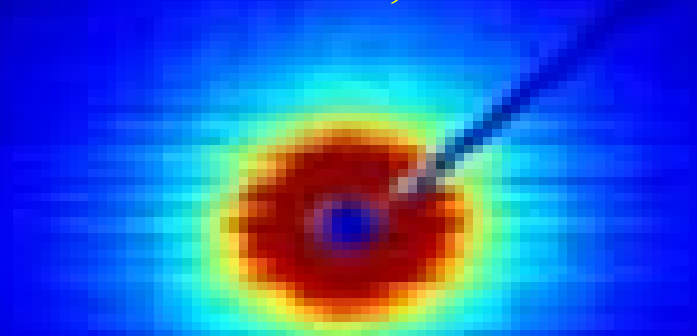
# Small Angle Scattering Platform for Structural Biology

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*

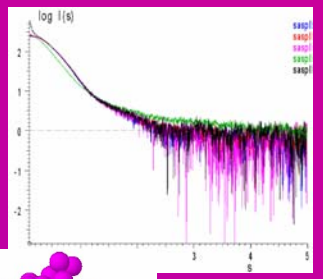
# *SAS PLATFORM*

- 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*

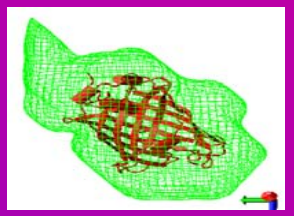
# *SMALL ANGLE SCATTERING*

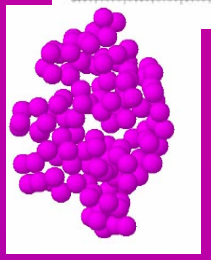
= a technique for studying structure and association at low resolution in solution under normal biochemical conditions



**Information from SAS:**

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





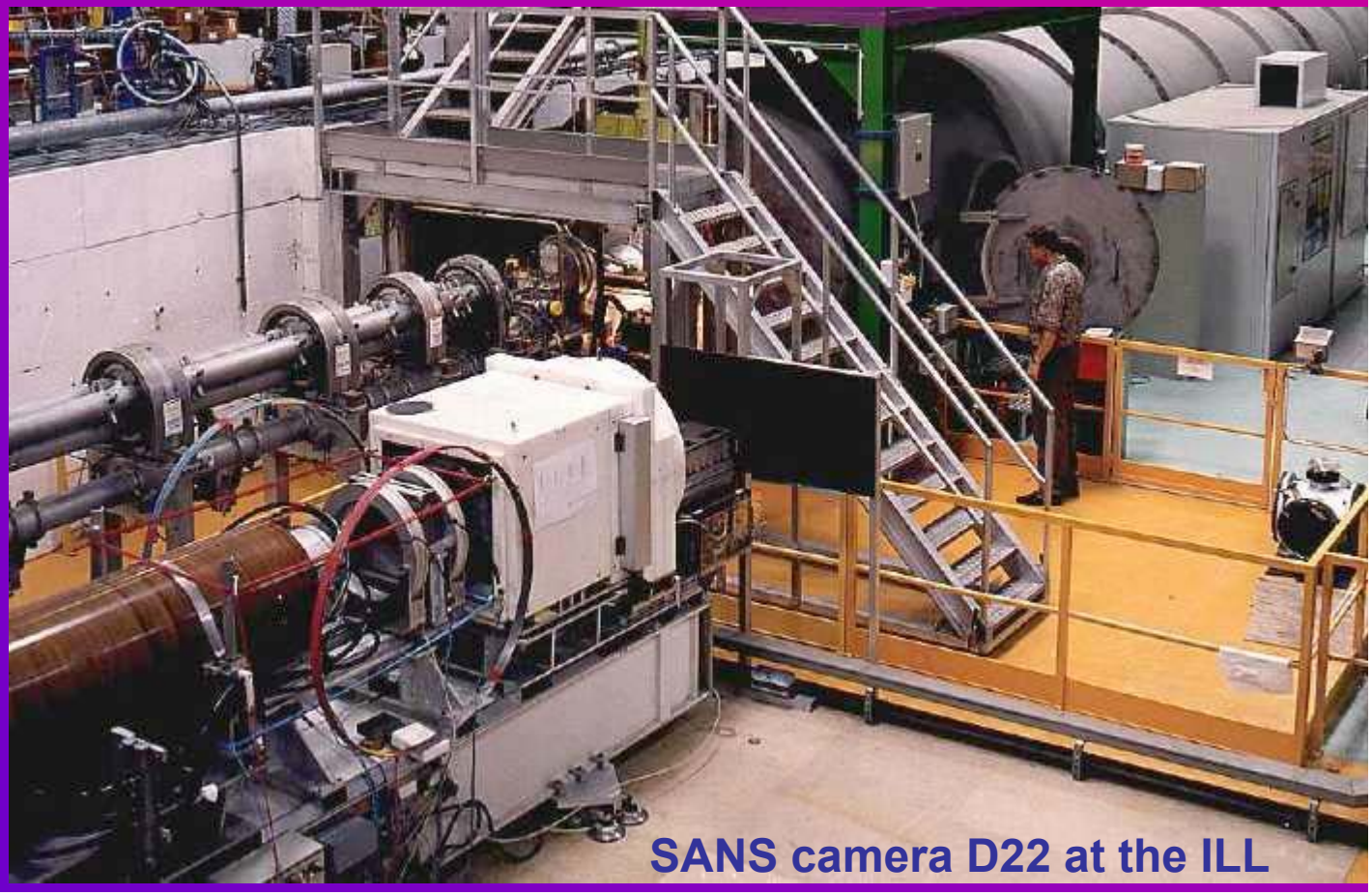
- molecular shape
- molecular interactions
- kinetics

