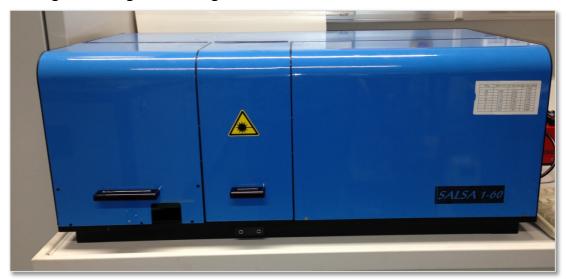




# **TU Graz - Light Scattering Laboratory equipment**

# **Small Angle Static Light Scattering**



The flat cell light scattering instrument consists of a GLG5360 Helium-Neon Laser (NEC Corporation, Tokyo, Japan, P=10mW,  $\lambda$ =632.8nm) and an array consisting of 160 photodiodes for simultaneously detecting scattering curves from 1° to 60° (Scattering vector in water  $2.3*10^{-4}-1.3*10^{-2}$  nm<sup>-1</sup>) . The sample cell has a variable thickness of 15 µm up to 5 mm. This allows to measure turbid samples with particle sizes between a few hundred nanometers up to about 10 µm.





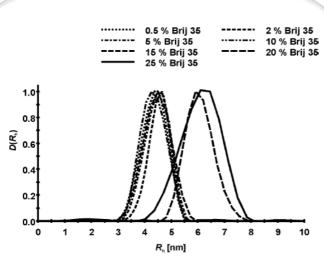
Information for users, on the requirements for measuring their samples.

#### DLS

By evaluation of the measured correlation function hydrodynamic radii from a few nanometers up to a few micrometers can be obtained. A minimum of 500  $\mu$ l of clear and dust free sample with sufficient concentration (typically several per mills, for micellar solutions higher than the cmc) is necessary. Biosamples (Proteins etc.) can be measured in a square cell at 90°C with only 30 $\mu$ l solution (typically per mill). Sample may be exposed to air during the measurement. Viscosity of the solvent should be known. Sample should not absorb light at 532 nm or 632.8 nm.

### **Examples:**

DLS can be used for the determination of hydrodynamic radii of proteins and lipoproteins [1] [2], vesicles [3] surfactant micelles [4], emulsion droplets [5] inorganic particles [6][7], etc.



**Figure 5.** The results of the volume-weighted inverse Laplace transformation of the DLS experimental data for the Brij 35/water binary system at different surfactant concentrations corrected for the concentration effect according to the linear law presented in Figure 4. The curves of the size distribution function  $D(R_{\rm H})$  are normalized to the same peak height.

Example: Determination of the size distribution of micells by DLS. Figure taken from [4]

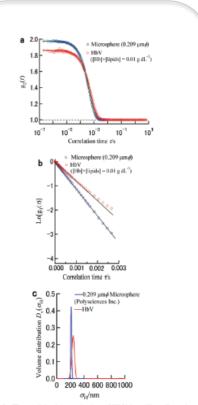


Figure 1. The particle characterization of HbV in a dilute dispersion as obtained by DLS. Intensity correlation functions,  $g_2(t)$ , of a dilute HbV dispersion ([Hb] =  $6.3 \times 10^{-3}$  g dL<sup>-1</sup>) and a 0.027 wt % dispersion of pseudomonodisperse microsphere with a nominal averaged diameter of 0.209  $\mu$ m measured at 25 °C (a), the cumulant fit to the same DLS data (b), and the particle size distribution of the HbV and the microsphere (c), obtained by using ORT.<sup>56</sup>

Example: Determination of the size of Hemoglobin vesicles by DLS. Figure taken form [3]





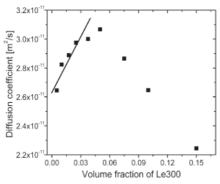


Figure 8. Collective diffusion coefficient for Le300 without surfactant as a function of particle content from 0.5% to 16%. The straight line is a visual guide and corresponds to the typical slope shown by charged repulsive systems at low volume fraction.

Example: Determination of the hydrodynamic radii of silica particles. Figure taken from [6]

# Multispeckle DLS:

Dynamics of ergodic non-ergodic systems can be measured. Correlation times up to  $10^4$  seconds can be achieved. A minimum of 20  $\mu$ l of sample is necessary. Flat cells are airtight but long-term stability against air cannot be guaranteed. Sample should not absorb light at 632.8 nm.

Multispeckle DLS can be used for the measurement of slow dynamics in highly concentrated emulsions, gels, etc.

#### Small angle static light scattering:

Particles sizes from hundreds of nanometers up to 10  $\mu$ m can be measured. A minimum of 20  $\mu$ l of sample with sufficient concentration is necessary. Turbid samples e.g. milk can also be measured. Multiple scattering can be reduced by decreasing the cell thickness (15 $\mu$ m up to 5mm). At Transmission be > 0.9 the effect of multiple scattering is negligible. Sample may be exposed to air during the measurement. Refractive indices of continuous and dispersed phase of the sample should be known.

#### Examples:

By means of small angle static light scattering the size and size distribution of emulsion droplets [8][9], inorganic particles [10], etc. can be determined.





