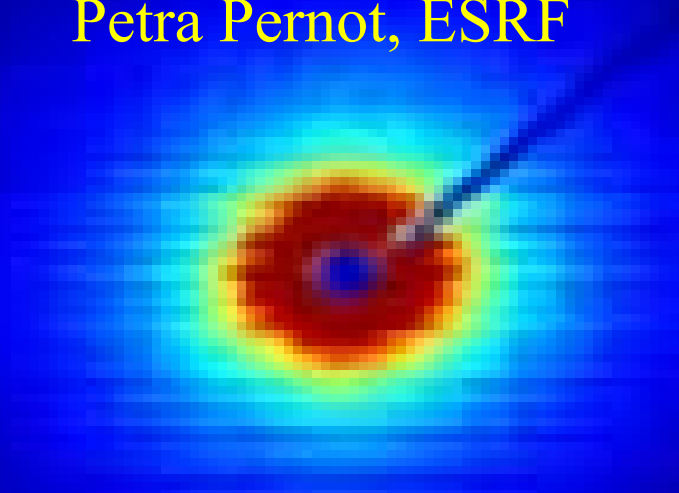


Small Angle Scattering Platform for Structural Biology

Petra Pernot, ESRF



OUTLINE:

- SAXS/SANS in Grenoble: new SAS platform of CISB
- Conversion of ID14-EH3 from MX to bio-SAXS

*MAXINF2 Integration Workshop & Coordination Meeting
Grenoble, November 28th -29th 2007*

SAS PLATFORM

- Instruments available to European biological community
- Joint proposals SAXS/SANS
- Expertise
 - use of instruments
 - experimental protocols
 - data interpretation

ILL: *Peter Timmins,
P. Callow,
R. May,*

IVMS: *M. Jamin,*

EMBL: *J. Marquez,*

IBS: *E. Pebay-Peyroula,
M. Blackledge, F. Gabel,*

ESRF: *S. Larsen, C. Ferrero,
P. Pernot, D. Spruce*

The screenshot shows a web browser window displaying the website for the Small Angle Scattering (SAS) Platform for Structural Biology at Grenoble, France. The browser title is "Untitled Document - Microsoft Internet Explorer fourni par ILL". The address bar shows "D:\SAS\boepp\to\du\ref\http". The website header includes the title "The Small Angle Scattering (SAS) Platform for Structural Biology - Grenoble, France" and lists the participating institutions: European Synchrotron Radiation Facility (ESRF), Institut Laue-Langevin (ILL), Partnership for Structural Biology (PSB), Institut de Biologie Structurale (IBS), Institut de Virologie Moléculaire et Structurale (IVMS), and European Molecular Biology Laboratory (EMBL). The main content area features a large image of the Institut Laue-Langevin facility with the text "Institut Laue-Langevin neutrons for science". Below this is a smaller image of a building with the PSB logo and the ESRF logo. The text reads: "Welcome to the the Small Angle Scattering Platform for Structural Biology!". A paragraph below states: "This site is being developed to promote the sharing of the small angle scattering expertise present at the ILL and the ESRF with the wider scientific community. In particular we will offer advice and support for sample preparation, data collection and data interpretation to maximise the potential of small angle scattering for the study of biological macromolecules in solution." The left sidebar contains a "Site Search" box and a list of navigation links: Platform Information, Introduction to Small Angle Scattering (SAS), Neutrons vs X-rays, SAS Instrumentation, Data Processing, Data Analysis, Modeling, Sample Requirements, Facilities Available as Part of the SAS Platform, Instruments (ESRF ID2, ESRF BM28, ILL D11, ILL D22), Deuterium Lab, Software, Computing Resources, Contacts, Glossary, and Useful Links (Manuals, Selected Publications, Proposal Submission). The footer includes "About Us", "Site Map", "Privacy Policy", "Contact Us", and "©2007 SAS Team".

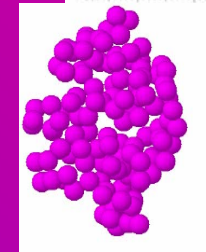
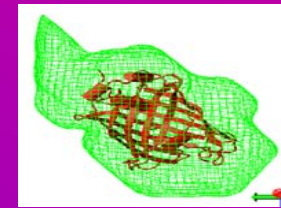
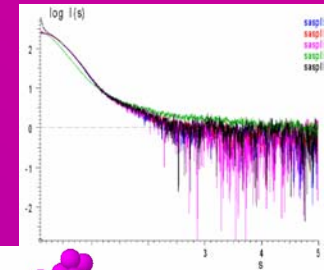
SMALL ANGLE SCATTERING

= a technique for studying structure and association at low resolution in solution under normal biochemical conditions

Information from SAS:

- model independent parameters R_g , $I(0)$
- *ab initio* shape determination
- rigid body modelling