- 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 |

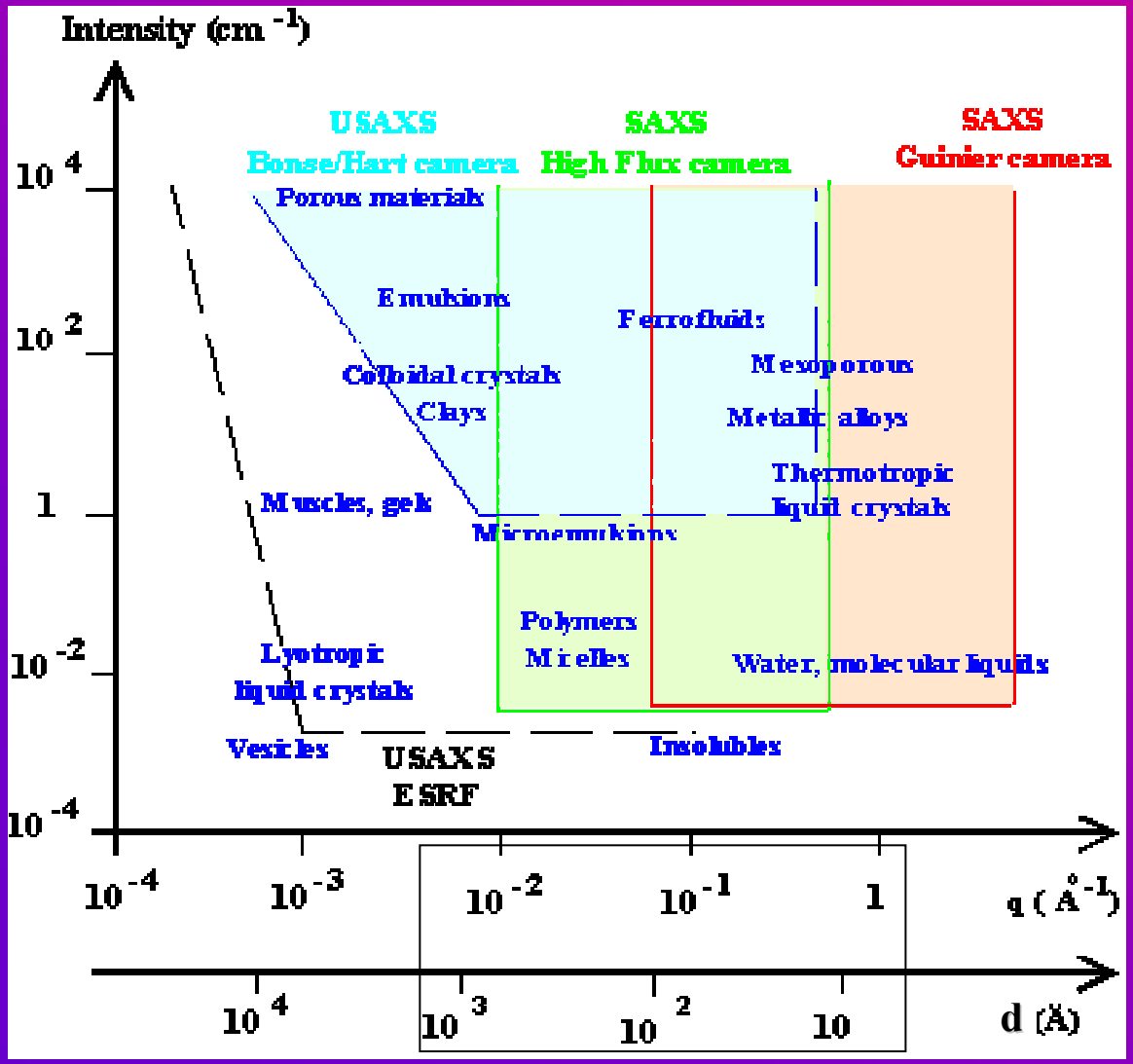
# *INSTRUMENTS FOR SAS*

- 