High Flux Neutron Diffractometers at ILL

Alan Hewat, ILL Grenoble

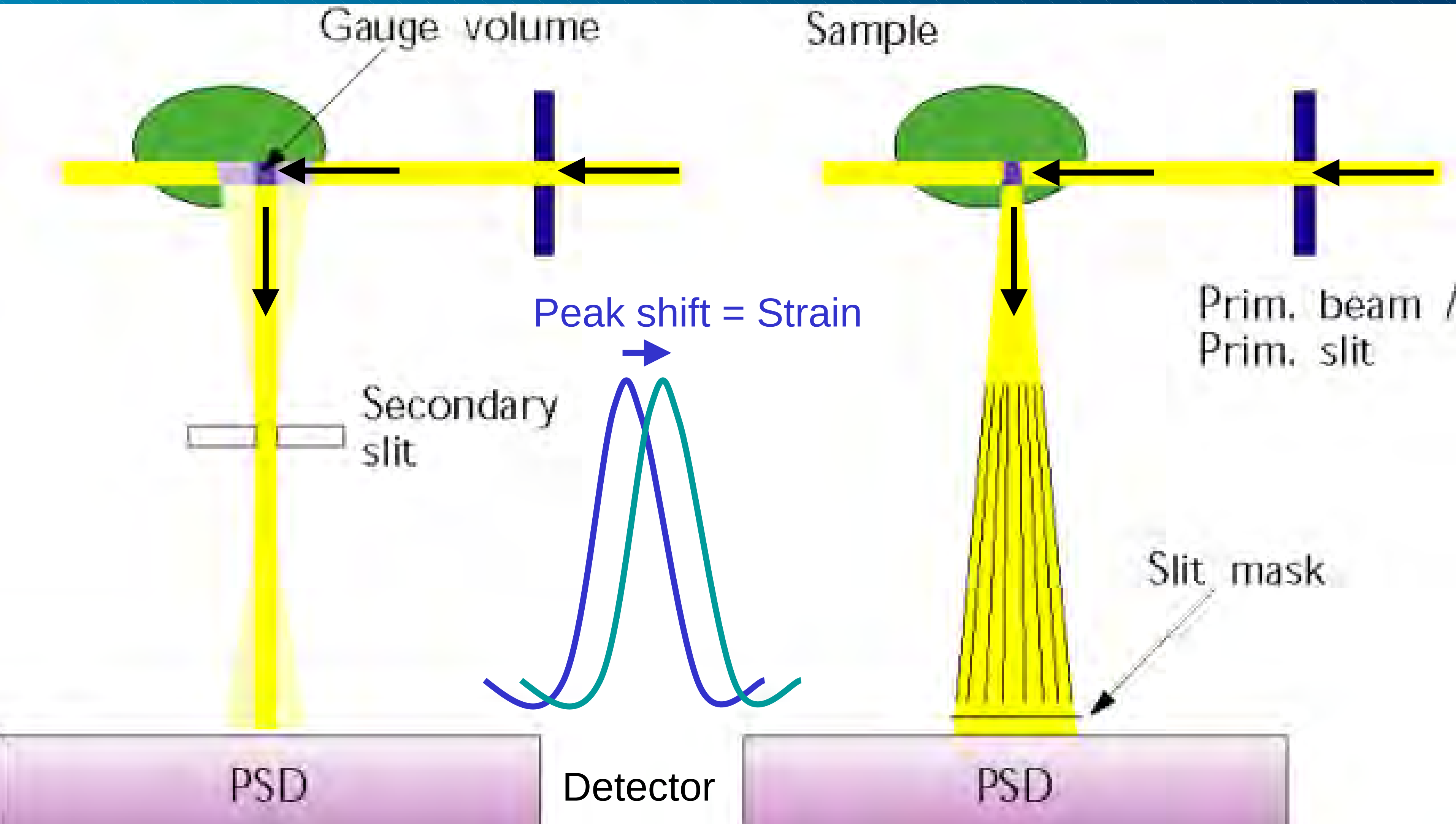
Newly available/upgraded diffractometers at ILL

- **SALSA** neutron strain scanner (Ph.Withers, Manchester)
- **FaME38** industrial support lab (N.James, Plymouth)
- **Super-D2B** high resolution powders (P.Attfield, Edinburgh)
- **D20** high flux powder diffractometer (Higher Resolution)
- **D19** 2D position sensitive detector (J.Howard, Durham)
- **VIVALDI** neutron image plate (C.Wilkinson, London)

New Proposals

- **DRACULA** (Diffractometer for Rapid ACquistion)
- **CYCLOPS** (CYlindrical Ccd Laue Optics Photo-Scintillator)

Neutron Strain Scanning

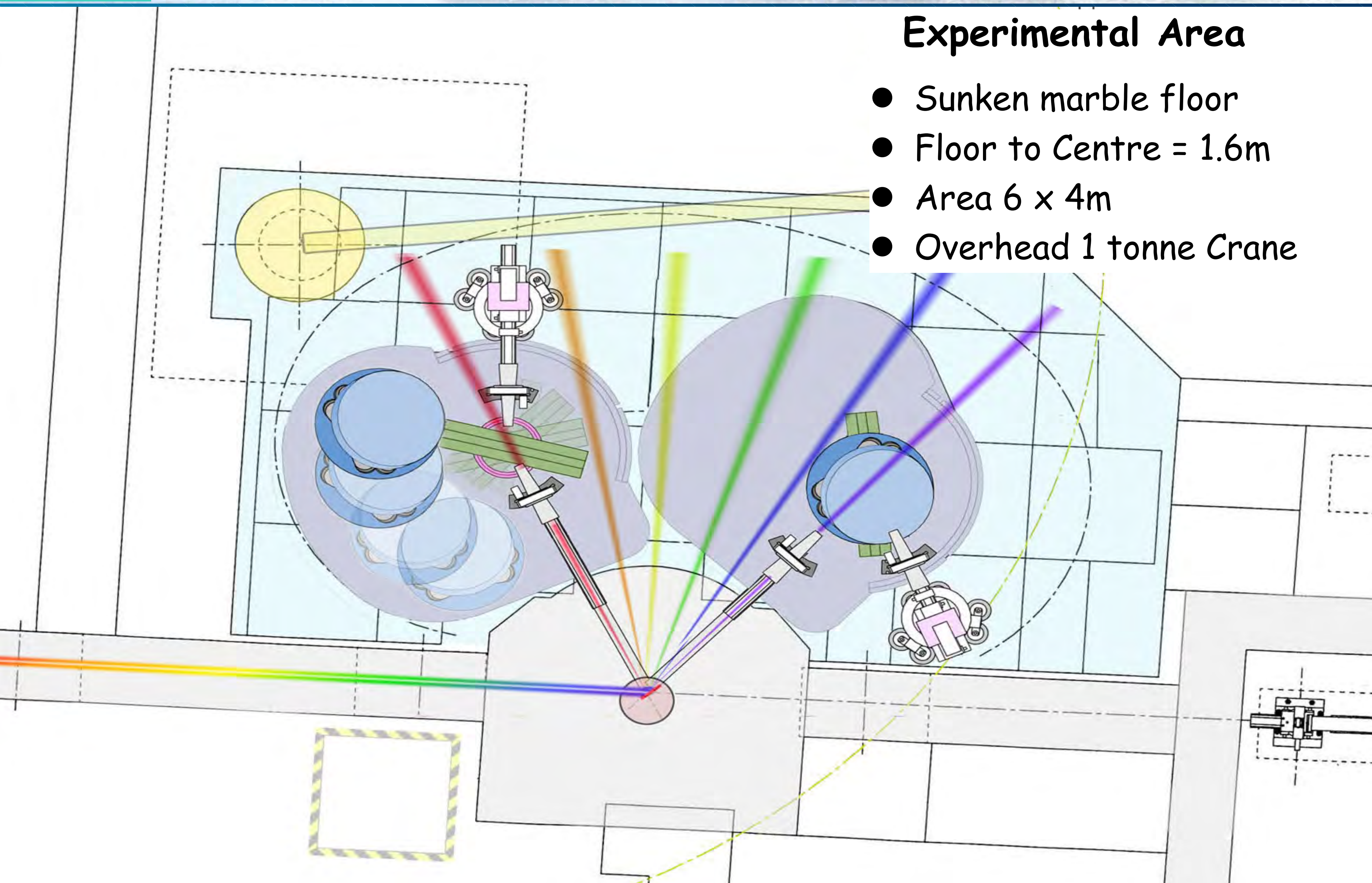


SALSA, A strain scanner using a hexapod

Phil. Withers (Manchester), P.J.Webster, G.A.Webster, A.M.Korsunsky,
G.M.Swallowe, L.Edwards, M.E.Fitzpatrick, G.Bruno, Th.Pirling (ILL)

Experimental Area

- Sunken marble floor
- Floor to Centre = 1.6m
- Area 6 x 4m
- Overhead 1 tonne Crane



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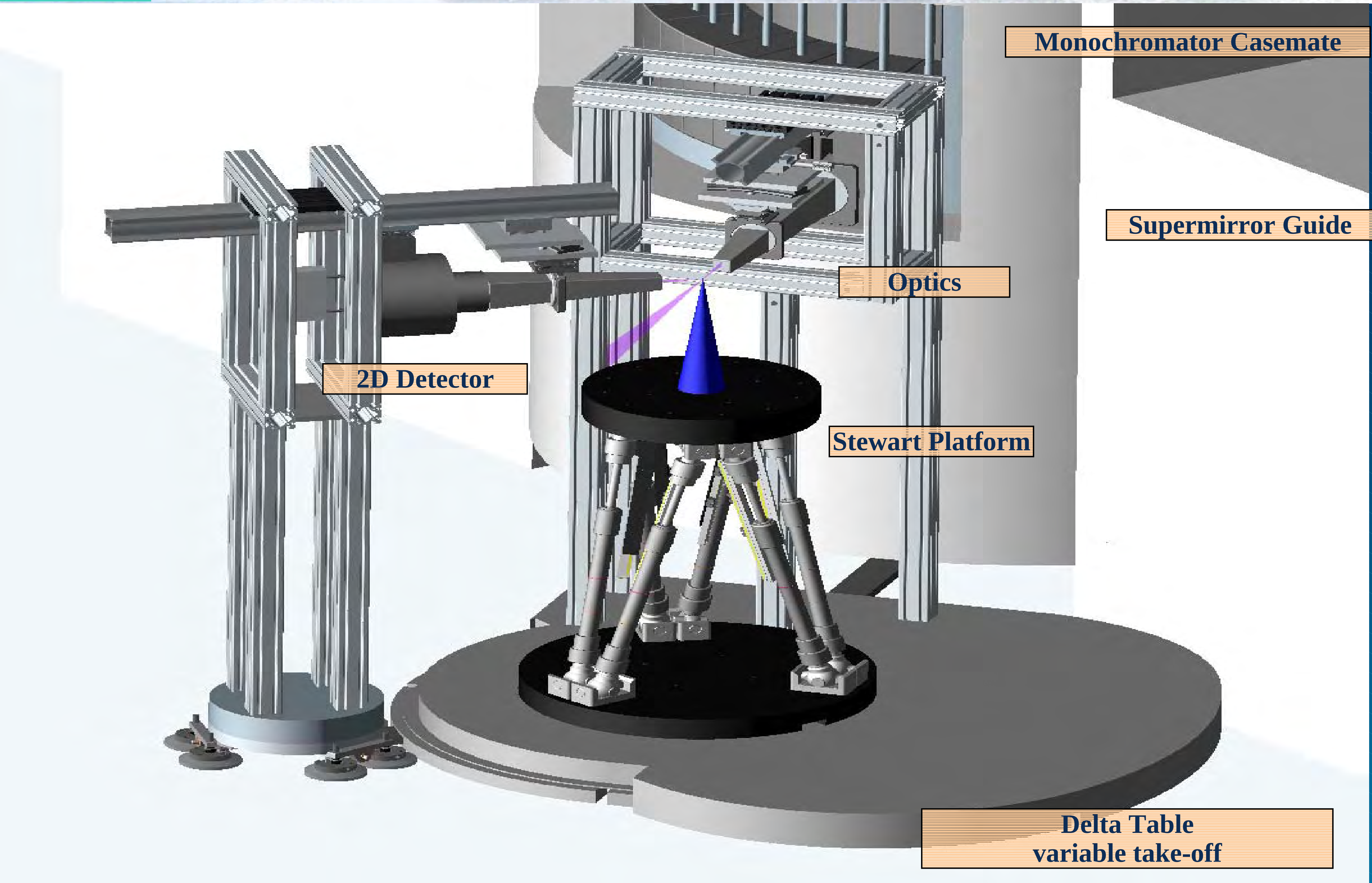

