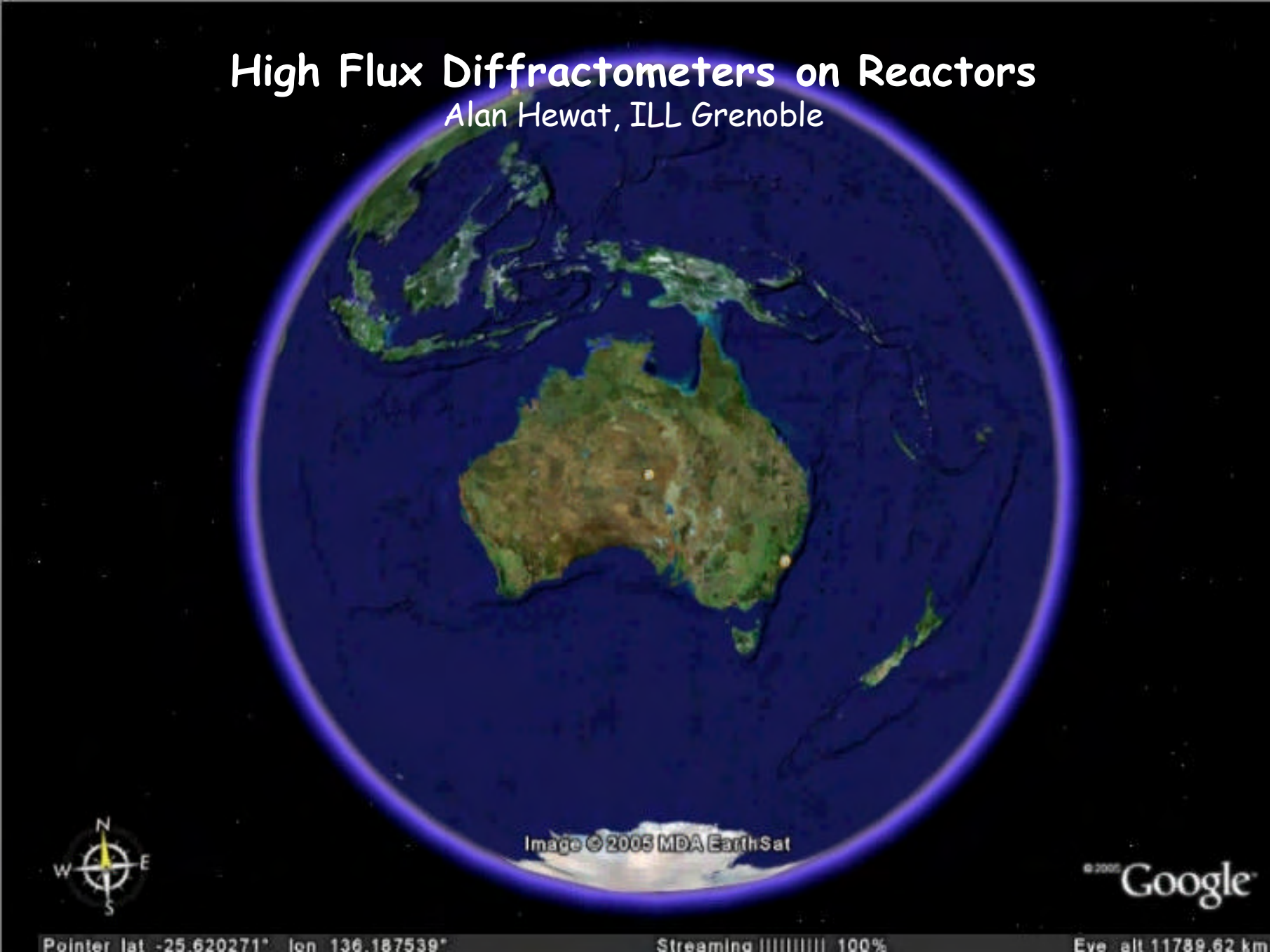
High Flux Diffractometers on Reactors

Alan Hewat, ILL Grenoble



High Flux Diffractometers on Reactors

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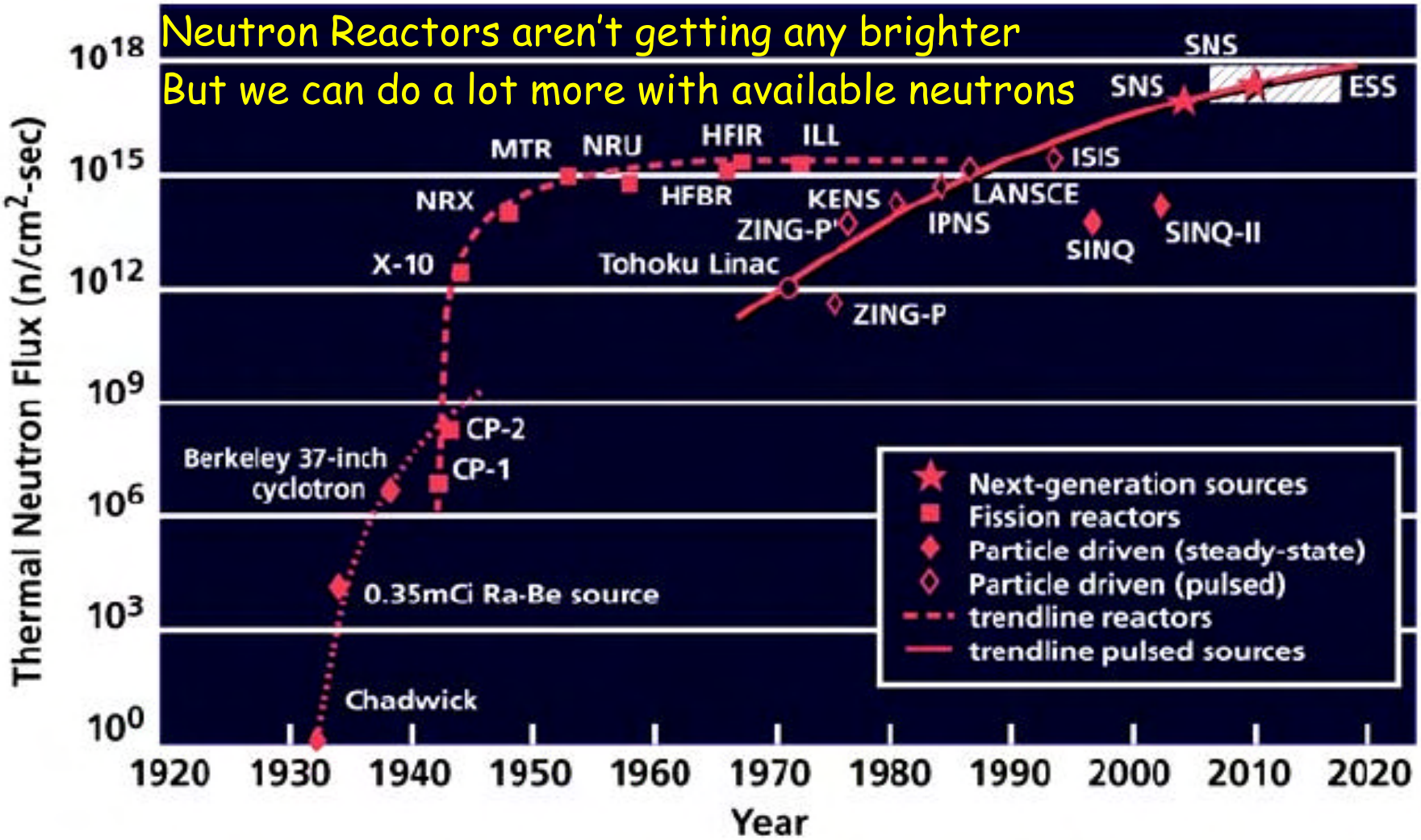
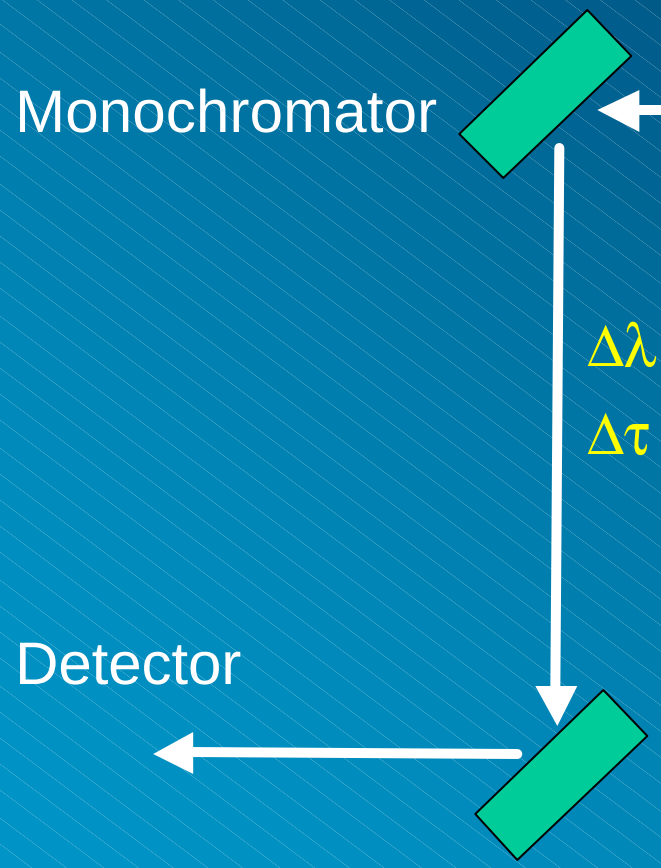


J.M. Carpenter, D.L. Price, R.A. Robinson and J. M. Rowe 2005 CCLRC Technology Roadmap

Classical Neutron Diffraction from a single crystal

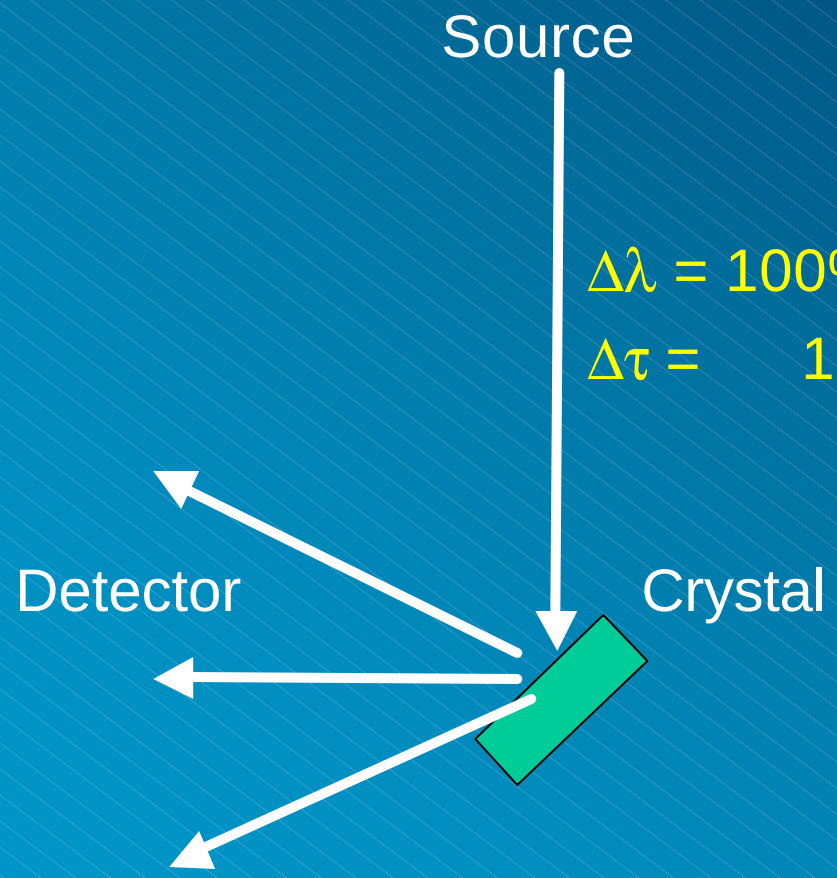


$\Delta\lambda$ = 1% (monochromatic beam)

$\Delta\tau$ = 100% (continuous source)

TOF Neutron Diffraction from a single crystal

Multiple reflections sorted by Time-Of-Flight

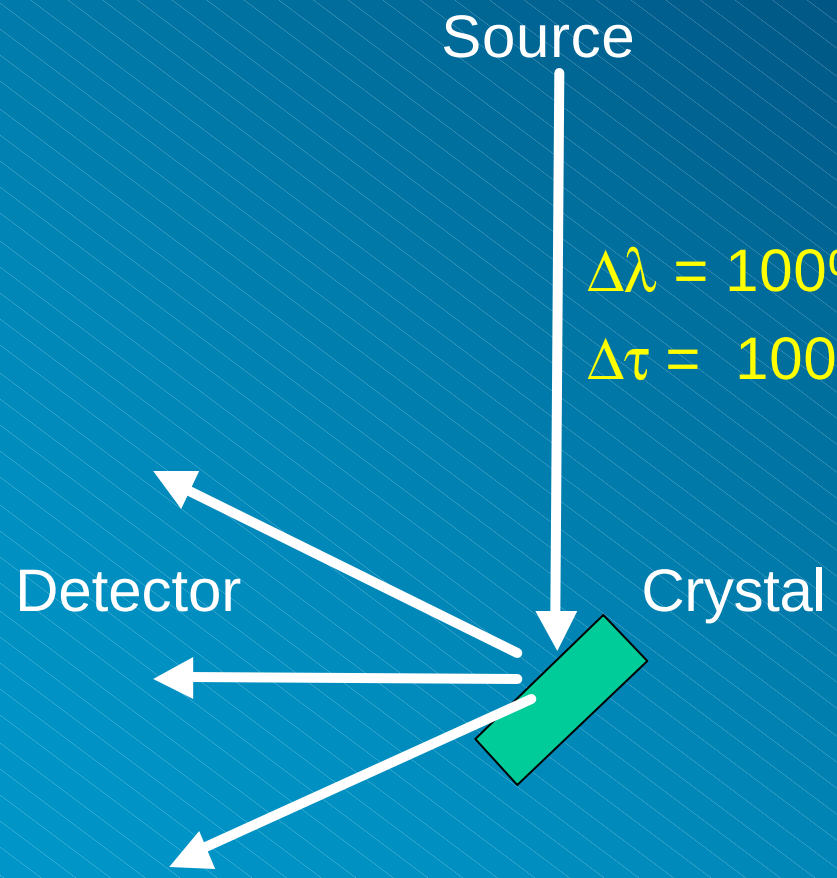


$\Delta\lambda$ = 100% (white beam)