- molecular shape
- molecular interactions
- kinetics

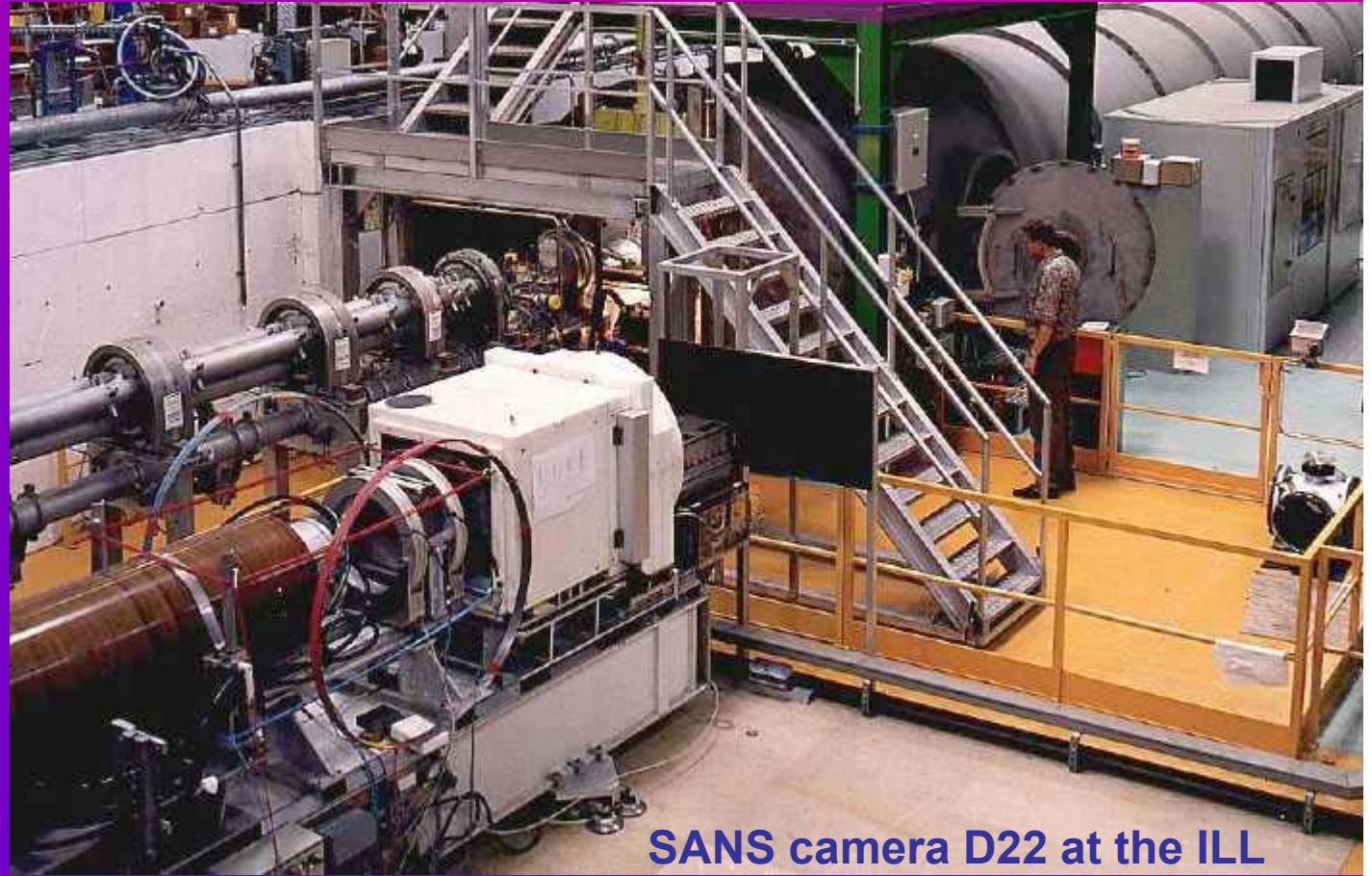


- complementarity of **SAXS** and **SANS**

	SAXS	SANS
volume	small < 50 ml	larger ~ 300 ml
concentration	> 0.1 mg/ml	> 1 mg/ml
measuring time	short ~ s	longer ~ m÷h
radiation damage	yes	no
contrast variation	no	yes
sensitive to salts, denaturants	yes	no