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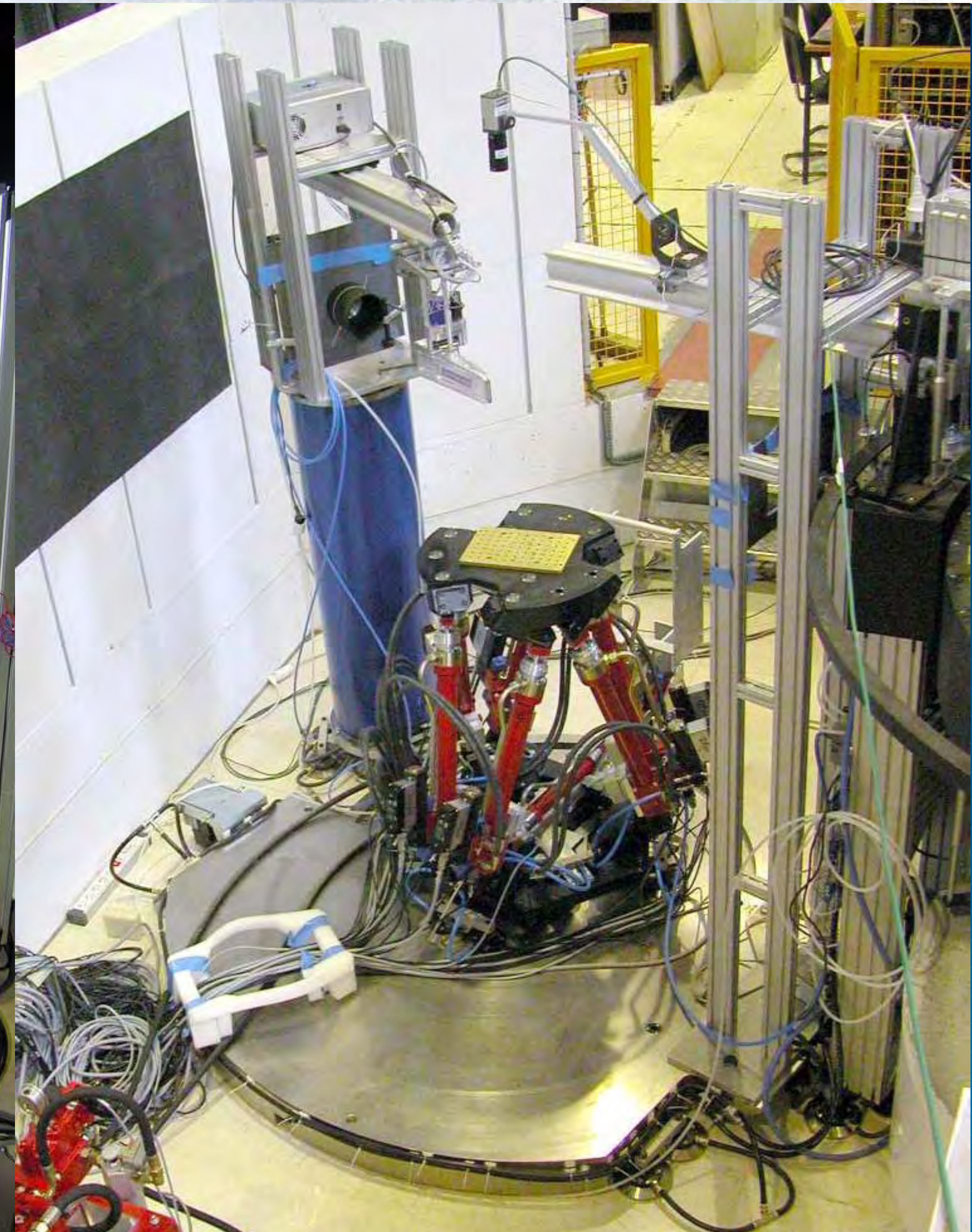

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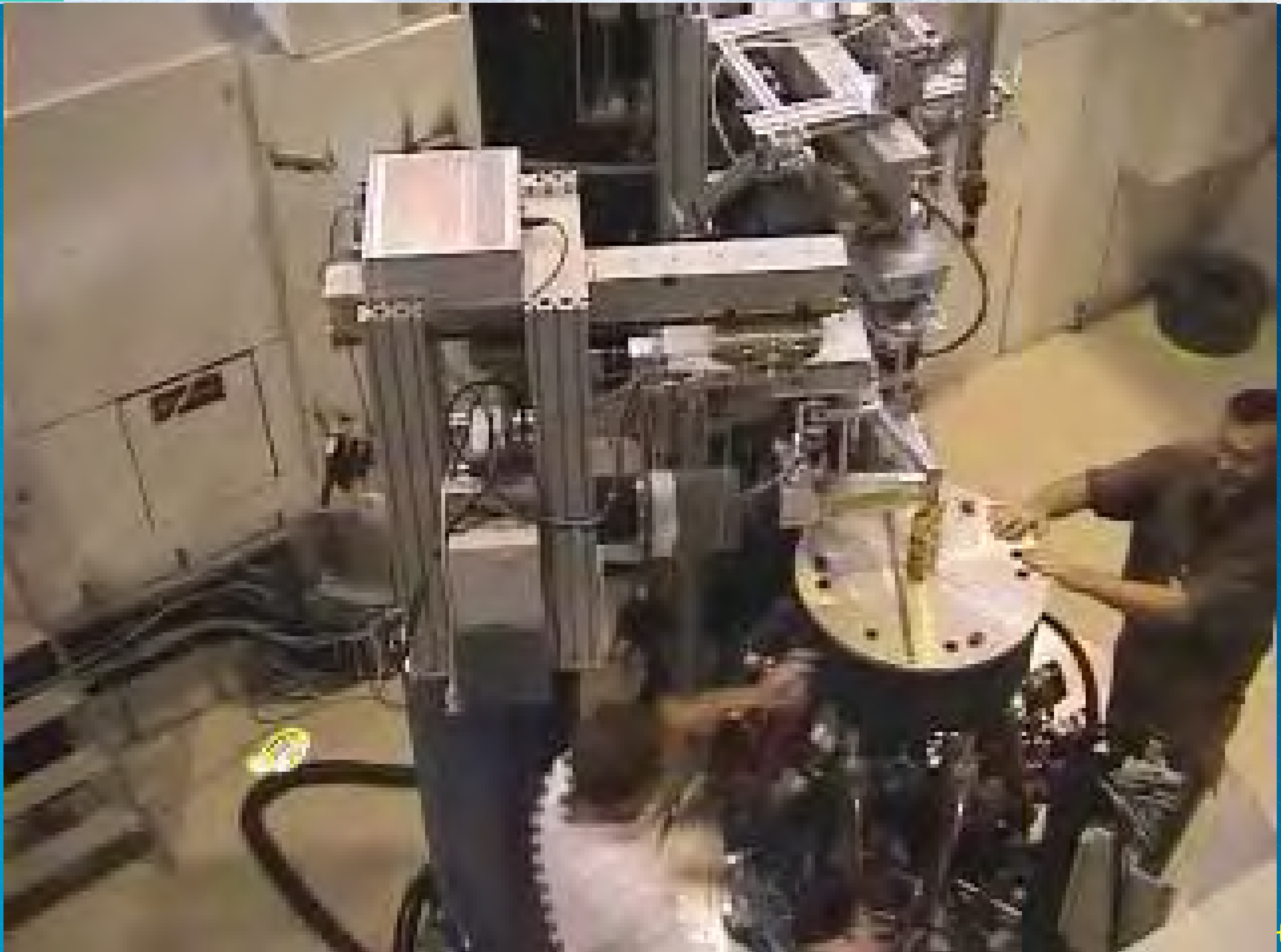

Hewat/

SALSA, A strain scanner using a hexapod

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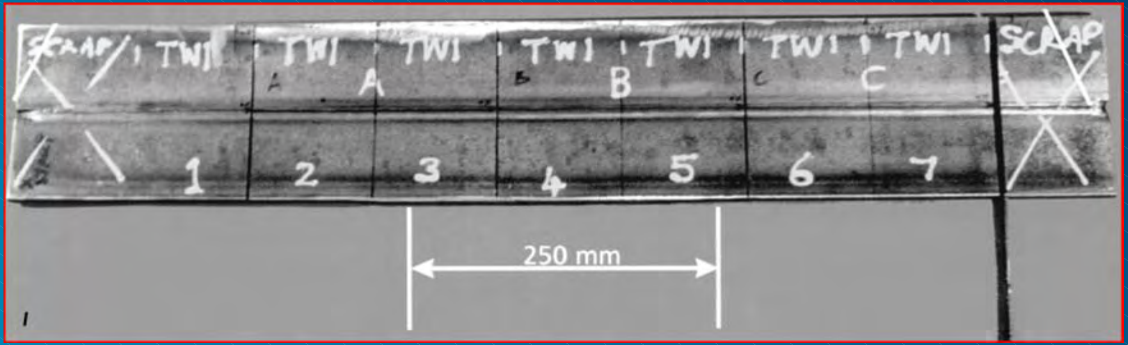
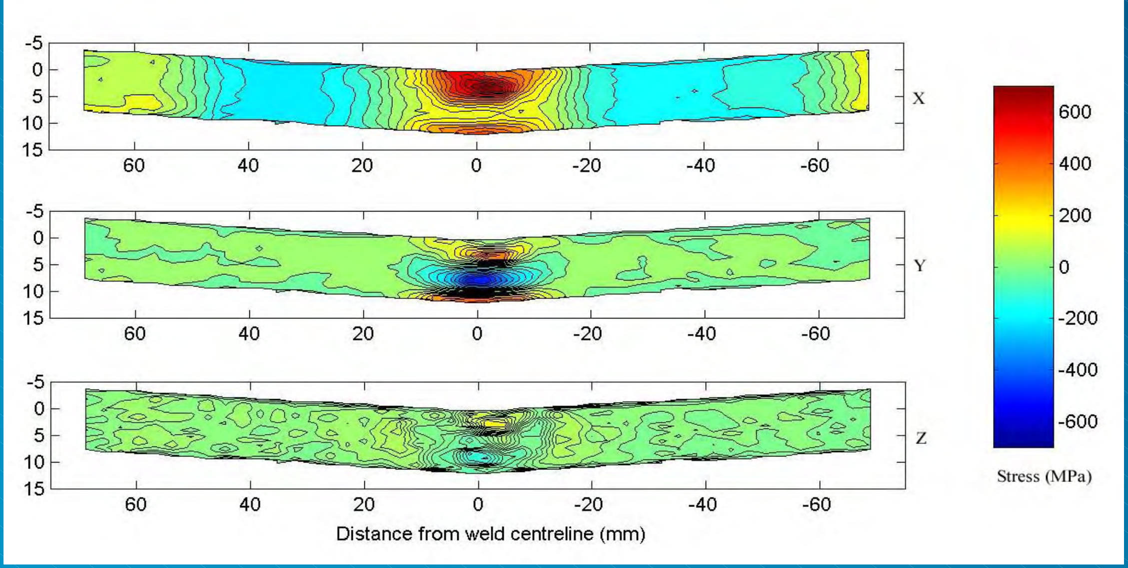
ISO standard VAMAS ferretic steel weld measured in many labs for comparison

Relationship between fatigue and residual stress



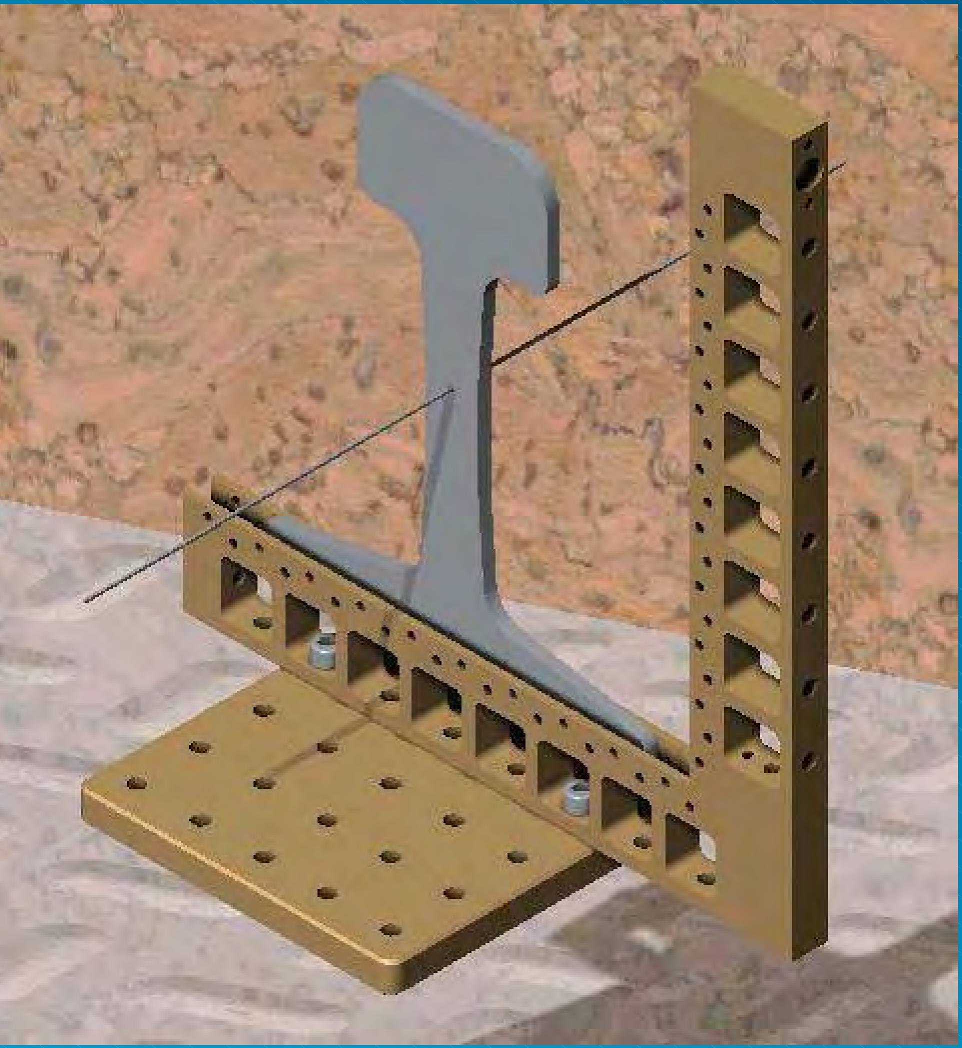


3 perpendicular components of stress x=longtitudinal y=transverse z=perpendicular

