



Cordouan Thetis — jedinečný systém pro měření velikosti i tvaru nanočástic

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THETIS™ : Víceúhlové DLS, SLS a D-DLS

Nejpokročilejší řešení pro charakterizaci nanočástic!

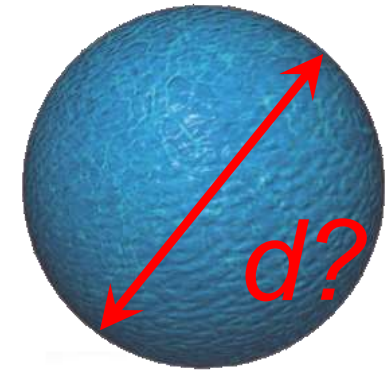


Proč je velikost nanočástic tak důležitá?

- Souvisí se specifickým povrchem částic
- Ovlivňuje schopnost procházet membránami a interagovat s povrchy
- Agregace je důležitá pro posouzení stability suspenzí
- Schopnost self-assembly
- Ovlivňuje optické, mechanické a elektrické vlastnosti
- Etc.

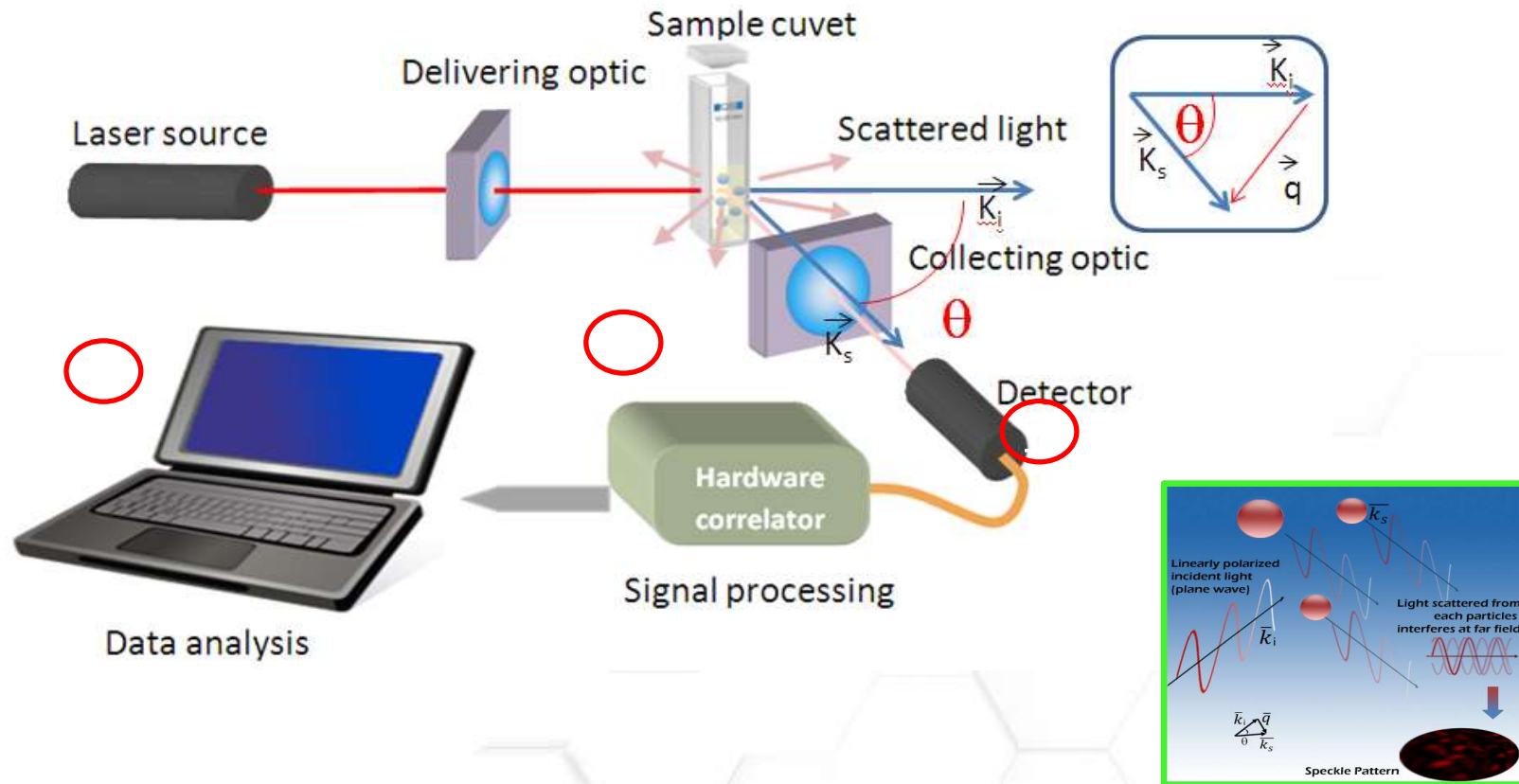
Pro charakterizaci je využívána řada technik:

- Elektronová mikroskopie: TEM,
- Electrozone Coulter counter
- Hmotnostní snímání: Diferenciální sedimentační centrifugace, rezonanční hmotnostní detekce
- X-Ray: SAXS,
- Optické metody: Sledování částic, Laserová difrakce, **Dynamický rozptyl světla (DLS)**



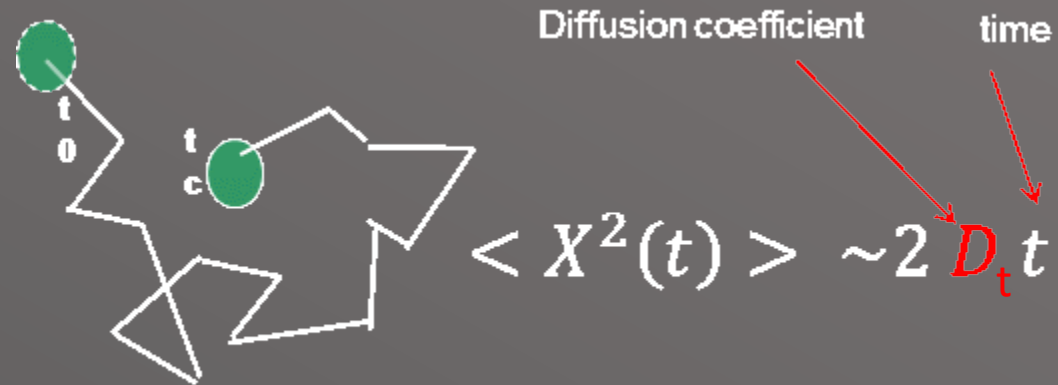
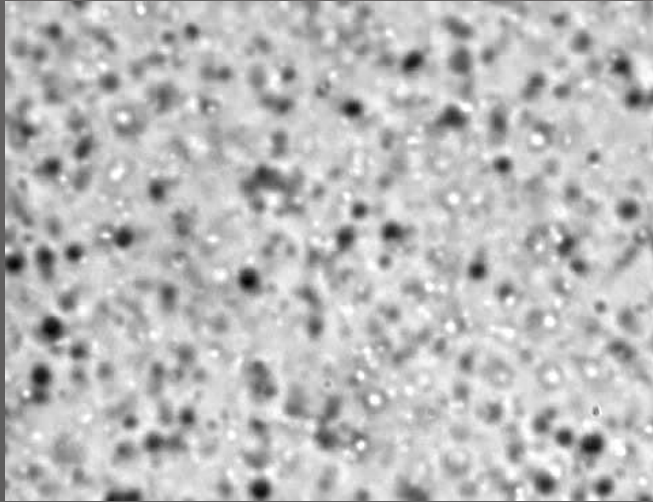
DLS Uspořádání měření:

- Měřena fluktuace rozptylu světla za účelem sledování Brownova pohybu NPs



Brownův pohyb: „znak“ velikosti NPs

Brownian motion= Random “walk”



L. BACHELIER (1901)

NPs:
hard spheres without
interactions

Viscosity Boltzmann Temperature

$$D_t = \frac{KT}{3\pi\eta \phi_H}$$

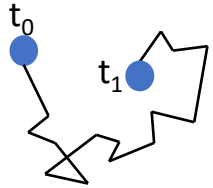
→

$$\phi_H = \frac{KT}{3\pi\eta D_t}$$

EINSTEIN (1905)

Princip měření DLS

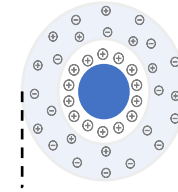
Brownian motion



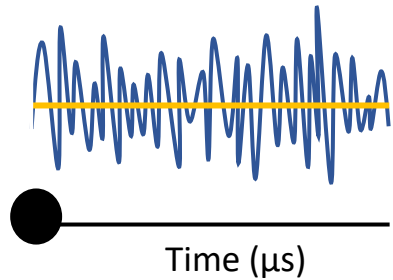
Translational Diffusion coefficient

Stokes-Einstein equations

Hydrodynamic diameter

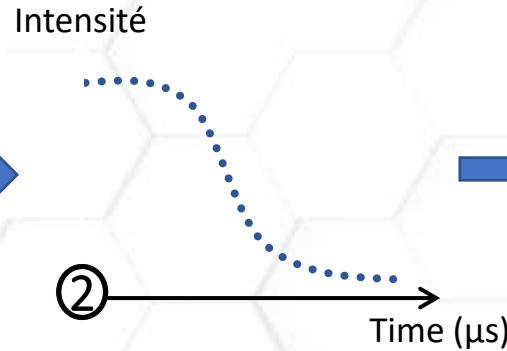


Light intensity



T (°C)

Autocorrelation



Size Distribution



EM field: $E(\omega t) = \sum E_i e^{-i(kr - \omega t)}$

$$G_2 = A + \beta \exp(-2\Gamma t)$$

Intensity: $I(t) = E(t) E^*(t)$

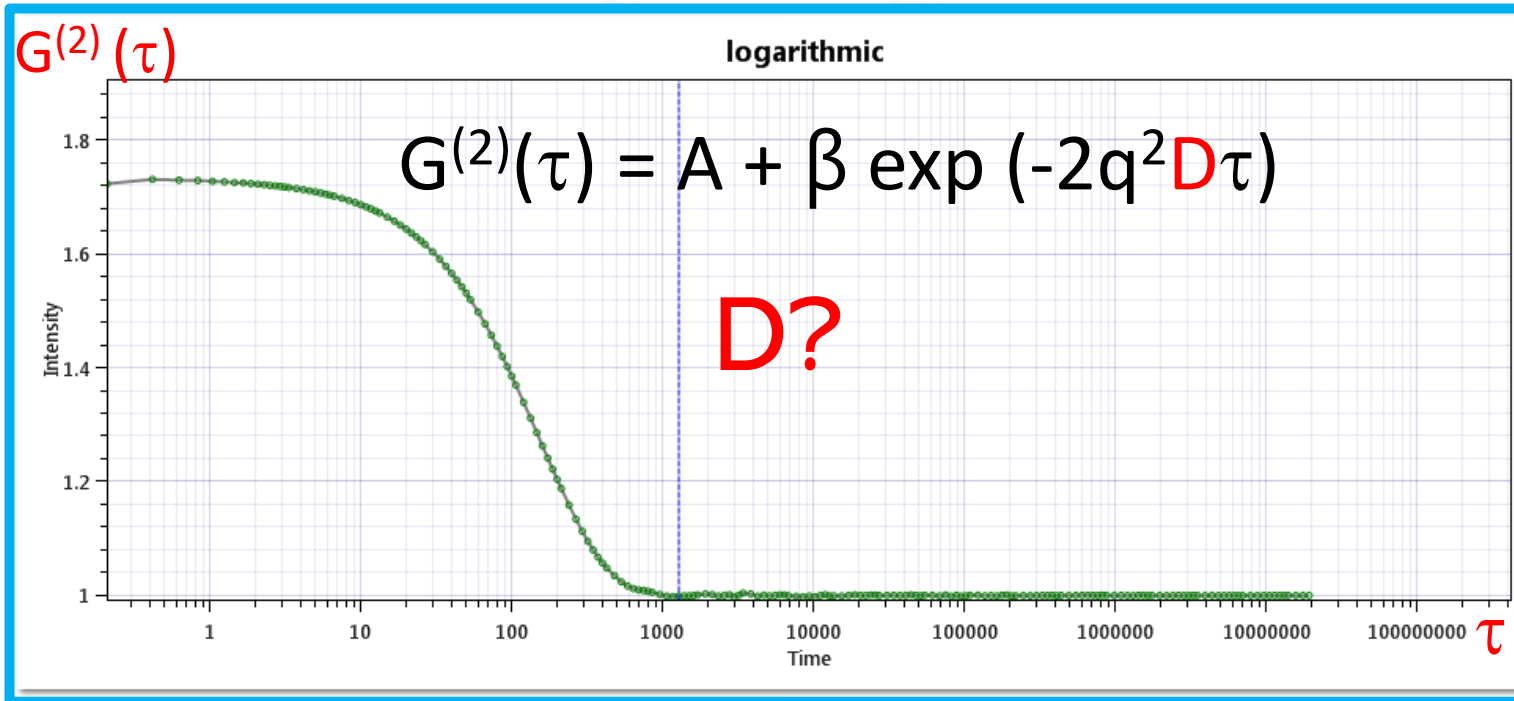
$$q = \frac{4\pi n_0}{\lambda} \sin \theta / 2$$

$$\phi_H = \frac{KT}{3\pi\eta D}$$

$$\Gamma = q^2 D$$

Correlogram & Zpracování dat

Inversion problem: Jak najít optimální exponenciální křivku odpovídající experimentálnímu korelogramu?

