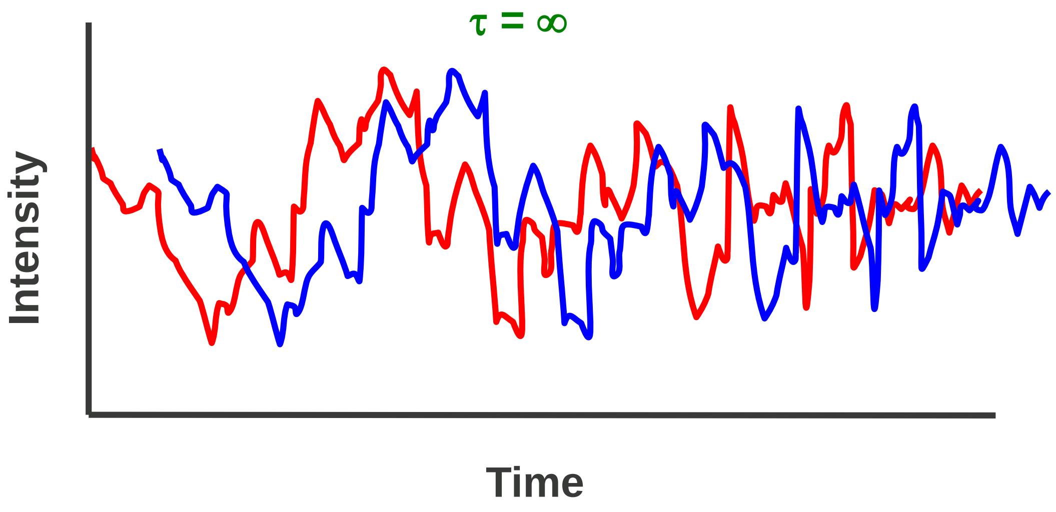
Correlation

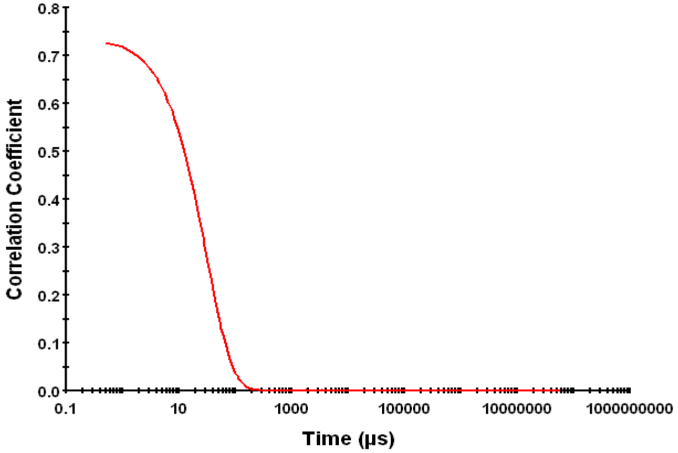


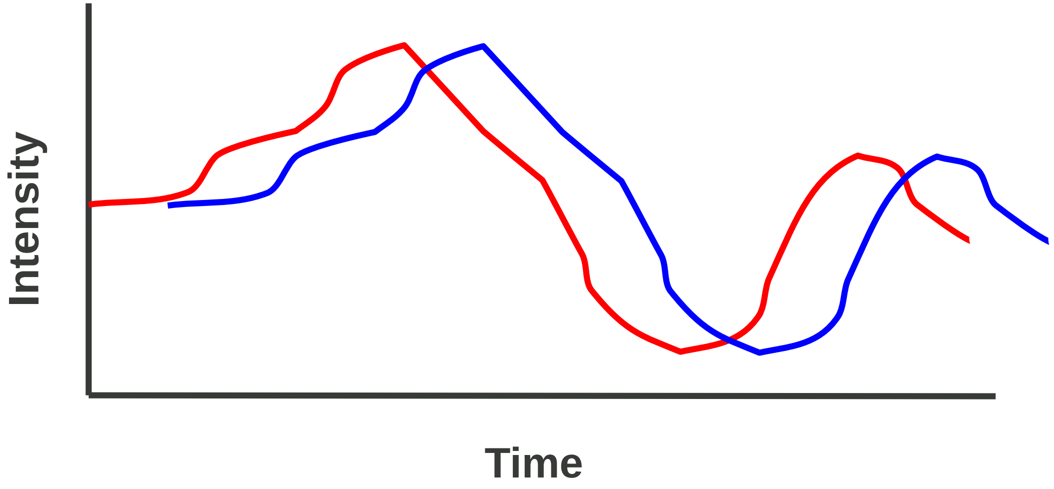
Correlation

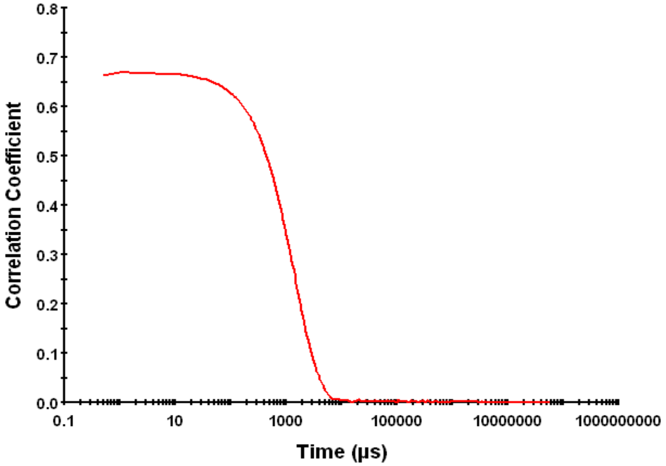


Correlation

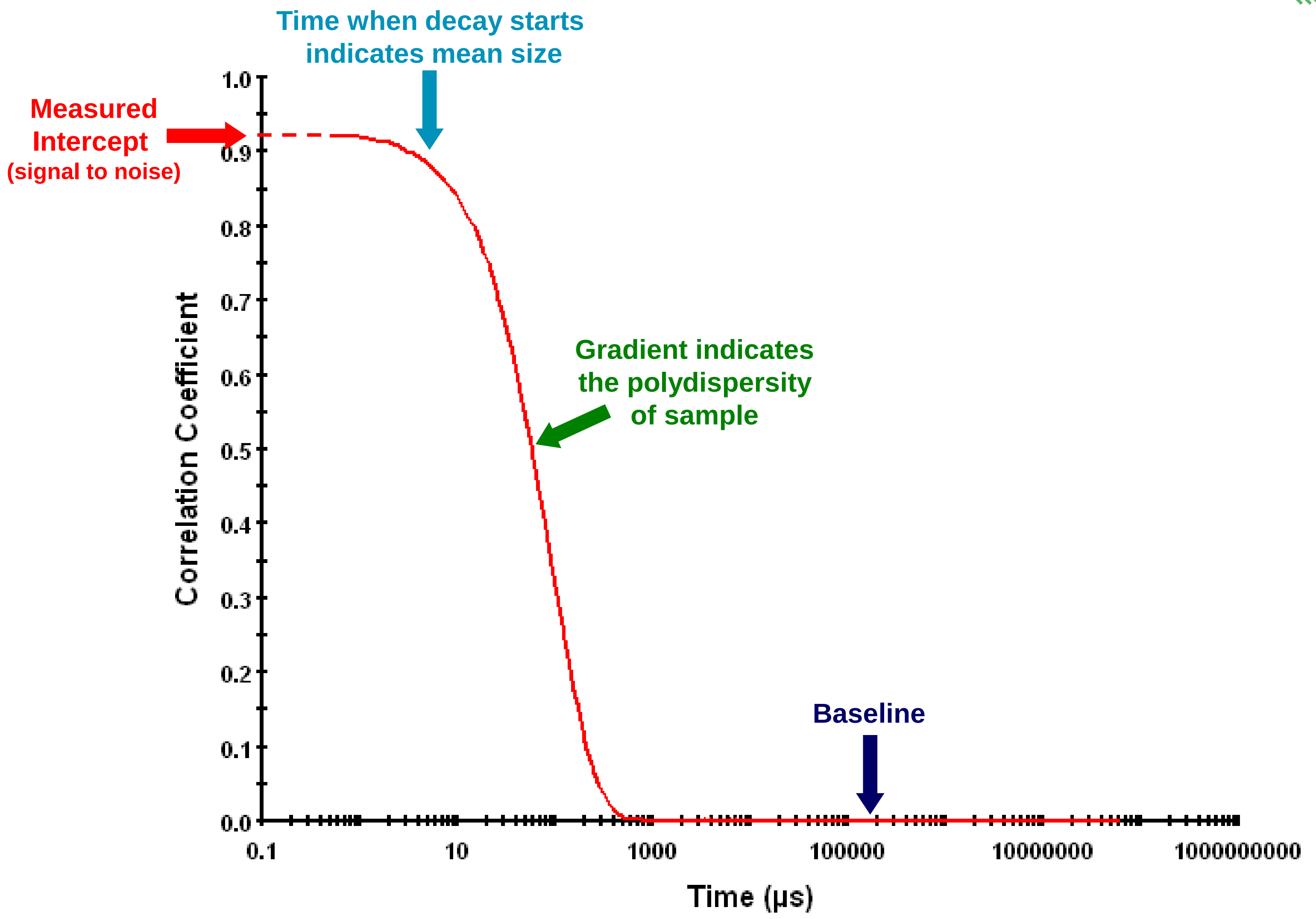








Correlation Functions



Agenda

1. Brownian Motion
2. Correlation
3. **Analysing the Correlation Function**

Correlation Functions

- The correlation function can be modelled with an exponential expression such as:

$$G(\tau) = B + A \sum e^{-2q^2 D \tau}$$

Where

B = baseline at infinite time

A = amplitude (or intercept)

q = scattering vector = $(4\pi\, n/\lambda_o) \sin(\theta/2)$

n = dispersant refractive index

λ_o = laser wavelength

θ = detection angle

