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PROJECT REPORT
SUMMER STUDENT PROGRAM 2008

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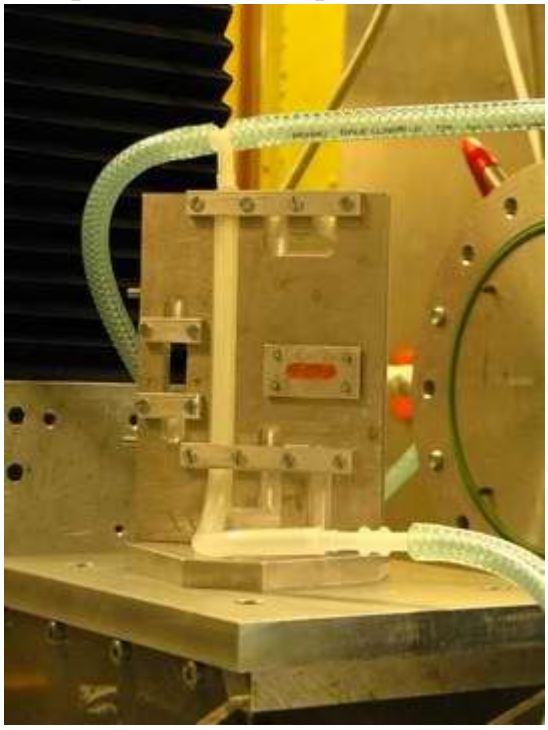
1. Introduction

My main work during the Summer Students Prorgam 2008 was testing a new sample holder to research chemical reaction of liquid samples. This new cell is usefull to mesaure liquid samples by X-ray techniques (for example XAFS). In an XAFS experiment, one measures an absorption spectrum. There are two regions in XAFS spectra. XANES: X-ray Absorption Near Edge Structure region and EXAFS: Extended X-ray Absorption Fine Structure. In the transmission mode absorption spectrum is measured as the portion of the signal that is absorbed (i.e. does not pass through) the sample. The sample holder have possibility to measure in the fluorescence mode, too. From the XAFS study we can obtain information about coordination environment or oxidation state (and its changes). The idea of XAFS measurements of liquid samples reaction is quite new and very interesting.



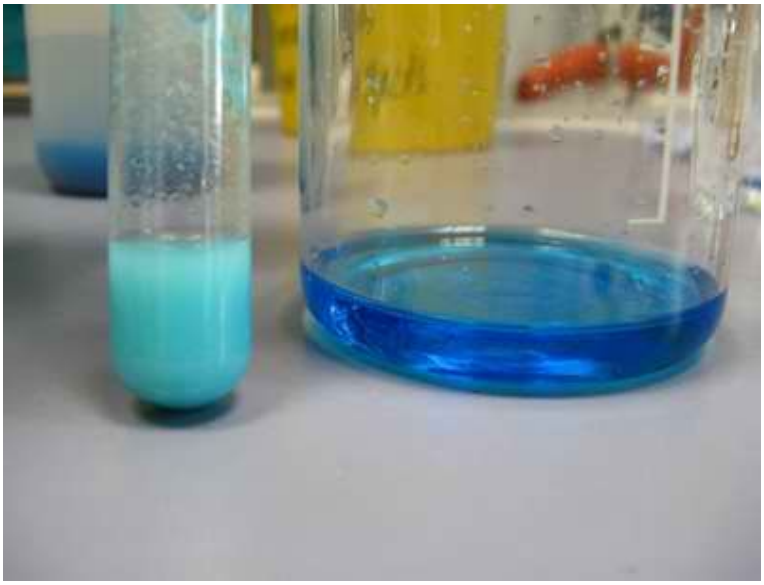
Pic. Sample holder.

Using the sample holder we can measure one liquid or reaction of two liquids. We can do our measurements in the time of the reaction. The flow speed of liquid/liquids can be control by speed of pomps.

2. 2. Project

At first, my partner and I have checked the reaction which can used to test sample holder. The XAFS measurements will be mostly done at beamline X so we decided to find the investigated atom with edge energy above edge energy of iron. We chose copper as an element and following reaction:

$$CuSO_4 + NaOH \rightarrow Cu(OH)_2 + Na_2SO_4$$

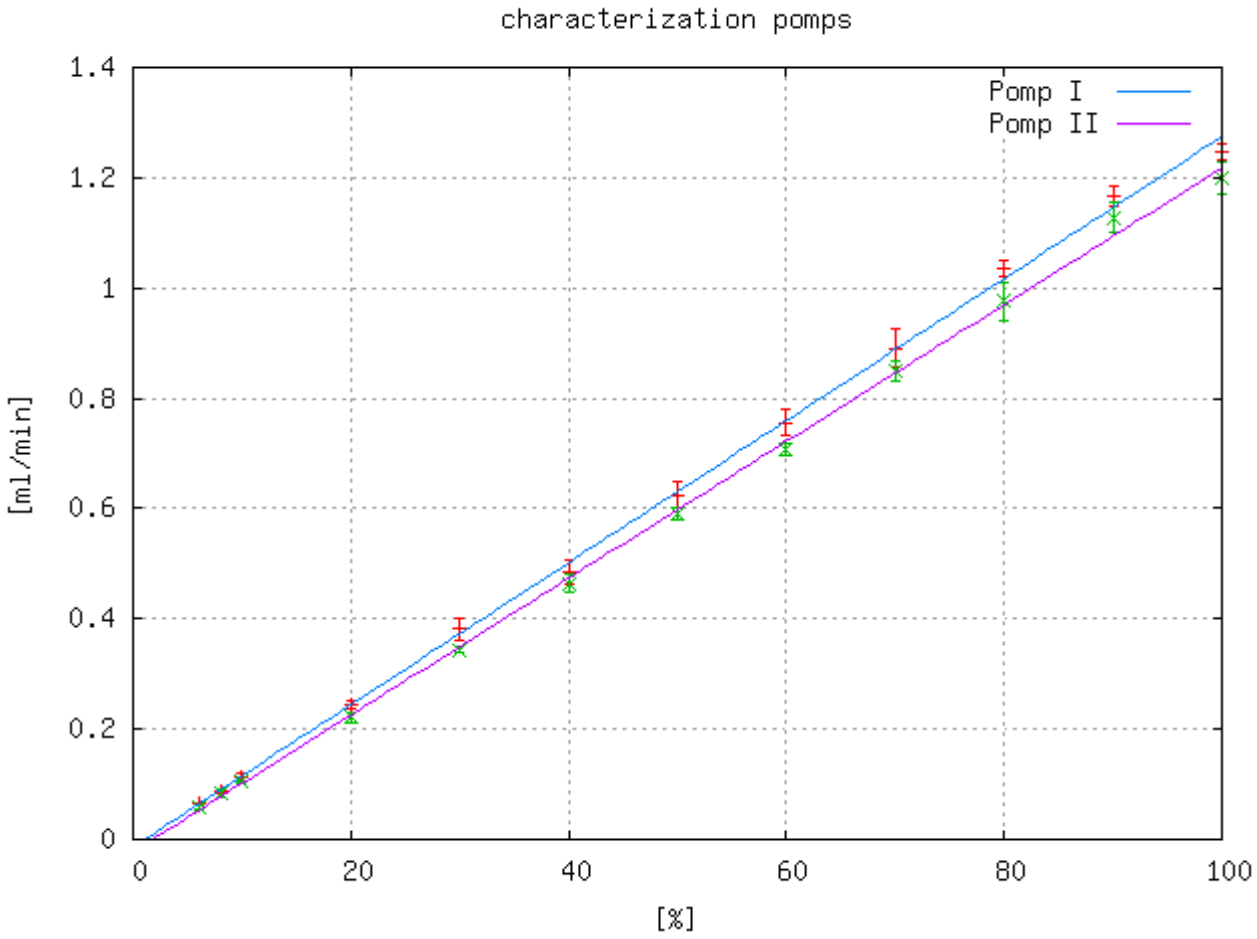


Pic. 1 $CuSO_4$ +NaOH -> $Cu(OH)_2$ + Na_2SO -> CuO_4

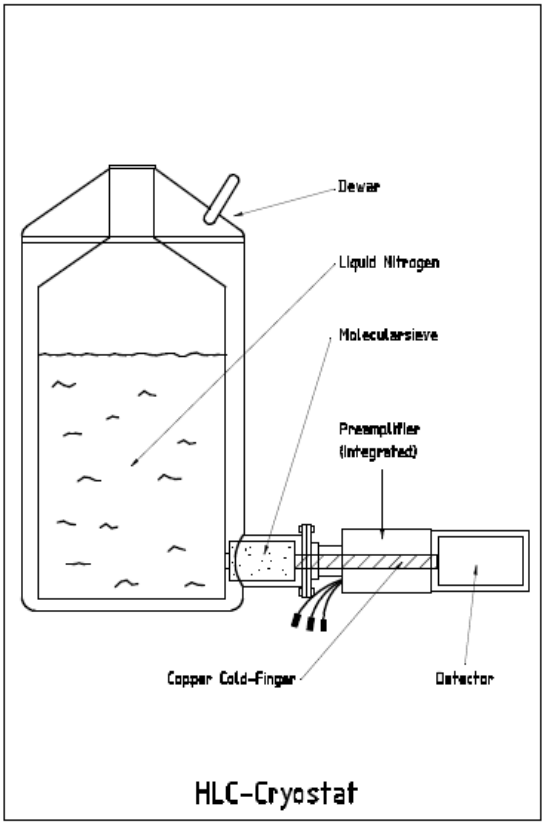
In chemical laboratory we tested this rection and we found the optimal concetration for the Cu. In this reaction under higher temperature and during the time we gets CuO from $Cu(OH)_2$. We set that the temperature of measurements shoud be above 40°C.

Than we characterise the speed of pomps, differences between pomps and real speed of pumping for various parameters. We made a table and graph to implement this information to program which control measurements at this device.

%	pomp 1 average	st. dev.	pomp 2 average	st. dev.
6	0,0648	0,0022	0,0578	0,0036
8	0,0888	0,0034	0,0846	0,0064
10	0,1140	0,0073	0,1045	0,0009
20	0,2436	0,0067	0,2209	0,0083
30	0,3824	0,0197	0,3432	0,0056
40	0,4846	0,0225	0,4646	0,0166
50	0,6249	0,0231	0,5892	0,0108
60	0,7561	0,0245	0,7075	0,0115
70	0,8906	0,0349	0,8485	0,0188
80	1,0355	0,0149	0,9754	0,0357
90	1,1681	0,0186	1,1282	0,0262
100	1,2466	0,0144	1,1984	0,0298



To get the different temperatures of reaction I designed the cooling device for liquids. As a base and storage of cryogenic liquid to cool down sample the old germanium detector was used. We unmounted electronic of detector with beryllium window to get cryostat with copper cold finger.



Pic. 2 Cryostat. Source: http://www.detector-systems.de

Pic. 3. In the laboratory.

3.Other activity

In the free time we have made pictures in Hasylab area and created 360º panoramas which can be used in future to visualisation for Hasylab (DORIS and PETRA III) web pages.

Pic. 4 Panorama of Hasylab area.