



Experiments Division

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Conceptual Design Report

Sample Environment at ALBA XAS beamline

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Overview

This document describes the sample environment to be realized at the ALBA XAS beamline. The proposed design comprises:

1. an in-situ chemical cell for high pressure / high temperature XAFS/XRD measurements;
2. a normal sample holder for in-vacuum XAFS measurements at low (LN2) / high temperatures;
3. a support structure compatible with the cell and the normal sample holder allowing for:
 - a. sample alignment with 6 degrees of freedom (aiming at reflXAFS measurements),
 - b. sample exchange among 7 sample positions in the normal sample holder,
 - c. optic paths for visual inspection or IR/Raman spectroscopy;
4. vacuum-isolated LN2 cooling scheme.

Fluorescence measurements with the in-situ cell are assumed to be conducted with the X-ray emission spectrometer [1] which collects fluorescence in the back scattering geometry.

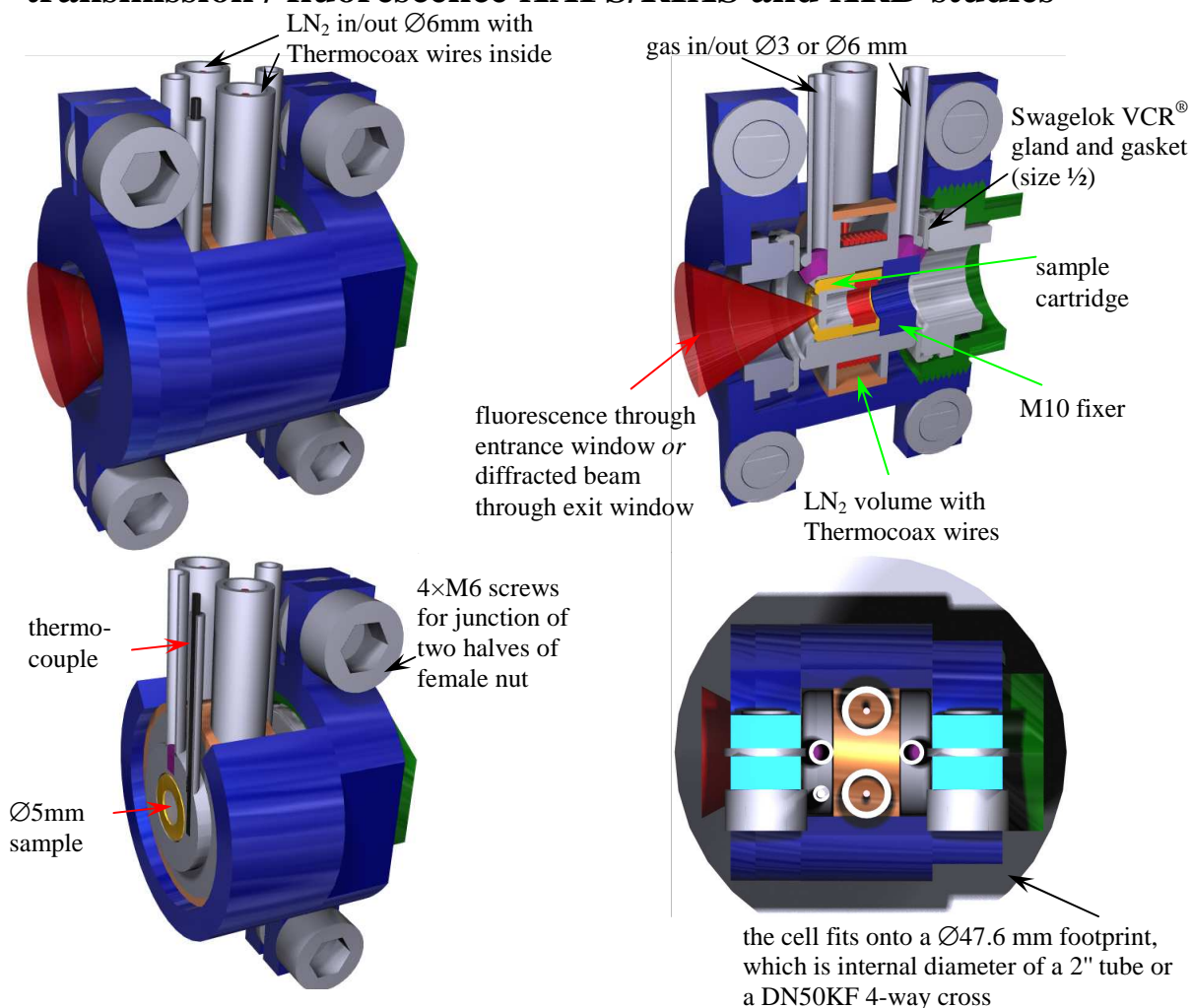
1 Mission

A good general purpose cell should span a possibly larger subspace in the multi-dimensional parametric space:

1. high/low(LN₂) temperature;
2. high(20–50 bar with corresponding safety precautions)/normal pressure;
3. low/high x-ray energies (window thickness);
4. self-supported pellets / powder samples;
5. possibility for fluorescence measurements;
6. combination with XRD;
7. combination with IR/Raman/UV-vis spectroscopies/(visual inspection);
8. small volume;
9. reliable thermometry.

The high pressure windows are intrinsically not suitable to low x-ray energy (items 2+3). All the other combinations are possible with the cell described below, except the usage of optical tools (item 7) with non-pressed powder samples which should be held by e.g. silica frits. In general, the usage of optical tools (item 7) with the proposed cell is questionable; your comments on this are welcome.

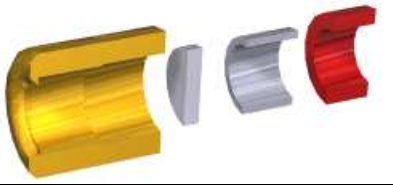
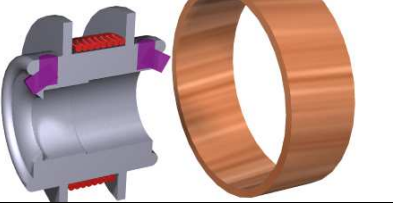
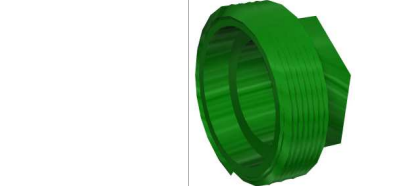
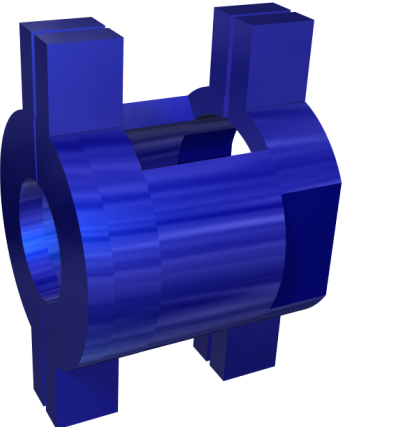
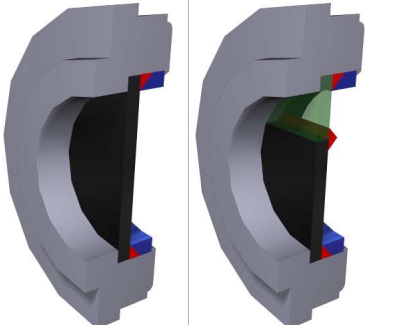
2 In-situ cell for high pressure, high/LN₂ temperature transmission / fluorescence XAFS/RIXS and XRD studies



Features:

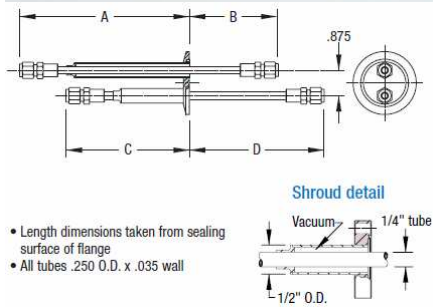
1. Temperatures from LNT up to ~700°C (as proved with a similar design with Swagelok VCR seal fittings [2]).
2. Small volume: sealed *either* with foils (Kapton or Al) at the position of the gaskets (energies down to 2.4 keV with thin windows and at normal pressure) *or* with standard ring gaskets and ad hoc Sigradur[®] windows glued by high temperature epoxy ($E > \sim 7$ keV). The sets of windows of different thickness are trivially exchanged. The mounting-dismounting cycles will not affect the windows (!) but solely the standard gaskets.
3. The sample is a self-supported pellet or a powder held by two silica frits. The gas goes *through* the sample, not around it.
4. The sample is in a cartridge: the sample mounting is simple, with the stationary cell, which is important e.g. for preparing the sample in a glove-box.
5. The opening angle for fluorescence collection (through the entrance window) or for XRD (when the cell is turned around) is 56° ($2\theta = 28^\circ$). This can be further increased up to $2\theta \sim 40^\circ$.
6. Reliable temperature measurements (thermocouple fixed near the sample).
7. Small size: the length of the cell is ~40 mm. The cell can be fitted onto a DN50KF flange.
8. A guard vacuum volume is foreseen for working (i) at low energies, (ii) at LNT without ice formation on windows and (iii) at high pressures to prevent the risk of explosion and/or contamination of ambient atmosphere.
9. The complete active volume is thermalized (there is no cold walls or windows).

