

# Recent developments of multilayer optics for SAXS in the laboratory

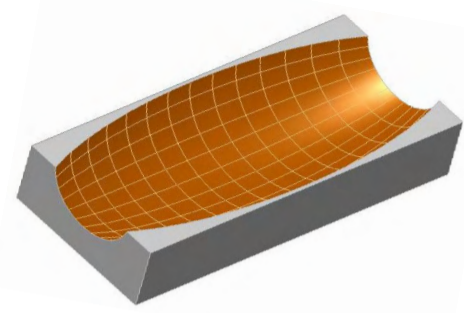
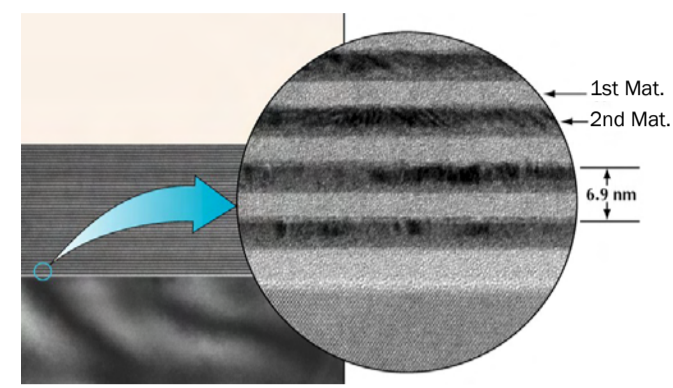
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## Aspheric multilayer coated optics preserves source brilliance

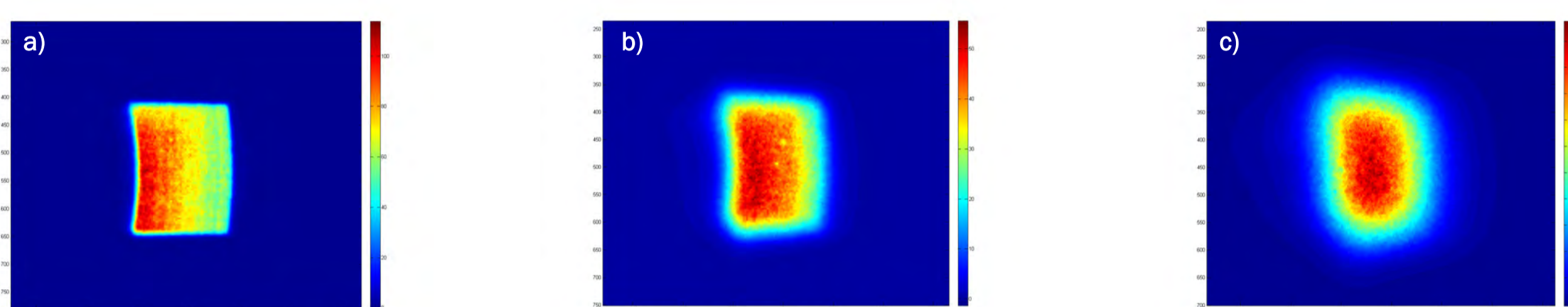
- X-ray multilayer coatings
- highly curved aspheric substrates

→ high reflectivity  
→ tailored spectral purity

→ large collection angles  
→ spot size  
→ easy to align



- FOX3D aspheric mirror delivers a lower divergence and higher beam homogeneity



Three log-scale images of the collimated Cu Ka X-ray beam taken with a CCD camera at different distances from the GeniX output to the detector (in air without collimation)

a) distance GeniX output - detector : 150 mm image size : 1.1 x 1.5 mm<sup>2</sup>  
b) distance GeniX output - detector : 650 mm image size : 1.1 x 1.5 mm<sup>2</sup>  
c) distance GeniX output - detector : 1350 mm image size : 1.1 x 1.5 mm<sup>2</sup>

