
Synchrotron radiation and the ESRF
Wim Bras
DUBBLE @ ESRF
Netherlands Organisation for Scientific Research (NWO)
NWO
FWO
VLAANDEREN

# Outline

- Short introduction synchrotron radiation
- European Synchrotron Radiation Facility
- DUBBLE
- Examples of research on Dubble

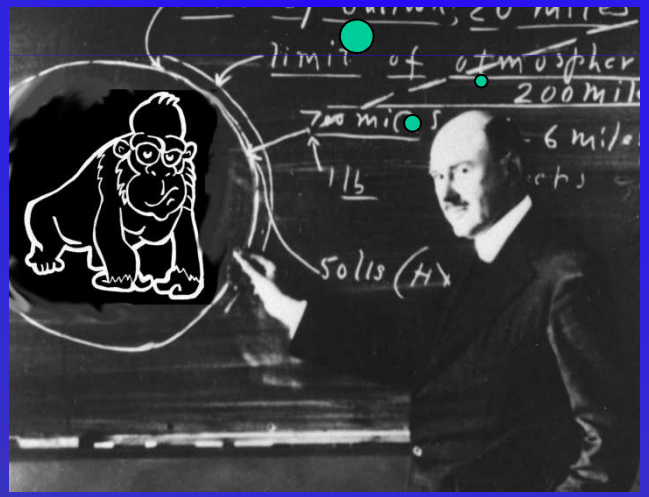
Commercial possibilities indicated in *Italics*

# Meet the Röntgens

So you think this is funny mr Röntgen?

I'll kick him out of my school.

Prof. Conrad Röntgen
First Nobel price winner
1845 - 1923
1848-1865 Apeldoorn




1864


Mrs. Röntgen

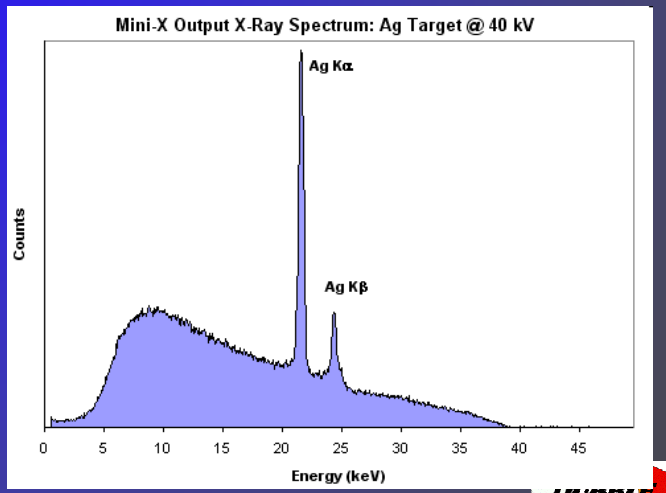
# How do we conventionally generate X-rays?

- With an X-ray generator


Panalytical Almelo

- Synchrotron radiation

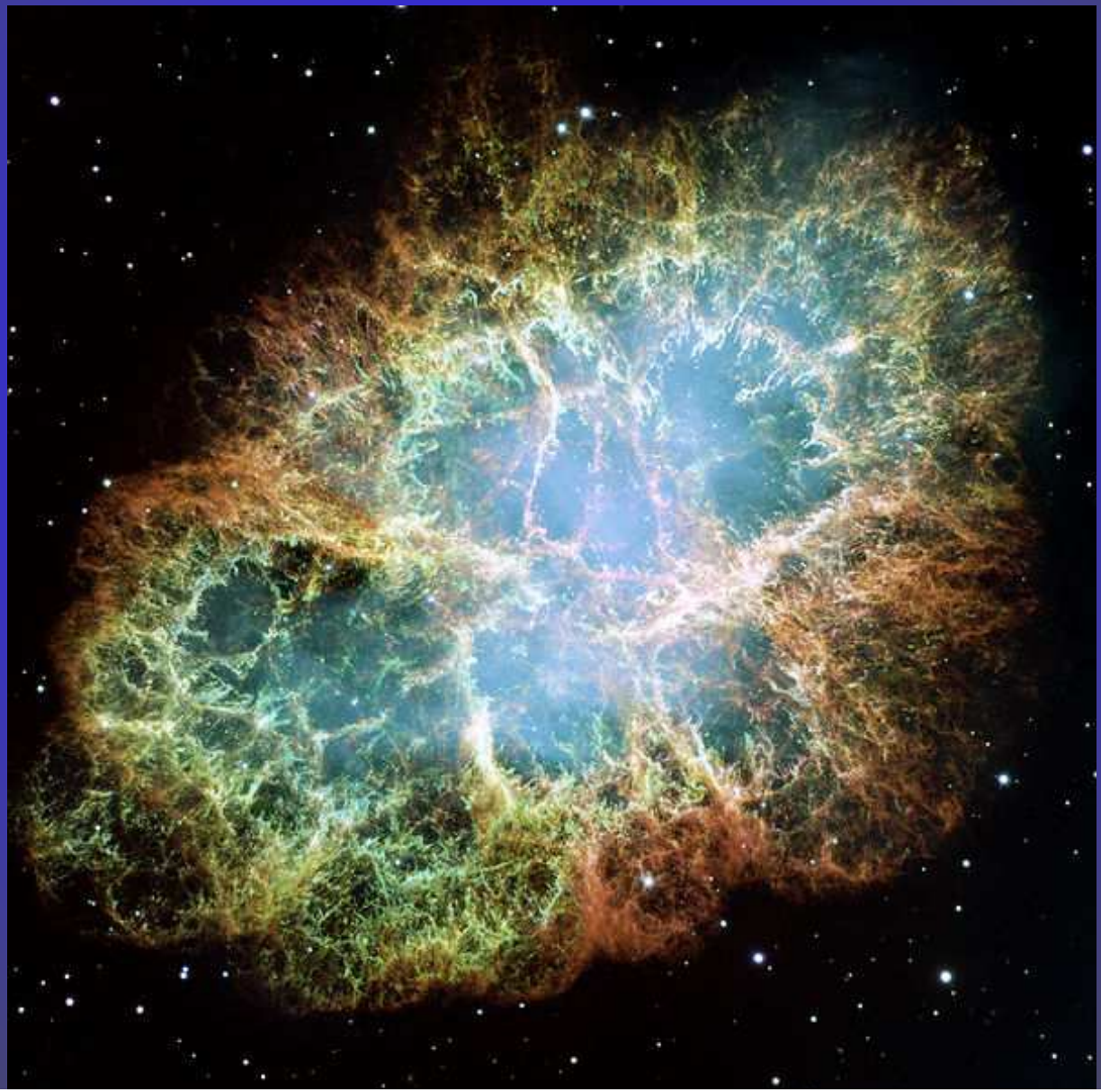
# “Conventional” X-ray tube

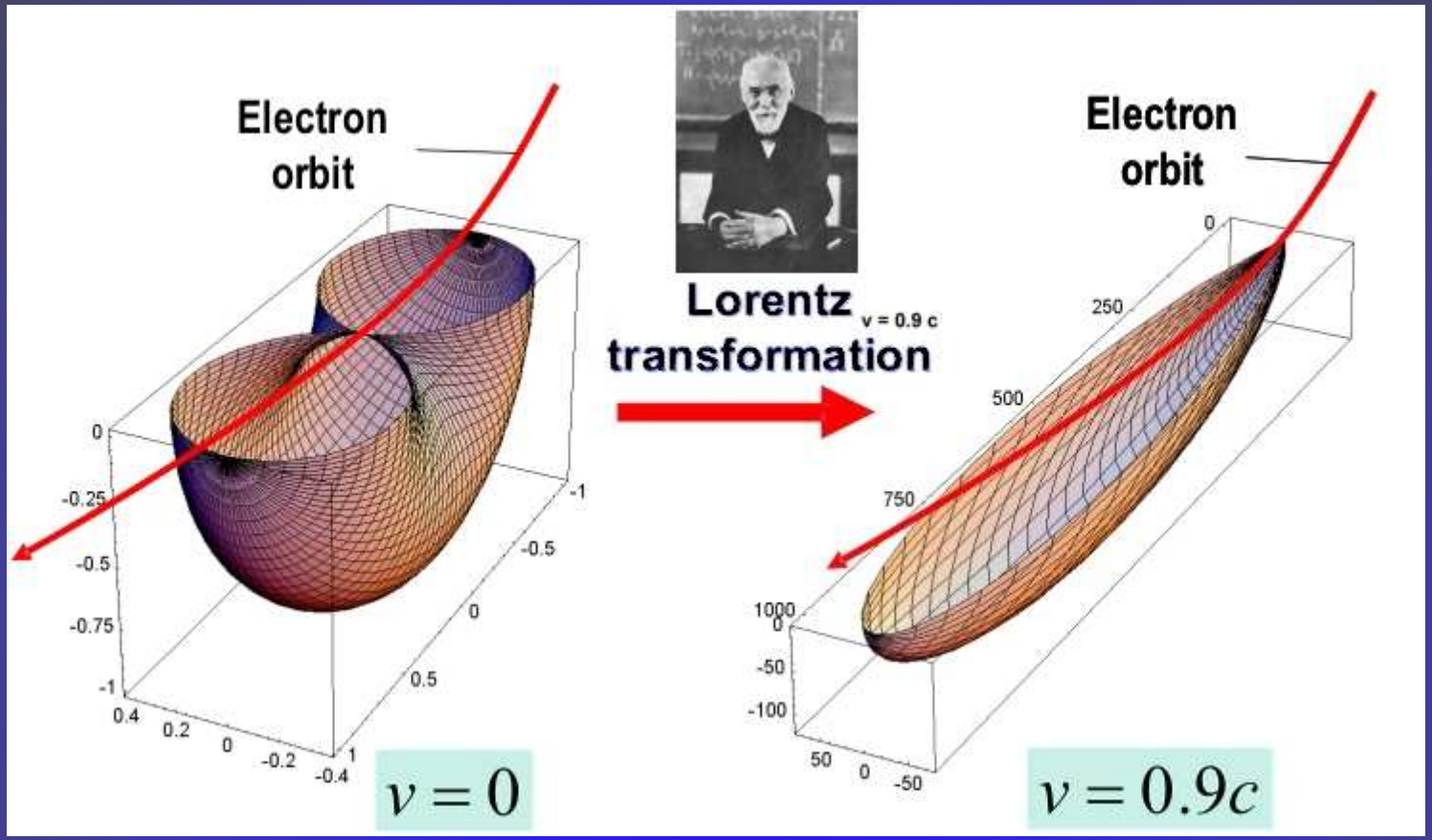


# Synchrotron radiation

- X-rays generated by fast moving electrically charged particles forced to change their direction
- Natural phenomenon

# Crab Nebula: Blue stuff is SR

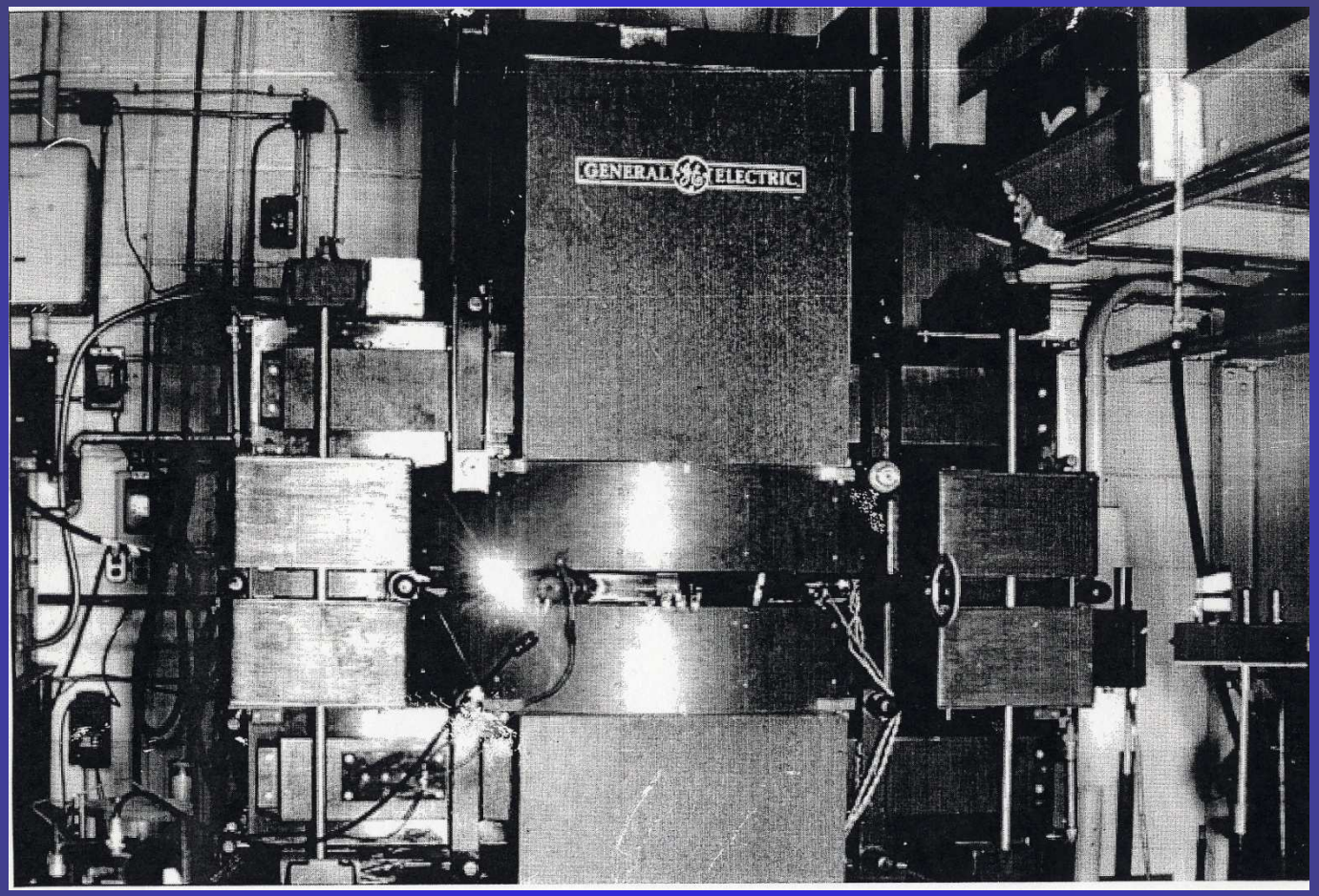




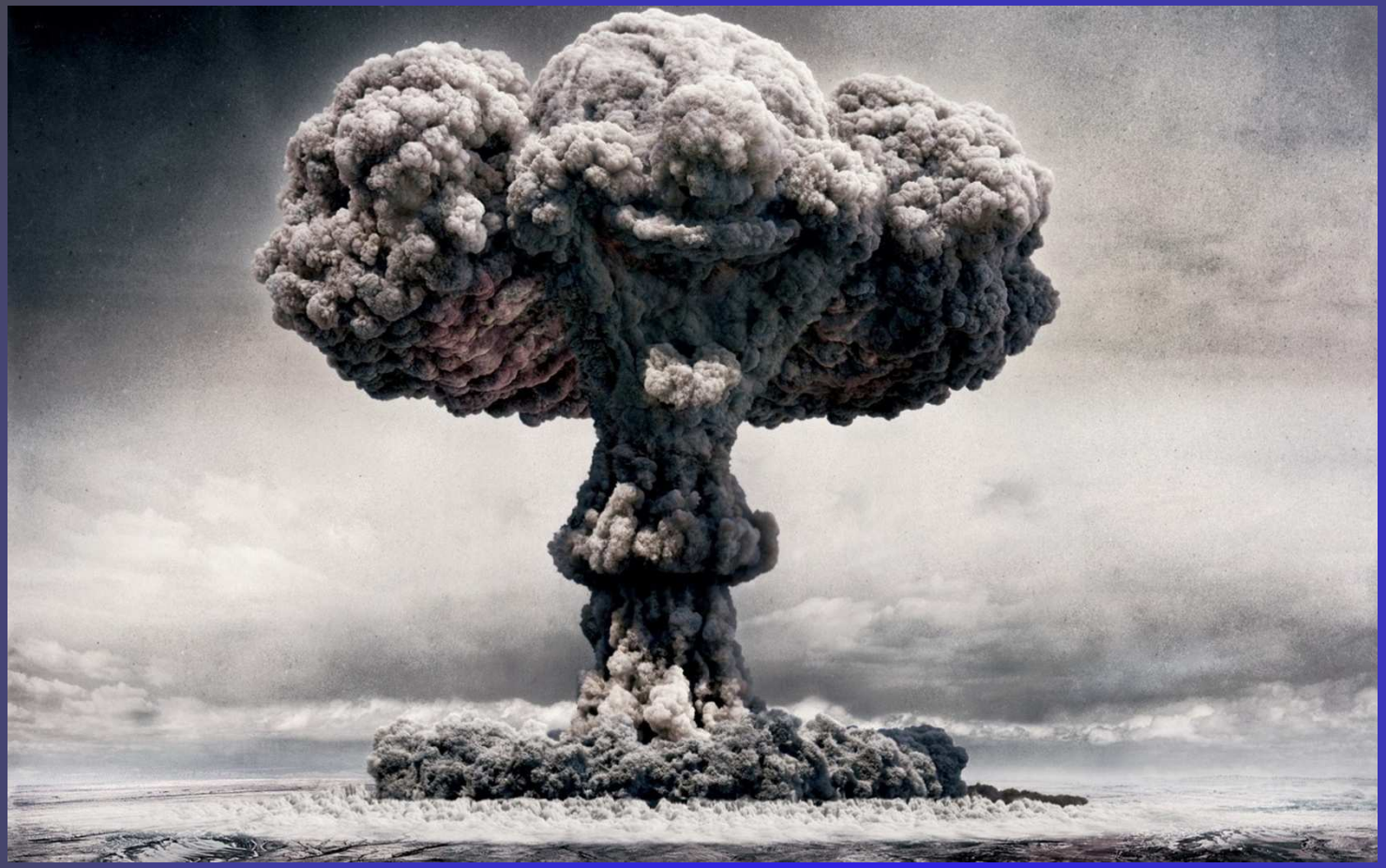

‘slow’ electrons
(dipole radiation)
(think of antenna)

‘fast’ electrons

# 1947




Why would General electric be interested……?