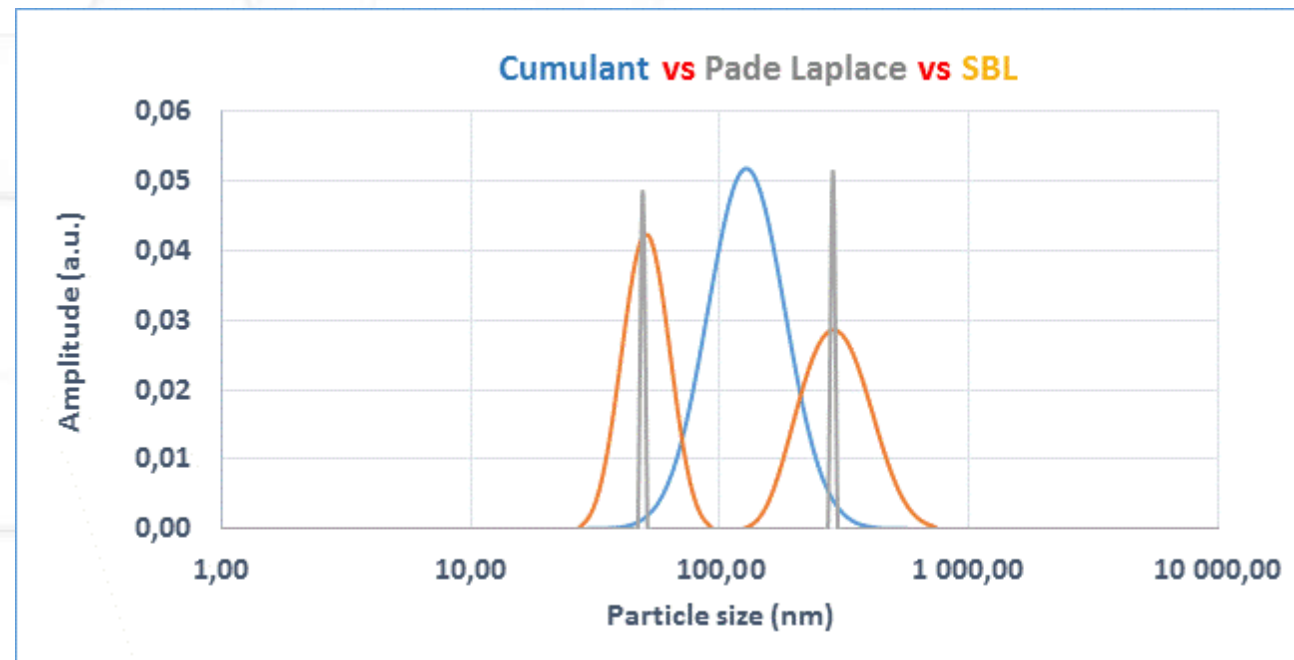


Fit leads to D , and D to the diameter of NPs ϕ_H .

$$\phi_H = \frac{KT}{3\pi\eta D}$$

Inverzní algoritmy pro stanovení velikosti NPs:

Algorithm	Number of populations	Distribution	Model
Cumulants	Monomodal continuous	Yes	$G(\tau) = A + B e^{-\Gamma\tau}$; Gaussian with Zavg & PDI
Pade Laplace	Multi modal discrete	No	$G(\tau) = A + \sum_{i=1}^{250} B_i e^{-\Gamma_i\tau}$
SBL	Multimodal continuous	Yes	$G(\tau) = A + \int_0^{10\mu m} B(z) e^{-\Gamma(z)\tau} dz$



DLS measurement

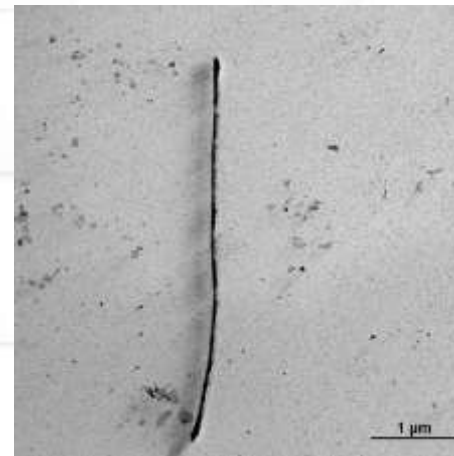
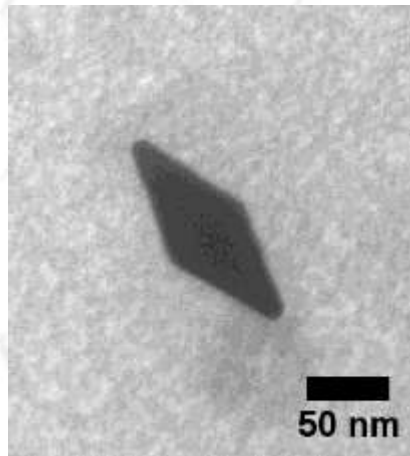
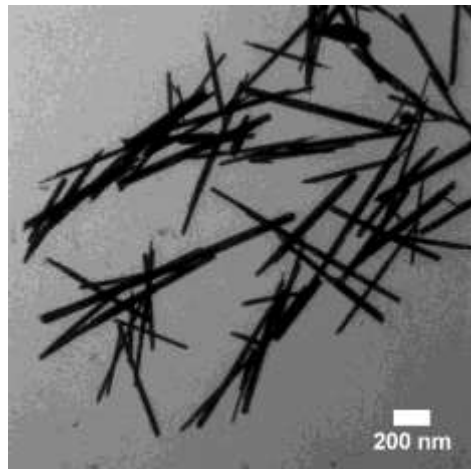
NanoKin v2.3.11.0

LUIS UK DDAO 10% LUIS UK DDAO 10%_20210329_120524.dat



Proč záleží na tvaru nanočástice?