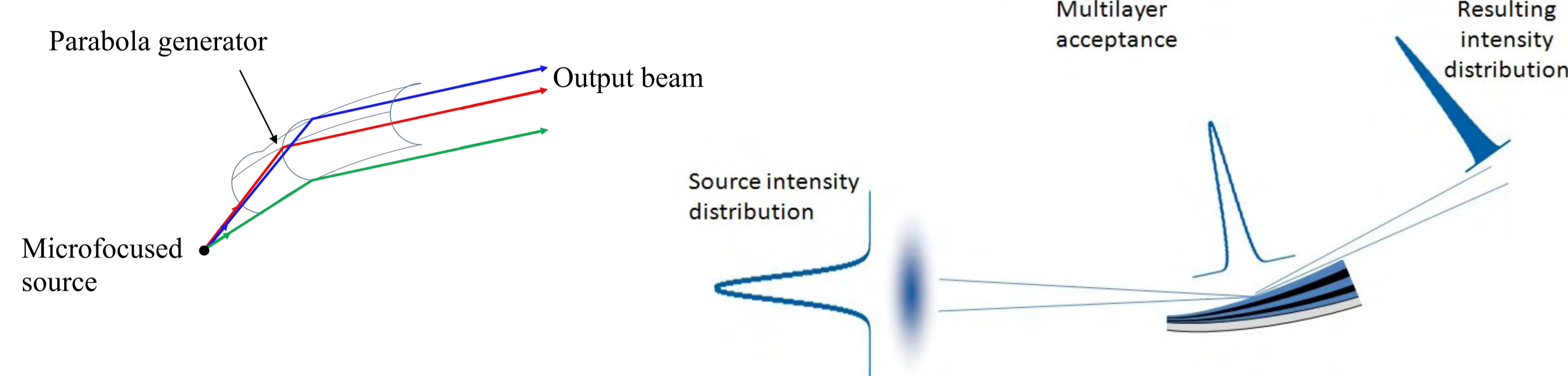
- Improved beam properties of FOX3D mirror offer higher potential for efficient collimation and accurate SAXS data acquisition
- Combination of last generation low divergence FOX3D Xenocs optic with scatterless slits will further improve the coupling of the source onto the sample.
- High brilliance low divergence optic opens new possibilities on the setup: GISAXS, WAXS...

## Highest coupling efficiency

- Comparative coupling efficiency: Montel VS FOX3D

Mirror Type	Montel Mirrors	FOX 3D Mirrors
Optic Efficiency (calculated from source-mirror distance 14cm and source size 70µm and over 100 mm mirror length)	Eff = 42 %	Eff = 62 %

Single Reflection multilayer optics provide higher flux compared to Montel mirrors



- Resulting flux outcome from the optic is a convolution of Source intensity distribution (S<sub>s</sub>) and acceptance size (S<sub>m</sub>) by the multilayer optic

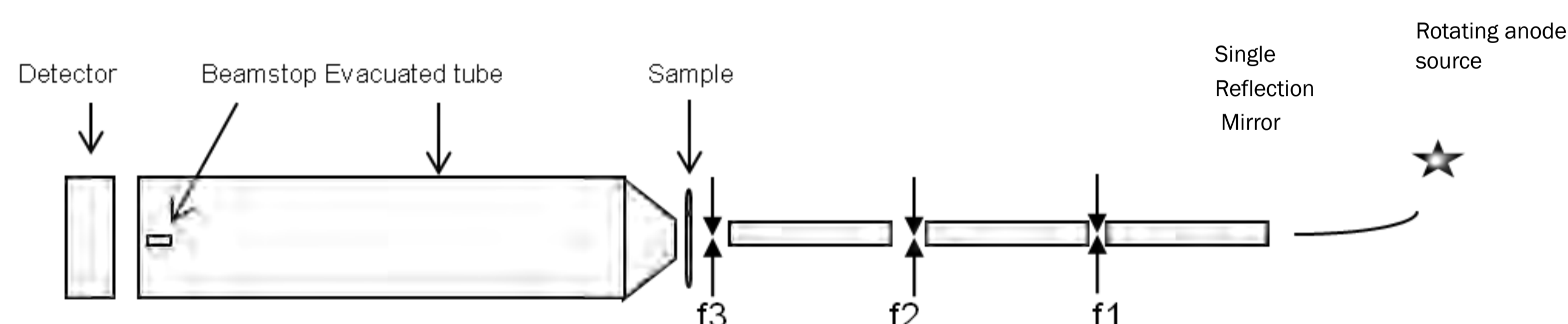
$S_m$  is a function of  $\Delta\theta$  (width of Bragg reflection of the multilayer optic) and  $p$  (source-optic distance) and is expressed as  $S_m = p \cdot \Delta\theta$