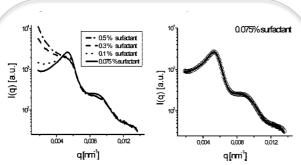
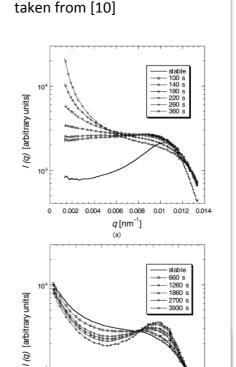


Figure 1. Results from static light scattering on dense (50 vol %) emulsions with various surfactant concentrations. Left: scattering patterns of oil-in-water emulsions with different amounts of added surfactant. The highest concentration of surfactant was  $\sim 0.5$  wt % (dash-dotted), followed by  $\sim 0.3$  wt % (dashed),  $\sim 0.1$  wt % (dotted), and finally  $\sim 0.075$  wt % (solid). Right: scattering pattern of an emulsion with 50 vol % oil and a surfactant concentration of 0.075 wt % (open circles), in comparison with its GIFT-fit function (solid line). The GIFT evaluation of the scattering data was based on a hard sphere structure factor model resulting in the correct volume fraction of 49.8%.

Example: Small angle static light scattering of concentrated emulsions. Figure taken from [8]



Example: Small angle static light

scattering of silica particles and

resulting size distribution. Figure

Fig. 4. Measured scattering curves. (a)  $\Delta I$  method (30 vol)% untreated silica, 30 wt% sucrose, 1.65 mol/l urea, 50 units/ml urease). (b)  $\Delta pH$  method (30 vol)% coated silica, 40 wt% sucrose, 1.07 mol/l urea, 0.5 units/ml urease). The curve of the stable suspensions were measured separately.

(b)

 $q [nm^{-1}]$ 

0.002 0.004 0.006 0.008 0.01 0.012 0.014

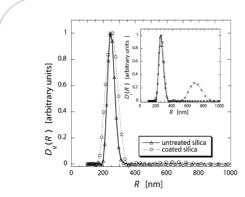


Fig. 3. Size distributions of the silica particles determined with static light scattering on diluted suspensions. Shown are the volume-weighted and the intensity-weighted (inset) distributions of both untreated and coated silica.

## References

- 1 Martin Gauster, Olga V. Oskolkova, Josef Innerlohninger, Otto Glatter, Gabriele Knipping, Saša Frank, Endothelial lipase-modified high-density lipoprotein exhibits diminished ability to mediate SR-BI (scavenger receptor B type I)-dependent free-cholesterol efflux. *Biochem. J.* **2004** 382, 75–82.
- 2 Manuela Augsten, Hubert Hackl, Birgit Ebner, Angela Chemelli, Otto Glatter, Günther Marsche, Uwe Lang, Gernot Desoye, Christian Wadsack, Fetal HDL/apoE: a novel regulator of gene expression in human placental endothelial cells. *Physiological Genomics* **2011**, 43, 1255-1262.
- Takaaki Sato, Hiromi Sakai, Keitaro Sou, Martin Medebach, Otto Glatter, Eishun Tsuchida, Static Structures and Dynamics of Hemoglobin Vesicle (HbV) Developed as a Transfusion Alternative. *J. Phys. Chem. B* **2009**, 113, 8418–8428





- 4 Matija Tomšič,\* Marija Bešter-Rogač, Andrej Jamnik, Werner Kunz, Didier Touraud Alexander Bergmann, Otto Glatter, Nonionic Surfactant Brij 35 in Water and in Various Simple Alcohols: Structural Investigations by Small-Angle X-ray Scattering and Dynamic Light Scattering. *J. Phys. Chem. B* **2004**, 108, 7021-7032.
- 5 Anniina Salonen, Christian Moitzi, Stefan Salentinig, Otto Glatter, Material Transfer in Cubosome-Emulsion Mixtures: Effect of Alkane Chain Length. *Langmuir* **2010**, 26, 10670–10676.
- 6 S. Ahualli, G. R. Iglesias, W. Wachter, M. Dulle, D. Minami, O. Glatter, Adsorption of Anionic and Cationic Surfactants on Anionic Colloids: Supercharging and Destabilization. *Langmuir* **2011**, 27, 9182–9192.
- Amin Sadeghpour, Franz Pirolt, Otto Glatter, Submicrometer-Sized Pickering Emulsions Stabilized by Silica Nanoparticles with Adsorbed Oleic Acid. *Langmuir* **2013**, 29, 6004–6012
- 8 Norbert Freiberger, Martin Medebach, Otto Glatter, Melting Behavior of Shear-Induced Crystals in Dense Emulsions as Investigated by Time-Resolved Light Scattering. *J. Phys. Chem. B* **2008**, 112, 12635–12643.
- 9 Gerhard Fritz, Elke M. Wagner, Helmut Lindner, Wolfgang Hofmann, Rudolf Zechner, Otto Glatter, Dependence of lipoprotein-lipase-catalyzed triacylglycerol hydrolysis on droplet size of synthetic monodisperse emulsions measured with static light scattering. *Journal of Colloid and Interface Science* **2004**, 275, 642–648
- Hans M. Wyss, Josef Innerlohinger, Lorenz P. Meier, Ludwig J. Gauckler, Otto Glatter, Small-angle static light scattering of concentrated silica suspensions during in situ destabilization. *Journal of Colloid and Interface Science* **2004**, 271, 388–399.