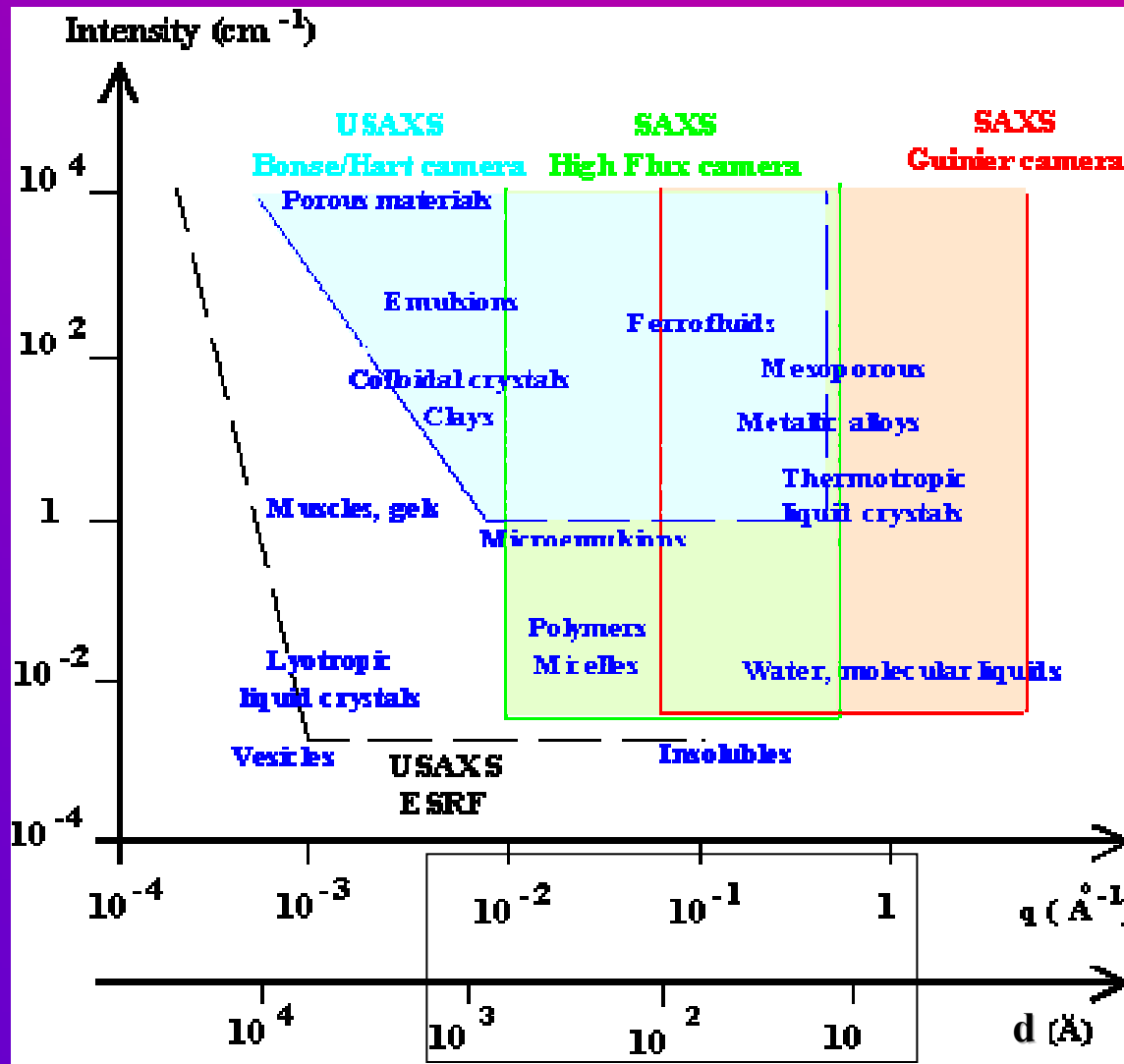
INSTRUMENTS FOR SAS

- SAXS – ESRF
 - ID2
 - ID14-3 conversion: budget ESRF, scientist EMBL
- SANS – ILL
 - D11
 - D22
 - D33 project
- Instruments available via proposals to ESRF and ILL



SANS camera D22 at the ILL

ID14-EH3 conversion to bio-SAXS



GOAL = investigation of biological macromolecular complexes in solution

q_{\min} and q_{\max} defined by the experimental setup:

- $\lambda = 0.931 \text{\AA}$, ($E = 13.3 \text{ keV}$)
- detector diameter $\sim 20 \text{ cm}$,
- sample-to-detector distance D : 1-3 m
- beam stop size $\sim 2 \text{ mm}$

	$D = 1 \text{ m}$	$D = 3 \text{ m}$
q_{\min}	0.013 \AA^{-1}	$4.3 \times 10^{-3} \text{ \AA}^{-1}$
d_{\max}	47 nm	140 nm
q_{\max}	1.34 \AA^{-1}	0.45 \AA^{-1}
d_{\min}	4.68 \AA	14 \AA

REQUIRED MODIFICATIONS

q resolution limited by beam divergence, beam size, parasitic scattering, detector PSF and dynamic range,...

