

# Current status of KWS-3

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**KWS-3 is a very small angle neutron scattering (VSANS) instrument running on the focusing mirror principle at the Research Neutron Source Heinz-Maier Leibnitz (FRM-II) in Garching-München. Standard configuration of the instrument with 9.3m sample-to-detector distances allows performing scattering experiments with a wave vector transfer resolution between  $10^{-4}$  and  $10^{-3} \text{ \AA}^{-1}$ , bridging a gap between Bense-Hart and pinhole cameras. Installation of new sample position at 1.1m distance transforms an important supplementary instrument to self-efficient one in case of many applications. Now KWS-3 approaches the end of a major upgrade aiming for proper use of the enhanced neutron flux after the instrument relocation from Jülich.**

Small-angle scattering is used for the analysis of structures with sizes just above the atomic scale, between 1 and about 100 nm, which cannot be assessed or sufficiently characterized by microscopic techniques. KWS-3 is an important supplementary instrument, which extends the accessible range of scattering angles to very small angles with a superior neutron flux when compared with a conventional instrumental setup with pinhole geometry. Thus the length scale that can be analyzed is extended beyond 1  $\mu\text{m}$  for numerous materials from physics, chemistry, materials science and life science, such as alloys, diluted chemical solutions and membrane systems.

An idea of the instrument on the focusing mirror principle has already been given by Alefeld et al. in 1989 [1]. Test experiment of high-resolution focusing SANS with a small toroidal neutron mirror in 1997 [2] has supported above-mentioned idea. The final decision to build KWS-3 instrument at the FRJ-2 reactor in Jülich took place in 2000 and final configuration was discussed by Alefeld et al. in [3]. KWS-3 became operational in Jülich in 2003 [4], and after the shutdown of the FRJ-II reactor was rebuilt at the FRM-2 reactor in Garching/München.

In Fig. 1 the layout of the VSANS instrument KWS-3 is shown. The basic elements are an MgLi velocity selector with a wavelength spread 20% [5], a double focusing mirror with a focal length of 11 m and a two-dimensional pixel detector (scintillator of lithium glass with 6.6% <sup>6</sup>Li mixed with cerium) of sufficient pixel resolution. The mirror is a 1.2 m long, 12 cm wide and 8 cm thick toroid coated with 80 nm of <sup>65</sup>Cu. At such a short mirror length with respect to the focal length, the toroidal shape is a good enough approximation to an elliptical shape. The reflection plane has been chosen to be horizontal, reducing the deterioration of the image due to gravity. The layout depicts the 1:1 imaging of the entrance aperture, located in the front focus just behind the velocity selector, to the rare focus located on the detector plane.

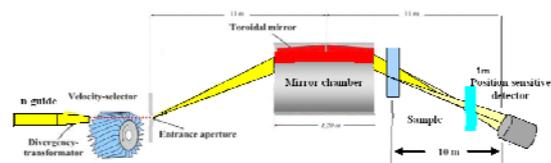


FIG. 1: Layout of the V-SANS experiment KWS-3.

After the transfer into the guide hall of the FRM II neutron source in Garching a major upgrade became necessary, with the aim to make proper use of the enhanced neutron flux, which is nearly one order of magnitude larger compared to FRJ-2. The mirror refurbishment, comprehensive upgrade activities in the vacuum system, electronics and programming have been performed with the aims of protecting the new mirror coating from aging, transforming the instrument into a user-friendly state and introducing conceptual improvements. Some details of finished upgrades have been published by Goerigk et. al (2011) [6].

Here we report about next important upgrades extending the Q-range, and usability of the instrument. These innovations will be finished in 2011 without disturbing of user operation program at KWS-3.

**Sample environment boosting at the standard sample chamber (9.3m).** A new more suitable and safe access to the sample chamber with a new adapter system (500 mm flange) for complex systems was designed and installed as shown in Fig.2. This adapter system could be used for installation of the complex sample environments like ovens, cryo-systems, magnets... A new crane has been already installed at the new wavelength chopper of KWS-2 and can be used to remove the flange and install complex (heavy) systems of the sample environment.

