X-TIP Workshop

*Coupling of Synchrotron Radiation IR and **X**-rays with **Tip** based Scanning Probe Microscopies*

16-18 November 2005

Edge Radiation IR end-station at ESRF

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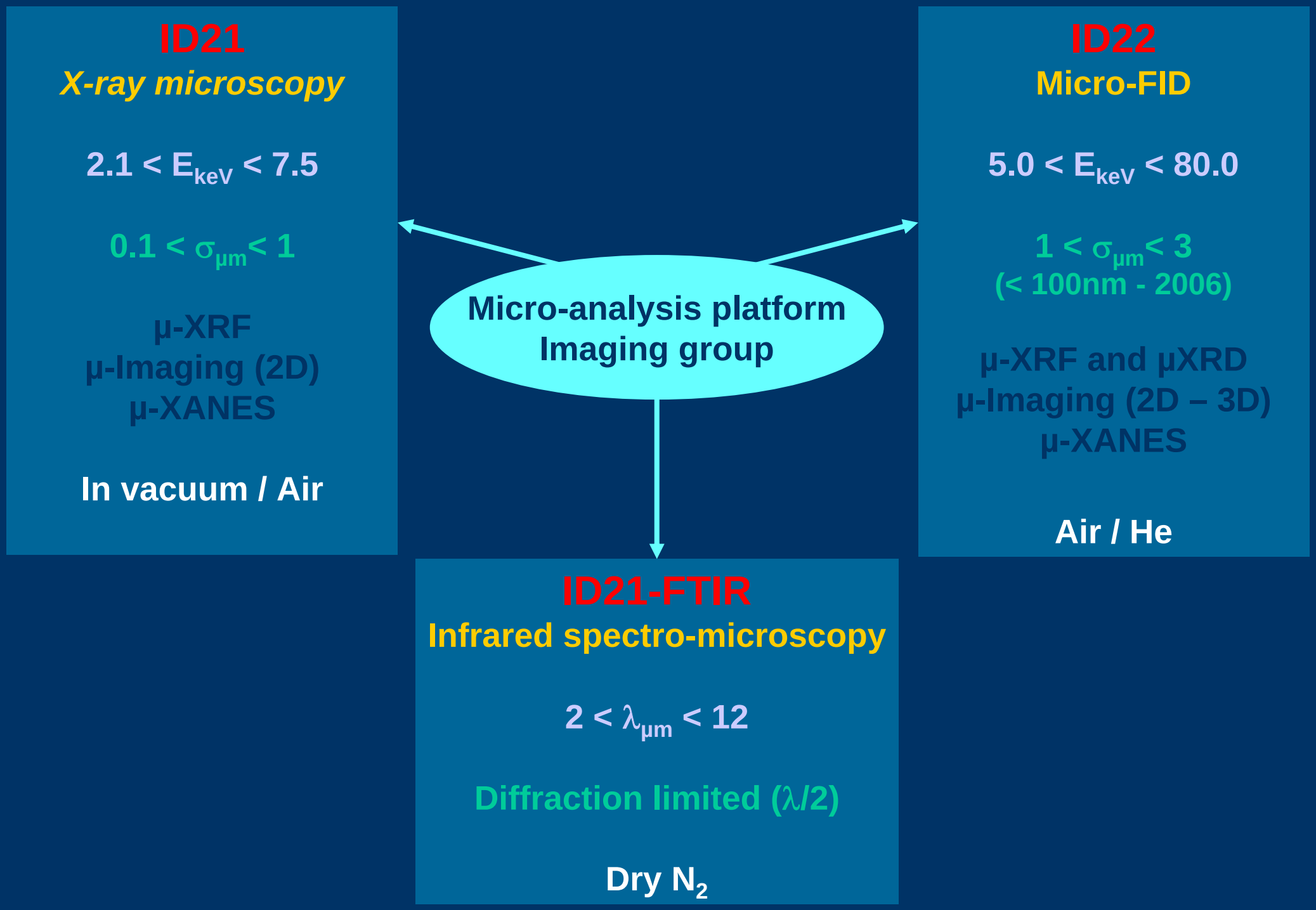
Outline

- **X-ray microprobes**
- **Some examples**
- **IR end-station**
- **Synchrotron based IR-SNOM?**

- M. Cotte (ESRF)
- M. Salomé (ESRF)
- R. Baker (ESRF)
- E. Gagliardini (ESRF)
- K. Scheidt (ESRF)
- P. Dumas (SOLEIL)
- O. Chubar (SOLEIL)

- N. Rochat (CEA)
- F. Bertin (CEA)
- A. Chabli (CEA)
- F. Comin (ESRF)
- M. Silveira (ESRF)
- N. Chevalier (UJF-CEA)
- S. Huant (UJF-CNRS)


ID21
X-ray microscopy
2.1 < E keV < 7.5
0.1 < σ µm < 1
µ-XRF
µ-Imaging (2D)
µ-XANES
In vacuum / Air
Micro-analysis platform
Imaging group
ID22
Micro-FID
5.0 < E keV < 80.0
1 < σ µm < 3
(< 100nm - 2006)
µ-XRF and µXRD
µ-Imaging (2D – 3D)
µ-XANES
Air / He
ID21-FTIR
Infrared spectro-microscopy
2 < λ µm < 12
Diffraction limited (λ/2)
Dry N2

Attributes of multi-keV XRM (2-30keV)

- **X-ray Fluorescence** → *Trace element detection & mapping*; *Quantitative fluorescence analysis*
- **Micro-spectroscopy (XANES)** → *Chemical state specificity*
- **Higher penetration**; **Phase contrast** → *Microscopy on thick samples*; *Lower radiation damage (?)*
- **Larger focal lengths (> 20mm)**; **Larger depth of focus (> 100µm)** → Space for sample environment; *3D imaging*

Multi-modal approach
- ***Micro-Fluorescence***
- ***Micro-diffraction***
- ***3D imaging***
- ***Spectroscopies***

⟷ **In-situ experiments** ***controlled sample environment***

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Multi-modal approach
- ***Micro-Fluorescence***
- ***Micro-diffraction***
- ***3D imaging***
- ***Spectroscopies***

⟷ **In-situ experiments** ***controlled sample environment***

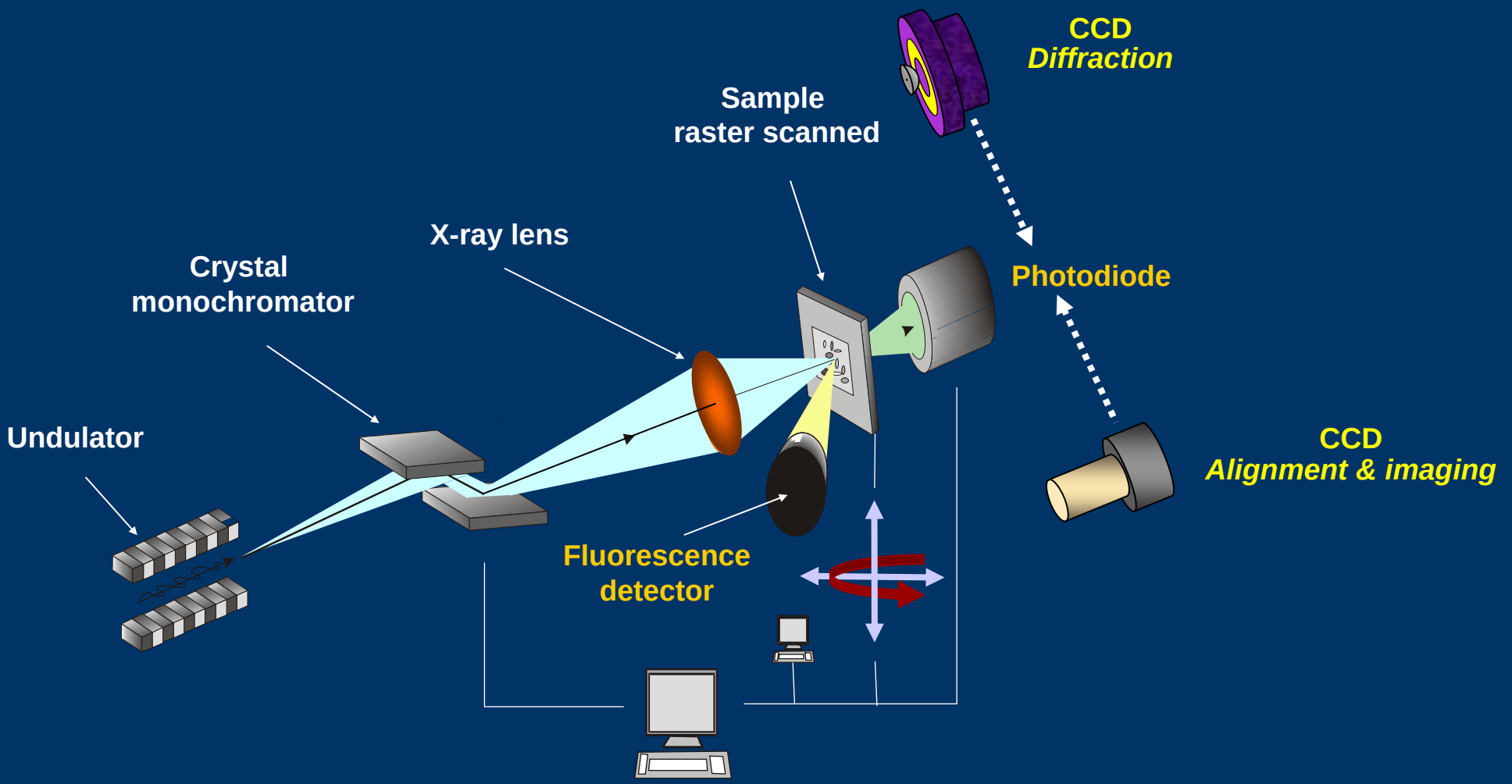
Imaging and micro-spectroscopy beamlines

ID21-FTIR

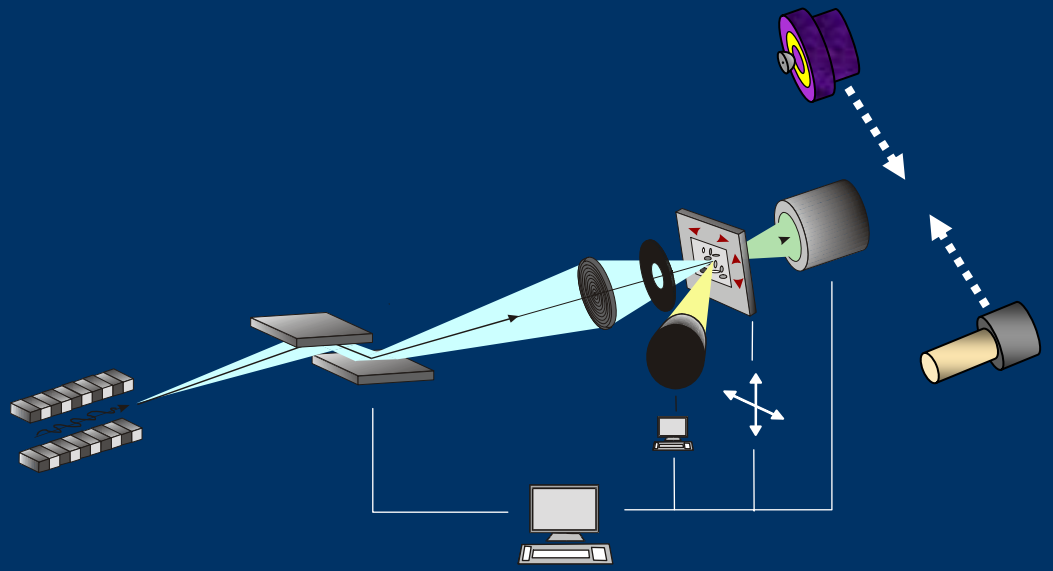
ID21

ID22

1 — 10 — KeV — 100

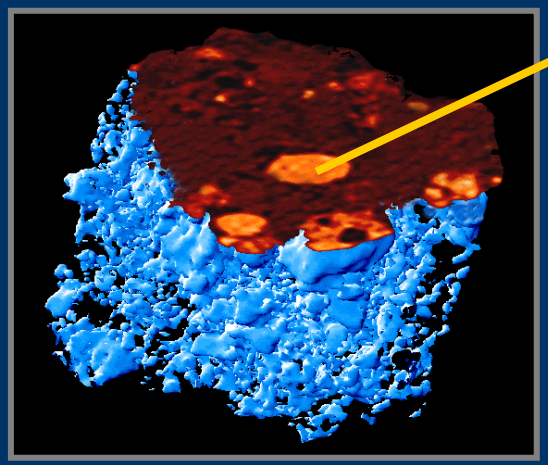


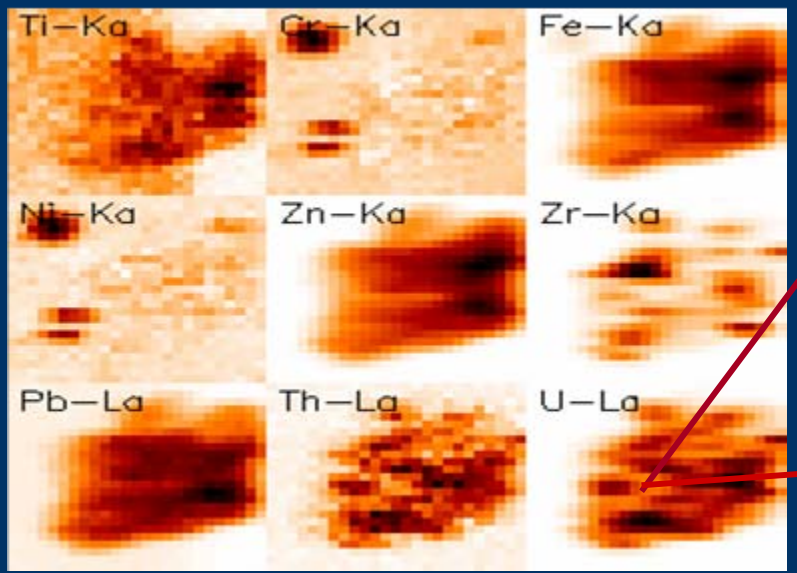
Several signals and information available