$\Delta\tau$ = 1% (pulsed source)

White Beam Neutron Diffraction from a single crystal

Multiple reflections sorted by the Crystal itself

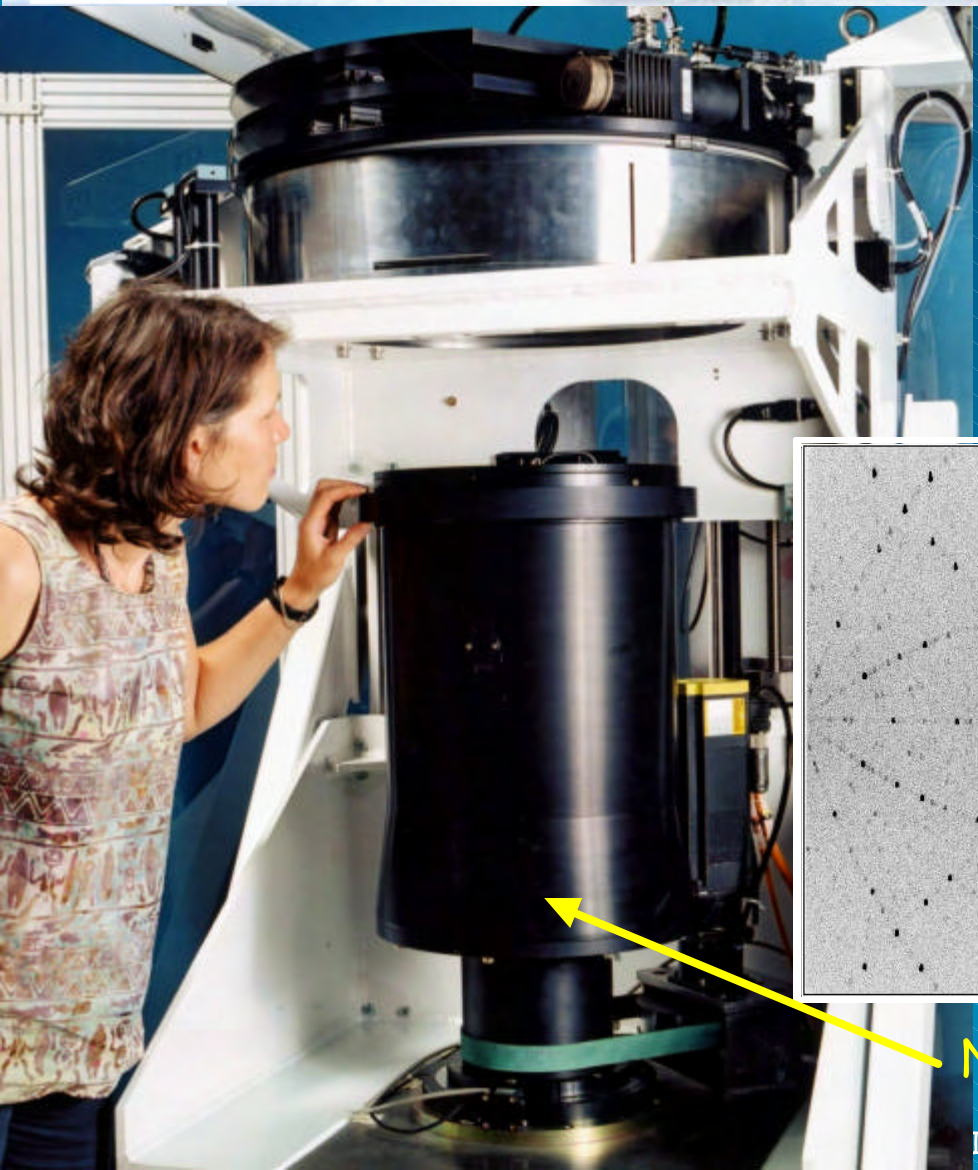


$\Delta\lambda$ = 100% (white beam)

$\Delta\tau$ = 100% (continuous source)

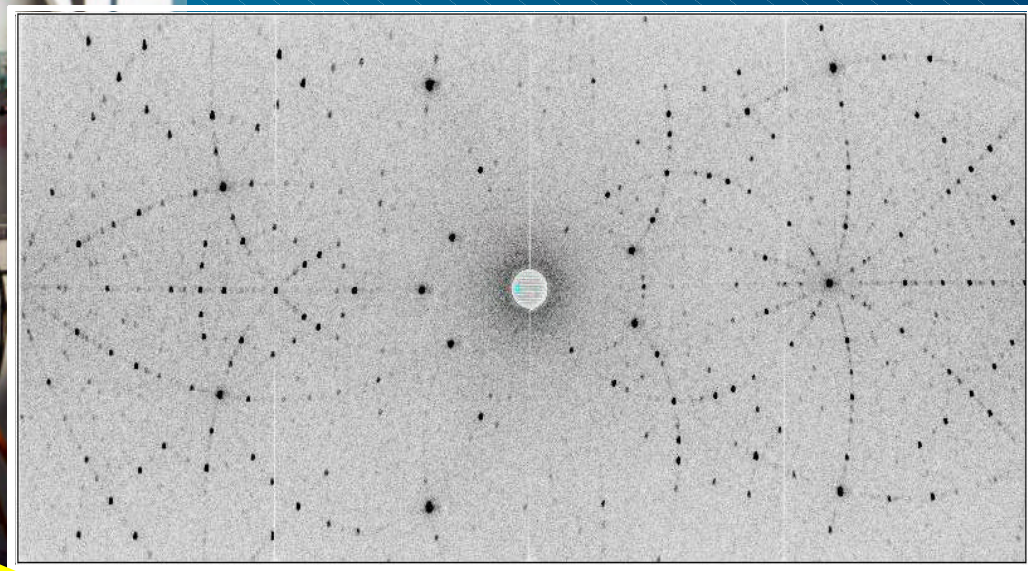
White Beam Neutron Diffraction-Vivaldi

G.McIntyre , M-H.Lemée-Cailleau , C.Wilkinson (ICNS-2005)



Multiple reflections
sorted by the Crystal itself

"Niimura Special"
Neutron Image Plates (NIP)



NIPs inside cylinder, Laser readout

ANSTO-KOALA Image Plate Detector

Wim Klooster with G. McIntyre & Maatel Grenoble

ANSTO-KOALA Image Plate Detector

Wim Klooster with G. McIntyre & Maatel Grenoble

KOALA – Australian VIVALDI

Flux from thermal neutron guide

High res. (l= 1.03Å) 1.29×10^{9} $n.cm^{-2}s^{-1}$

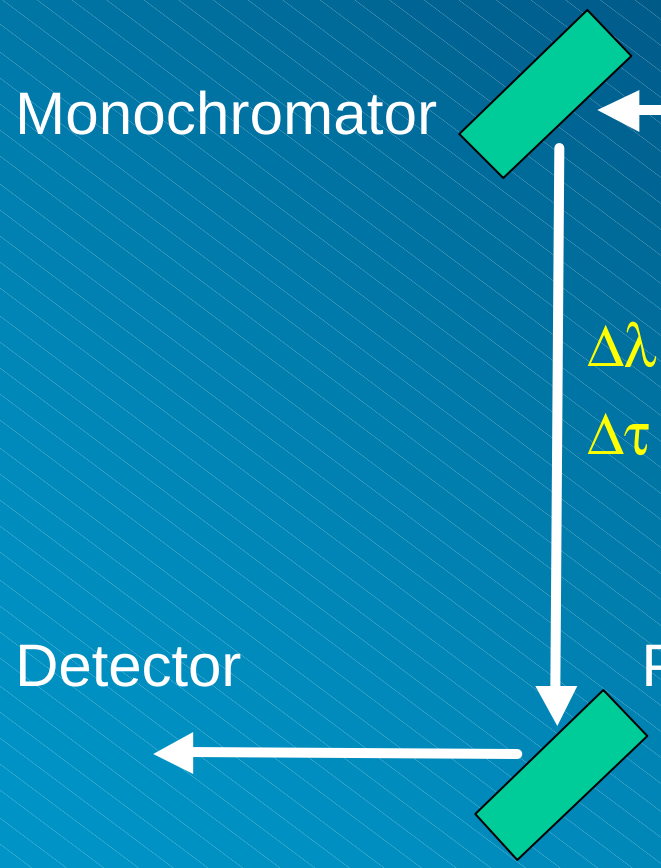
Flux comparable to that on Vivaldi

KOALA also uses Niimura Special IPs

Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focussing)
- A wide band of wavelengths
- A very high flux on the sample
- A very large area detector

What about Classical Neutron CW Diffractometers



$\Delta\lambda$ = 1% (monochromatic beam)

$\Delta\tau$ = 100% (continuous source)

Comparison of TOF & CW Diffractometers

Large detectors + high flux on the sample

Efficiency for a given resolution = * time averaged flux on the sample
* sample volume
* detector solid angle
* detector efficiency

Jorgensen, J.D., Cox, D.E., Hewat, A.W., Yelon, W.B.

"Scientific opportunities with advanced facilities for neutron scattering"
Shelter Island Workshop, 1984
Nuclear Instruments and Methods in Physics Research B12 (1985) 525-561

Comparison of TOF & CW Diffractometers

The time-averaged **Flux*Detector** criterium

	D20	GEM
Flux average on sample	5×10^7	$\sim 2 \times 10^6$
Detector solid angle	0.27 sr	4.0 sr
Efficiency=Flux*Detector	1.7	1

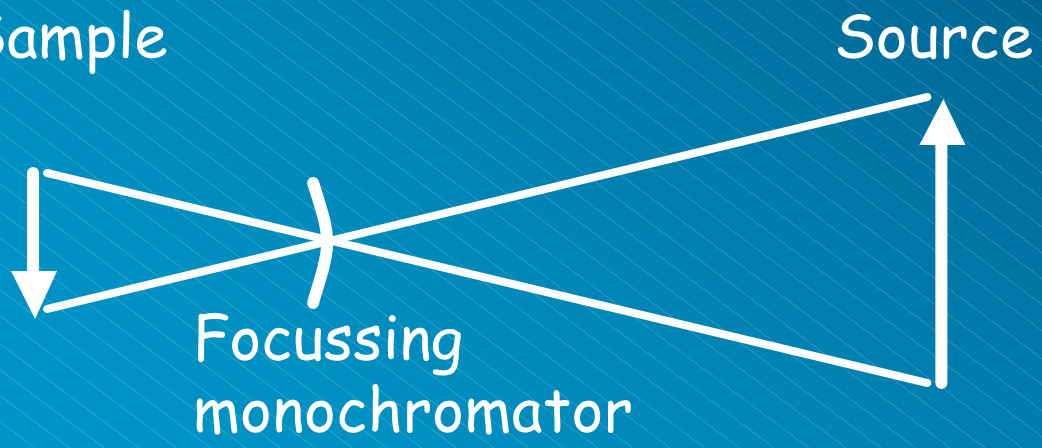
The CW and TOF machines have similar efficiencies

The time-averaged flux is very high on a CW machine (D20)
The detector is very large on a TOF machine (GEM)

Why is sample flux so high from a reactor?

A: Large vertically focusing monochromators ?

No ! Focusing in real space only
gives a factor of x2 to x4

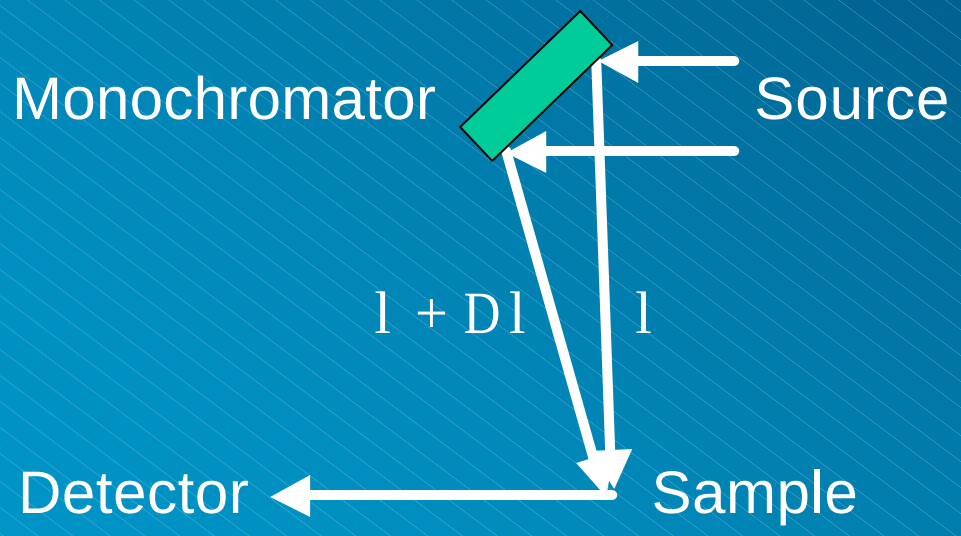


cf use of convergent guide with TOF

Why is sample flux so high from a reactor?