D = diffusion coefficient

τ = correlator delay time

Analysing The Correlation Function

- Correlation function contains the **diffusion coefficient** information required to be entered into the Stokes-Einstein equation
- The **diffusion coefficients** are obtained by fitting the correlation function with a suitable **algorithm**

Analysing The Correlation Function

Two different analyses are performed:

Cumulants analysis

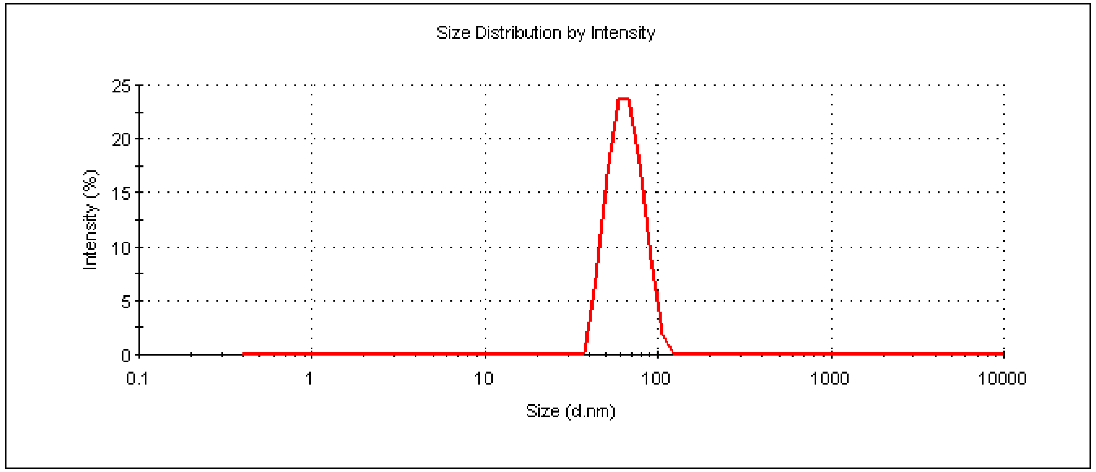
- Mean size (z-average)
- Polydispersity index

Distribution analysis

- Distribution of sizes

Z-Average (d.nm):	63.13
PdI:	0.029
Intercept:	0.965
Result quality :	Good

	Diam. (nm)	% Intensity	Width (nm)
Peak 1:	65.94	100.0	14.35
Peak 2:	0.000	0.0	0.000
Peak 3:	0.000	0.0	0.000



Cumulants Analysis

- Defined in the International Standards ISO13321 (1996), ISO22412 (2008) and **ISO22412 (2017)**
- Only gives a **mean particle size (z-average)** and an **estimate of the width of the distribution (polydispersity index)**
- Only the dispersant refractive index and viscosity are required for this analysis

The z-Average Diameter

- Definition of the z-Average Diameter (Z_D):

The intensity-weighted mean diameter derived from the cumulants analysis

- Specific to light scattering
- Very sensitive to the presence of aggregates or large contaminants due to the inherent intensity weighting

Polydispersity Index

- Definition of the Polydispersity Index (PdI):