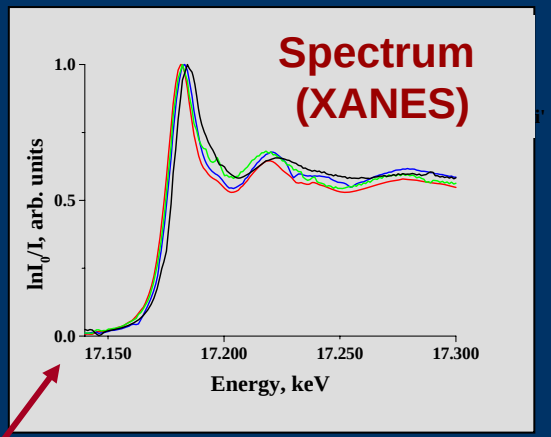


Trace element imaging - Fluorescence

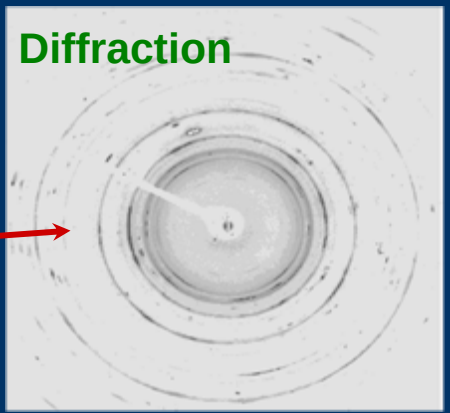
Tomography - Absorption



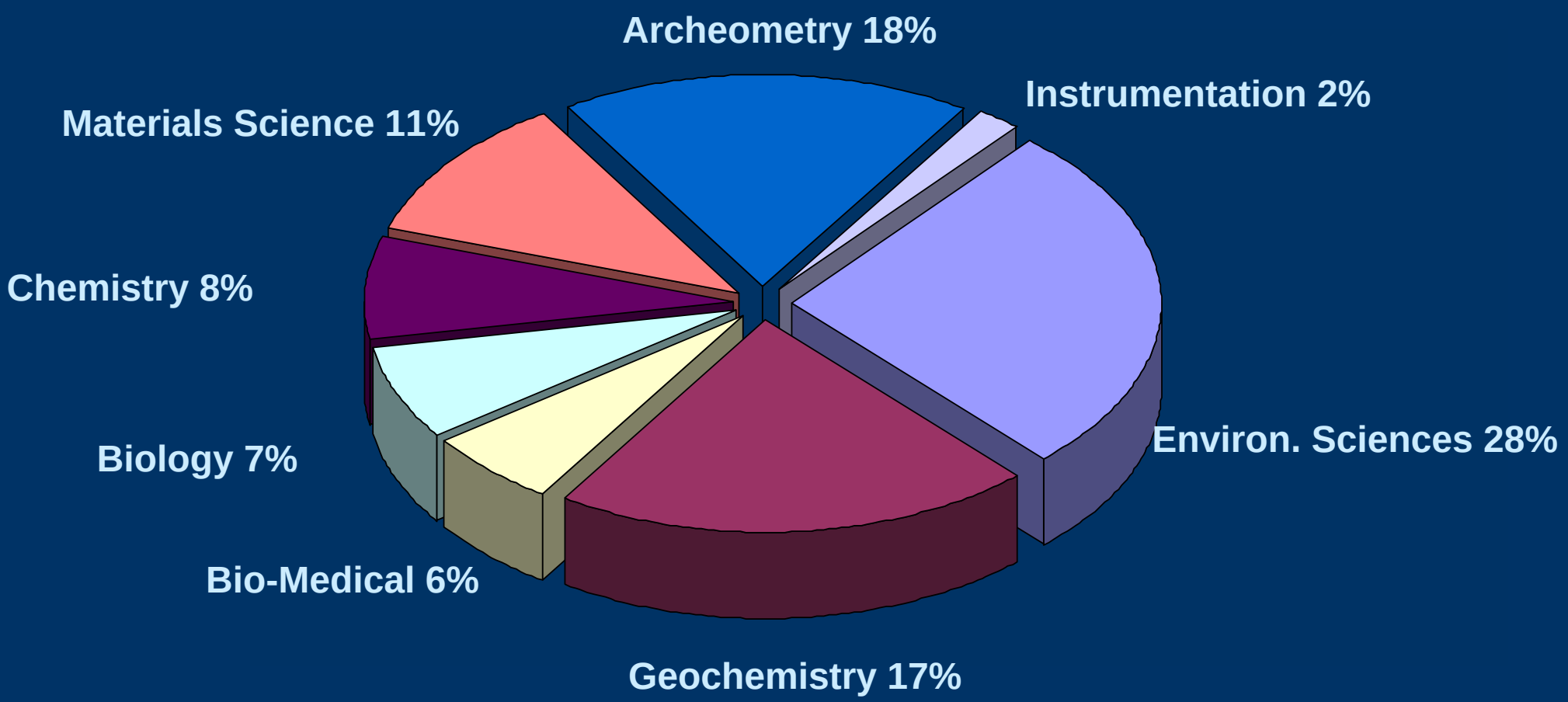




Diffraction

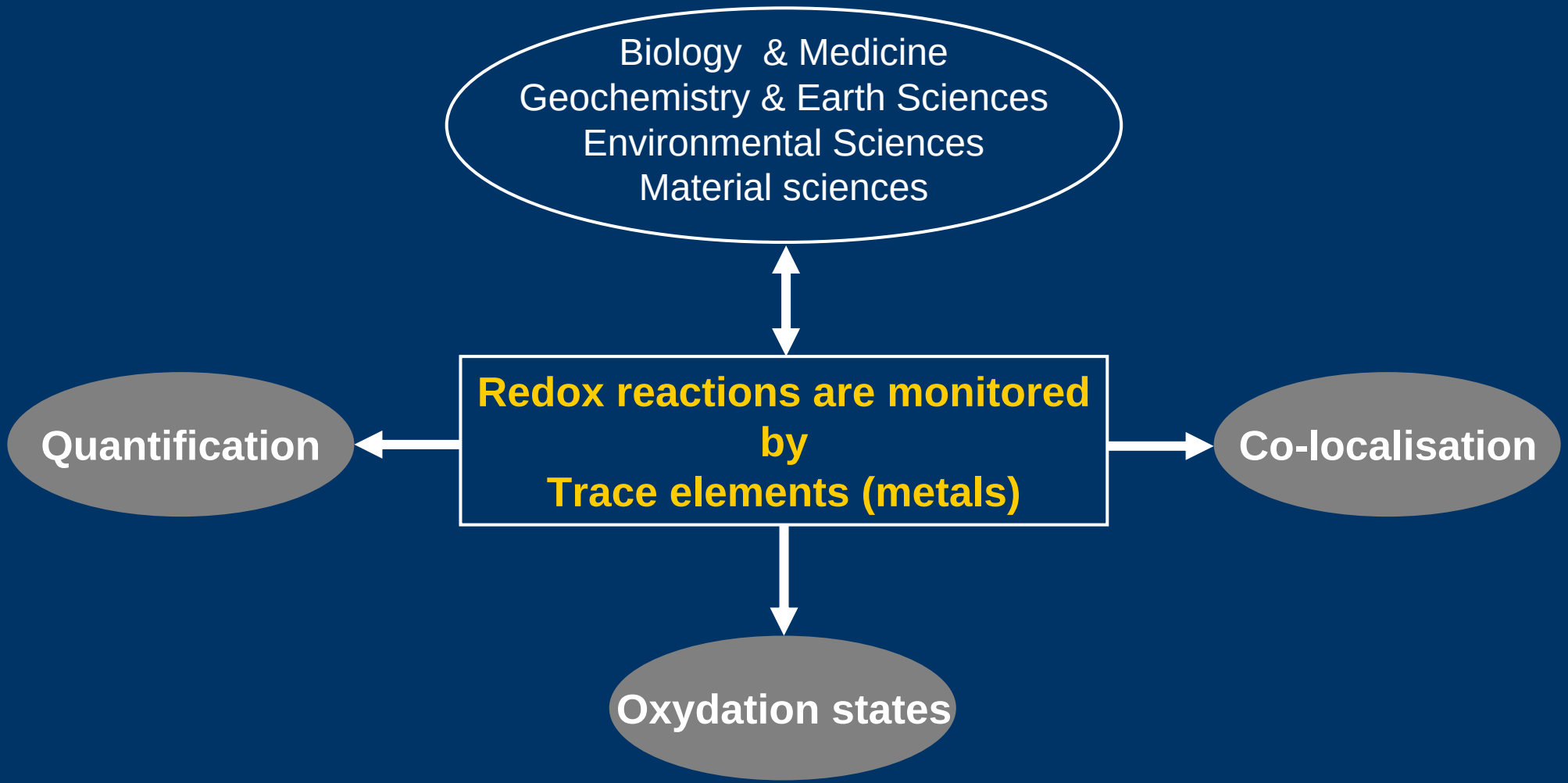


Science at ID21 and ID22/ID18F



Period: 2002-2005: > 210 experiments

Need for trace element analysis in heterogeneous systems



- chemical speciation → **XANES**
- detection and quantification → **X-ray fluorescence**
- element co-localisation: → **2D/3D mapping**