1953

# Family tree

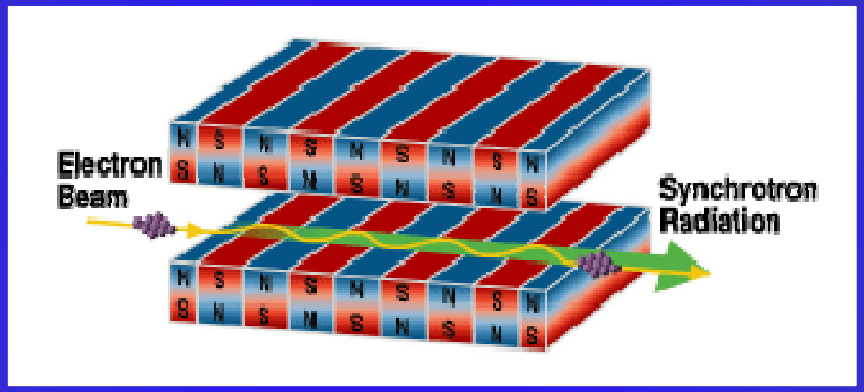
CERN

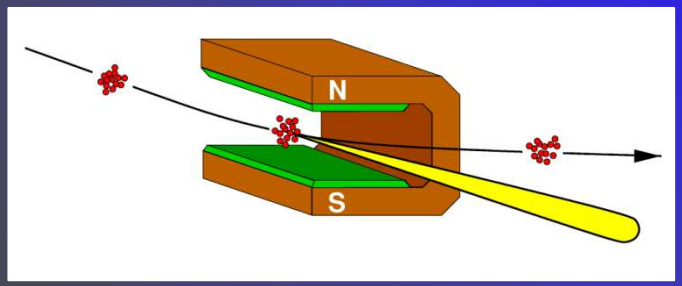
1936

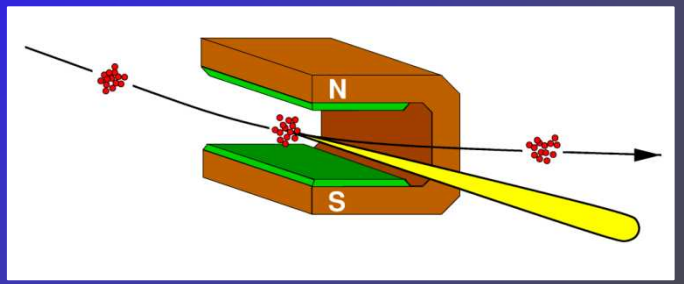
ESRF

# Magnet lattice

Undulator or insertion device



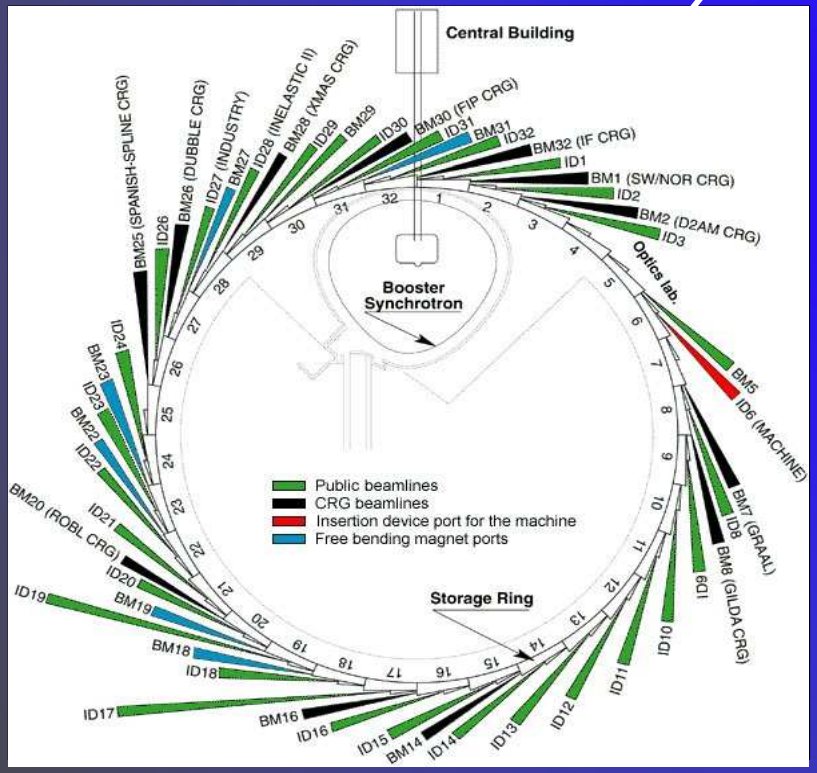



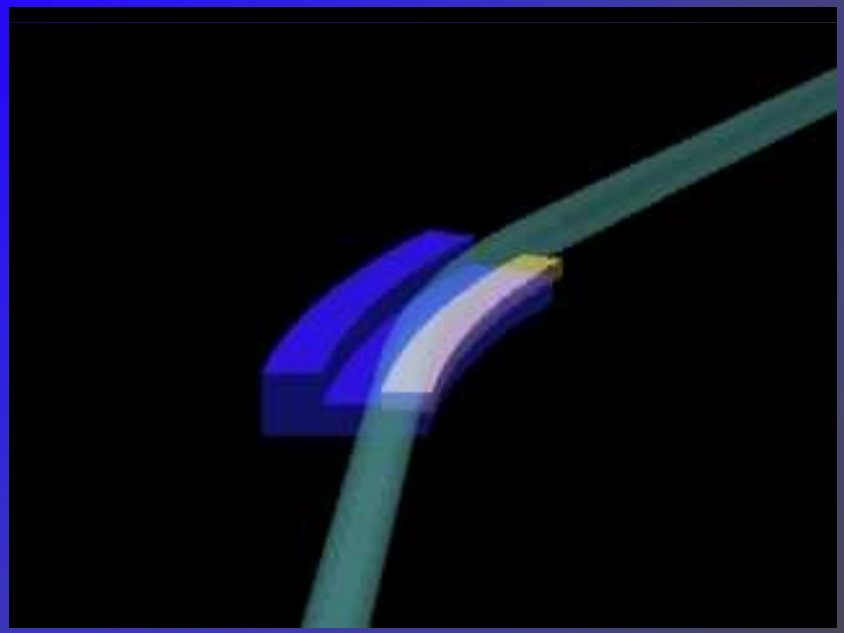




Bending magnet

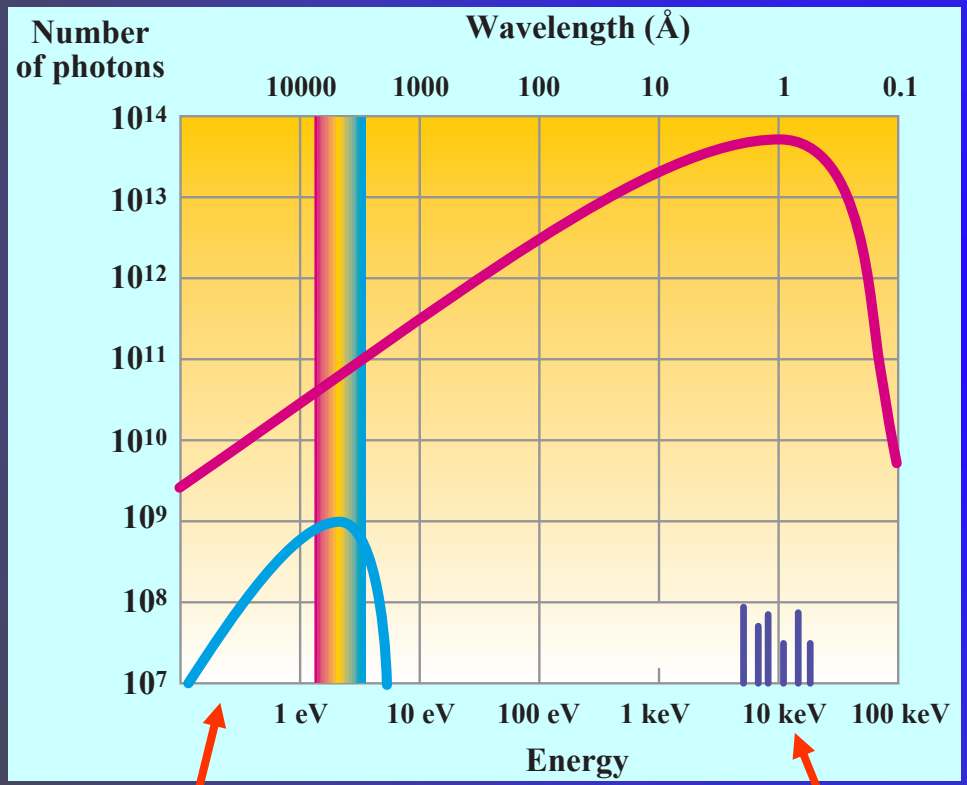
# Synchrotron radiation sources

Bending magnet



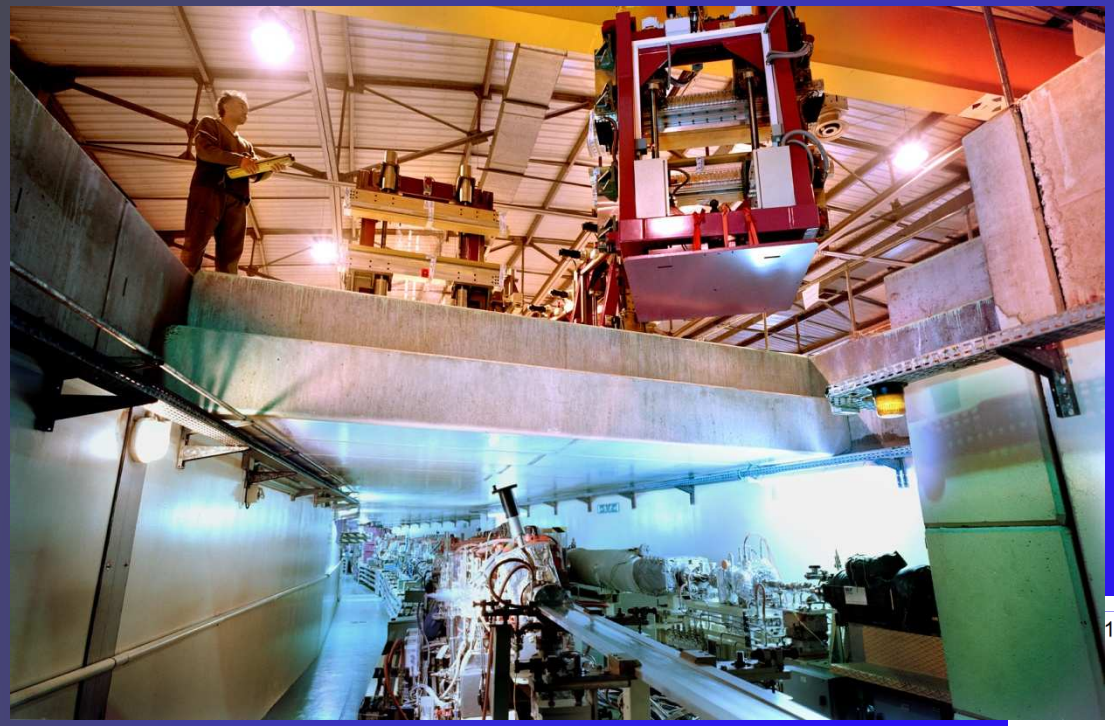
# What is the advantage of SR?




sunlight

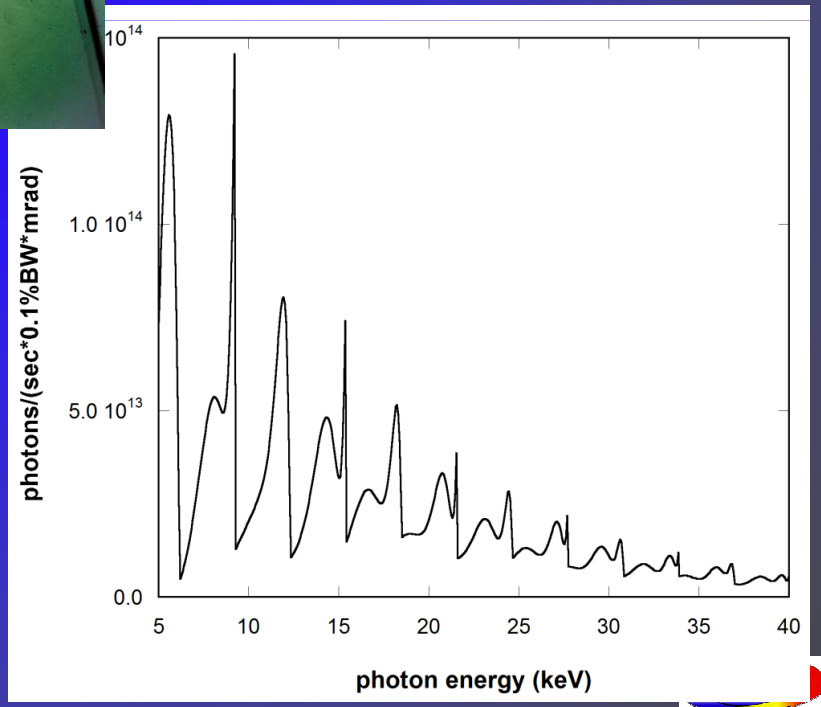
conventional
X-ray generator

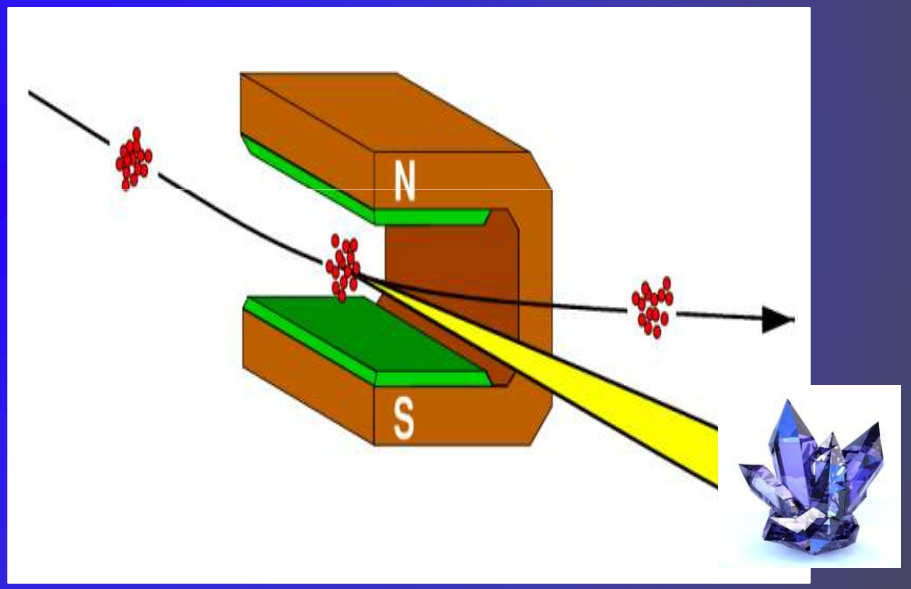
- High flux
- Continues spectrum
- Good collimation
- Beams 1 – 300 micron



*Boessenkool BV Almelo*

# undulator




N
S

# Synchrotron labs

- These labs are set up as user facilities
- They are relatively large scale
- Belgium and the Netherlands have made the choice to participate in international facilities

# Dutch ESRF participation

- 6% of the experimental time on public beam lines is for the Benesync consortium (50/50 Be/Nl)
- This is not sufficient for the Dutch/Belgian demand for some techniques
- Two ‘own’ beam lines
- DUBBLE (part of the Dutch research infrastructure)

- *6% of ESRF expenditure should be in Belgium-Netherlands*

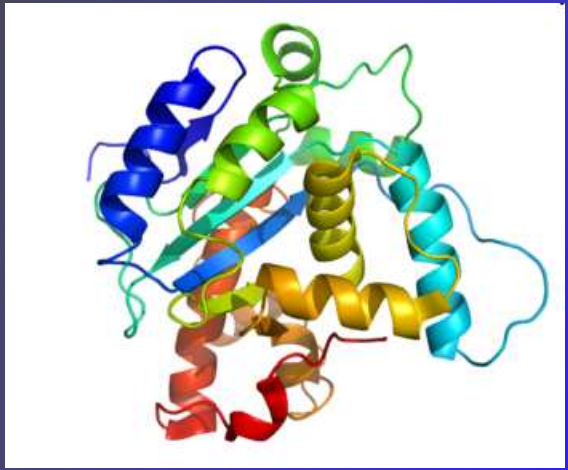
# European Synchrotron Radiation Facility

Grenoble France

# European Synchrotron Radiation Facility

- ~ 50 beam lines (= experimental set ups)
- ~ 25 different techniques
- 6 days/ week, 24 hours/day
- 270 days/year (minus strike days, after all it is France….)
- ~ 1500 publications/year

# Many different experiments

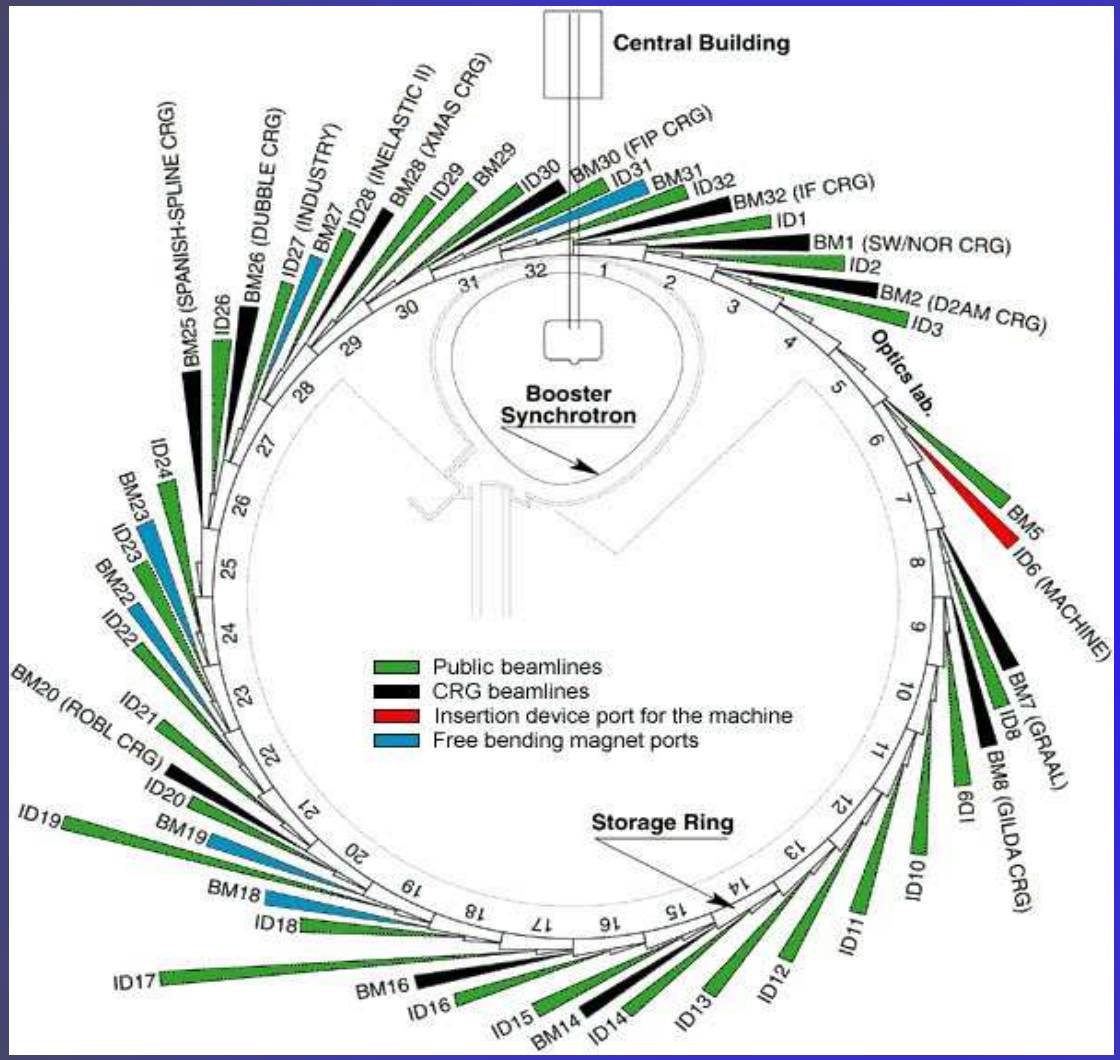


Protein crystallography
About 25% of the beam lines
About 35% of publication output