A dimensionless measure of the broadness of the size distribution calculated from the cumulants analysis

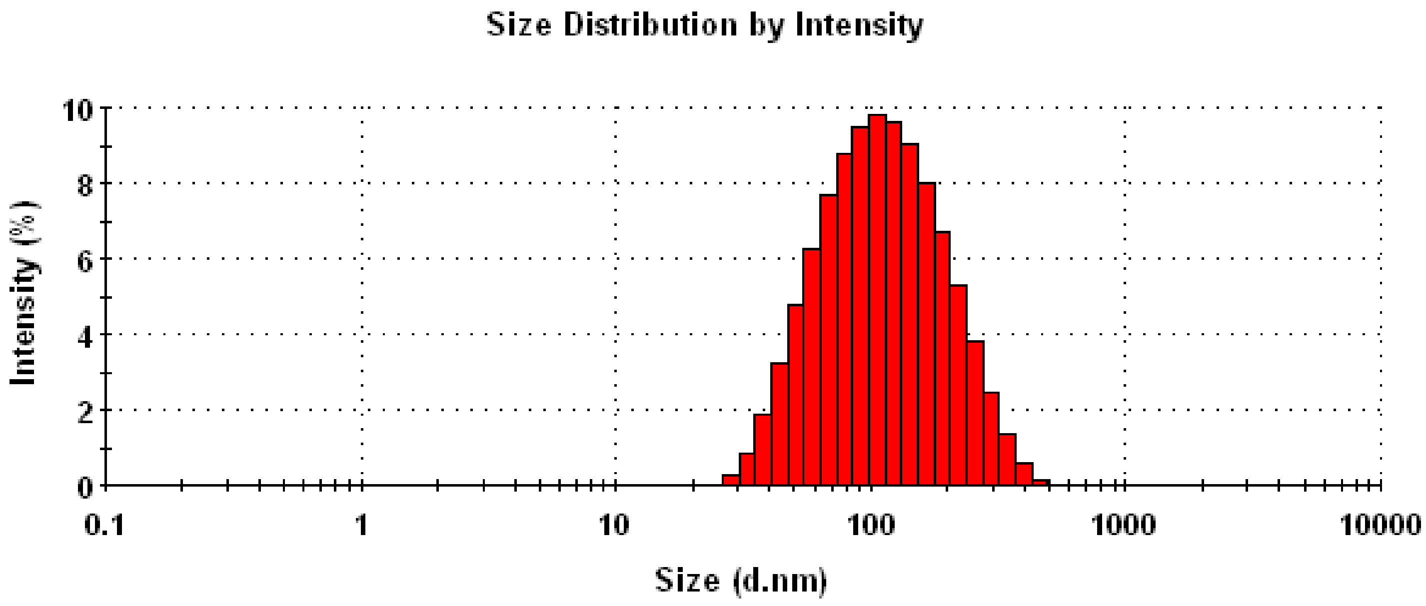
- Ranges from 0 to 1 in the Zetasizer software
- Values > 1 indicate that the distribution is so polydisperse, the sample may not be suitable for measurement by DLS

Zetasizer Distribution Algorithms

- **General Purpose**
  - Suitable for the majority of samples where no knowledge of the distribution is available
  - Will give broad, smooth distributions
- **Multiple Narrow Modes**
  - Suitable for samples suspected to contain discrete populations
  - Will give narrow peaks
- **Protein Analysis**
  - Best suited for protein samples – will give narrow peaks – can be used for any sample type
  - Automatically picks the optimal distribution

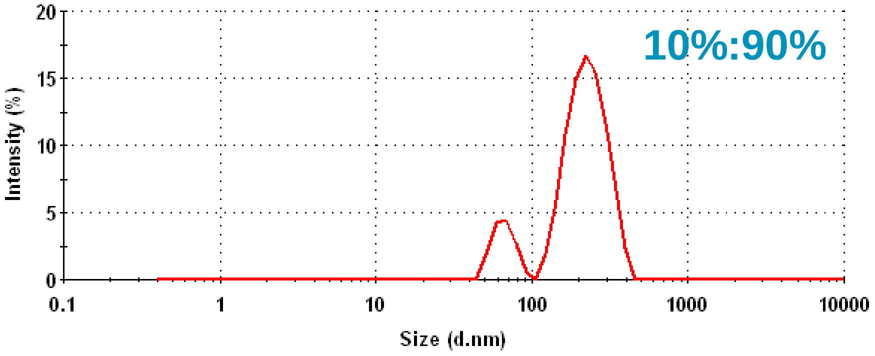
Size Distributions in the Zetasizer Software