EXPERIMENTAL HUTCH

MEZZANINE

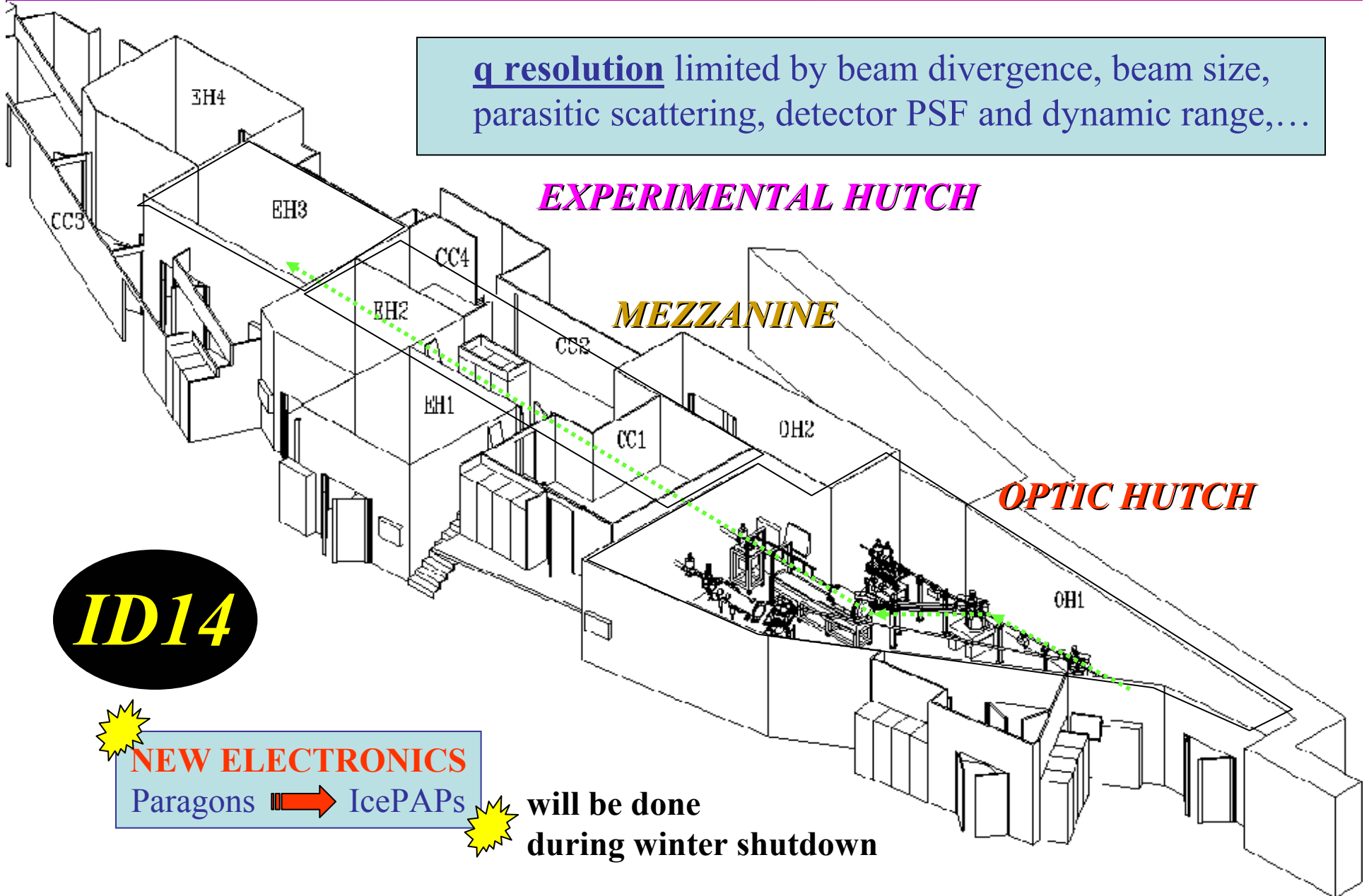
OPTIC HUTCH

ID14

NEW ELECTRONICS

Paragons → IcePAPs

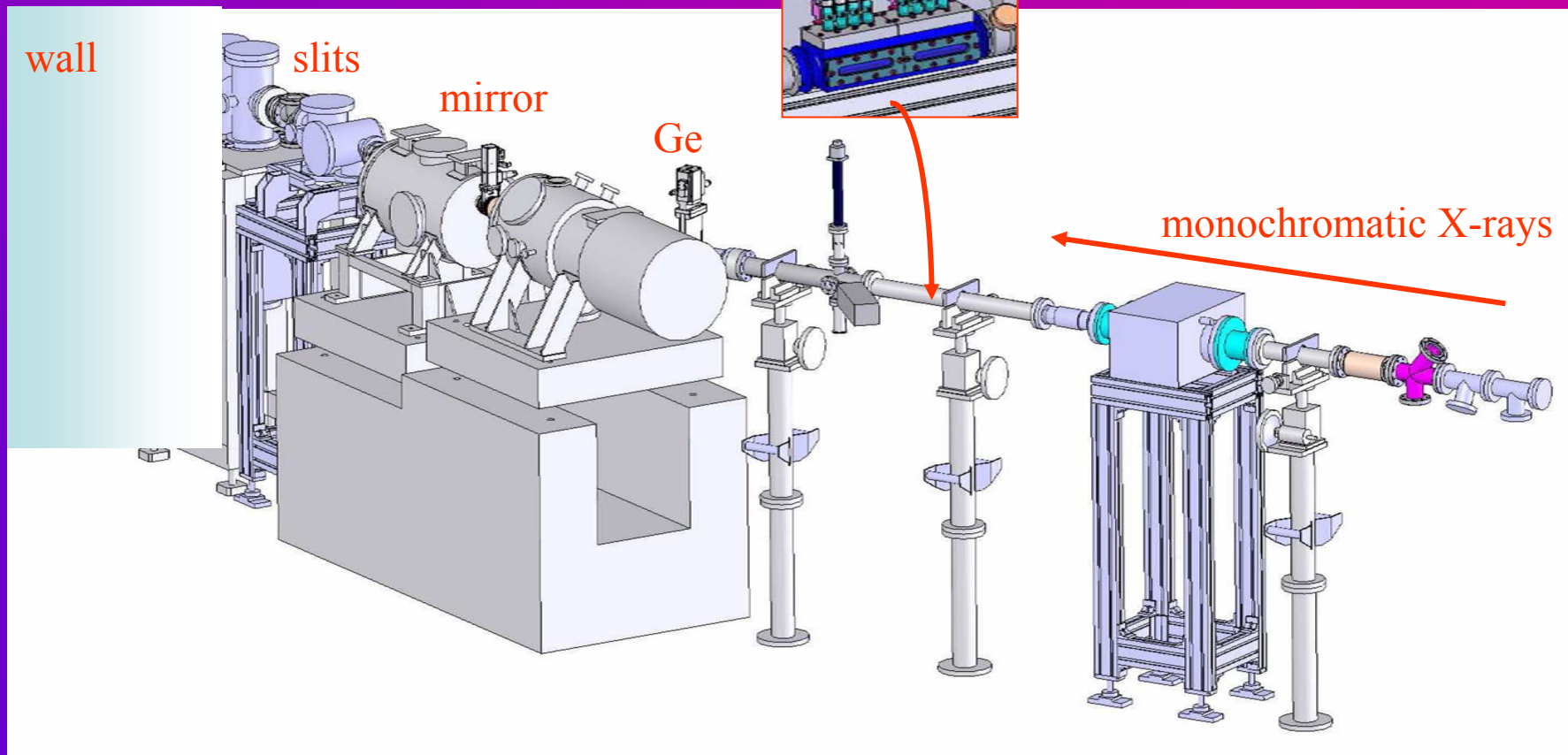