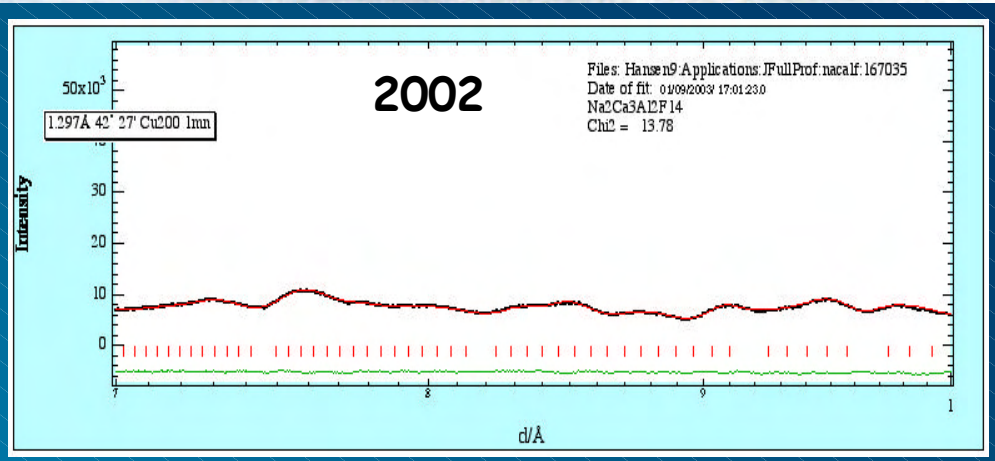
A: Large wavelength-band focusing monochromators ?

Yes ! Focusing in reciprocal space can give a factor of x10

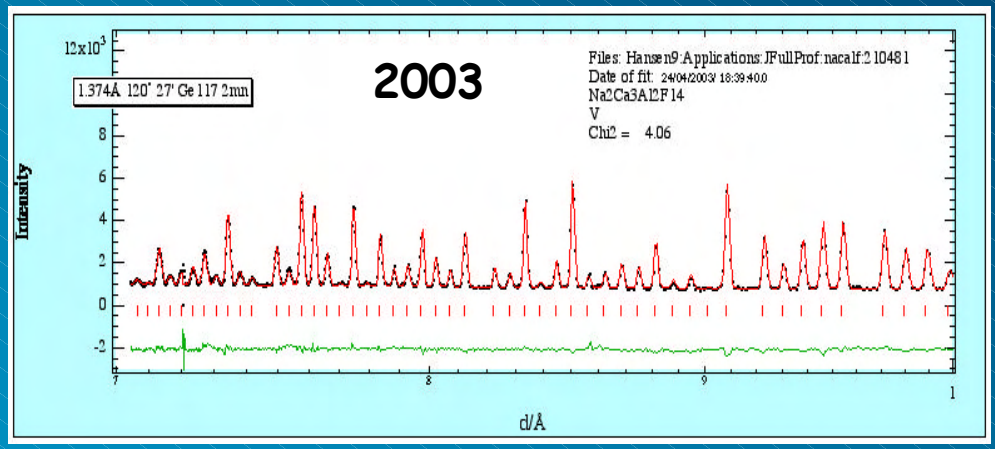


$\Delta d/d \sim 0.1\%$ for $\Delta\lambda/\lambda \sim 1\%$

High flux compatible with good Resolution
High take-off option on D20



Before and After (data in 2 min.)



Higher D20 resolution since 2003

Applications - fast detectors, small samples

Eric Kisi (ICNS Sydney)

Very fast chemical and electrochemical kinetics

(Ti,Si,C) reaction -> Ti_3SiC_2

University of Newcastle, Australia

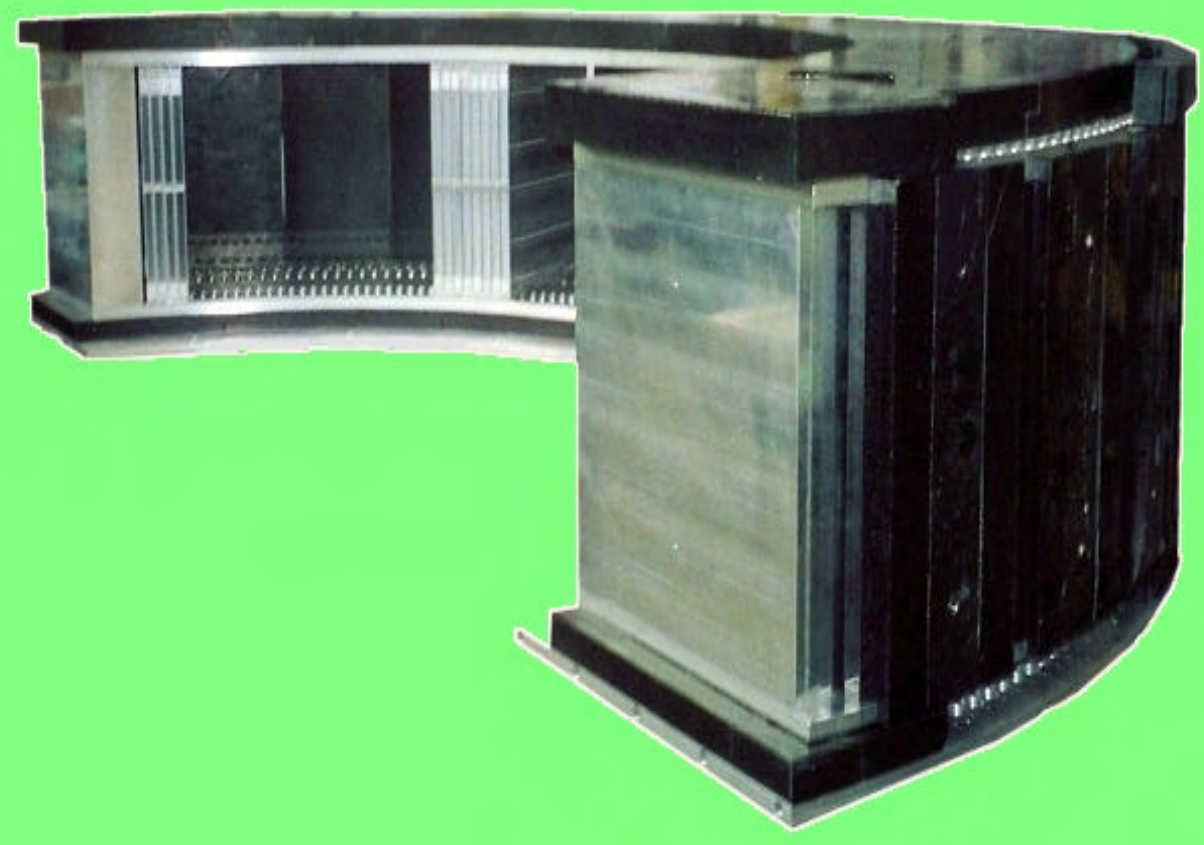
- The explosive SHS reaction was studied in real time with neutrons
- The reaction is exothermic, & heats the sample to 2200°C in <1 sec
- The complete diffraction pattern (left) is collected at 300 ms intervals - **A World Record**

D.Riley, E.Kisi, T.Hansen, A.Hewat (2002)

2D detectors for CW Powder Diffraction

E. Suard, C. Ritter, A. Hewat, P. Attfield et al.

Super-D2B – a new High Resolution Powder Diffractometer

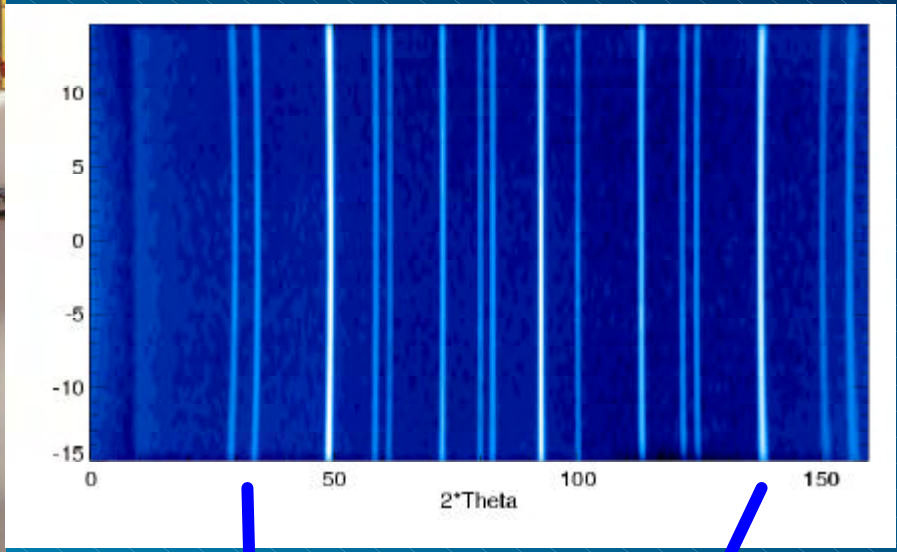


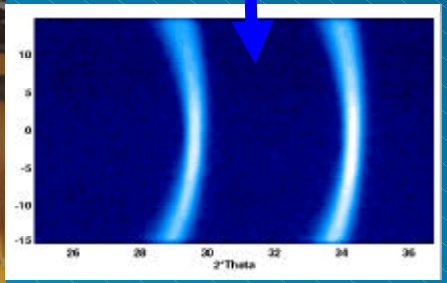
128 x 300 mm high resistive wire detectors, high resolution collimators

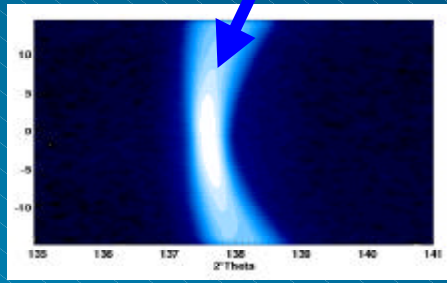
2D detectors for CW Powder Diffraction

E. Suard, C. Ritter, A. Hewat, P. Attfield et al.

UK-EPSRC Super-D2B project at ILL





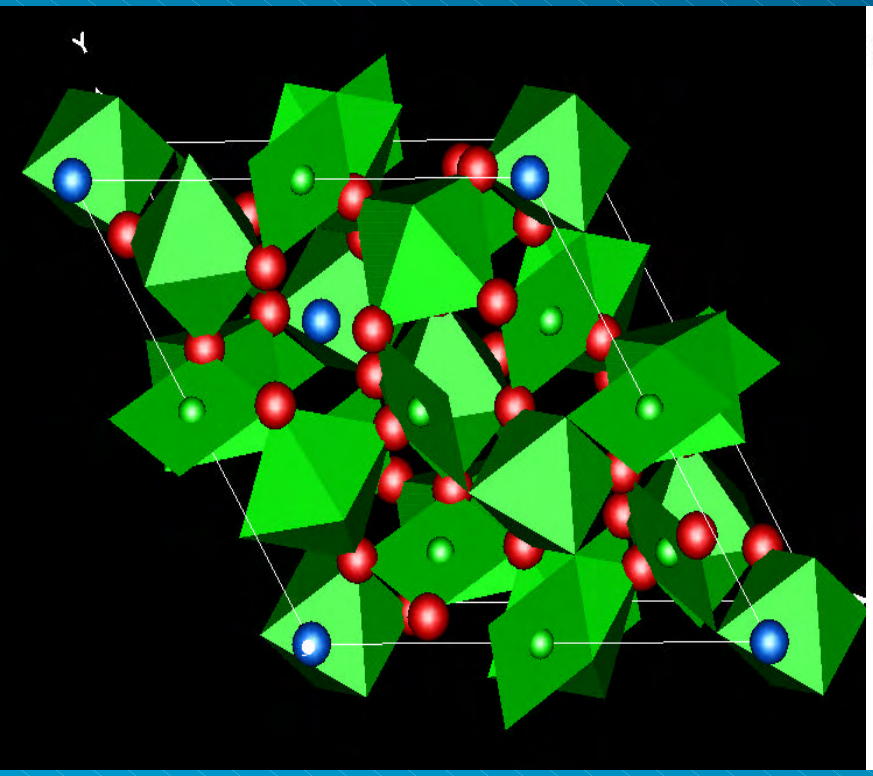


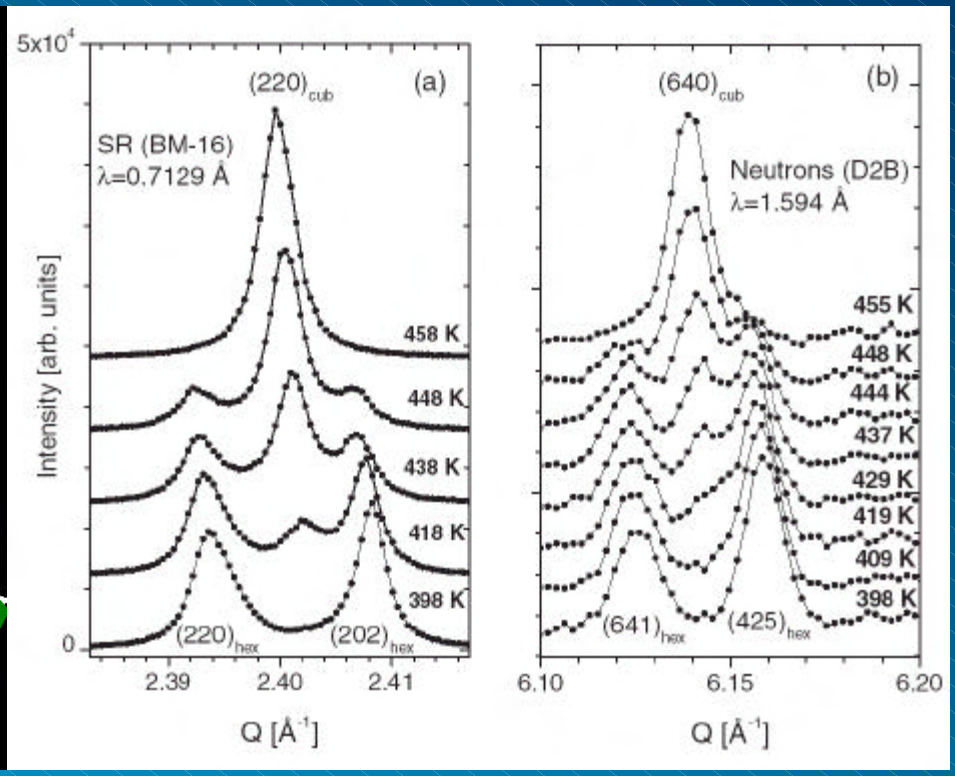
D2B, Charge Transfer & Magnetic Order

R Przenioslo, I Sosnowska, E Suard, A Hewat & A N Fitch

Phase co-existence in the charge ordering transition in $CaMn_7O_{12}$

R Przenioslo, I Sosnowska, E Suard, A Hewat & A N Fitch (2002)
J. Phys. Condens. Matter **14**, 5747-5753