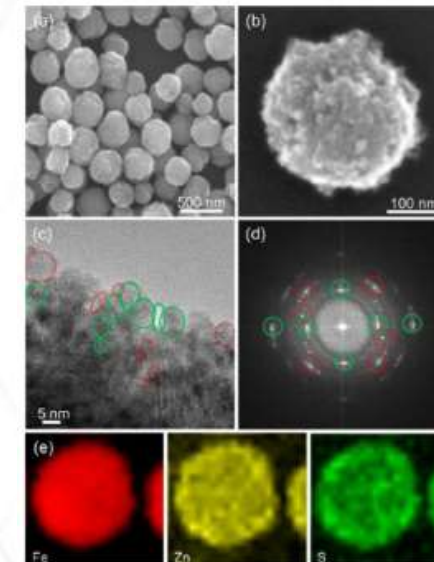
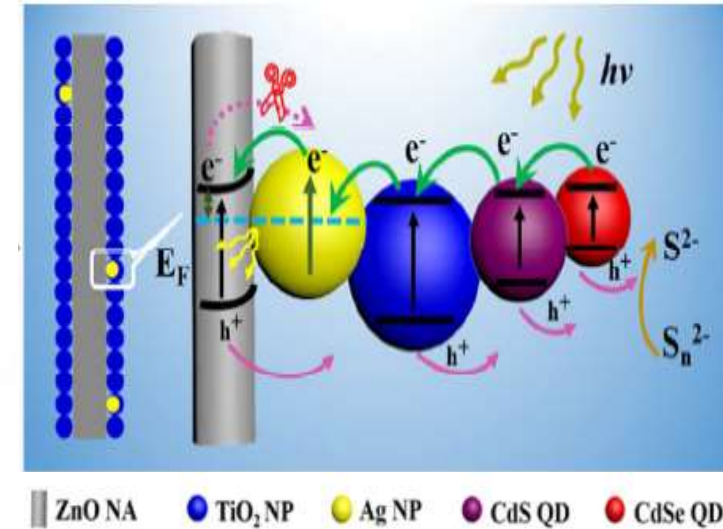
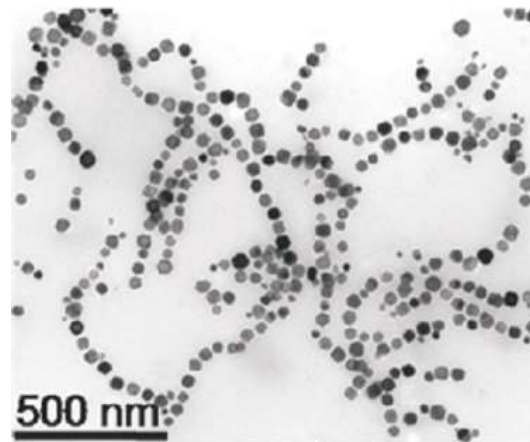
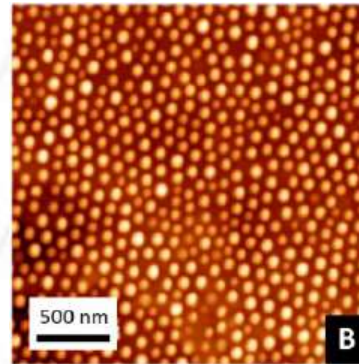
1. Pokročilé materiály vyžadují nové NPs
2. Grafen, uhlíkové a stříbrné nanotrubičky jsou příští generací elektrických transponderů
3. Syntéza nových tvarů nanočástic



Příklady anizotropních NPs:

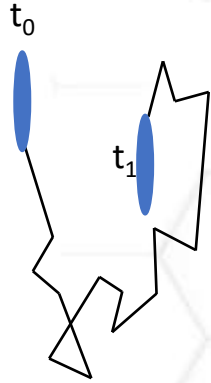
1. Hybridní nanomateriály:

1. Efektivní materiály solárních článků
2. Nové optické materiály
3. Nanokrystaly vylepšující strukturu
4. Magnetické nanočástice pro zobrazovací metody

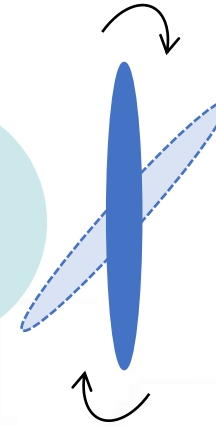


Chování anizotropních částic

Translational Diffusion
coefficient
 D_t



Rotational
Diffusion coefficient
 D_r



1. 2 rozdílné pohyby: translační a rotační
2. Vedou ke 2 rozdílným difúzním koeficientům!
3. Vyžaduje polarizaci dopadajícího světla

Anizotropní difúzní koeficienty:

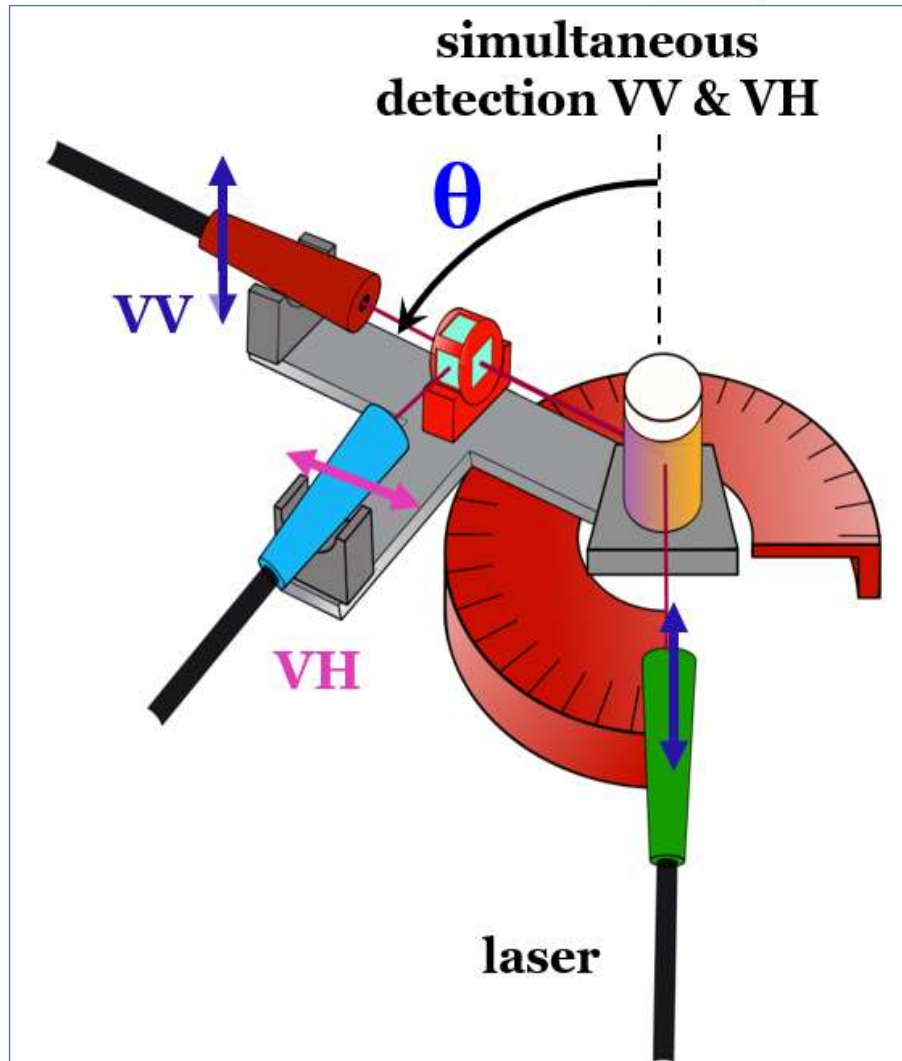
$$D_t = \frac{k_B \cdot T}{3 \cdot \pi \cdot \eta \cdot L} \cdot \left[\ln \left(\frac{L}{w} \right) + \gamma \right]$$

$$D_r = \frac{3 \cdot k_B \cdot T}{\pi \cdot \eta \cdot L^3} \cdot \left[\ln \left(\frac{L}{w} \right) + \varepsilon \right]$$

