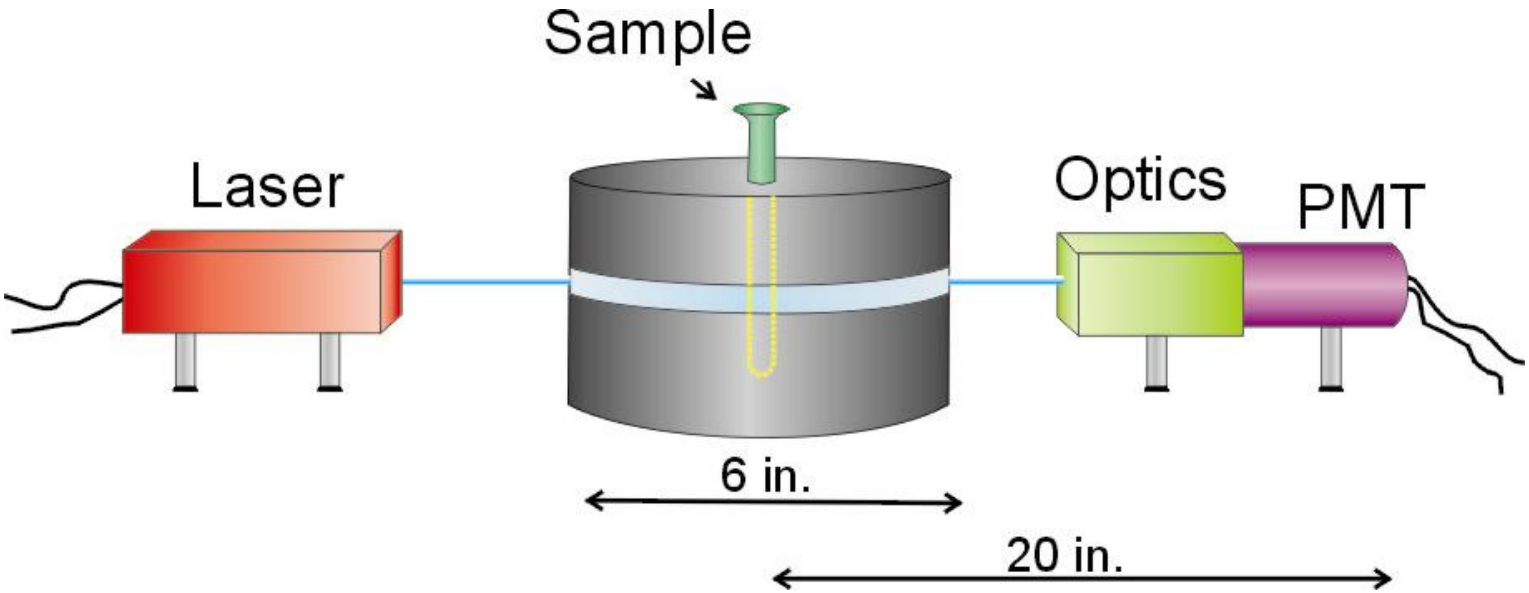
Miniaturized Dynamic Light Scattering for High Throughput Measurements