Neutrons measure small changes in M-O distances – charge transfer

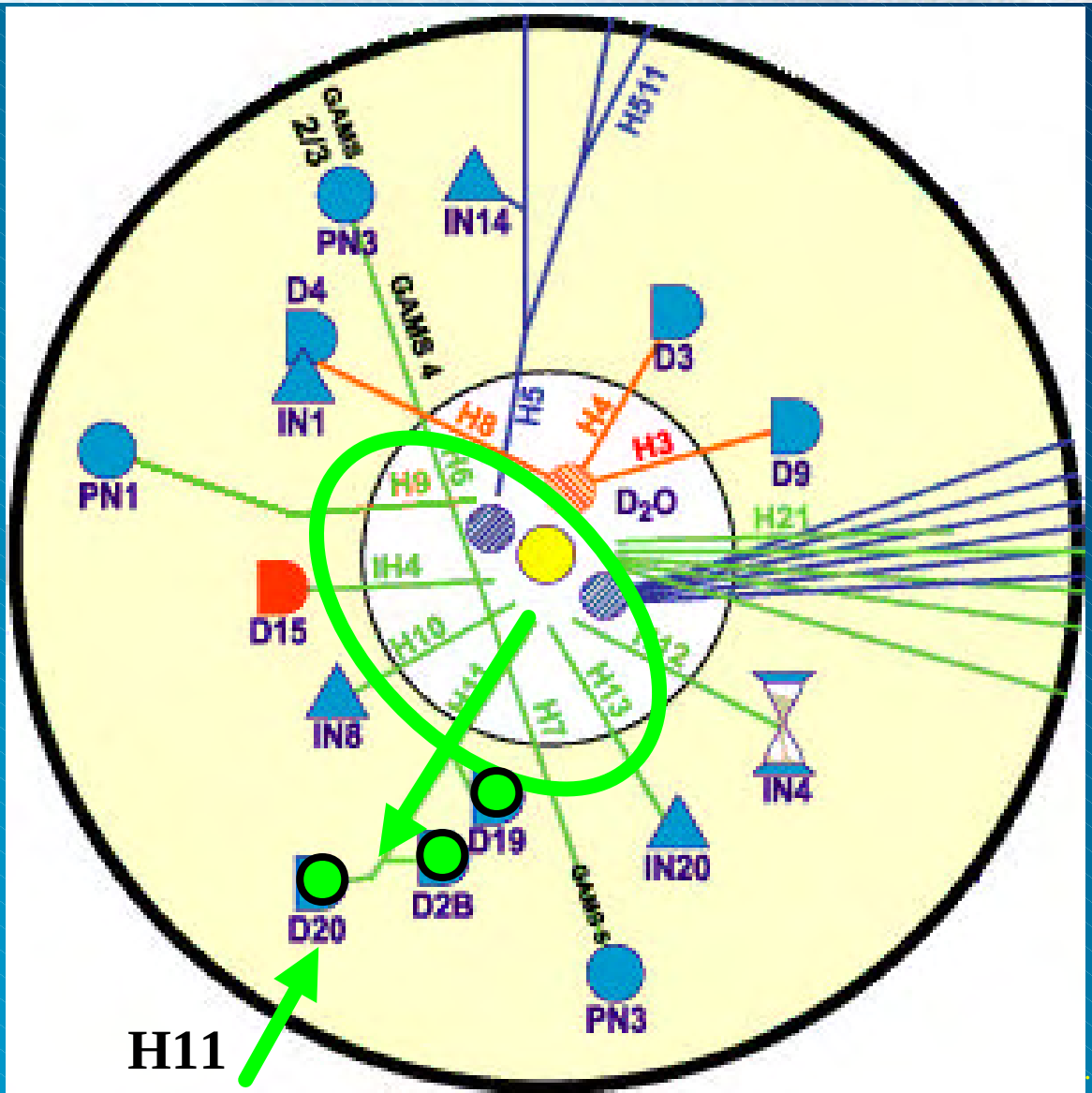
ANSTO-ECHIDNA HRPD diffractometer

Klaus-Dieter Liss with EuroCollimators & AZ-Systèmes

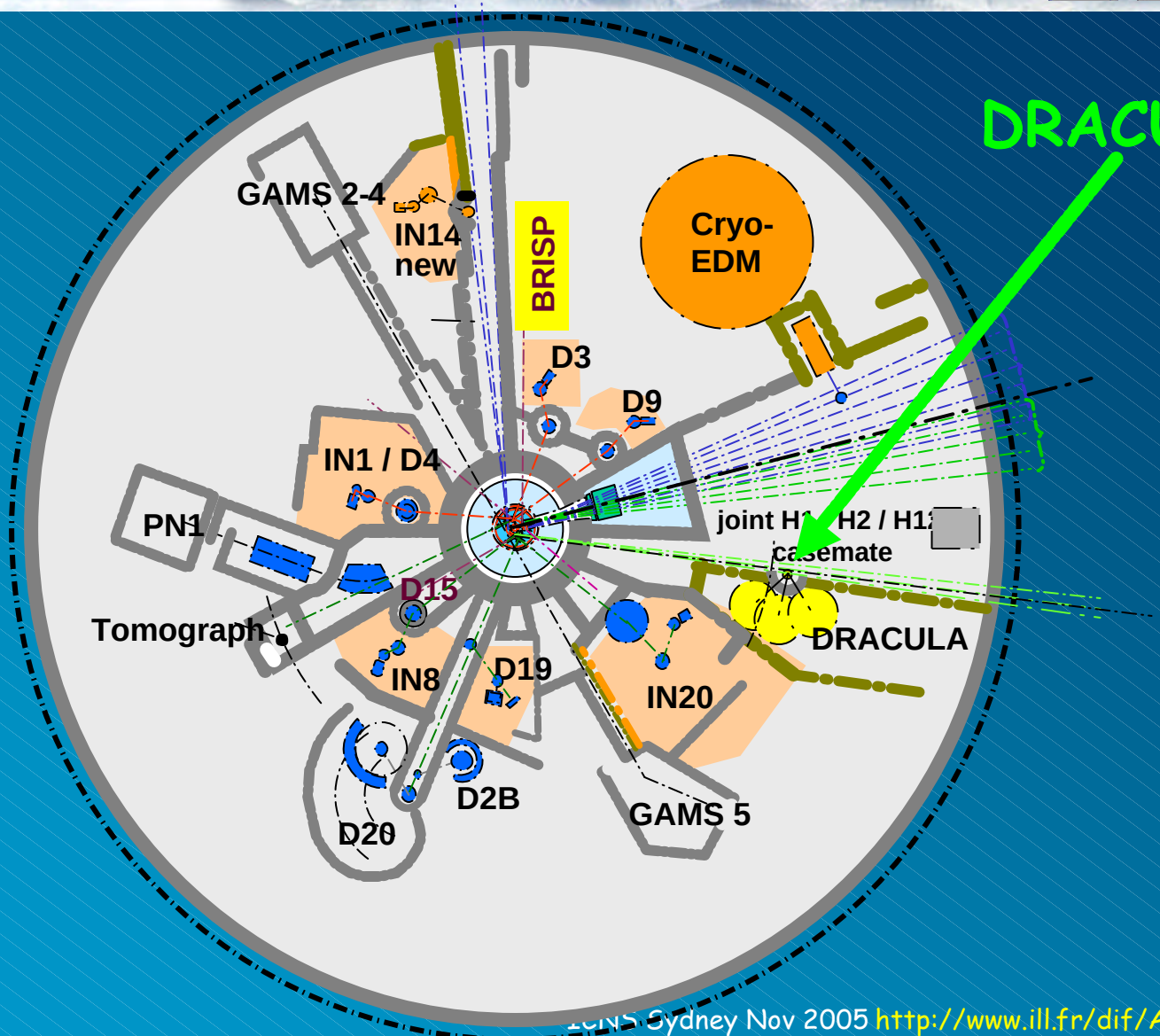
FRM-II SPODI HRPD diffractometer

Ralph Gilles, M.Schlapp, M.Hölzel, H.Fuess, H.Boysen

Only 1 ILL Thermal Beam for Diffraction (H11)



A New ILL Thermal Beam for Diffraction



A New ILL Thermal Beam for Diffraction

Alan Hewat & Henry Fischer, ILL

Diffractometer for
Rapid
ACquisition over
Ultra
Large
Areas

First presented at the ILL "Instrument Day" 26 Feb 2002

Highest priority for Instrument Committee 17 Oct 2003

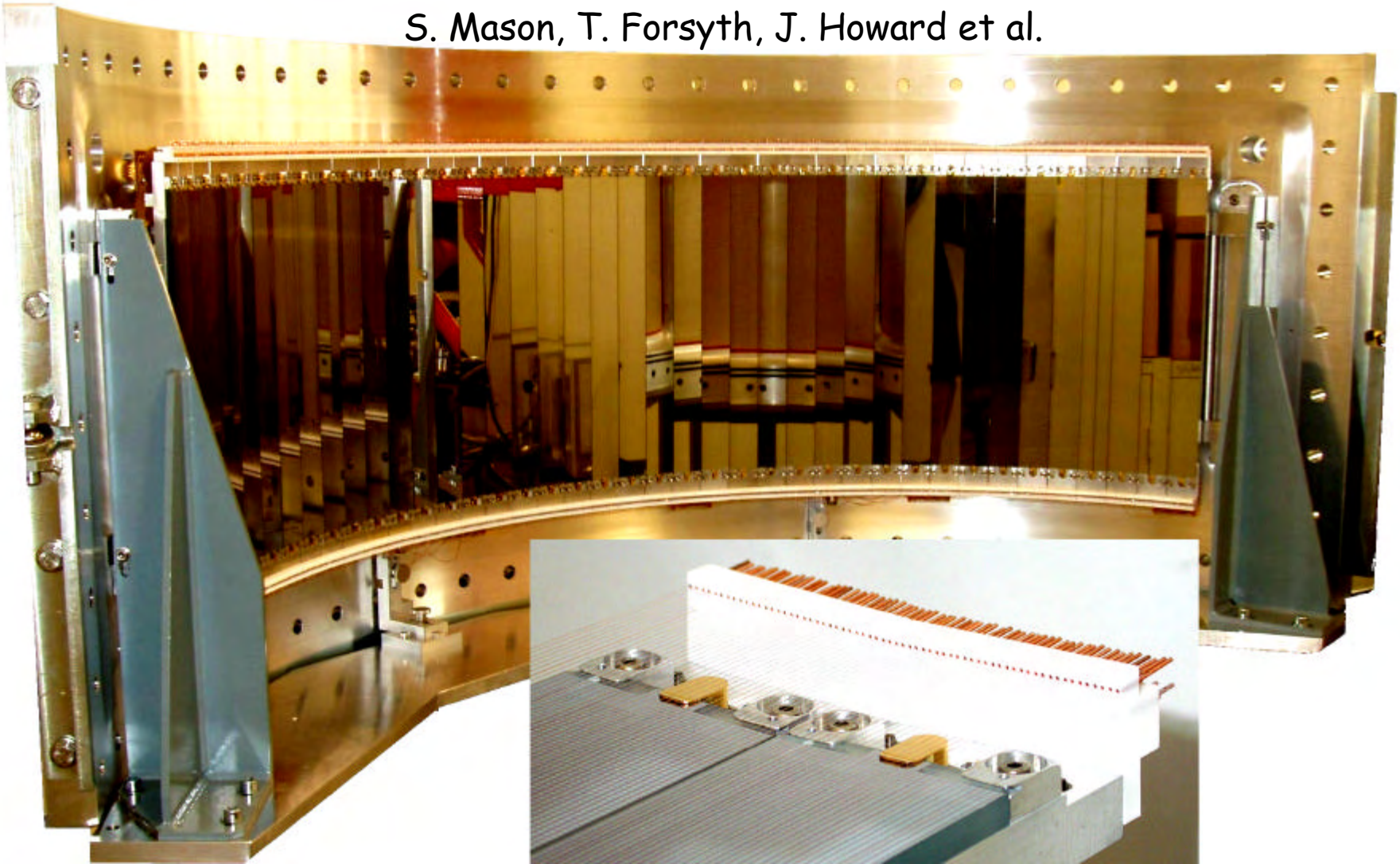
DRACULA construction scheduled for 2006 Millennium Programme

