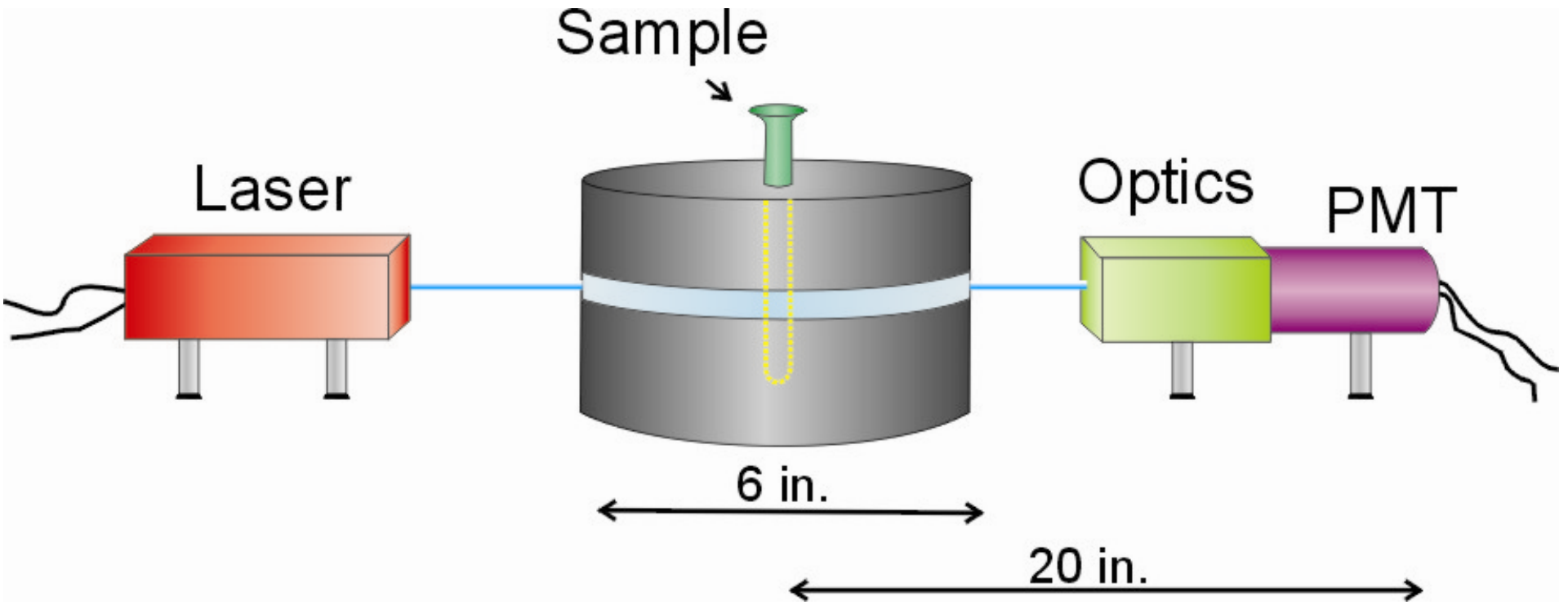
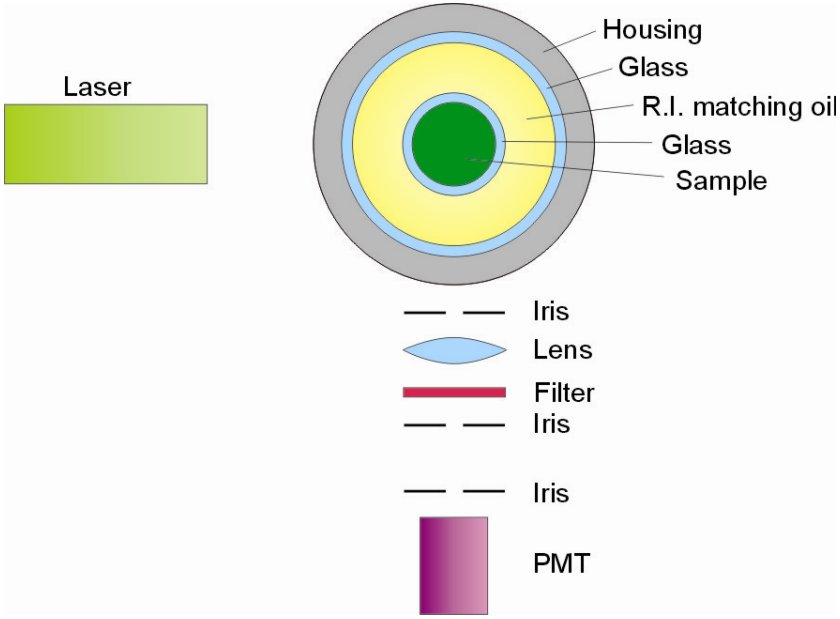
Microfluidic Dynamic Light Scattering

Tom Chastek

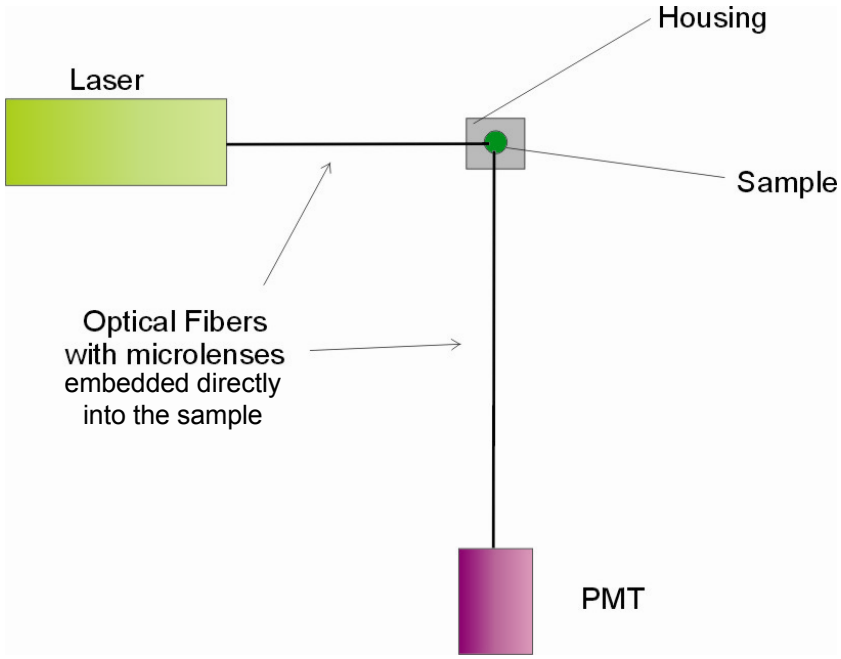
Traditional dynamic light scattering instrument