will be done during winter shutdown



OPTIC HUTCH

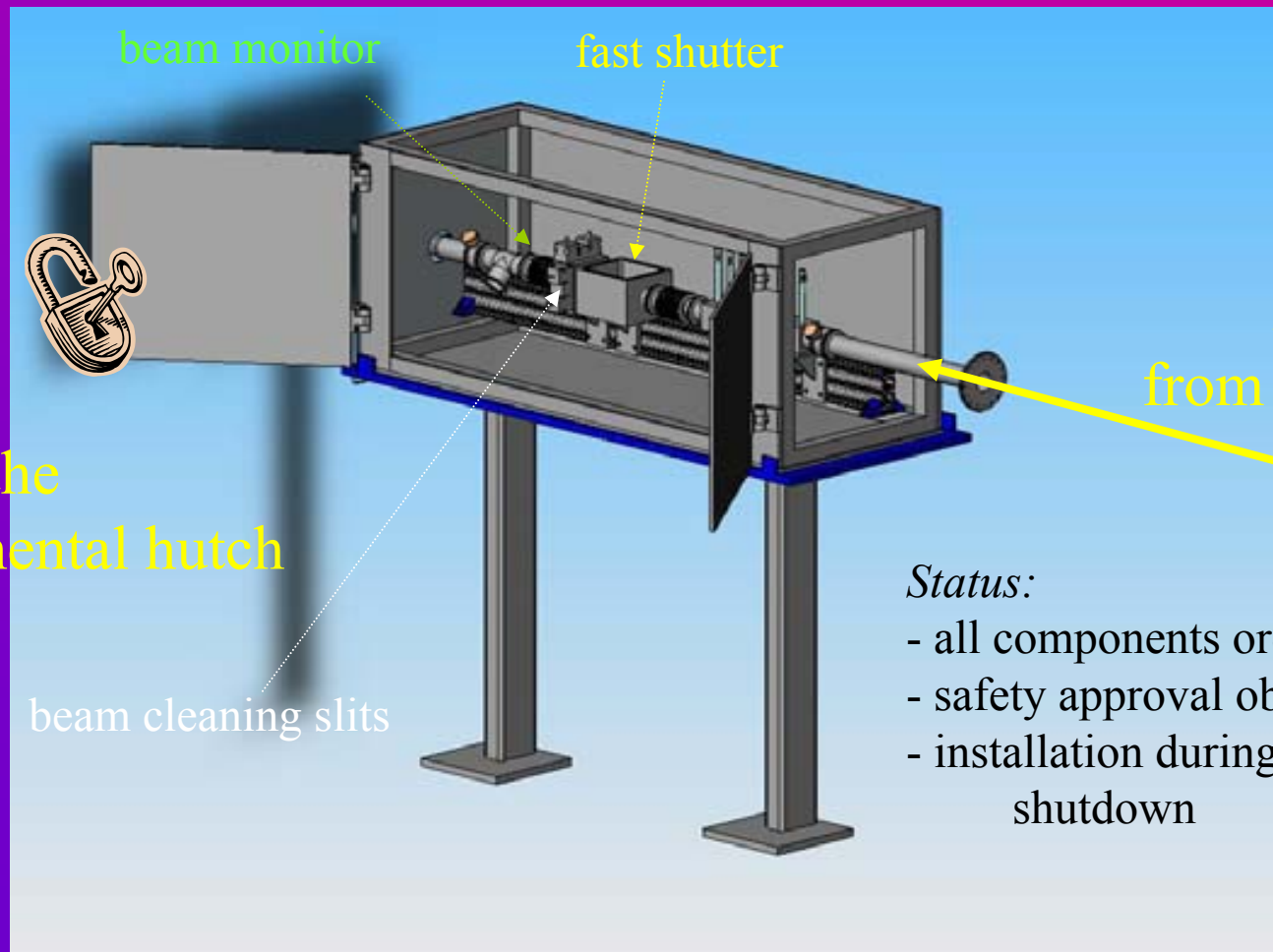
- beam defining slits after the mirror
- fluorescent screen after the slits
- attenuators before Ge crystal