- Primary size distribution is **intensity**-weighted
- A plot of the **relative intensity of light scattered** by particles (on the Y axis) versus various **size classes** (on the X axis) which are logarithmically spaced



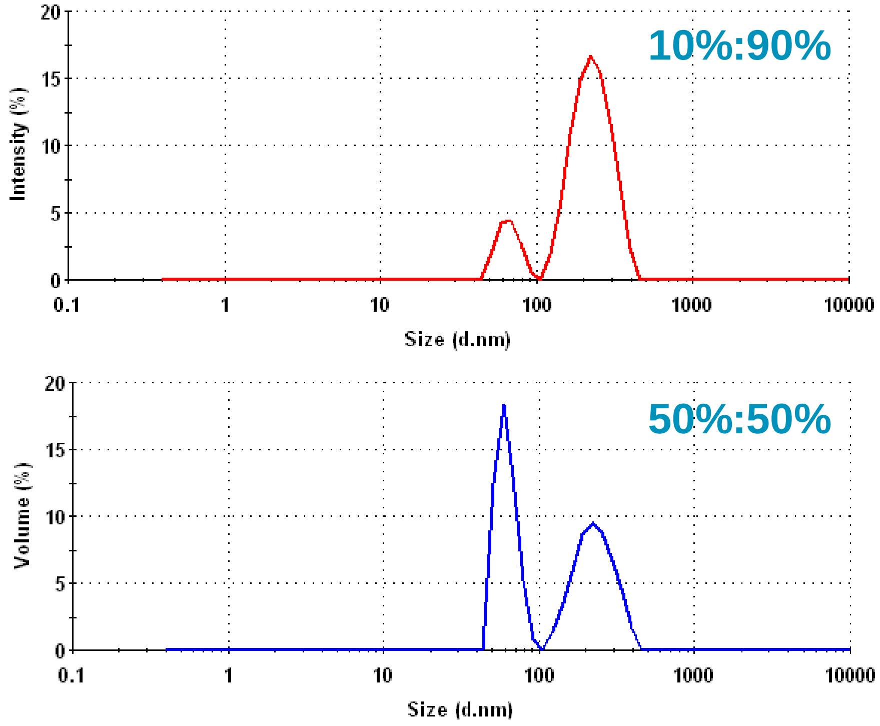
Intensity Size Distributions

- Primary result
- Based upon the intensity of light scattered by particles
- Sensitive to the presence of large particles/aggregates /dust
- Only the dispersant viscosity and refractive index values are required