Need for complementary techniques

C, H, O, N

Mg, Na, S, **P, Cl, Ca, V, Cr, Fe, Cu, Zn**, …

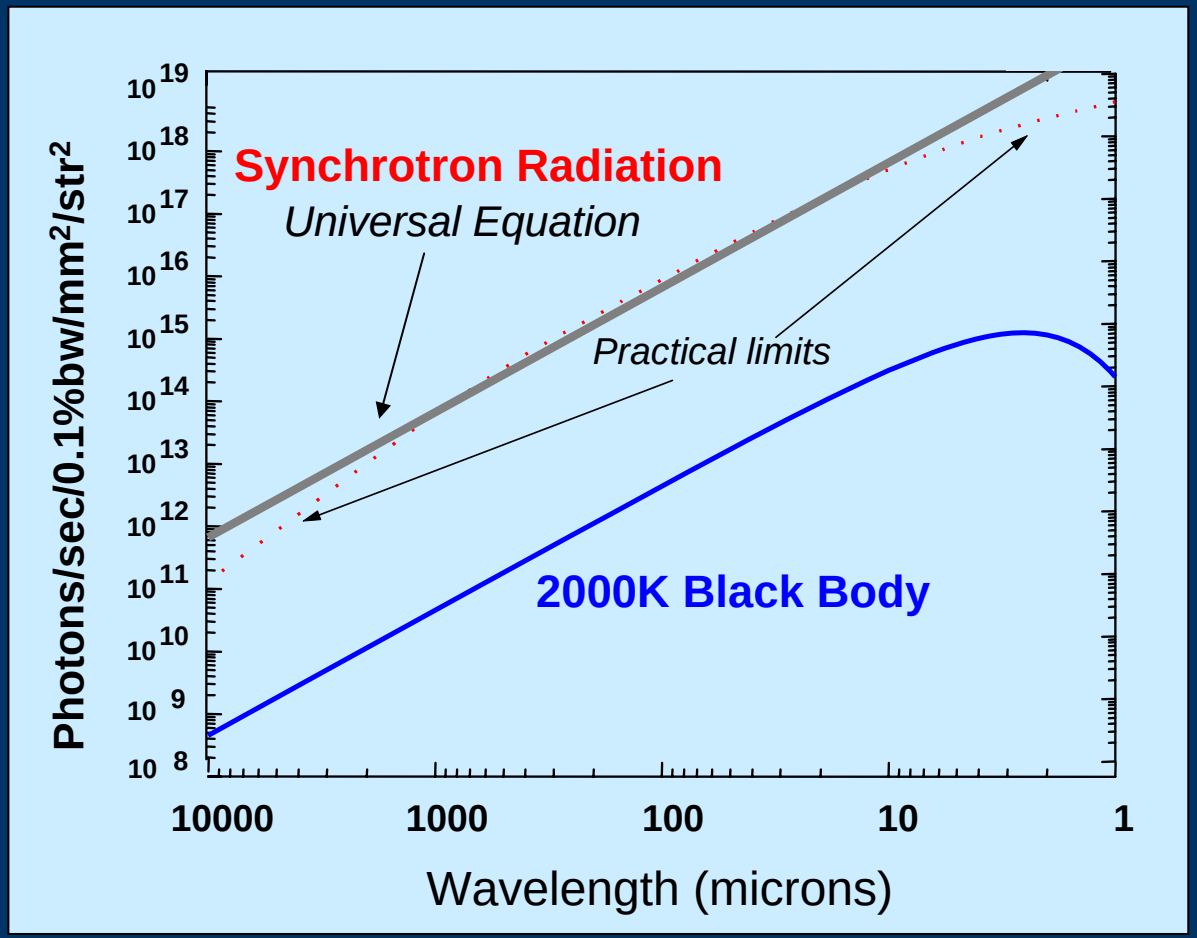
IR spectro-microscopy

- resolution **+**
- detection limit **+**
- chemical selectivity **+++**
  - ➢ functional groups

X-ray spectro-microscopy

- resolution **+++**
- detection limit (fluorescence) **+++**
- chemical selectivity (XANES) **+++**
  - ➢ oxidation states

Synchrotron source: brightness advantage



BRIGHTNESS

Signal-to-Noise

Data Collection

Spatial Resolution

Spectro-microscopy

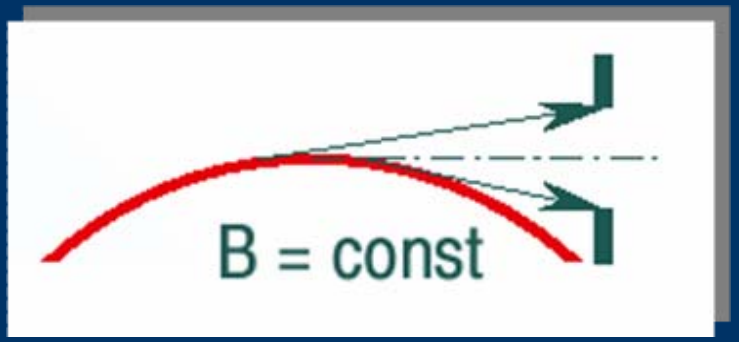
Chemical mapping

BROADBAND

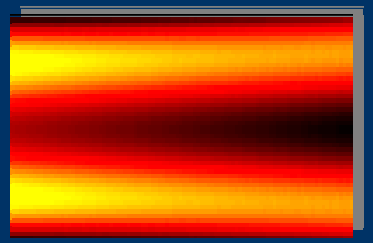
Spectroscopy

Two main modes of infrared emission

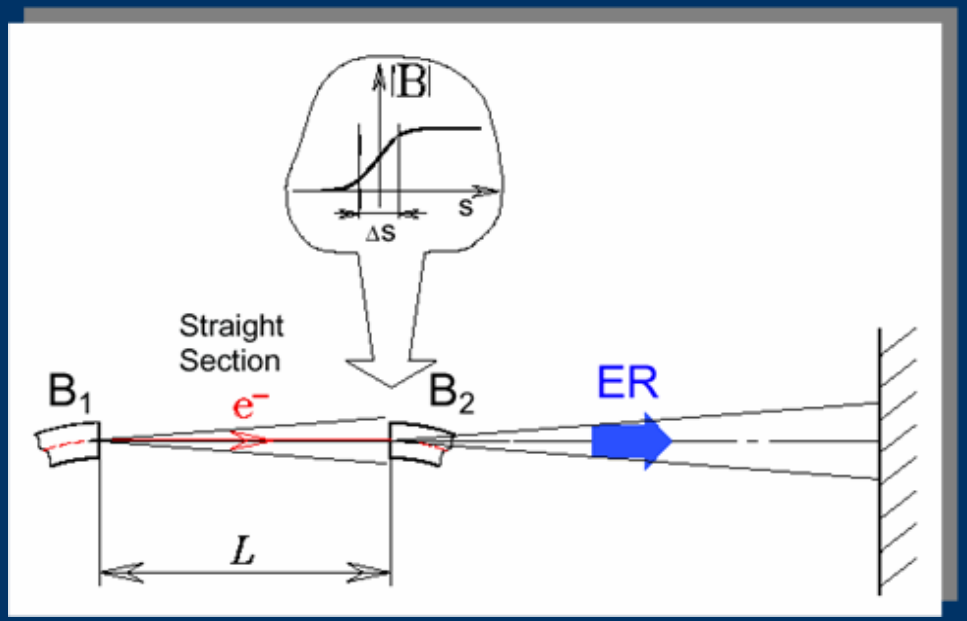
Bending magnet emission



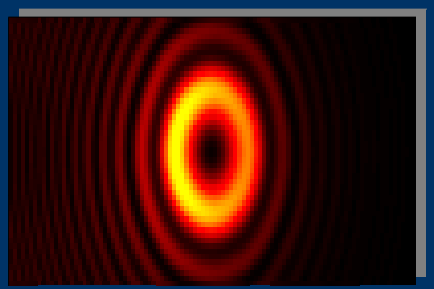
10 μm



Edge emission



10 μm

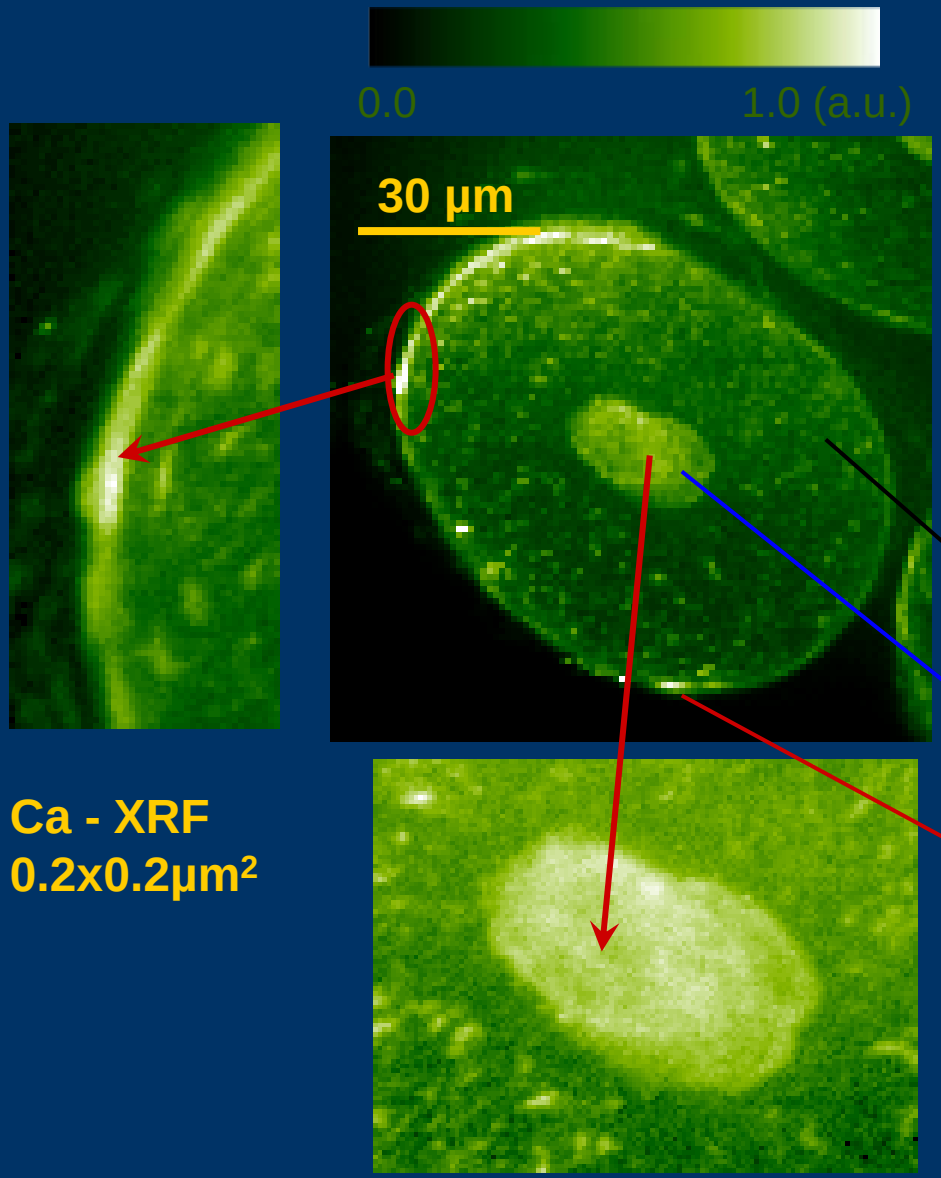


ID21 FTIR Microscopy end-station: Combined studies with X-ray microscopy

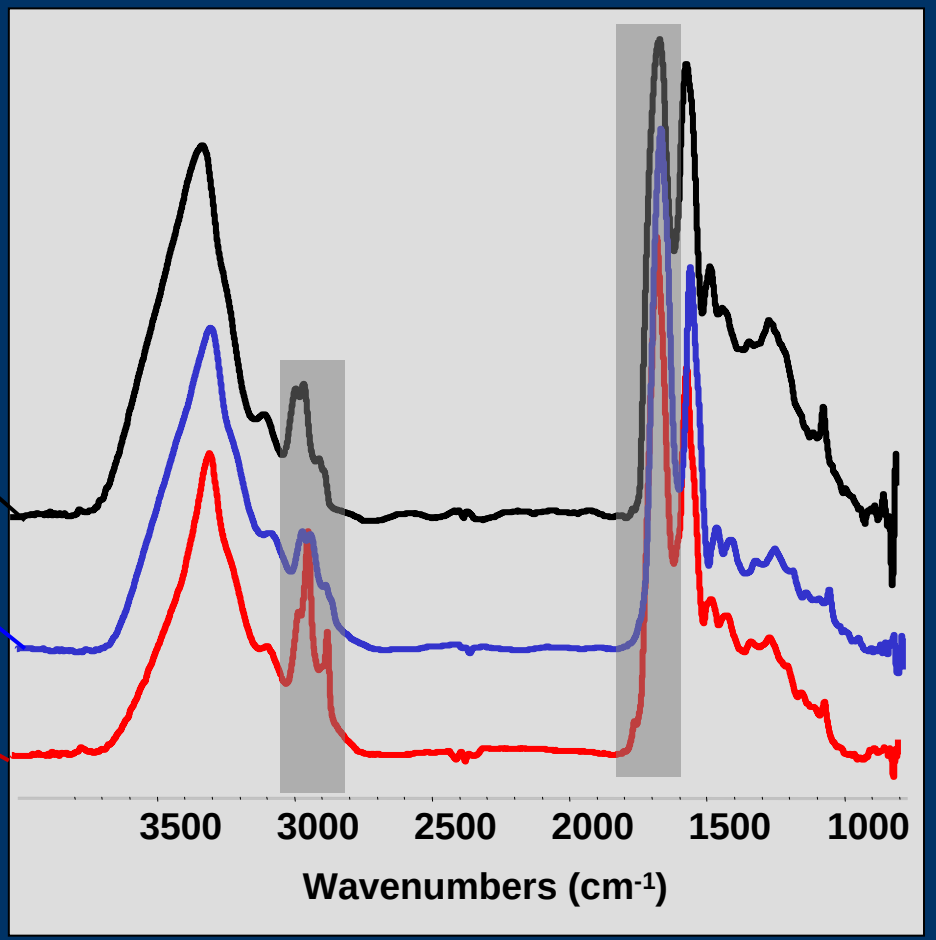
Compatibility with X-ray microscope sample holder

