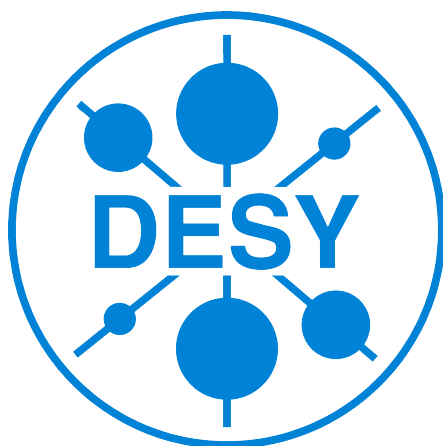


# DESY summer student program 2011



a report by

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[this is not an established emblem, so it should be considered as an unofficial one]

## Acknowledgements

I really should express my gratitude to all the organizers of the program, including Olaf Behnke, Doris Eckstein and Andrea Schrader with whom we communicated for great labour invested in establishing the program.

I would also like to thank all the lecturers for their input in our education and especially Rainer Gehrke, Oliver Seeck and Heinz Graafsma for rather inspiring lectures and also Catherine for the German classes.

My special thanks are addressed to people who guided me during my studies and work at DESY: to Jens Viefhaus and Leif Glaser who made a good introduction to DESY and good overall feeling, to Sascha Deinert for long detailed explanations of things and introduction to experimental science at DESY, to Markus Ilchen for guidance in coding; and to Ulla Vainio for the experimental week that she managed to make quite informative and entertaining at the same time. Also I'd like to thank all of them for kind treatment and willingness to answer all the questions raised.

Finally, I thank my partners, Pavel Volkov, with whom I was working during the exercise week, and Evgeniy Khramov, with whom I was working for the main group, and also other summer students for discussions, inspiration and other communication which are necessary for work and progress.

## Preface

As the time and my attention at DESY was scattered among many activities, I'd like mention most of them and not concentrate on the main one in the group. This will represent the feel of the program better.

My main activities were attending the lectures, working in the main scientific group, learning basics of the German language and practicing at B1 DORIS line. Also, I've contacted my Russian colleagues and asked if any synchrotron experiments are of interest and, given a positive answer, I've prepared and sent a proposal to the DOOR system. My side activities were mostly some more learning physics and coding, sports and some other things for rest.

# Main group activity: time-of-flight electron-ion spectroscopy

## Introduction

The basic idea of the electron-ion time-of-flight spectrometry is to study the results of inelastic scattering of photons on atoms by measuring the energies of electrons and ions generated during the process. The main process with the sample which is of consideration here is the photoionization of the specimen atoms, but also other ways to emit an electron, like Auger effect.

After an electron leaves an atom, it is separated from the generated ion with electric and magnetic field and then can be detected. What is measured in our case is the time of flight of each electron from the specimen to the detector as well as the ion time-of-flight (ToF). Considering this data one can determine the energies of the electrons, the same thing for the ions and also obtain some time resolution of the processes and analyse the coincidence structure.

One of the main products of the experiment is the two-dimensional spectra which represents the dependence of the number of events (electron reaches the detector) on the time elapsed since the XUV-pulse hit an atom and on the wavelength of incident photon. By analysing this spectra one can see what electrons have reached the detector. For instance, in the figure 1 one can see electrons kicked out different shells (four lines) and no Auger electrons (the latter have the energy and the time-of-flight which doesn't depend on the photon energy).

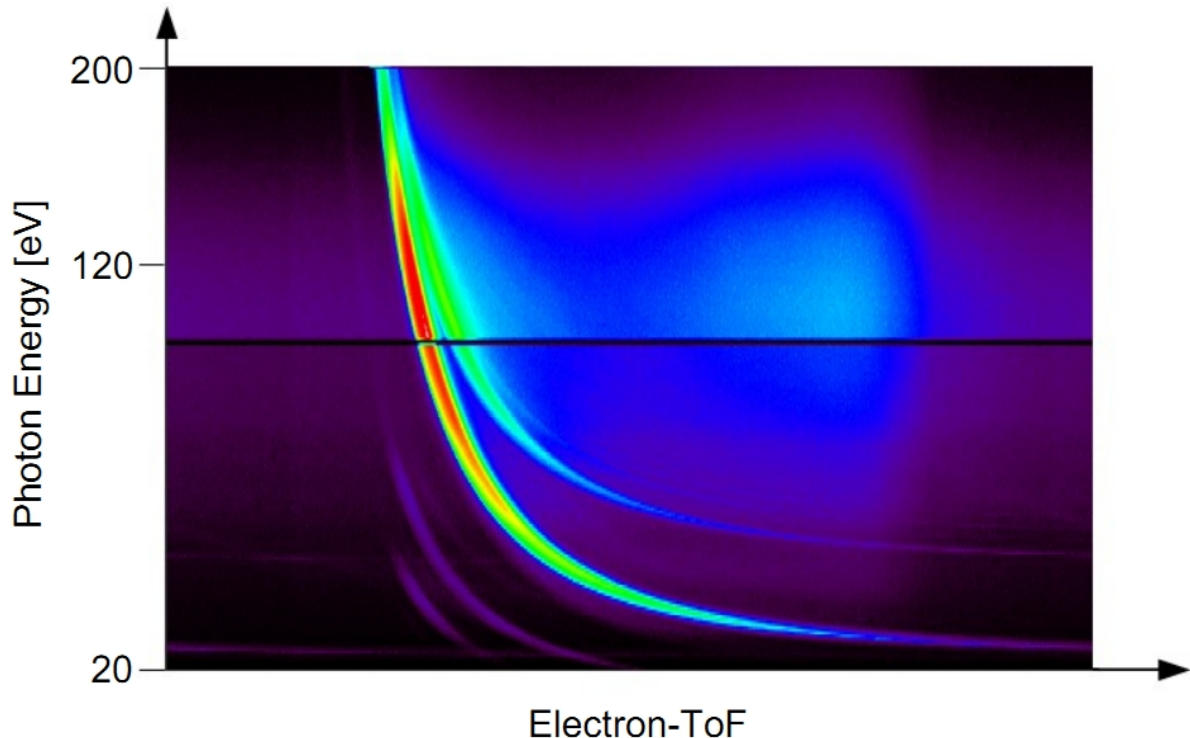


Figure 1. Electron 2D ToF spectrum. Sasch Deinert, [1]

The aim of the work in this year was to create a ToF spectrometer with a higher resolution of electron ToF. This means, for instance, that the lines on the picture should be

easily distinguishable within higher energy range.