Crystal distortions due to high (30 Tesla) magnetic fields


Chocolate butter crystallisation

Spider silk




270 meter

the ESRF

# ESRF accelerator

- 1 km circumference
  - *1 km high vacuum*
  - *1 km of electronics controls required*
  - *1km of diagnostics of all kinds required*
  - *metrology*

  - *Feenstra (Dalfsen), supports for vacuum vessels*

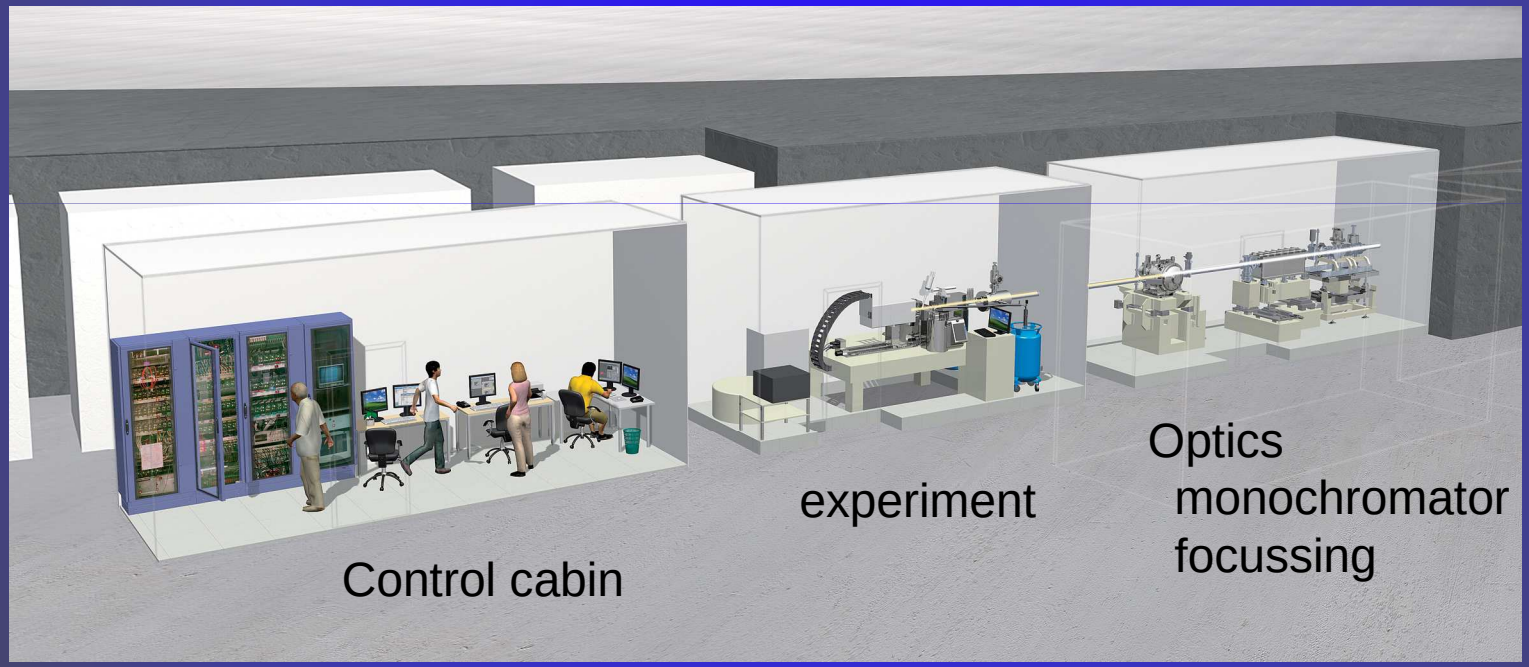
# Around 50 experimental stations

- Beam line optics
  - *High vacuum*
  - *Fine mechanical engineering*
  - *Mostly custom built*
- Experimental station
  - *Large variety of requirements/sales opportunities*

# X-ray optics

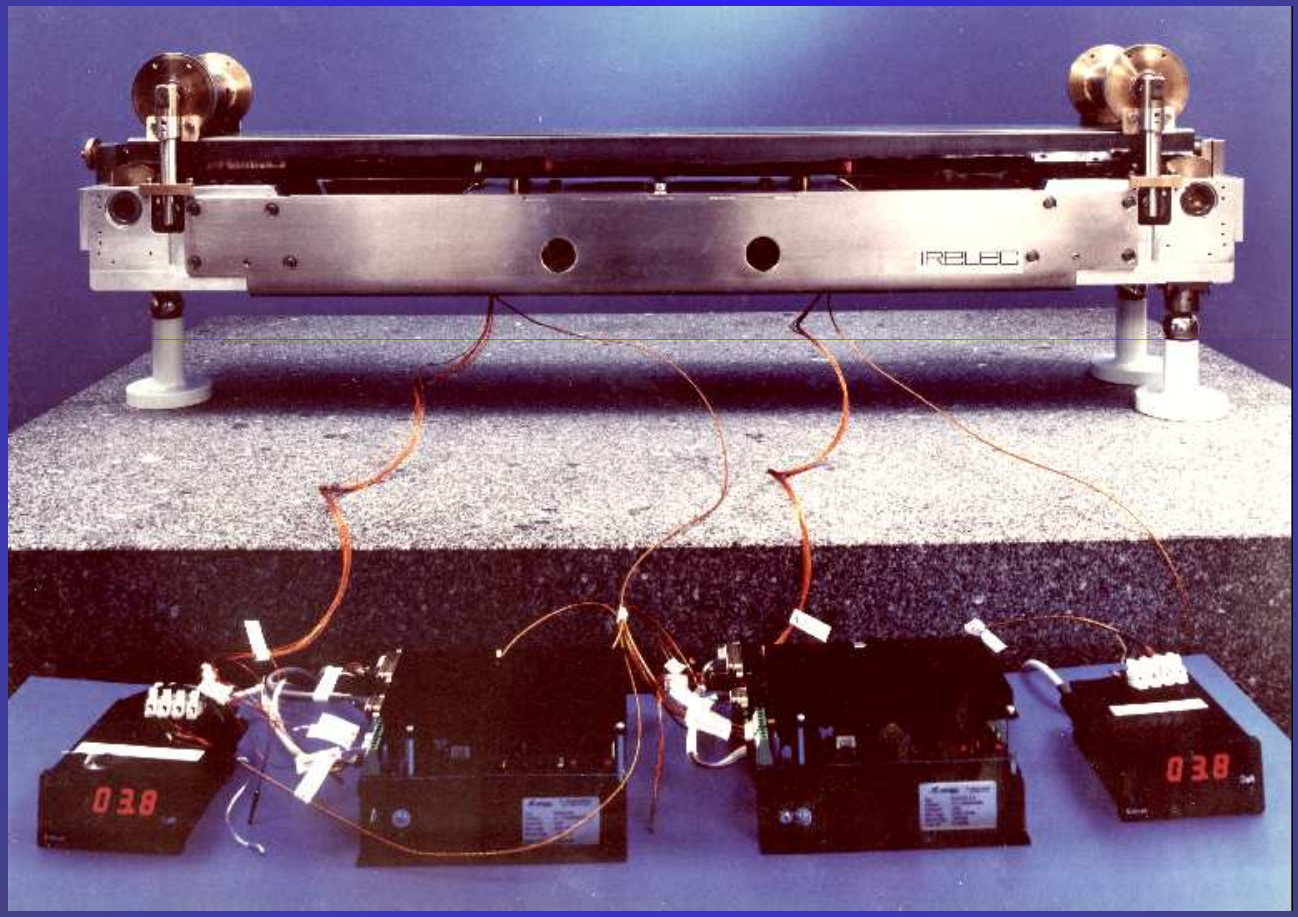
- X-rays come out of machine
  - Slightly divergent
  - Polychromatic
- For most experiments
  - Requires monochromatic
  - Requires focusing

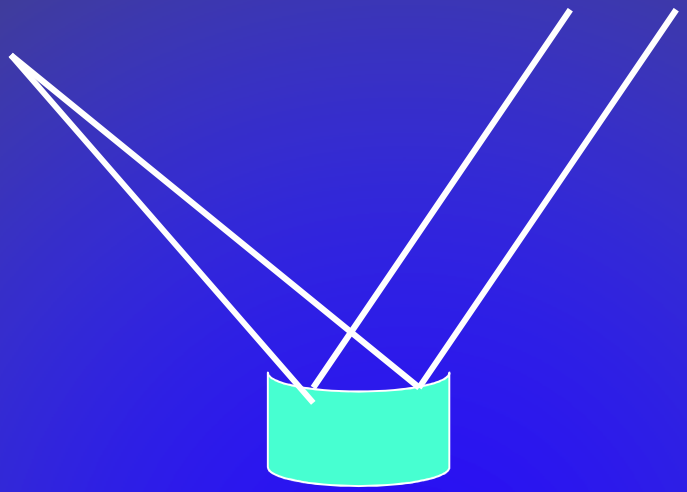
# Scheme beam line




storage ring

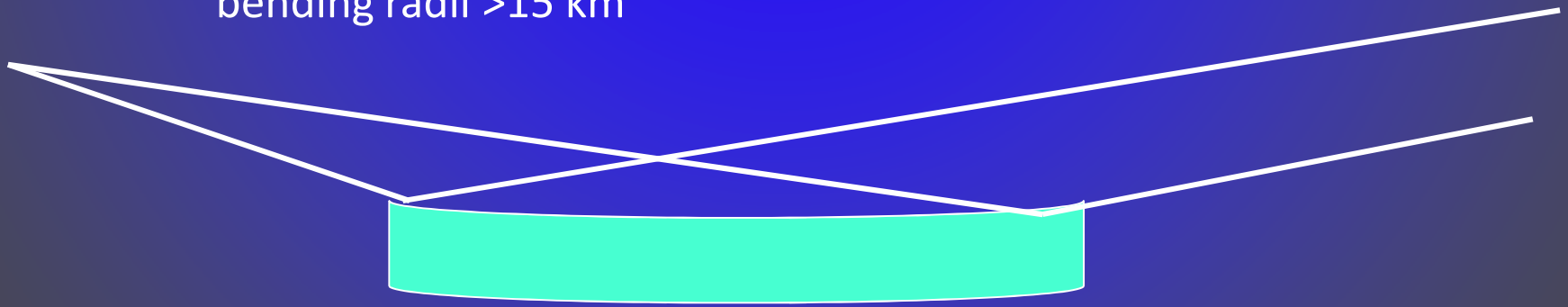
# Mirror: grazing incidence: focussing





Conventional mirror

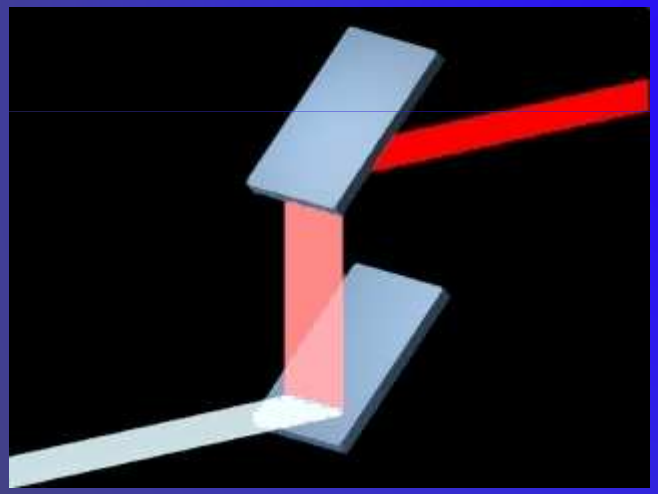
X-ray mirror, Si of semiconductor grade, shallow angles, otherwise absorption
Length up to 1.5 m
roughness < 1.5 Å
slope error < 1.5 mrad
bending radii >15 km



# In real life

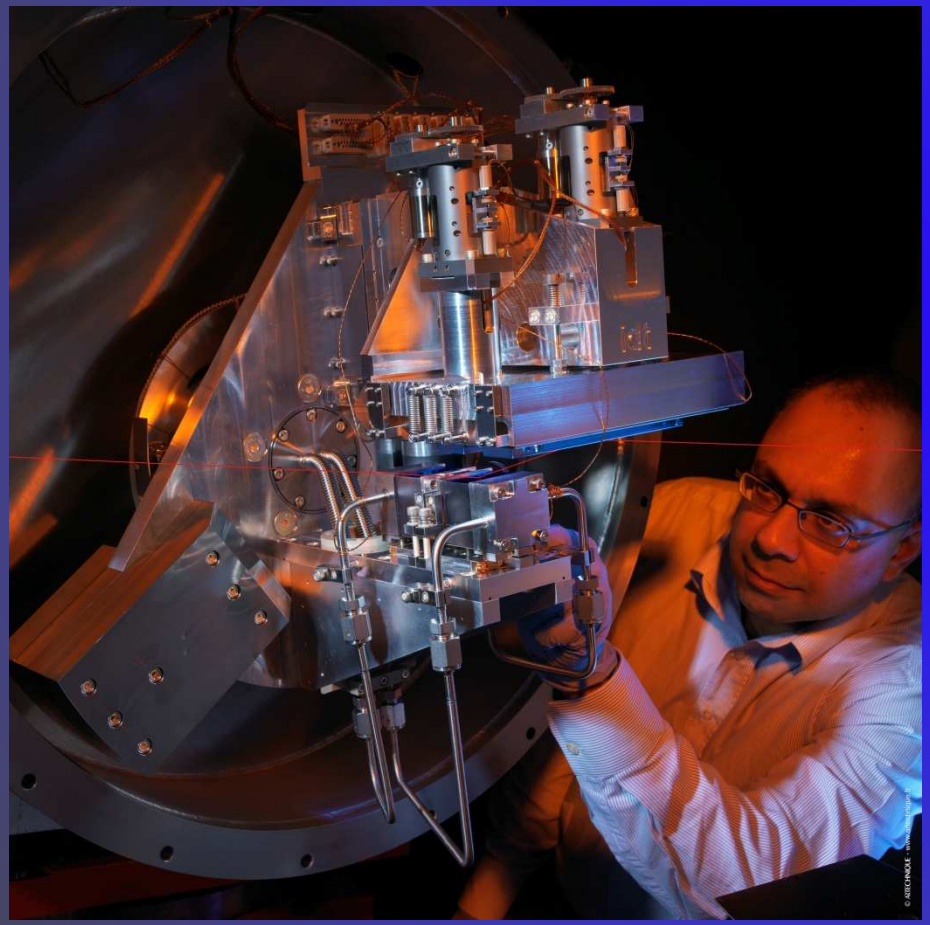
Positioning accuracy, angular  mrad, linear  2-5 micron, bending very accurate

# Monochromator



- Most often two pieces of silicon
- Positioning accuracy to beam, angular very high
- To each other high
  - Roll
  - Yaw
  - Offset
  - Pitch

# Monochromator



Some specialized companies exist
Relatively small market

# Monochromators and mirrors

- *High vacuum ($10^{-7}$ – $10^{-9}$)*
- *High radiation environment*
- *Positioning all done remotely*
- *Positioning all required to be vacuum compatible and accurate*
- *Cooling issues*
- *Most vacuum vessels custom made*

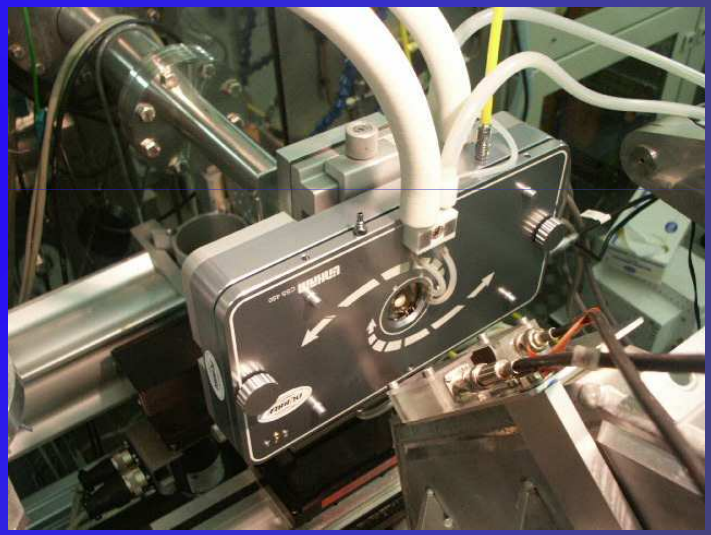
# Sample environment

- *Large variation in requirements*
- *Pressure/temperature control*
- *Gas mixtures/controlled flow*
- *Stop-flow, temp jump, pH control*
- *Magnetic/electric fields*
- *Deformation rigs*

- *Etc. etc.*

# Sample environments

3.0 m



0.2 m

# Technique combinations

Some experiments:

- *Raman*
- *UvVis*
- *Mass Spectrometry*
- *DSC/DTA*
- *Etc. etc*

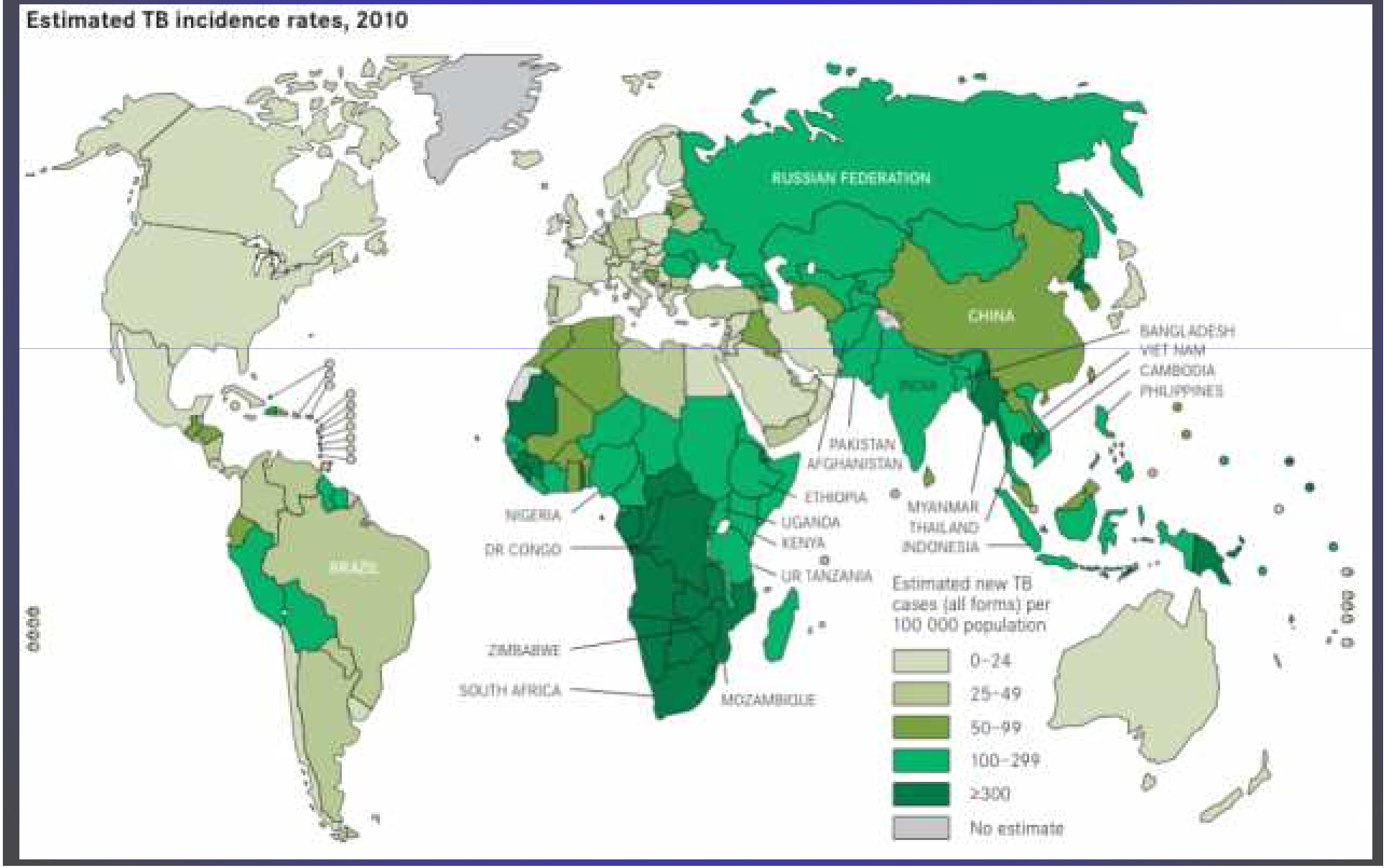
# Sample stages

- Samples + sample environment have to be mounted and positioned
- *Fine mechanical engineering*

# Biochemical research

- Most important single subject of ESRF
- Strong connections with pharmaceutical industry
- About 25% of ESRF beamlines dedicated to protein crystallography

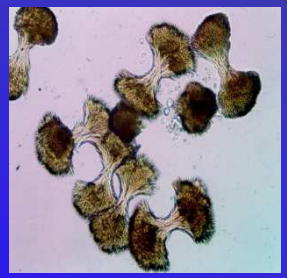
# tuberculosis

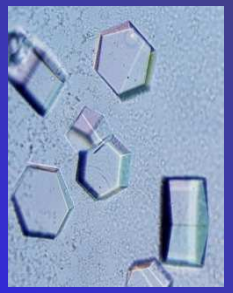


# TB Crystals


Rv2610


Rv3628


Rv1908c


Rv2428

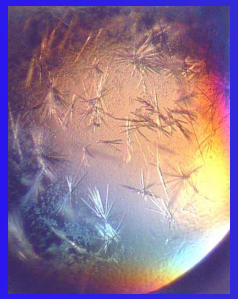

Rv2991


Rv2438c


$Rv0014c_{331}$


$Rv0014c_{279}$


Rv0813c


Rv0018c


Rv1846c


Rv0877


Rv2461c


Rv2276


Rv2883c

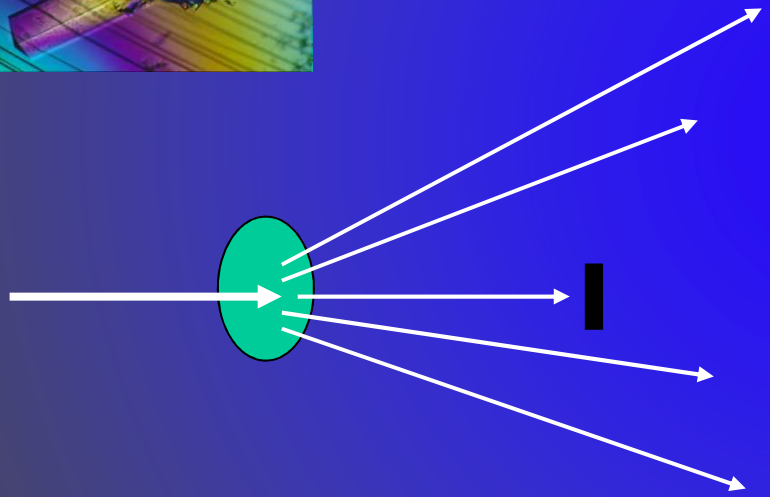

ML2640

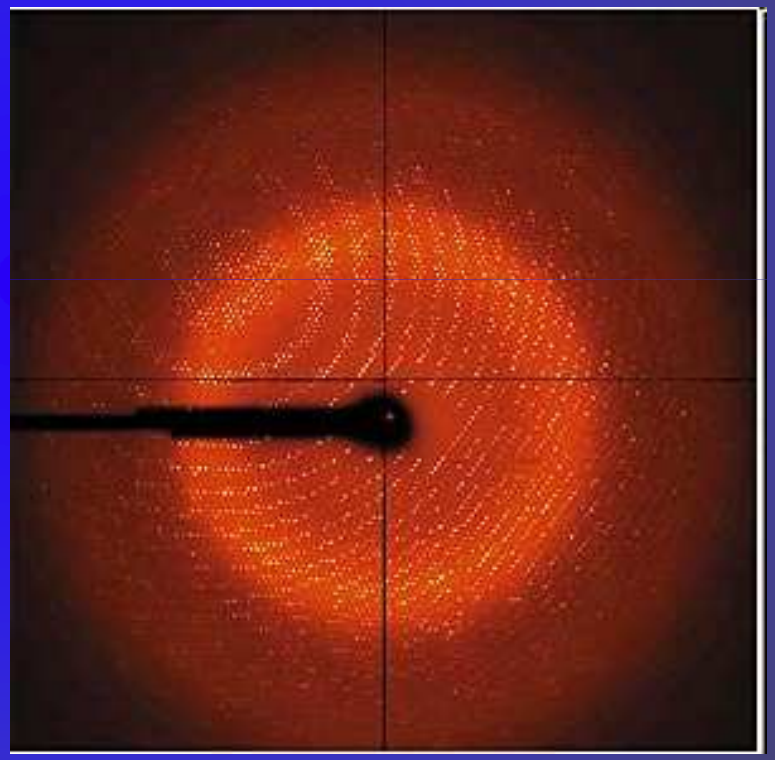

Rv2667

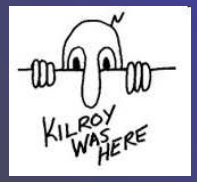

Rv0733

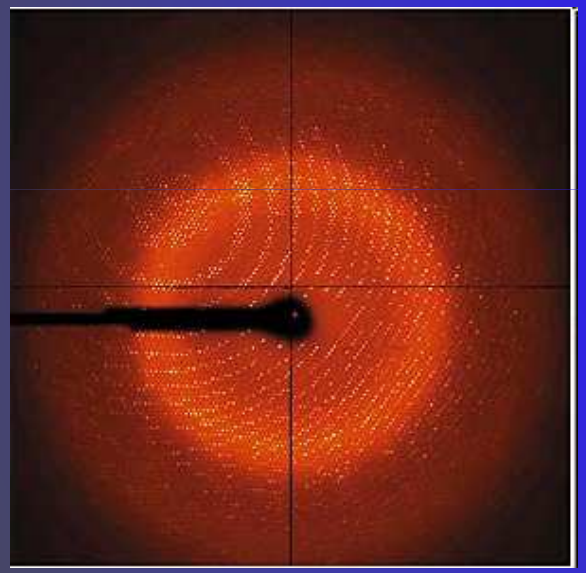

Rv2[illegible]

# Protein crystallographie

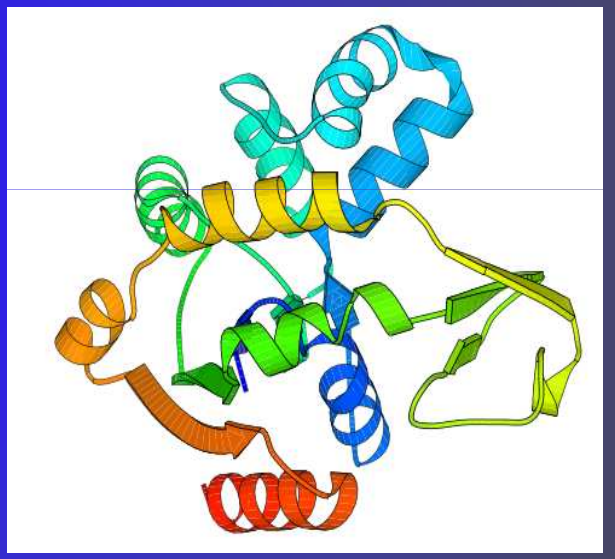


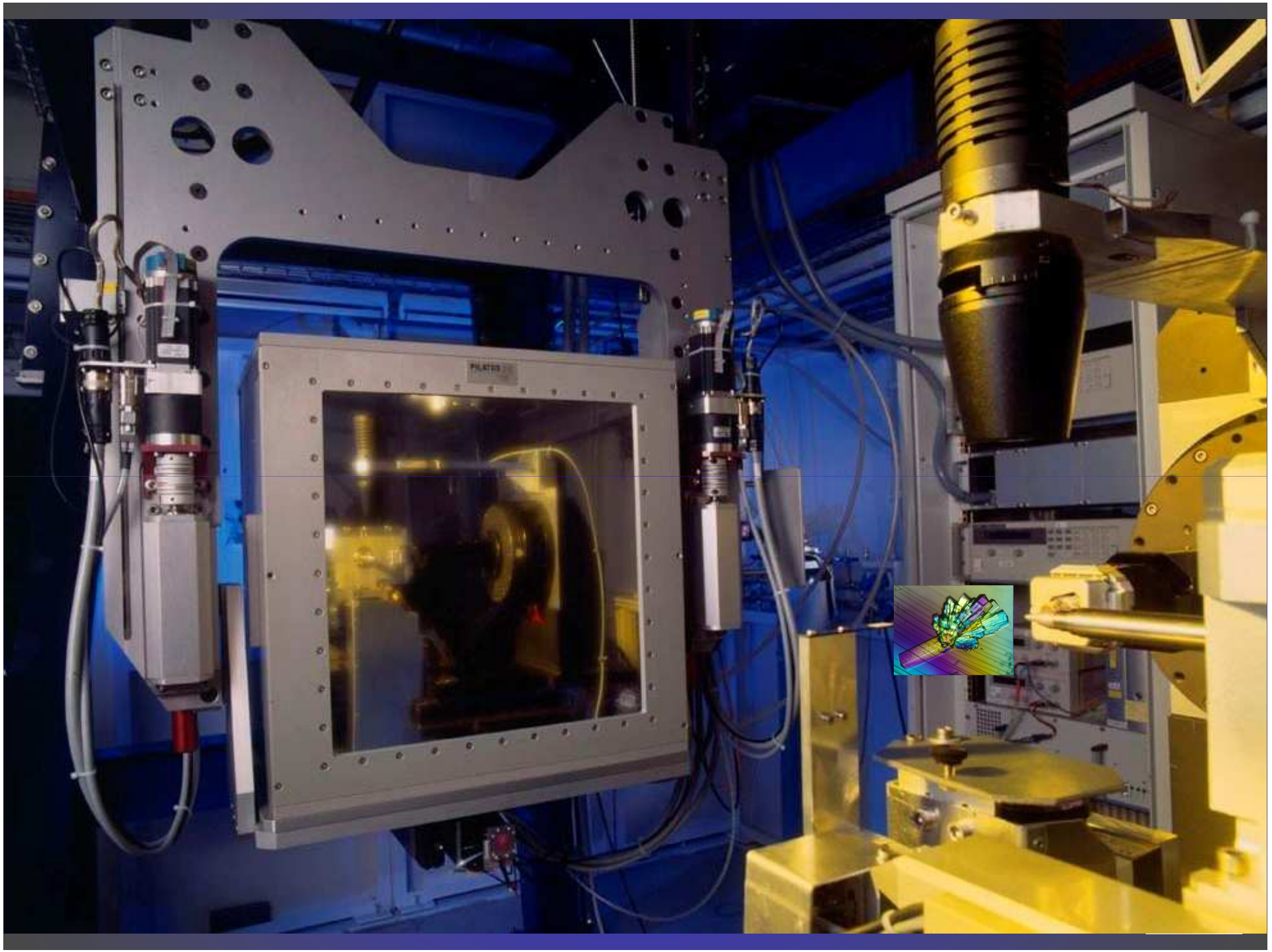




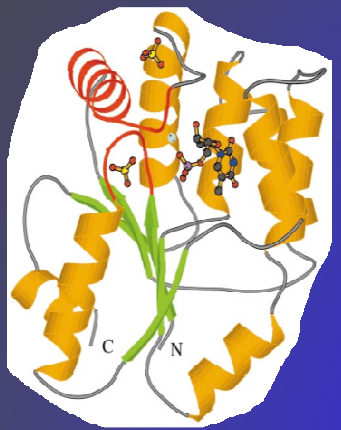


"=" Fourier transform
of electron density

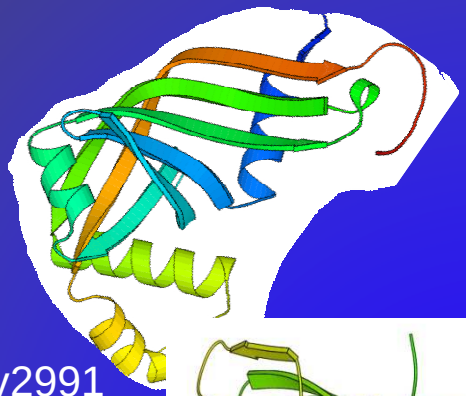




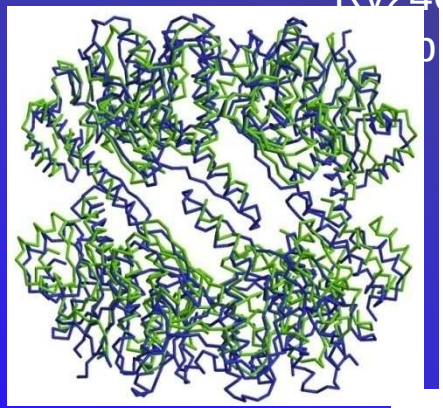
# 3D Structures

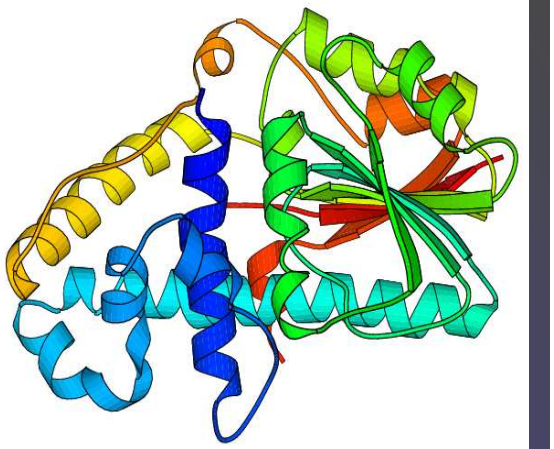


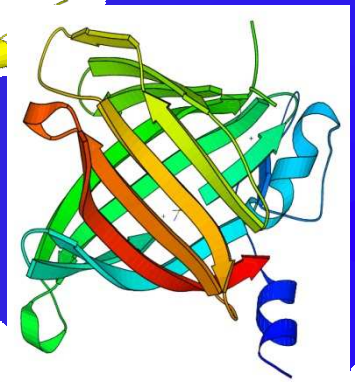

Rv3247c
tmK

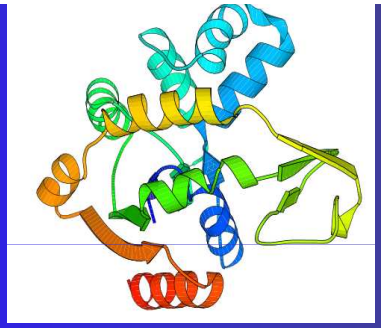


Rv2991

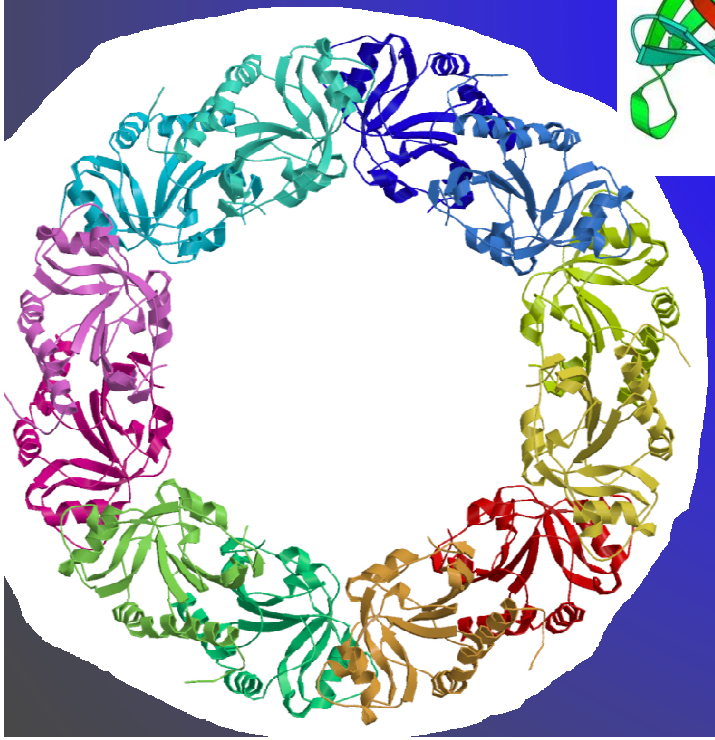








Rv0733
adK




Rv0014c
pknB

# What can we do with this?

- Understanding live cycle of bacteria
- Better understanding about action of antibiotics
- Maybe, just maybe, one day, design new drugs to combat diseases

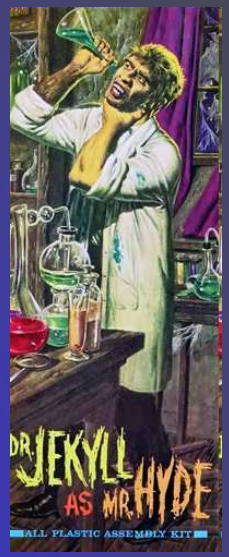
- If no new drugs have been developed due to PX what is the use now?