} DONE during October shutdown



MINIHUTCH in mezzanine

~ 4 m from the sample



beam monitor

fast shutter

X-rays
from Optic hutch

wall of the
Experimental hutch

beam cleaning slits

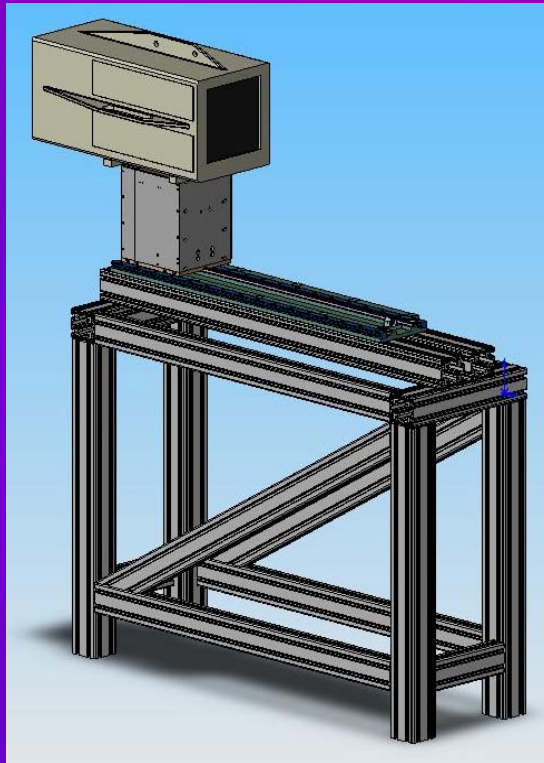
Status:

- all components ordered
- safety approval obtained
- installation during the winter shutdown

EXPERIMENTAL HUTCH

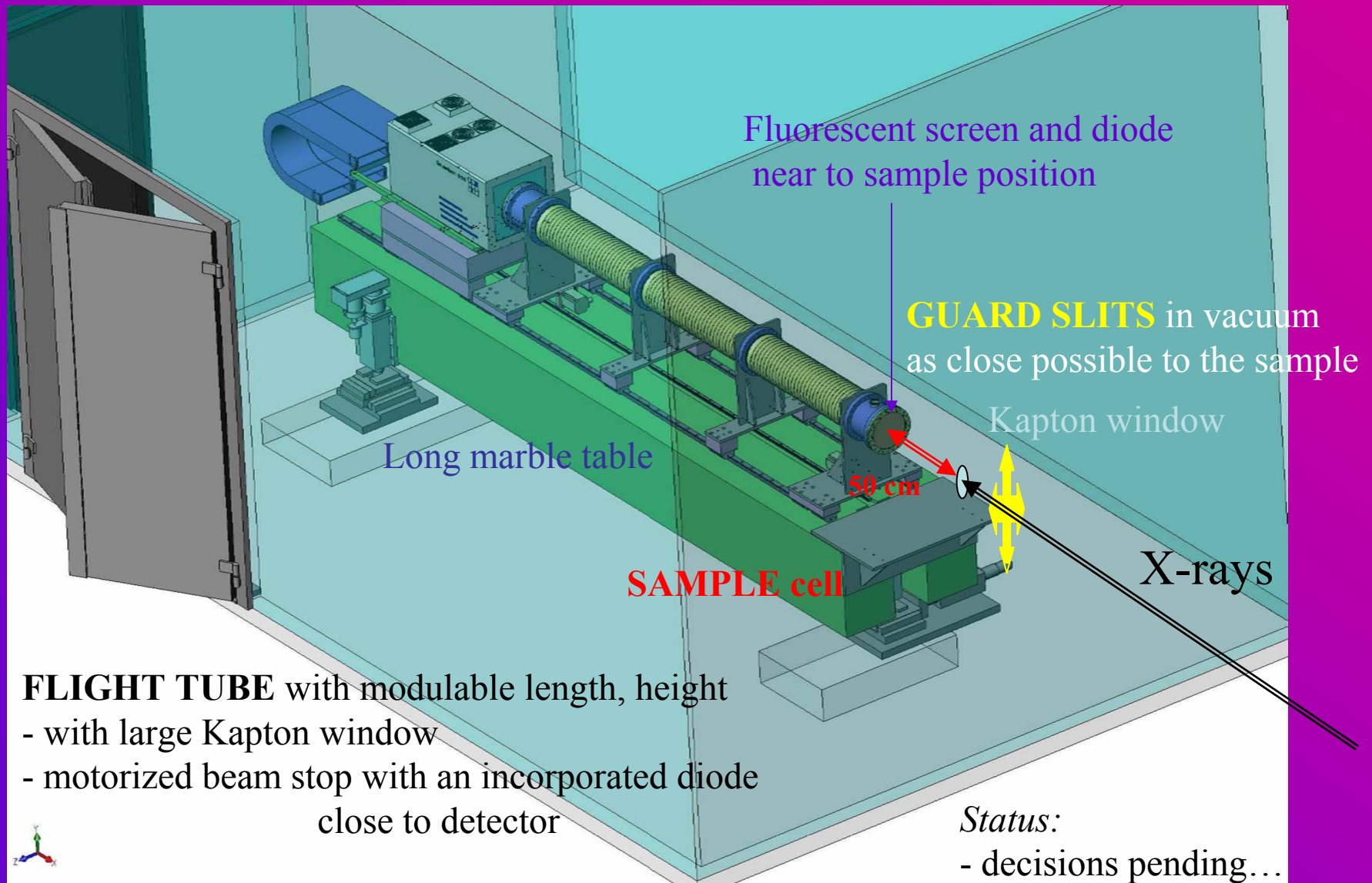
January-March 2008

- commissioning of new electronics of Optic hutch, minihutch equipments and feasibility SAXS tests
- equiped as it is, however without Be-window, with new table to hold detector ~ 2.6 m from the sample (small cell on microdiff), flight tube, motorized beamstop



EXPERIMENTAL HUTCH

from March 2008: installation of new equipments and commissioning



DETECTOR(S)