FaME38, Facility for Materials Engineering

Neil James, (Plymouth), P.J.Webster, Axel Steuwer, Mat. Peel, Darren Hughes,
Benoît Malard, Nicolas Ratel, Zhaohui Chen, Thanh Giang, Husin Sitepu

Sample Positioning and Alignment



Multi-zone furnace, other furnaces, ovens

ISO/VAMAS positioning plate
± 100 microns and ± 0.10° routinely
± 10 microns and ± 0.02° with care

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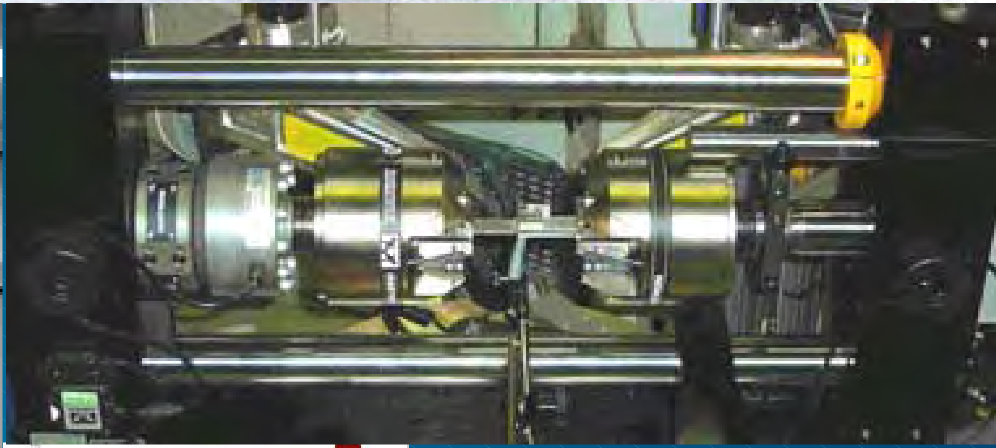
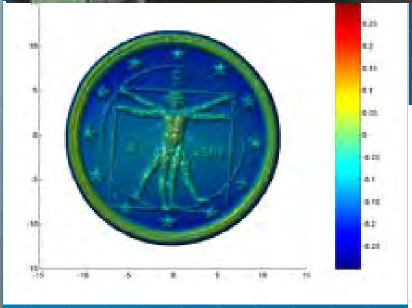
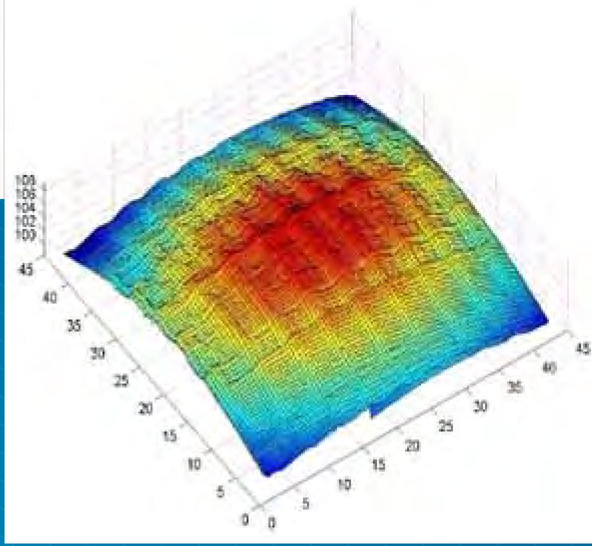
Sample characterisation & environment



50KN hydraulic tension/compression machine

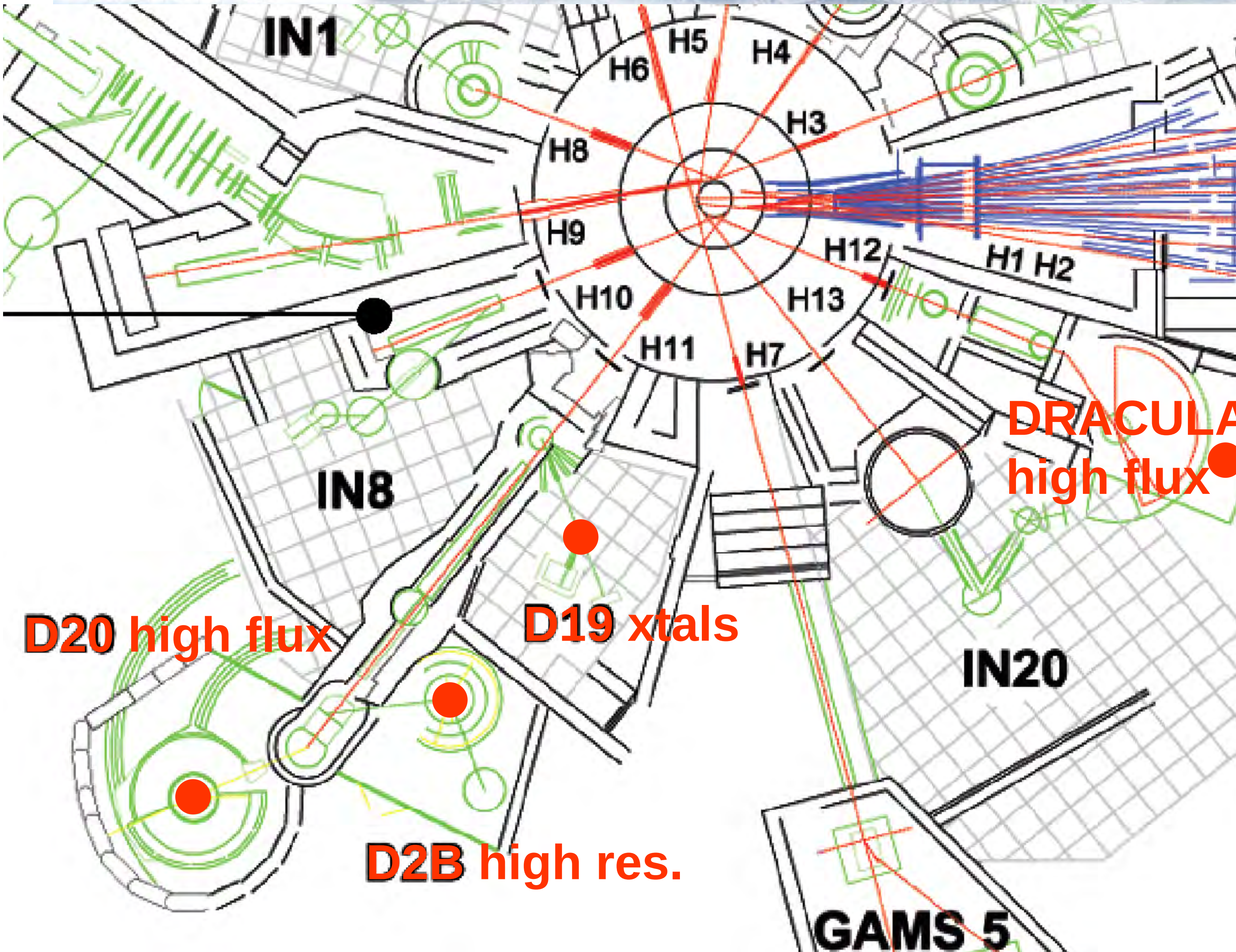


Coordinate Measuring Machine (left)

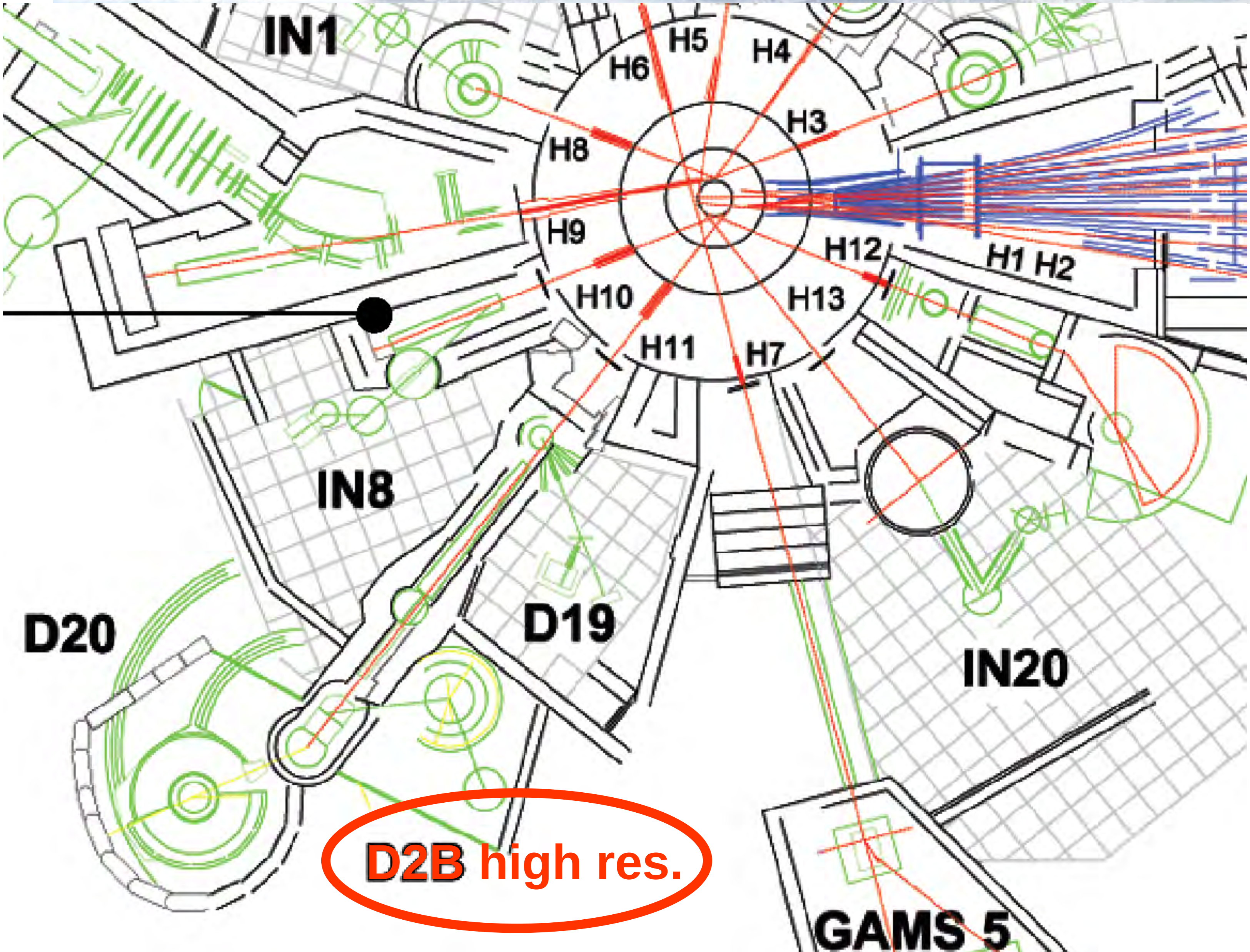


CMM surface scans of a Euro coin and of a laser shock peened plate (above)

D2B,D19,D20,DRACULA
High Flux-High Resolution Diffractometers



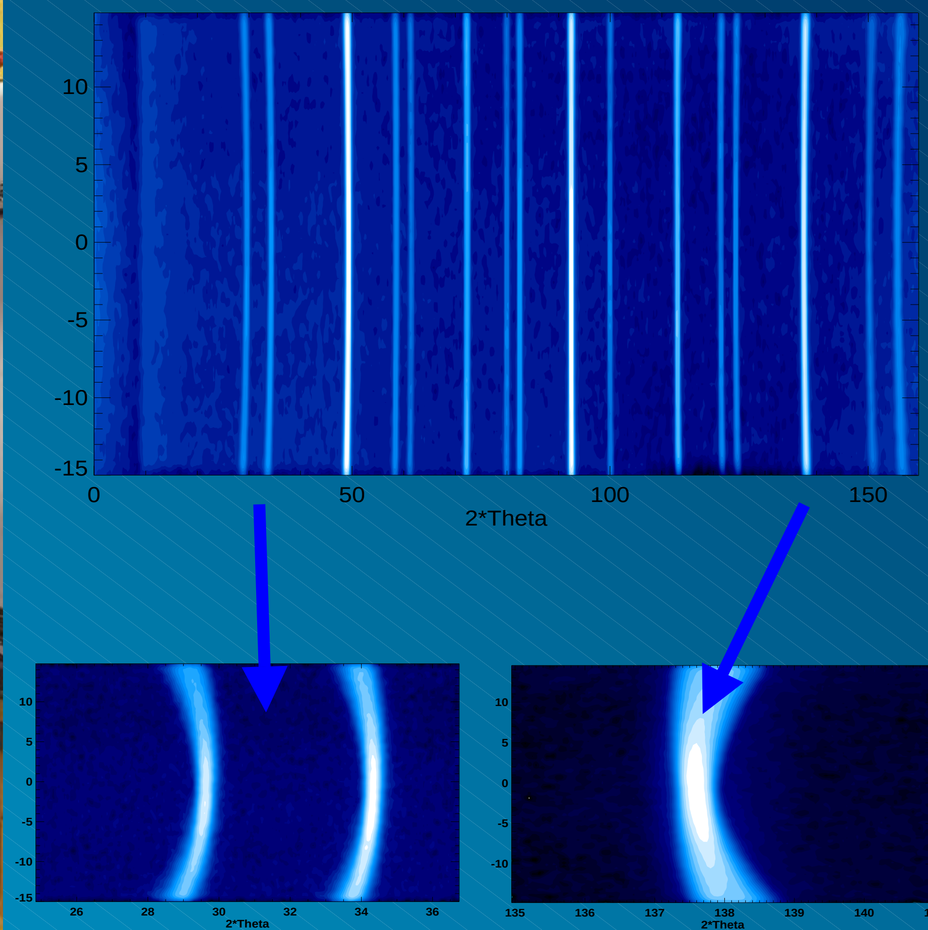
D2B,D19,D20,DRACULA
High Flux-High Resolution Diffractometers



