Microfluidic Dynamic Light Scattering

Motivation/Objective

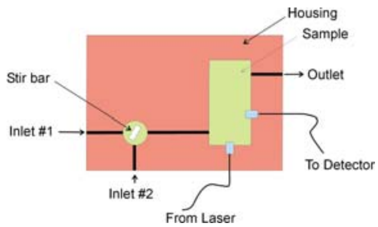
Motivation: Industrial formulations such as coatings, paints, personal care products, food and cosmetics increasingly incorporate nanostructures such as micelles and particles to enhance performance. To more rapidly discover and optimize these innovative products, industry requires high throughput measurements of fluid nanoparticle size and structure.

Goal: To develop and demonstrate microfluidic devices for conducting dynamic light scattering (DLS) measurements. This new platform will enable high throughput measurements of small volumes of nanostructured and nano-particle laden fluids. An important aspect of this work is to integrate DLS with other microfluidic tools, such as synthesis and fluorescence spectroscopy, so that the resulting device is widely useful in addressing measurement needs related to formulations.

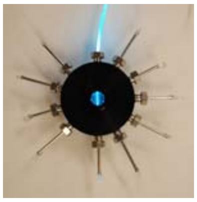
NIST Role/Approach

We developed new dynamic light scattering (DLS) instrumentation

The measurements are conducted using small fiber optic probes. Analysis volumes can be significantly reduced, well below 100 μL without compromising data quality.

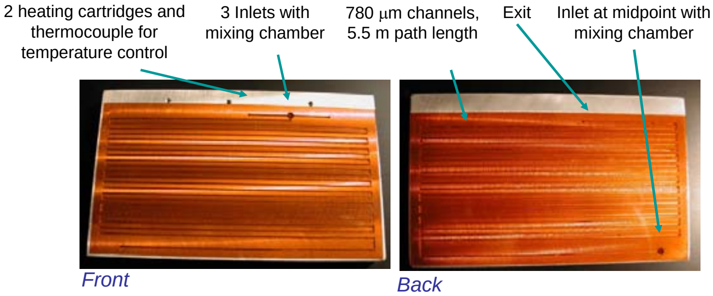


Schematic of an example DLS Device



Multiangle DLS

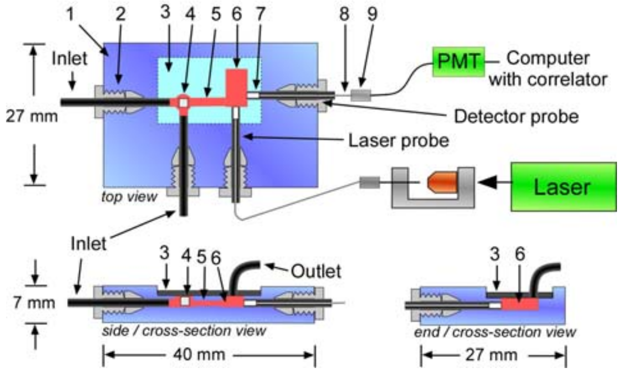
We developed a new microfluidic framework with several advantages



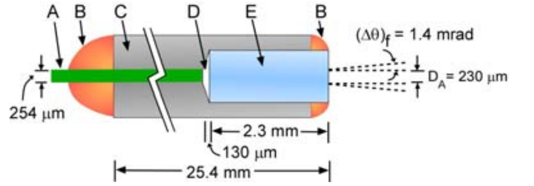
- These devices are compatible with the dynamic light scattering
- No leaking occurs at elevated temperature or with organic solvents