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

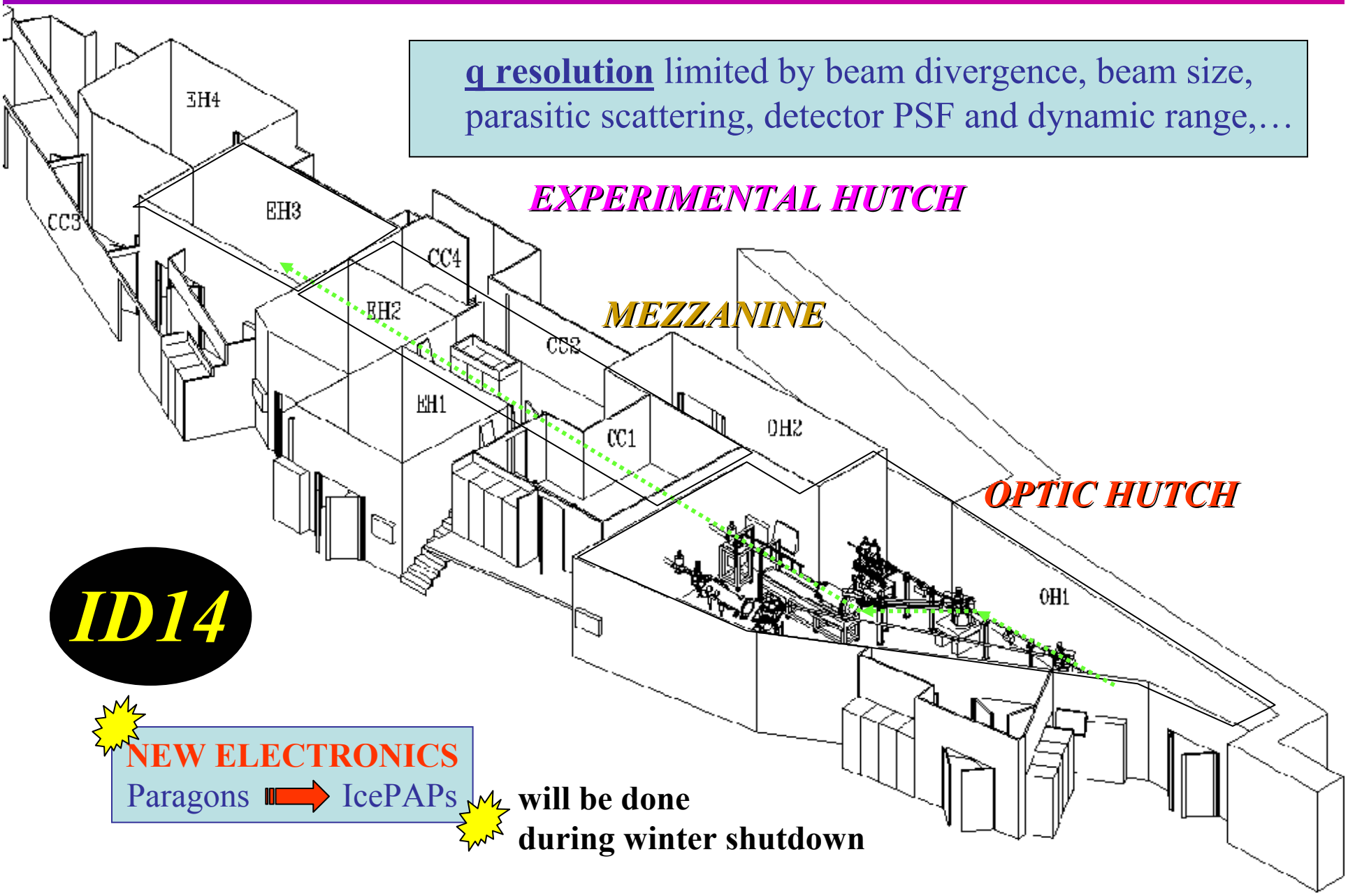
$q_{min}$ and $q_{max}$ defined by the experimental setup:

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

| | D = 1 m | D = 3 m |
|---|---|---|
| $q_{min}$ | 0.013 Å$^{-1}$ | 4.3 × 10$^{-3}$ Å$^{-1}$ |
| $d_{max}$ | 47 nm | 140 nm |
| $q_{max}$ | 1.34 Å$^{-1}$ | 0.45 Å$^{-1}$ |
| $d_{min}$ | 4.68 Å | 14 Å |

# REQUIRED MODIFICATIONS

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




**ID14**

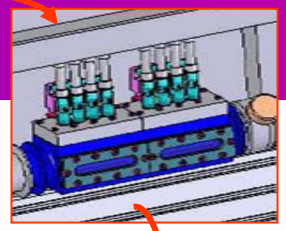
**NEW ELECTRONICS**
Paragons ⇒ IcePAPs

**will be done during winter shutdown**

# *OPTIC HUTCH*

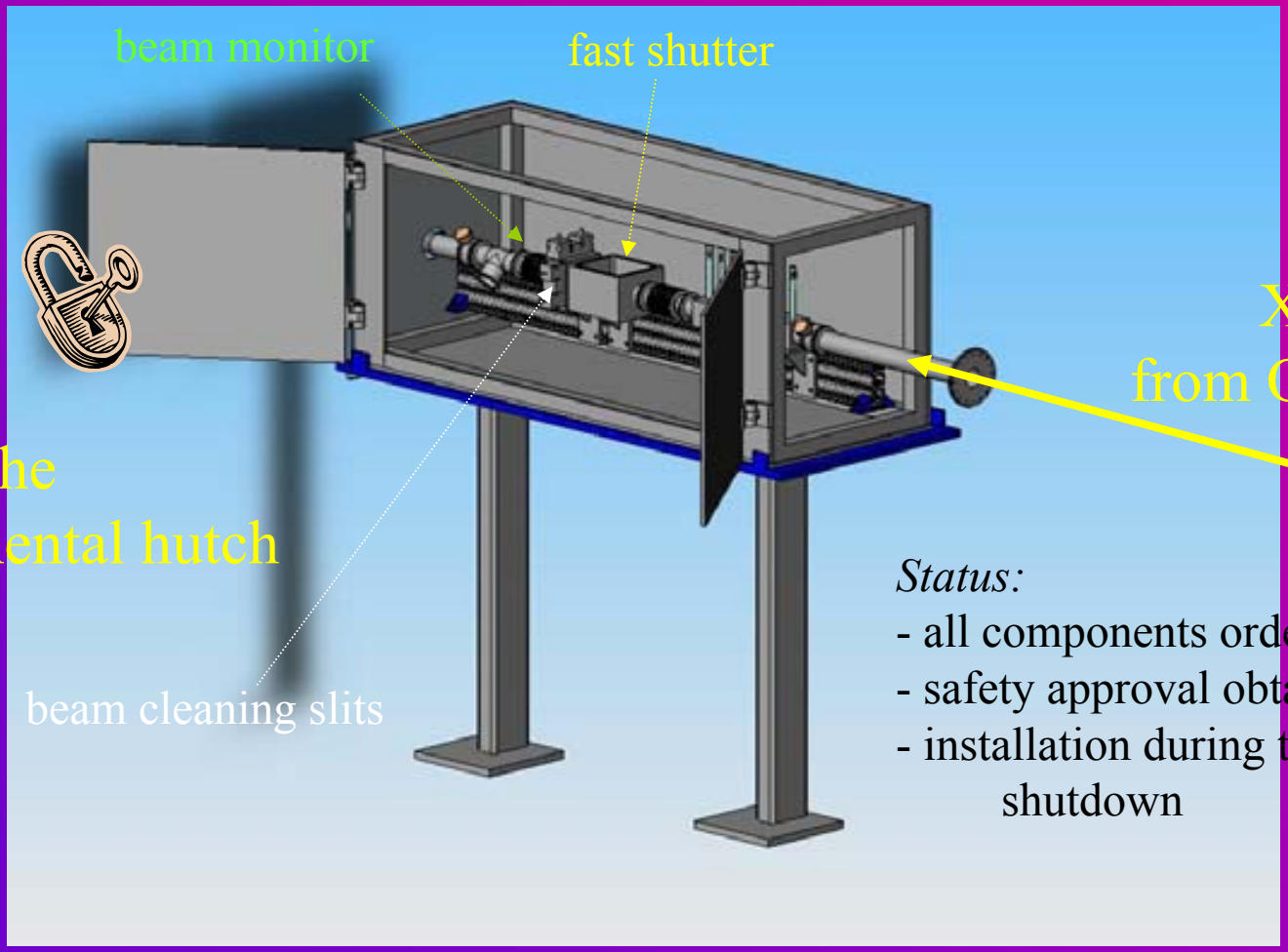
- beam defining slits after the mirror
- fluorescent screen after the slits
- attenuators before Ge crystal

