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Parallel SANS/SAXS and DLS at nm - µm Scale: Quantitative and Experimental Strategy for Pharmaceutical Nanoparticles and Polymers

Biological, polymer and medical samples may depict a wide particle size spectrum from nm to μm scale. The particle structure in solution can be studied without radiation damage by SANS up to 1 μm size (ILL-D11, 30m distance, 15Å), and at high intensity by SAXS. The particle size distribution is available by dynamic light scattering DLS, while this requires with concentrated original samples of SANS and SAXS the application at backscattering (173°; NIBS , No-Invasive Back-Scattering) and focusing to the front layer of the sample to avoid multiple scattering and special optics.

At ILL-D11 we have developed a strategy, setup and theory for a quantitative SANS/SAXS-DLS coupling. The SANS/SAXS beam and the laser of DLS hit the same point of a quartz cuvette/capillary. The DLS laser beam (Tm00) is focused to the 100 μ m front layer of the cuvette to avoid multiple scattering. The DLS size range is extended from the usual 5 μ m to >100 μ m by a special long focus (150 mm) optics in a dual optical bench device (Nanovel ProSpecD). In structure dynamics applications SANS and DLS are operated time resolved (SANS-DLS film).

The evaluation of SANS and DLS data is done following the same principles and scaling: The raw DLS data IDLS(r) represent the amount of scattering yielded by the particle size spectrum r. Here the large particles are tremendously over-estimated by the dependence of IDLS(r) on r6. The evaluation of the contributions with respect to mass contribution Cm(r) requires a scaling according to the particle type, as usual in SANS and SAXS in the Guinier- and Kratky-Porod-plots for spherical, flat / liposomal or stick-type particles. A theory and formula set for quantitative DLS in parallel to SANS/SAXS with pharmaceutical nanoparticle examples, and optics setup calculation is presented.

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