## The setup

Let's consider the design of the setup prepared by Sascha Deinert [1]. Schematic layout is represented in the figure 2.

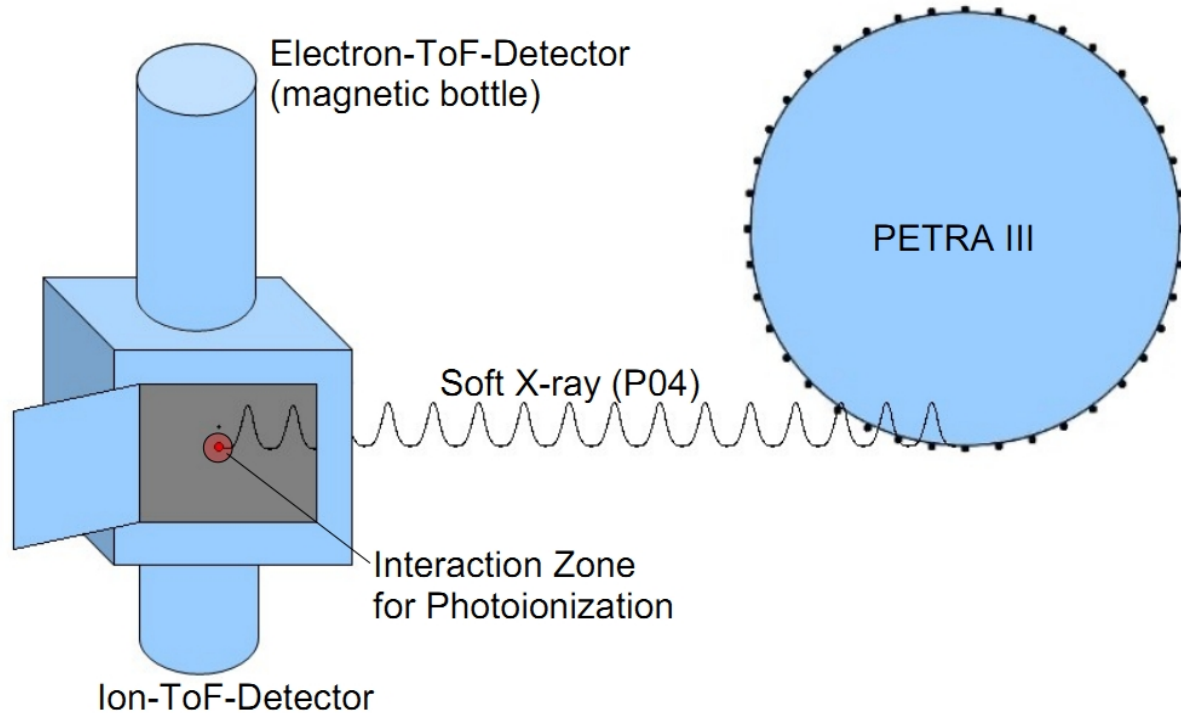


Figure 2. Schematic illustration of the setup layout. Sascha Deinert, [1].

The main principle of gathering the electrons is the principle of magnetic bottle which is used to turn all the electrons to the drift tube (see Figure 3). The higher is the gradient of the magnetic field, the the better is the ToF resolution since the electrons pass the distance which becomes closer to identical.

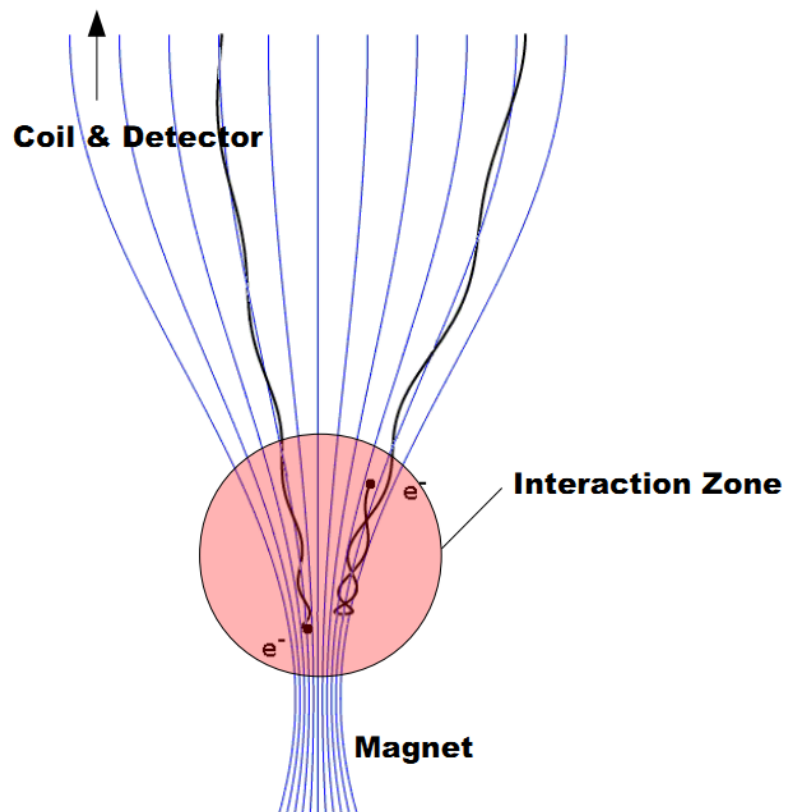
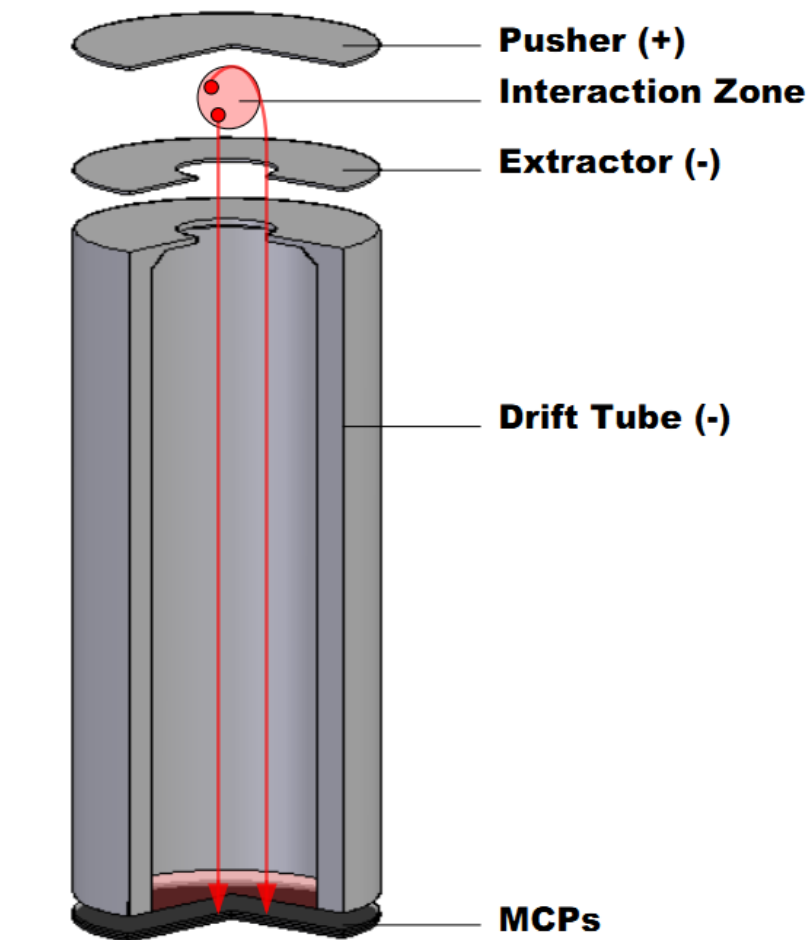