Highlights

Instrumental Details of High Throughput DLS Instrument



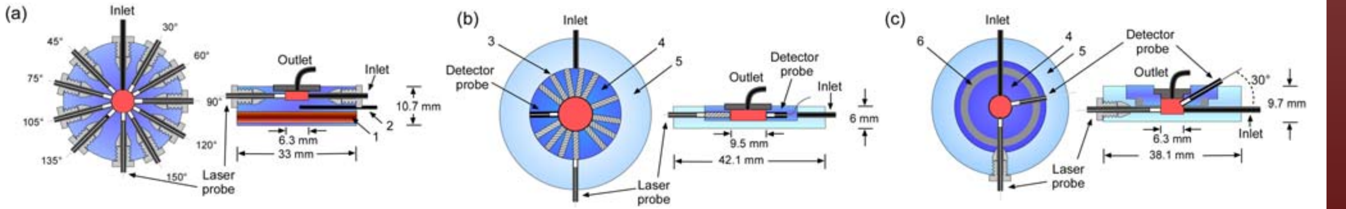
Fiber Optic Probe



On left, a typical device for high throughput measurements is depicted to scale. Multiple stock solutions can be mixed immediately prior to entering the measurement chamber. A key component is the fiber optic probe, depicted above. The small size of the probe allows for the measurement volume to be considerably reduced.

Labeled details are, (1) aluminum housing, (2) HPLC fittings, (3) cover, (4) mixing chamber with magnetic stir bar, (5) internal channel, (6) measurement chamber, (7) GRIN microlens, (8) optical fiber, (9) fiber couplers, (A) fiber optic (**B**) epoxy (C) stainless steel tube (O.D. = 1.6 mm, I.D. = 254 mm), (D) air gap (E) GRIN microlens

Multi-angle DLS Instruments



This general design of DLS devices is amenable to multiangle measurements. Devices with multiple stationary detectors and single rotating detectors show the versitility of this design. Labeled details are, (1) heating cartridge, (2) thermocouple, (3) 1 of 13 open channels to allow the laser light to reach the sample as the detector probe is rotated, (4) rotating ring that contains the detector probe (5) stationary cup-shaped base with laser probe, (6) Teflon o-ring.

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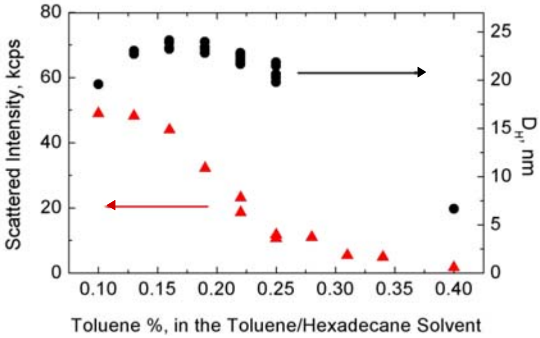
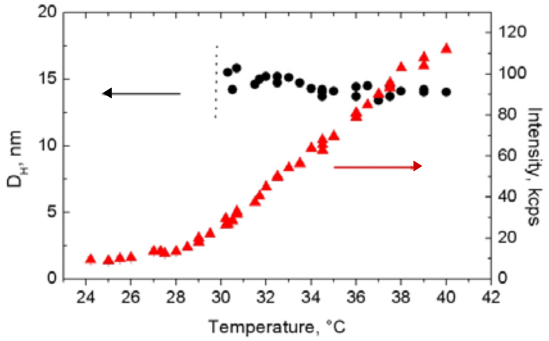
Highlights

Mapping of Critical Micelle Concentration



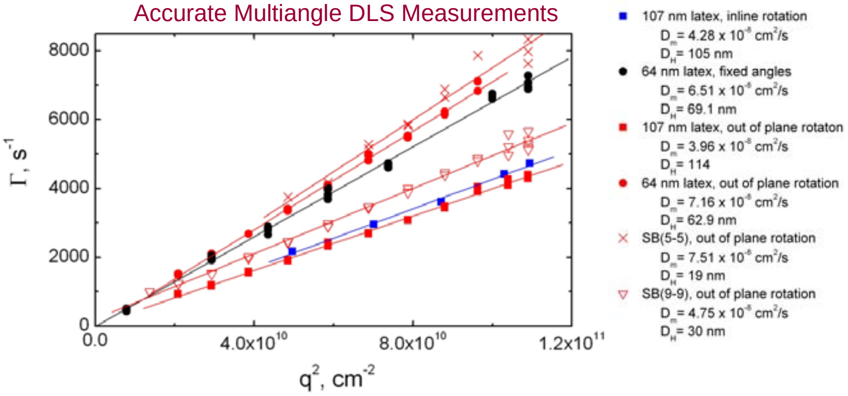
• The high throughput ability of the devices is demonstrated by blending a block copolymer, poly(styrene-b-isoprene), stock solutions. One is a neutral solvent yielding unimers and the other is selective yielding micelles. The critical solvent composition was rapidly identified as 25% toluene, 75% hexadecane.