- **L**: length
- **w**: width
- **L/w**: aspect ratio

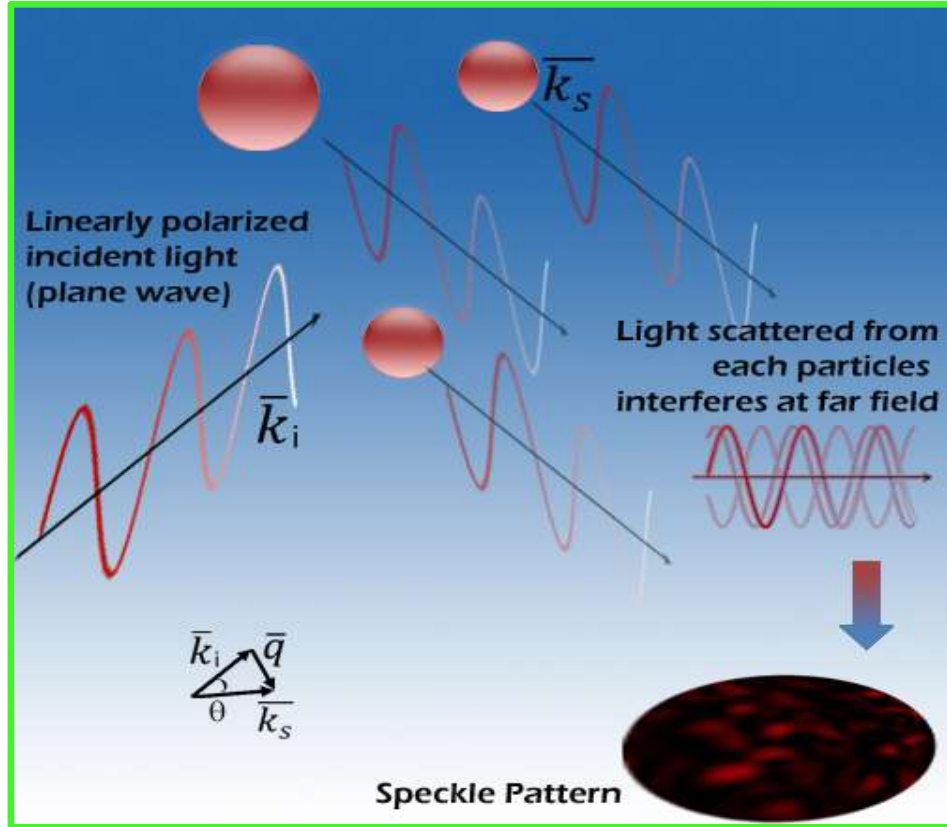
1. Výpočet je proveditelný
2. Získáme parametry L a W

D-DLS princip:

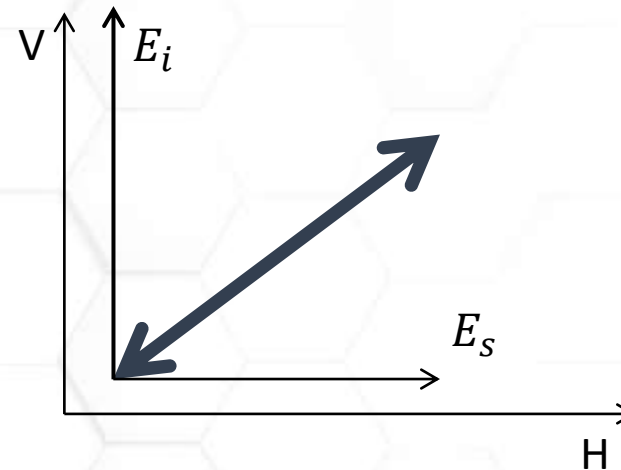


1. Detecting intensity on VV
2. Detecting intensity on VH

D-DLS princip:



1. Pro sférické částice žádné fotony na VH nevychází
2. Anizotropie nanočástic vede k průchodu fotonů na VH



VV a VH korelogramy:

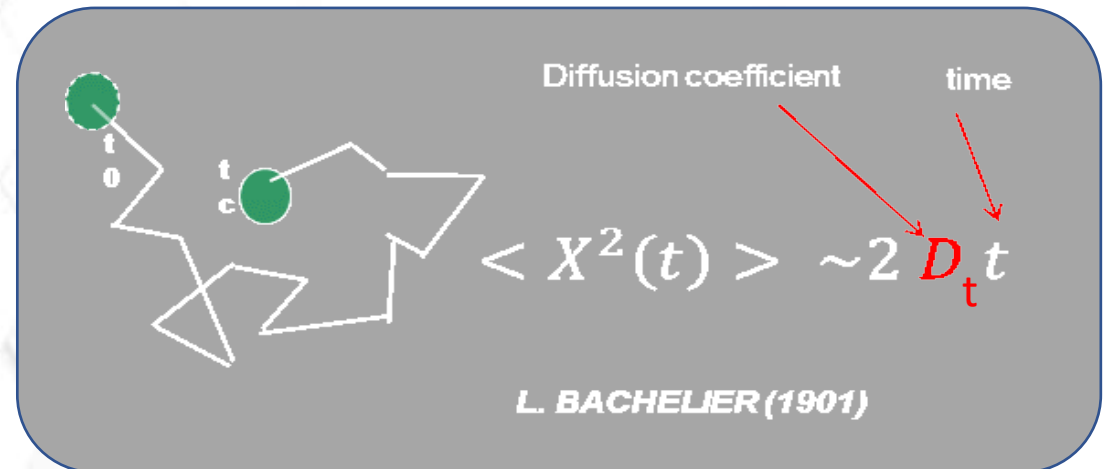
$$G_{(VV)}(\tau) = A_1 \cdot \exp^{-(\Gamma_t + \Gamma_r)\tau} + A_2 \cdot \exp^{-(\Gamma_t)\tau} + B$$