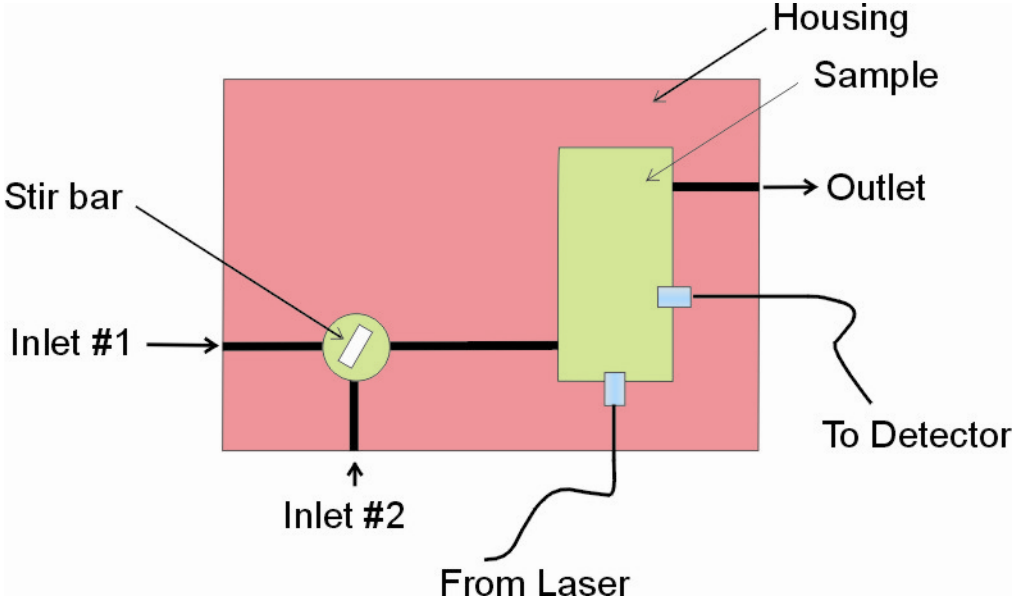


Traditional DLS instrument – TOP VIEW



Microfluidic DLS instrument





Dimensions

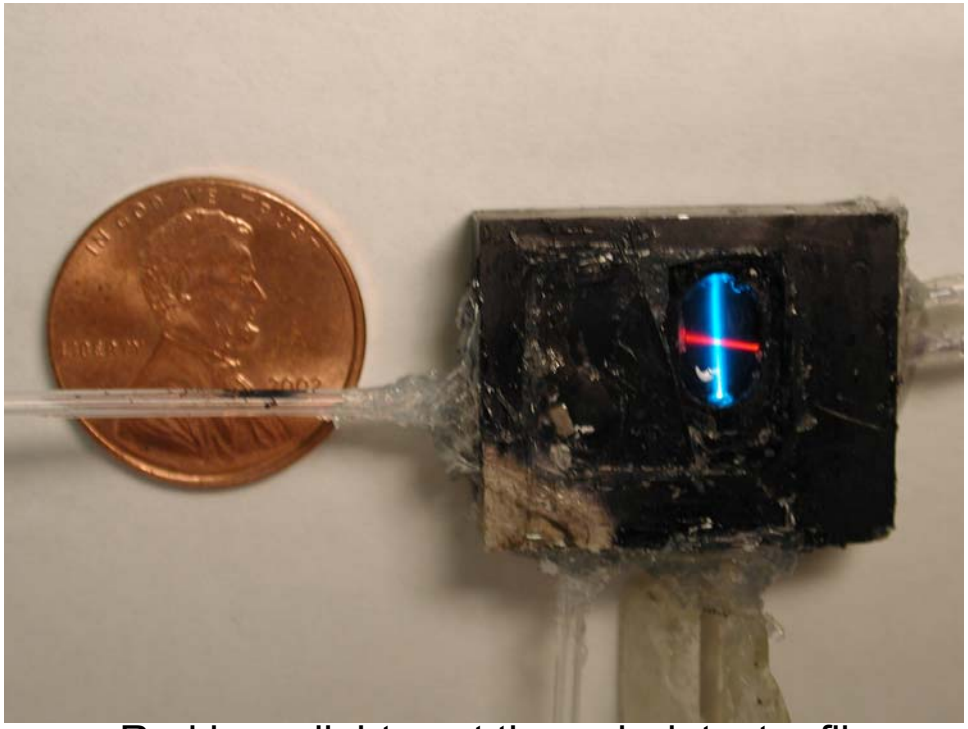
Overall: 20 x 25 x 7 mm

Detecting cavity: 7 x 5 x 2 mm, 70 μL

Mixing cavity: 5 mm diameter x 2 mm = 40 μL

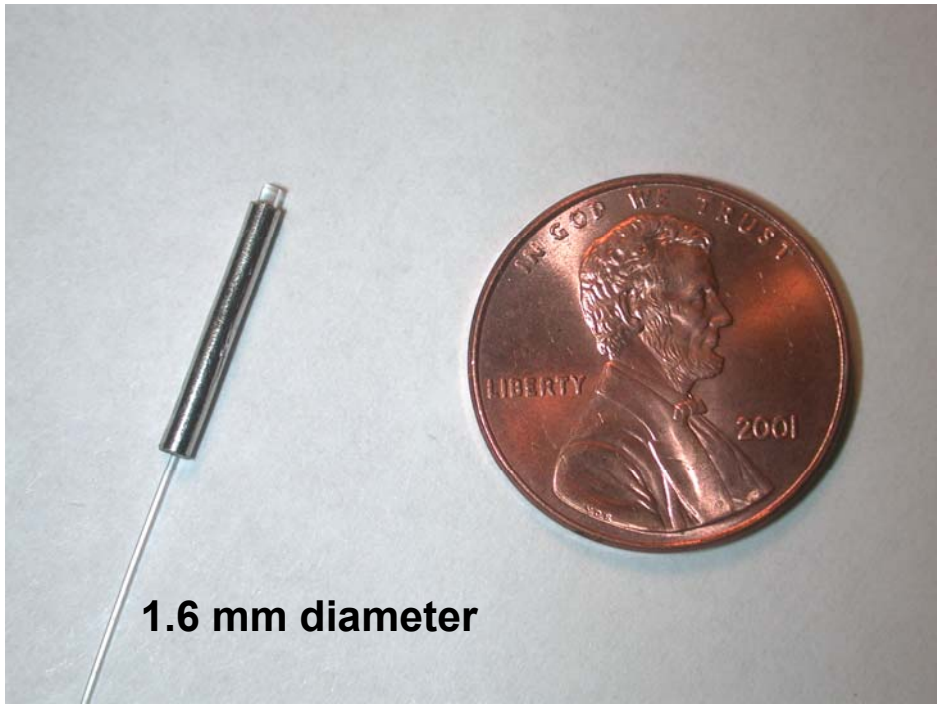
Microlens: 1 mm diameter, 3.1 mm long

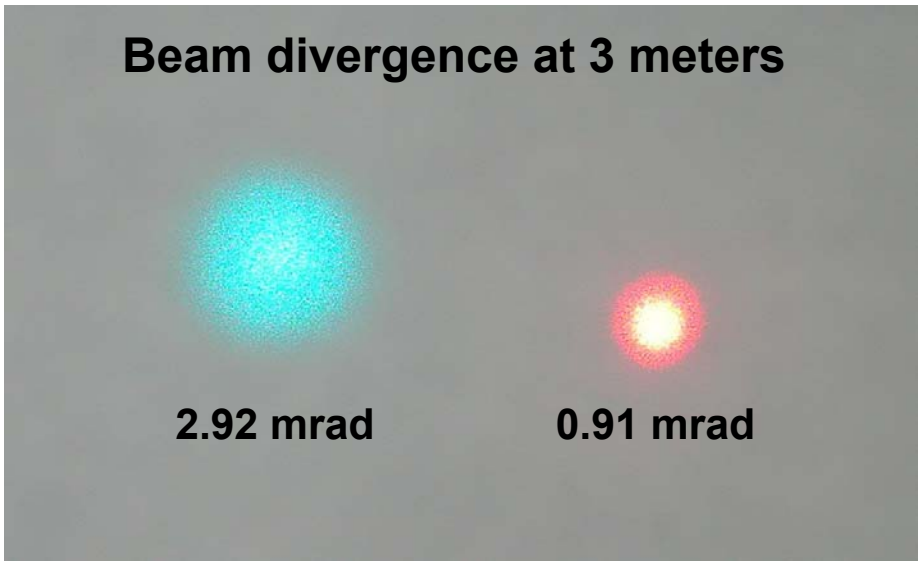
Fiber optic: overall – 254 μm
active area - ~5 μm