$S_r$  is resulting from the convolution of both size distribution

- Coupling efficiency is therefore expressed as (gaussian hypothesis):  $C_e = \frac{S_m}{\sqrt{S_s^2 + S_m^2}}$

## 3 pinhole SAXS camera with Cu µfocus RAG source

- Small angle x-ray scattering at the Institut Français du Pétrole, Rueil Malmaison, France



Typical distances : Sample detector: 60cm ou 160cm; Source-mirror : 12cm; mirror-f1 : 8cm f1-f2 : 80cm; f2-f3 : 67cm

Description of the experimental set up based on MM07 and Xenocs single reflexion mirror.

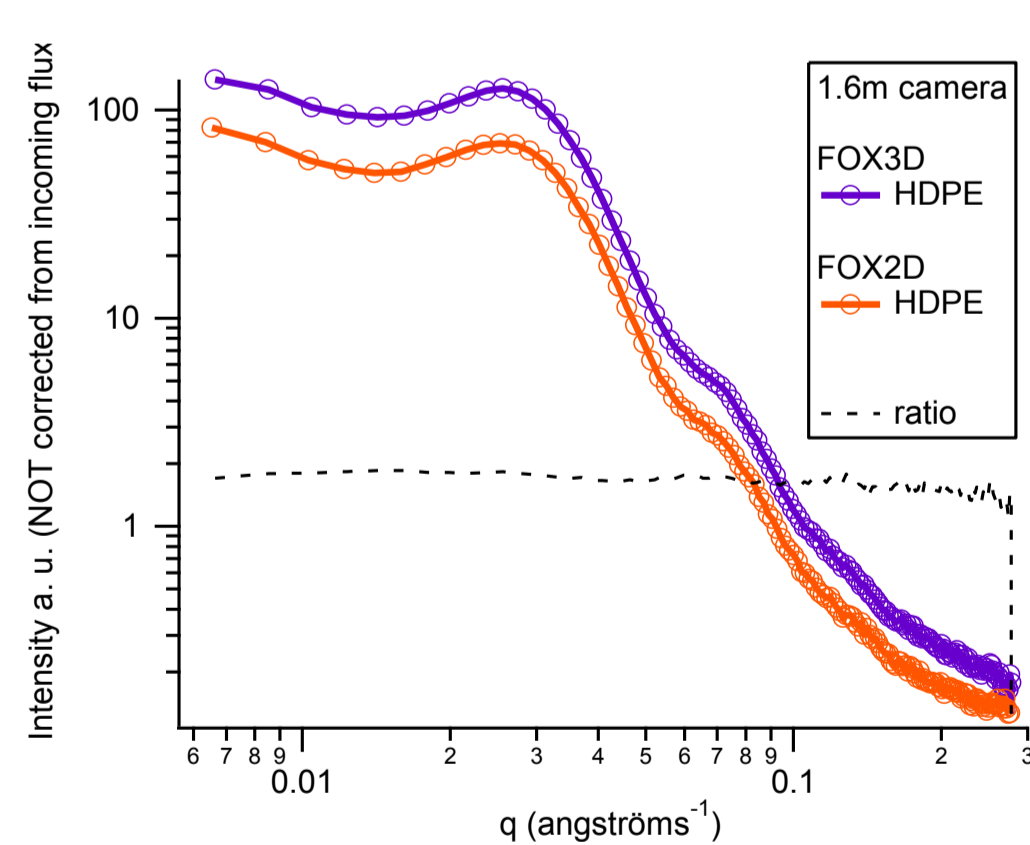


Sample changer at IFP

Slit #	Low resolution	High resolution low noise	High resolution high flux
Sample -detector distance	60 cm	160 cm	160 cm
Flux FOX2D at Sample	16.5 Mph/s	3.4 Mph/s	Not possible