Tom Chastek, Kathryn Beers, Eric Amis

National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

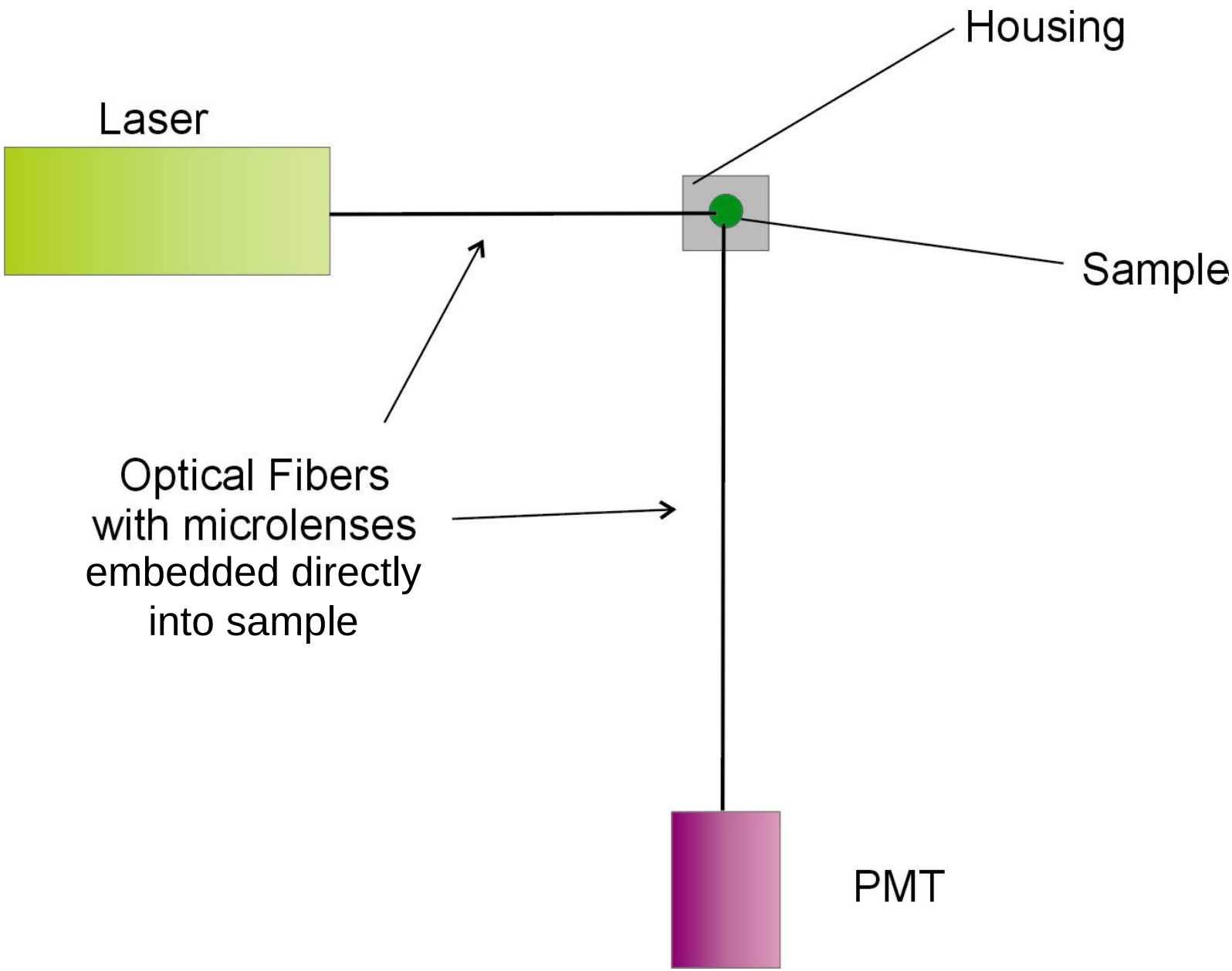
Polymers Division



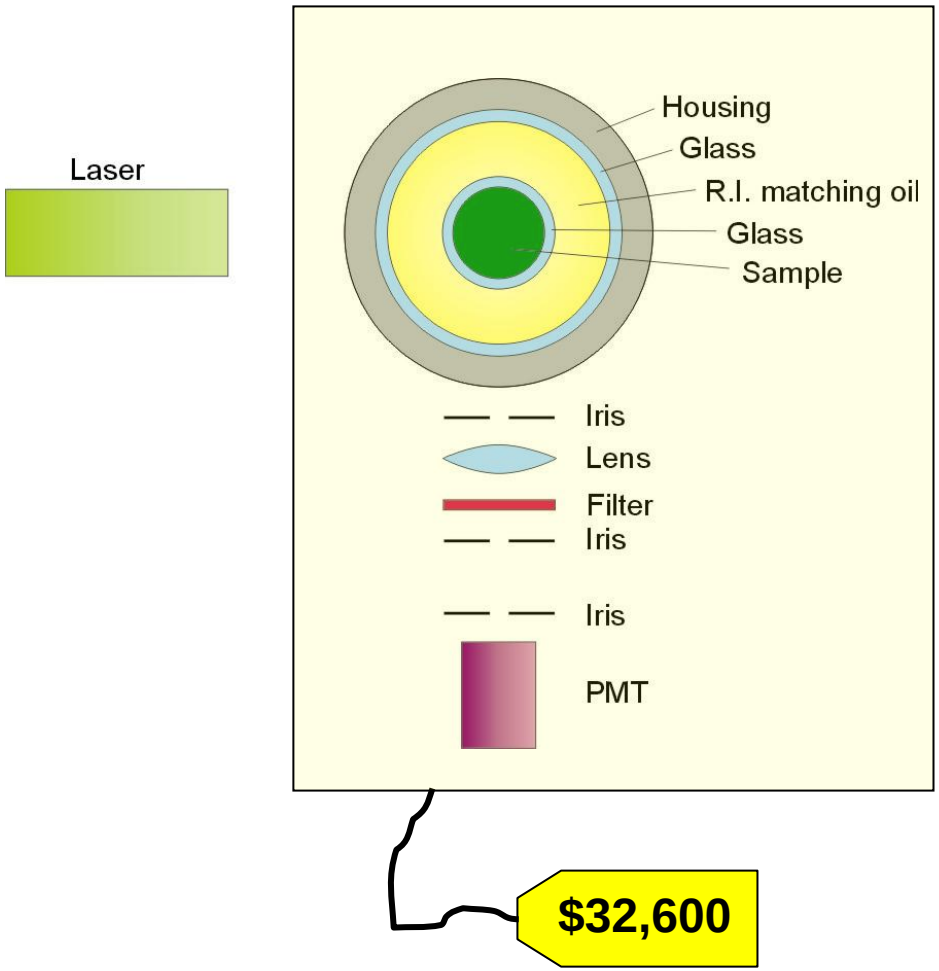
Traditional dynamic light scattering instruments