Custom parts (pictures are not in common scale)

	<p>Sample cartridge:</p> <ol style="list-style-type: none"> 1) sample volume with OD=9mm, ID=5mm on one end and M6 on the other end; 2) sample Ø5mm with two optional silica frits. 3) pusher OD=5mm, ID=4mm (of various lengths for samples of different thickness); 4) M6 screw with ID=4mm hole and with a hexagonal or plain socket.
	<p>Main part of the cell:</p> <ol style="list-style-type: none"> 1) a turned body with ridges corresponding to Swagelok VCR® gaskets and holes for gas paths (shown by magenta) 2) the Thermocoax is wound around the body in the LN₂ volume; 3) the LN₂ volume is then welded to a cylindrical cover. 4) the pipes are welded to the body and the cover.
	<p>Male nut</p>
	<p>Female nut in splitted form, assembled with 4×M6 screws.</p>
	<p>High-pressure windows based onto Swagelok VCR® (size ½) glands:</p> <ol style="list-style-type: none"> 1) the inner diameter of the glands is enlarged (the blue edges are drilled out); 2) The glassy carbon Sigradur® (www.htw-germany.com) disks (black) of various thickness (depending on x-ray energy) are glued by a high temperature epoxy (red). 3) (A very preliminary idea) for simultaneous IR spectra the window consists of two segments: a smaller segment of BaF₂ or CaF₂ and a larger segment of Sigradur®. However, frits must be excluded here. May be, there should be two transparent segments, at the top and at the bottom.

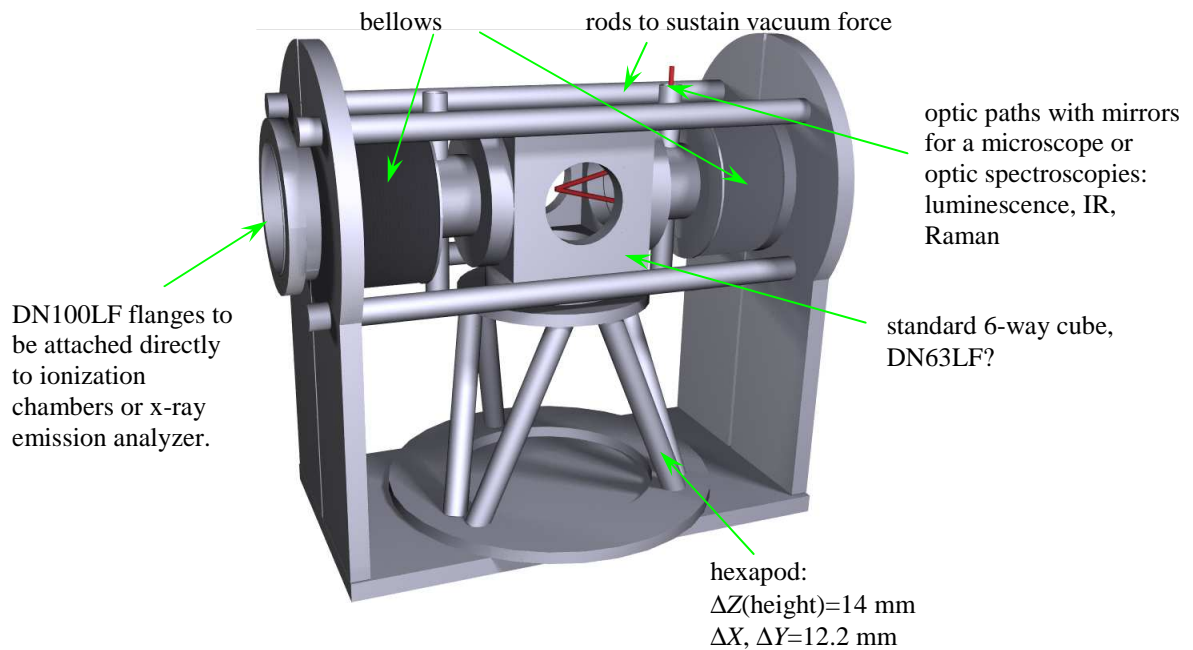
Standard parts

- 1) Swagelok VCR® size ½ gland (Ordering Number 6LV-8-VCR-3S-8TB2) shortened as required;
- 2) Swagelok VCR® size ½ gasket assembly (Ordering Number CU-8-VCR-2-GR)