} DONE during October shutdown



# *MINIHUTCH in mezzanine*

~ 4 m from the sample



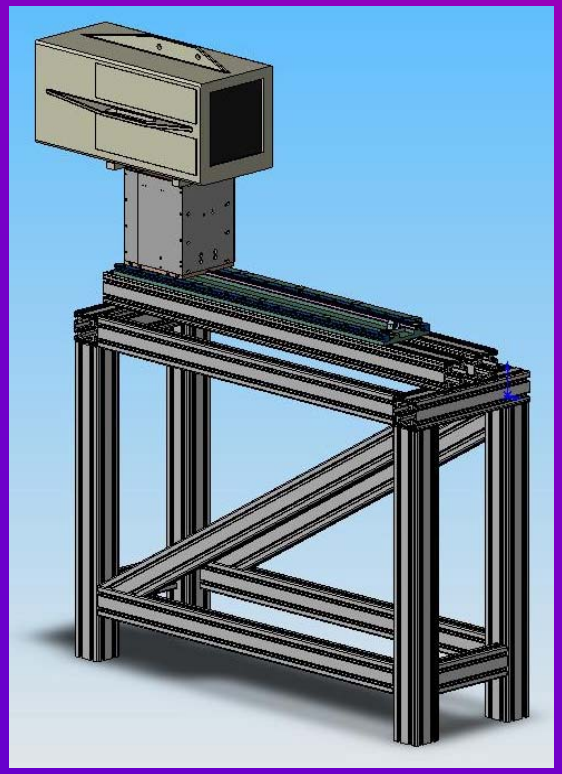

*Status:*
- all components ordered
- safety approval obtained
- installation during the winter shutdown