Principle: Measure the time-dependence of the scattered intensity
: Look at various timescales (1 μs to 1 s), and the scattered intensity will be correlated at time scales shorter than the dynamics

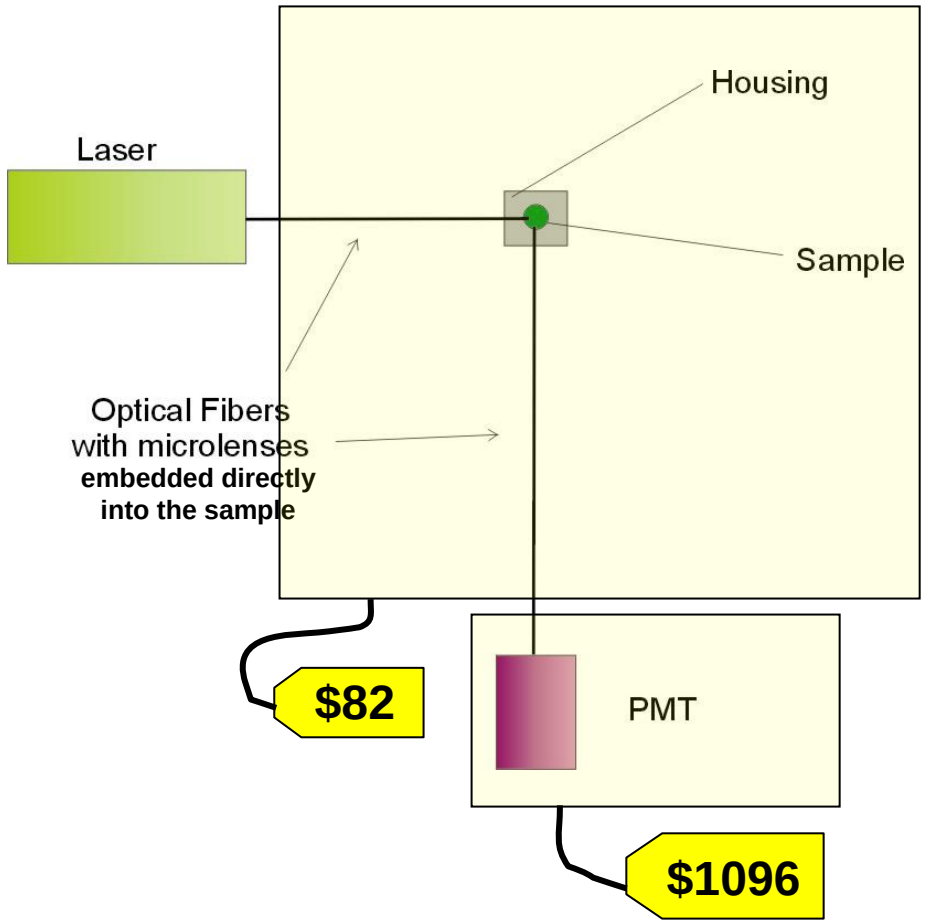
Use: To determine the diffusion coefficients of polymers or nanoparticles in solution
: The size of the particles can typically be determined from the diffusion coefficient

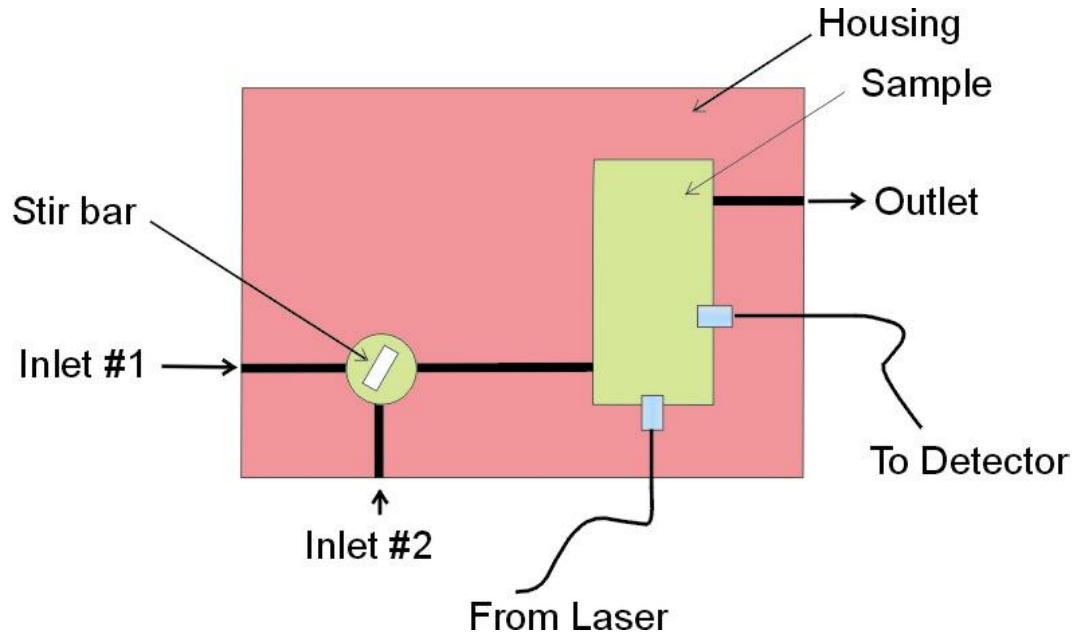

Housing
Laser
Sample
Optical Fibers
with microlenses
embedded directly
into sample
PMT