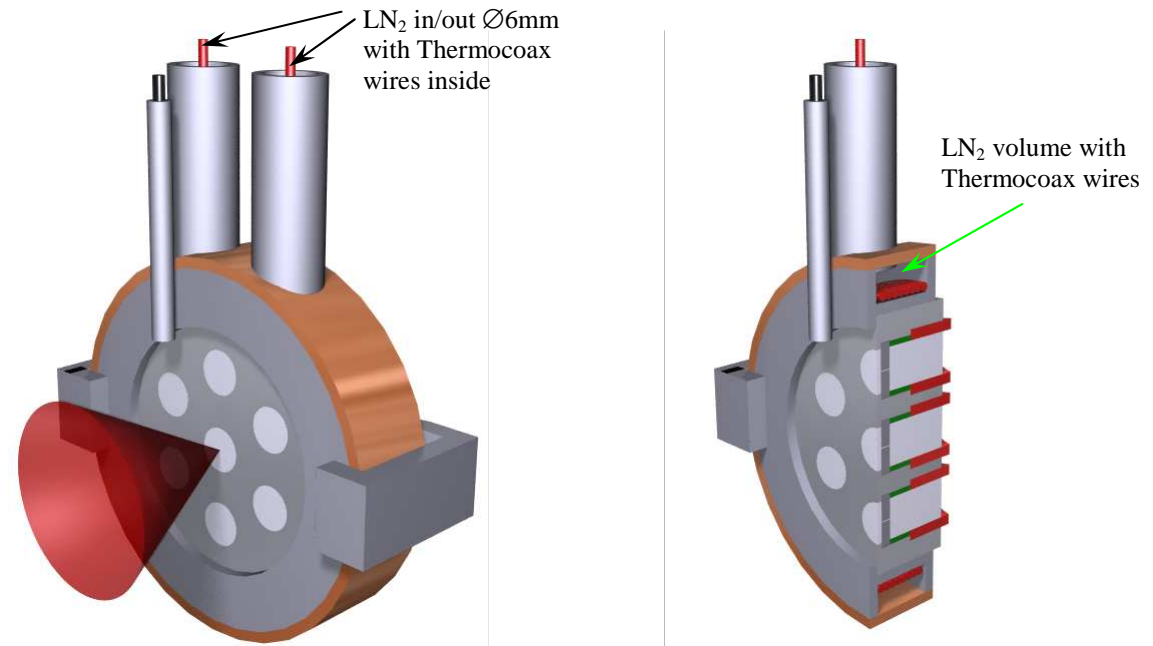
The cell is suspended by the fluid feedthroughs designed for good temperature isolation: MDC Vacuum Products Corporation (www.mdcvacuum.com).

3 Sample environment support with 6 degrees of freedom and optic paths



Distance sample-to-upstream flange = as short as possible to have the possibility to move the x-ray emission spectrometer to the sample.

4 Sample holder for in-vacuum cooling and heating



samples=small disks of $\text{Ø}5$ pushed by small cylinders of $\text{Ø}5$ and held by M6 with inner bore $\text{Ø}4\text{--}4.5$