Flux FOX3D at Sample	28.4 Mph/s	6.2 Mph/s	29 Mph/s
Slit f1	0.6x0.6 mm <sup>2</sup>	0.3x0.3 mm <sup>2</sup>	0.5x0.5 mm <sup>2</sup>
Slit f2	0.4x0.4 mm <sup>2</sup>	0.3x0.3 mm <sup>2</sup>	0.5x0.5 mm <sup>2</sup>
Slit f3	1.2x1.2 mm <sup>2</sup>	0.8x0.9 mm <sup>2</sup>	0.9x0.9 mm <sup>2</sup>

Various resolution configuration depending on slit settings and sample-detector distances.

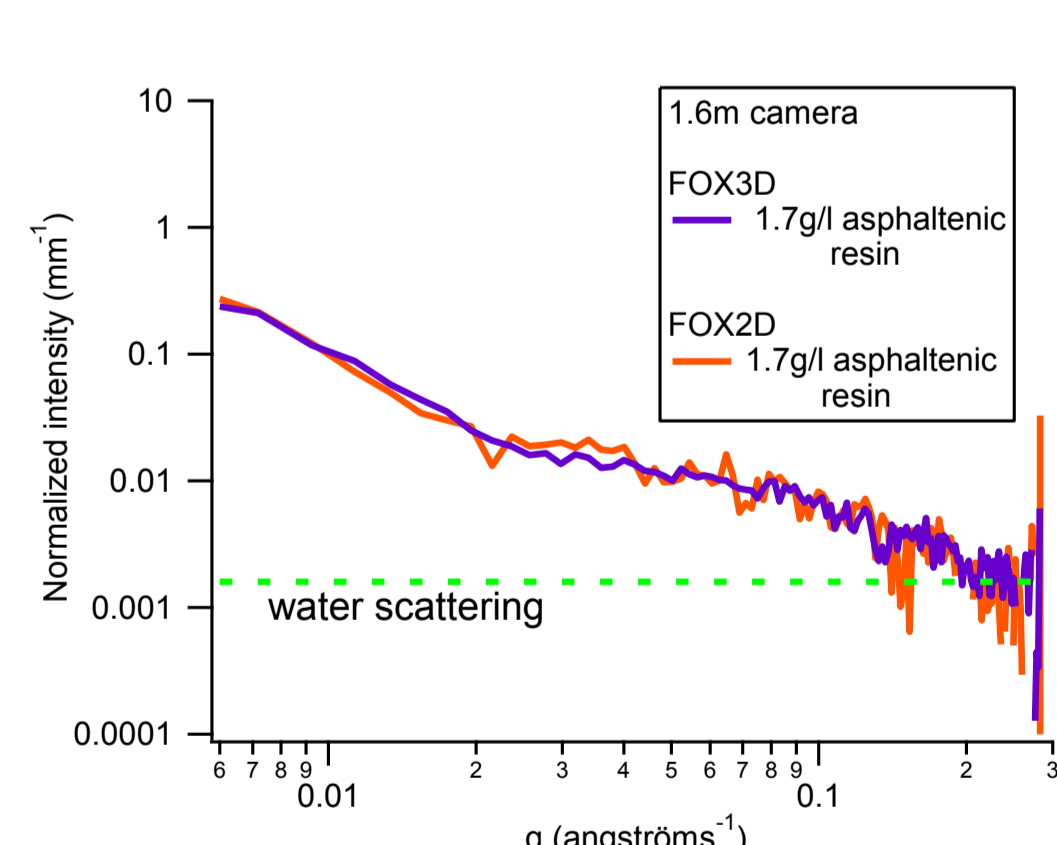
## Results on standard and typical challenging samples



Scattering curves of 1mm high density polyethylene for 1.6m long SAXS camera collected with the 2 different mirrors and with identical slit setting. Intensities are not corrected from incoming flux and show an increase of scattered intensity of around 75%. The dotted line is the ratio of FOX3D/FOX2D intensities.