Present on the beamline: **Q4R ADSC**

- 2×2 array with no more than 400μ slit between detecting areas
- 2304×2304 pixels, $82 \mu\text{m}^2$ each
- active area: $188 \times 188 \text{ mm}^2$
- readout time: lowest noise $\sim 9 \text{ s}$
high speed $\sim 3 \text{ s}$
- 16 bits



FRELON

Fiber optically coupled (taper) Frelon CCD based on **Kodak KAF-4320** image sensor

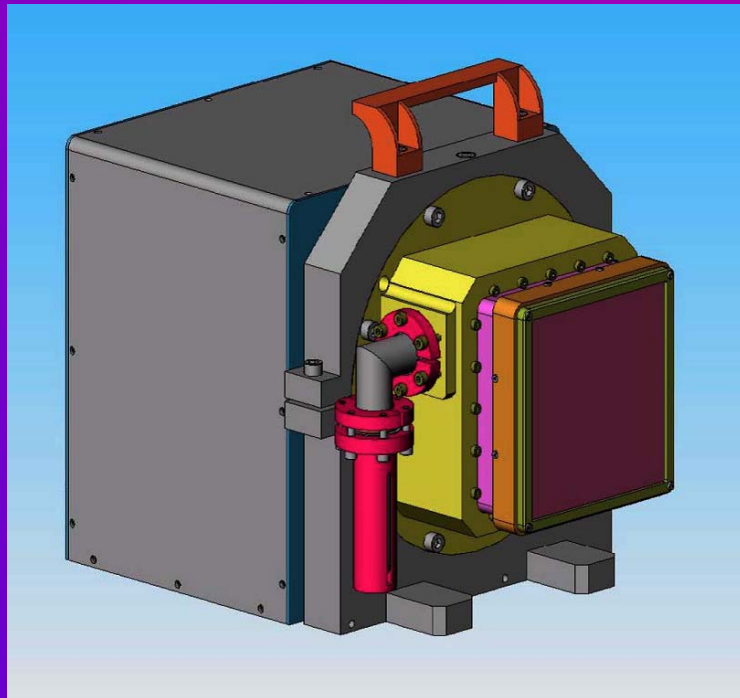
Active area: 10 cm × 10 cm

Full dynamic range: 16 bit (14,5 bit above the noise)

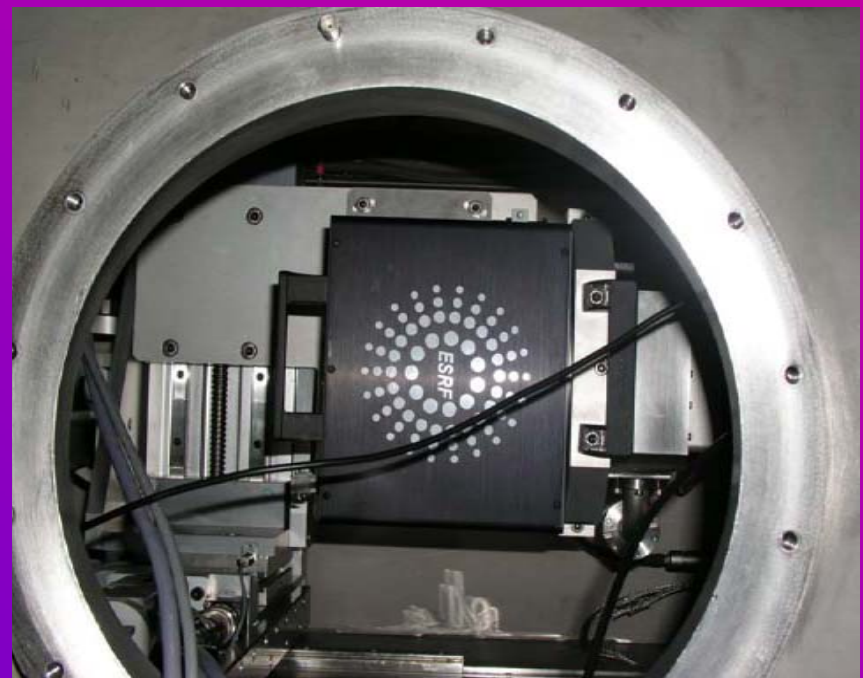
Spatial resolution: 80 μm (49 μm pixel)

Full frame rate of 3 frames/sec (2048 × 2048)

Detector translation table to cover required q-range



ID02 installation



Bruker AXS VANTEC-2000



Gas-filled detector bought by Detector pool

14 cm x 14 cm active area
100 msec Snapshot
spatial resolution = 70 μm
local and global count rate of $>800,000$
high dynamic range of $>10^7$
high area uniformity

Inert counting gas

 **no maintenance required**

Radiation hard

 **can withstand primary beam**

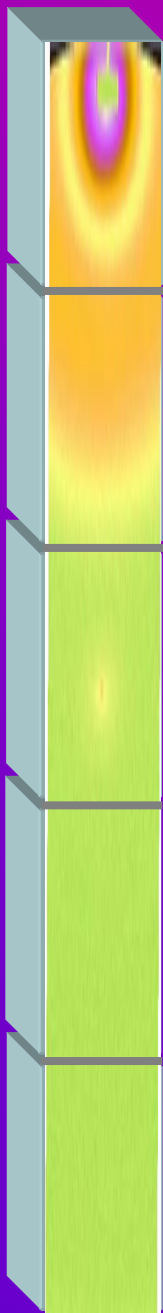


all advantages of a sealed, gaseous, photon-counting detector with real-time mode