5 Liquid nitrogen cooling

5.1 Option A: In-air cooling



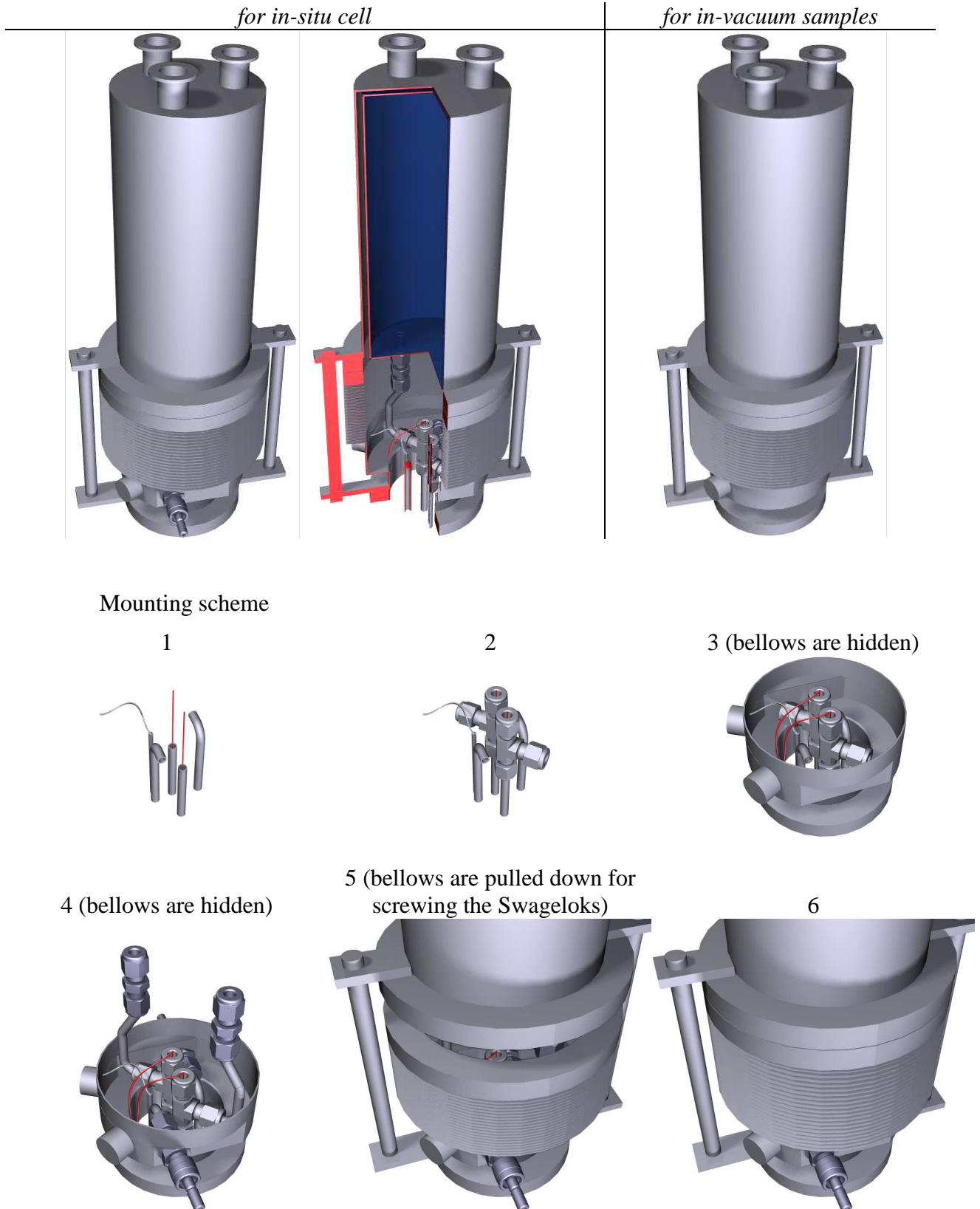
A liquid nitrogen container with gravity driven circulation. [3,4]. The isolation against moisture condensation is done by an insulation material (e.g. polyisocyanate foam).

Advantage: cheap;

Disadvantages: intensive boiling, quick run out of nitrogen (~ 1 hour for $\sim 3\text{L}$ container), the nitrogen feedthroughs have to be made of double wall pipes in order to protect the mounting flange from freezing/overheating.

5.2 Option B: Vacuum-isolated cooling

One filling of the LN₂ tank (~1L) is expected to suffice for 4-5 hours. The isolation vacuum is common with the sample vacuum. The gas connection can be done with Swagelok® Quick-Connects, as shown below.



6 3D files

The 3D files (*.rh) of all the assemblies shown above are sent in a separate zip-file. The rh-files can be viewed by a freeware viewer [Deep View \(www.righthemisphere.com\)](http://www.righthemisphere.com) with possibility of hiding/showing the parts, cross-sections, free rotating, zooming etc.

References

- [1] K. Klementiev, *Conceptual Design Report “X-ray emission spectrometer at the ALBA XAS beamline”* (2007) (see web page: <http://www.cells.es/Beamlines/XAS/>).
- [2] *unpublished(?)*, In-situ cell design of T.Ressler’s EXAFS group from Fritz-Haber Institute and TU Berlin, Germany.
- [3] F. W. H. Kampers, T. M. J. Maas, J. van Grondelle, P. Brinkgreve, D. C. Koningsberger, *An in situ cell for transmission EXAFS measurements on catalytic samples*, *Rev. Sci. Instrum.* **60** (1989) 2635.
- [4] S. Hannemann, M. Casapu, J.-D. Grunwaldt, P. Haider, P. Trüssel, A. Baiker and E. Welter, *A versatile in situ spectroscopic cell for fluorescence/transmission EXAFS and X-ray diffraction of heterogeneous catalysts in gas and liquid phase*, *J. Synchrotron Rad.* **14** (2007) 345–354.