Red laser light sent through detector fiber

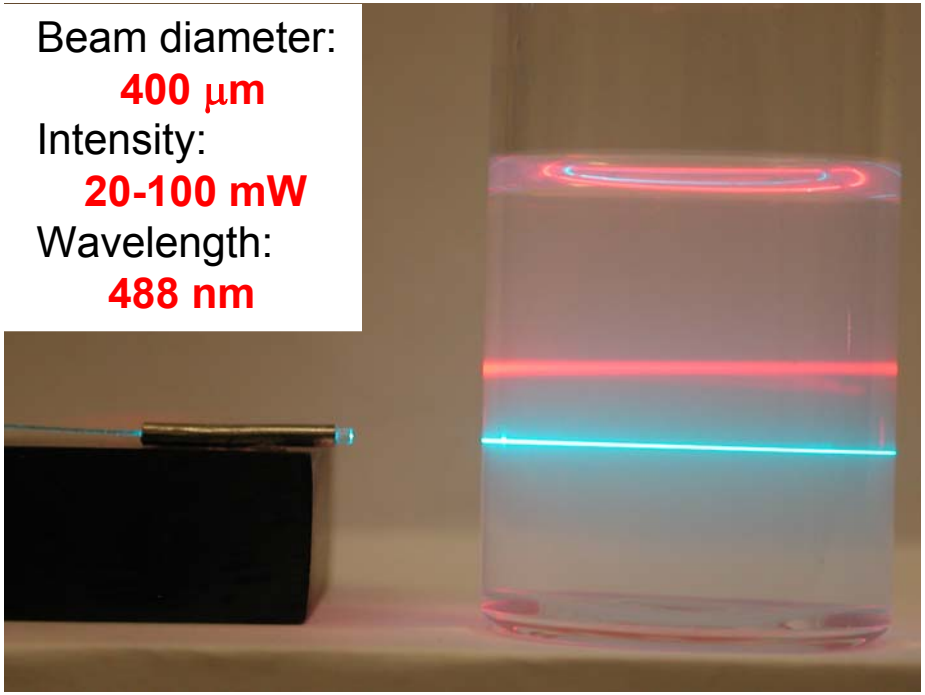
Fiber optic probe with microlens





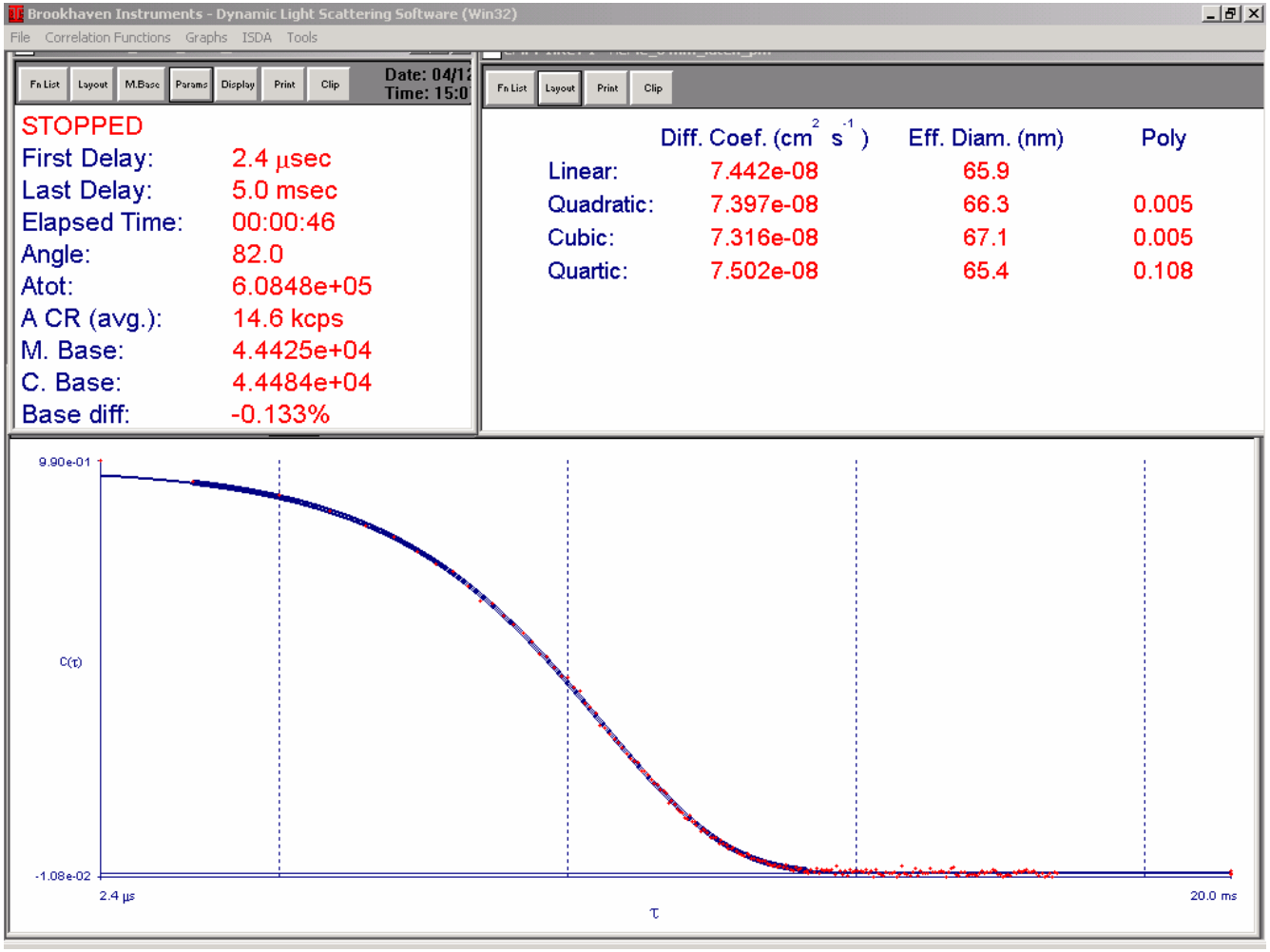
Red helium-neon laser used as reference

Beam diameter:
400 μm
Intensity:
20-100 mW
Wavelength:
488 nm

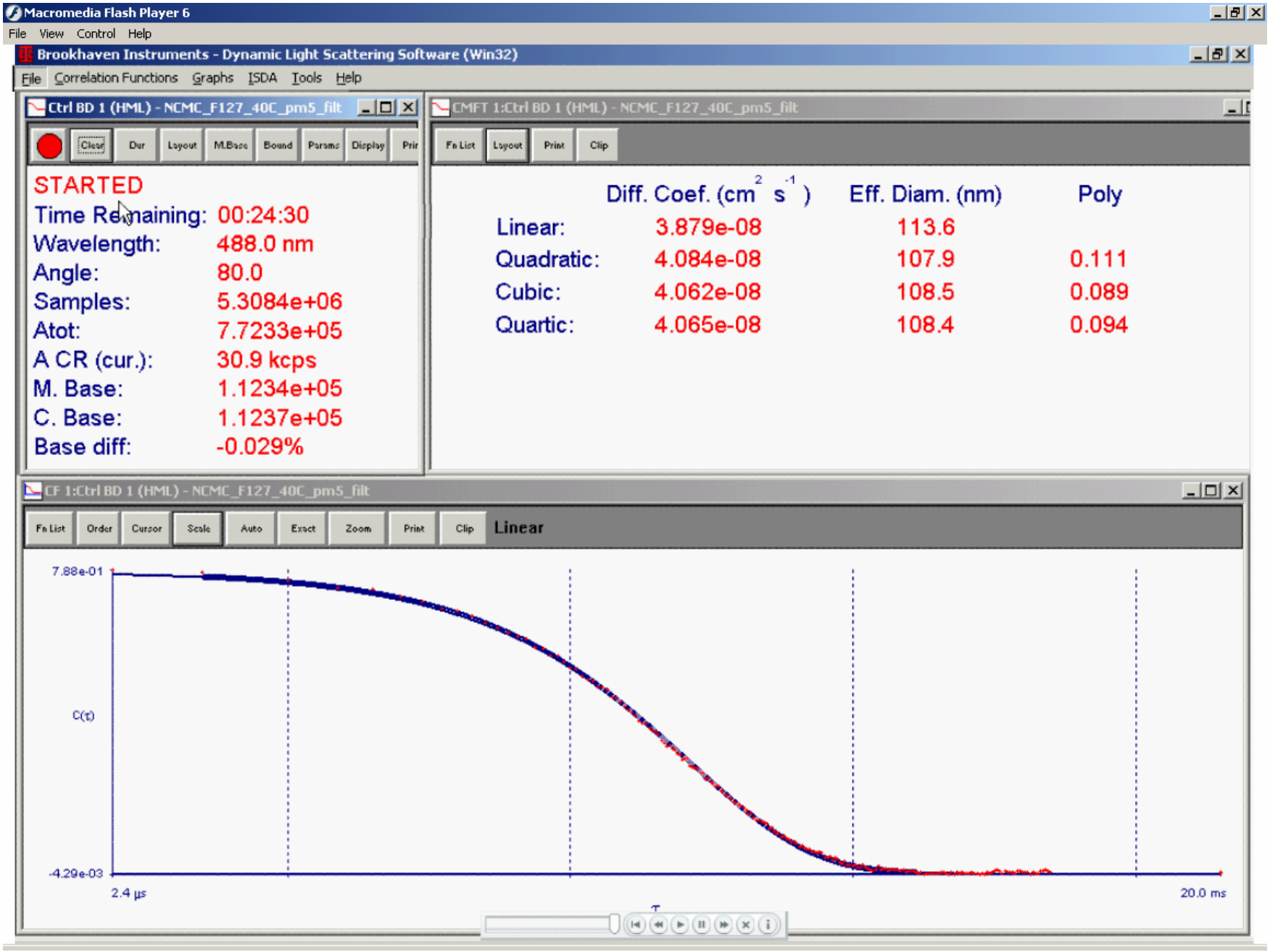


Particle sizing of aqueous polystyrene latex solutions

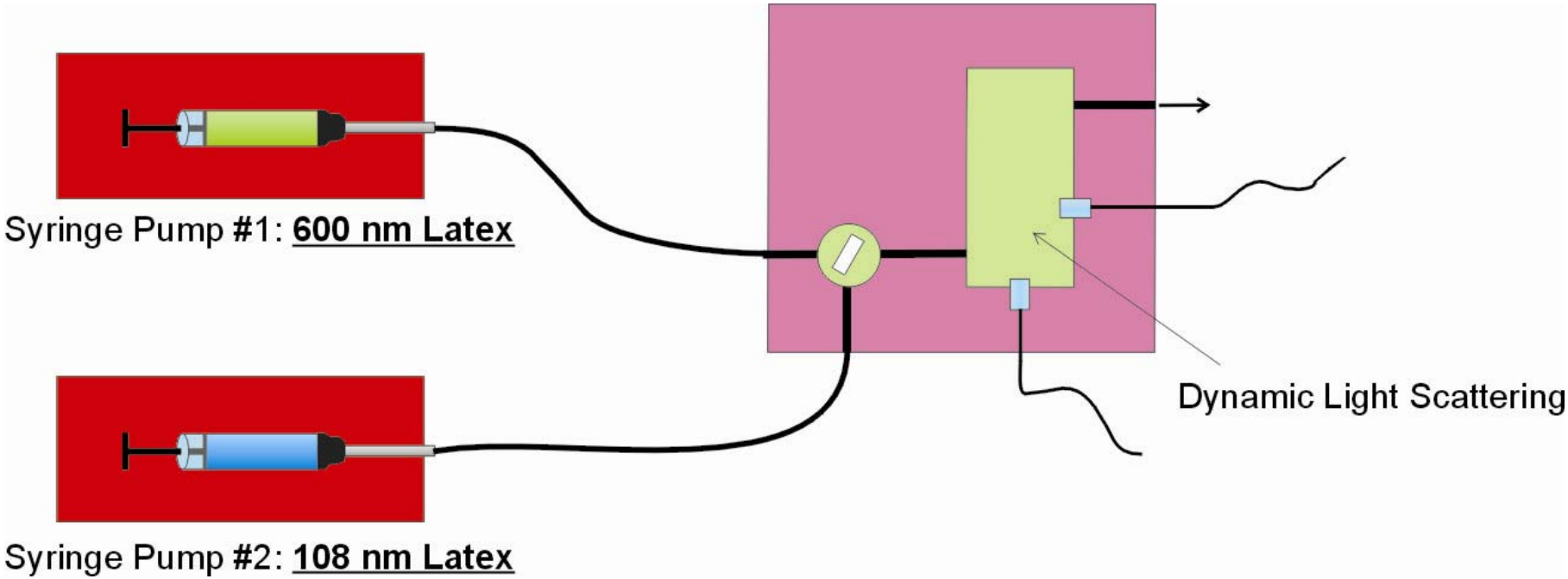
Manufacturer's size		Measured size
64 nm latex	=	65 nm, poly 0.069
108 nm latex	=	103 nm, poly 0.117
600 nm latex	=	569 nm, poly 0.007