PILATUS

- high dynamic range: $> 1:1\,000\,000$ (20bits)/pixel/image
- readout time ~ 3 ms
- no electronic noise
- point spread function = 1 pixel
- electronically gateable:
 - no mechanical shutter needed
- pixel size: $172 \times 172 \mu\text{m}^2$
- single module: 195×487 pixels
- active area single module:
 - $33.5 \times 83.8 \text{ mm}^2$
- counting rate: $> 2 \times 10^6/\text{s}/\text{pixel}$
- framing rate: 200 Hz
- energy range: 3 –30 keV

440 mm



PILATUS 2M

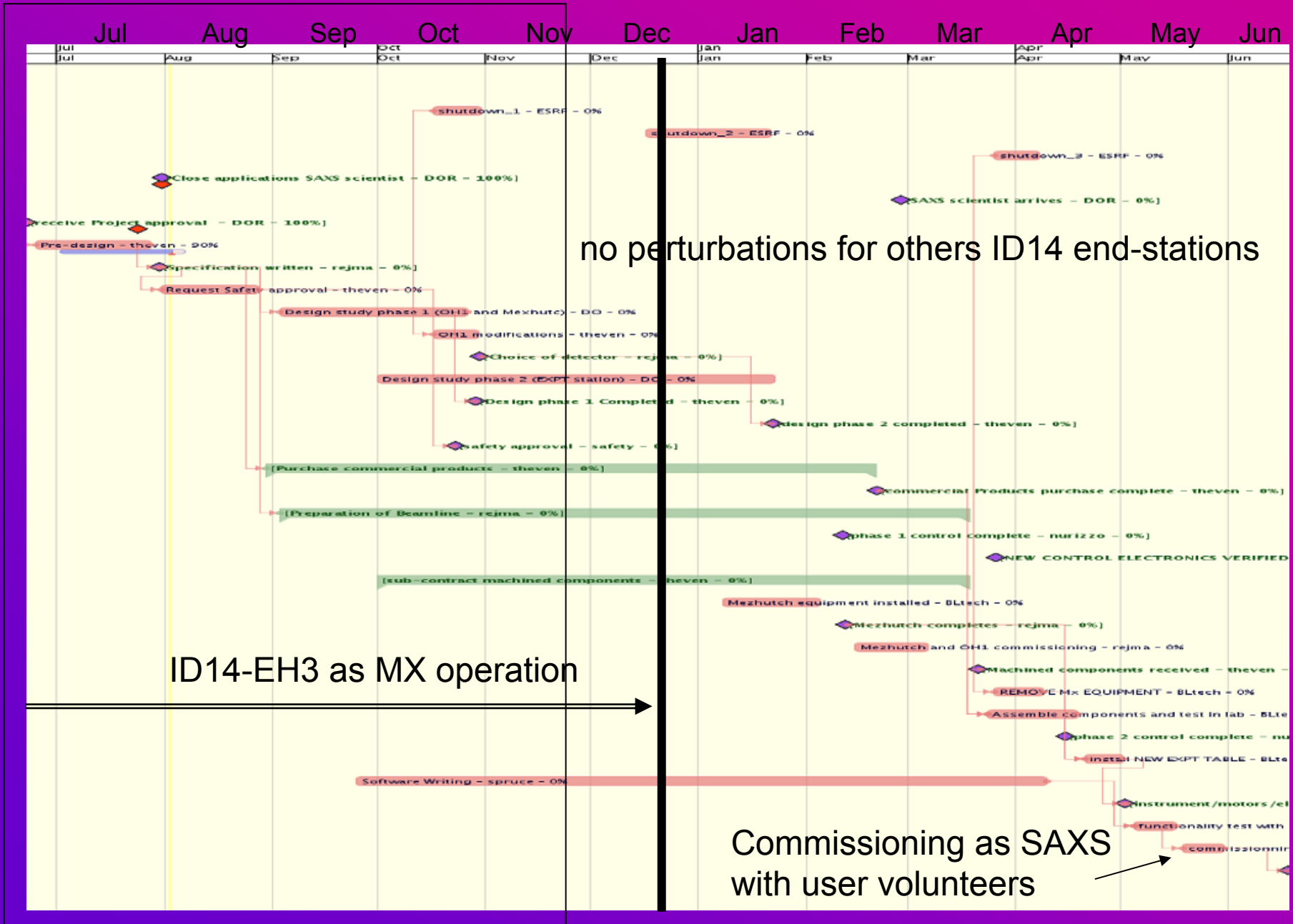
24 modules

active area = $254 \times 289 \text{ mm}^2$

price ~ 600 k€, delivery time ~ 1 year

‘Stripe’ (5 modules, 500K) bought by the bio-SAXS beamline at EMBL Hamburg  borrow to the ESRF from January to September 2008

Timetable 2007-8



ID14-EH3 conversion to bio-SAXS

people involved

Cooperation with EMBL Hamburg: *D. Svergun, M. Rössle*
- actual bio-SAXS beamline at Doris, future at PETRA III
- PILATUS, 'mini' Sample Changer, data analysis software, etc.
Via EMBL Grenoble: *F. Cipriani, A. McCarthy*

Advice from ID02 and BM26 ESRF: *N. Theyencheri, P. Bösecke, and W. Bras*

MX 'realisation team': *P. Theveneau, D. Spruce, T. Mairs, D. Nurizzo*

Still waiting for EMBL expert...: *NN*