Traditional DLS instrument

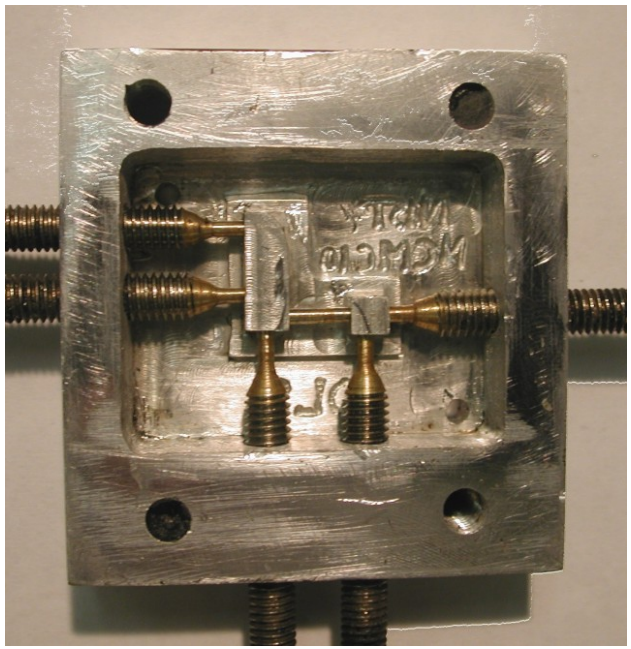


Miniaturized DLS instrument

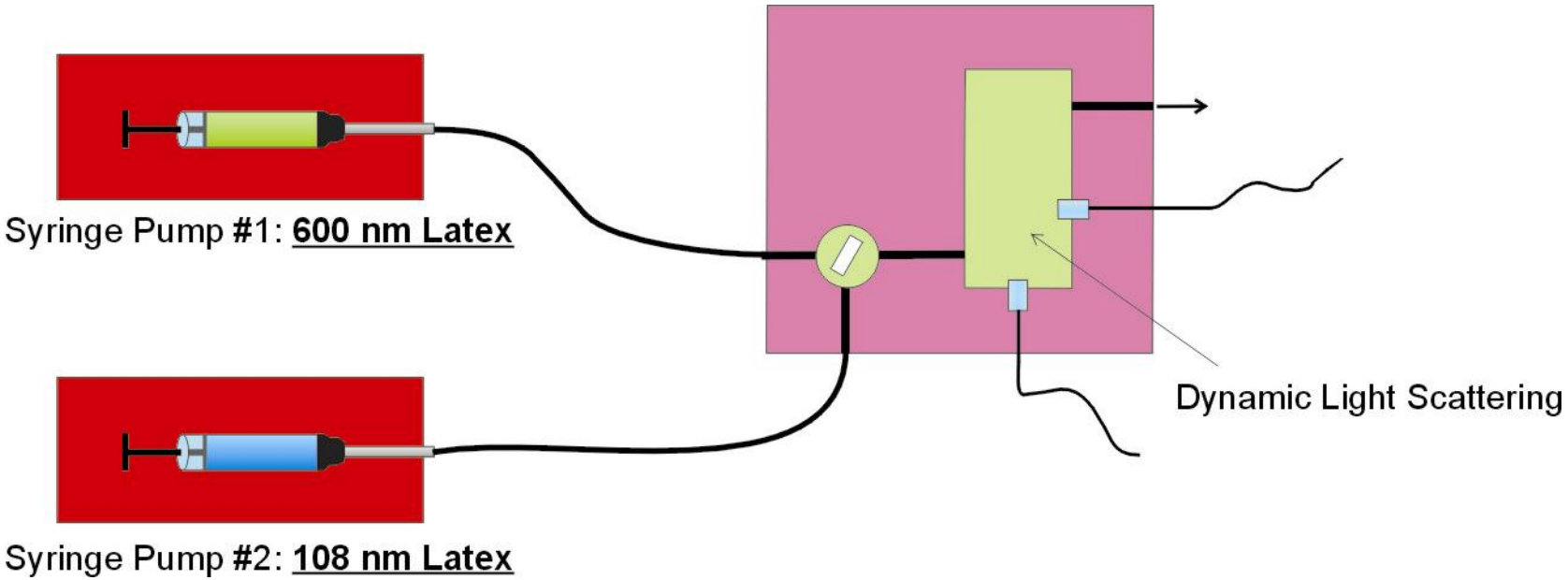



Housing
Sample
Outlet
Stir bar
Inlet #1
To Detector
Inlet #2
From Laser


NIST
NCMC10
DLS



Demonstration of DLS measurements



Overview of what will be demonstrated:

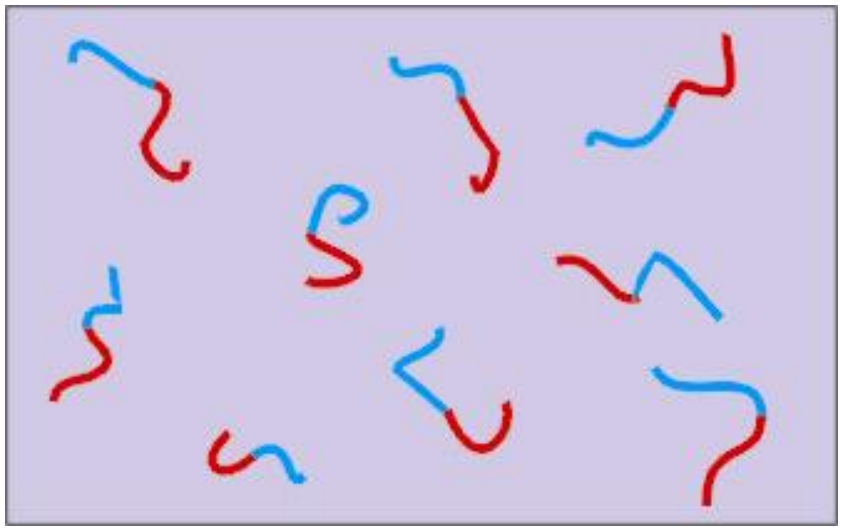
- The measurement chamber is initially filled with 600 nm latex solution
- A syringe pump is used to flow 108 nm solution into the chamber
- 135 μL are flowed over a 10 s period

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

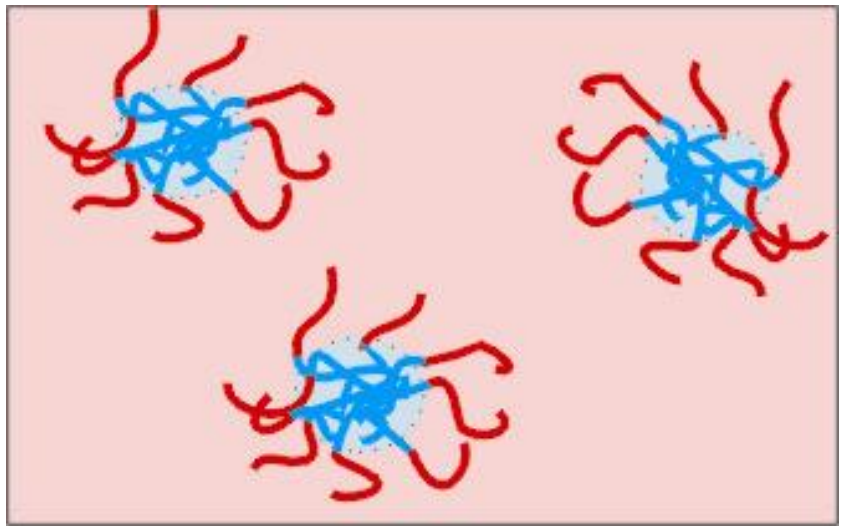
Initially 600 nm latex, until 135 μL of 108 nm latex pumped into measurement chamber

Altering solvent composition to dissolve block copolymer micelles

Poly(styrene-b-isoprene)