Figure 3, the magnet bottle principle for electron capture. Sascha Deinert, [1]

The ions are detected in the opposite direction and are captured using electrostatic field (which is possible since they have a very low velocity after the ionization). The layout is illustrated in the figure 4. Long drift tube provides better resolution while short one avoids heavy ions to be overtaken from later pulses.



The basic improvements that were proposed to be done were introducing a better magnet for the electron capturing, a longer drift tube and also pusher and extractor of a bigger diameter which was meant to provide the electric field to be more homogeneous.

Basically the whole setup chamber was created during this summer. I participated in some stages of preparation and learned the whole structure of it. During first launches to test the detectors it was shown that the electron ToF resolution was improved.

## Data representation

The main idea about the software development was that some parts of it were previously written in IDL and because most of the group members know only Python (and may be for some other reasons) it was proposed that those pieces of software should be written in Python.

So, I learned some basics of Python first. It seems to be quite useful since Python is good for many scientific applications (including tweaking some parameters when plotting functions because of the variable scope design) as well as others, which is supported by easy-to-read syntax, huge amount of libraries (including easy-to-use web-protocol support). However, Python is not very easy to install properly. For instance, even the basic examples from the Mayavi (a plot library) web site were not working right as they are expected to.

What I've done was the tool for creating the diagrams like that in the figure 1. An example of its usage on some elder data is shown in the figure 5. However, the new actual

data wasn't ready when I was at DESY and any further development of the software wasn't done. Also, one can notice a worse resolution which was because just one spectrum is evaluated but not a stack of many ones.

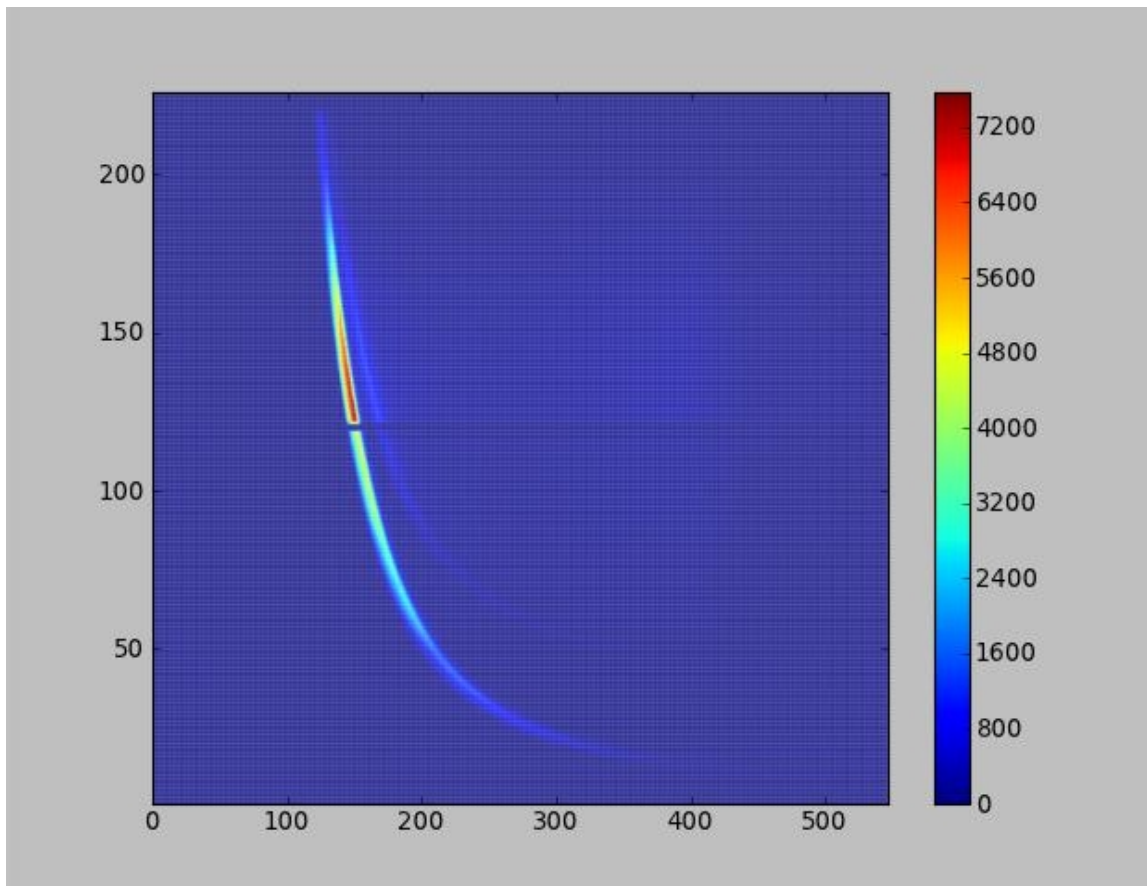


Figure 5. An example of data evaluation with the new Python software.

# Experimental week activity: anomalous SAXS investigation of Au particles in toluol.

## Introduction to ASAXS

Generally, SAXS (small angle x-ray scattering) is a variety of methods that are distinguished because of historical development of x-ray scattering. The latter began from wide angle x-ray scattering (WAXS) but then some new patterns were discovered in the beginning of the 20<sup>th</sup> century and in the 1940-1960 Guinier, Debye, Luzatti, Porod and others developed the interpretation for the basic features in SAXS patterns.

