Journal of large-scale research facilities

Journal of large-scale research facilities, 1, A29 (2015) http://dx.doi.org/10.17815/jlsrf-1-27

Published: 19.08.2015

KWS-2: Small angle scattering diffractometer

Heinz Maier-Leibnitz Zentrum
Forschungszentrum Jülich, Jülich Centre for Neutron Science

Instrument Scientists:
- Aurel Radulescu, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH, Garching, Germany, phone: +49(0) 89 289 10712, email: a.radulescu@fz-juelich.de
- Noemi Kinga Szekely, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ),
Forschungszentrum Jülich GmbH, Garching, Germany, phone: +49(0) 89 289 10739, email: n.szekely@fz-juelich.de
- Marie-Sousai Appavou, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH, Garching, Germany, phone: +49(0) 89 289 10747, email: m.s.appavou@fz-juelich.de

Abstract: KWS-2, operated by JCNS, Forschungszentrum Jülich, represents a classical pinhole SANS instrument where, combining the pinhole mode using different neutron wavelengths and detection distances with the focusing mode using MgF_2 lenses, a wide Q-range between 1 x 10^{-4} and 0.5 $Å^{-1}$ can be explored.

1 Introduction

KWS-2 (Radulescu, Pipich, Frielinghaus, & Appavou, 2012) is dedicated to high intensity/ wide-Q investigation of mesoscopic structures and structural changes due to rapid kinetic processes in soft condensed matter, chemistry, and biology. The high neutron flux, comparable with that of the world leading SANS instruments, which is supplied by the neutron delivery system (cold source, selector, guides) (Radulescu & Ioffe, 2008; Radulescu, Pipich, & Ioffe, 2012), and the possibility to use large sample area using focussing lenses, enable high intensity and time-resolved studies. On demand, the instrument resolution can be tuned using the double-disc chopper with adjustable opening slit, which allows the variation of the wavelength spread between 2 and 20 %. This offers a high flexibility in optimising the instrument performance towards improved characterisation of structural details and accurate beam characteristics (avoid the gravity and chromatic effects while using the lenses).

Figure 1: Instrument KWS-2.

2 Typical Applications

- Colloids, nanocomposites, polymer gels, networks
- Polymer blends, diblock copolymers
- Microemulsions, complex fluids, micelles
- Membranes, films; in-situ adsorption – desorption/ humidifying – drying phenomena
- Kinetics of demixing, formation, aggregation
- Shear induced micelle deformation, rubber network deformation, nanocomposite ordering
- Protein structure and folding/ unfolding
- Pressure dependence of phase diagrams, fluctuations, molecular interactions
- In-situ crystallisation semi-crystalline polymer

Self-assembly of block-copolymers in micellar structures is a widely studied topic at KWS-2. The properties of block-copolymer micelles tuned by changing e.g. solvent quality, temperature, solvent selectivity, block copolymer composition, and molecular weight are investigated thoroughly benefiting from the adjustable instrumental resolution between 2 and 20 %.

Another kind of typical application relate to fast structural changes of micellar systems (formation, transformation or chain exchange at equilibrium) or polymer crystallisation which are investigated by time-resolved SANS in the second or sub-second (up to 50 ms) regimes. More recently, the determination and control of the morphological parameters of biocompatible gels and amphiphiles became an important topic of study stimulated by the demands from nanomedicine related to the design of new functional drug delivery vehicles.

3 Sample Environment

- Anton-Paar fluid rheometer
- Stopped flow cell
- Sample holders: 9 horizontal x 3 vertical (temperature controlled) for standard Hellma cells
- Oil & water thermostats (typical 10 °C ...100 °C)

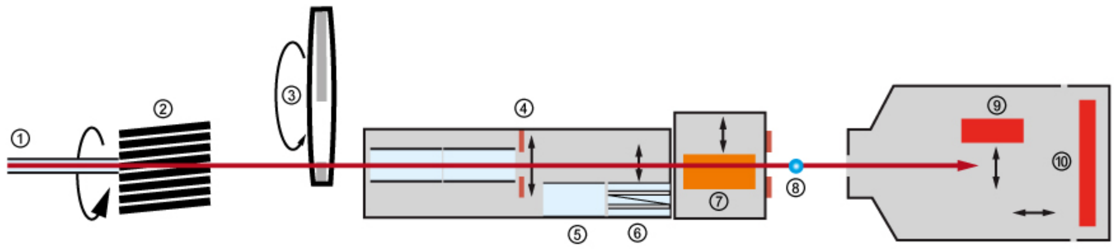

Figure 2: Schematic drawing of KWS-2.

