	Experiment title: In-situ characterization of aerosol nanoparticles with low volume fraction at ambient pressure with SAXS	**Experiment number**: MI-1330
Beamline: ID02	**Date of experiment**: from: 04.07.2018 to: 06.07.2018	**Date of report**: 21.12.2018
Shifts: 6	**Local contact(s)**: Dr. Michael Sztucki	*Received at ESRF:*

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Report:

In experiment MI-1330 we were successfully measuring airborne nanoparticles with ultra-low volume fraction with SAXS at the ID-02 beamline. The technique and the equipment, which enables these very sensitive SAXS measurements, was developed in previous SAXS measurments at ELETTRA synchrotron. While improving the technique, we realized that a high brilliance and stable beam, as provided at ESRF, is a prerequisit for gathering the small changes between signal and background. A stable and low background hence play an important role.

To this end, the setup, consisting of a flow tube, was redesigned by taking all findings from previous experiments into account. Figure 1 shows a schematic drawing of the setup. The aerosols with a concentration in the order of $\sim 10^6$ particles per cubic centimeter were generated by a tungsten hot wire generator operated with helium as carrier gas. The use of helium as carrier gas allowed us to perform SAXS measurements of nanoparticles at ambient pressure and temperature. The resulting aerosol or particle free helium was then routed alternatingly by the switching module into the inner flow tube. The switching was synchronized with the acquisition cycle of the SAXS system. Thereby, SAXS signals can be obtained alternately of particles or pure helium background. By this means small fluctuations e.g. by deposition at the windows can be corrected, because exterior long-term influences cancel out by averaging. The inner flow is superimposed by a laminar sheath flow shortly before the SAXS measurement point keeping the particles away from the Kapton windows. Electrostatic charging of the windows by the x-ray beam and hence particle deposition is prevented by using conductive Kapton. As reference the aerosol nanoparticles will be guided through a core sampling system to a Differential Mobility Particle Sizer (DMPS) for measuring the size distribution. In addition, transmission electron microscopy (TEM) samples where taken in parallel. These instruments were adopted for the operation with helium.

The set-up day before the beamtime was necessary to install our setup in the hutch and adjust the DMPS mesaurement system for helium. We set up two sample-to-detector distances of about 2m and 10m for the SAXS experiments and calibrated the absolut intensity scale with a capillary of water. The absolut calibraiton is crucial for retrieving the volume fractions of the particles from the SAXS signal. After the calibration was done we were ready to measure airborne nanoparticles with SAXS.

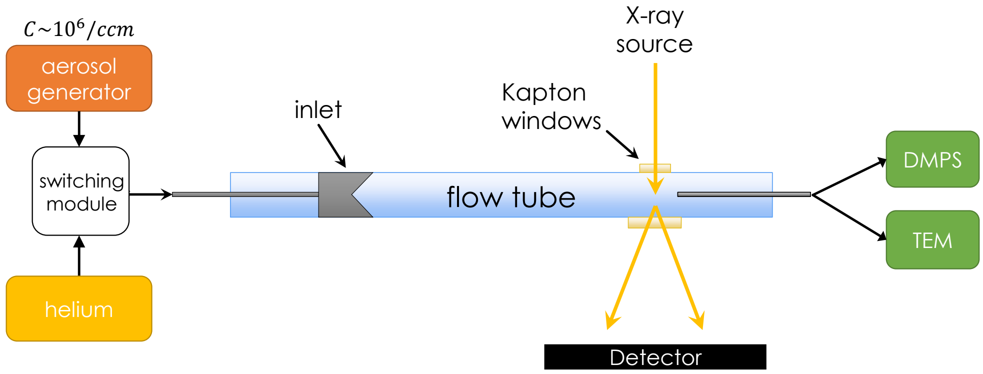

Figure 1: SAXS setup used at the ID-02 beamline for measuring airborne nanoparticles with ultra-low volume fraction.