D2B High Resolution Powder Diffractometer

J.P.Attfield, P.D.Battle, J.L.Finney, C.Greaves, K.Prassides,
S.A.T.Redfern, B.Raveau, A.W.Hewat, E.Suard, C.Ritter

UK-EPSRC Super-D2B project at ILL



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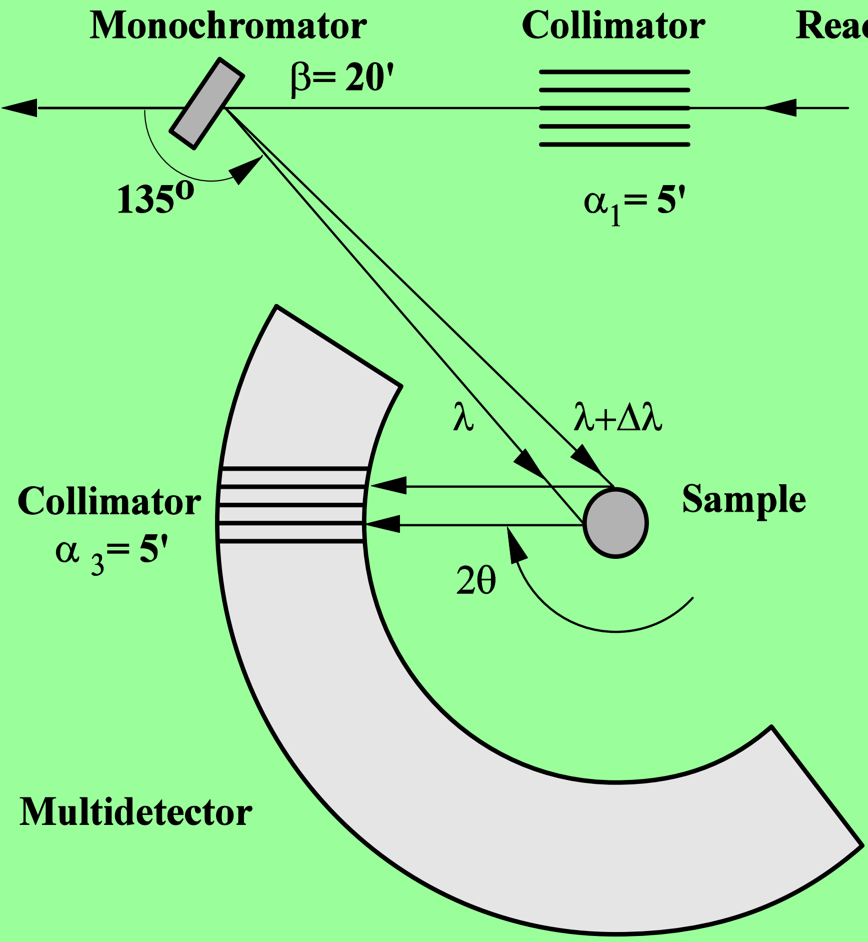

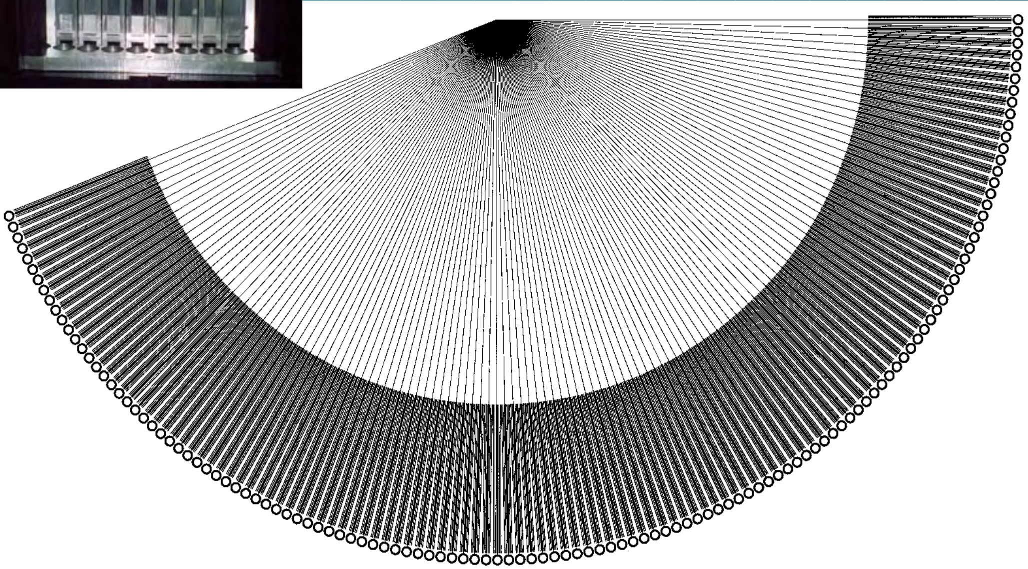

Array of 128 Collimators & Linear-Wire PSD Detectors

D2B High Resolution Powder Diffractometer

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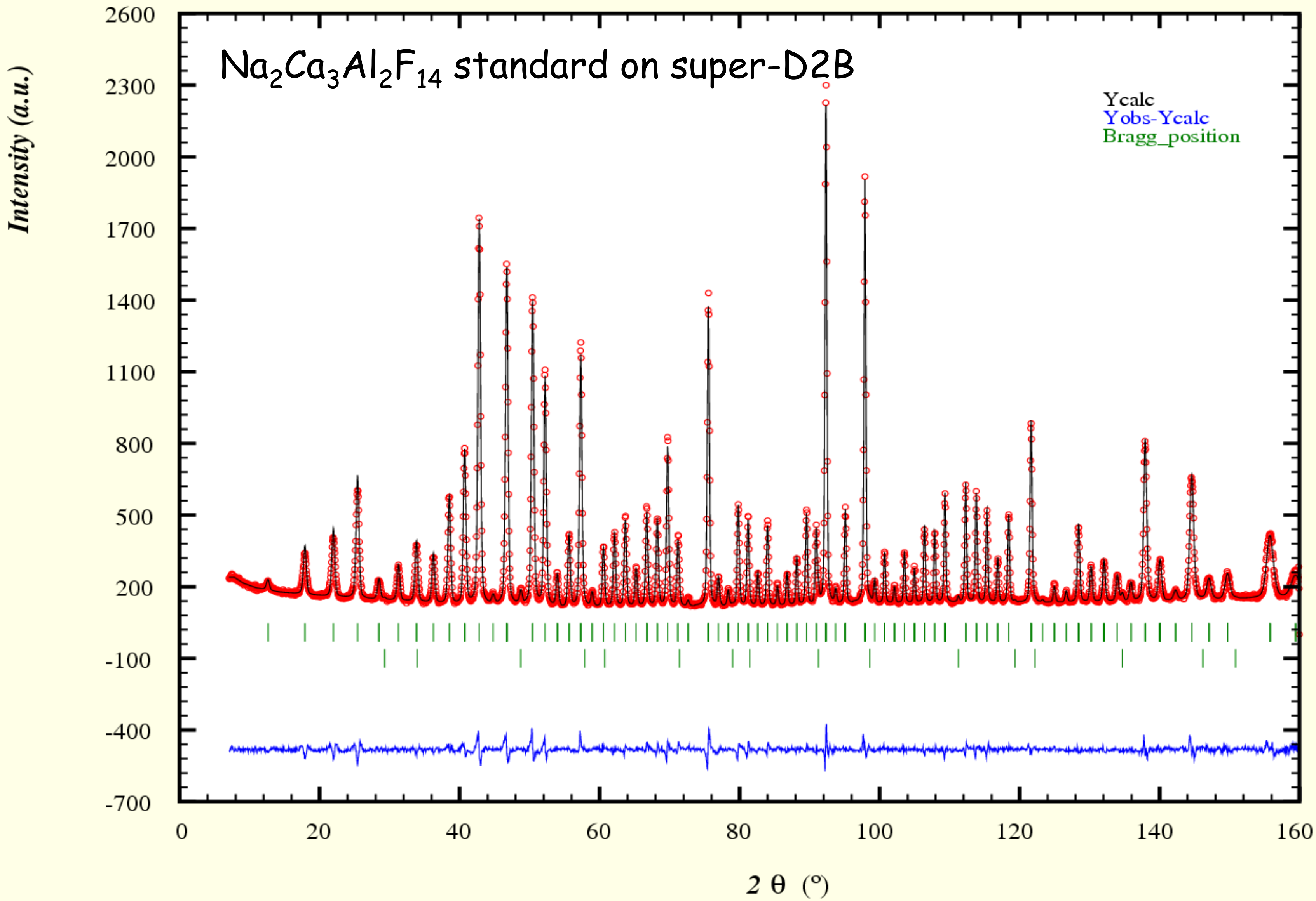

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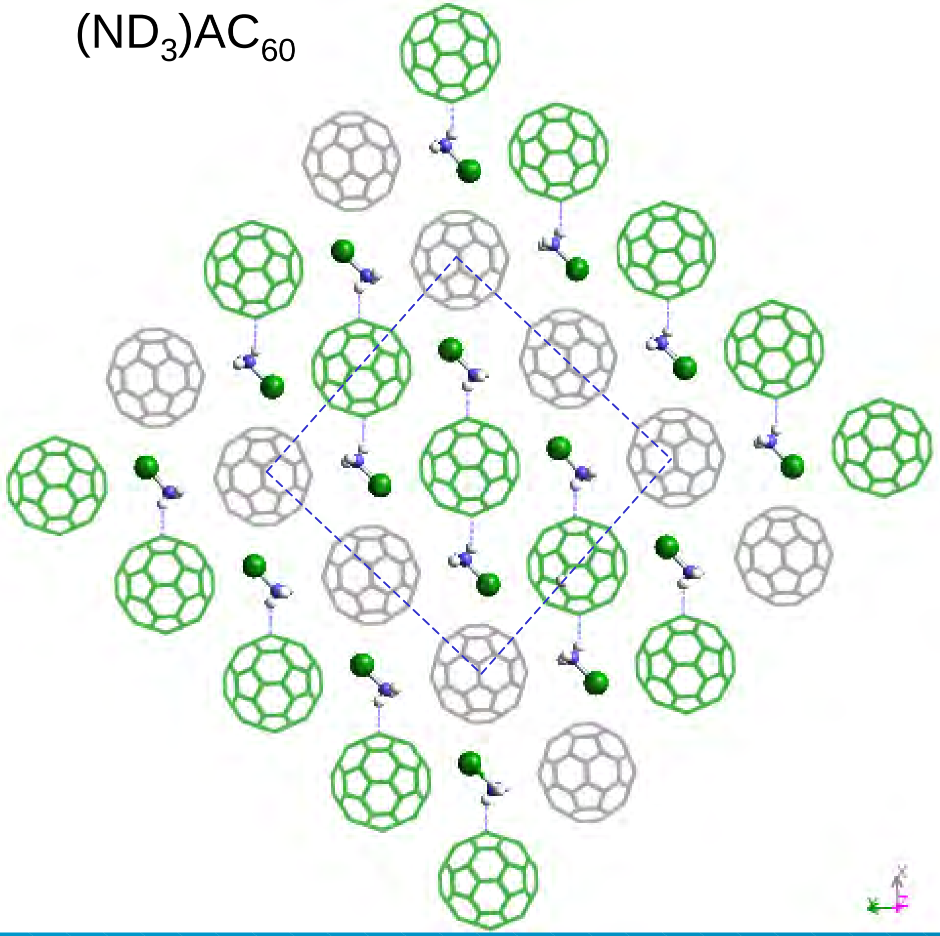
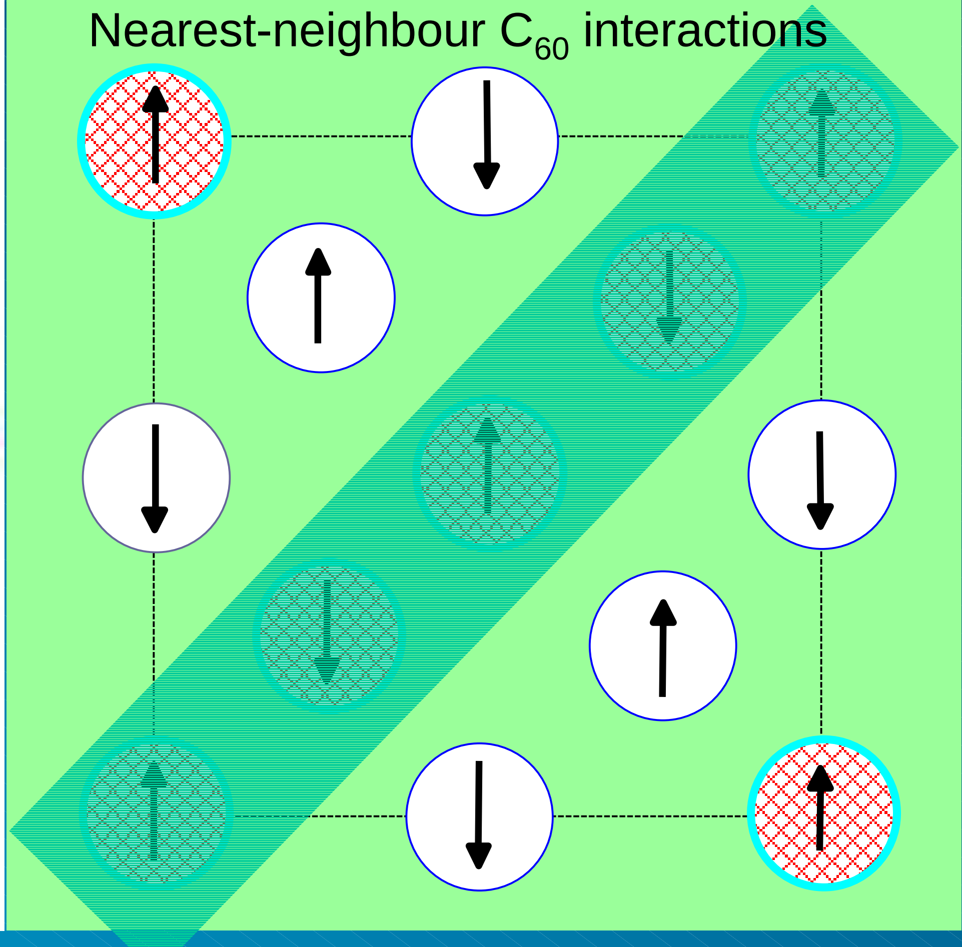
Fullerides & Framework Materials - S.Margadonna (Edinburgh)