SAXS methods study elastic scattering of x-rays in two geometries: grazing incidence SAXS, which is used for studies of surfaces and transmission SAXS (figure 6). The latter can deliver information about different particles or other deluted inclusions like macromolecules and pores – the information like size, shape and arrangement of those. What is actually measured is just the intensity of the scattered x-rays within different directions.

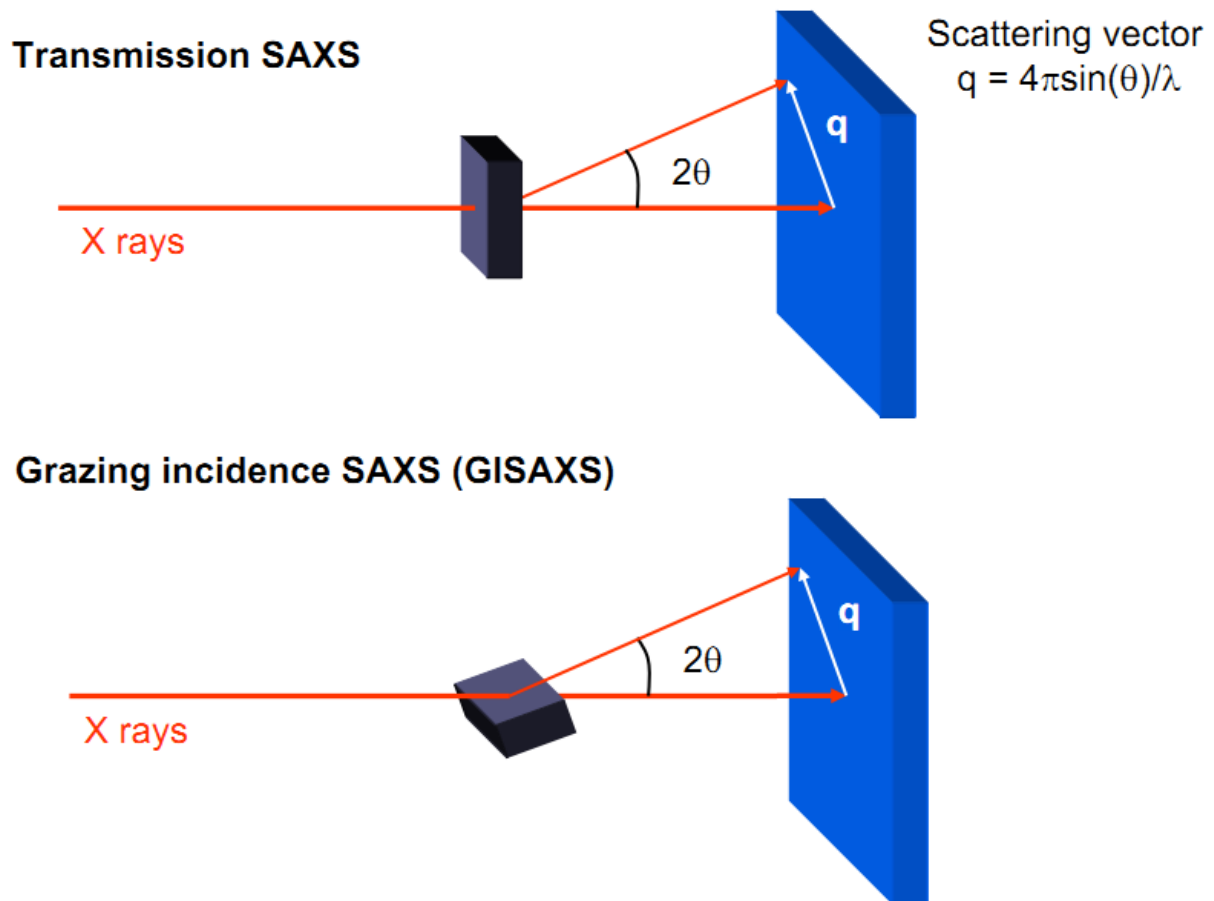


Figure 6. Transmission and grazing incidence SAXS. Ulla Vainio, [2].

The basic method of getting those parameters of the particles is to use some model of the scatterers and then to try to fit the experimental scattering curve with the modeled one, taking into account background and other details.

Anomalous SAXS is the method which allows to obtain element resolution of the



SAXS patterns. The physical background is the following.

Different elements have so called absorption edges which usually coincide with the anomalous scattering region (see figure 7) which means that near some energy absorption increases and the refraction index decreases and hence the scattering does so.

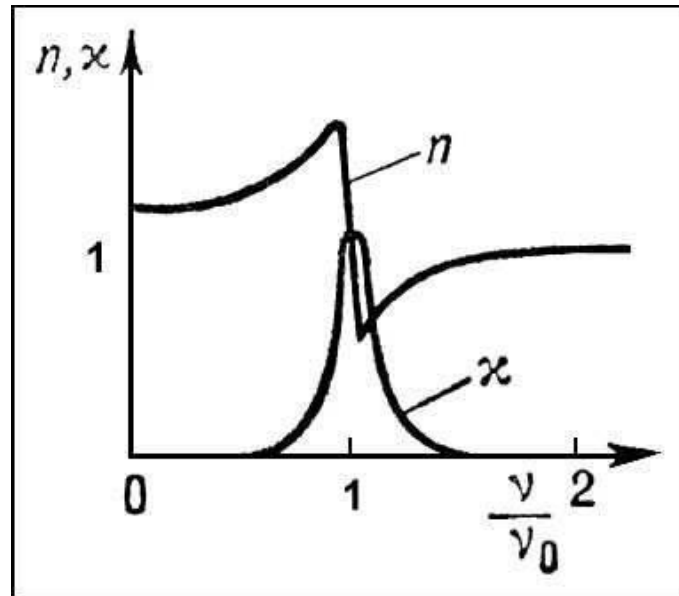


Figure 7. A sketch of the behavior of the refraction index ( $n$ ) and the coefficient of absorption ( $\chi$ ) near the absorption edge and the anomalous scattering region. An illustration from the internet.

If the SAXS data is taken in two different points near the edge, it can be subtracted one from another and the scattering from other elements is cancelled as well as the background while for the main element scattering is changed significantly and hence can be derived. This is the basis of ASAXS and this allows to achieve the element resolution.