first in ESRF

# *EXPERIMENTAL HUTCH*

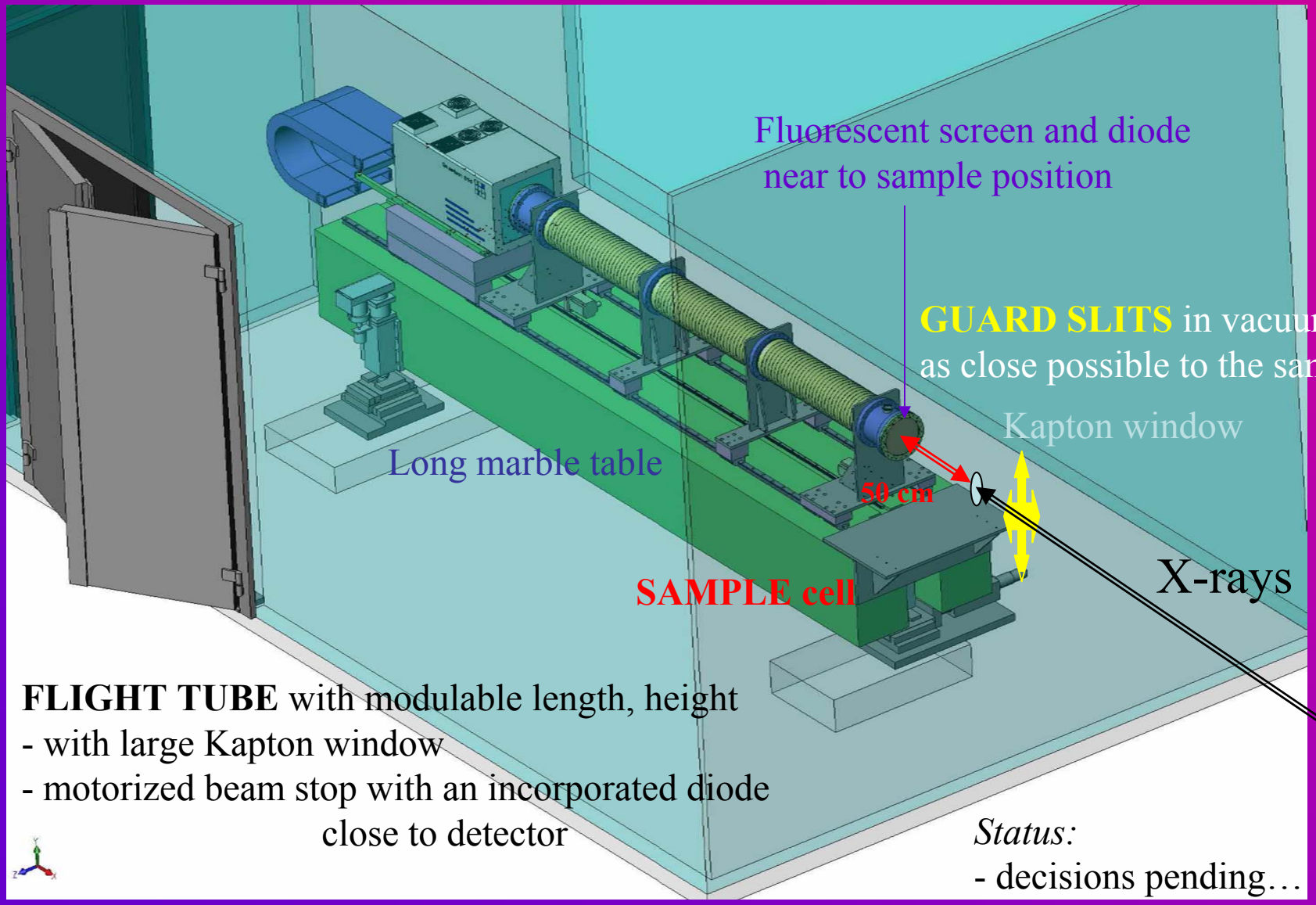
***January-March 2008***

- 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




**FLIGHT TUBE** with modulable length, height
- with large Kapton window
- motorized beam stop with an incorporated diode close to detector

*Status:*
- decisions pending…

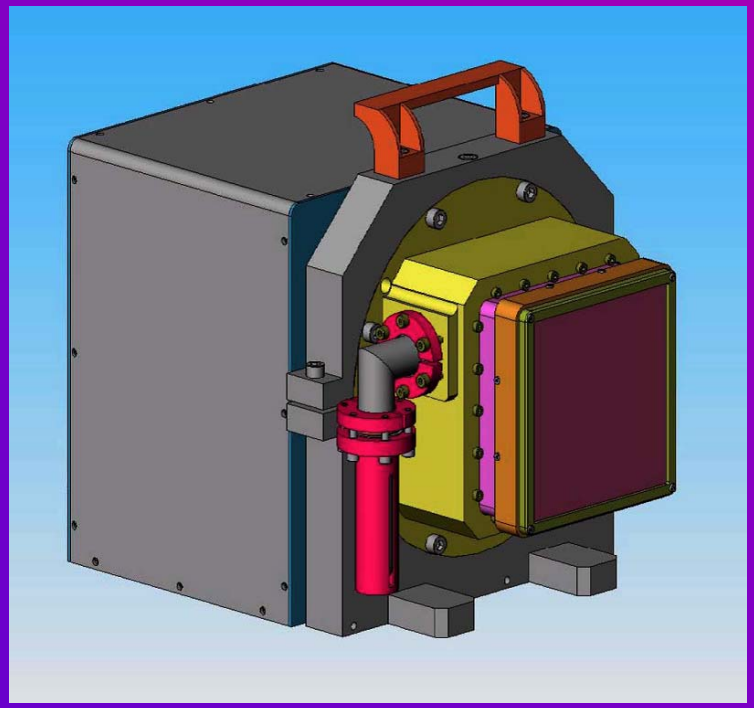
# DETECTOR(S)