Network of ND•••p(6:6) interactions - orthorhombic [110] plane
Influence of ND_3-C_{60} close contacts on magnetic exchange

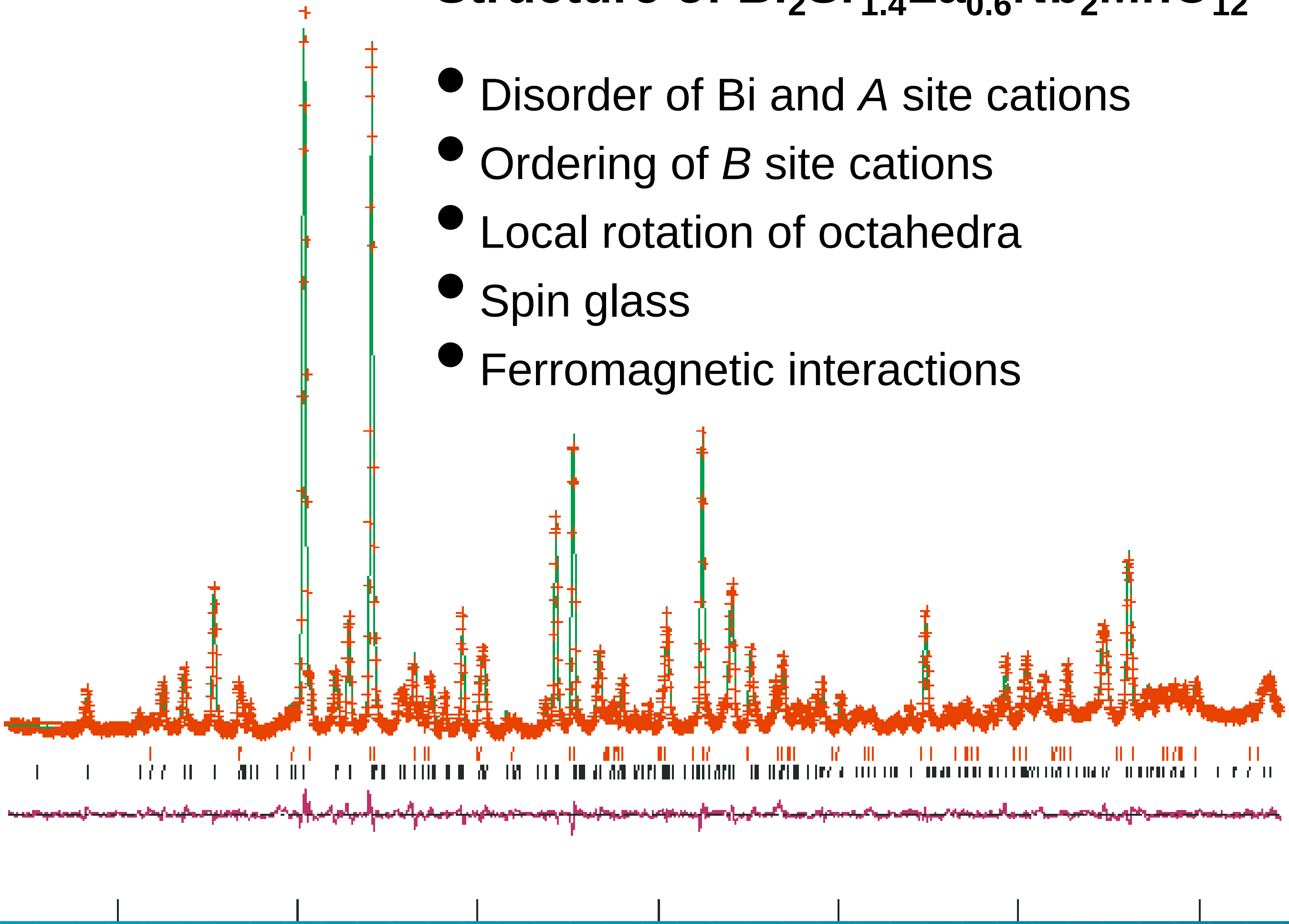
Strong effect on *nn* AF interactions along ferrorotationally ordered C_{60} stripes

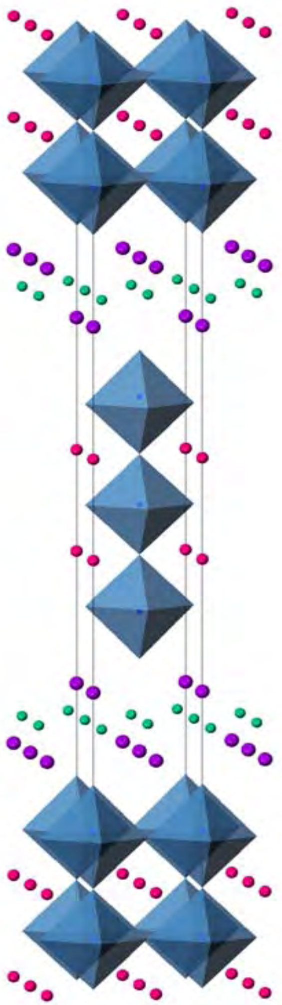
J.P.Attfield, P.D.Battle, J.L.Finney, C.Greaves, K.Prassides,
S.A.T.Redfern, B.Raveau, A.W.Hewat, E.Suard, C.Ritter

Aurivillius Phases - E. McCabe (Birmingham)

Structure of $Bi_2Sr_{1.4}La_{0.6}Nb_2MnO_{12}$

- Disorder of Bi and *A* site cations
- Ordering of *B* site cations
- Local rotation of octahedra
- Spin glass
- Ferromagnetic interactions