# taxol

source

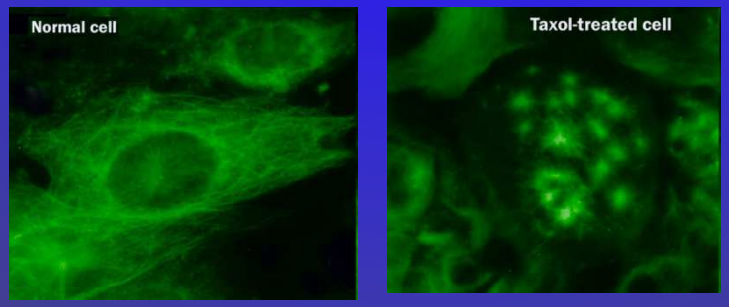
Finish line:
Approval for patients use

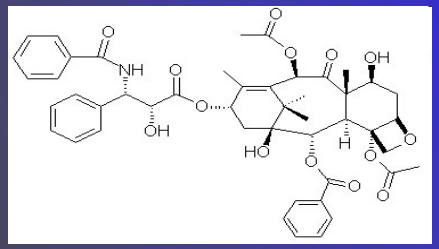
Human tests




Animal tests

Cell cultures



Isolation compound

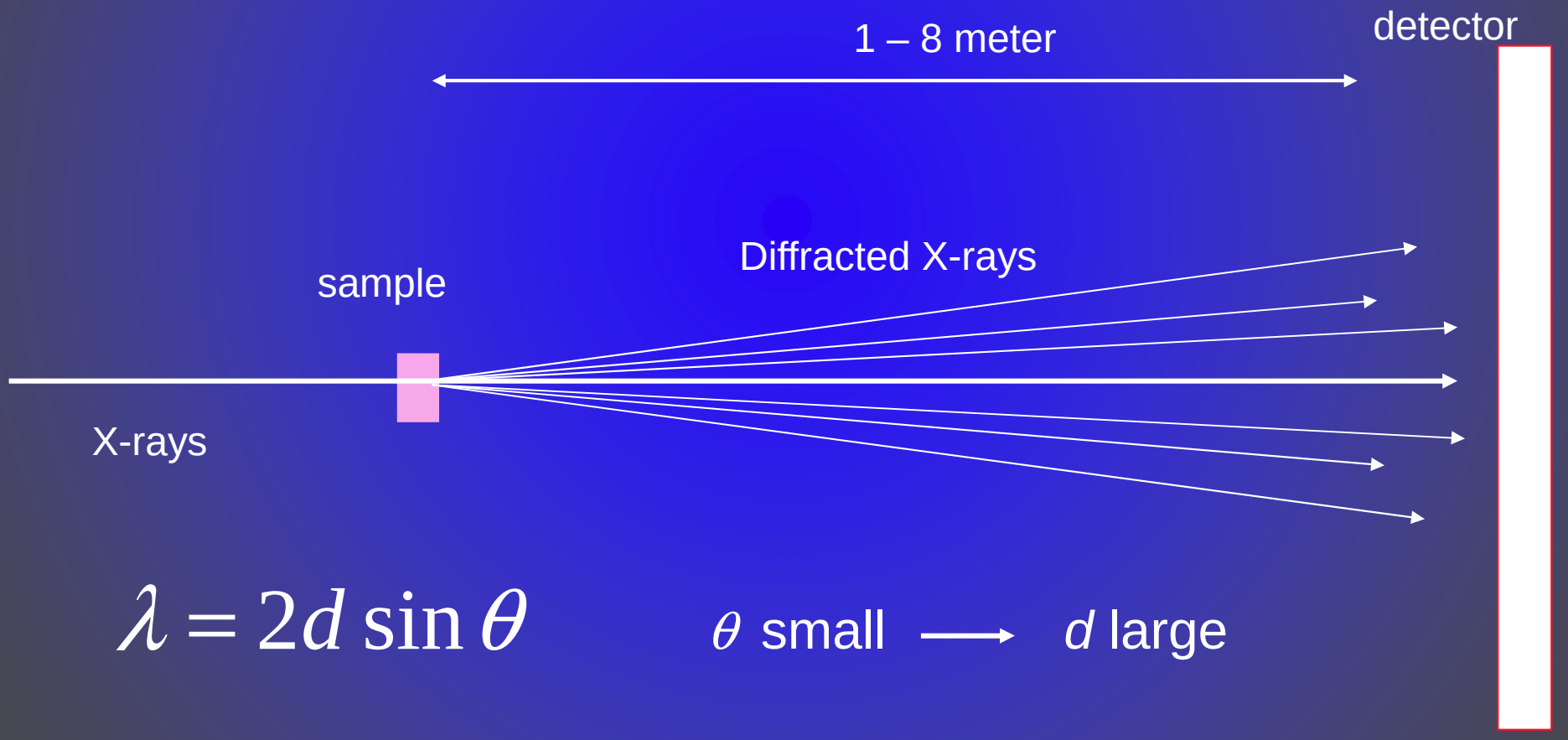
# The time path

1962
discovery taxol

1992
Approved for treatment of
ovarian cancer

Thanks to better understanding of protein structures
nowadays such a time path is reduced to about 15 years

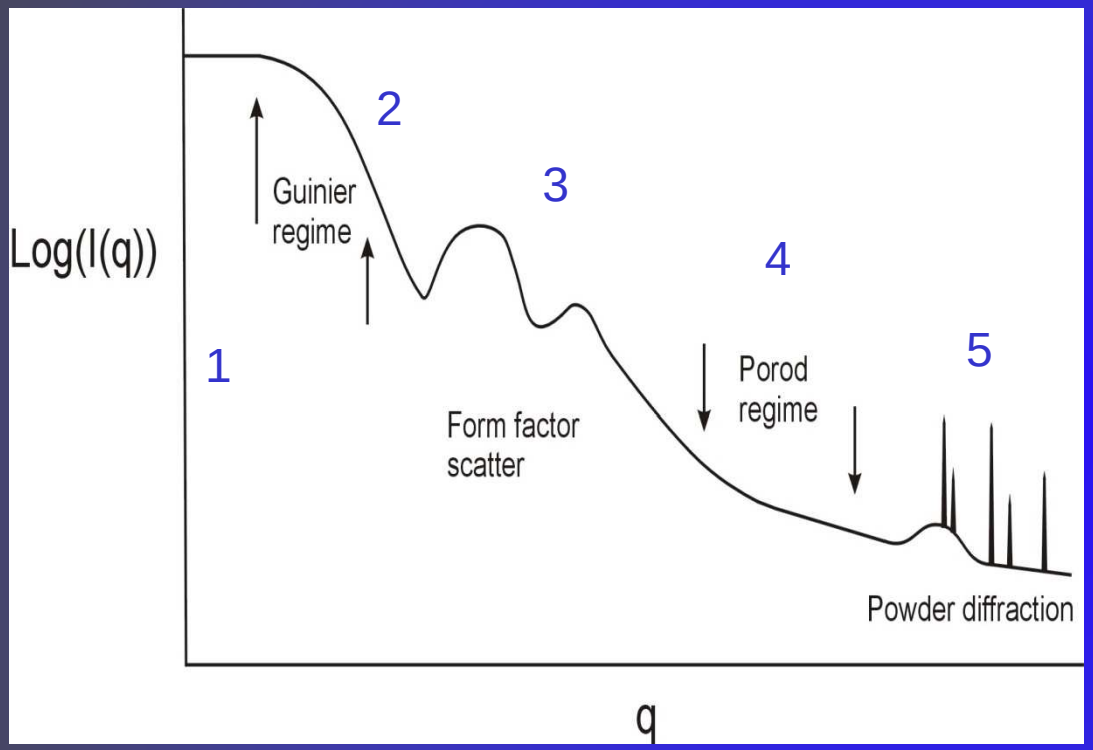
# X-ray scattering




$$\lambda = 2d \sin\theta$$

$\theta$ small ⟶ $d$ large

# SAXS/WAXS




500 nm

0.2 nm

1 limit $q \rightarrow 0$
electron density contrast
density fluctuations

2 Guinier range
particle size

3 particle shape

4 Porod range
particle surface

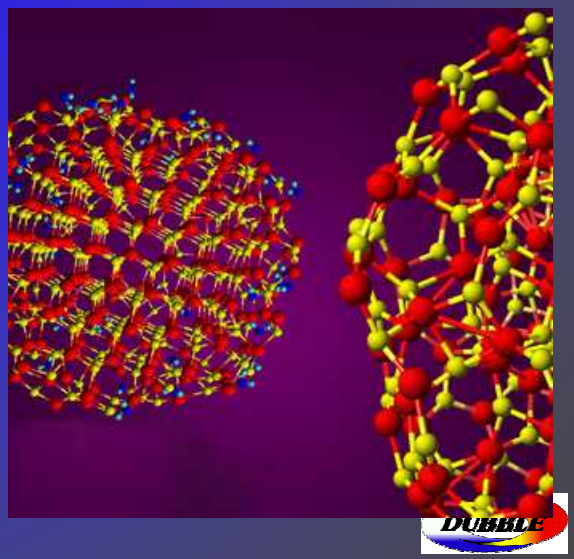
5 Intermolecular/atomic ordering

# X-ray scattering and diffraction

- Combined SAXS-WAXS experiments
- WAXS crystalline structure
- Small angle: shape and size of clumps of atoms

- SR not only static
  also time-resolved

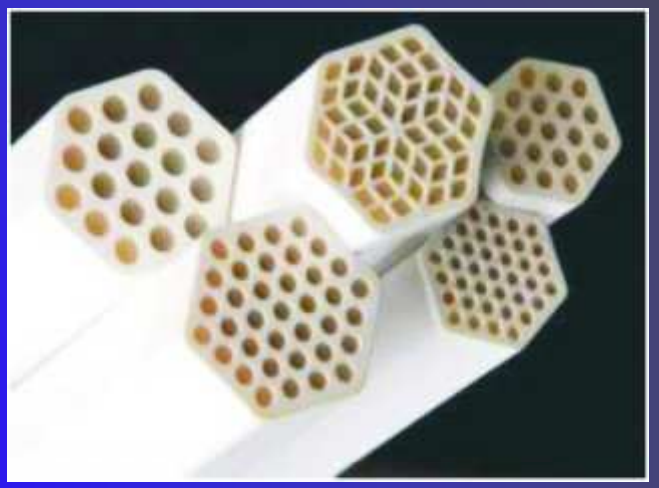
- 0.4 – 200 nm ‘visible’



# Multidisciplinary applications

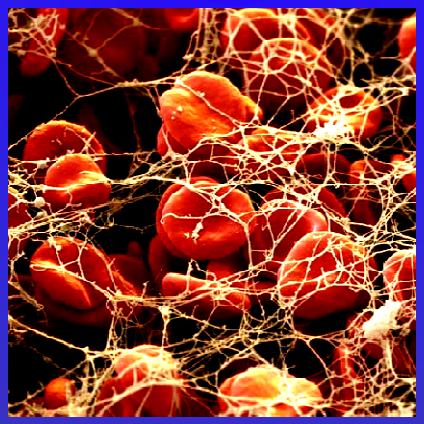
hip replacements

polymer fibers



Ceramic membranes

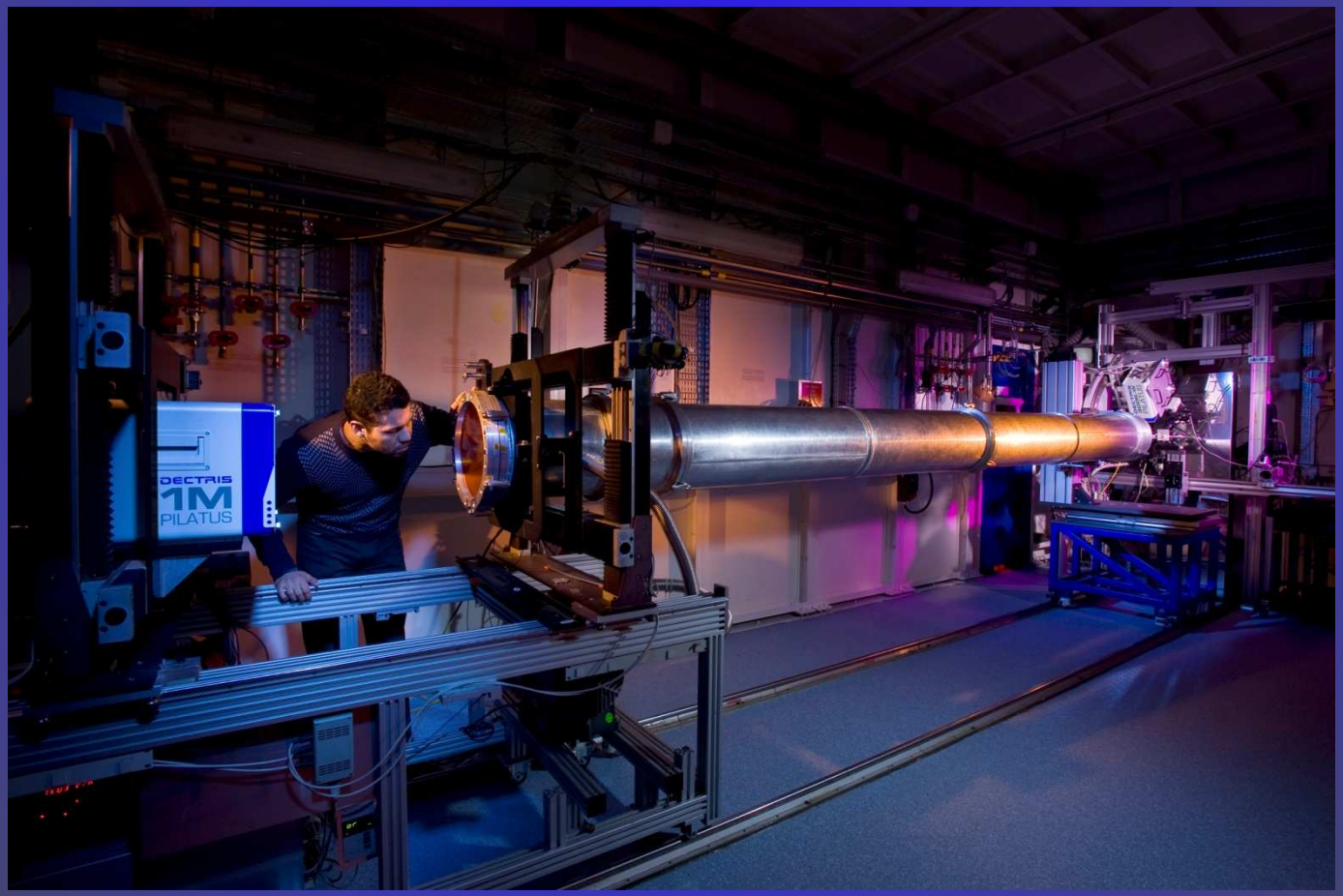
Unwashed Eskimo hair

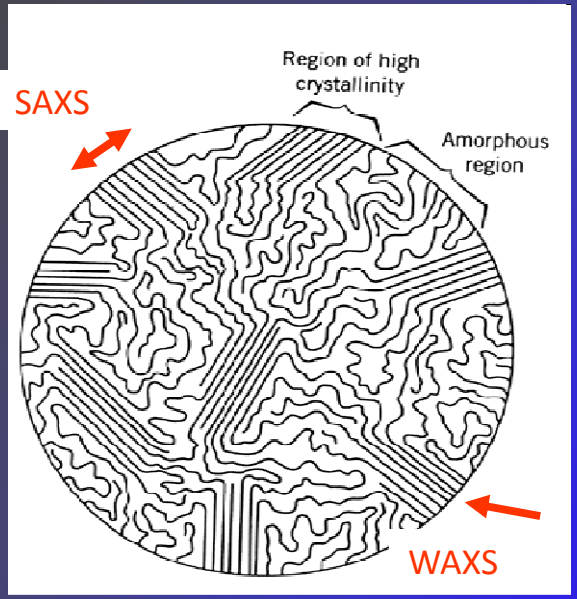


fibrinogen,

Etc., etc., etc….