Present on the beamline: **Q4R ADSC**

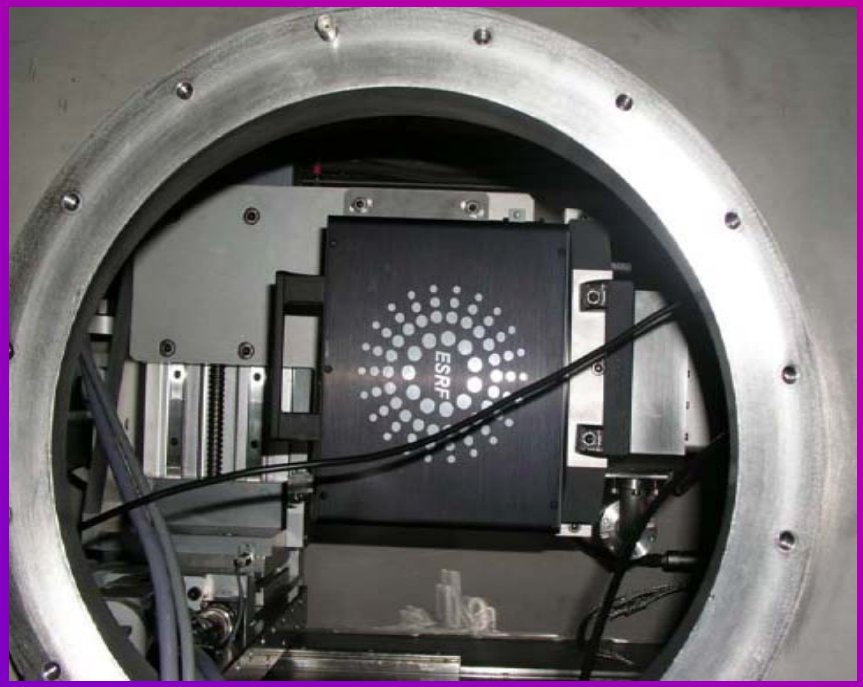
- 2 × 2 array with no more than 400 μ slit between detecting areas
- 2304 × 2304 pixels, 82 μm² each
- active area: 188 × 188 mm²
- readout time: lowest noise ~ 9 s
  high speed ~ 3 s
- 16 bits

# FRELON

Fiber optically coupled (taper) Frelon CCD based on **Kodac 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
# VÅNTEC-2000

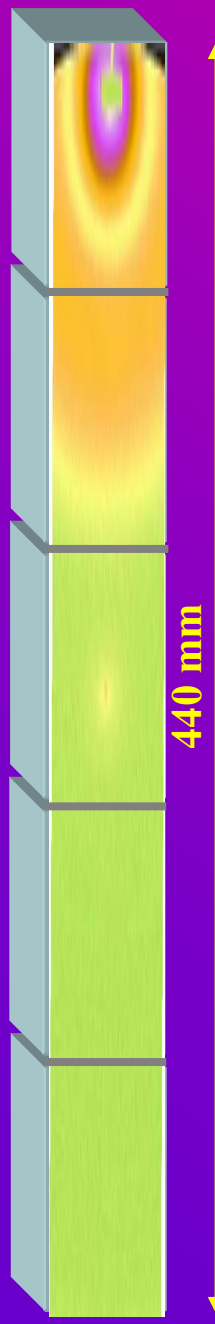
*Gas-filled detector bought by Detector pool*