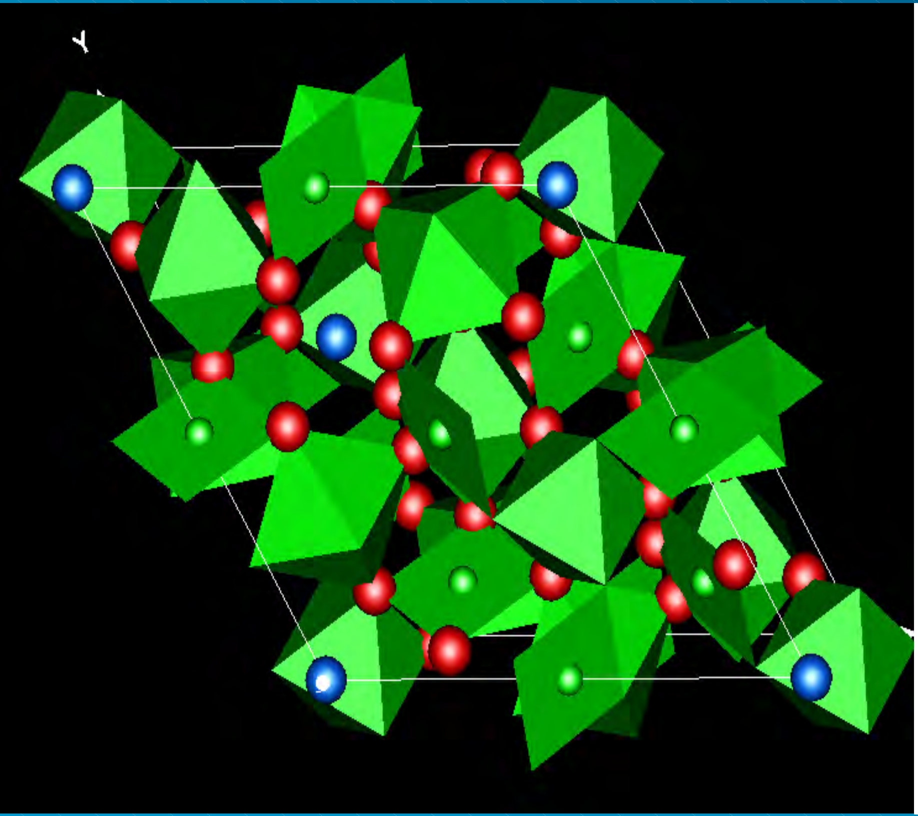


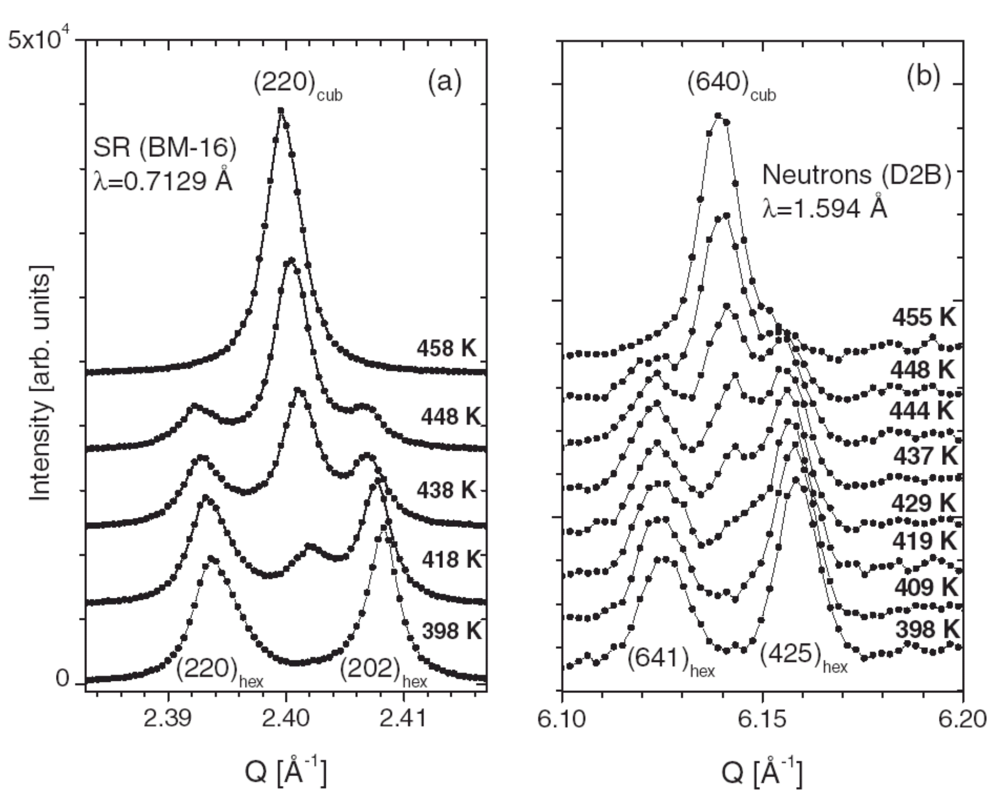


Phase co-existence & charge ordering transition in $CaMn_7O_{12}$

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

J. Phys. Condens. Matter **14**, 5747-5753





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

D2B High Resolution Powder Diffractometer

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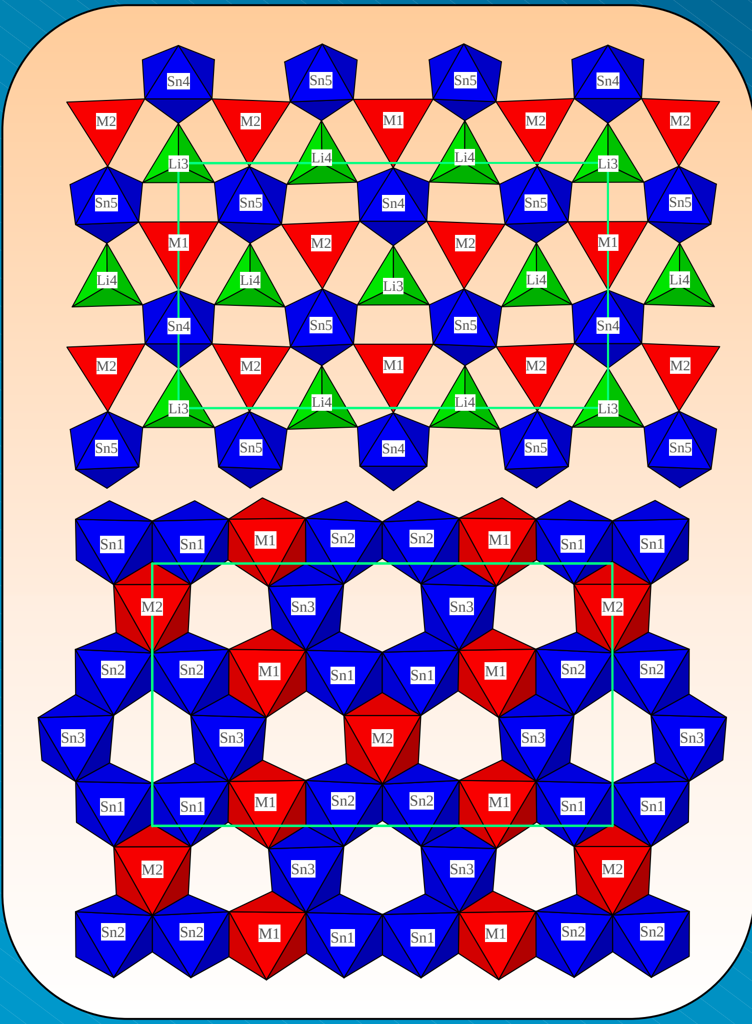
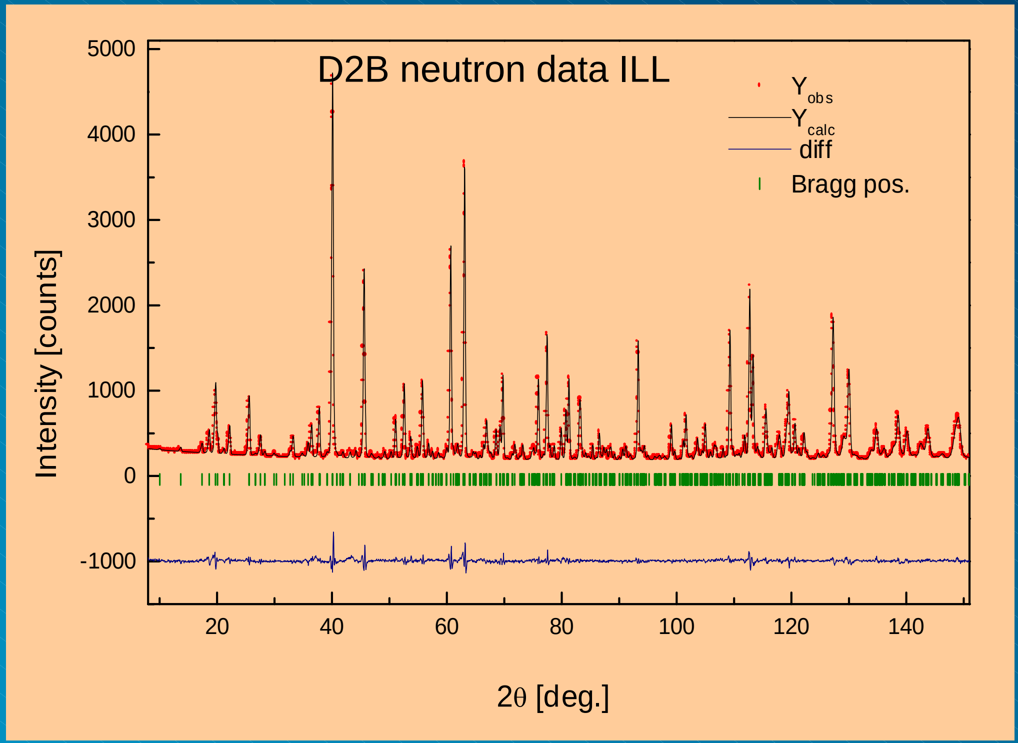
S.A.T.Redfern, B.Raveau, A.W.Hewat, E.Suard, C.Ritter

Cation distribution in $Li_2M(II)Sn_3O_8$, M(II)=Mg, Co, Fe.

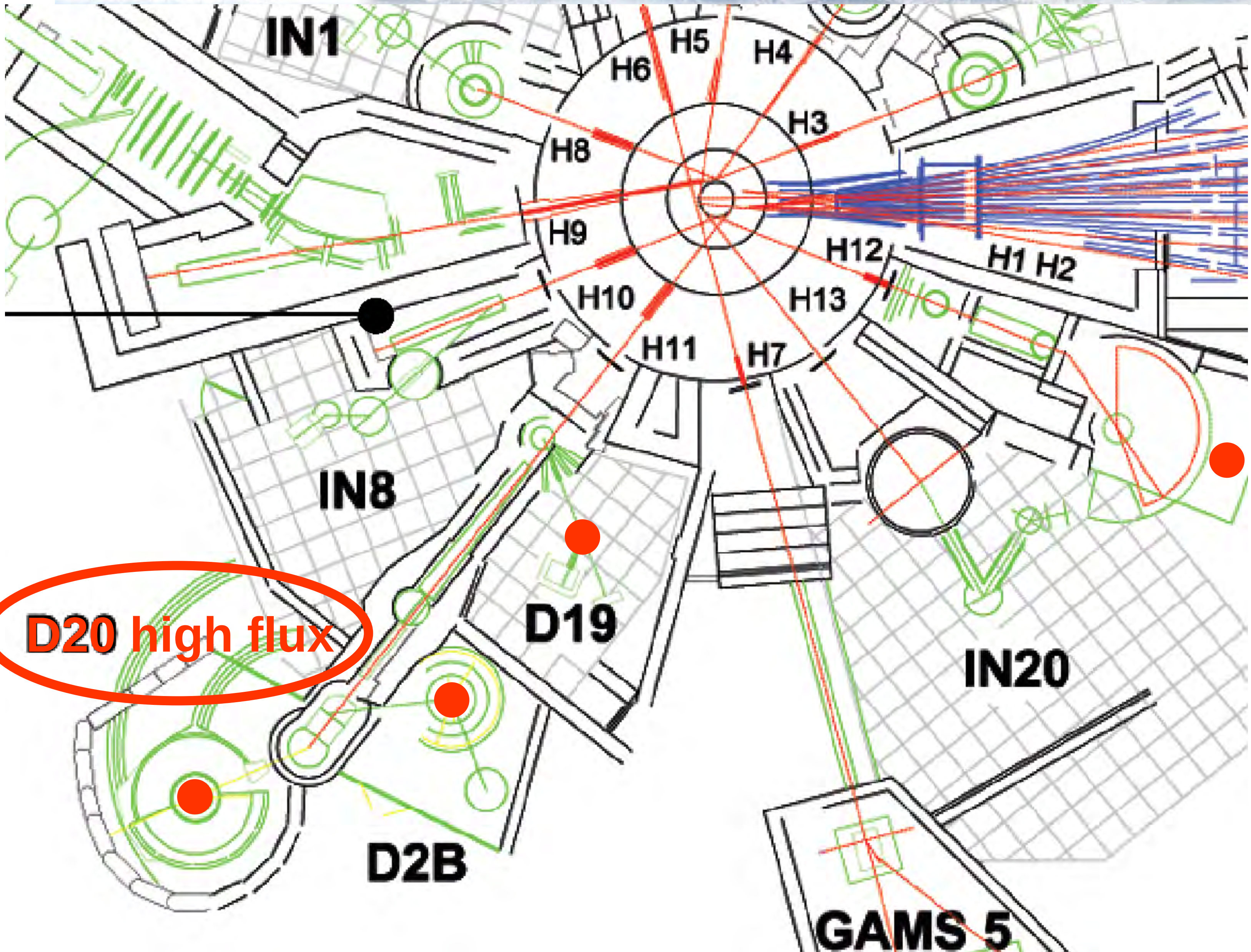
T. Trendafilova, D. Kovacheva, K. Petrov & A. Hewat (2004) EPDIC-IX, Prague.



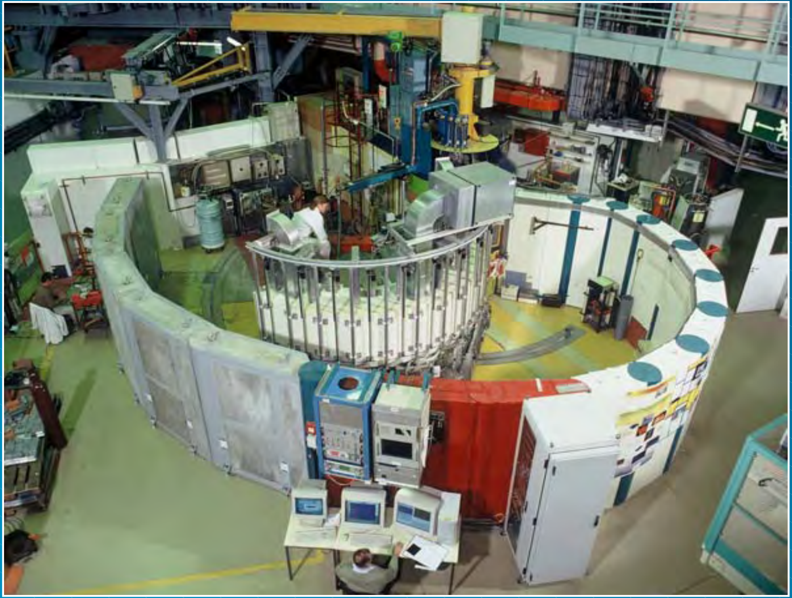
- Complex oxide structures containing lithium were solved using both ILL-ESRF neutron and synchrotron data

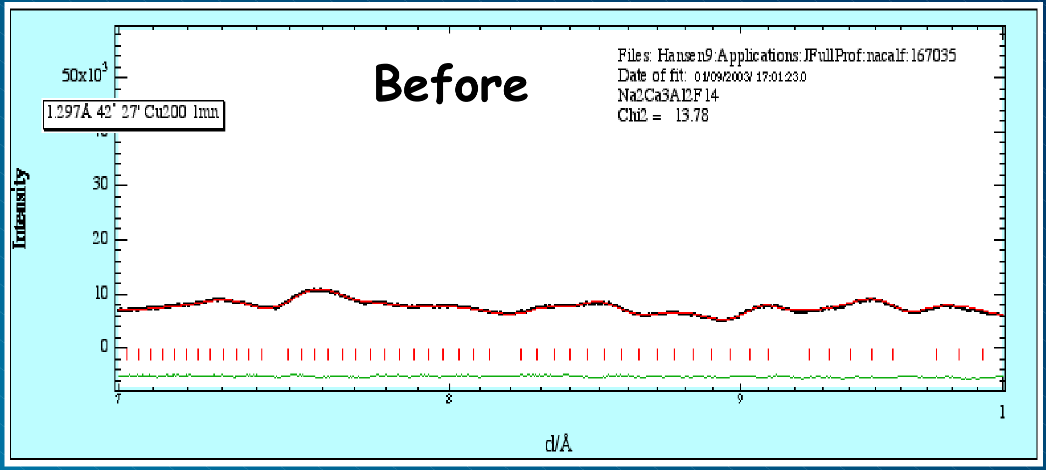


D2B,D19,D20,DRACULA
High Flux-High Resolution Diffractometers

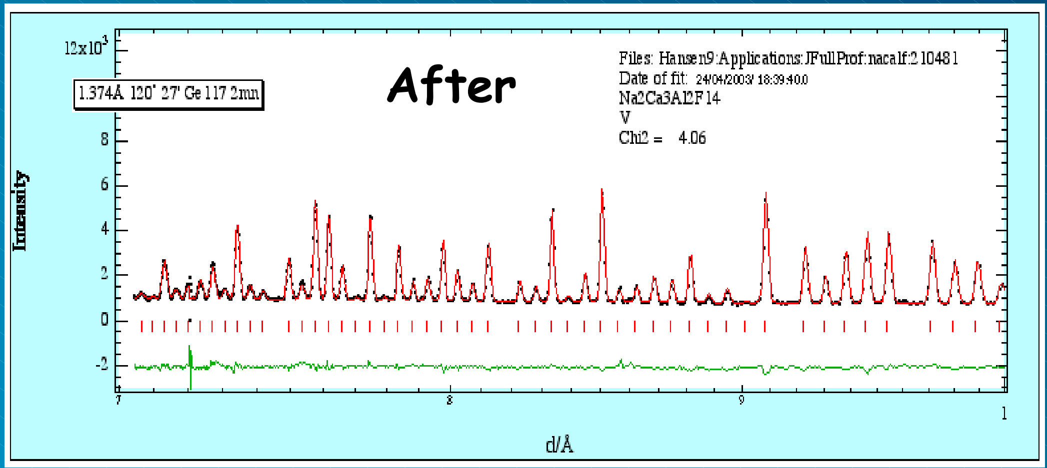


D20 High flux compatible & Good Resolution
High take-off option on D20





Before and After (data in 2 min.)