Example measurement of a 108 nm latex solution



Combined: particle sizing and sample flow

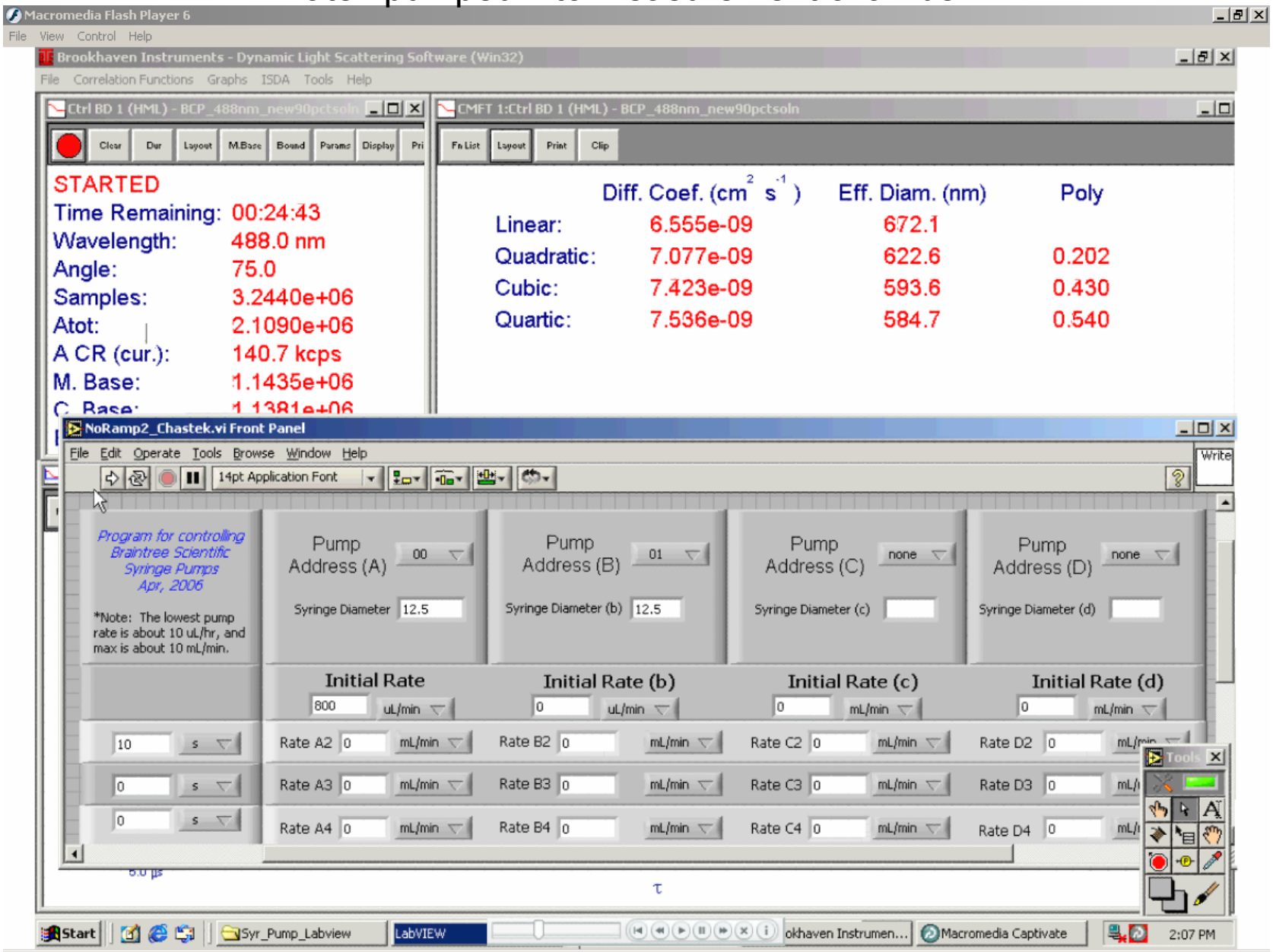


Overview of what will be demonstrated:

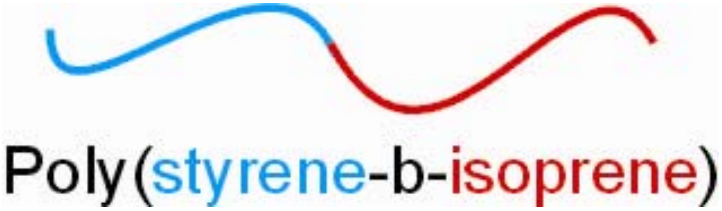
- The measurement chamber is initially filled with 600 nm latex solution
- A syringe pump is used to flow 108 nm solution into the chamber
- 135 μL are flowed over a 10 s period

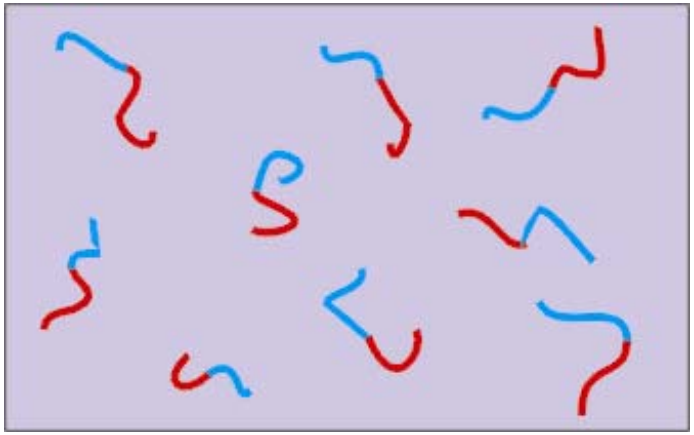
These results will demonstrate the ability of this instrument to perform high throughput nanoparticle size measurements

Initially 600 nm latex, until 135 μL of 108 nm latex pumped into measurement chamber