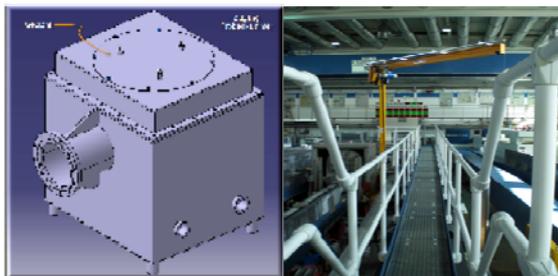


FIG.2: New safe access to sample chamber with suitable adapter for complicated sample, new access platform to sample chamber, and crane for heavy sample environment

The sapphire sample cells so far used at KWS-3 are not vacuum-sealed. Additionally the cross section of the cells (window diameter 15 cm) is nearly an order of magnitude larger compared to the beam ( $10 \times 2 \text{ cm}^2$ ) and by this a ten times larger sample amount has to be provided, which almost is not needed for the scattering experiment. Thus a new cell development with more suitable sizes using improved sapphire windows (chemo-mechanically polished) had been started. In a 1st step sapphire cells (gas-sealed) have been developed with a cross-section of  $10 \times 3 \text{ cm}^2$ , which can be used under atmospheric pressure (Fig.3). In a 2nd step these cells will be mounted as an inset in a vacuum sealed housing by using larger sapphire windows of  $13 \times 11.4 \text{ cm}^2$  size with the possibility to thermostat the whole system. The sapphire insets, the vacuum-sealed housing with thermostat systems, as well as sample holder for Helma cells are already available for users.

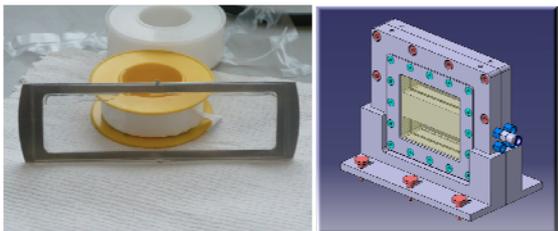


FIG.3: New safe access to sample chamber with suitable adapter for complicated sample.

**New sample position at 1.1m distance.** Additionally, conceptual improvements have been introduced to KWS-3, in particular, a new sample chamber at a distance of 1.1m with respect to the detector. Fig. 4 represents the SANS curve of silica particles (in H<sub>2</sub>O) with a diameter of 300 nm and a narrow size distribution obtained from measurements at the two distances. The implementation of the second sample position has allowed extending the Q-range of KWS-3 by an order of magnitude to cover more than two orders of magnitude, and transforms an important supplementary instrument to self-efficient one in case of many applications.

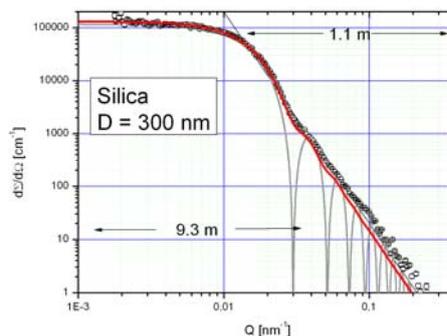


FIG. 4: Silica spheres of 300 nm diameter measured at 9.3 and 1.1 m (new sample chamber) at KWS-3 (black symbols: 10mg/ml). The grey curve is the theoretical scattering curve (form factor of 300nm spheres) calculated from the contrast of the (silica) particles with respect to the solvent (H<sub>2</sub>O) and the particle concentration. The minima of the Bessel functions are not resolved because of the instrumental resolution [6].

Fig.5 depicts the new layout of the second sample chamber, which is under construction in ZAT and will soon become available. The new sample chamber will be adapted to the flight tubes by additional vacuum valves and will be fully equipped with vacuum systems and an additional sample stage.