Higher D20 resolution since 2003

Applications - fast detectors, small samples

Eric Kisi et al. (ICNS-2005 Sydney)

Very fast chemical and electrochemical kinetics

(Ti,Si,C) reaction -> Ti_3SiC_2

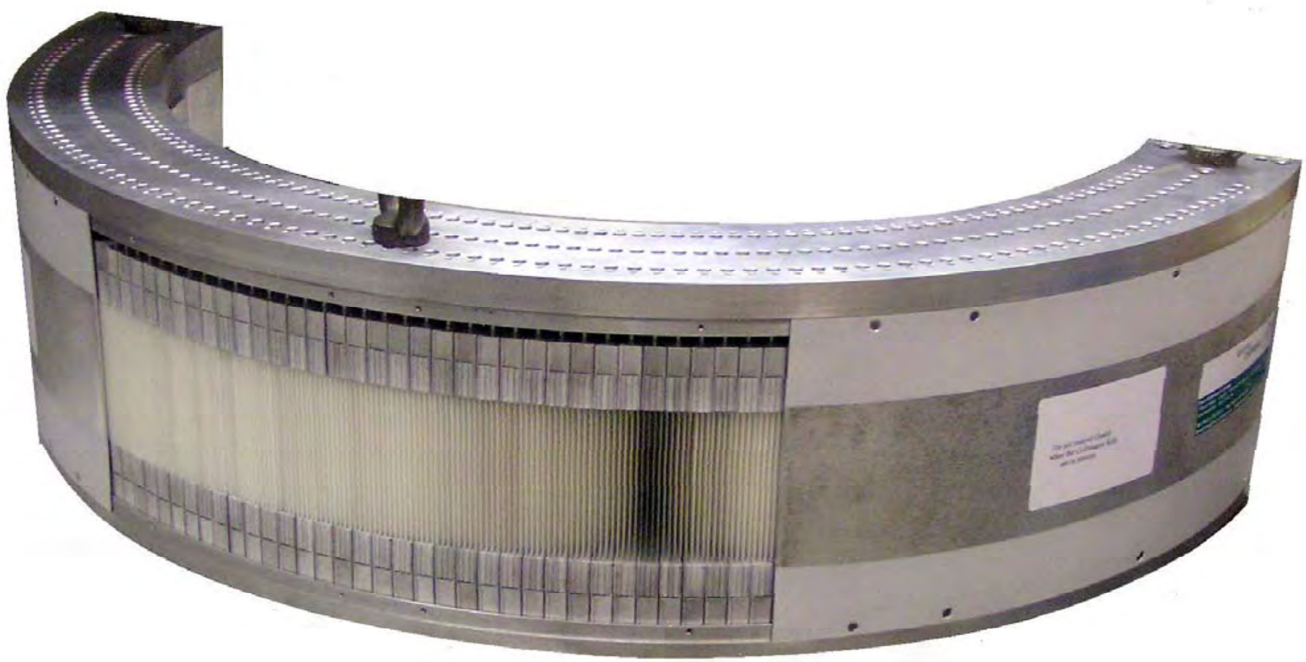
University of Newcastle, Australia

- 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)

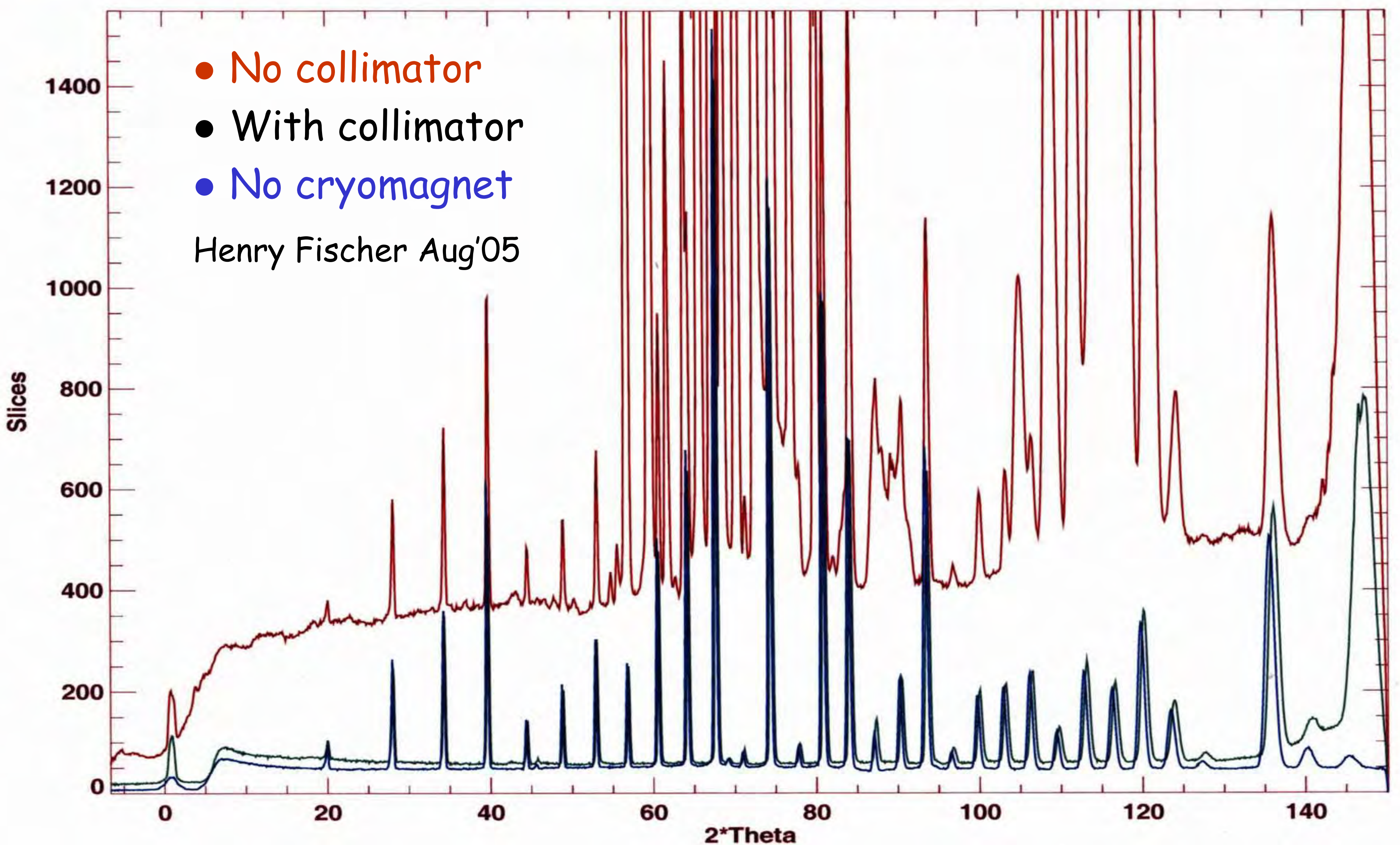
Sample Environment
Radial Collimator, Cryomagnet & Pressure

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



Sample Environment
Radial Collimator & Cryomagnets on D20

Radial Collimator = 95% transmission, 3% background



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	1.0

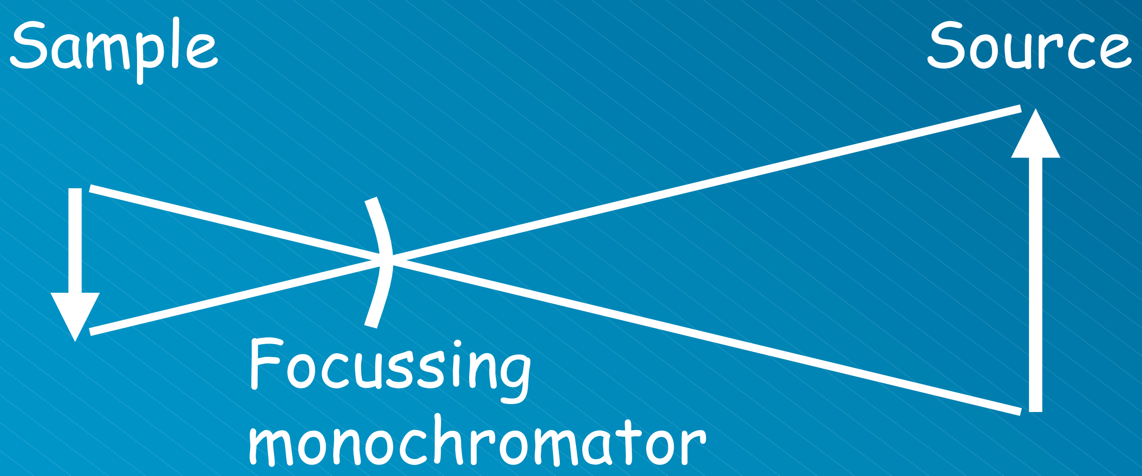
The CW and TOF machines have similar efficiencies

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

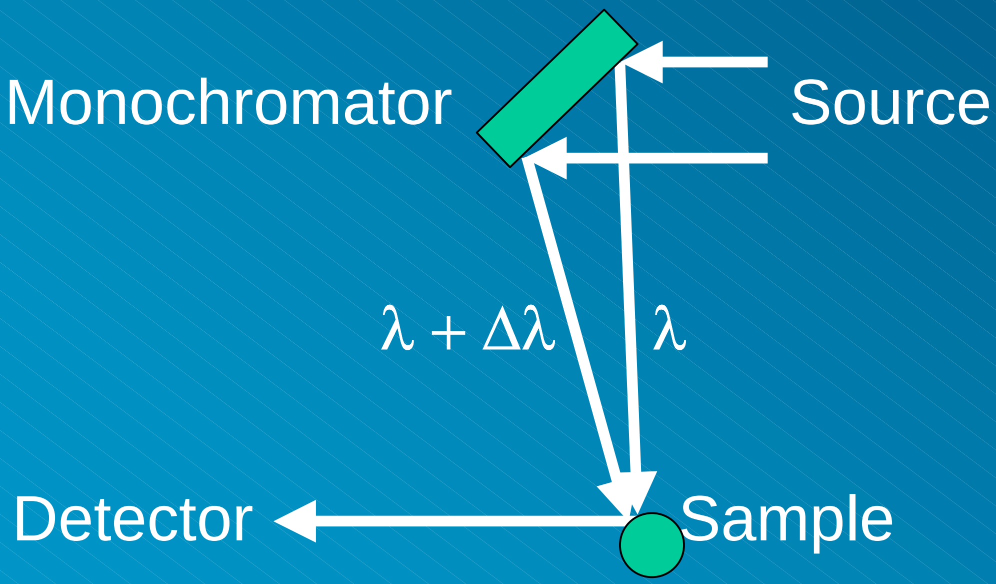


cf use of convergent guide with TOF

Why is sample flux so high from a reactor?

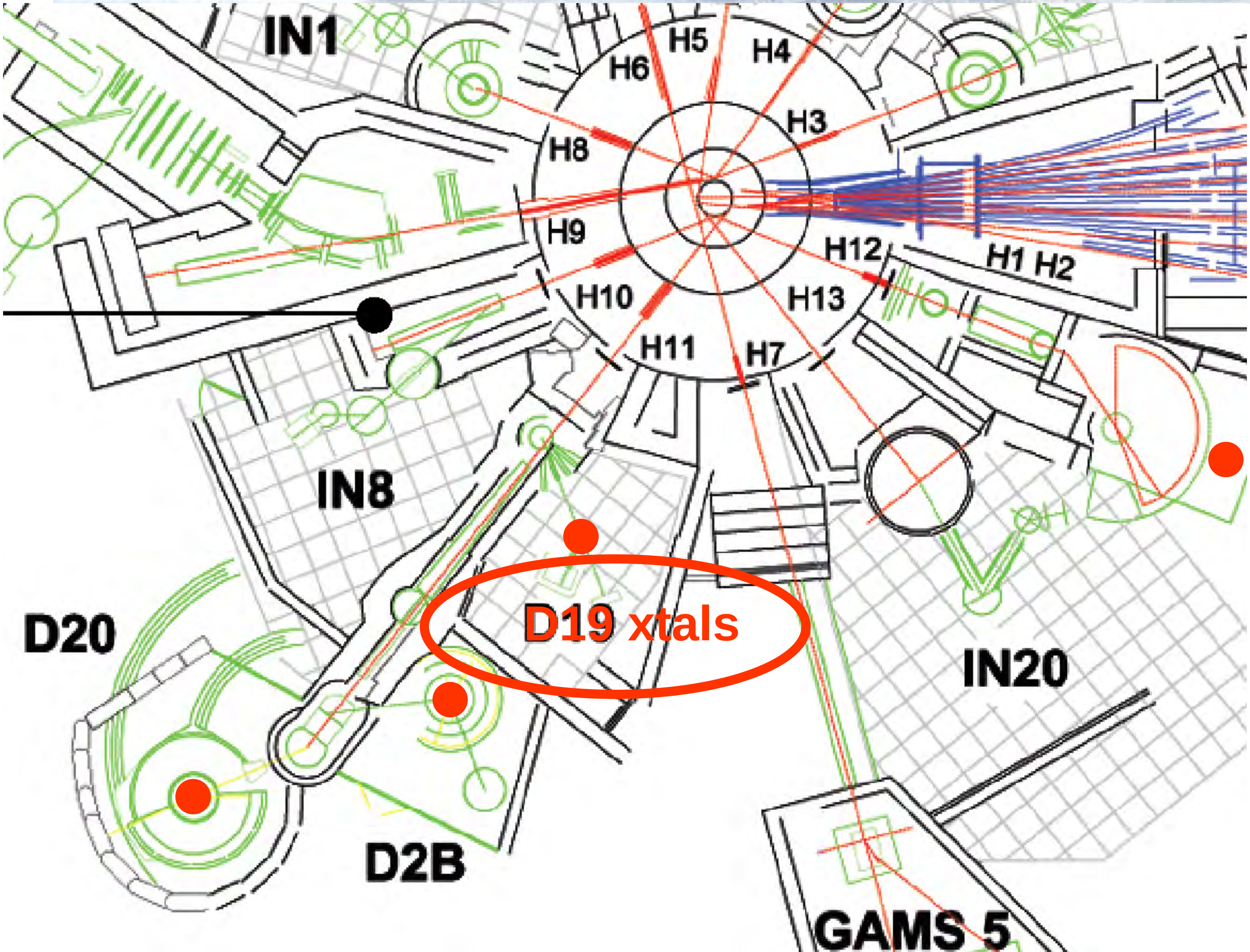
A: Large wavelength-band focusing monochromators ?