$$G_{(VH)}(\tau) = A_3 \cdot \exp^{-(\Gamma_t + \Gamma_r)\tau} + B$$

$$\Gamma_t = D_t \cdot q^2$$

$$\Gamma_r = 6 \cdot D_r$$

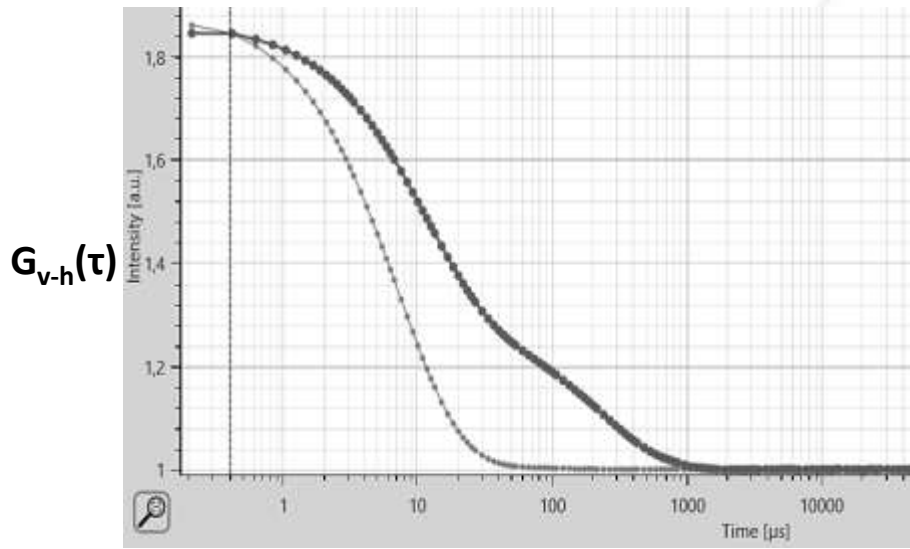
For isotropic particles : A_3 & $A_1 \ll A_2$



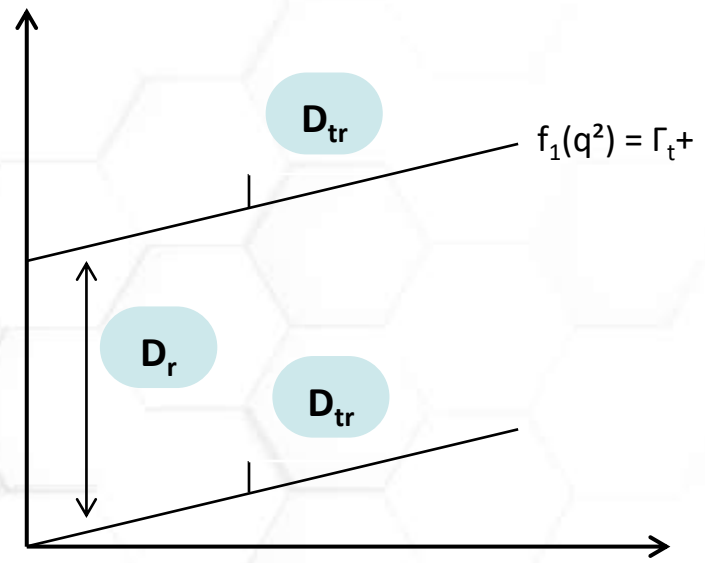
Analytický postup

① $G_{(VV)}(\tau) = A_1 \cdot \exp^{-(\Gamma_t + \Gamma_r)\tau} + A_2 \cdot \exp^{-(\Gamma_t)\tau} + B$