Car exhaust
Soot of diesel

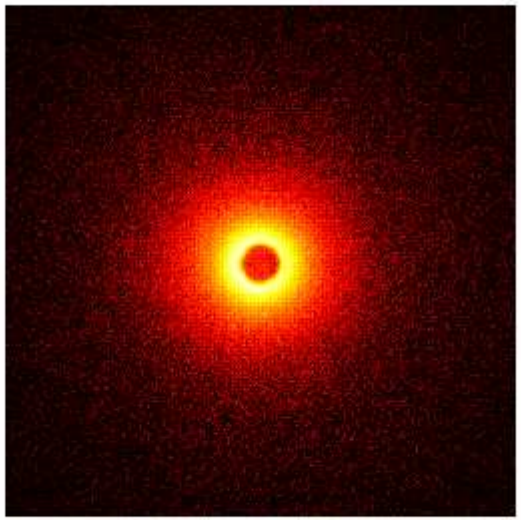

DECTRIS
1M
PILATUS

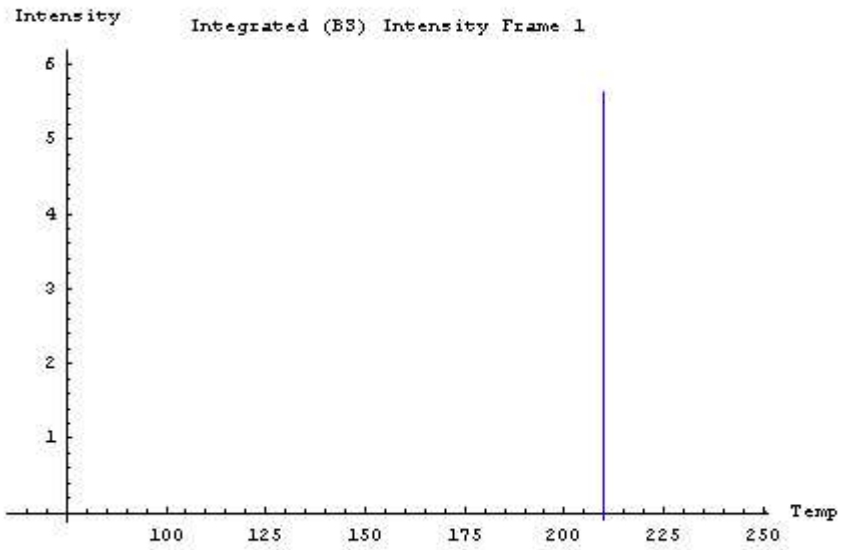



melt

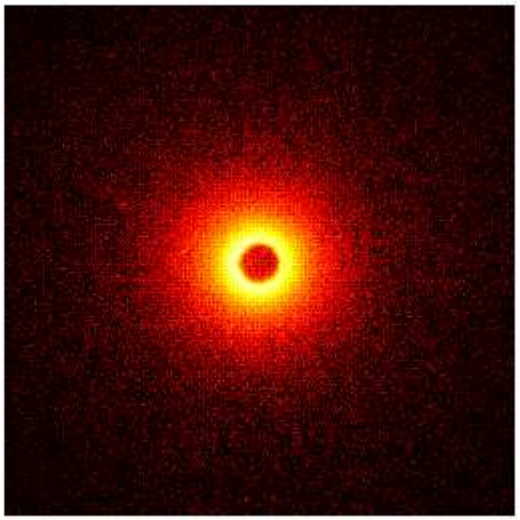
uni-directional

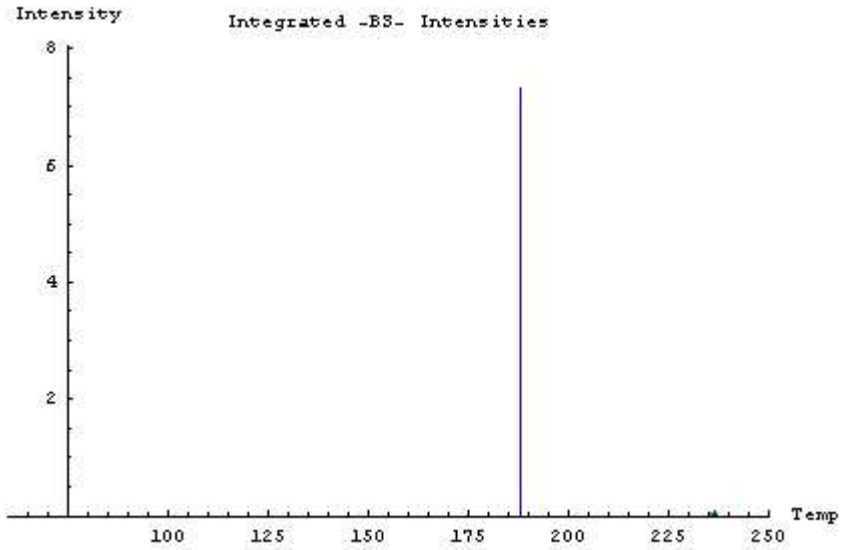
T= 251.25

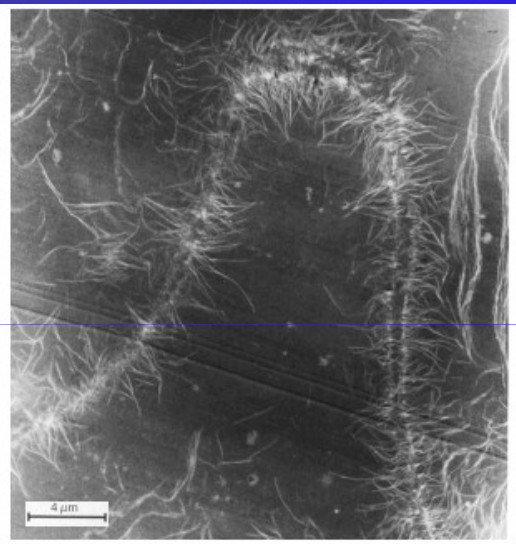


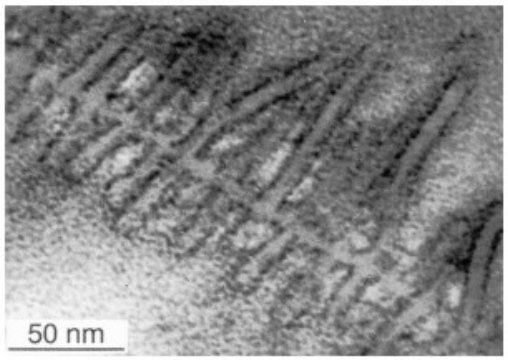


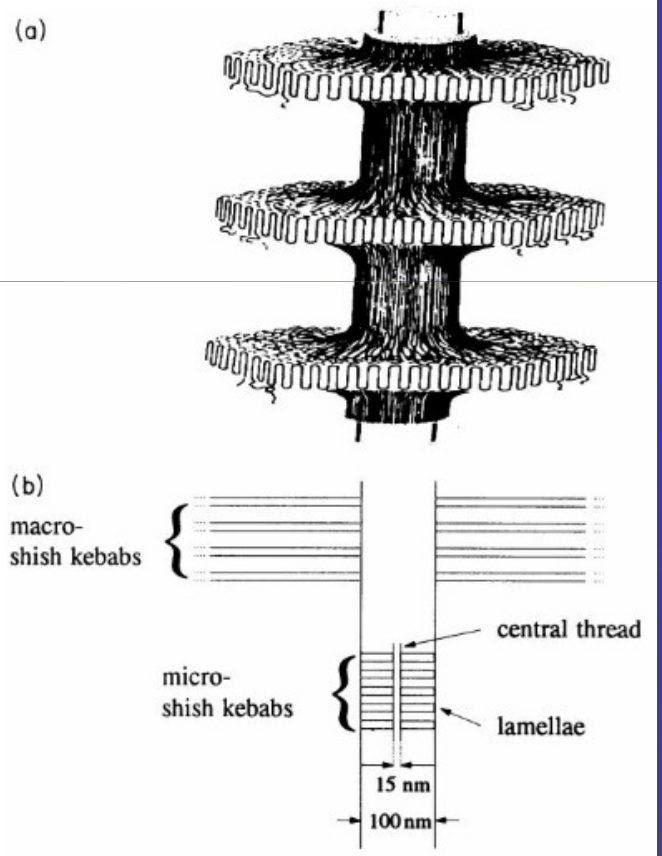

T= 236.432



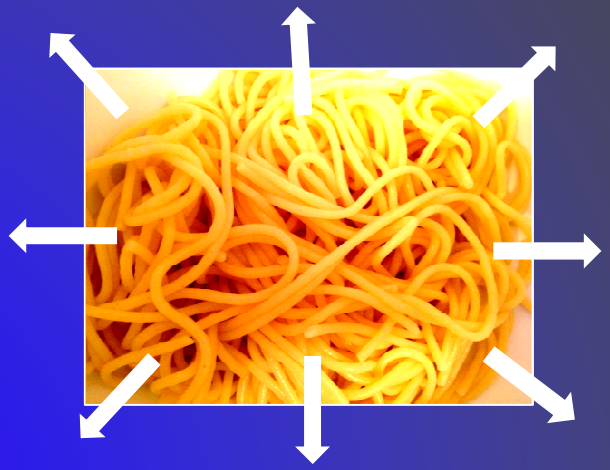


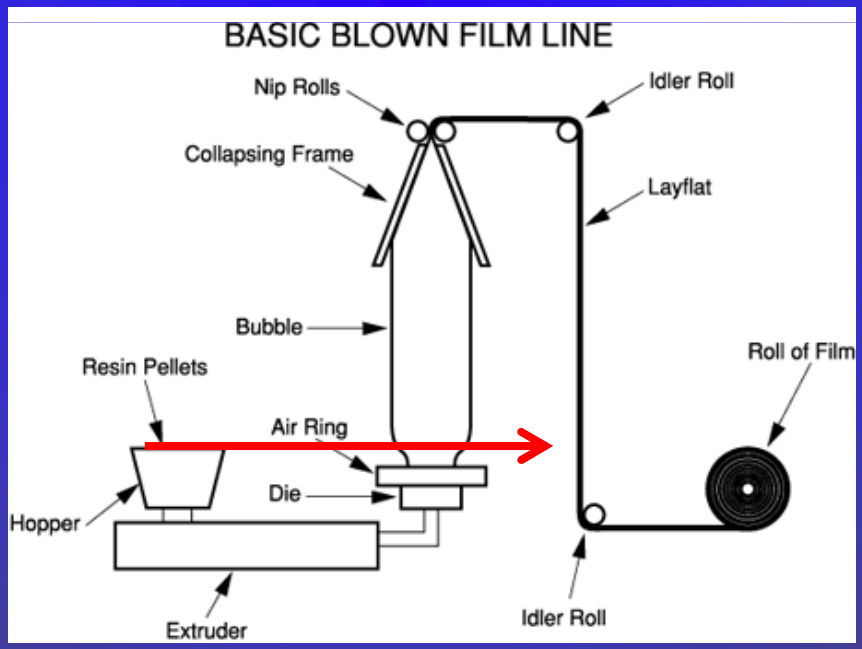

4 μm


50 nm


(a)
(b)
macro-
shish kebabs
central thread
micro-
shish kebabs
lamellae
15 nm
100 nm

melt




BASIC BLOWN FILM LINE
Nip Rolls
Idler Roll
Collapsing Frame
Layflat
Bubble
Resin Pellets
Roll of Film
Air Ring
Die
Hopper
Extruder
Idler Roll

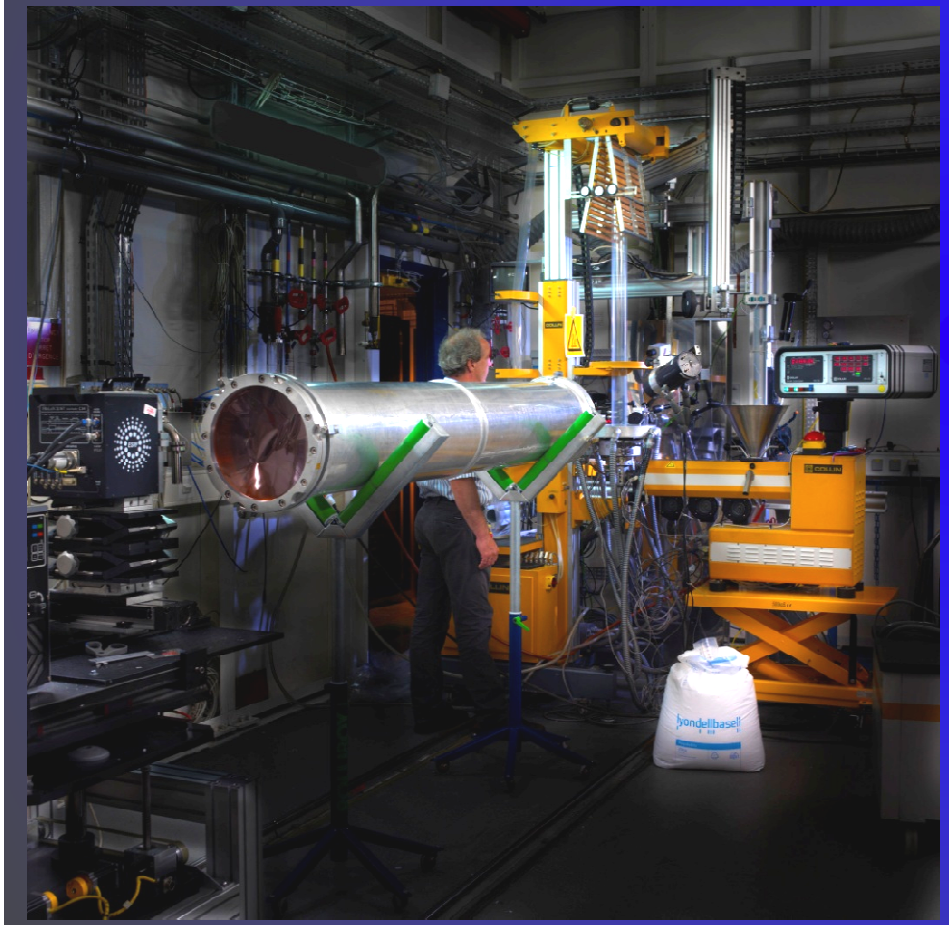
# The perfect garbage bag


TU/e Technische Universiteit Eindhoven University of Technology

Groepen:
P. Lemstra
H.E.H. Meijer

DUBBLE

# Fundamental research with industrial undertones





1974


A plastic bag


2011

This bag is 6 x lighter but just as strong
6x less oil
6x less pollution

This is not irrelevant: we produce enough of this material to cover the earth 3 times/year