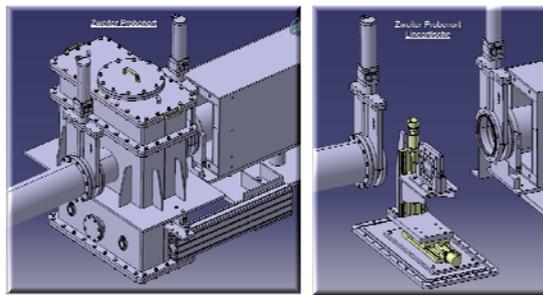


FIG. 5: New sample chamber of KWS-3 at 1.1m.

**New beam stop.** KWS-3 has two standard modes, namely high-resolution and high-intensity one. In case of the high-resolution mode  $2 \times 2 \text{ mm}^2$  aperture is used, and  $5 \times 5 \text{ mm}^2$  for the high-intensity. Therefore, two beam stop absorbers should be available simultaneously. Also, the whole system should have easy

access for maintenance to change active beam-stop. In new conception the detector is spitted from the rear flight as shown in Fig. 6. Detector could be easily moved out from shielding (not shown in figure) and needed beam-stops could be installed.

Additional option of given detector configuration is possibility to measure strongly scattered samples in Q-range of  $10^{-2}$ - $10^{-1}\text{\AA}^{-1}$  ( $\lambda=12.3\text{\AA}$ , sample-to-detector distance 12cm, flux  $>1.2 \times 10^5$  n/sec/cm<sup>2</sup>). As well as one will be able to use this short configuration to measure sensitivity of detector with Plexiglass directly at KWS-3 beam line without complicate movement of detector to the sample position of the KWS-2, as we do it now.

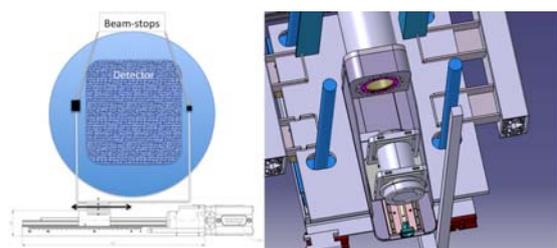


FIG. 6: (left) Linear table ( $\pm 6\text{cm}$ ) will insert with two beam-stops of different size; (right) new conception of detector-flight-tube connection.

**Electronics and detector upgrade.** The software for electronic setup and readout of the two-dimensional pixel detector needed comprehensive diagnosis and programming activities when implemented onto a Linux platform. Calibration tables obtained from the measurement of pixel-related pulse-height spectra for the removal of events have been established and tested. The dead time of the pixel detector was determined with a 'non-paralytic model' to a value of 2.9 ms (Fig. 7).

Subsequently, comprehensive measurements for the characterization of the pixel detector have been performed, including sensitivity measurements (for correction of the two-dimensional scattering pattern) and calibration measurements of the detector pixel resolution ( $dx = 2.74$ ,  $dy = 2.55$  pixel/mm).

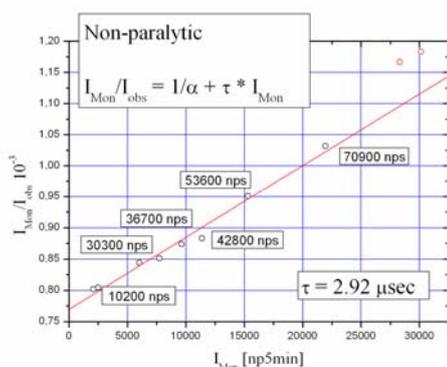


FIG. 7: Dead time of the KWS-3 pixel detector. Data are normalized to the monitor count in units of neutrons per 5 min.

The 'non-paralytic model' shown in the figure was employed. Beyond 70 kHz the detector becomes partially saturated (red circles).

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