### The background and the aim of the experiment

It is expected that LCLS will make it possible to resolve nanosecond scale processes and some proof-of-concept experiment is desirable to be made. Studies of Brownian motion of some scattering particles using bunch splitter and a delay scheme are proposed to be such an experiment. It is necessary, however, for such an experiment, that the particles shouldn't form clusters and the approximate size of the scatterers should be known. SAXS is just a proper method to figure whether this requirements are satisfied.

The scatterers are proposed to be golden particles. We have got eight specimens to examine. The colloids were produced using different methods and were represented as golden particles in toluol. Because of this, there were no need to use ASAXS but the subtraction of the background and the whole procedure was actually the same as in ASAXS.

### The setup

The setup we used was the B1 beamline of the DORIS III synchrotron which is dedicated for ASAXS experiments. The layout is illustrated in the figure 8.

Layout of ASAXS beamline B1 at HASYLAB, Hamburg with PILATUS 1M and MYTHEN detectors 2010

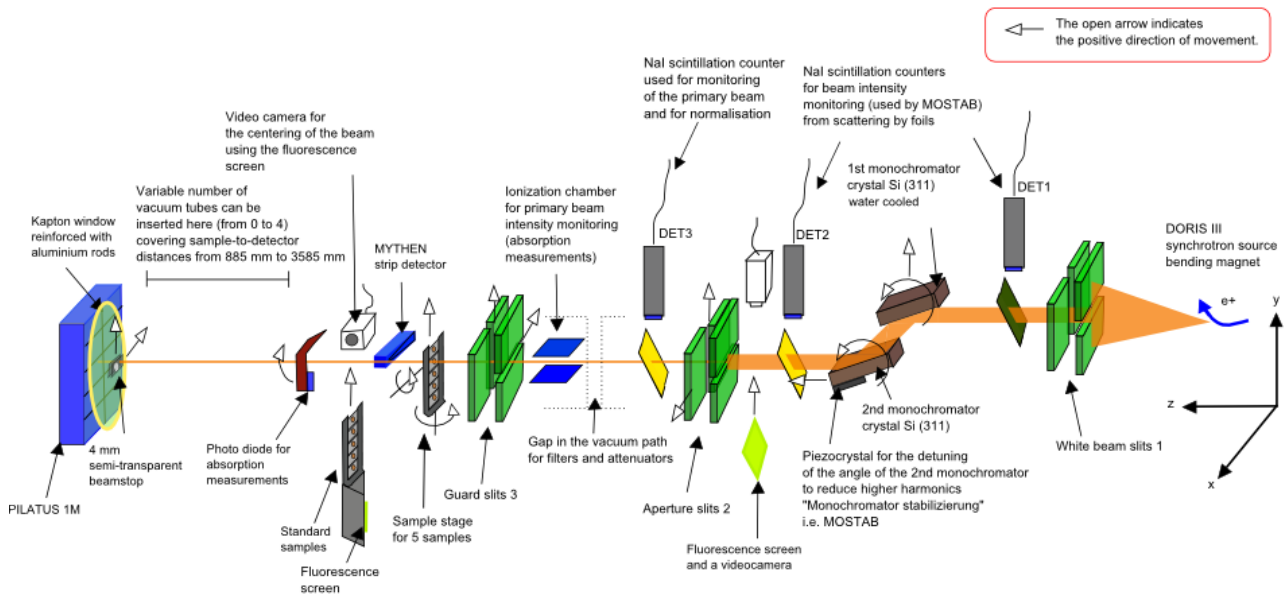


Figure 8. The setup layout. From hasylab.desy.de, [3].

Aside the optical system (slit system and the monochromator) and the sample system (multiple sample holder and the reference sample) the setup contains the following important elements: ionization chamber which controls the photon flux before it reaches the specimen, an additional WAXS detector, a diode detector which can be used to measure the intensity behind the specimen, the sliding tube between the sample and the main detector which allows measure scattering in different angle ranges and finally the main 2D detector itself with a semitransparent beamstop (which allows to easily justify the center of the beam). See the actual outlook of the setup in the figure 9.

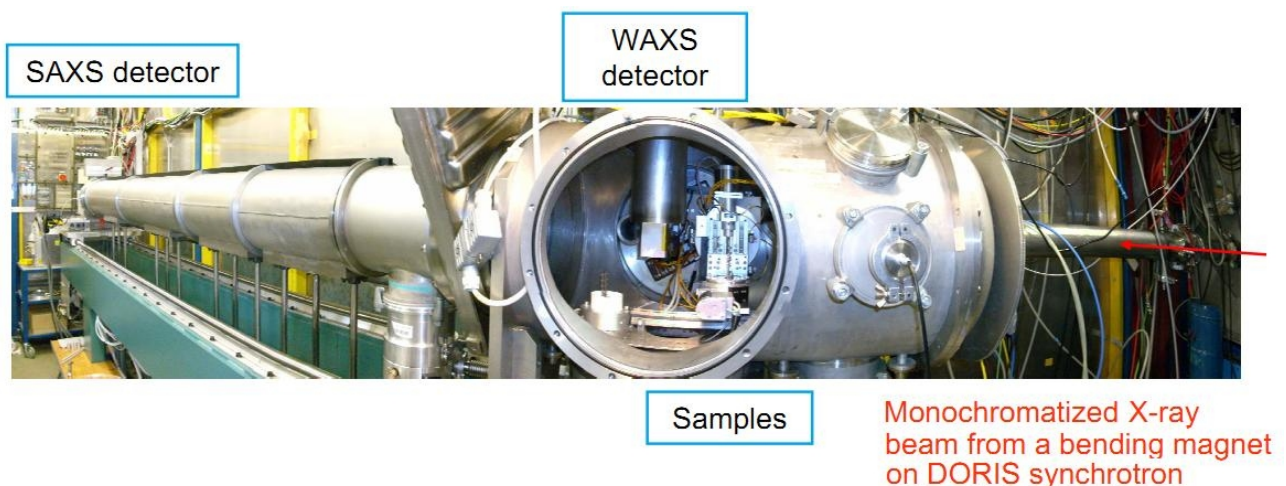


Figure 9. The setup outlook. Ulla Vianio, [2].