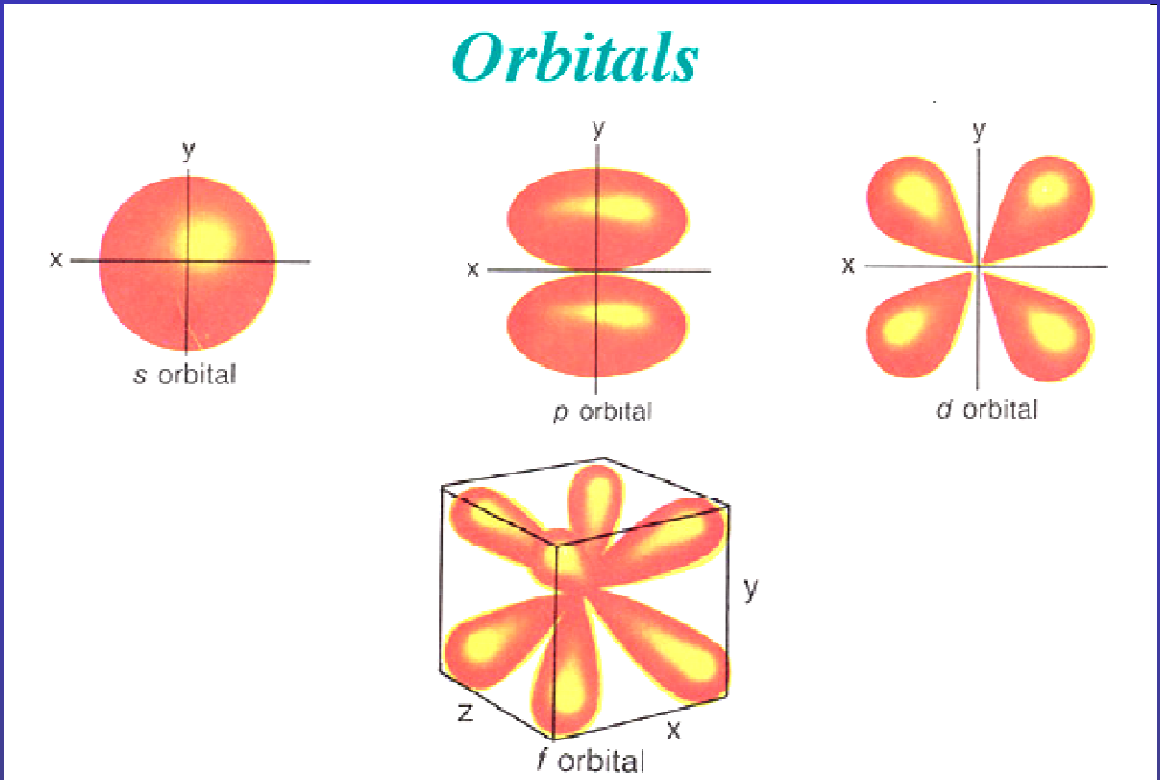
# Dutch Polymer Institute support

- To obtain funding also industry support required

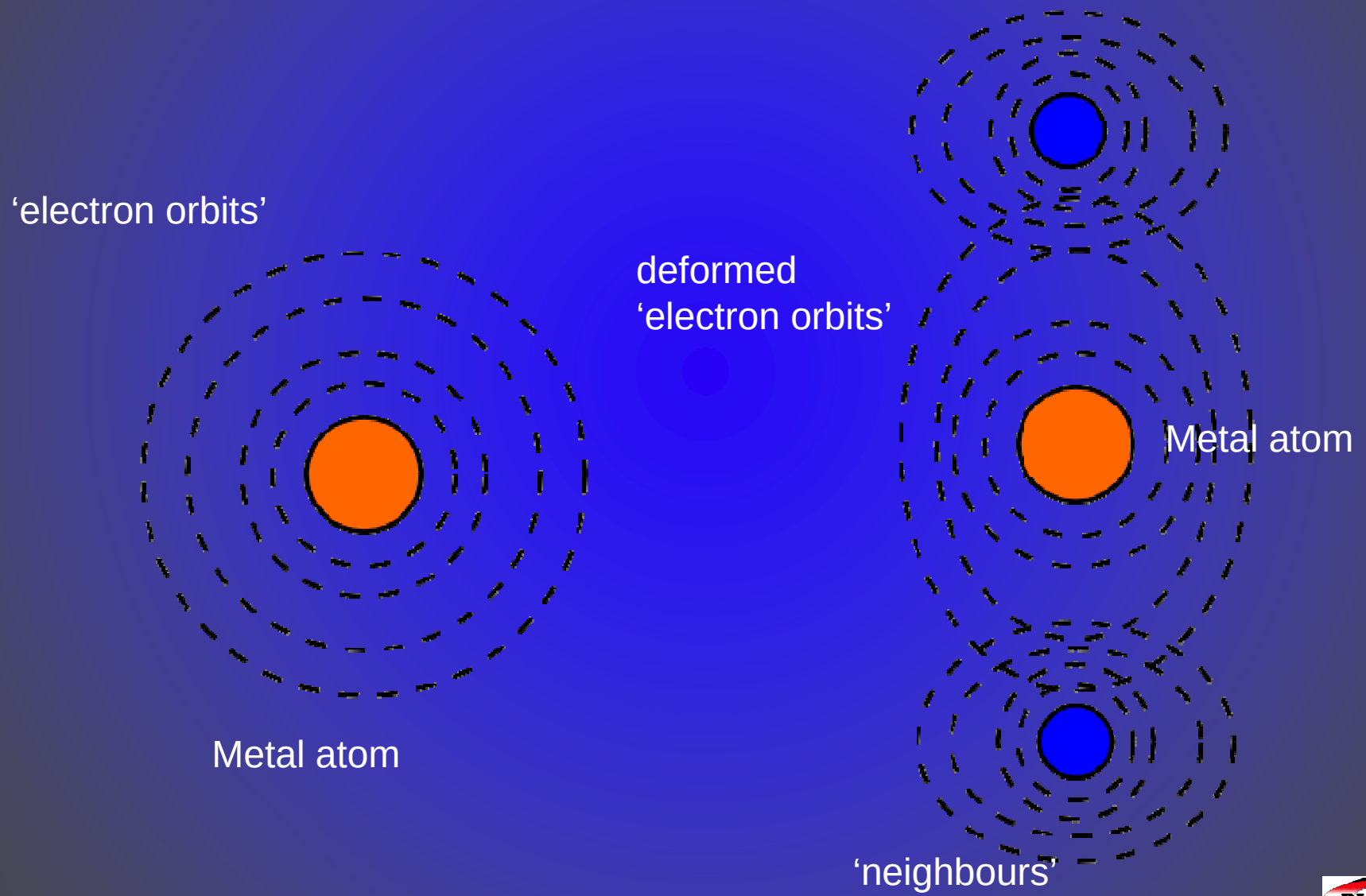


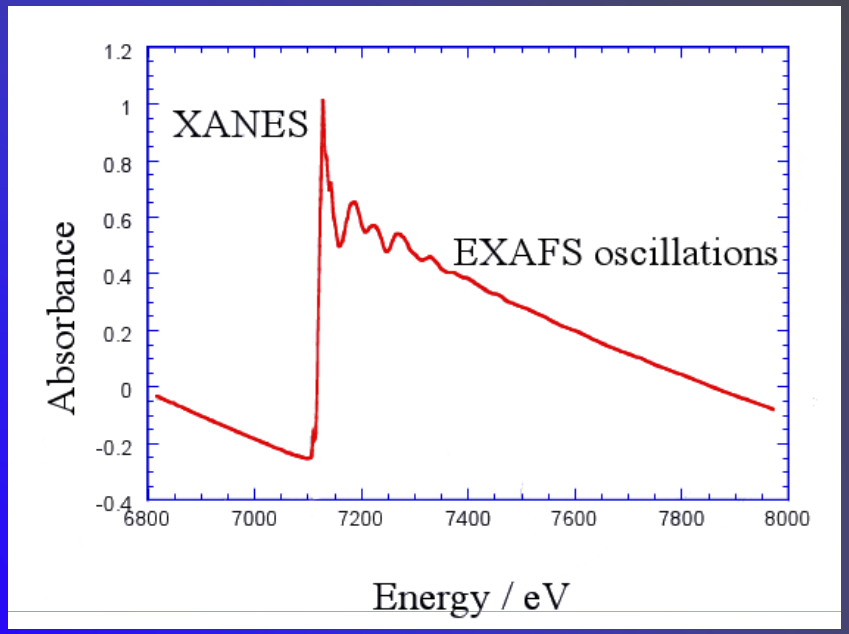
- so far 14 postdoc years, 4 PhD years funded
- some sale of beam time
- even proprietary research largely published

# X-ray spectroscopy



# X-ray spectroscopy

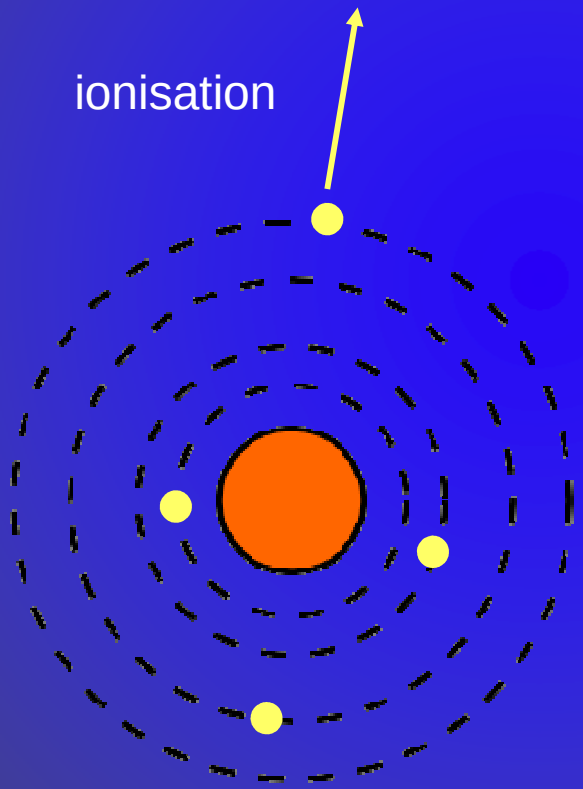





ionisation

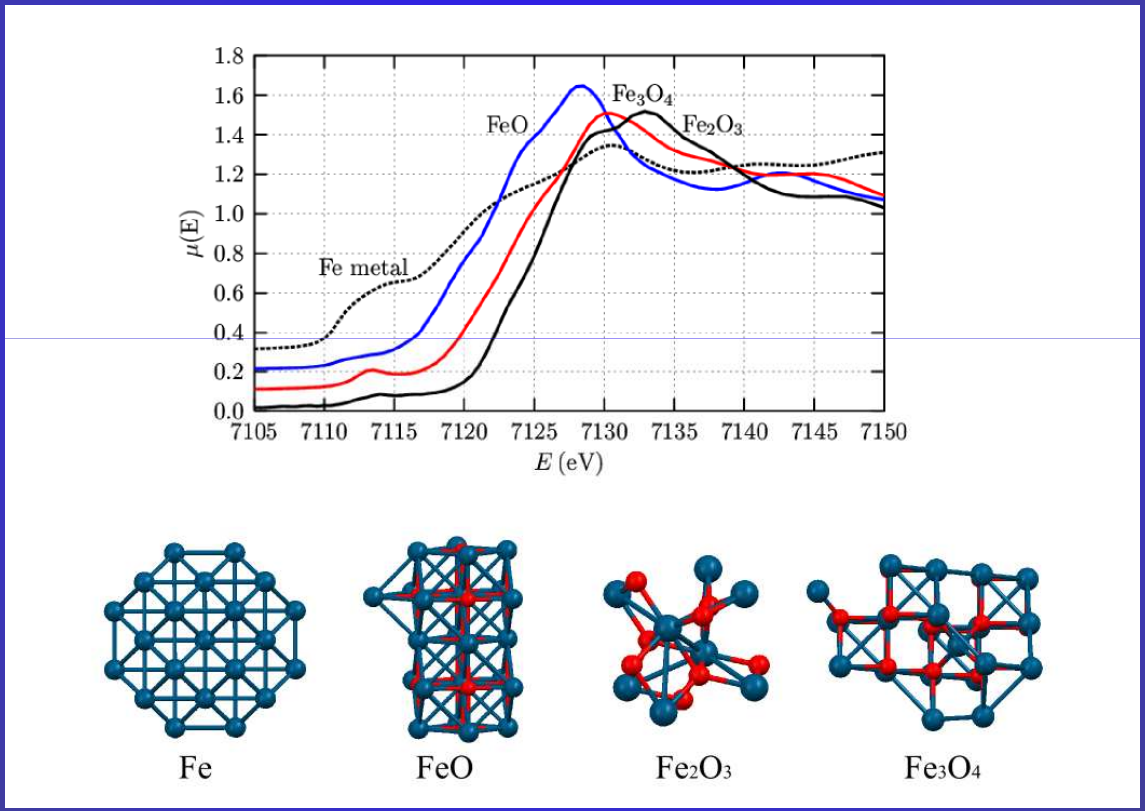
Röntgen photon

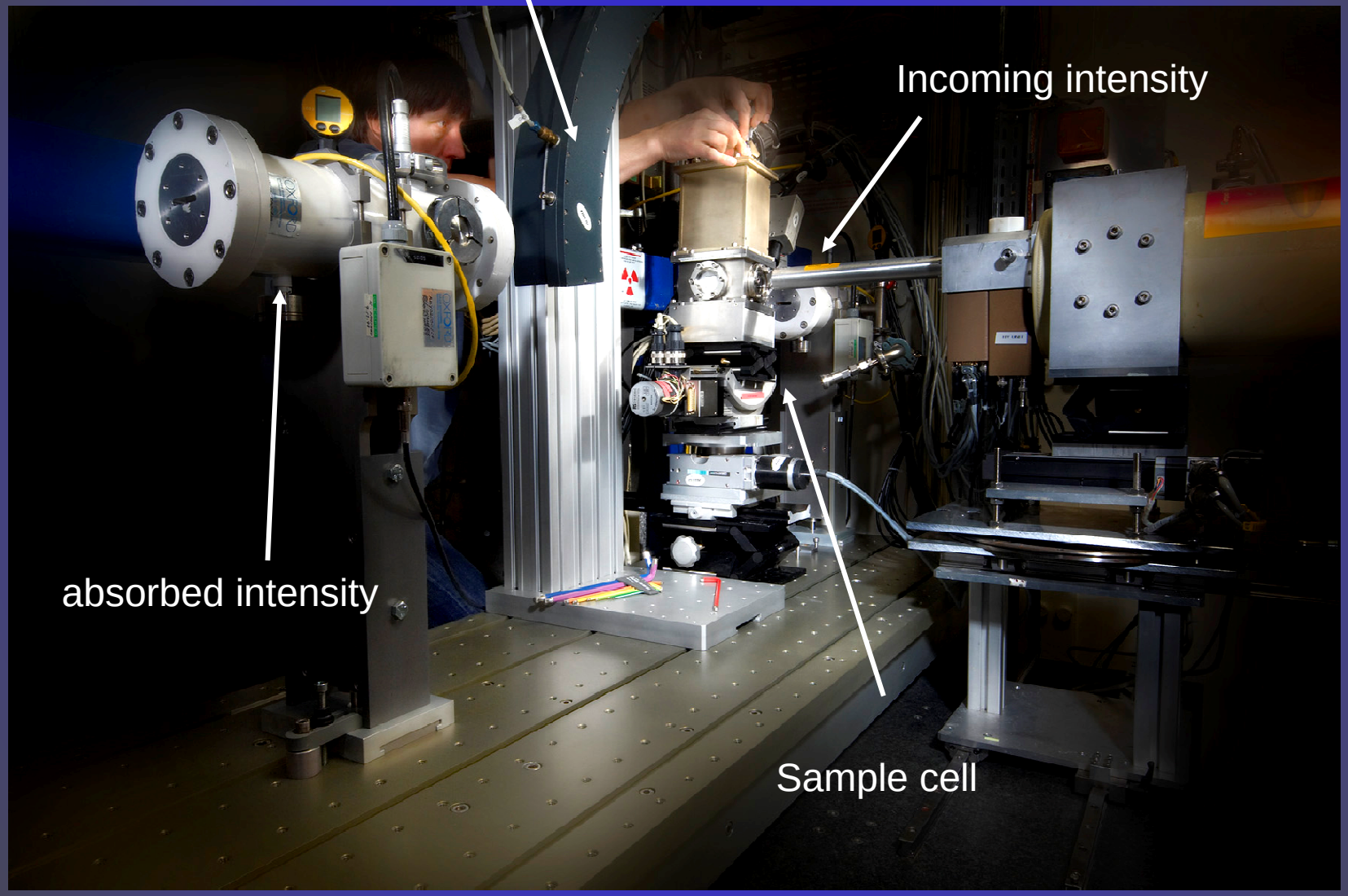
absorbed



$$\frac{I_t}{I_0}(E)$$

# Difference in surroundings of metal atom; difference in absorption spectrum




Powder diffraction
Incoming intensity
absorbed intensity
Sample cell

# Again multidisciplinary

catalysis

Hydrogen storage

liquid metals

pharmaceuticals

Environmental pollution

electro chemistry
cultural heritage

Etc., etc., etc….

# Technique combinations

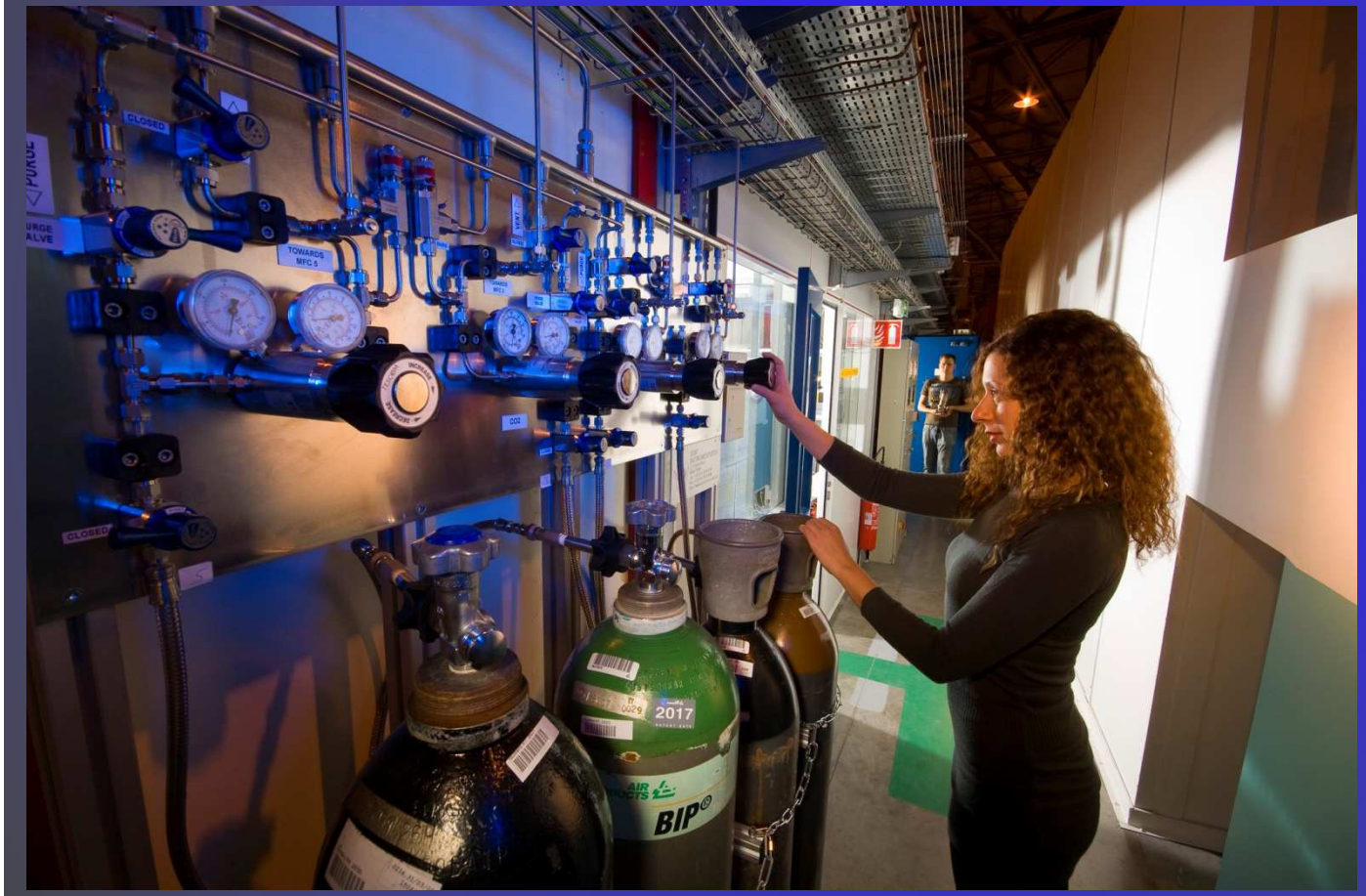

How do I make a working catalyst?