$G_{(VH)}(\tau) = A_3 \cdot \exp^{-(\Gamma_t + \Gamma_r)\tau} + B$



③ coefficients D_t and D_r



② $\Gamma_{tr} + \Gamma_{rot} = D_t q^2 + 6D_r$

$\Gamma_{tr} = D_t q^2$

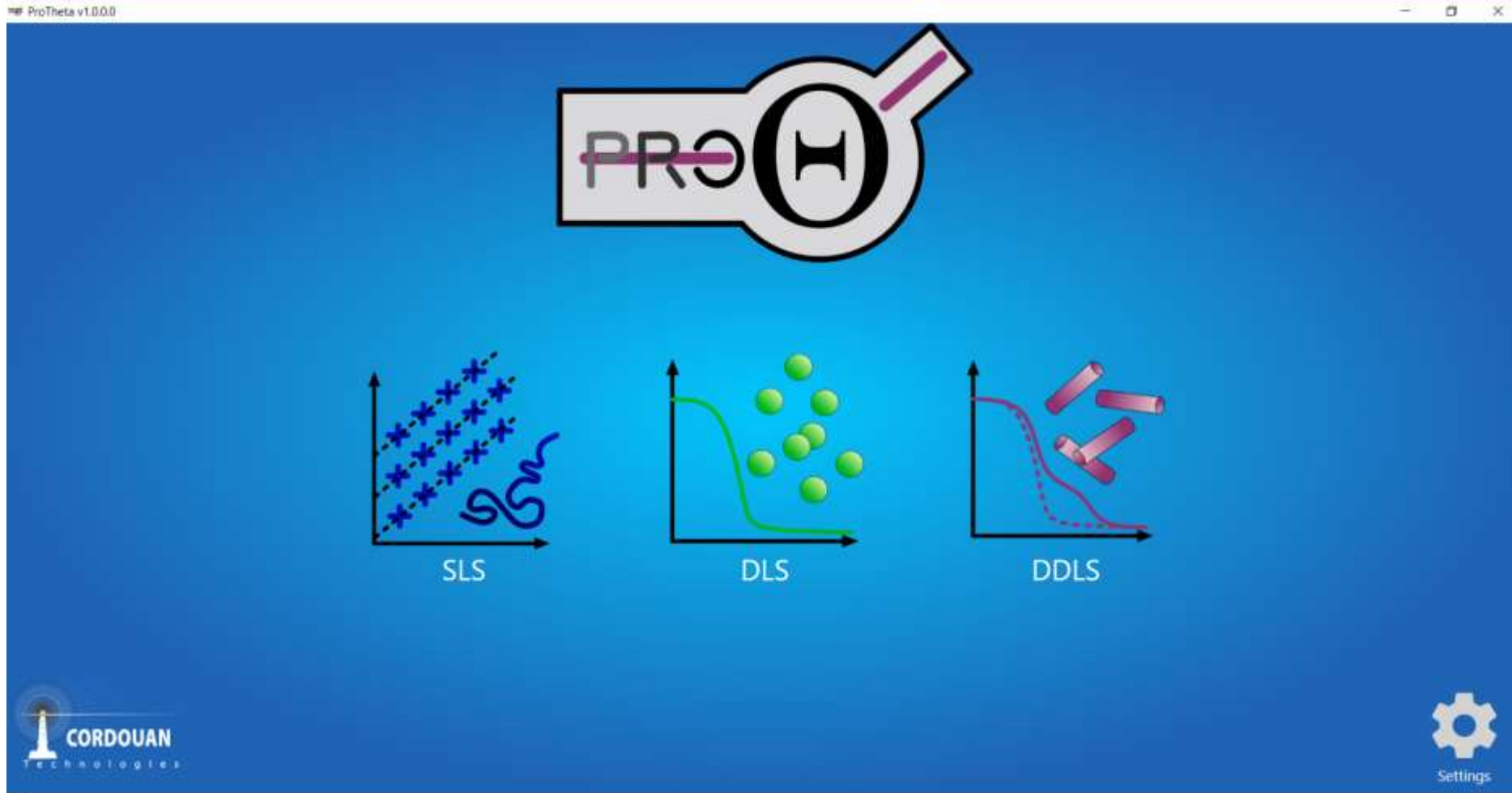
④ Length L, Aspect Ratio AR and width w

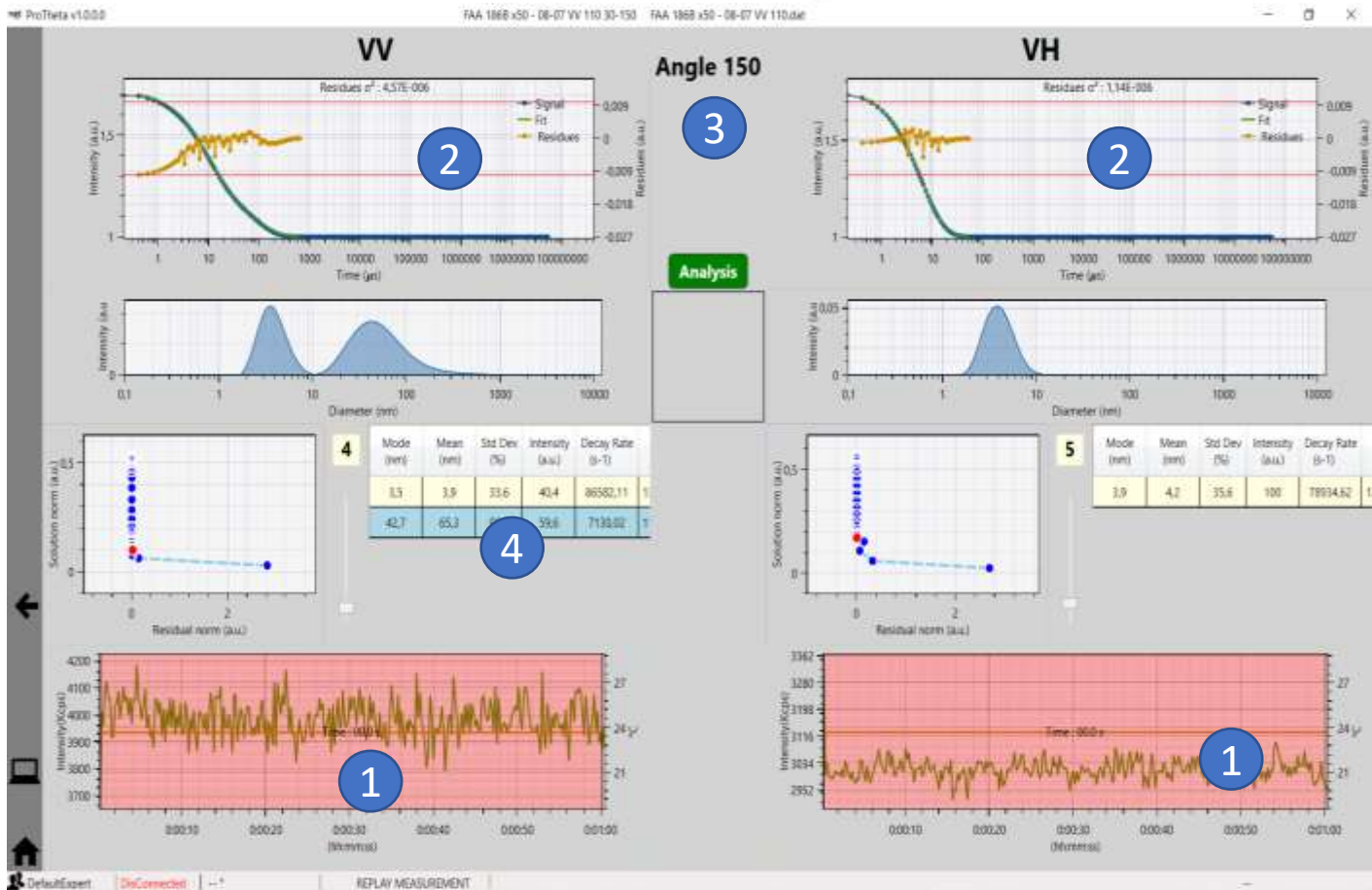
THETIS: A multi-angle DLS, SLS & D-DLS

- DLS, DDLS and SLS in one instrument
- Multi-angle scattering measurement system: 30 to 160 degrees
- Real time and Time resolved Software correlator
- High Power single mode laser @ 635 nm
- Scattered Intensity measurements on 2 perpendicular polarizations
- Temperature controlled from 1 to 70 degrees Celsius
- Molecular weight, concentration and replay modes for DLS and DDLS data
- 3 different algorithms for particle size measurements