- Enlarge the palette of micro-analysis techniques available at the ESRF
- A unique facility for a coordinated use of IR and X-ray microscopes (physically close)
  - a clear demand from the user's community:
    - *Archaeology, Environmental sciences, Earth sciences, Biology, Polymers, Cosmology*
  - stimulate the in-house research programme by developing a new "culture" IR+X-rays
  - high potential for industrial applications

Various calcium sites in human hair shaft



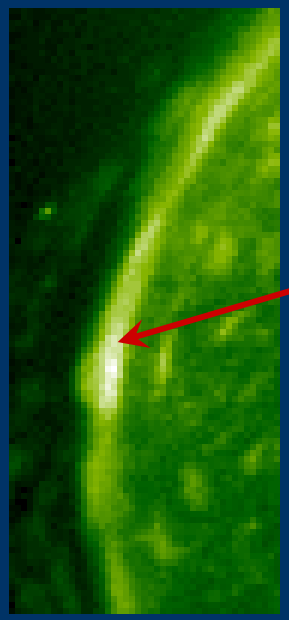
Ca - XRF
0.2x0.2µm²

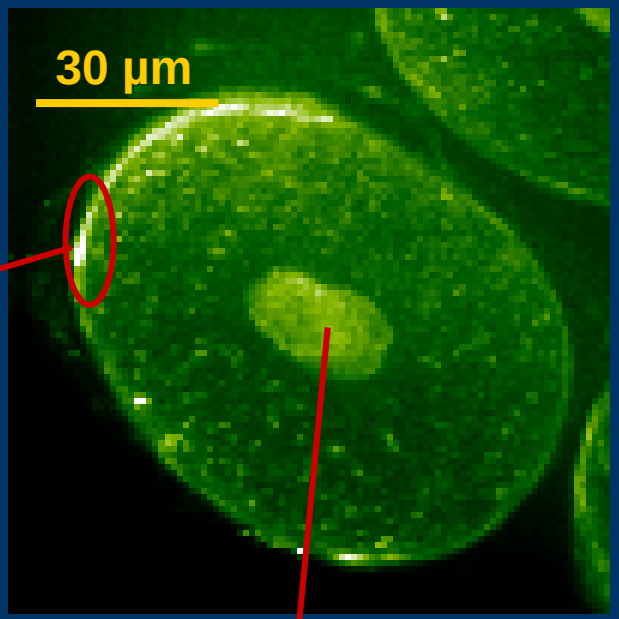


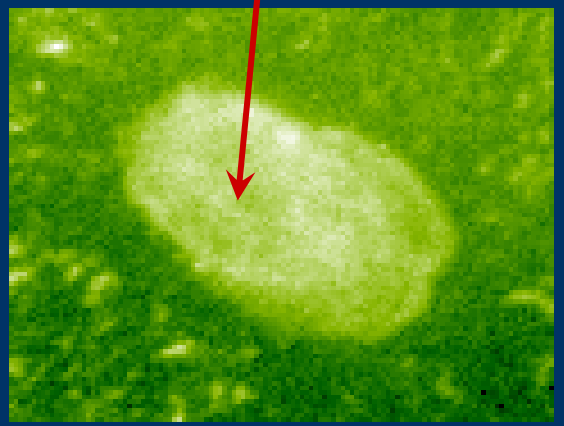
Infrared Spectra

C. Merigoux *et al.*, 2003

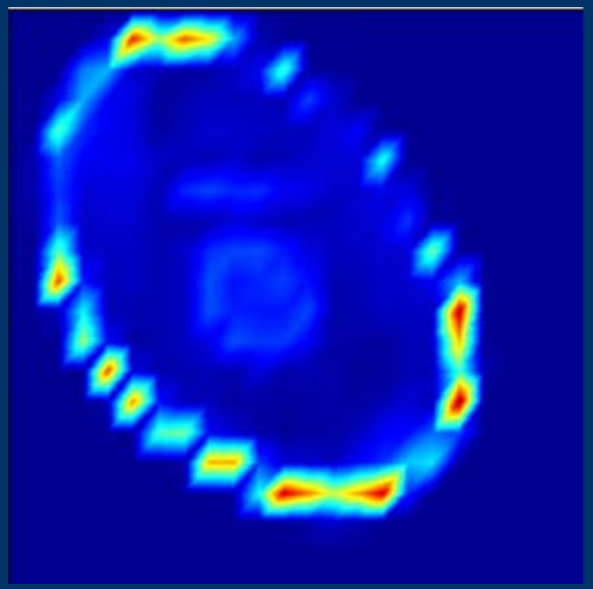
Various calcium sites in human hair shaft

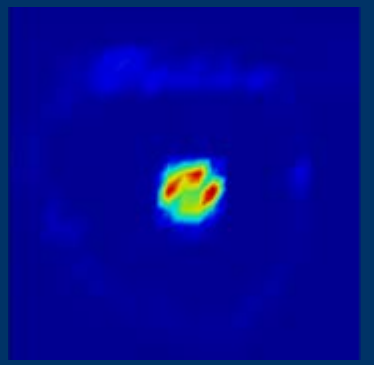




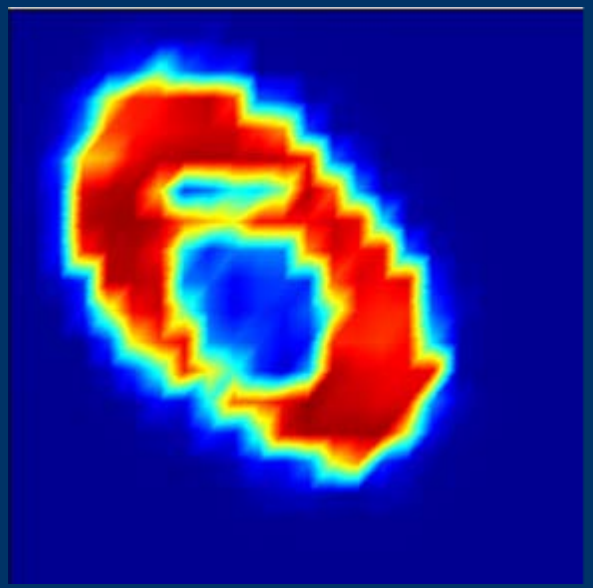


Ca - XRF
200x200nm²





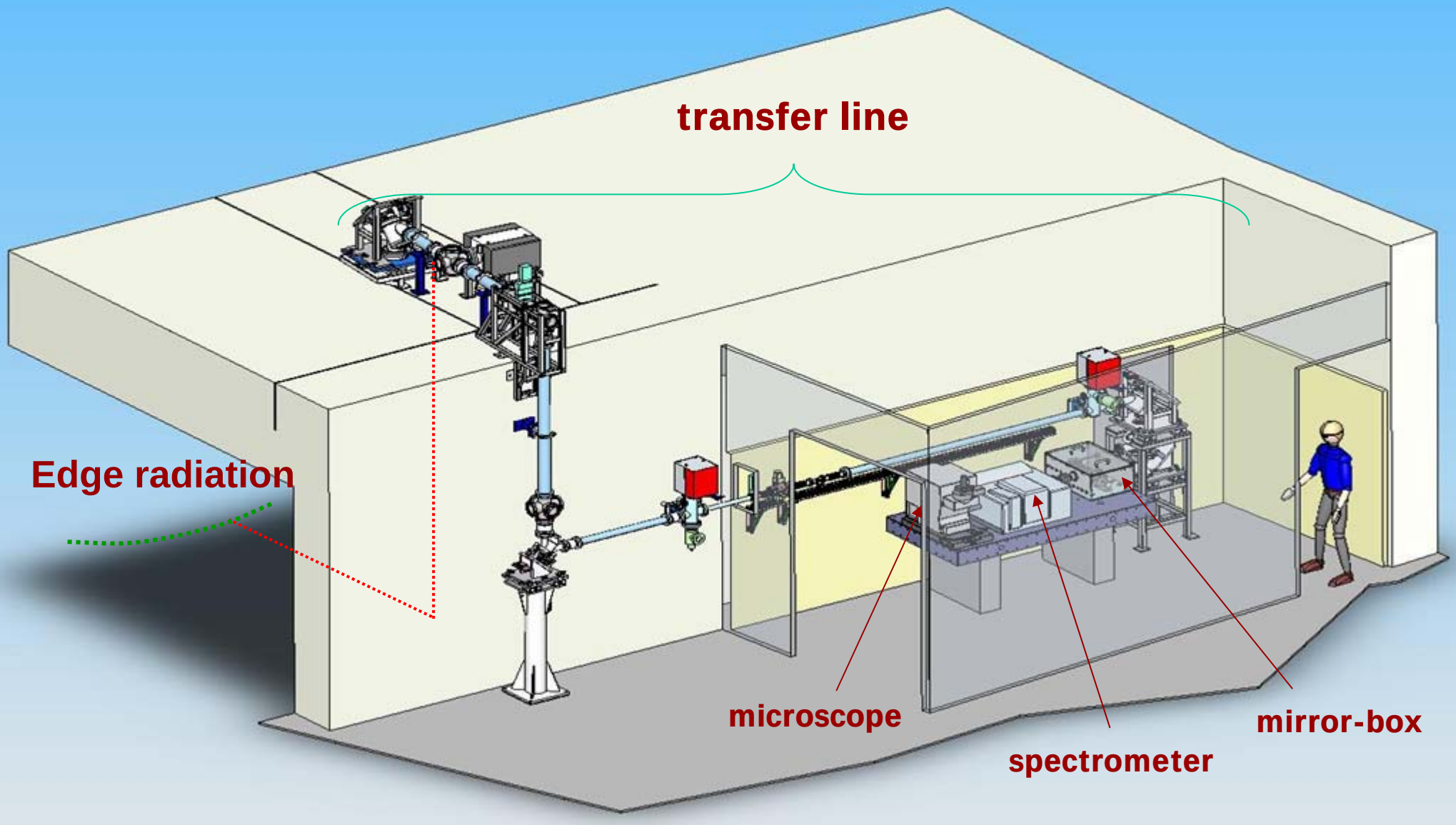
Two different « types » of lipids in cuticule and medulla