Dracula Ultra Large Area 2D Detector

B. Guérard, J-F. Clergeau, P. Van Esch, G. Viande et al.

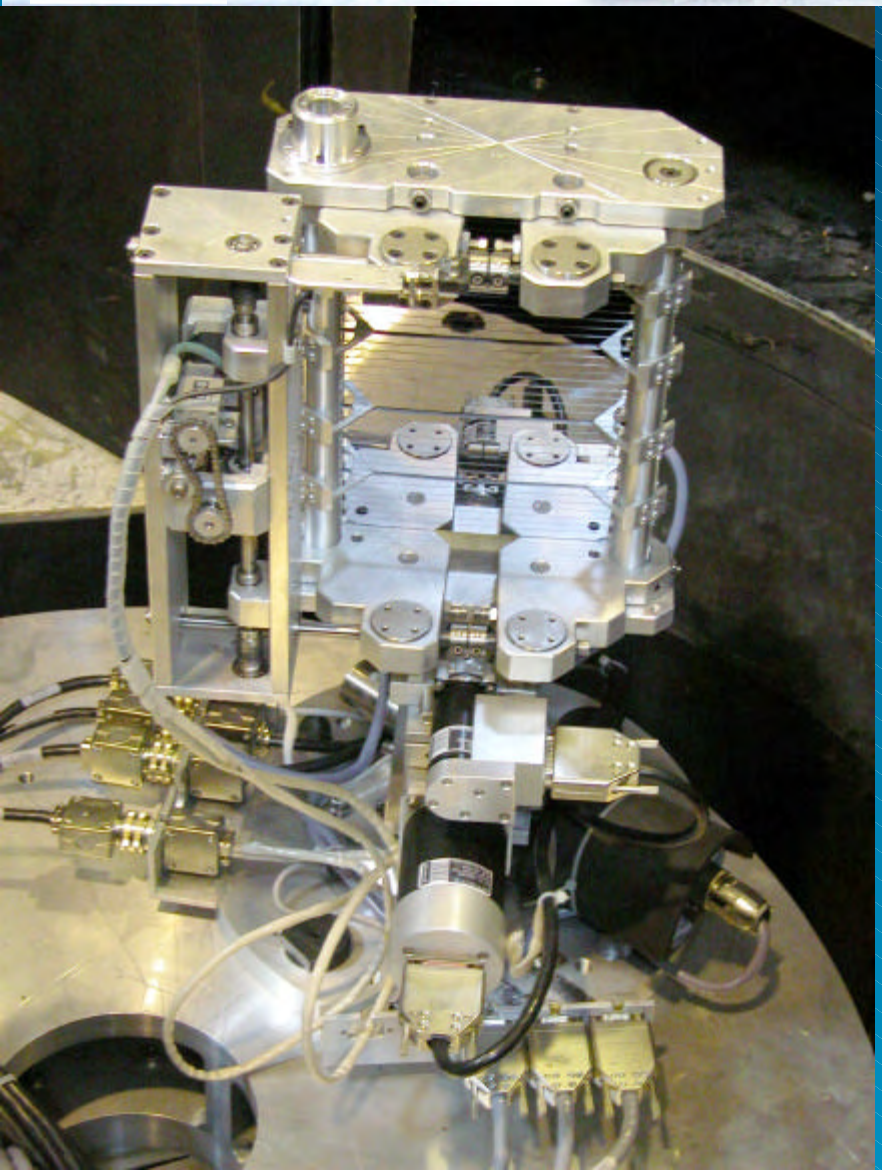
Based on new $120^0 \times 30^0$ 2D-detector for D19

S. Mason, T. Forsyth, J. Howard et al.



Dracula Focussing Geometry

Doubly focussing monochromator, l-focussing



Si[hhl] $2T_M=90^0$ monochromator

Doubly focusing bent wafers (SALSA)

D-spacings in the range $2T = 60^0\text{-}120^0$

- [115] l=1.54 Å d=0.89 Å to 1.54 Å
- [113] l=2.44 Å d=1.39 Å to 2.44 Å
- [111] l=4.61 Å d=2.66 Å to 4.61 Å

Comparison of TOF & CW Diffractometers

The time-averaged **Flux*Detector** criterium

	D20	GEM	DRACULA	SNS
Flux average on sample	5×10^7	$\sim2\times10^6$	$\sim10^8$	$\sim2.5\times10^7$
Detector solid angle	0.27 sr	4.0 sr	1.5 sr*	3.0 sr
Efficiency=Flux*Detector	1.7	1	18	9

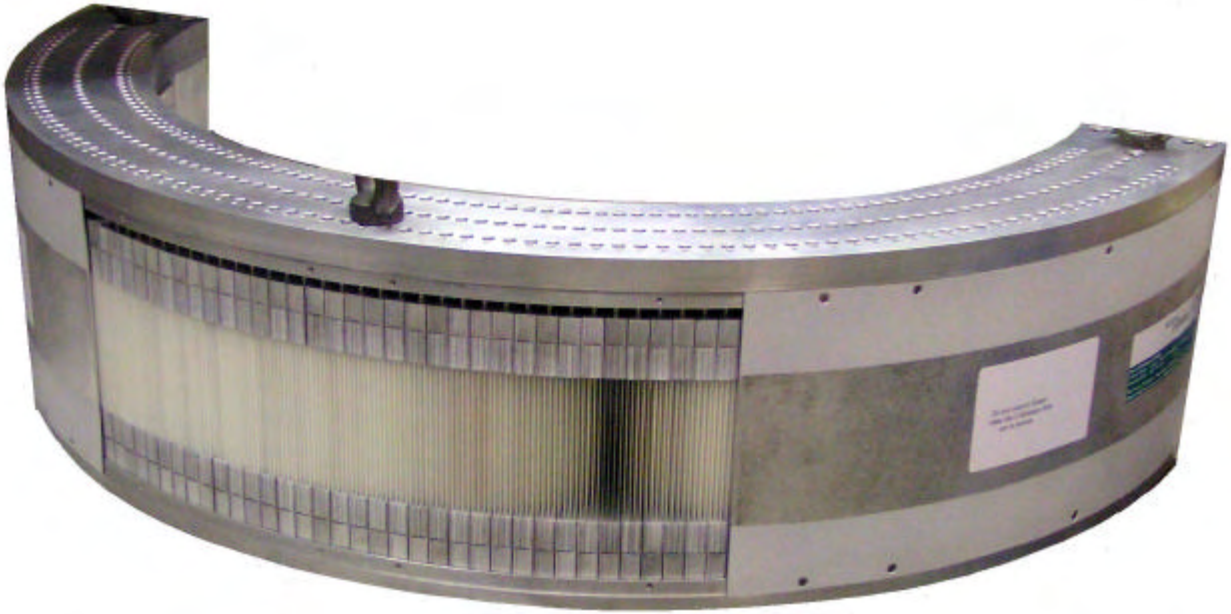
* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires 30°x160°

TOF machines already have big detectors

With big detectors ILL can compete with SNS intensity

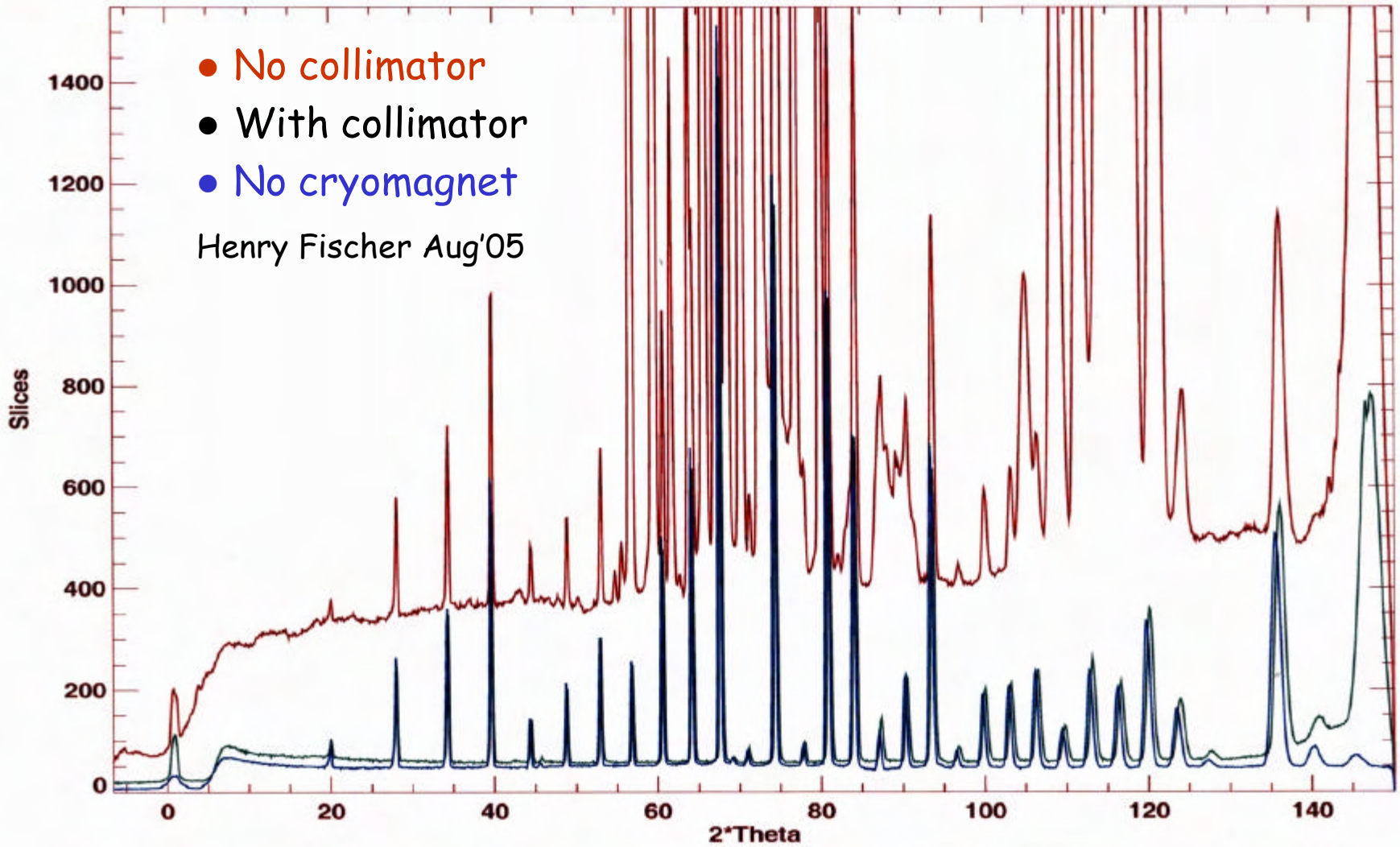
Dracula Sample Environment
Radial Collimator, Cryomagnet & Pressure

- 100Kbar ILL Paris-Edinburgh cell (N. Kernavanois et al.)
- D20 Radial Oscillating Collimator (H. Fischer, Th. Hansen, P. Henry)



Dracula Sample Environment
Radial Collimator & Cryomagnets on D20

Radial Collimator = 95% transmission, 3% background



New Science on Dracula

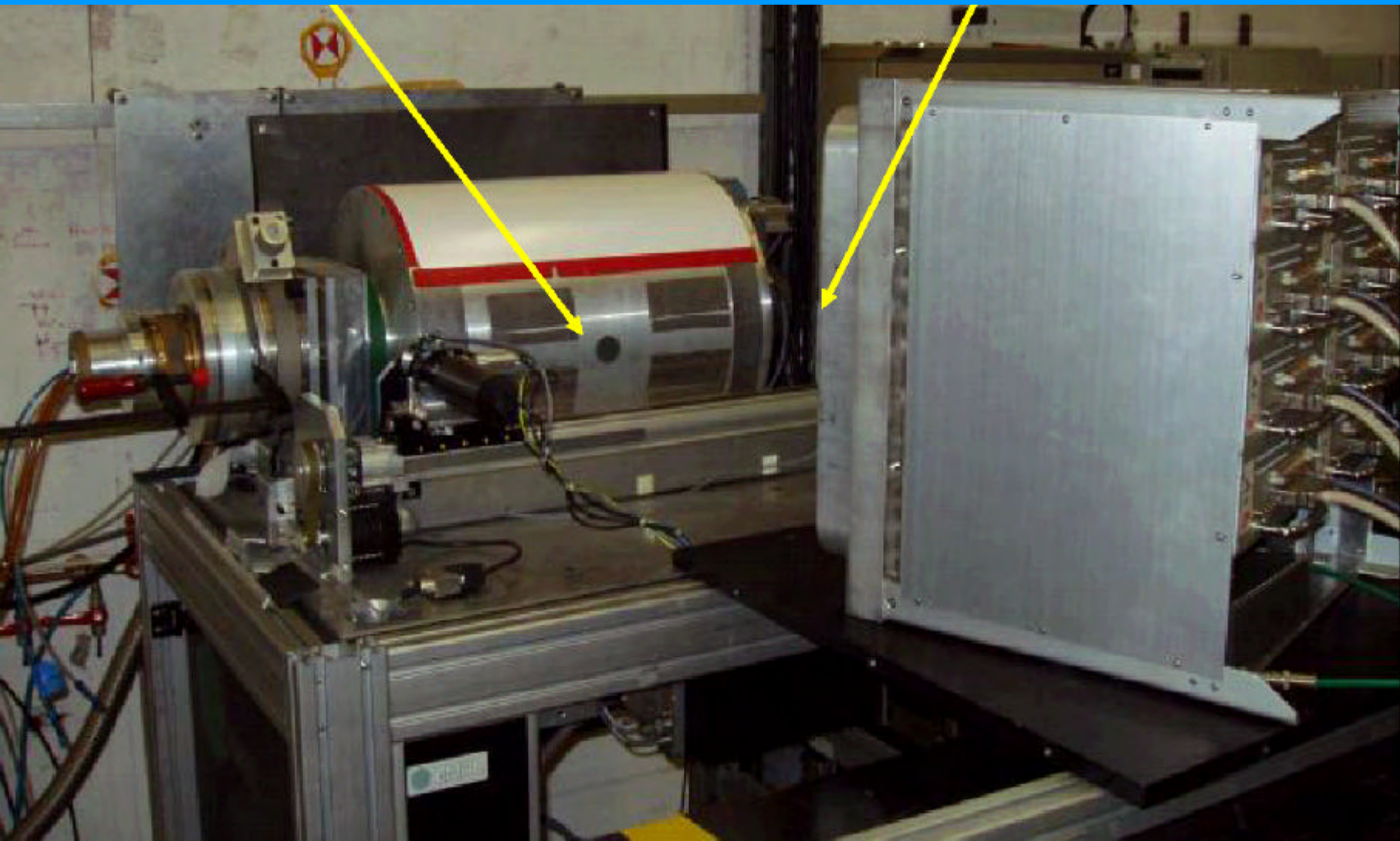
New science with a very high intensity neutron diffractometer