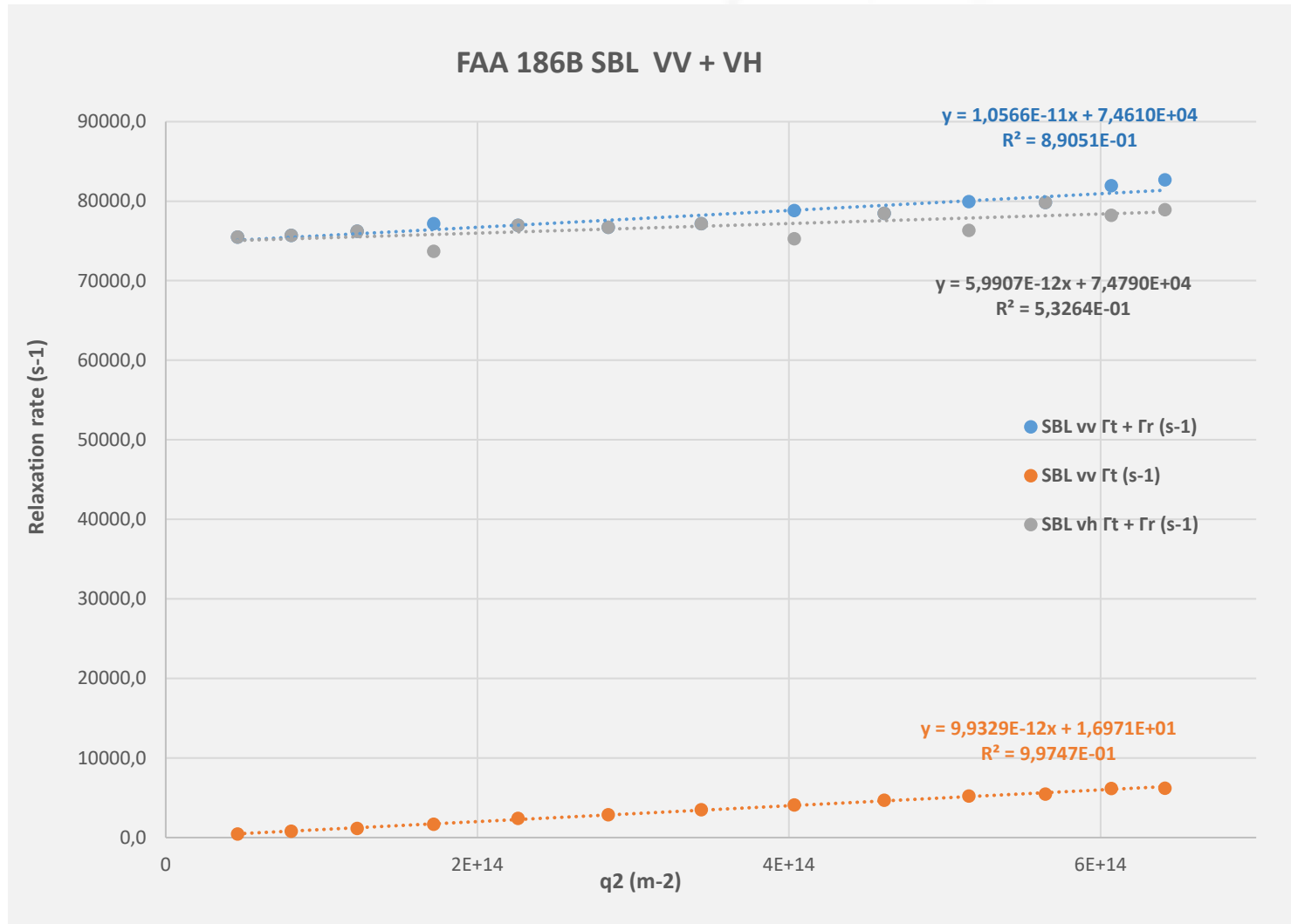
ProTheta software



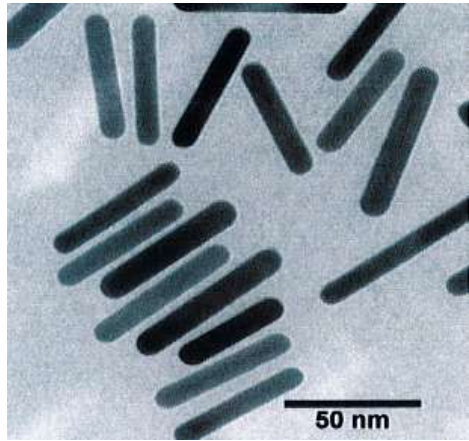


- 1 2 Channels (VV, VH) Time resolved D-DLS
- 2 Both VV and VH correlogram
- 3 User selected/automatic scattering angle setting
- 4 Automatic calculation of length, width & Aspect ratio / Rotational & Translational Diffusion Coefficient measurements

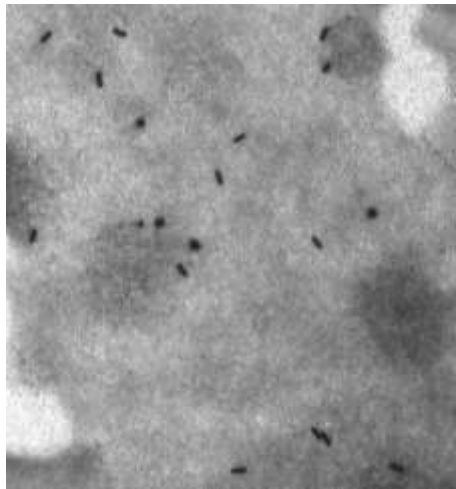
Experimentální výsledky měření nanotrubiček



Experimentální výsledky

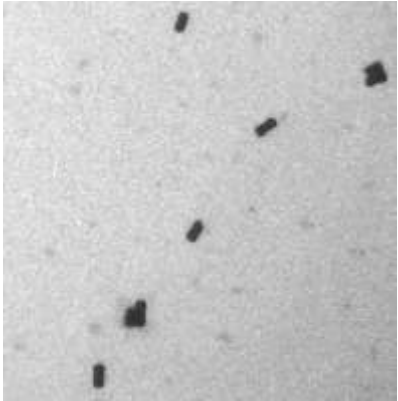


Parameter	D-DLS THETIS	TEM
L (nm)	70,7	66,4
L/w	5	4
w (nm)	14,2	16,5



Parameter	D-DLS THETIS	TEM
L (nm)	28,5	28,3
L/w	1,7	2
w (nm)	16,8	14,5

Experimentální výsledky



Parameter	D-DLS THETIS	TEM
L (nm)	46	47
L/w	1.4	1,8
w (nm)	34,8	26

THETIS SPECIFICATIONS

Particle size range	Particle size : 1 nm up to 1 μm
Sample concentration	0.0001% to 10% w/% (solvent dependent)
Temperature control range inside the cell	10°C to 70°C; +/-0,1°C (depending on cuvette cell material)
Sample cell	Cell : optical quality QS cylindrical ; 10 mm light path;
Sample volume	>400 μL
Sample type	Aqueous & organic solvents; pH: 1-14 (depending on cuvette cell material)
Anisotropic range	Anisotropic Ratio from 2 to 100

MEASUREMENT CONFIGURATION & PROCESSING

Measurement technology	Static Light Scattering (SLS), Depolarized Dynamic Light Scattering (DDLDS)
Polarization	VV - VH
Laser source	Highly reliable 50 mW diode @635
Measurement angles	All angles from 30° to 150°, step = < 0,1°
Data processing algorithm	Real time and Time resolved autocorrelation (TR -DLS)
Detector	High sensitivity Avalanche Photodiode (APD)

GENERAL

Computer interface	USB 2.0 – Windows 10 32 & 64 bits
Dimensions/ Weight	33 cm x 33 cm x 38 cm (HWD) / 17 kg
Power supply	100-115/220-240 VAC, 50/60 Hz, 100 W max



Our instruments

2007

VASCO

DLS for high concentration and absorbing samples



2013

WALLIS

Zeta potential



2017

VASCO KIN

In situ and software correlator



2019

AMERIGO

Standard cell, *In situ* Time resolved DLS & Zeta potential



2021

THETIS

Continuous Multi angle Time resolved DLS, SLS and DDLS