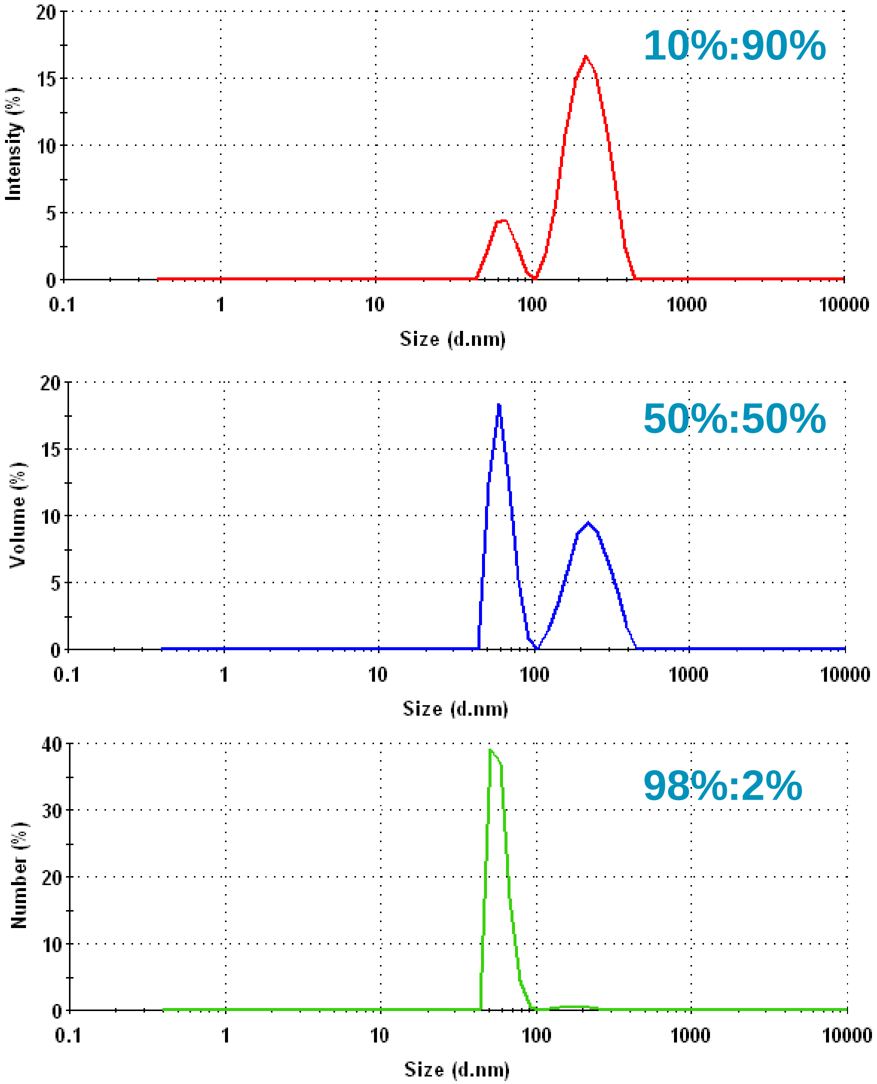
Volume Size Distributions

- Derived from the intensity distribution using Mie theory
- Equivalent to the mass or weight distribution
- Particle optical properties required to make this transformation
  - Particle refractive index
  - Particle absorption



Number Size Distributions

- Derived from the intensity distribution using Mie theory
- Particle optical properties required to make this transformation
  - Particle refractive index
  - Particle absorption



Size Distributions From DLS

- DLS technique tends to overestimate the width of the peaks in the distribution
- This effect is magnified in the transformations to volume and number

- The volume and number size distributions should only be used for estimating the **relative amounts** of material in separate peaks as the means and particularly the widths are less reliable

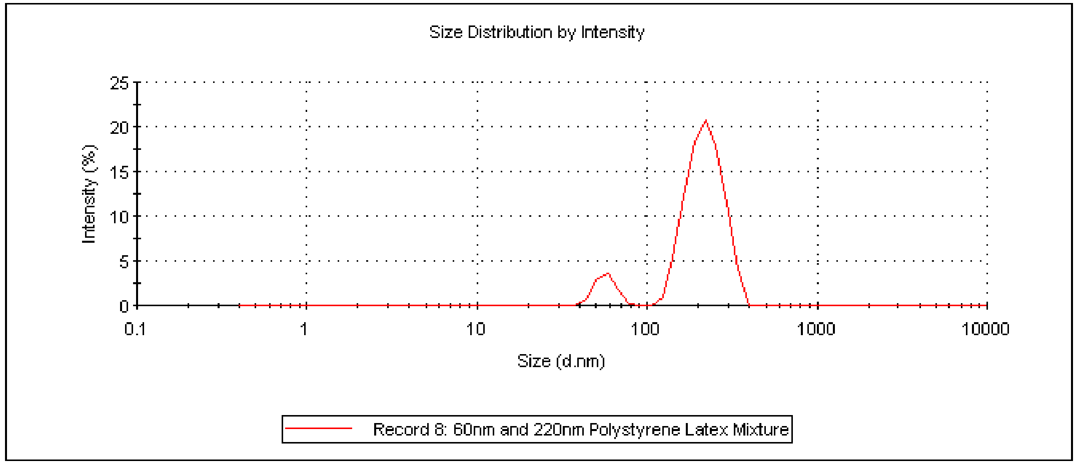
Volume/Number Distributions: Recommended Use

- Use the **Intensity PSD** for reporting the size of each peak in the distribution
- Use the **Volume** or **Number PSD** for reporting the relative amounts of each peak in the distribution

(Modal Size Report)

Z-Average (d.nm):	175.1
PdI:	0.191
Intercept:	0.866
Result quality :	Good

	Diam. (nm)	Width (nm)	% Intensity	Volume	Number
Peak 1:	222.9	51.67	90.8	52.3	2.0
Peak 2:	57.07	7.544	9.2	47.7	98.0
Peak 3:	0.000	0.000	0.0	0.0	0.0



Any Questions?

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