- For a given slit setting the brilliance of the source is better conserved. In this high resolution/low noise mode viz. 1.6m camera length, the intensity increases by around 75%. The intensity increase allows reinforcing data interpretation by a significant reduction of the signal to noise. It opens new possibilities for the exploration of low concentrated samples.

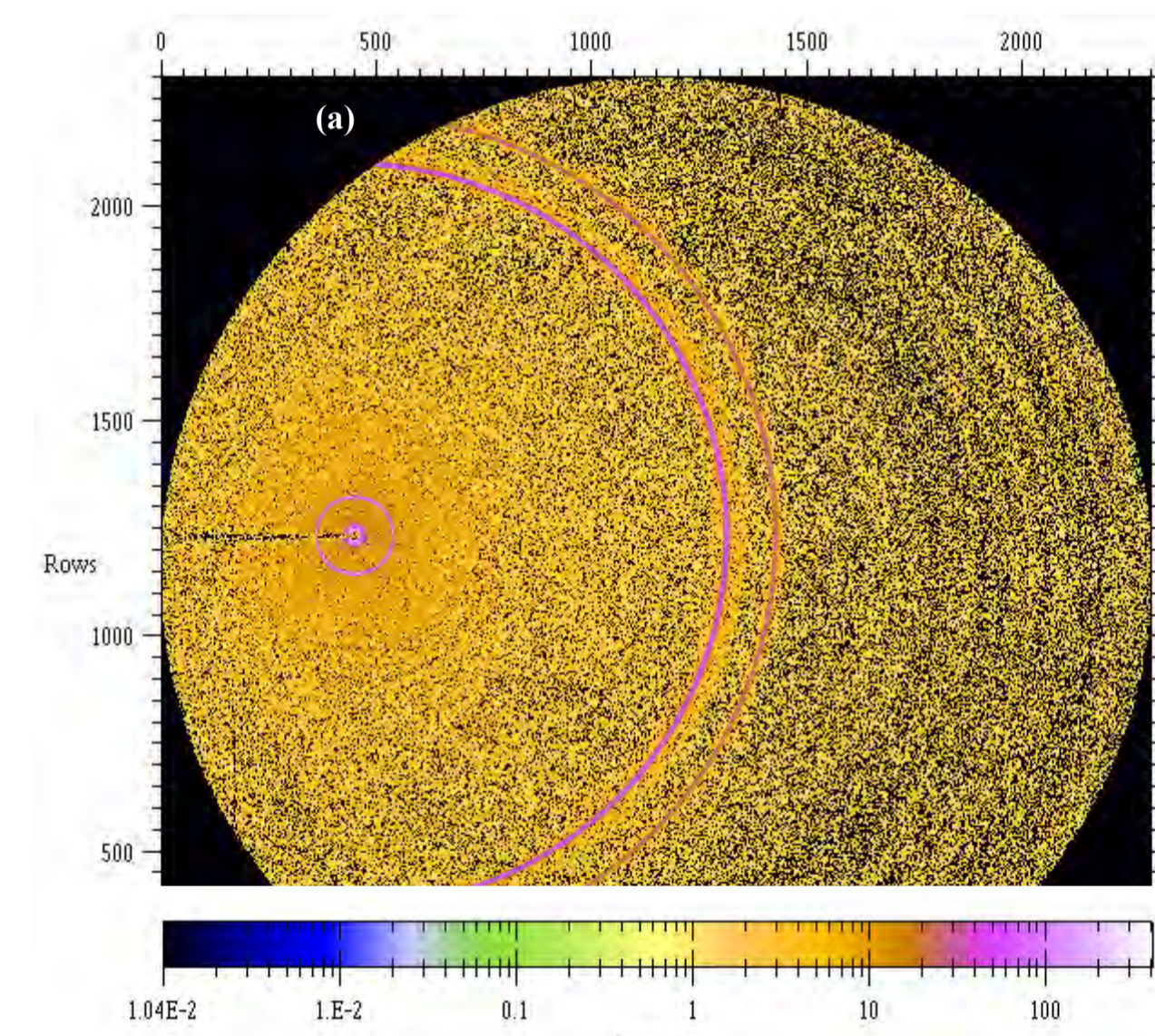
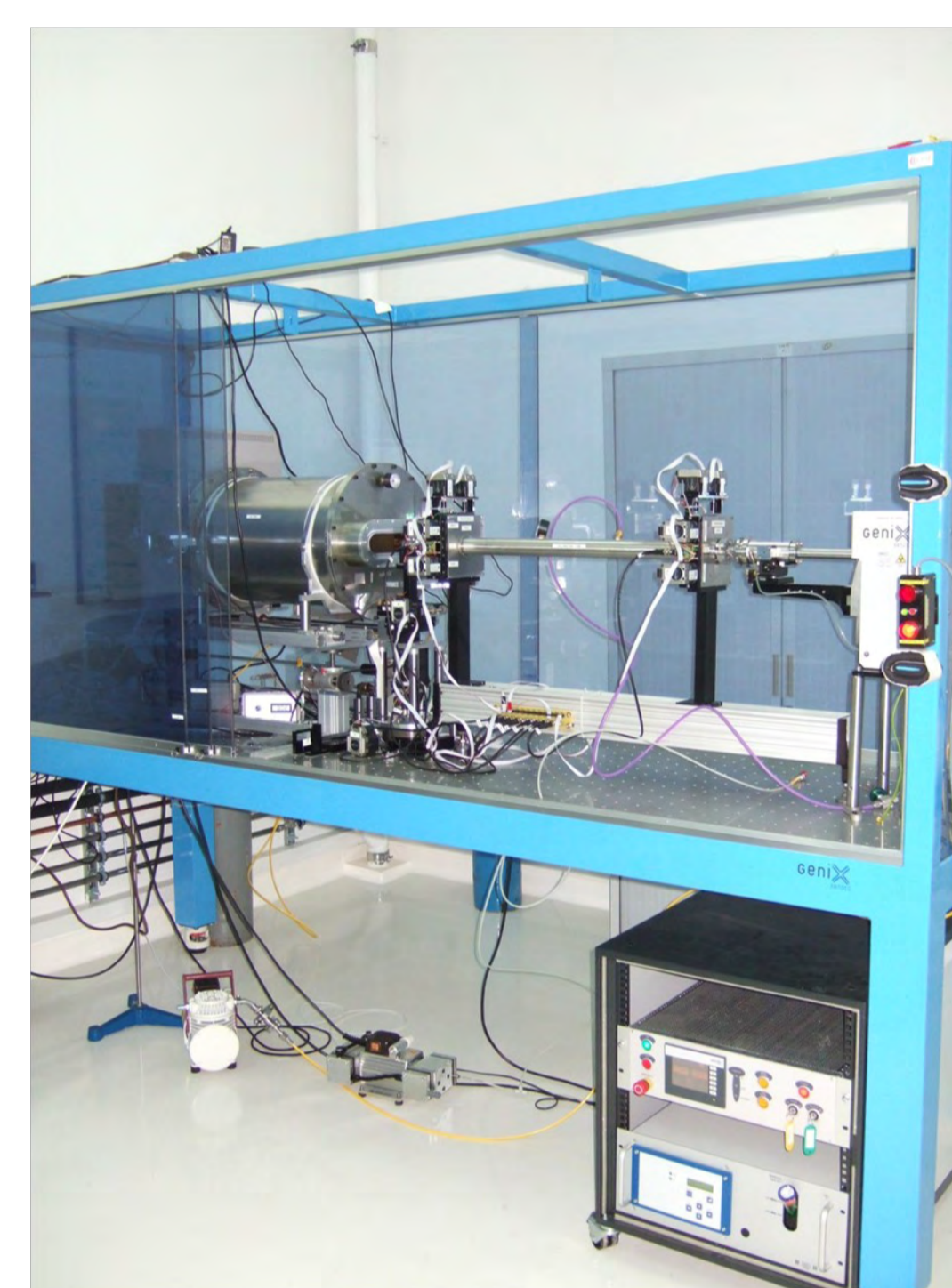
- Future combination of Xenocs FOX3D single reflection paraboloid mirror with scatterless slits optimized for Cu radiation will allow shorter collimation scheme. This will enable extension of the detection tube.