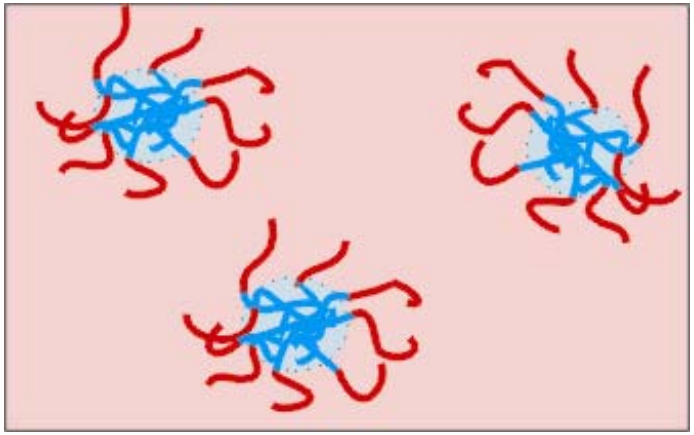


Altering solvent composition to dissolve block copolymer micelles



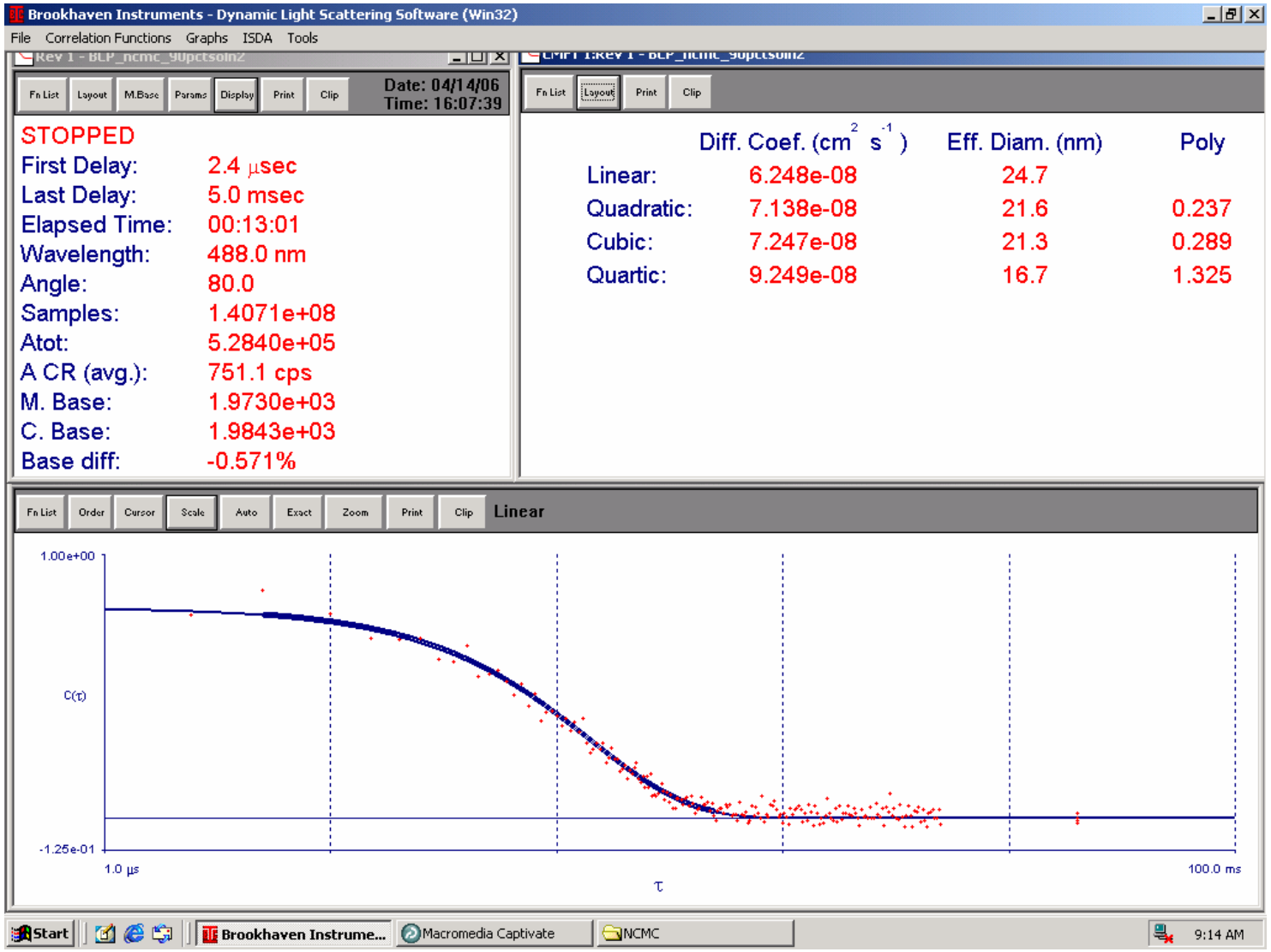


Unimer state in neutral solvent (e.g., toluene)



Micelles form in selective solvents (e.g., hexadecane)