## Data processing

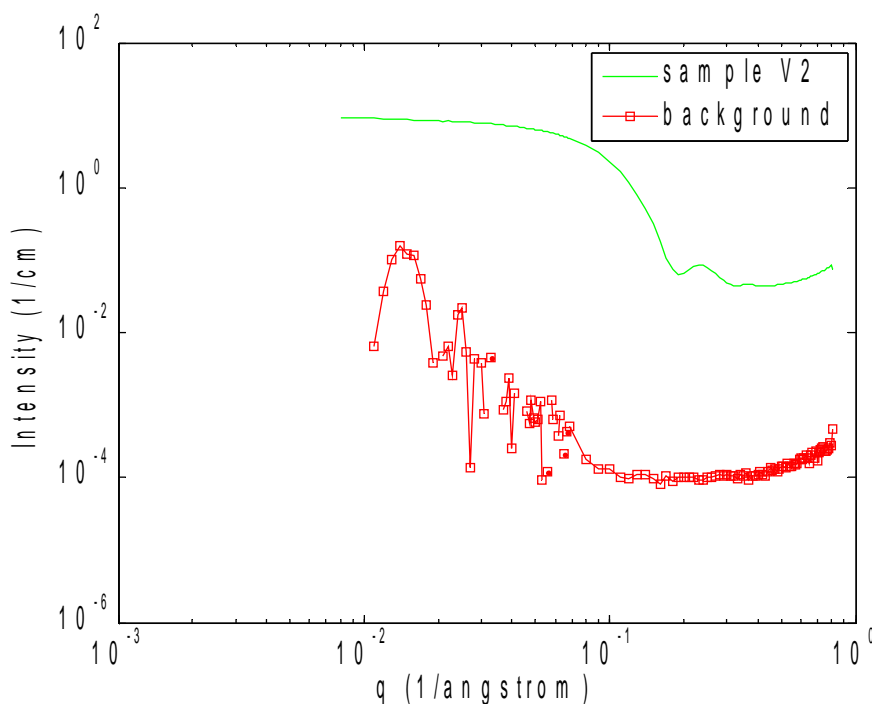
Most of the data processing is done using MatLab. First, some preprocessing like

integration along the angle and background substitution is done. In our case the experiment with the solvent (toluol) happened to be bad, so we found the measurements of toluol done some time ago at the B1 and used them. Then the main fit process with different models and cut of noisy data near the resolution edges is done. Basically we used two models: monodisperse spheres and spheres with the lognormal size distribution.

Also, the ATSAS package created by Dmitri Svergun and his scientific group can be used for reconstruction of the particle shape from the scattering data. The output .pdb files should be visualized with some other software – in our case it was the RasWin application [4] However, this shape reconstruction was rather a thing to play around since this Monte-Carlo simulation should be repeated many times and then the results should be averaged but in our case each simulation took about an hour and this was used just for reference (see the results).

## The results

Basically, the specimens can be divided into three classes: good ones, bad for the Brownian motion research ones and hard to interpret ones. The last group includes the measurement of the toluol which happened to be very noisy. We haven't figured the cause but, like it was mentioned before, we found the data toluol measured in the B1 station which we used for further data processing (figure 10).



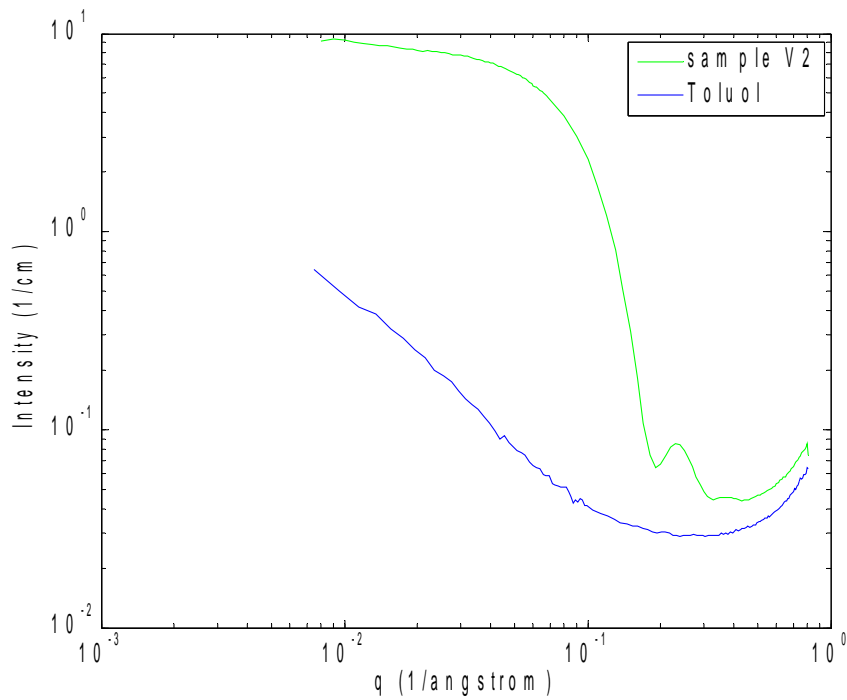


Figure 10. The data from one of the samples and of the backgrounds: the measured in our experiment and the measured before.

A couple of the bad for the Brownian motion research specimens had the scattering data shown below. The problem with them is that the increasing of the intensity for larger size scales and the peaks point to the presence of some clusterization which is not wanted for the experiment (figure 11).