Mapping of Micelle Temperature Behavior



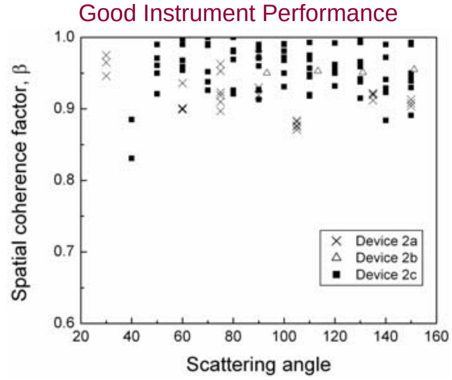
• DLS provides a simple and clear means of determining critical micelle temperatures. In this case PEO-PPO-PEO micelles in water form above 30 °C. The transition is evidenced by the scattering intensity and the fits of the autocorrelation functions.

Accurate Multiangle DLS Measurements

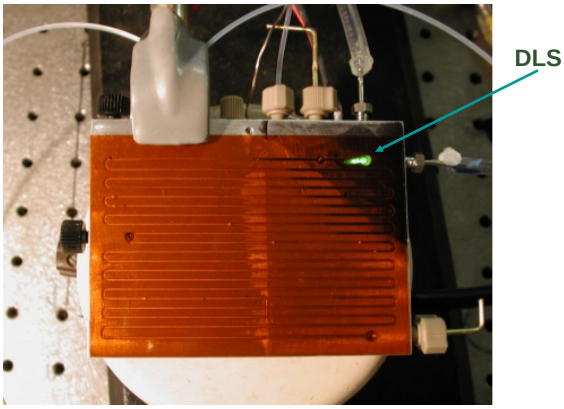


• The functionality of the multiangle DLS instruments is demonstrated with plots of the linewidth, Γ, vs. q^2. The accuracy in describing latex particles and block copolymer micelles is quite good.

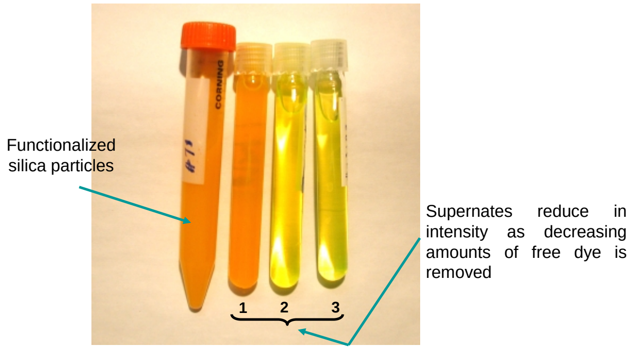
Good Instrument Performance



• A key measure of the quality of DLS data is the β value. All of the devices have consistly high β values, often >0.9.



• We have created a microfluidic device with a continuous reaction channel that incorporates DLS. Thus far we have synthesized monodisperse silica nanoparticles ranging from 90-185 nm, characterizing them as they exit the device.



• The silica nanoparticles can be functionalized. In this case, fluorescein triethoxy silane was covalently bonded to the surface. Free dye can be removed by centrifuging and redispersing the particles in new solvent. The particles remain intensely fluorescent, demonstrating the dye's attachment to the particles.

Contributors

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