Protein distribution in cortex

C. Merigoux *et al.*, 2003

ID21 – Infrared microscopy end-station



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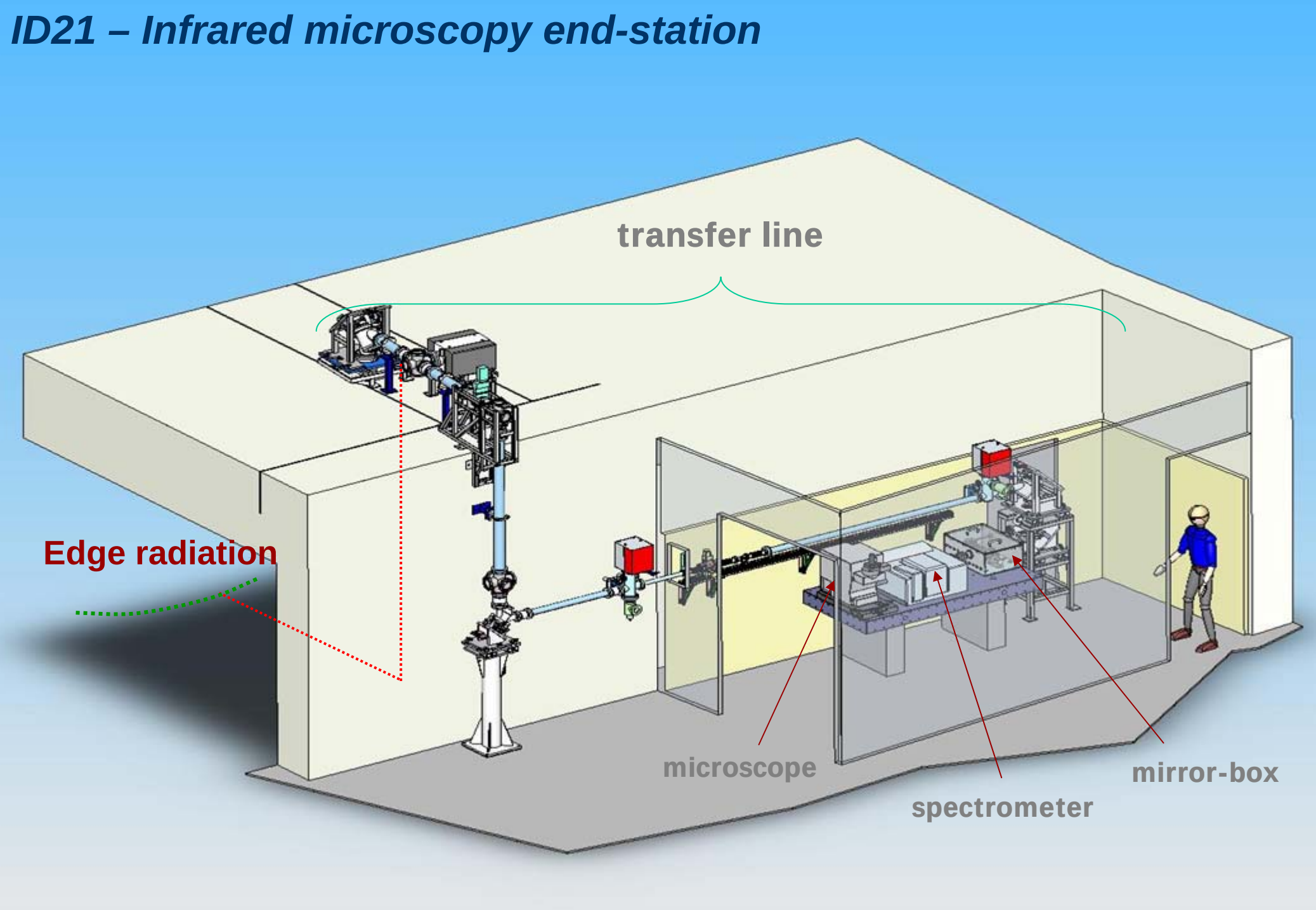
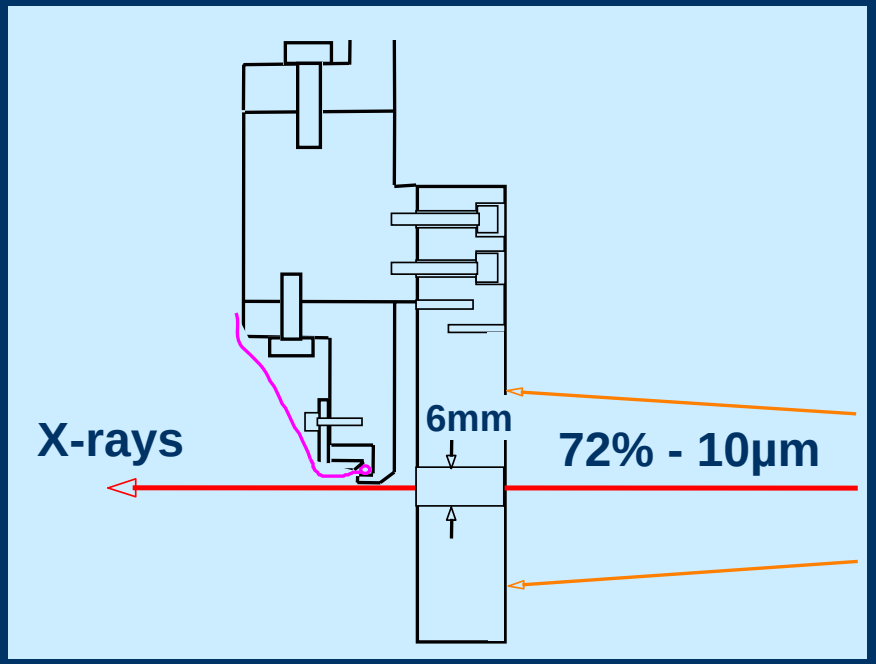
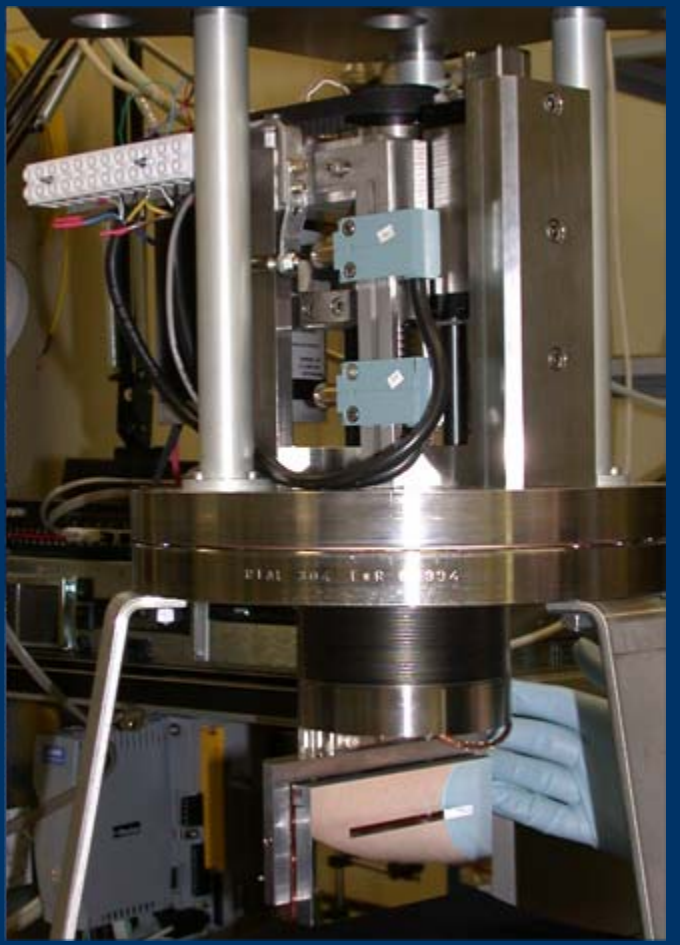
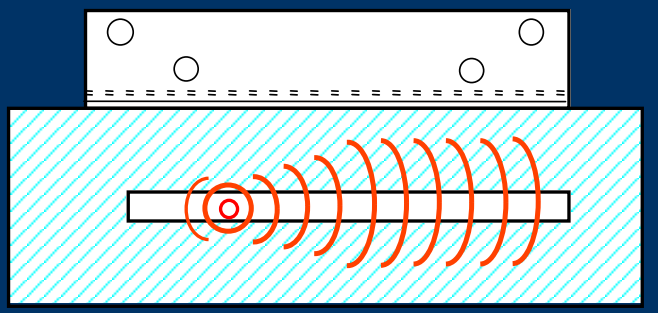


Extracting mirror (K.Scheidt, DIPAC'05, June 2005)





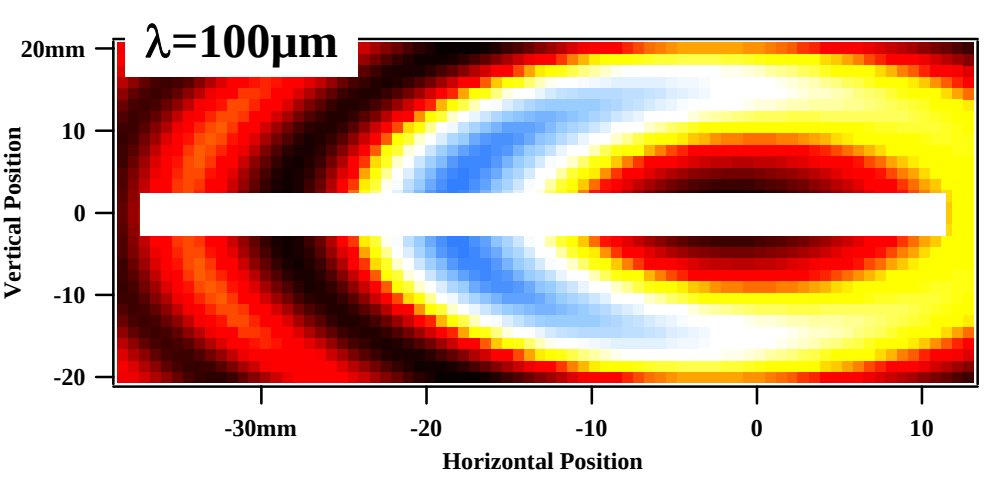
- flat un-cooled aluminium mirror, with a 5mm horizontal slot.
- lets the energetic part of the synchrotron light go through the optic without heating it.
- vertically movable, centered on the heart of the X-ray beam in a slow feed-back loop by the use of thermo-probes.