- 8-positions thermostated (Peltier) sample holder (-40 °C ... 150 °C)
- Magnet (1.5 T, vertical)
- Cryostat with sapphire windows
- Pressure cells (500 bar, 5000 bar)
- Humidity chamber, 5 % … 95 % for 10 °C … 60 °C

Complementary in-situ techniques (optional at sample aperture, see instrument plan)
- Anton-Paar fluid rheometer
- FT-IR spectroscopy
- DLS & SLS
- ^{3}He spin analyzer (SEOP)

4 Technical Data

4.1 Overall performance

- Q = 0.0001… 1 Å^{-1} (with tilted selector)
- Maximal flux: $2 \cdot 10^8$ n cm^{-2} s^{-1}
- Typical flux: $2.5 \cdot 10^7$ n cm^{-2} s^{-1} (collimation 8 m, aperture 50 x 50 mm^2, λ = 5 Å)

4.2 Velocity selector

- Astrium, $\Delta\lambda/\lambda$ = 20 %, λ = 3… 20 Å

4.3 Chopper

- Tunable $\Delta\lambda/\lambda$: 20 %... 2 % (TOF analysis)

4.4 Polariser

- Transmission, $P > 95$ % for $\lambda > 4.5$ Å

4.5 Active apertures

- 2 m, 4 m, 8 m, 14 m, 20 m, sample position

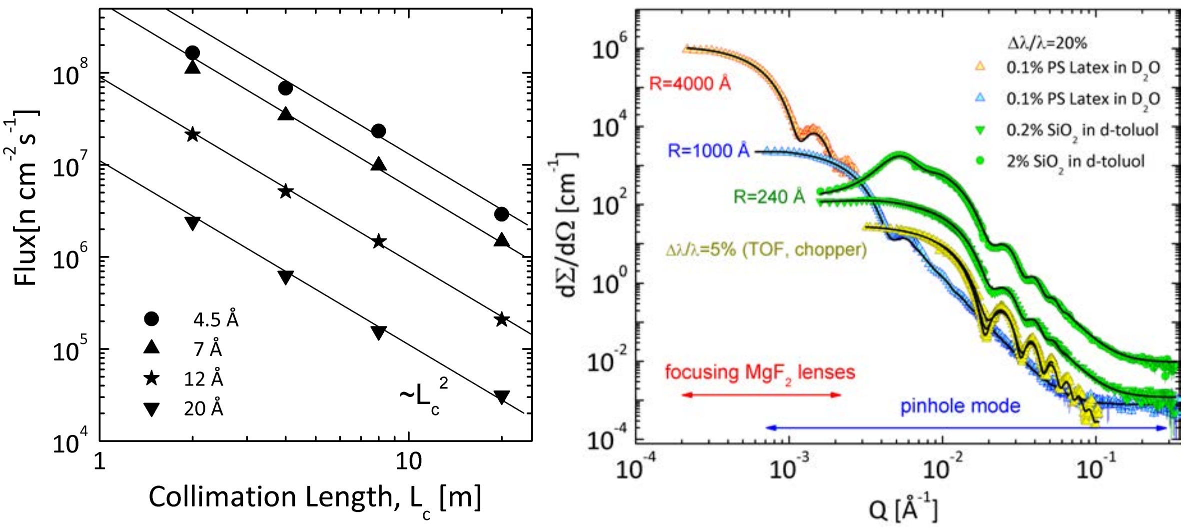

Figure 3: Left: the absolute neutron flux at the sample position; Right: the available range of momentum transfer Q for a selection of instrument configurations (the experimental data are described by model function convoluted with the instrument resolution).

4.6 Aperture sizes

- Rectangular 1 x 1 mm^2 – 50 x 50 mm^2

4.7 Neutron lenses

- MgF_2, diameter 50 mm, curvature 20 mm
- Packs with 4, 6, 16 lenses

4.8 Sample stage

- XYZθ translational-rotational stage + craddle
- Accuracy better than 0.01°, 0.01 mm

4.9 Detector 1

- Detection range: continuous 1 – 20 m
- ^{3}He tubes array, active area $\sim$ 0.9 m^2, count rate for no deadtime $>$ 2 MHz, resolution = $<$ 8 mm, stability of pixel response $\sim$ 0.1 %, efficiency 85 % for 5 Å

4.10 Detector 2 (high res.)

- Spatial resolution 0.45 x 0.45 mm^2
- Active area: Ø = 8.7 cm
- ^{6}Li-Scintillator 1 mm thickness
- Fixed position: 17 m after sample position

References

Radulescu, A., & Ioffe, A. (2008). Neutron guide system for small-angle neutron scattering instruments of the Jülich Centre for Neutron Science at the FRM-II. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *586*(1), 55-58. (Proceedings of the European Workshop on Neutron OpticsNOP '07) http://dx.doi.org/10.1016/j.nima.2007.11.039

Radulescu, A., Pipich, V., Frielinghaus, H., & Appavou, M. S. (2012). KWS-2, the high intensity / wide Q -range small-angle neutron diffractometer for soft-matter and biology at FRM II. *Journal of Physics: Conference Series*, *351*(1), 012026. http://dx.doi.org/10.1088/1742-6596/351/1/012026

Radulescu, A., Pipich, V., & Ioffe, A. (2012). Quality assessment of neutron delivery system for small-angle neutron scattering diffractometers of the Jülich Centre for Neutron Science at the FRM II. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *689*, 1-6. http://dx.doi.org/10.1016/j.nima.2012.06.027