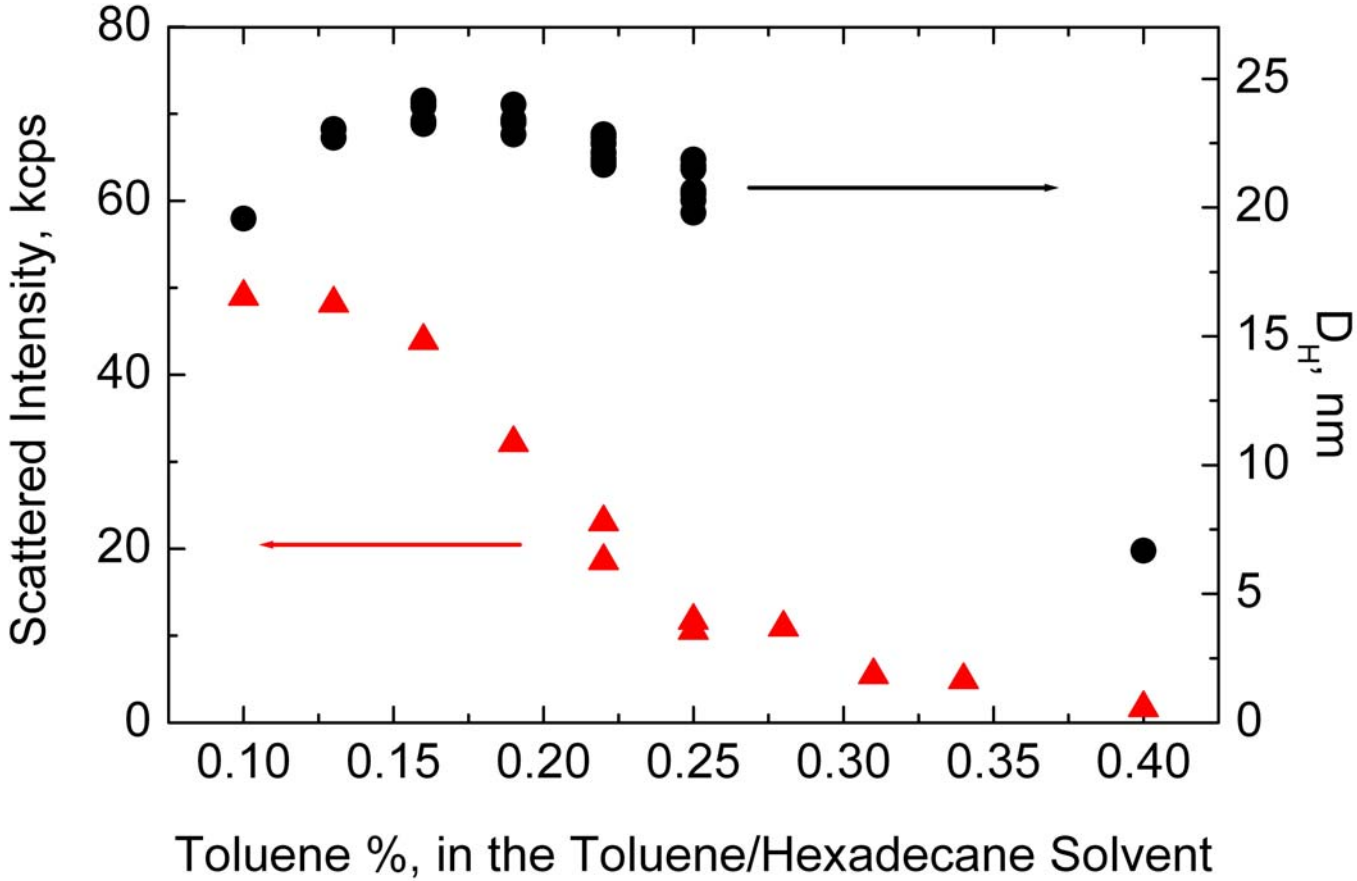
Unimer state in neutral solvent (e.g., toluene)



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

- polyisoprene corona
  polystyrene core

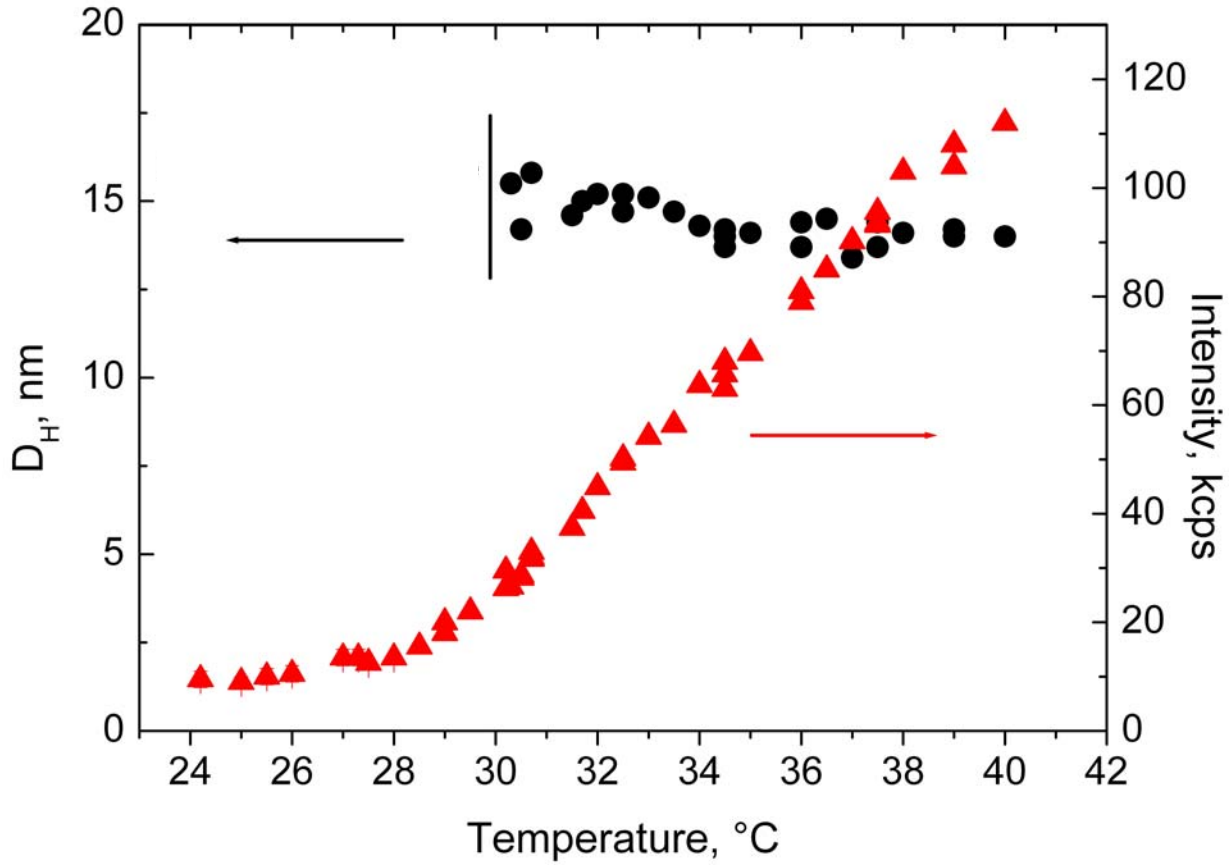
DLS data from PS-b-PI in mixed hexadecane/toluene solvent