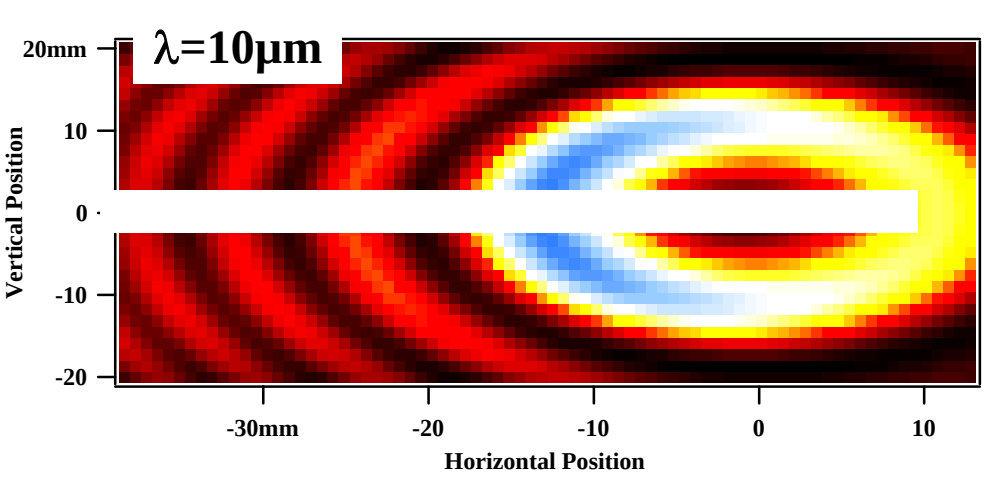


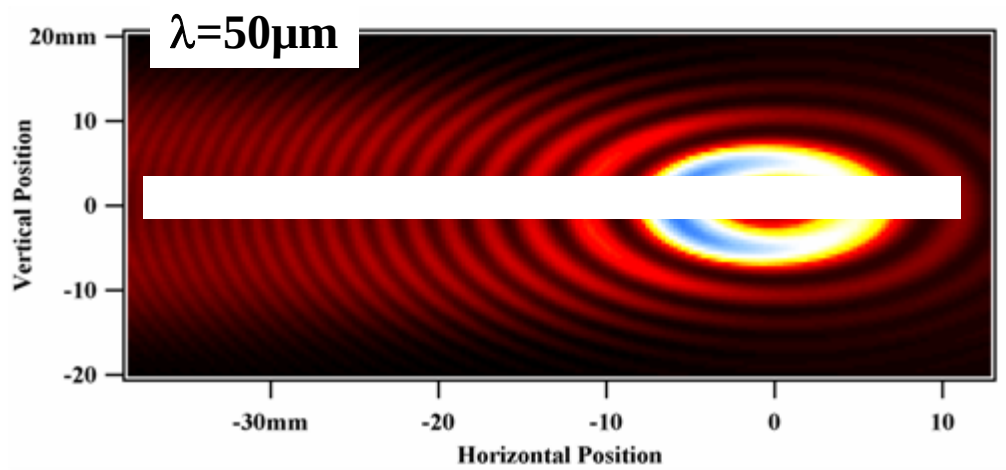
slotted mirror with 6mm vertical slot

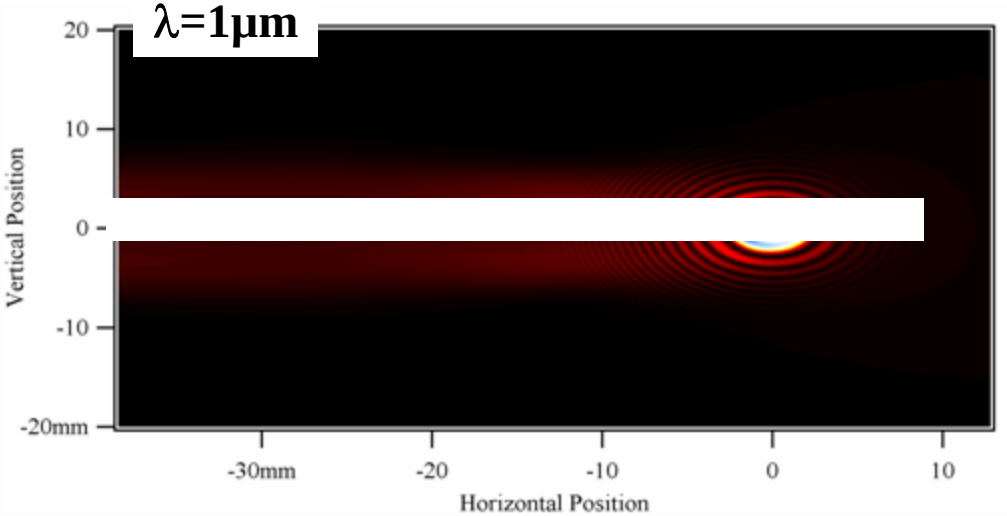
Slotted extraction mirror

Absorbed power: 1.5KW → a few watts



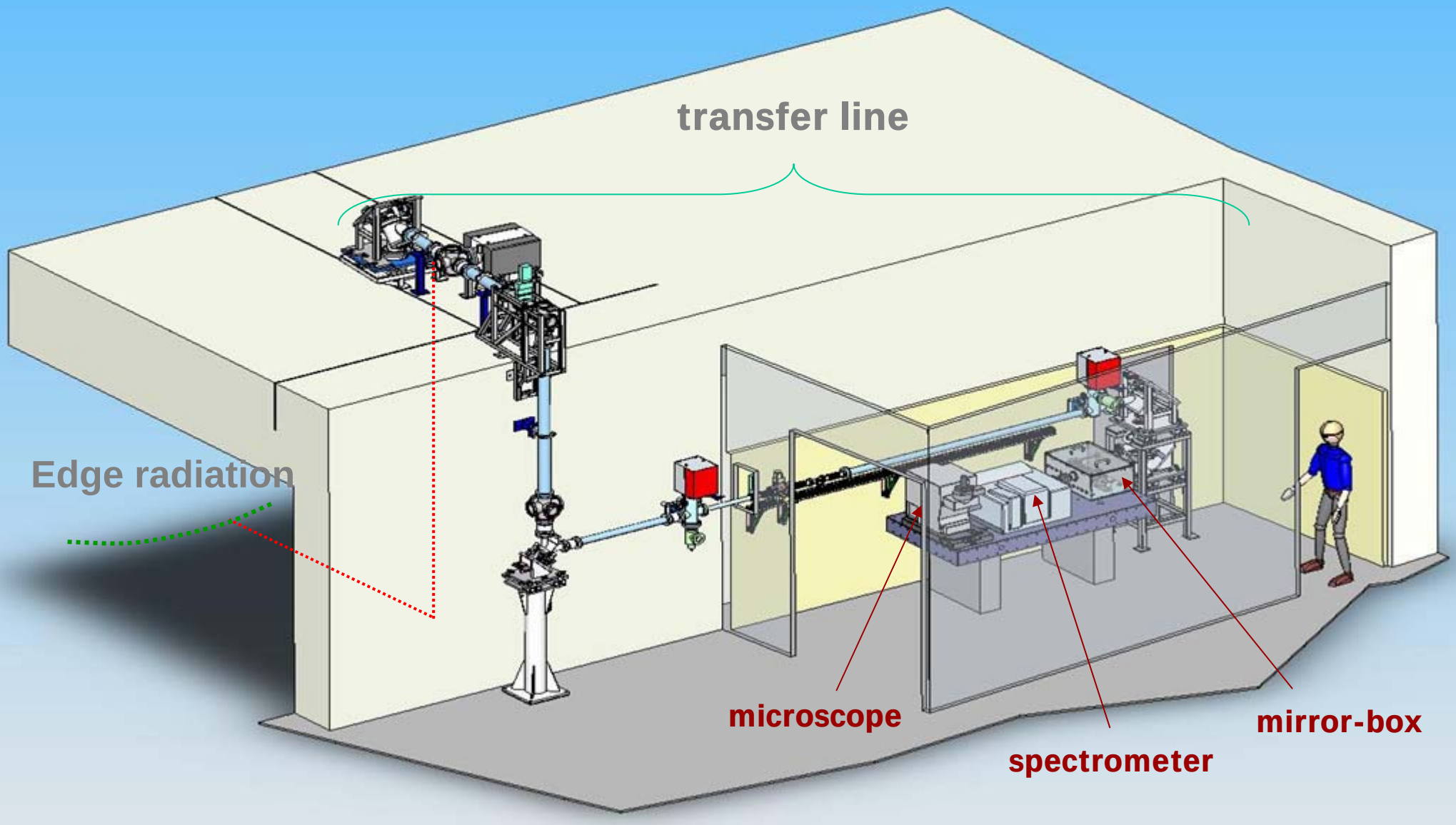




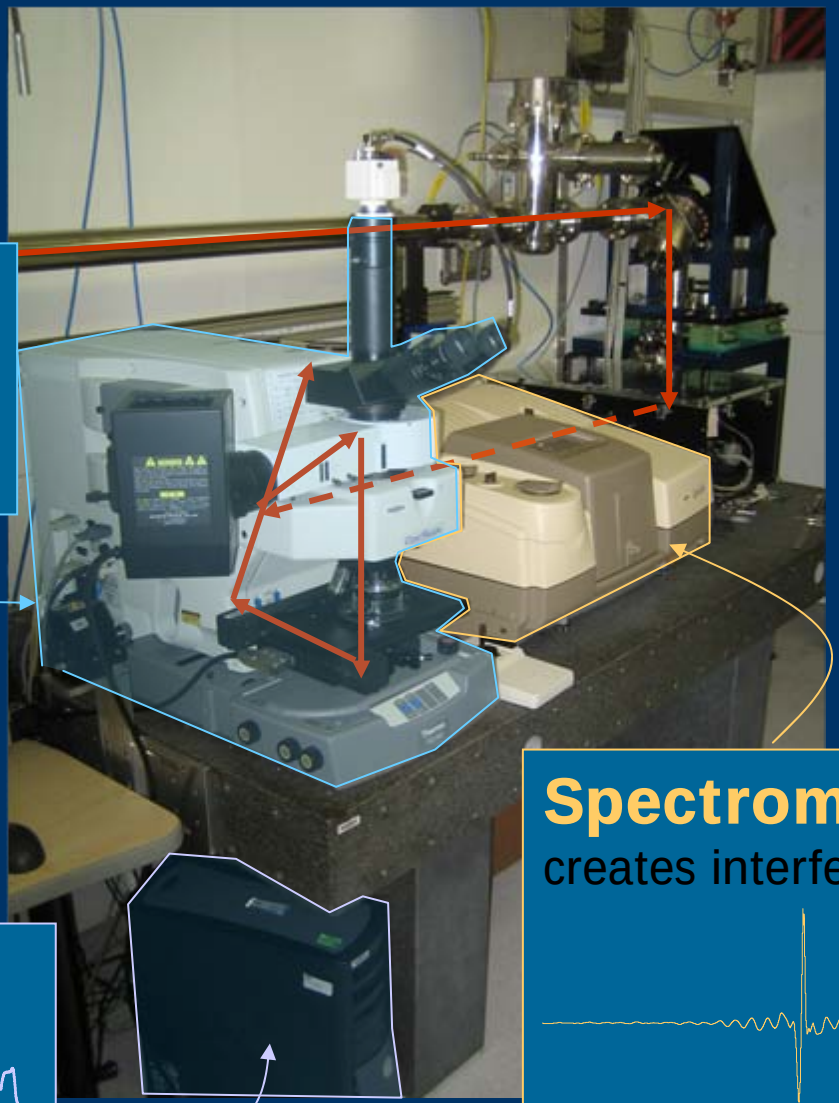


Beam profile at 3.2m from entrance main dipole

ID21 – Infrared microscopy end-station



The infrared end-station



Microscope

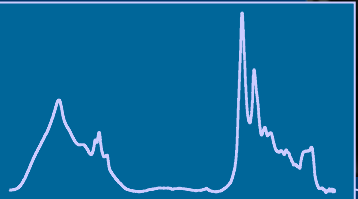
- focuses the beam on the sample
- collects and detects the transmitted or reflected beam

Spectrometer

creates interferograms

Computer

- Fourier Transform
- Data processing

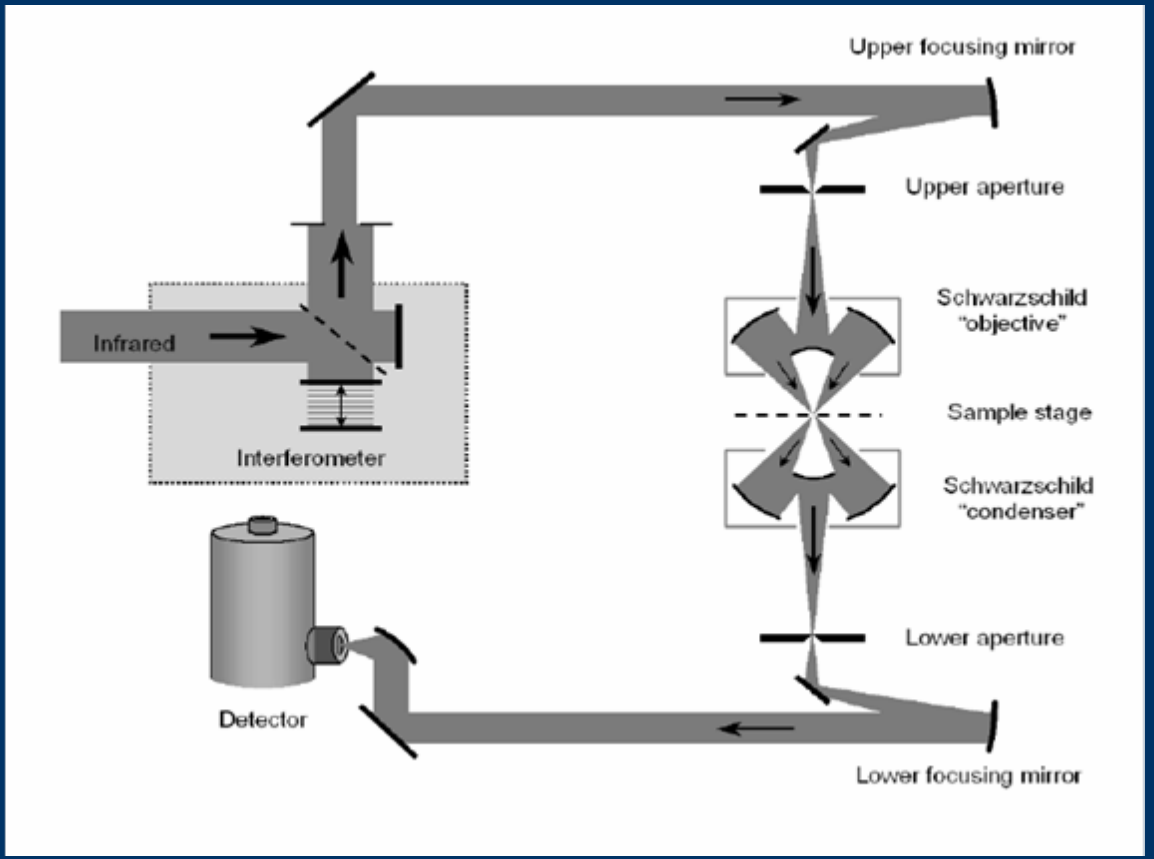


Inside the microscope

Confocal microscope:

Two confocal Schwarzschild objectives:

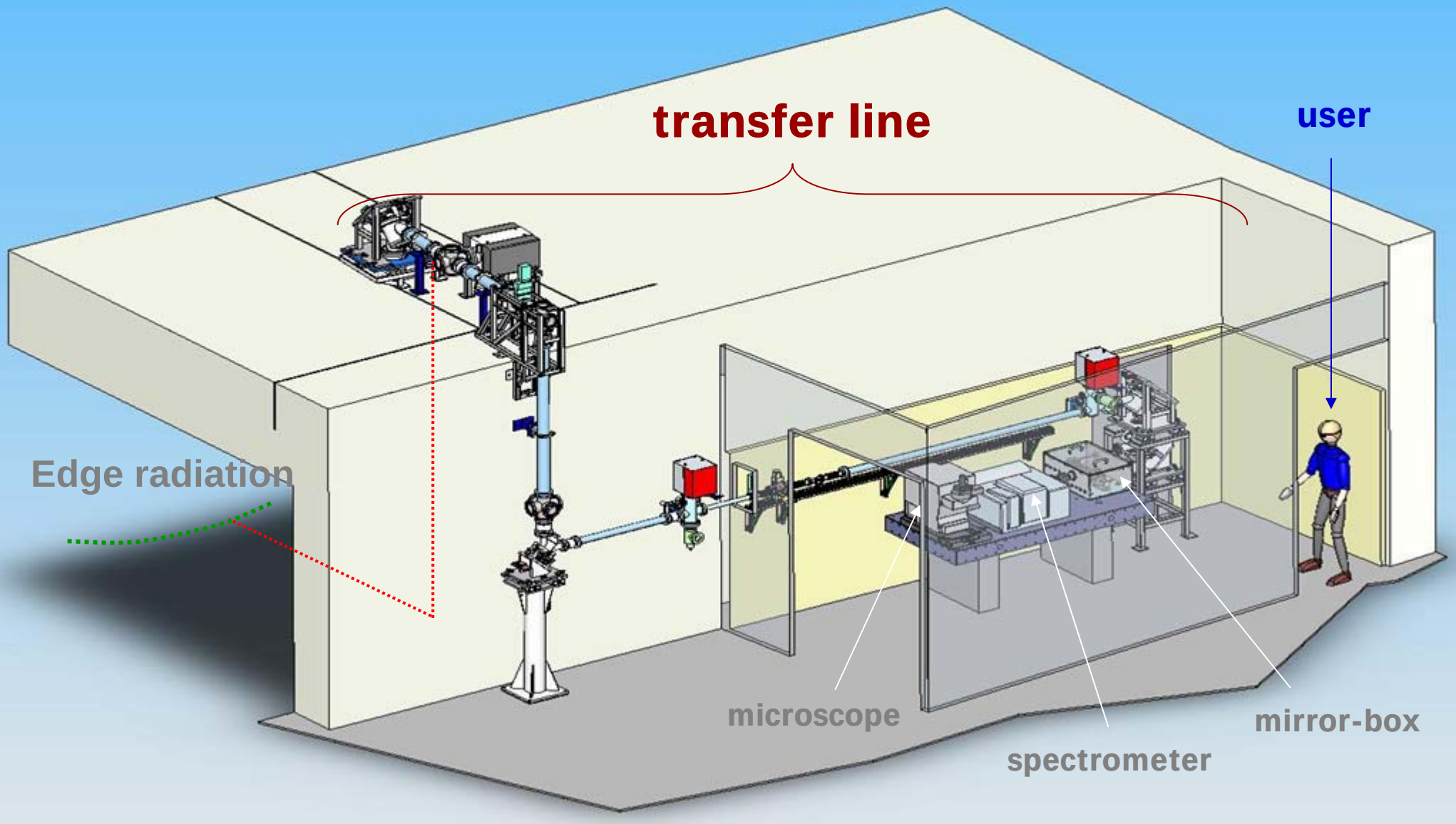
- focus the light onto the sample
- collect the light and relay it to the detector.



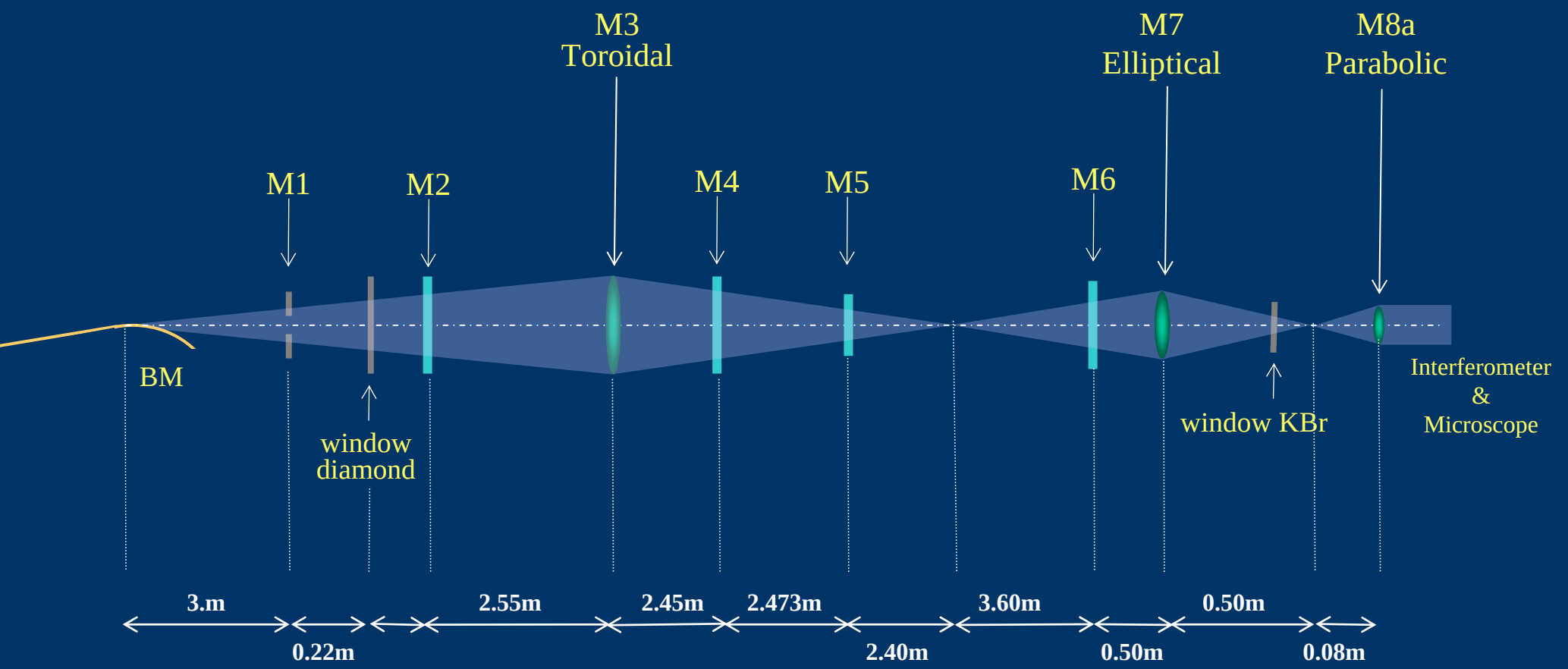
Diffraction-limited resolution of λ/2 (λ: 2→12μm)