Groep Bert Weckhuijsen, Utrecht (Spinoza price 2013)

# Structure grows

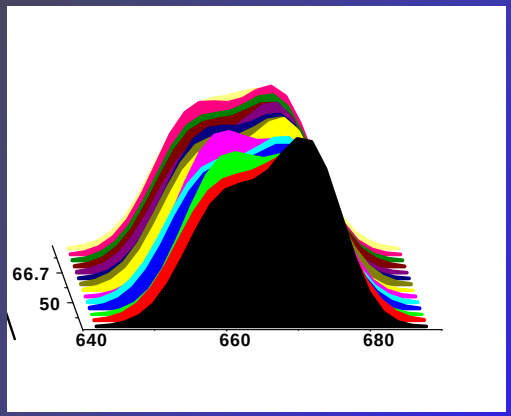
1. Early in reaction, no crystal, X-ray spectroscopy (XAFS)
2. A little later, X-ray diffraction WAXS
3. When it has grown sufficient, SAXS
4. In the end catalytic activity XAFS, (UvVis, Raman spectroscopy)

# All these steps one wants to study on-line

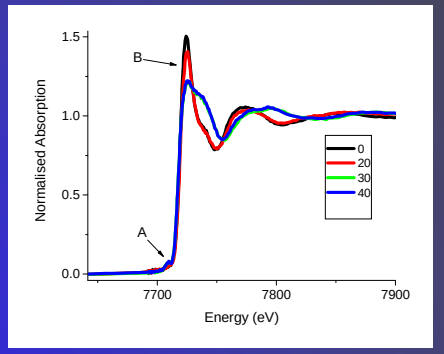



- Infrastructure
- Industrially relevant conditions
- High gas pressure
- Flow
- Operando conditions

# In-situ multi-technique approach of zeolite synthesis

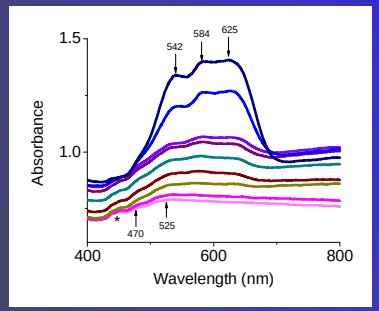



Raman

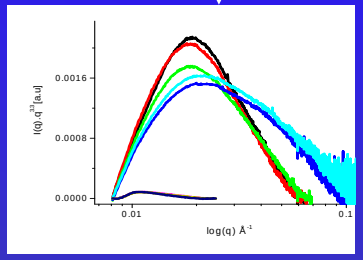



XAFS

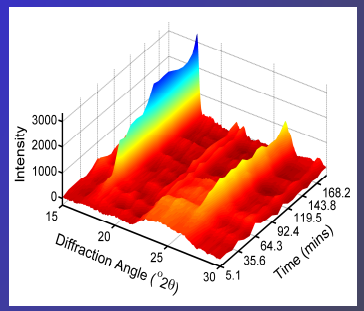
UV-Vis




SAXS



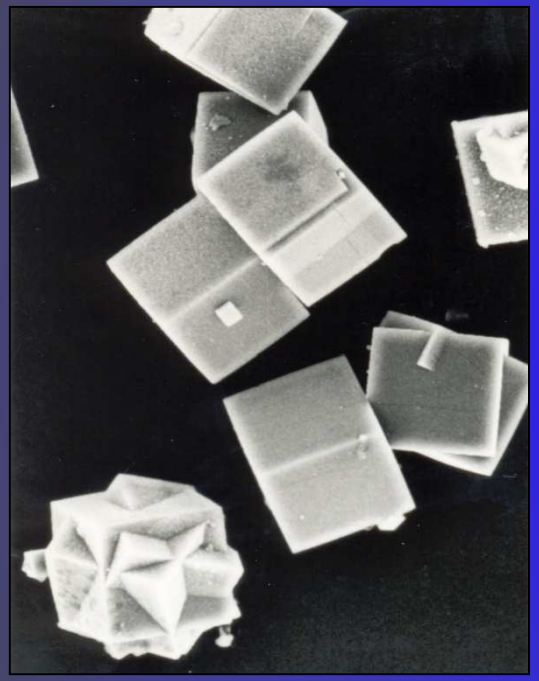

WAXS



# Unique instrumentation

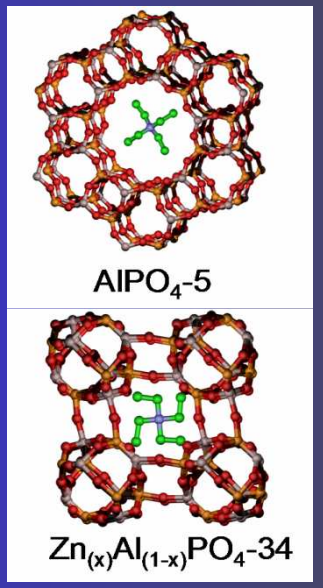
- Sample at high pressure/temperature
- Simultaneous 5 techniques possible
- All aspects of the sample and kinetics can be studied simultaneously

# We can:



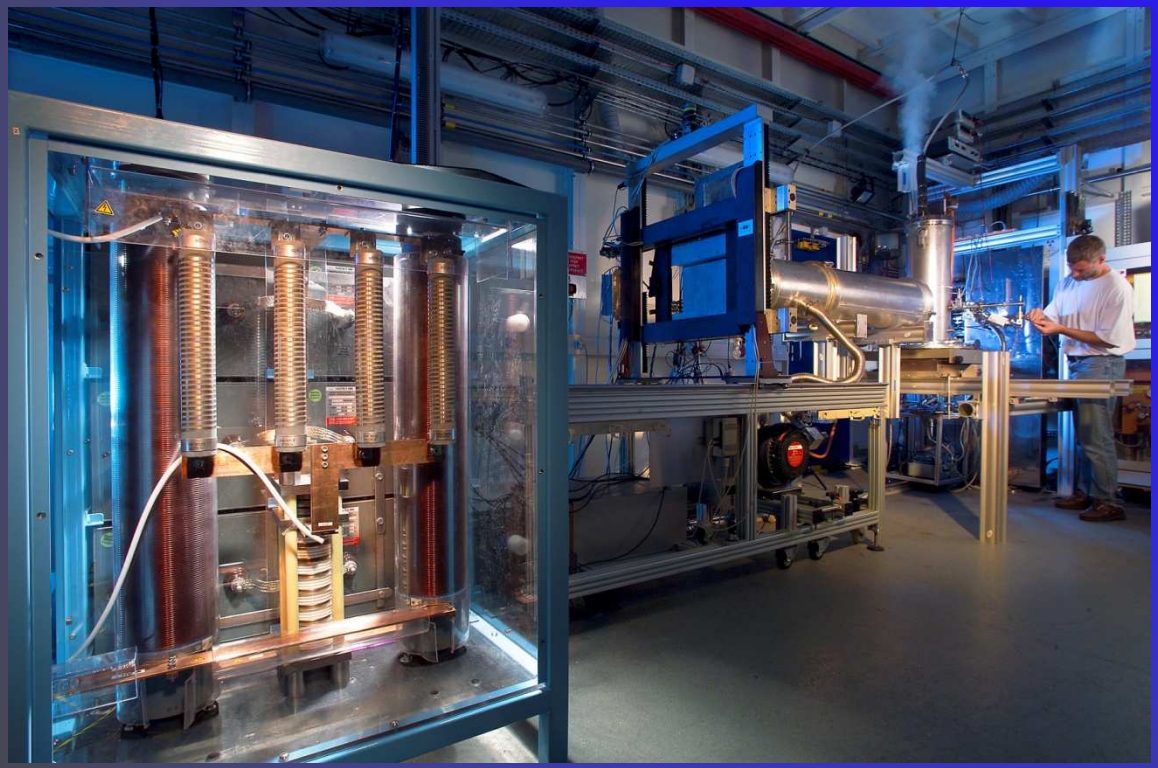
Catalyst crystals

- Follow particle growth
- Study the catalytic actions
- Follow the life cycle of the catalyst

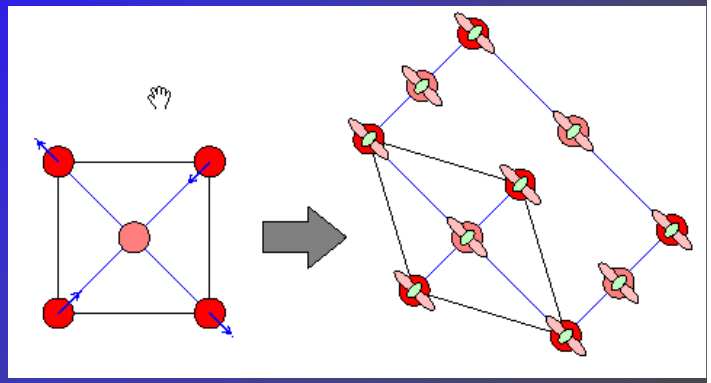



All in a single experiment !

# Also very fundamental work



Distortions of crystalline lattices in 30 Tesla pulsed fields

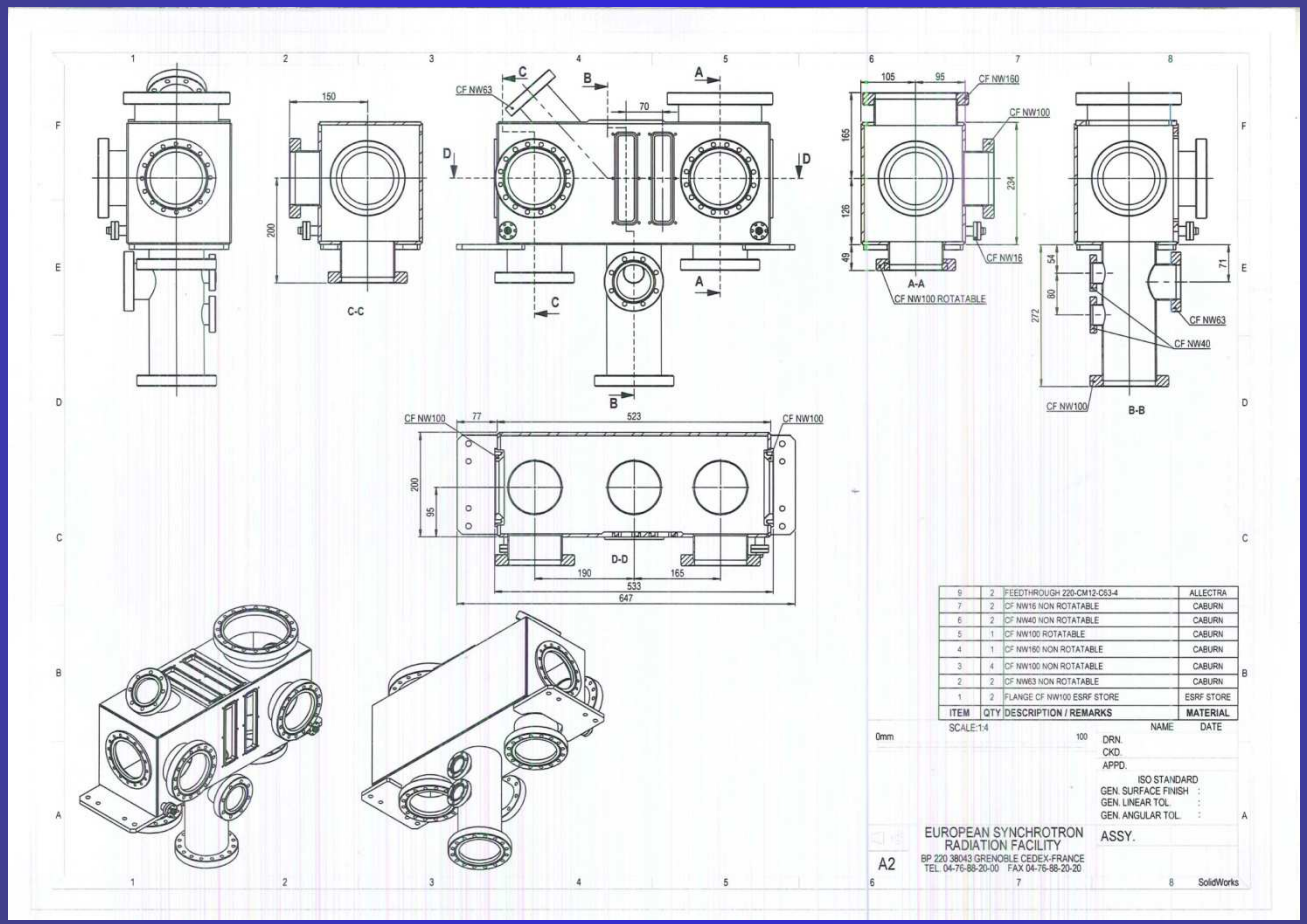


# Dus je wilt de ESRF als klant?

Drie belangrijke zaken

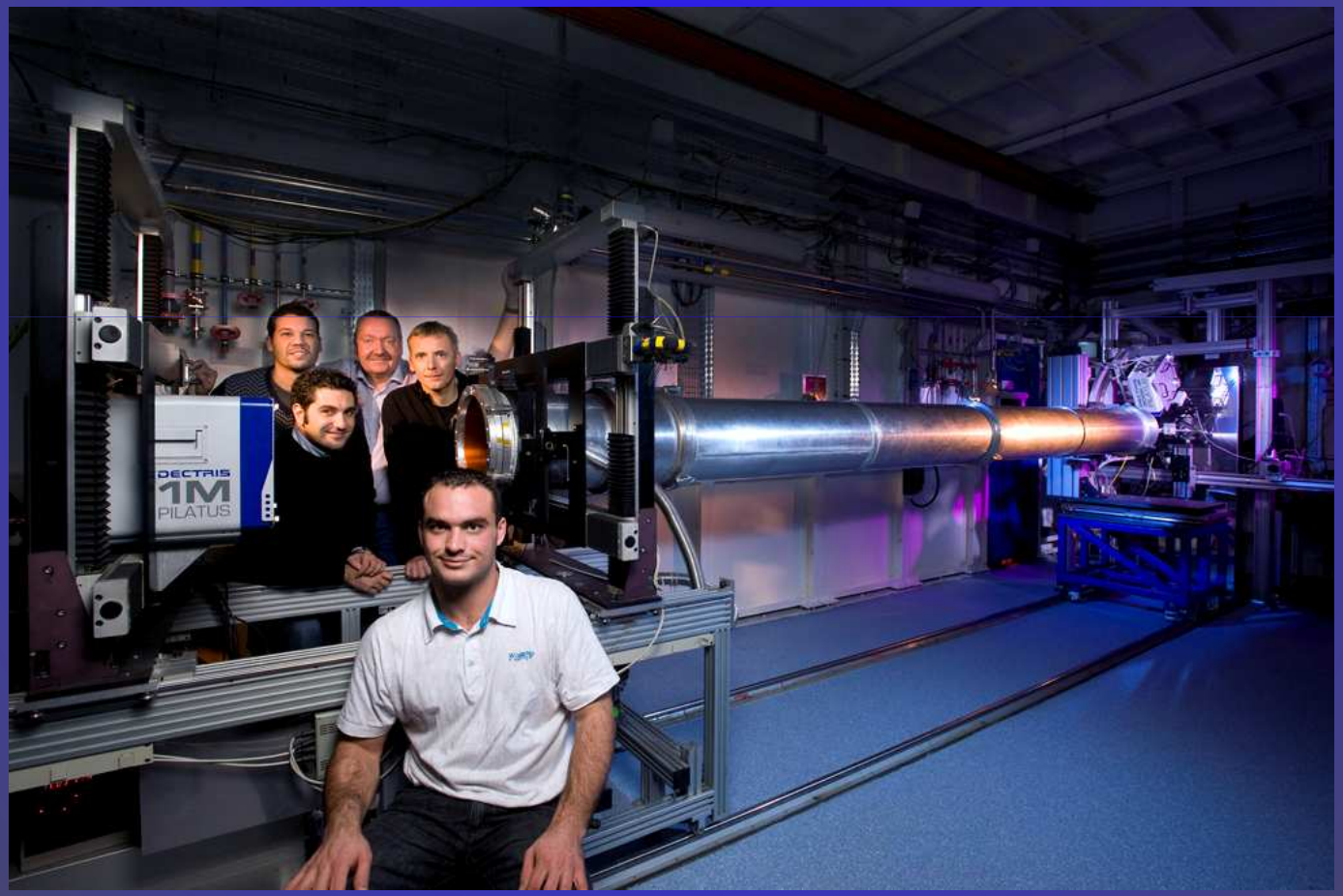
Frans, Frans en Frans

# Dubble: vacuum chamber




Drawings exist: ready for tender

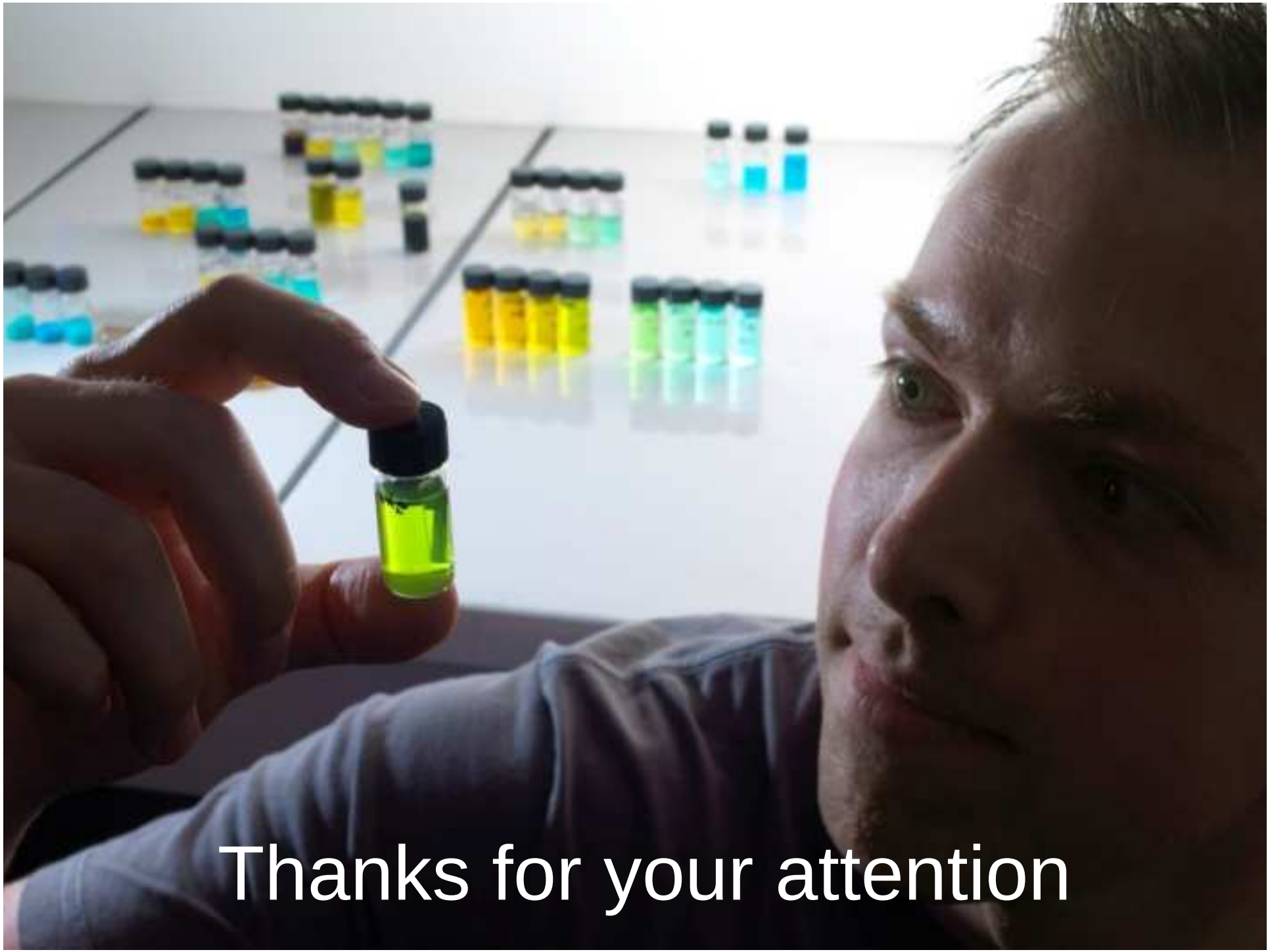
# Bellows: large diameter; edge welded; support structure needed




Please remember:

-I don't work for the ESRF
- I work for NWO and don't have much influence on the ESRF buying process

- Sometimes we can help a bit…


Thanks for your attention