Result :At 25 °C, PS-b-PI micelles dissociate in hexadecane/toluene if the solvent is composed of **25% toluene**.

: Each data point required only 180 μL of solution

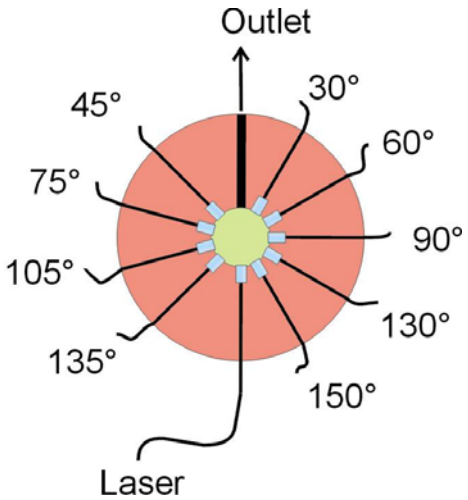
Temperature scan of an aqueous block copolymer solution

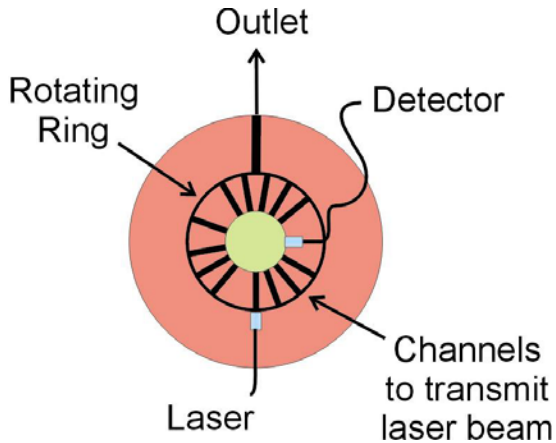


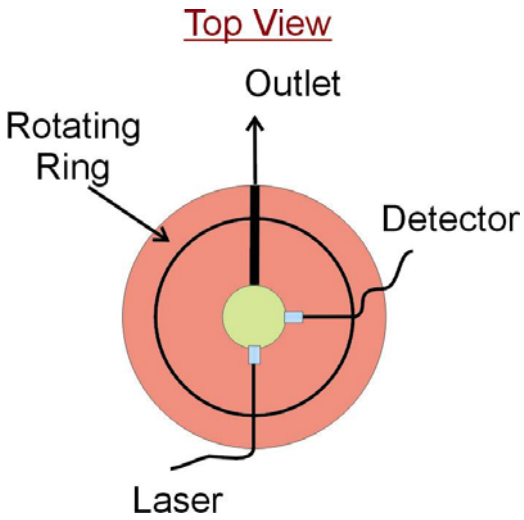
Result: The critical micelle temperature (CMT) of Pluronic P85 (2% in water) is 29-30 °C

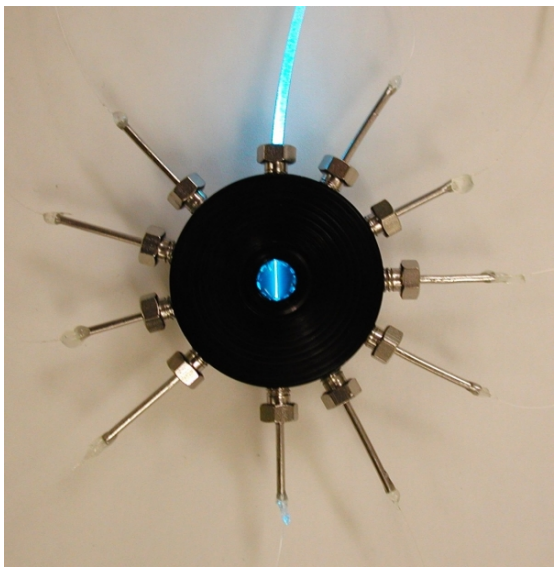
Multi-angle instruments

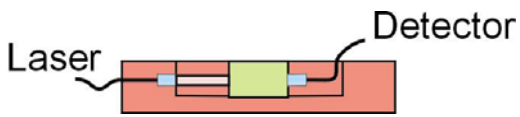
Several stationary detectors or Single rotating detector probe