Absolute intensity scattering curves of diluted (1.7g/l) asphaltic resin solution in toluene. Data are subtracted from pure solvent (toluene). Data are collected on a 1.6m long SAXS camera for 10mm with the 2 different mirrors and with identical slit setting. The dotted line corresponds to the water scattering level illustrating the low level of resin scattering.

## Short SAXS/WAXS camera with Moly µFocus Tube

- Small angle X-ray scattering and Wide angle scattering at the Institut Chimie Séparative, CEA Marcoule, France is used for the development of advanced materials for the 4th generation nuclear plants in a sustainable development.

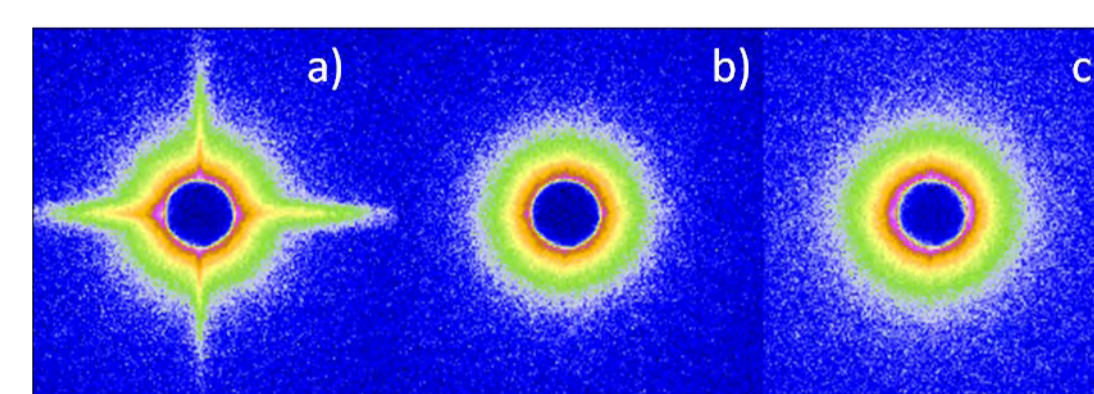


## Set-up

- GeniX Mo Low divergence: here the divergence is minimal, focusing is considered at  $\infty$
- Anti-scattering vacuum slits (special mounted edges) and flight tube
- Large (350 mm diameter) Image Plate detector from MAR Research for small and wide angle regime acquisition
- Motorized sample changer

## Scatterless slits optimized for Mo radiation

- Hybrid blades allows for background free collimation, even at intense beams



Images of empty SAXS camera of the D2AM beamline at ESRF, France. Collected on a 1.67m SAXS camera and 3mm beamstop, energy set to 17.48keV.

(a) Standard collimation, flux maximized to 3.4e10 ph/s @17.48keV on sample

(b) Same collimation, last anti scatter slits replaced by Xenocs' scatterless slits, same flux

(c) Collimation scheme simplified including Xenocs' scatterless slits allows to increase the flux up to 9.6e10ph/s with same beam focus on detector

- Hybrid blades allows increased useful flux on sample and offers a potential to further decrease the minimum q reachable by a reduction of beamstop size and/or increase of camera length (work in progress)

## Conclusions:

- FOX3D aspheric mirror delivers a « cleaner » beam with lower divergence
- FOX3D mirror enables a 50% to 80% flux gain depending on targeted Q range
- FOX3D mirror allows simpler and shorter collimation schemes
- FOX3D mirror makes lower Qmin possible and/or lower collection times.