- High Pressure and Extreme Sample Environments
- New Materials from High Pressure Synthesis
- Isotope replacement contrast
- Strongly absorbing elements
- In-situ chemical kinetics
- Very fast reactions
- Very weak magnetic order and polarised neutrons
- Texture
- Even Single crystal diffraction

2D Electronic Detectors & Image Plates

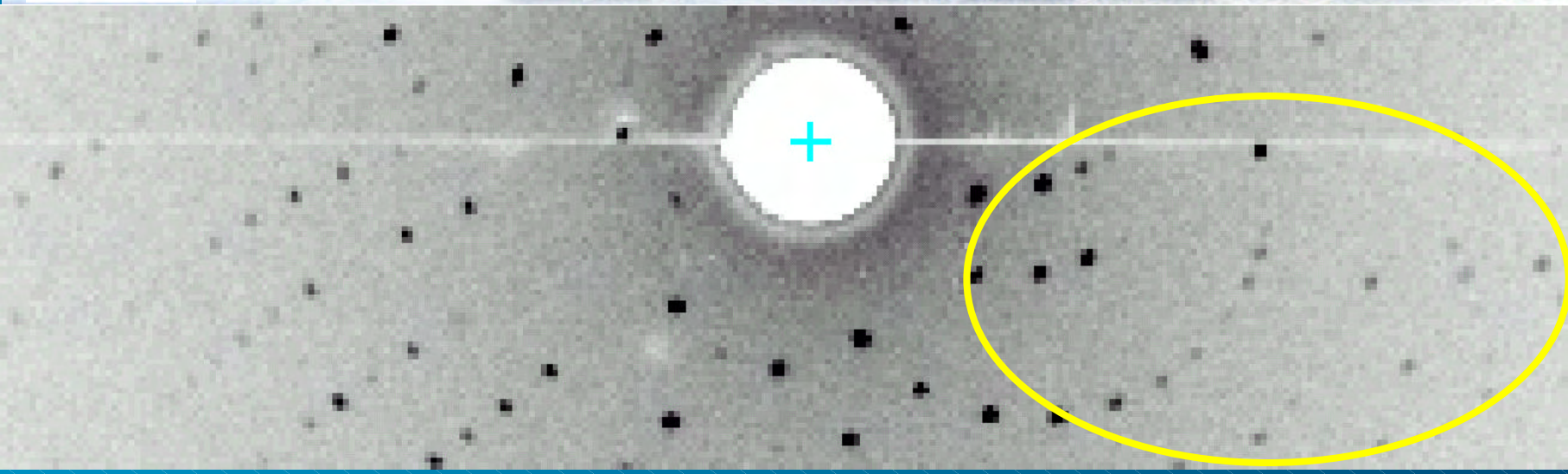
8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

LADI image plate vs D19 proto detector

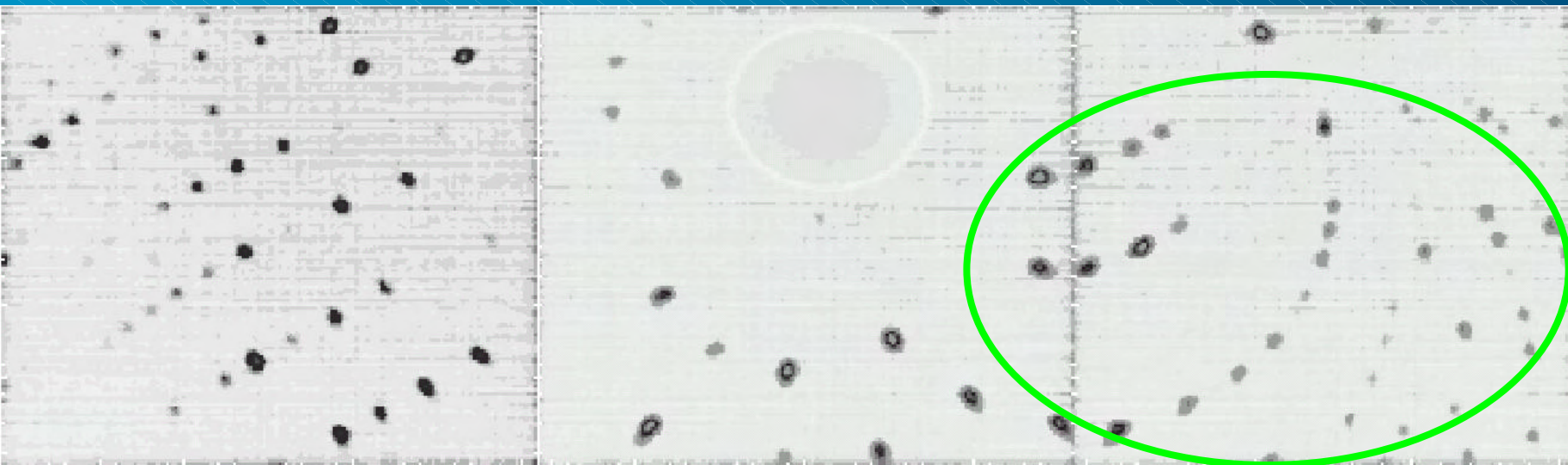


2D Electronic Detectors & Image Plates

8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

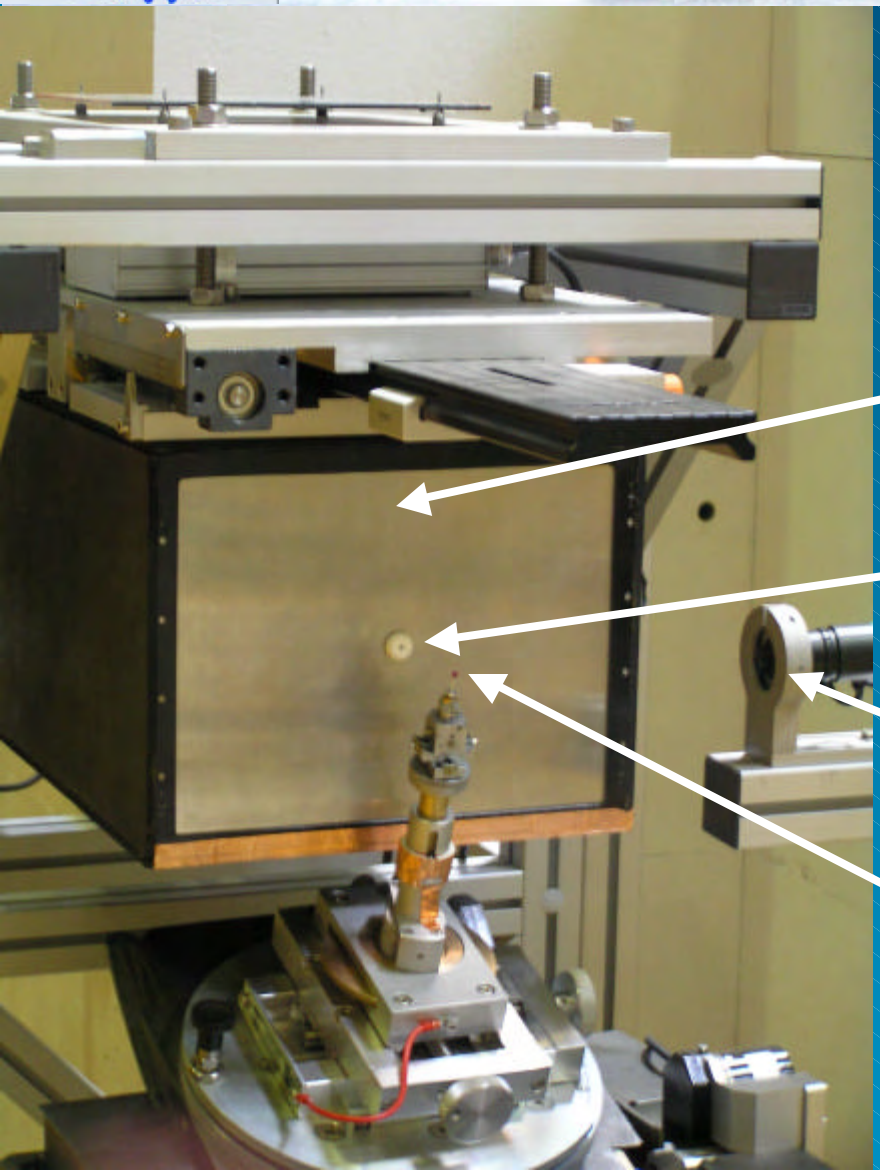


LADI image plate (12 hours above) vs D19 proto detector (1 hour below)



ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



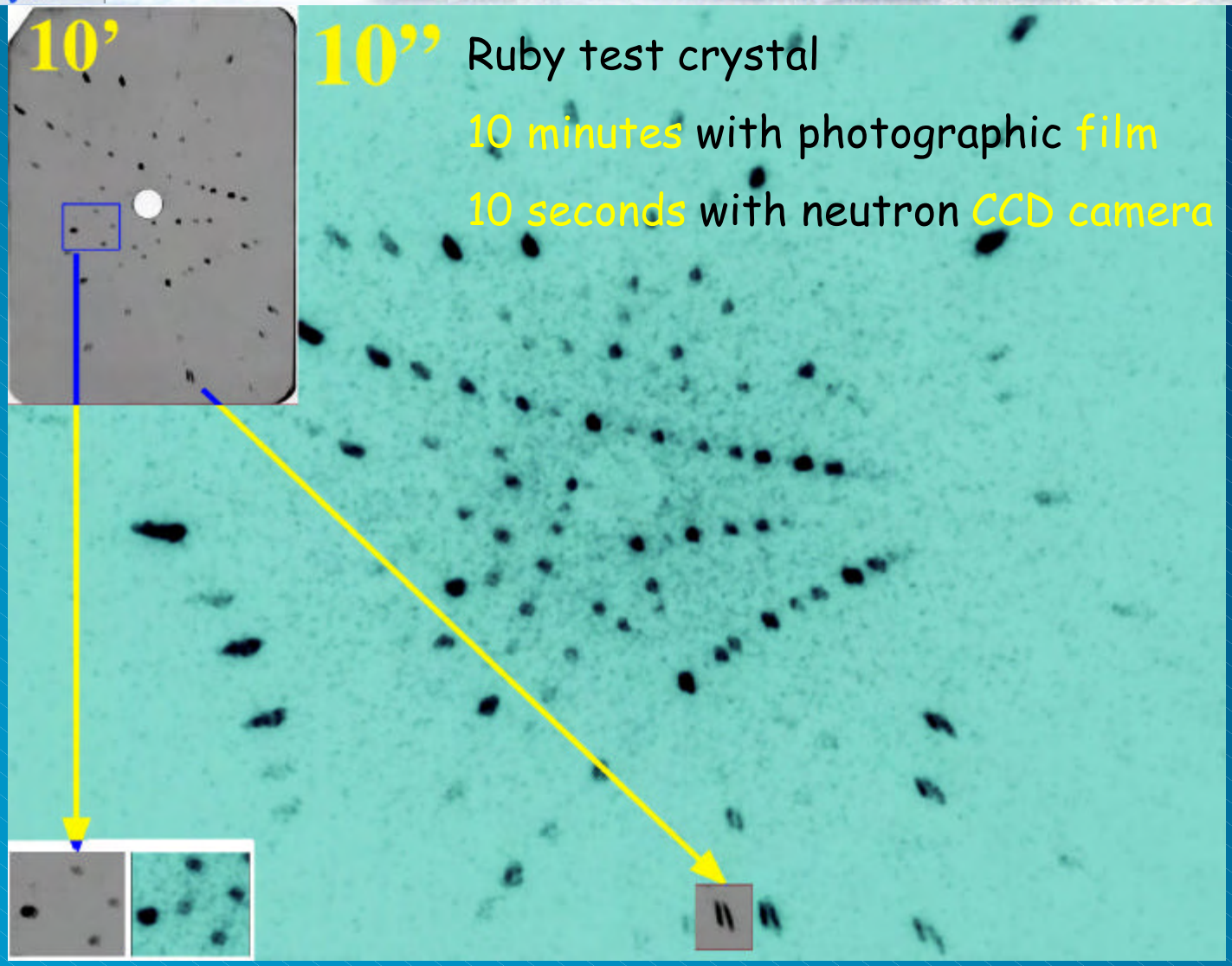
Orient Express

On H24 thermal neutron guide

Dual lens-coupled chilled ICCD's

ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



Ruby test crystal

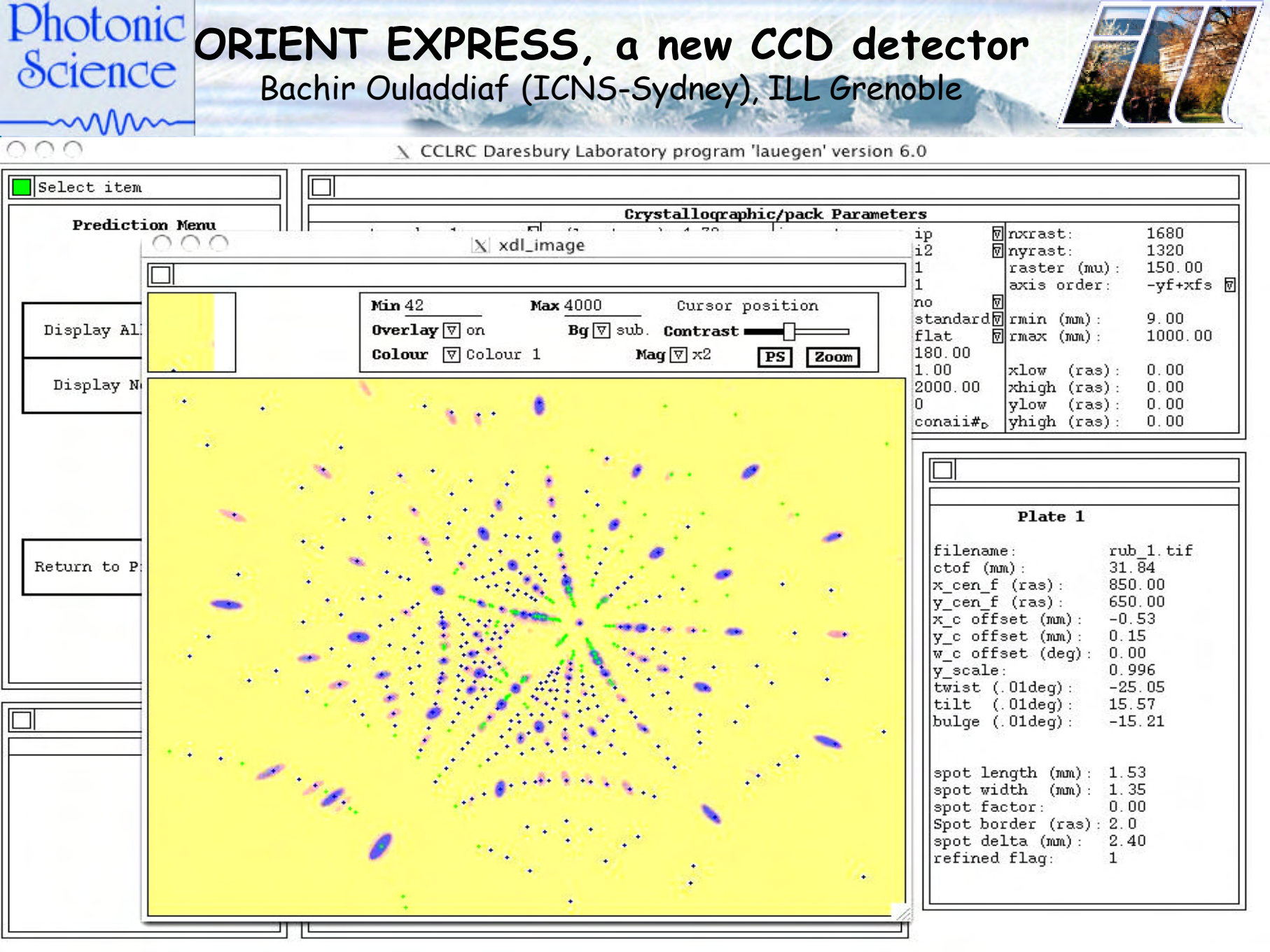
10 minutes with photographic film

10 seconds with neutron CCD camera

Photonic Science

ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focussing)
- A wide band of wavelengths
- A very high flux on the sample
- A very large area detector

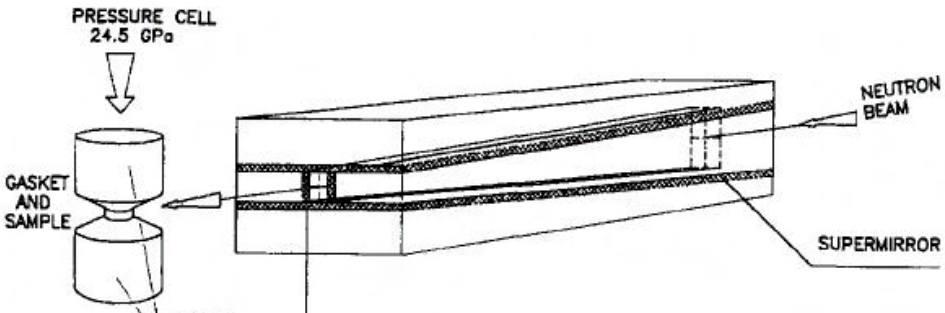
Next Generation Neutron Diffractometers

A large incident solid angle (focussing)

Focusing neutrons to study small samples

I.N. Goncharenko[a,b,*], I. Mirebeau[a], P. Molina[c], P. Böni[d]

[a] *Laboratoire Léon Brillouin, C. E. A.-C. N. R. S., C. E. Saclay, 91191 Gif-sur-Yvette, France*
[b] *Russian Research Center Kurchatov Institute, 123182 Moscow, Russian Federation*
[c] *Ecole Centrale de Nantes, 1 rue de la Noé, 44000 Nantes, France*
[d] *Laboratory for Neutron Scattering ETH&PSI, CH-5232 Villigen PSI, Switzerland*



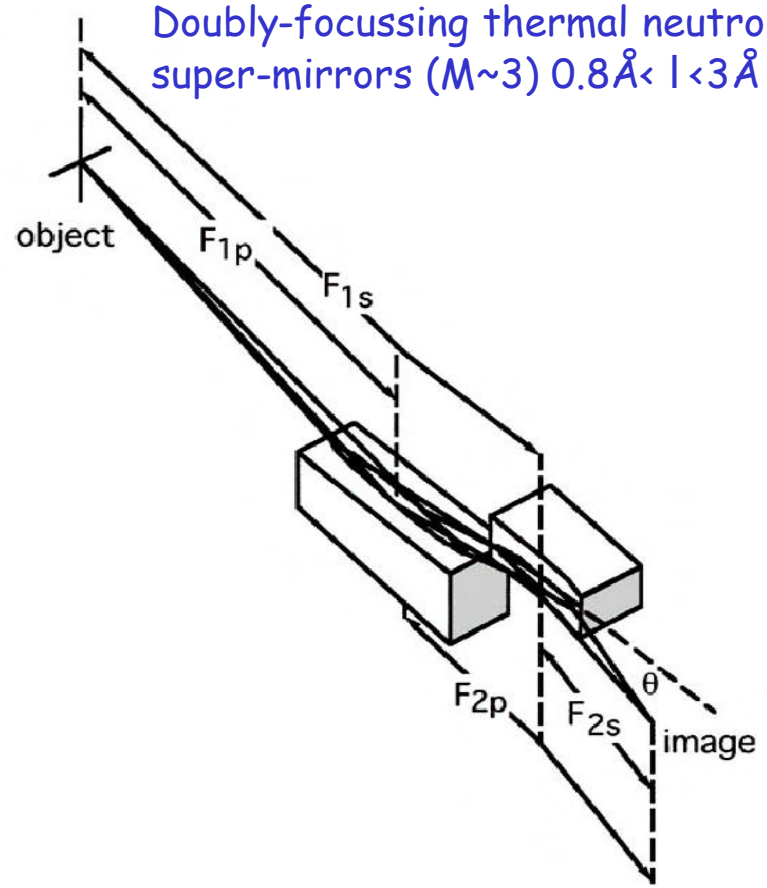
Kirkpatrick–Baez microfocusing optics for thermal neutrons

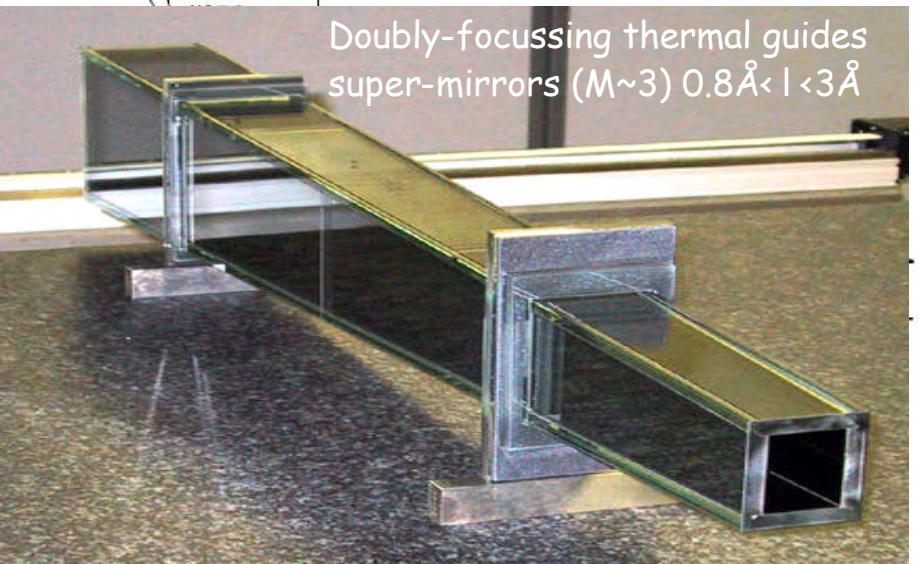
Gene E. Ice[a,*], Camden R. Hubbard[a], Bennett C. Larson[a], Judy W. L. Pang[a], John D. Budai[a], Stephen Spooner[a], Sven C. Vogel[b]

[a] *Oak Ridge National Laboratory, Rm R260, P.O. Box 2008, Bldg. 4500S, MS-6118, Oak Ridge, TN 37831-6118, USA*
[b] *Los Alamos National Laboratory, USA*

Received 8 April 2004; received in revised form 19 July 2004
Available online 18 November 2004

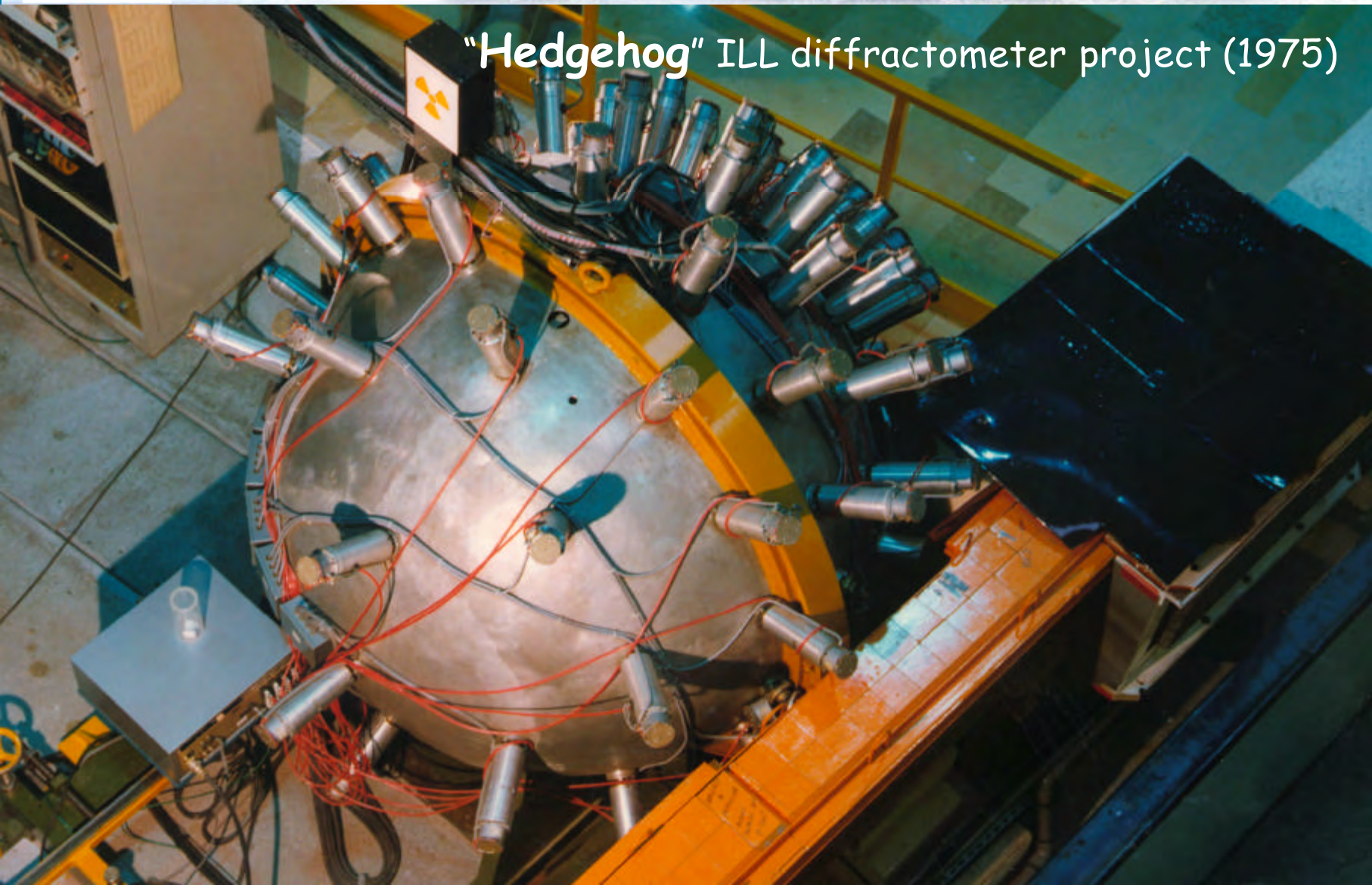
Doubly-focussing thermal neutron super-mirrors (M~3) 0.8Å< l <3Å