- polyisoprene corona
  polystyrene core

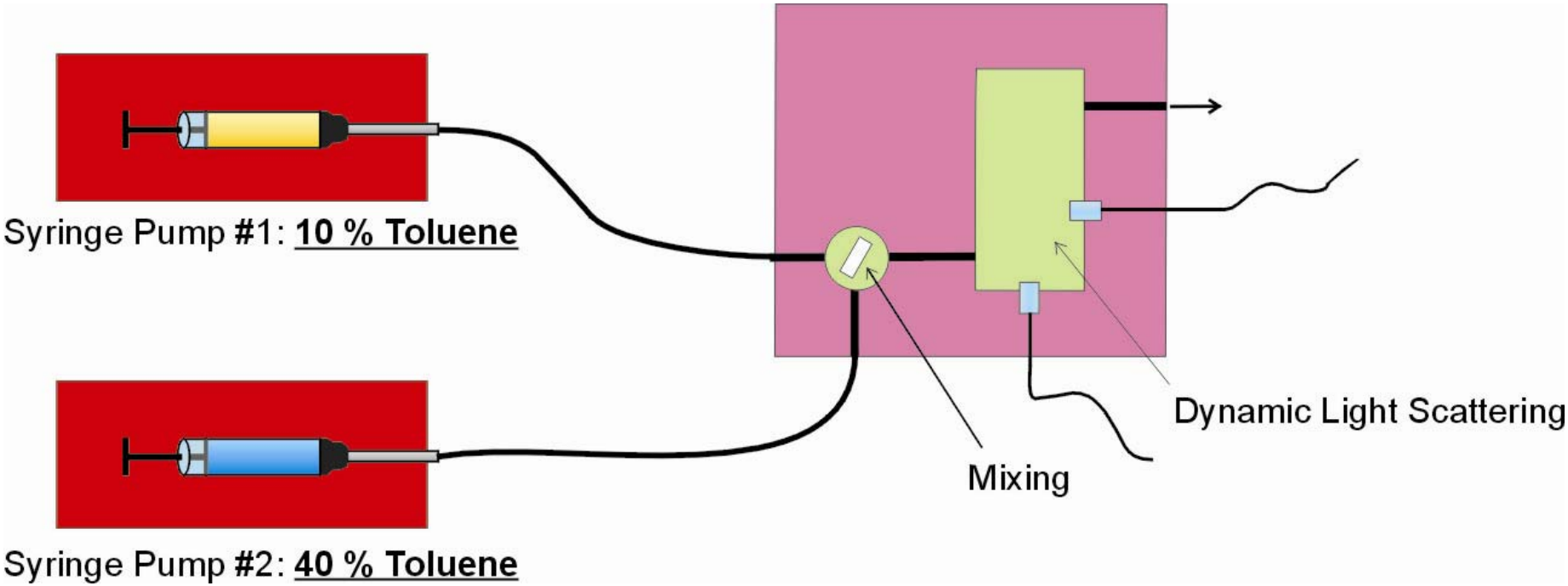


PS-b-PI, symmetric, 20 kg/mol

2% polymer, 10% toluene, 88% hexadecane

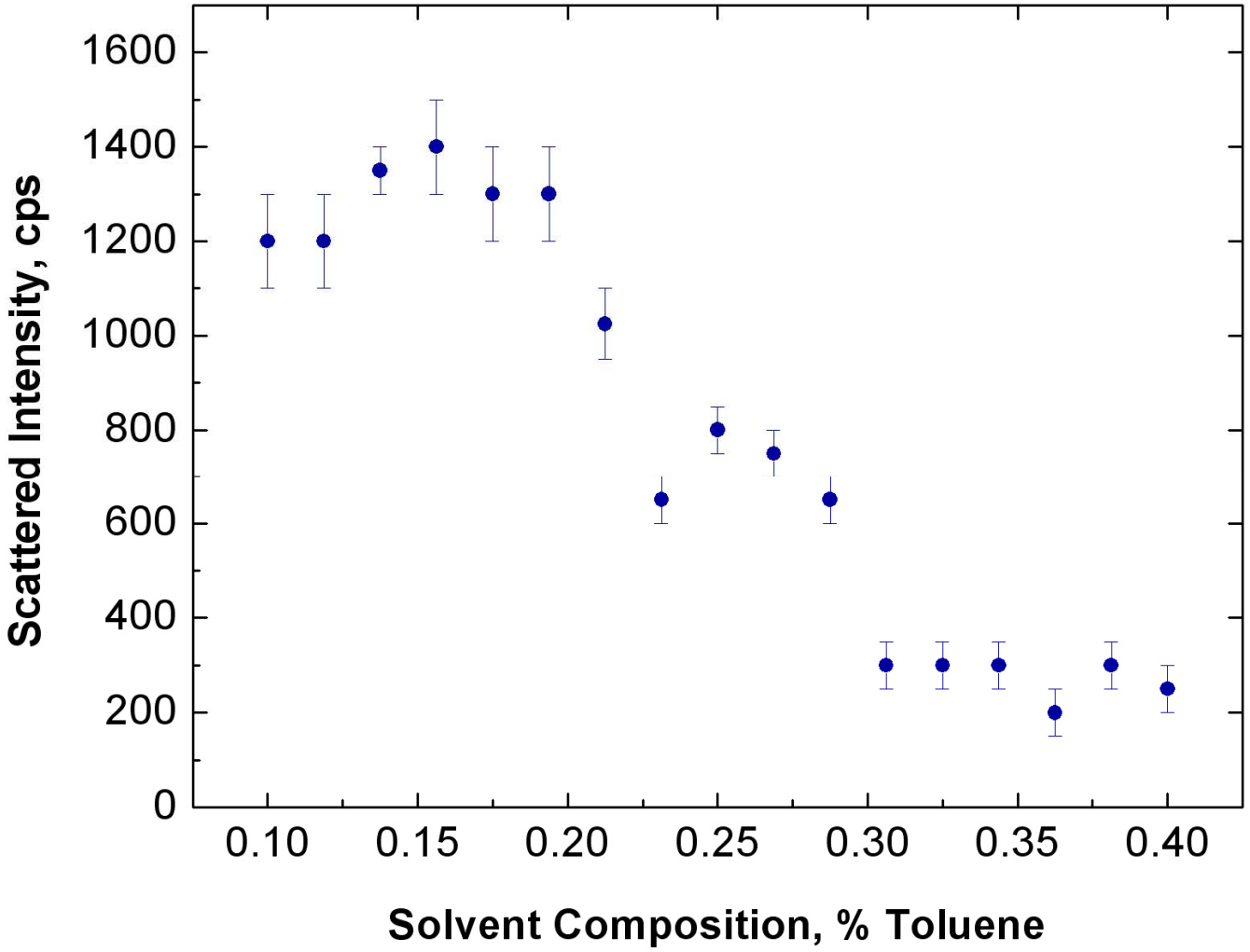
~ 21 nm micelles form

Experimental setup for flowing block copolymer solutions



Measurements are made by flowing each solution at **0-80 μL/min** for **2 min**, giving a volume of **160 μL**

e.g., when both are flowed at 40 μL/min, the blended sample will have 25% toluene

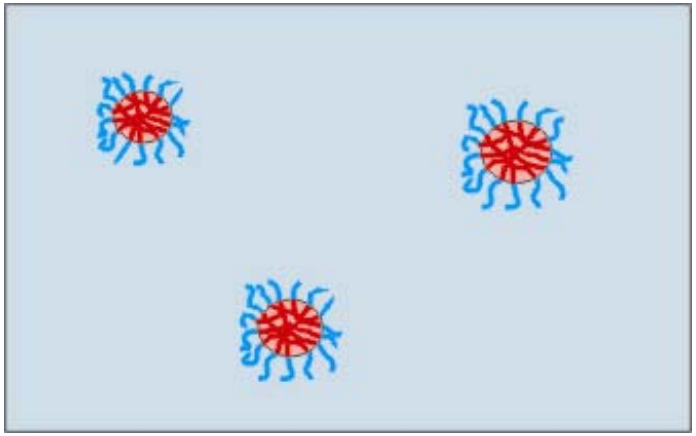