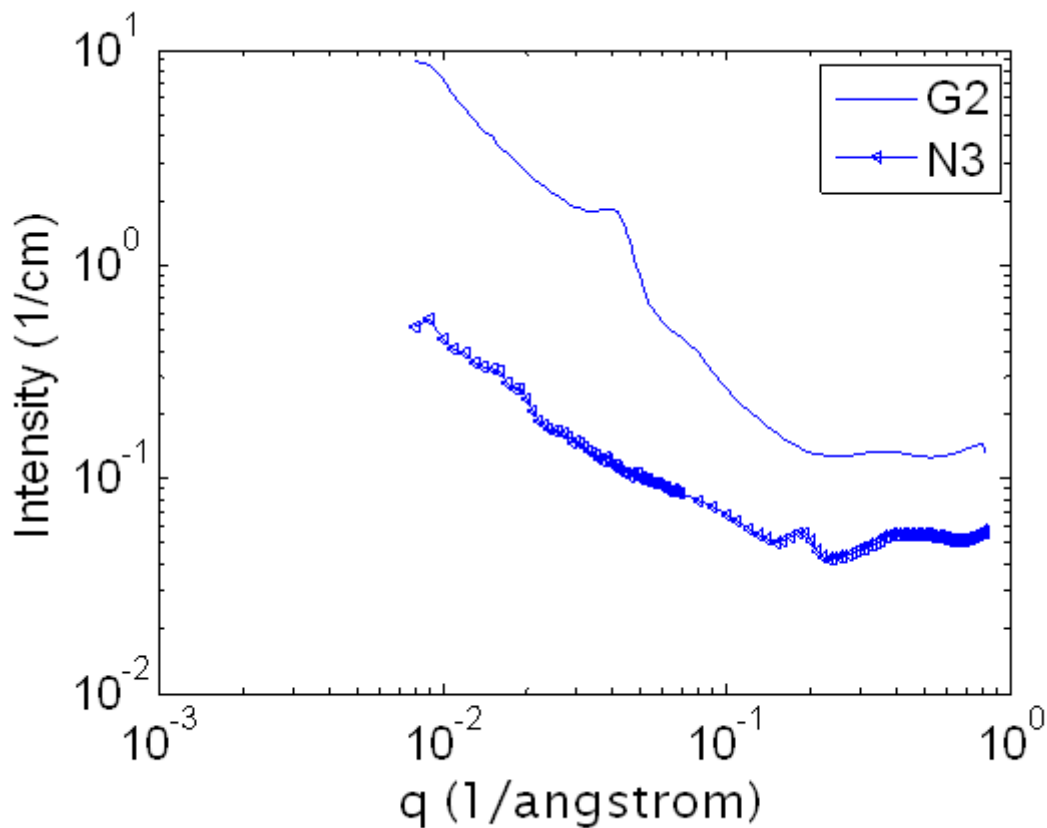


Figure 11. Scattering from specimens with structure factor.

Finally, one specimen happened to be good and was successfully fitted with monodisperse spheres model and with polydisperse spheres model. The size (the mean radius of the particles) was estimated as 23,4 Å with dispersion of about 2,2 Å. The fittings and the lognormal distribution used in modeling is shown below (figures 12-14).

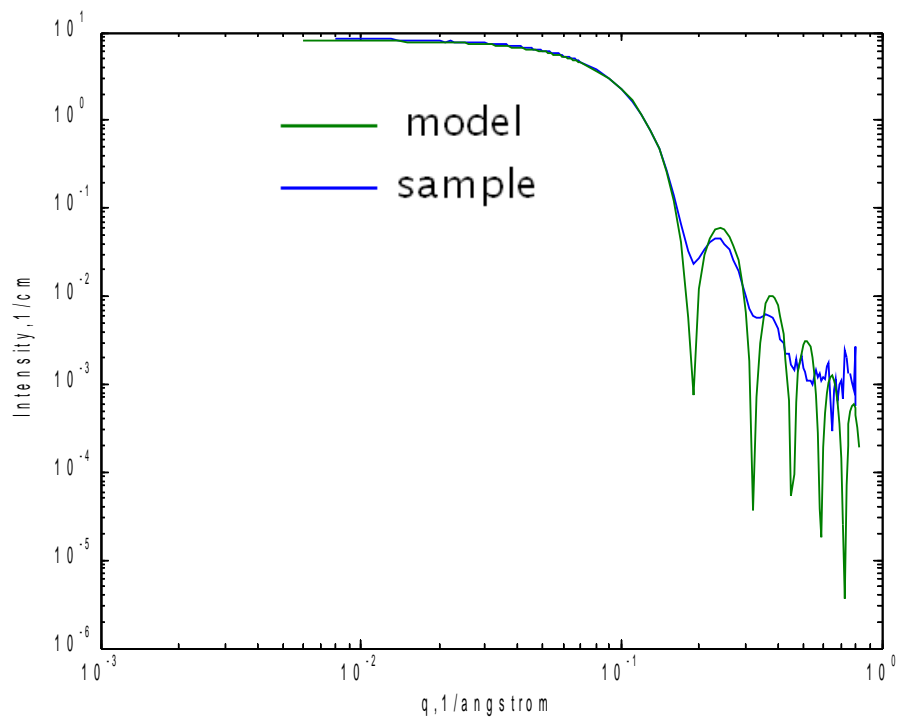


Figure 12. The monodisperse spheres model fit.

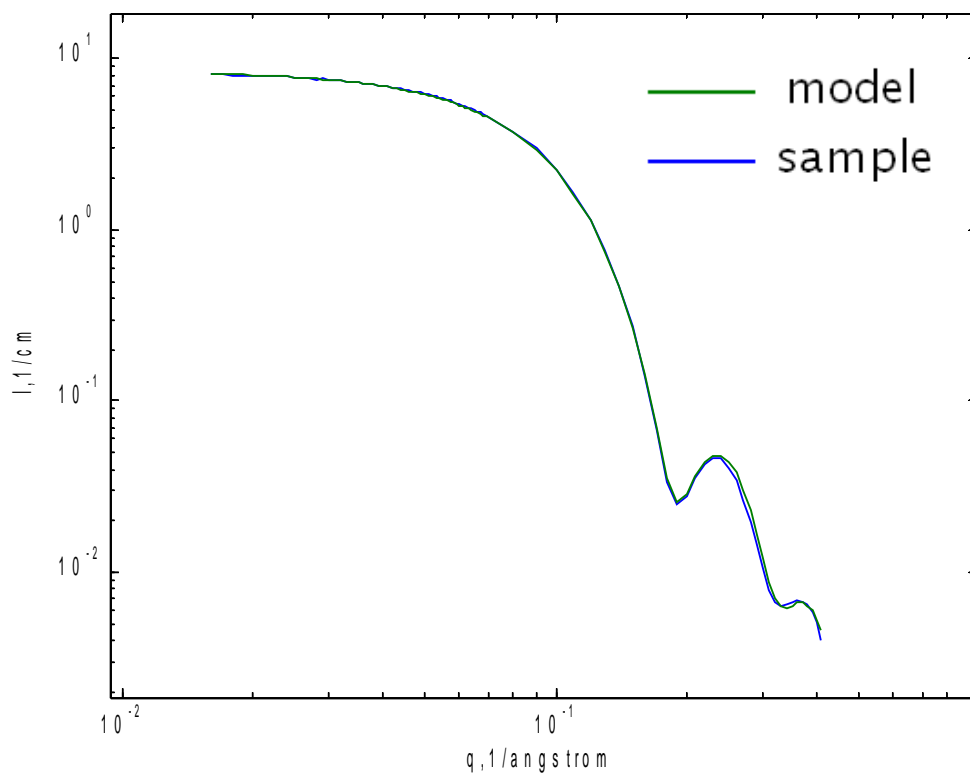


Figure 13. The polydisperse spheres model fit.