G.L. Carr, *Rev. of Scientific Instruments*, **2001**, 72, 1613

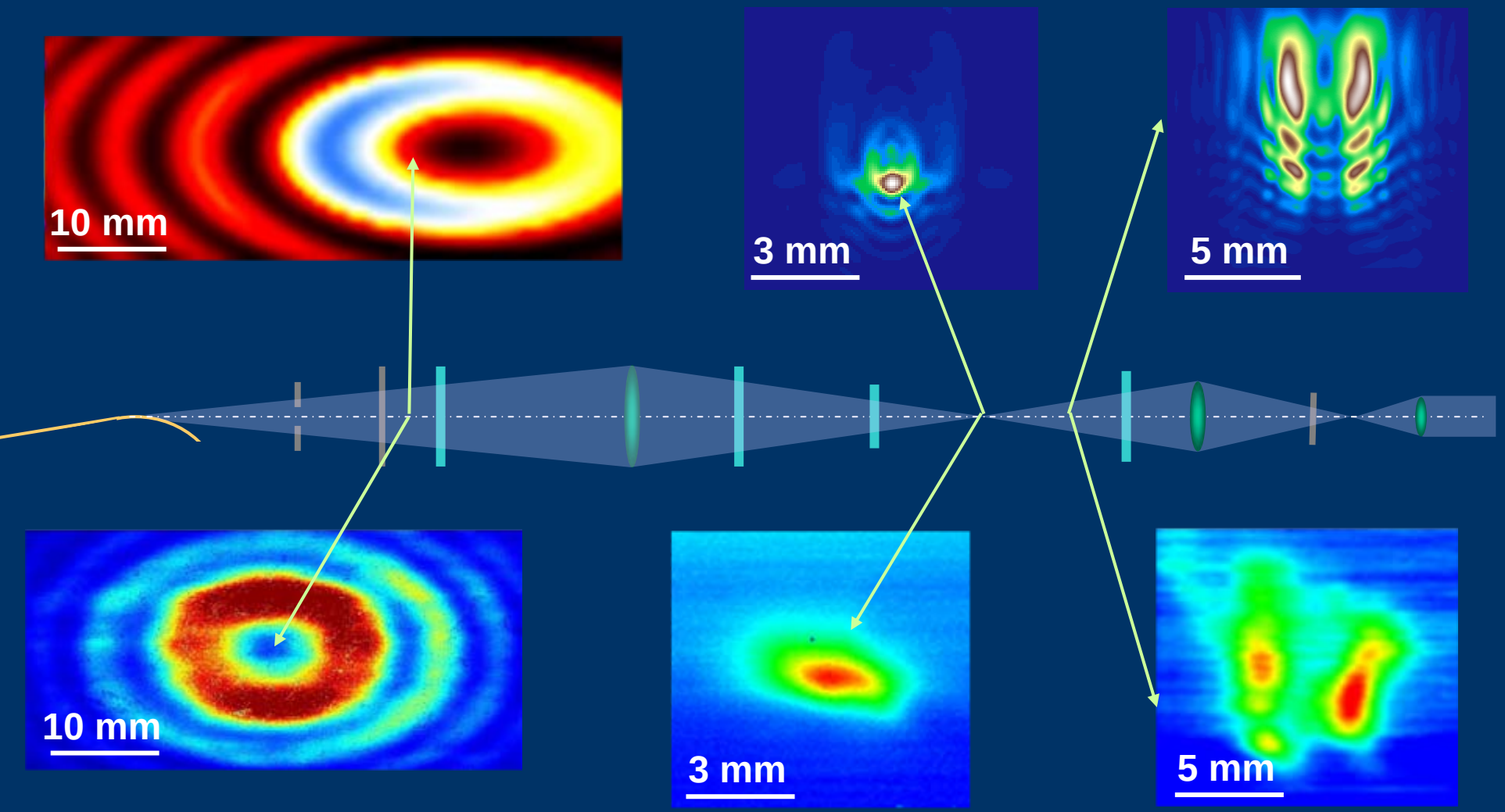
ID21 – Infrared microscopy end-station



ID21-IR: Optical pathway



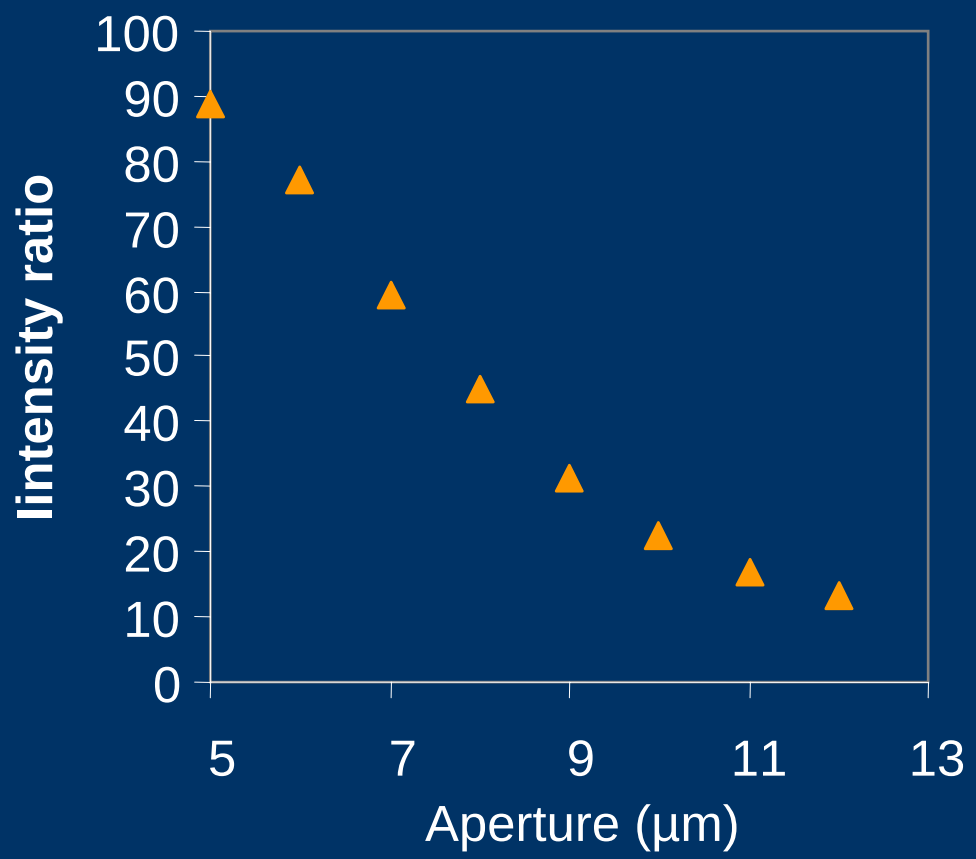
SRW code computations at 10 μm

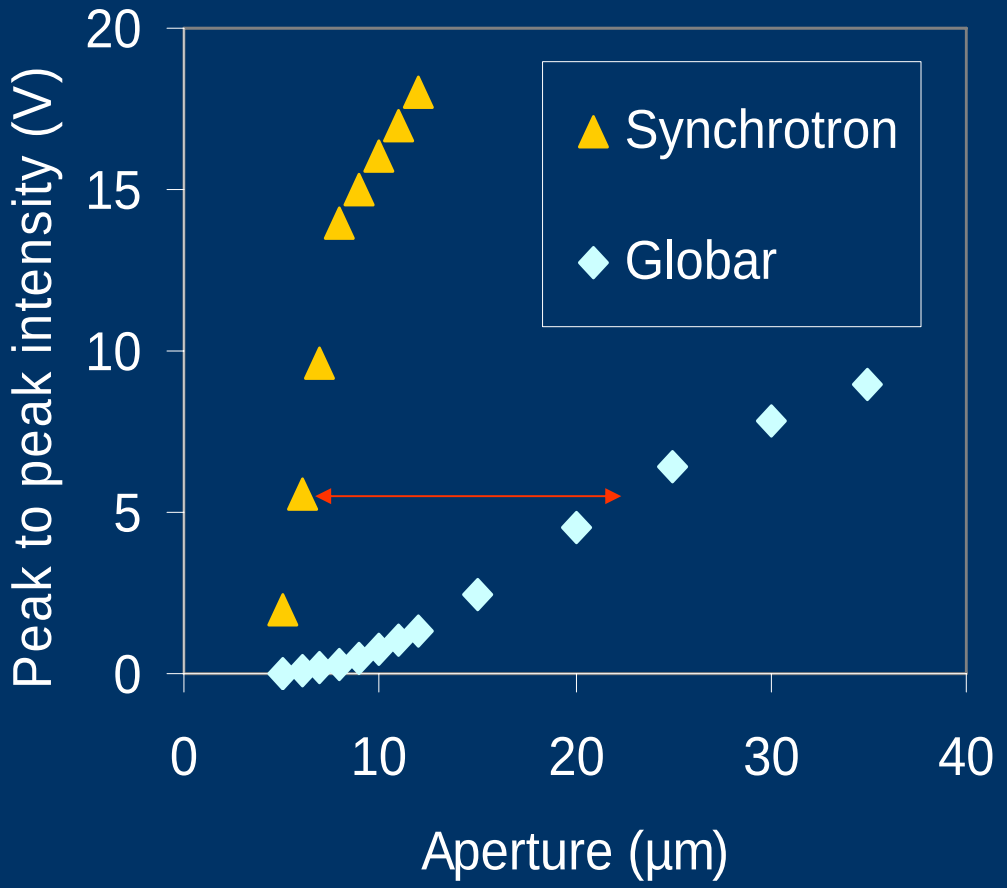


Measured intensity maps (integrated from 2 to 12 μm)

Synchrotron vs Globar

ID21-IR





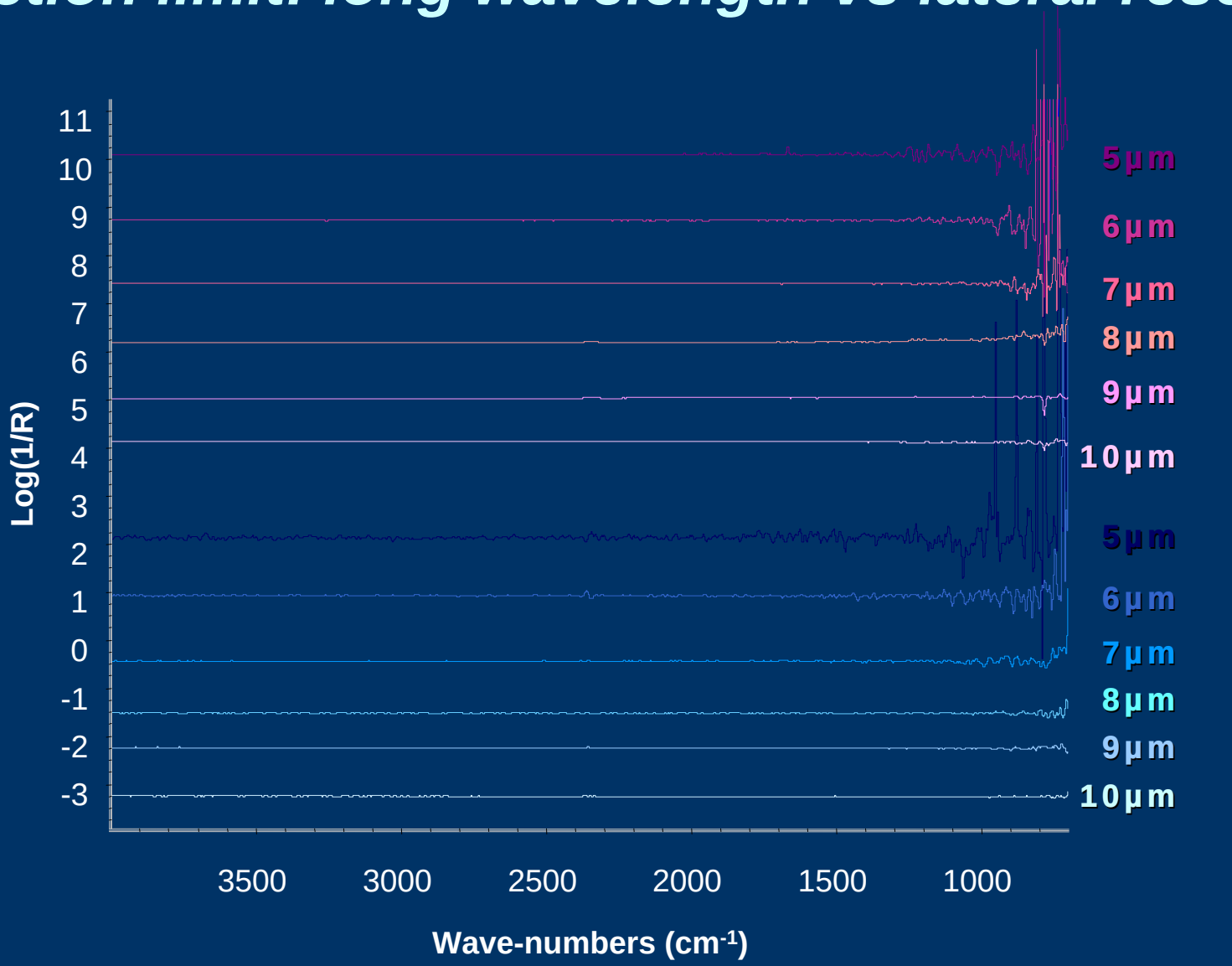
Synchrotron

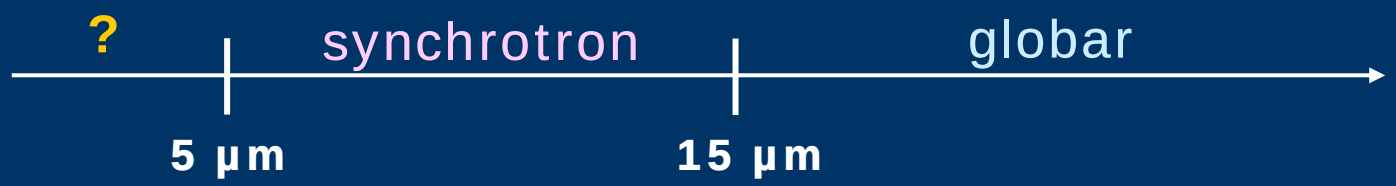
6×6 µm²

Globar

22×22 µm²

Diffraction limit: long wavelength vs lateral resolution





Synchrotron based IR-SNOM ?

6/12/2004

- F. Bertin (CEA)
- R. Casalegno (SPECTRO)
- A. Chabli (CEA)
- F. Comin (ESRF)
- M. Cotte (SPECTRO)
- P. Dumas (SOLEIL)
- M. Faucher (CEA)
- E. Garcia-Caurel (EP)
- S. Huant (SPECTRO)
- R. Ossikovski (EP)
- C. Rambaud (SPECTRO)
- N. Rochat (CEA)
- P. Royer (LNIO)
- J. Susini (ESRF)
- JJ. Yon (CEA)
- *P. Chaton (CEA)*
- *JM. Ortega (CLIO)*
- *B. Drevillon (EP)*

- Source ?
- Illumination ?
- Collection ?
- Excitation ?
- Detection ?
- Diapason ?
- Aperture ?
- Modeling ?

- N. Rochat (CEA)
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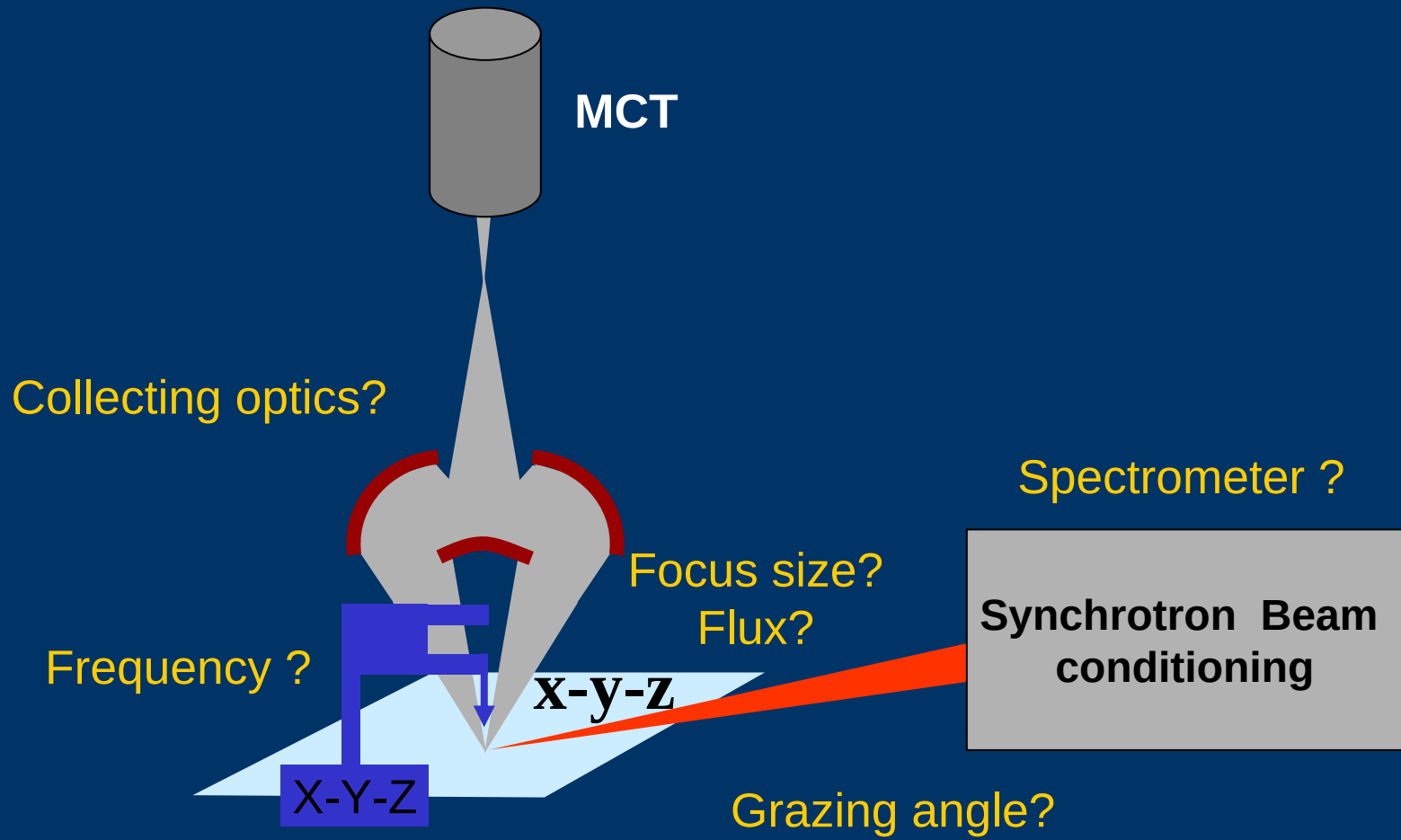
- M. Silveira (ESRF-UJF)
- N. Chevalier (CEA-UJF)
- S. Huant (UJF-CNRS)

Prototype with diapason
Feasibility tests at ID21

IR-SNOM: Test of principle

as of Nov. 2005



- **Minimizes modification of the current microscope configuration**
- **Benefits from existing equipment (microscope + spectrometer)**