At 22 °C, PS-b-PI micelles dissociate in hexadecane/toluene if the solvent is composed of **22-28% toluene**.

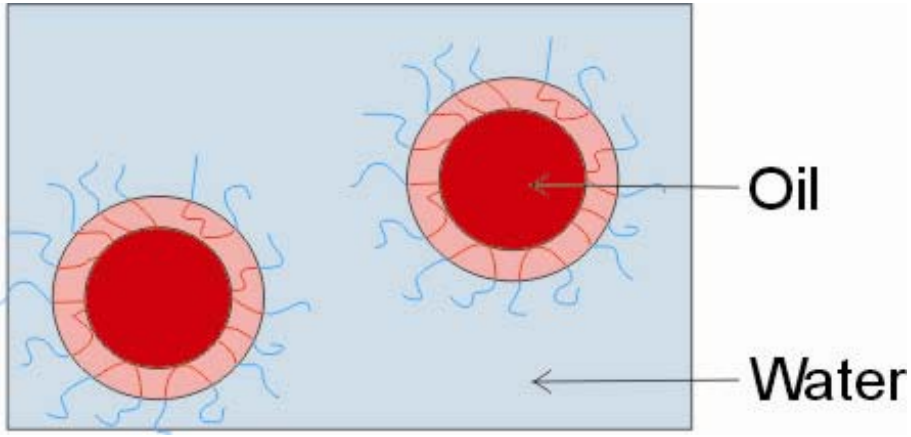
- Each data point required only 160 μL of solution

Block copolymer – Oil – Water equilibration

Poly(ethylene oxide-b-propylene oxide-b-ethylene oxide)



In pure water, pluronic polymers form micelles (at elevated temperature)

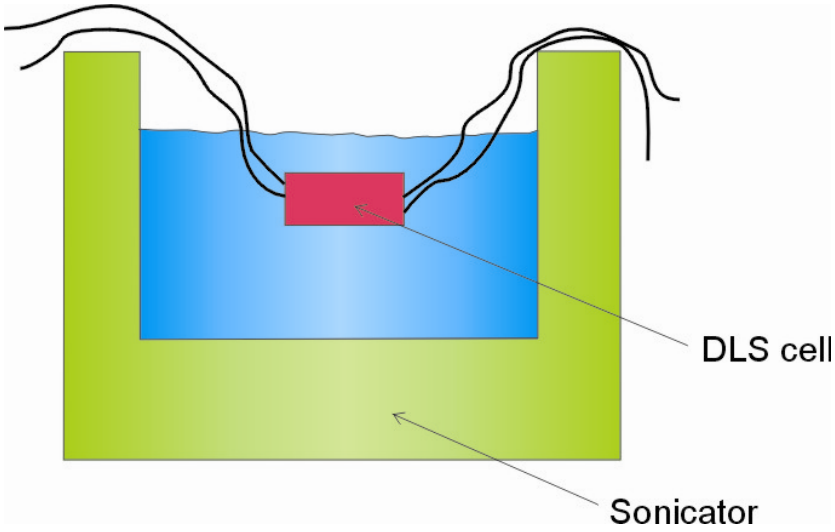


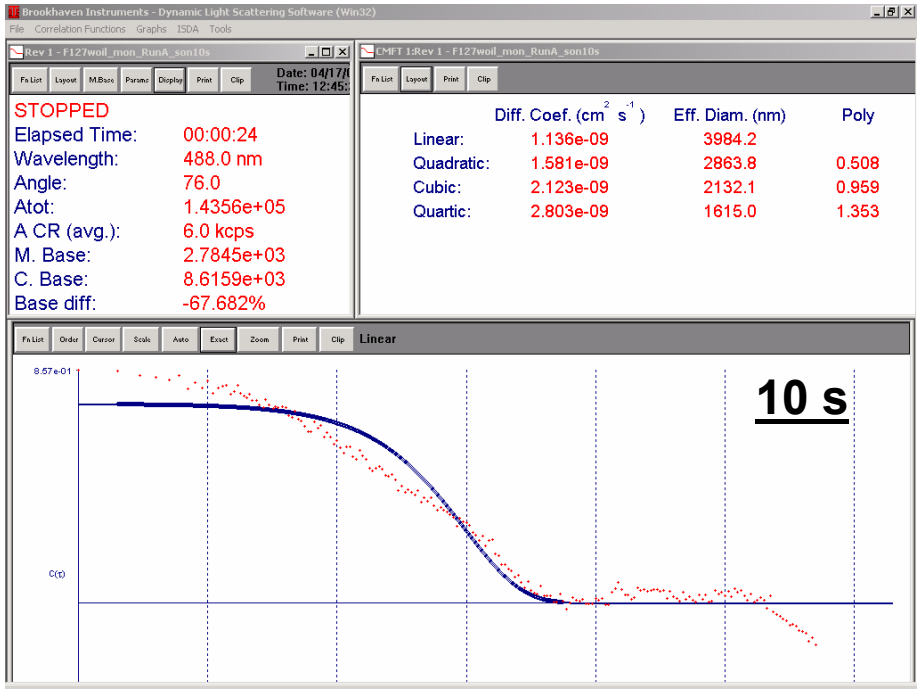
Added oil forms an emulsion
2% polymer, 1% oil, 97% water