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 x 172 $\mu m^2$
- single module: 195 x 487 pixels
- active area single module: 33.5 x 83.8 $mm^2$
- counting rate: > 2 x $10^6$/s/pixel
- framing rate: 200 Hz
- energy range: 3 –30 keV

PILATUS 2M

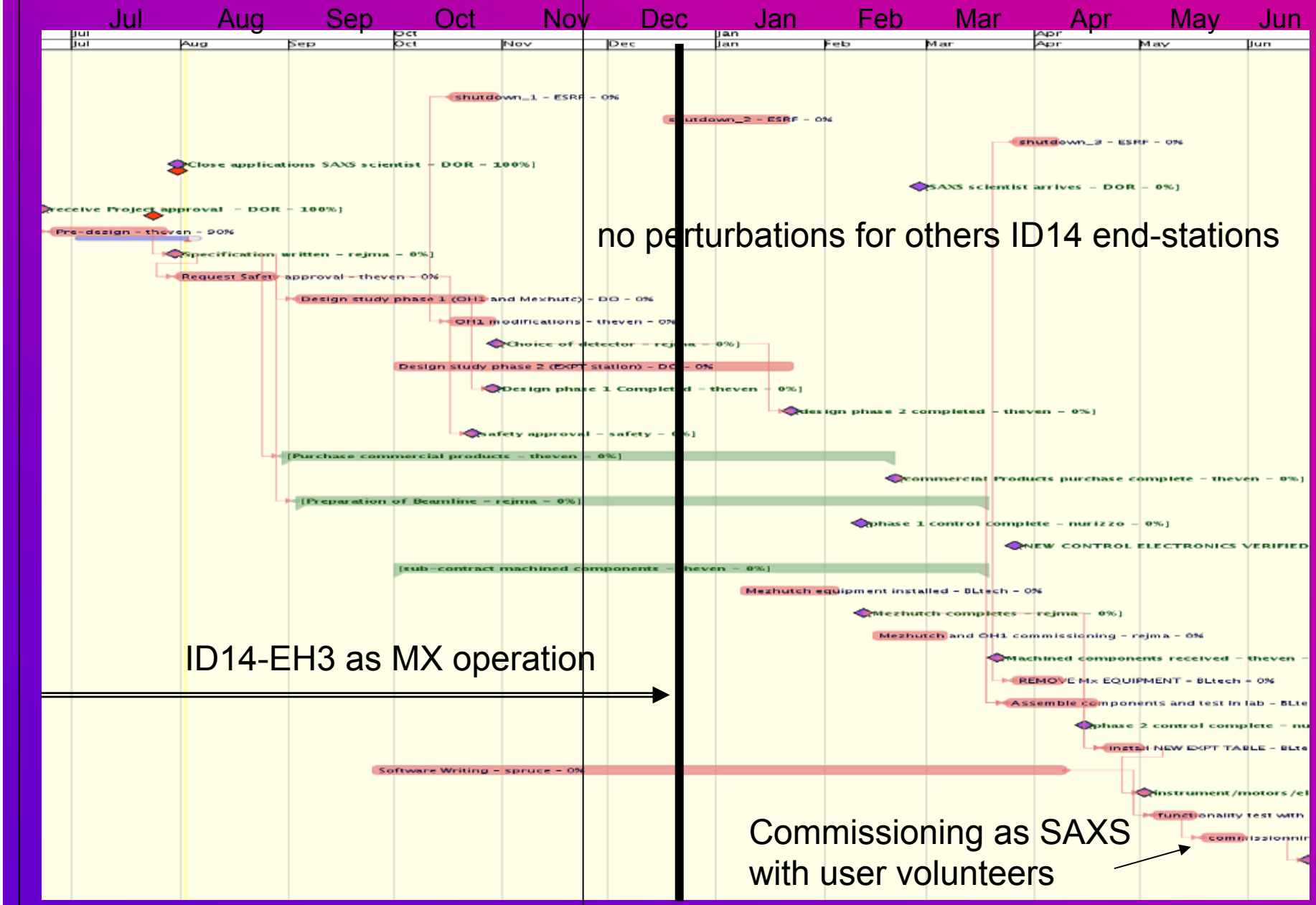
**24 modules**

active area = 254 x 289 $mm^2$

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*