Figure 2 shows a SAXS signal aquired at the ID-02 for airborne tungsten nanoparticles. The first graph presents the absolut calibrated raw signal and raw background for a sample-detector-distance of 2m. The background is an average of the background taken before and after the signal. The signal of the particles is so small, that it is only visible in a detail, displayed in the second graph. The third graph shows the difference between the signal and the background. The maximal intensity difference coming from the particles is in the order of $\sim 10^{-4}$ cm^{-1}. This leads to the assumption that the volume fraction of the particles is in the order of $\sim 10^{-10}$ for tungsten particles. This agrees well with the results of the volume fraction aquired with the DMPS system. We have taken several measurements for different tungsten hot wire generator adjustments to retrieve the best overlap for the SAXS and DMPS technique.

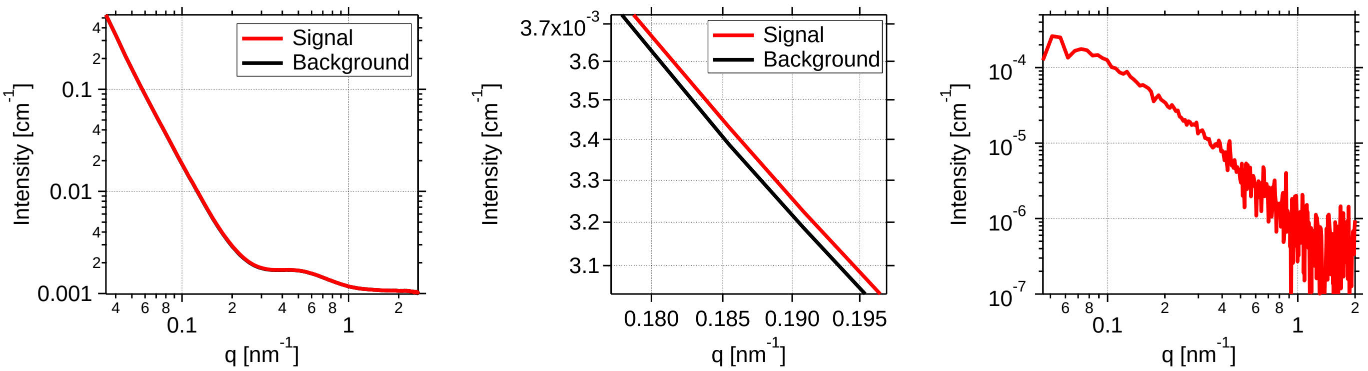

Figure 2: Absolut calibrated SAXS signals of tungsten nanoparticles with a volume fraction of about 10^{-10}. The first graph shows the full range of the aquired SAXS signal. The second graph displays a zoom of the first graph to see the small difference between the signal of the particles and the background. The third graph presents the difference between the signal and the background of the first graph. The maximal intensity difference coming from the particles is only $\sim 10^{-4}\ cm^{-1}$, which results in a volume fraction of $\sim 10^{-10}$ for tungsten nanoparticles.

To summarize, we obtained for the first time quantitative data from airborne tungsten nanoparticles in helium with ultra-low volume fraction using SAXS at the ID-02 beamline. This was only possibly by the high brilliance and stability of the ID-02 beamline. A crucial part of the technique is a stable background since the maximal intensity difference is only of the order of $\sim 10^{-4}\ cm^{-1}$. The measurments where done in helium under ambient pressure and temperature and with concentrations of the order of $\sim 10^6$ particles per cubic centimeter. With these settings it was possible to measure in parallel with a helium adjusted DMPS system. In addtion TEM samples were taken, which will help to examine the structure of the particles. This combination of thechniques is a unique way to characterize nanoparticles in the airborne phase. The results of the beamtime MI-1330 are already included in a publication currently submitted to Nature Communications. Further details on the publication will follow, when a decision is available.

Suggestions for further studies are the usage of other materials for the windows to reduce scattering losses and get an even more stable background. This might enable measurements of airborne particles with lighter materials than tungsten or to use air as carrier gas. Since the beam stablitiy is very crucial, measurements with Top-Up mode instead of the 7/8-Bunch mode would be very interesting.