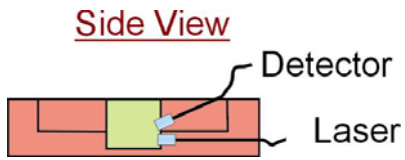




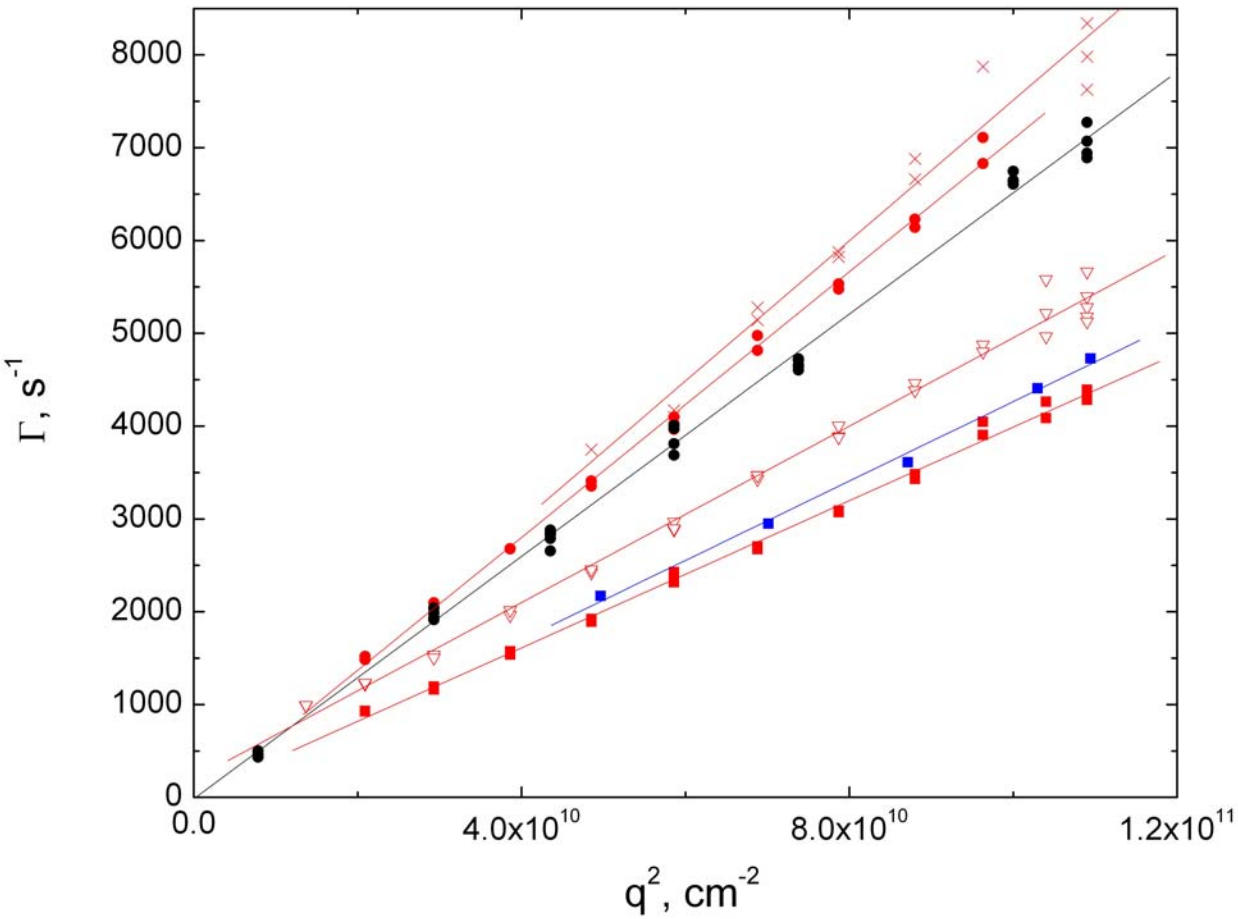








Data from multiangle instruments



- 107 nm latex, inline rotation
  D_m = 4.28 x 10^{-8} cm^2/s
  D_H = 105 nm
- 64 nm latex, fixed angles
  D_m = 6.51 x 10^{-8} cm^2/s
  D_H = 69.1 nm
- 107 nm latex, out of plane rotaton
  D_m = 3.96 x 10^{-8} cm^2/s
  D_H = 114
- 64 nm latex, out of plane rotation
  D_m = 7.16 x 10^{-8} cm^2/s
  D_H = 62.9 nm
- SB(5-5), out of plane rotation
  D_m = 7.51 x 10^{-8} cm^2/s
  D_H = 19 nm
- SB(9-9), out of plane rotation
  D_m = 4.75 x 10^{-8} cm^2/s
  D_H = 30 nm

Summary

A new miniature dynamic light scattering instrument was presented

- Accurate particle sizing from 10-600 nm on timescales as short as 5 s in both organic and aqueous solutions
- The flow-through design allows for high throughput measurement of sample aliquots of approximately 100 μL
- The critical solvent composition for the dissolution of poly(styrene-b-isoprene) micelles was measured by high throughput measurement of solutions blended together
- Determined a CMT for an aqueous block copolymer solution
- Demonstrated instruments for continuing this work with multiangle DLS measurements