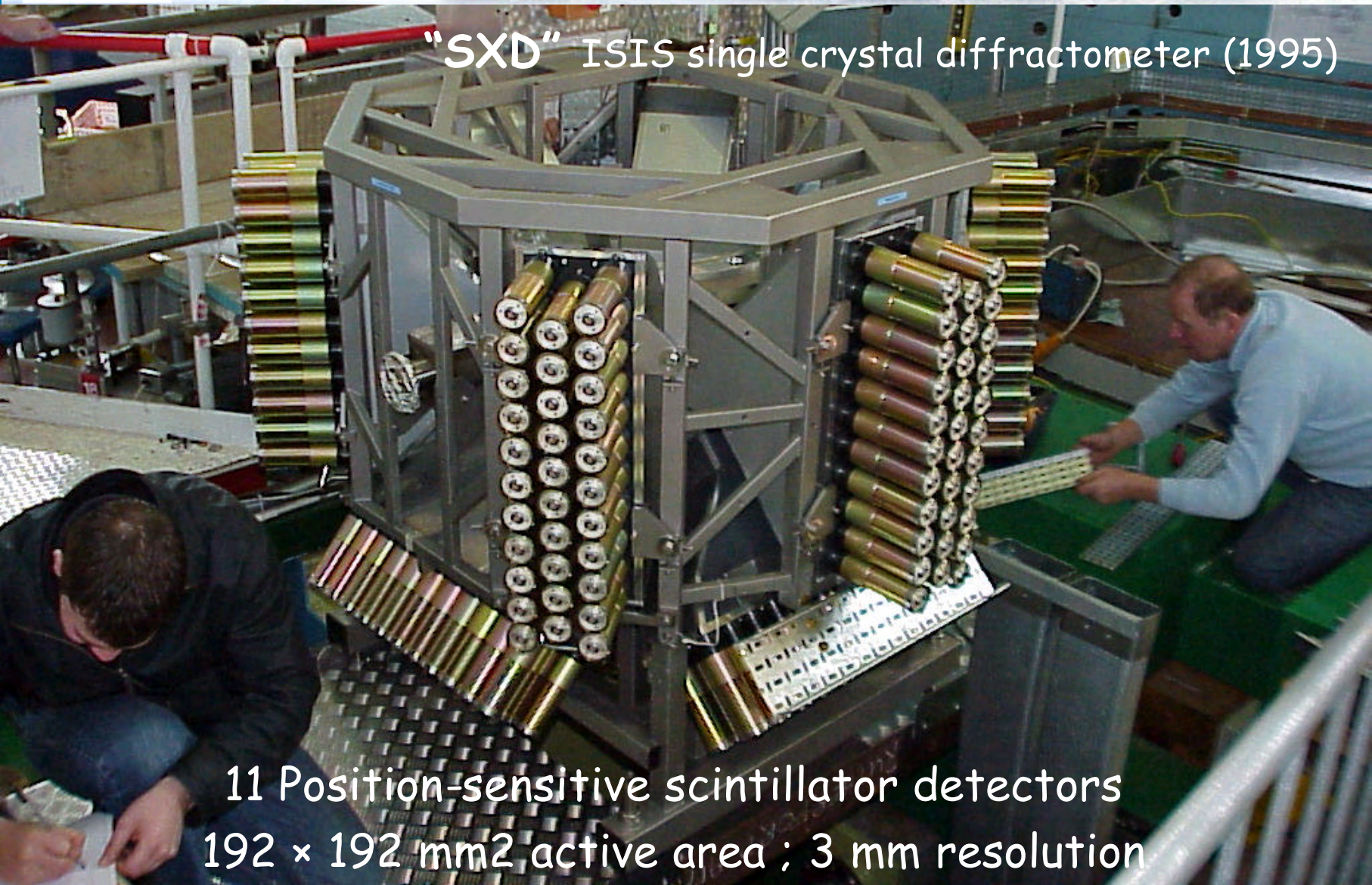
Next Generation Neutron Diffractometers

A very large area detector



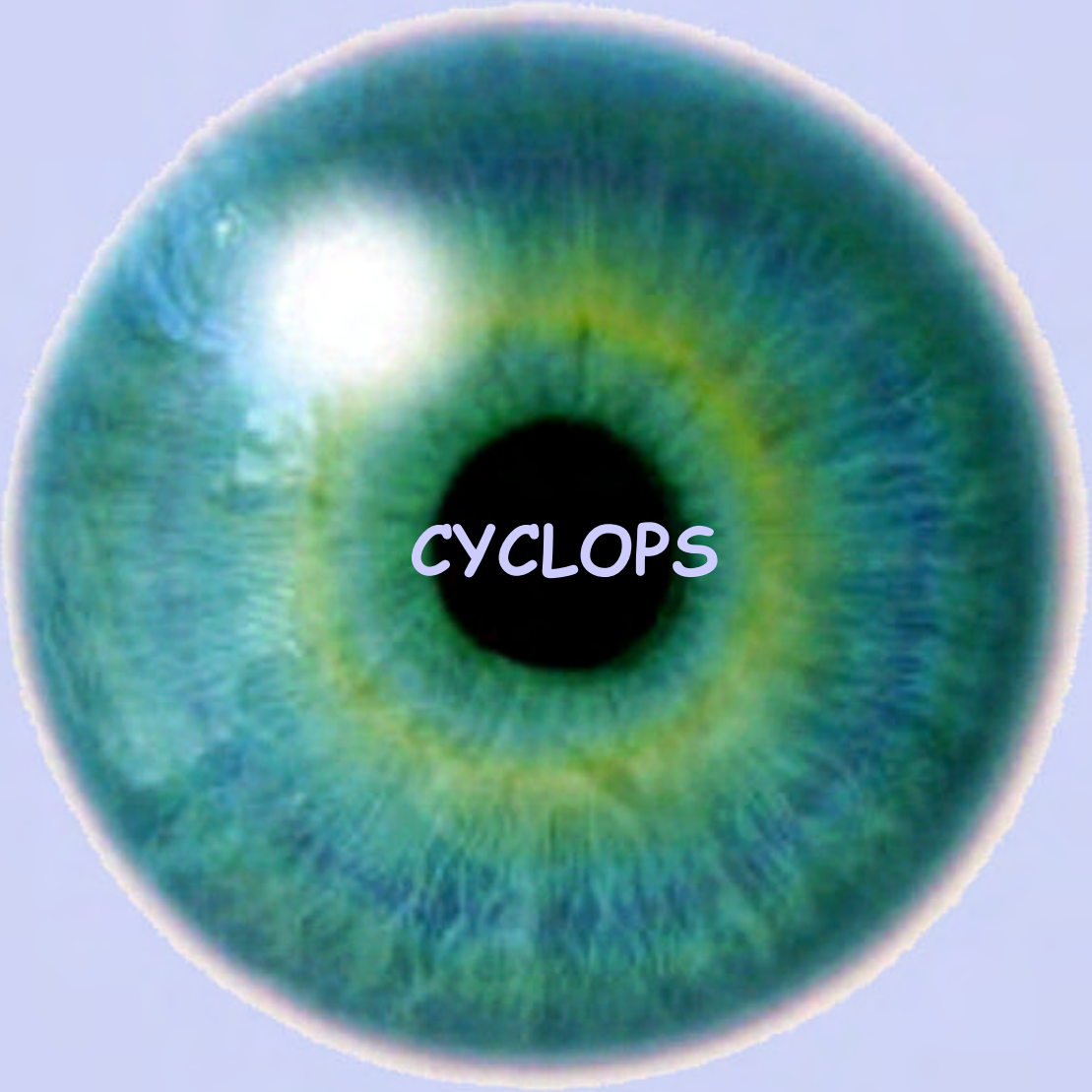
Next Generation Neutron Diffractometers

A very large area detector



Next Generation Neutron Diffractometers

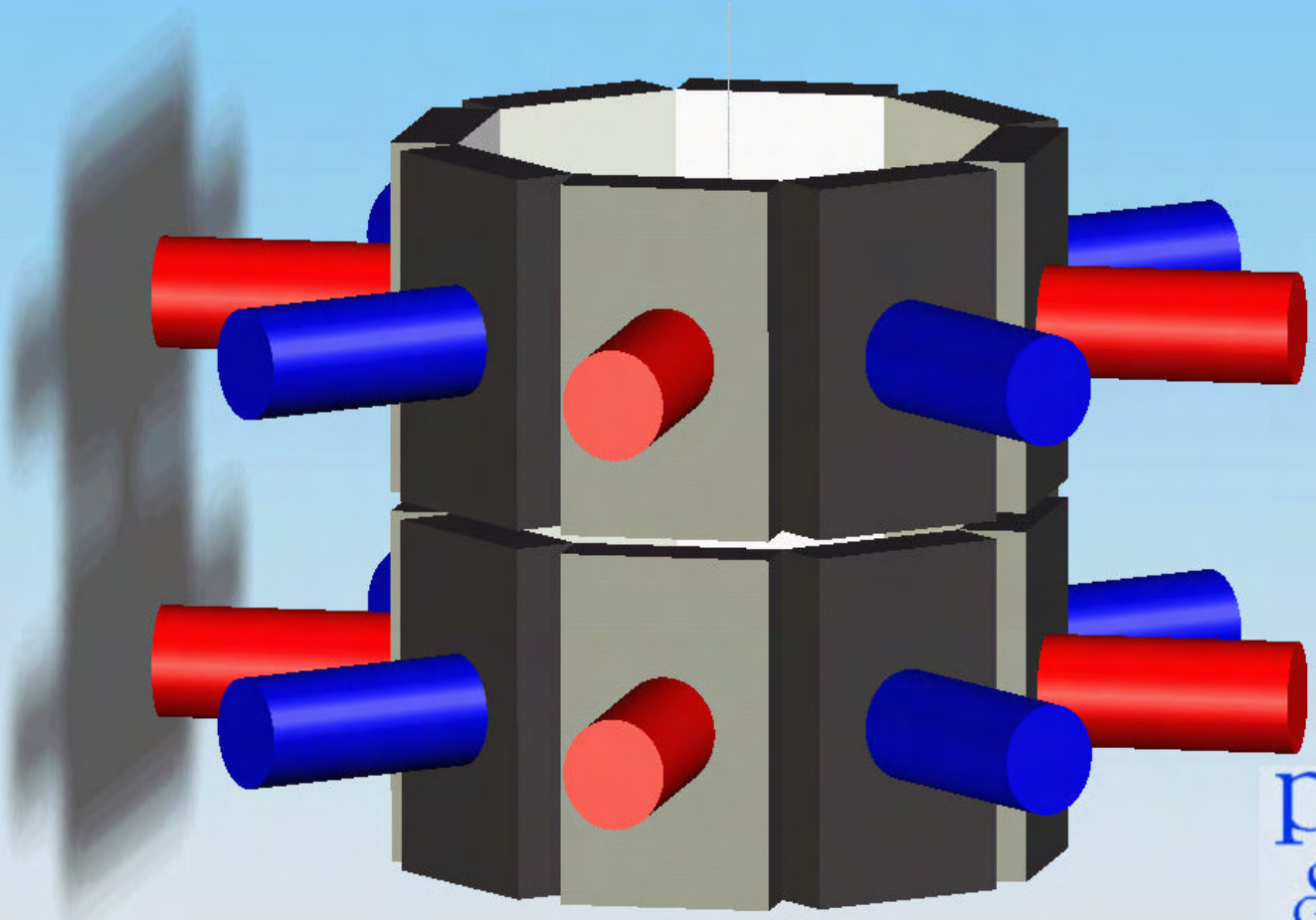
CYlindrical **C**CD **L**aue **O**ptics **P**hoto **S**cintillator



Next Generation Neutron Diffractometers

CYlindrical **C**CD **L**aue **O**ptics **P**hoto **S**cintillator

Double octagonal array of CCD neutron cameras



Photonic Science

Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focussing)
- A wide band of wavelengths
- A very high flux on the sample
- A very large area detector

CW diffractometers finally have what it takes

High Flux Diffractometers on Reactors

Alan Hewat, ILL Grenoble

ILL Diffraction Group Scientists

Jane Brown (Emeritus)
Giovanni Bruno (Salsa)
Tapan Chatterji (D10)
Gabriel Cuello (D4)
Marie-Theresa Fernandez-Diaz (D9)
Henry Fischer (D20, Dracula)
Trevor Forsyth (D19, D-Lab)
Thomas Hansen (D20)
Paul Henry (D20)
Nolwenn Kernavanois (D3)
Marie-Helene Lemée-Cailleau (Vivaldi)
Sax Mason (D19)
Garry McIntyre (D10, Vivaldi)
Bachir Ouladdiaf (D10, Orient Exp.)
Thilo Pirling (SALSA)
Clemens Ritter (D2B)
Anne Stunault (D3)
Emmanuelle Suard (D2B)
Clive Wilkinson (Vivaldi)

A Special Thanks to ICNS-2005

Brendan Kennedy, ICNS Chairman
Evan Grey, Programme Committee
Stewart Campbell, ANBUG users
Dennis Mather, AINSE & sponsors
Rob Robinson, Bragg Institute

Steve Hull, ISIS Session Chairman