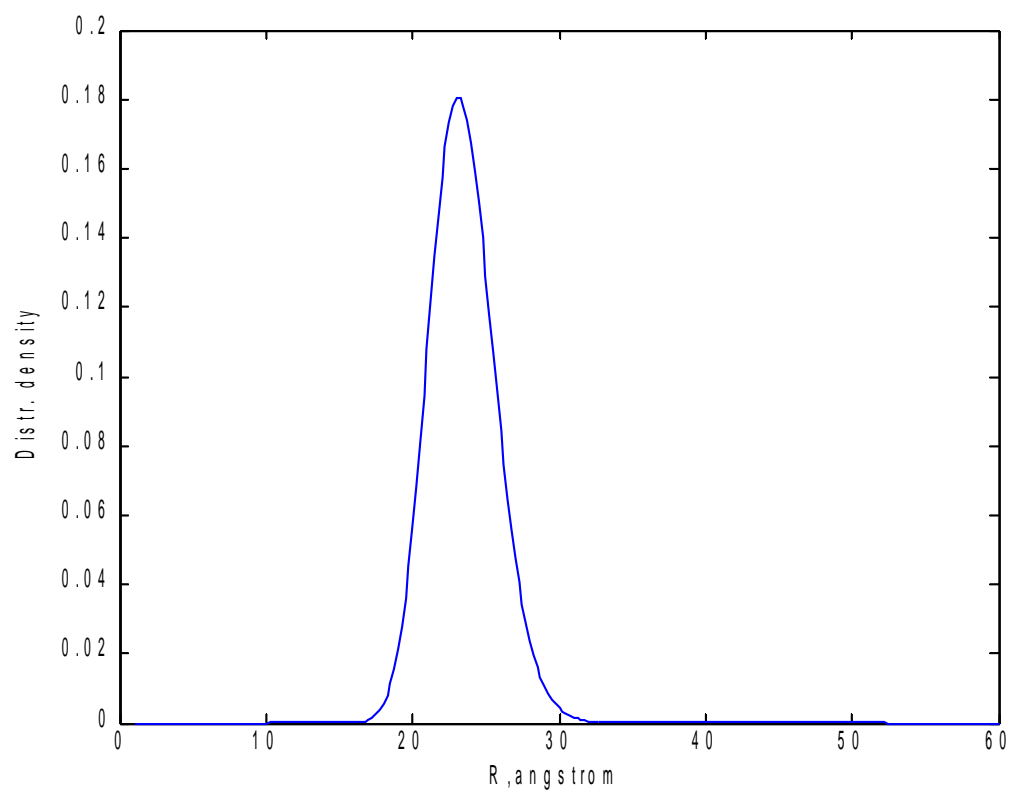


Figure 14. Lognormal distribution used in fitting.

# Choosing the setup for investigation of self-assembling microstructures in solutions of chiral matters

## Introduction

Solutions of a broad variety of chiral organic matters with very low ( $10^{-3} - 10^{-2}$  mol/L) concentrations are detected to solidify and turn into gel-like media. Studies with optical microscopy revealed the cause of the phenomenon. A system of anisometric one dimensional entities with a diameter of about  $1\mu\text{m}$  and with lengths up to some mm which has mechanical rigidity is formed in the solution, creating a framework.

Studies with atomic probe microscopy made it possible to distinguish the smallest strings – with the length from 50 to 500 nm and diameters of about 1 nm – in the residues of evaporated solution.

All the strings are detected to have helical structure and the bigger ones consist of twisted smaller ones; the process of the twisting has several phases. X-ray structural analysis showed that the strings have some crystal structure. Studies of optical properties (rotation of plane of polarization, circular dichroism, infrared and UV scattering, fluorescence) of a solution depend on the concentration and the temperature revealed that before the strings become visible other structures are formed which seems to be connected with a rise of linear associations of chiral molecules of the dissolved matter.

See [5]-[7] for details.

The aims of the experiment to be proposed were:

- to study the dependencies of the structure of strings and associations on the concentration of the solute matter and the temperature of the solution
- to examine the kinetics of forming of the strings and associations

## The setup choice

The setup choice tends to be a difficult problem. If one tries to overview all the experimental stations and to choose one then, he or she would probably end with still too much options and not enough information to decide. Many stations can do something that seems ok for the investigation (unless it is a study of a matter in extreme conditions or something else exotic).

On the other hand, one can figure the right choice if he or she knows what should be learnt about the specimen in the beginning of the program (which wasn't my case), as it can be somehow found out basing on the lectures. In this case, though, one should ask many additional questions, especially because good experimenters would say that it is possible to measure this or that on their station.

Anyway, it happened that the SAXS lecture occurred after I got to know about the necessity of experiment, so I asked Stephan Roth who works with and talked about SAXS if it can be used for the investigation and he answered that it should be so.

Then I chose the ASAXS beamline for my experimental week which is rather close to this matter but also because the anomalous SAXS is somewhat new and that was interesting to learn about. There I learned that SAXS does let to study scale features of deluted

specimens, but also that it is not that easy to interpret.

Finally, there remained less than two weeks before the deadline of sending the proposals so I contacted Stephan Roth to asked some more detailed questions and coordinate the details further with my colleagues. Stephan confirmed that all the scale features can be resolved and kinetic studies can be done on the P04 beamline of the Petra III synchrotron and that was actually the end of choosing. Also, Stephan confirmed that it is ok to left an open question of some extra studies for comparing with the main experiments, so I also wrote that some sort of imaging can be useful here.



## Conclusion and references

### Conclusion

The DESY summer student program happened to be quite rich with different experiences. I've attended to many lectures which introduced many fields and aspects of photon science. I've participated in work of two scientific groups and learned much about two methods, learned a new programming language, started learning German, faced real difficulties with proposal formulation, met many new people and much more. This was really amazing.

### References

- [1] Sascha Deinert, "Development and First Measurement of a Highly Efficient Electron-Ion-Coincidence-Spectrometer", Hamburg, 19.04.2011
- [2] [presentation by Ulla]
- [3] [http://hasylab.desy.de/facilities/doris\\_iii/beamlines/b1/experimental\\_station/index\\_eng.html](http://hasylab.desy.de/facilities/doris_iii/beamlines/b1/experimental_station/index_eng.html)
- [4] <http://www.umass.edu/microbio/rasmol/getras.htm#raswin>
- [5] Stovbun S.V., Mikhaylov A.I., Zanin A.M., Kostyanovsky R.G. / MSRU bulletin. «Natural science» series, №3, 2011, p. 92-97.
- [6] Stovbun S.V., «Chemical physics», Vol30, №8, 2011, p. 1-8.
- [7] Stovbun S.V., Zanin A.M., Skoblin A.A., Mikhaylov A.I., Kostyanovsky R.G., Grishin M.V., Shoob B.R., «Chemical physics», Vol30, № 9, 2011.