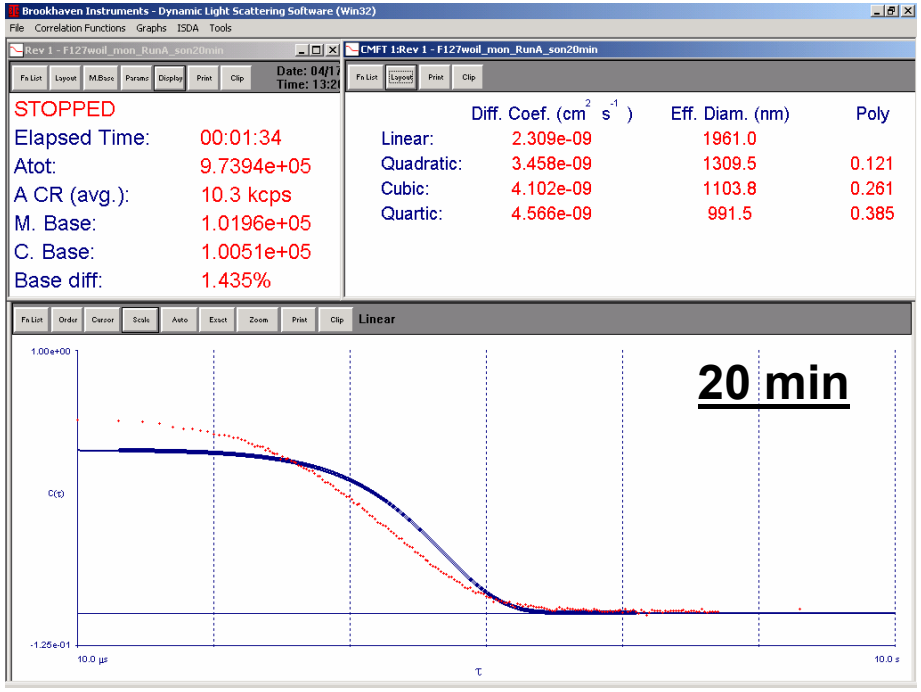
The emulsion particle size is several hundred nanometers, but equilibrating the solution requires appropriate mixing

How to equilibrate – Sonication? **No.**

DLS cell was submerged under water in a sonicator to allow for *in situ* particle sizing.





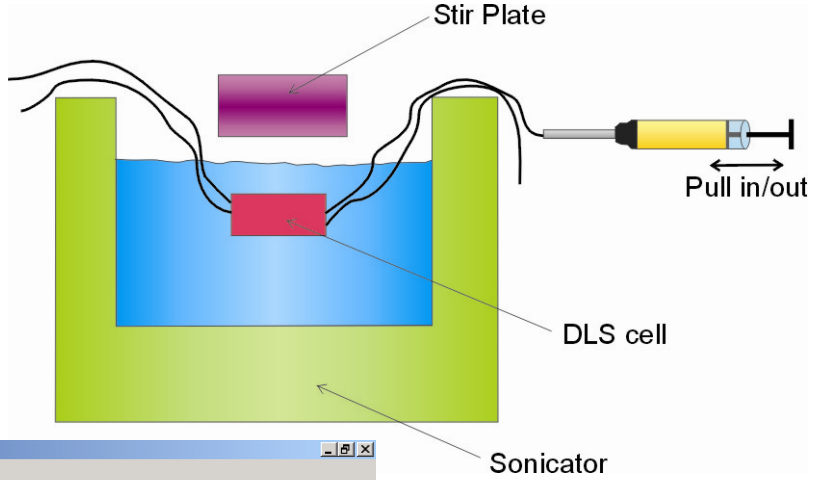


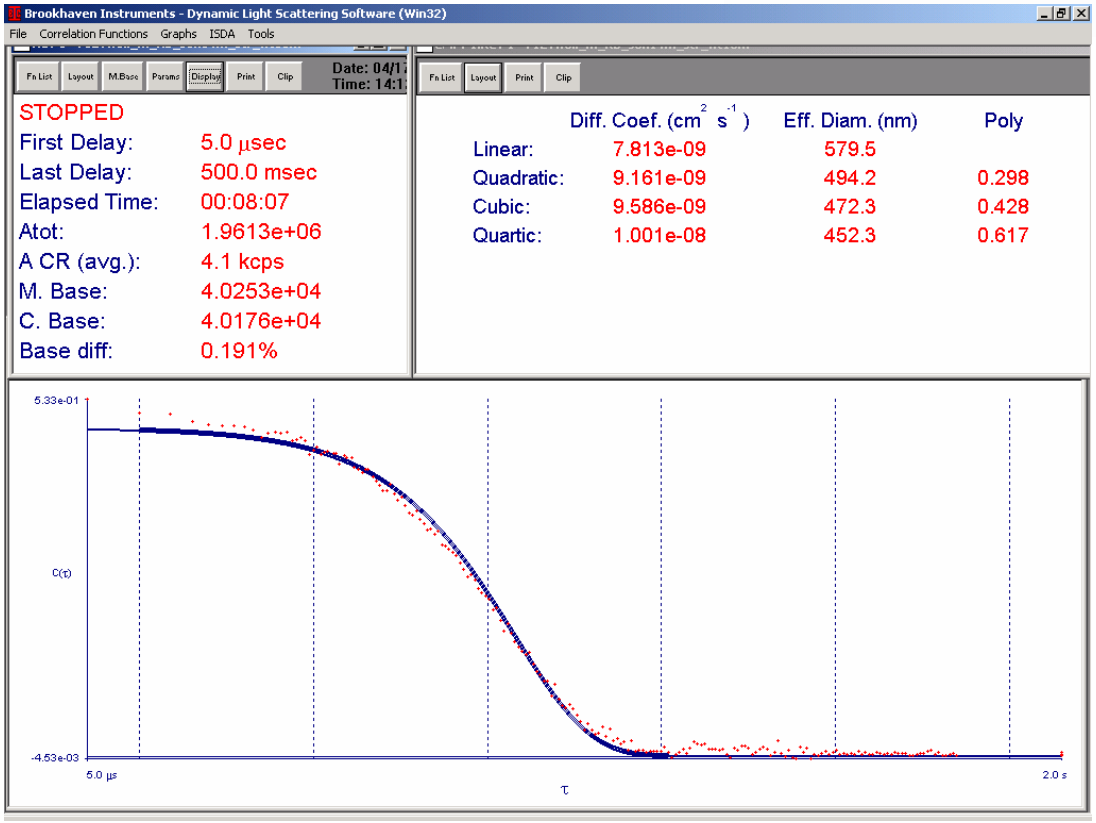
Particle size is broadly distributed even after 20 min of sonication

How to equilibrate – Sonication & Mixing? **Yes.**

Sonicating & mixing with a stir bar

- The sample is repeatedly pulled/pushed through the mixing chamber with a syringe
- 14 min of sonication and mixing gives a uniform particle size of ca. 500 μm





Summary

Fully operational microfluidic dynamic light scattering instrument has been made

- Demonstrated quantitative particle sizing from 20-600 nm on timescales as short as 5 s in both organic and aqueous solutions
- Demonstrated particle sizing of a 135 μL aliquot of sample flowed into the measurement chamber
- Demonstrated solution blending to determine the affect of solvent composition on micelle formation
- Demonstrated *in situ* sonication and mixing as a means to equilibrate an oil-water-block copolymer emulsion

Future improvements

- Accurate temperature control
- Reduction in volume
- Multi-angle measurements

Demo in room B223