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



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

D2B, D19, D20, DRACULA
High Flux-High Resolution Diffractometers



The Future - Bigger Detectors

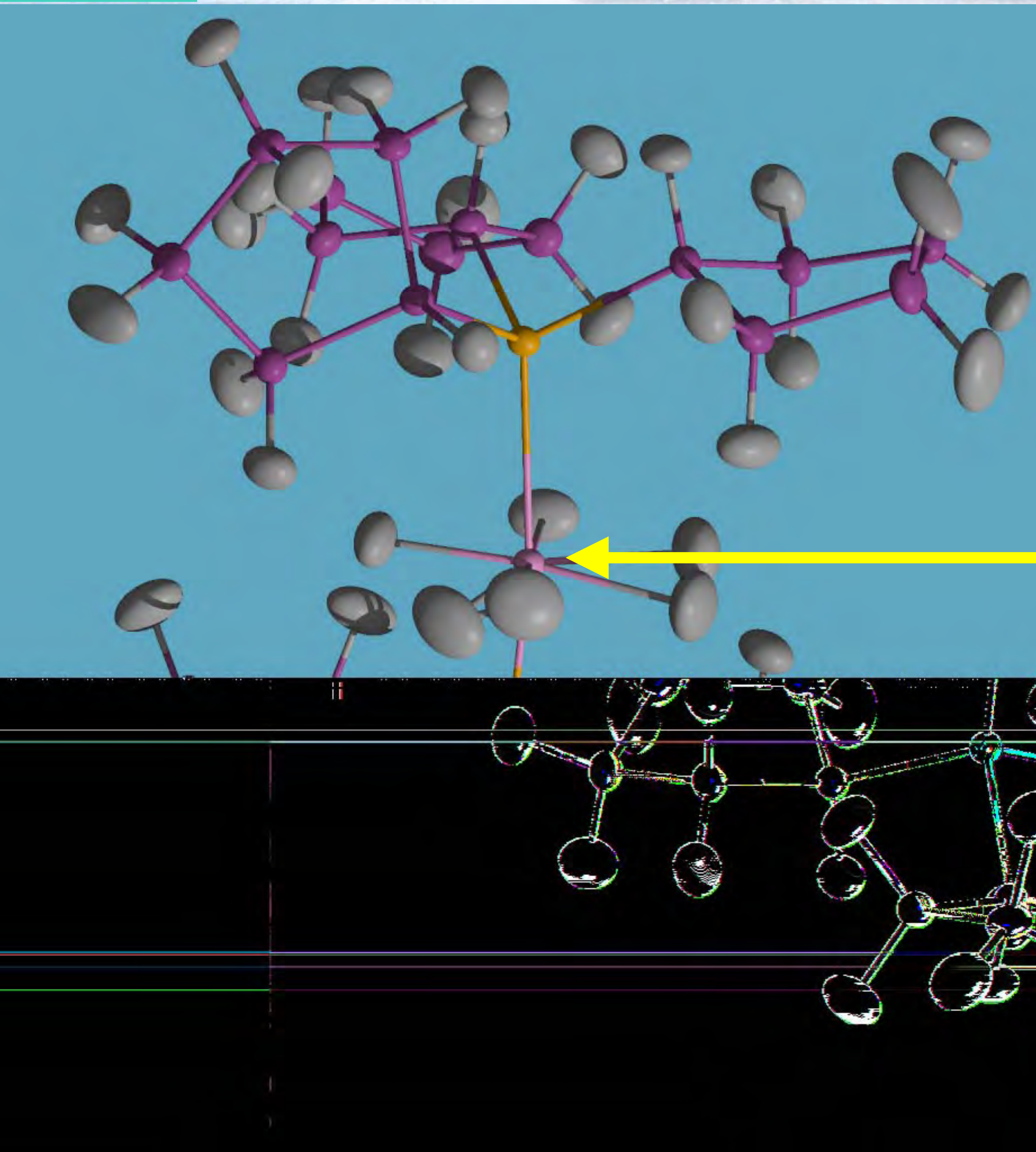
New D19

The Future for ILL

DRACULA, D9, D16

Organometallic Catalysts on D19

Albinati, Chaudret, Mason et al (2004)



Transition metal hydrides

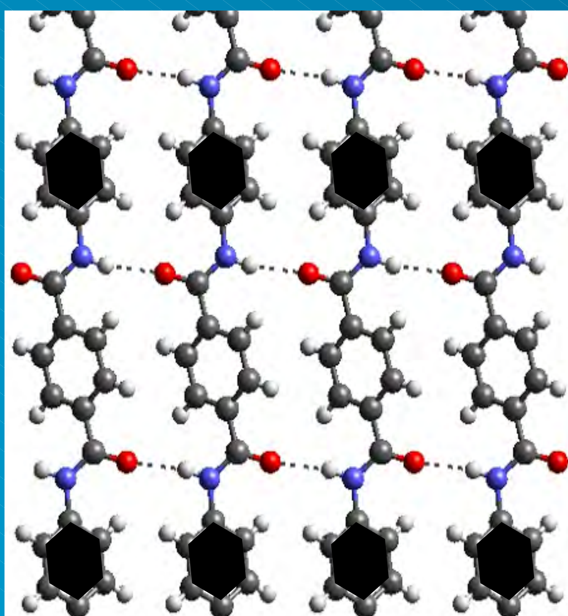
- Organometallic catalysts
- $RuH_2(H_2)_2((PCyp)_3)_2$
- NMR, X-rays already done
  but
- Need Ru-H coordination

Geometry of a key Ru precursor complex for hydrogenation catalysis as a function of T

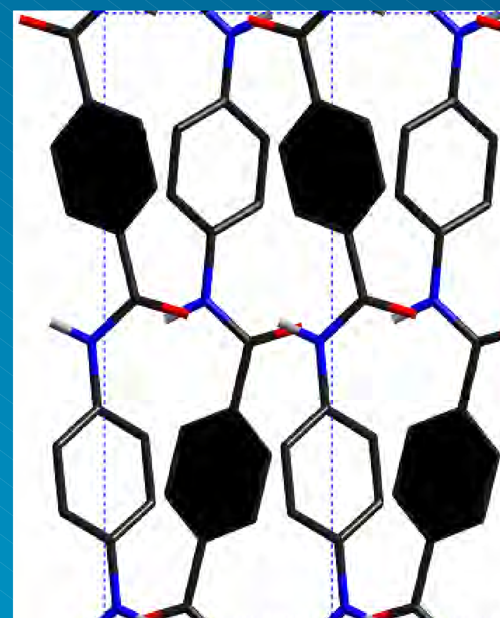
PPTA: poly(p-phenylene terephthalamide) KEVLAR

New insights into the structure of poly(p-phenylene terephthalamide) from neutron fiber diffraction studies.

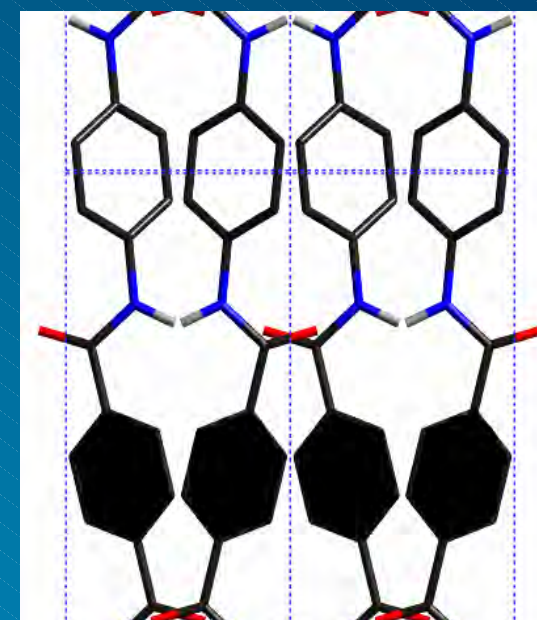
Gardner K H, English A, Forsyth V T, (2004) Macromolecules



How do H-bonded sheets stack along the c-axis ?

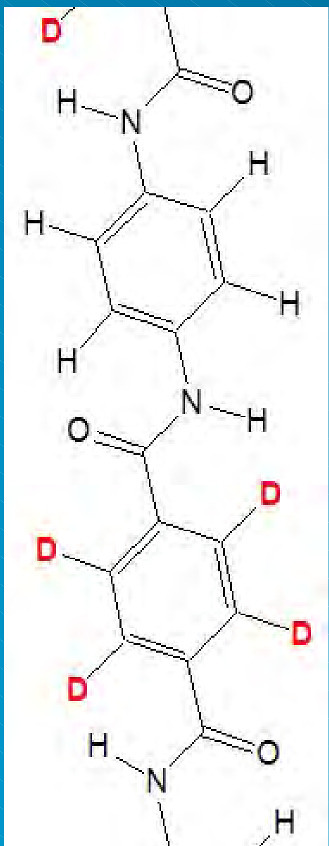


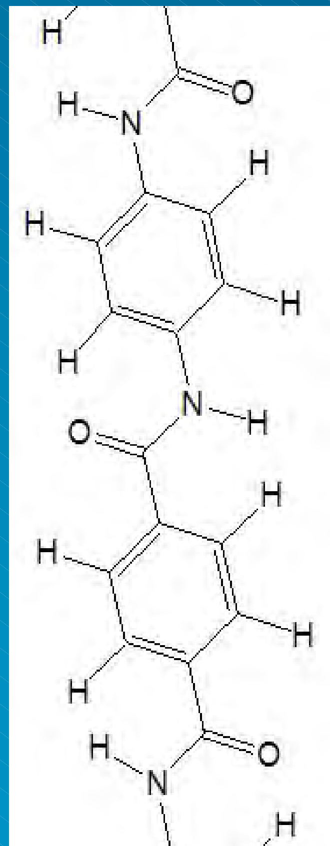
Models since 1970s

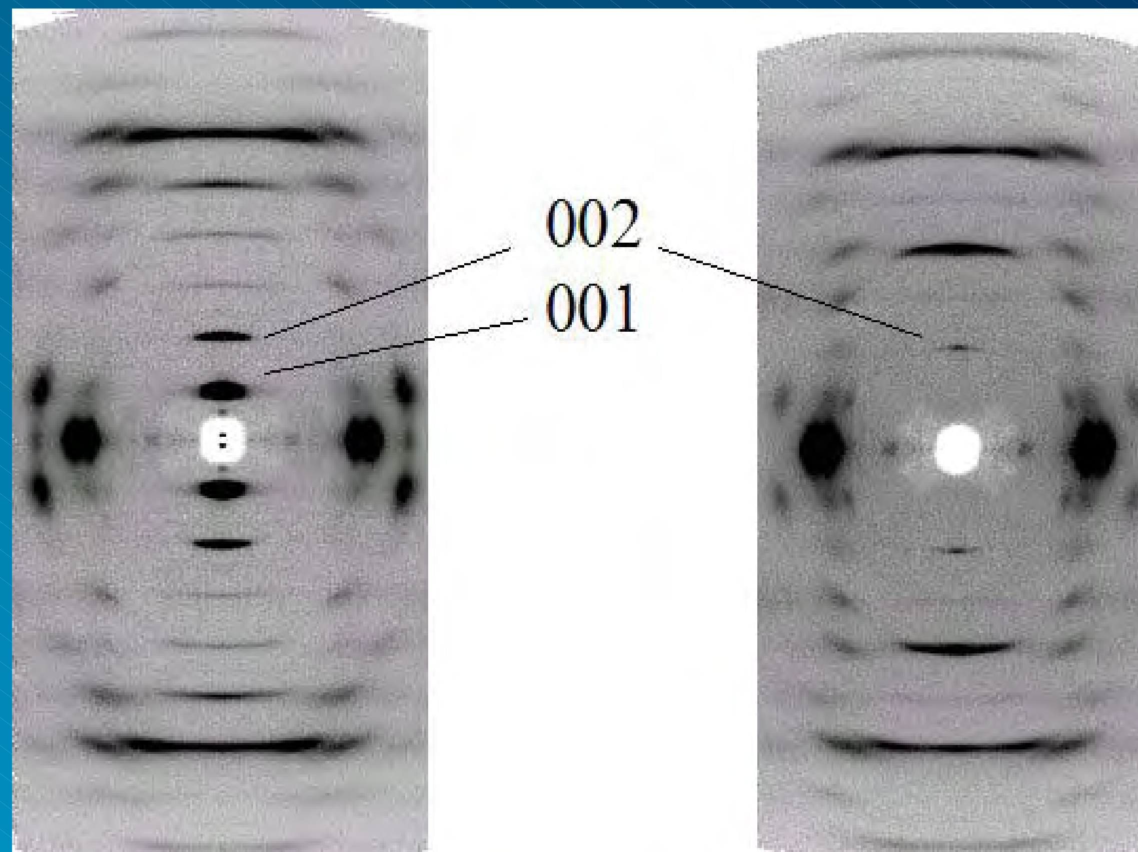


Correct model

PPTA: poly(p-phenylene terephthalamide) KEVLAR





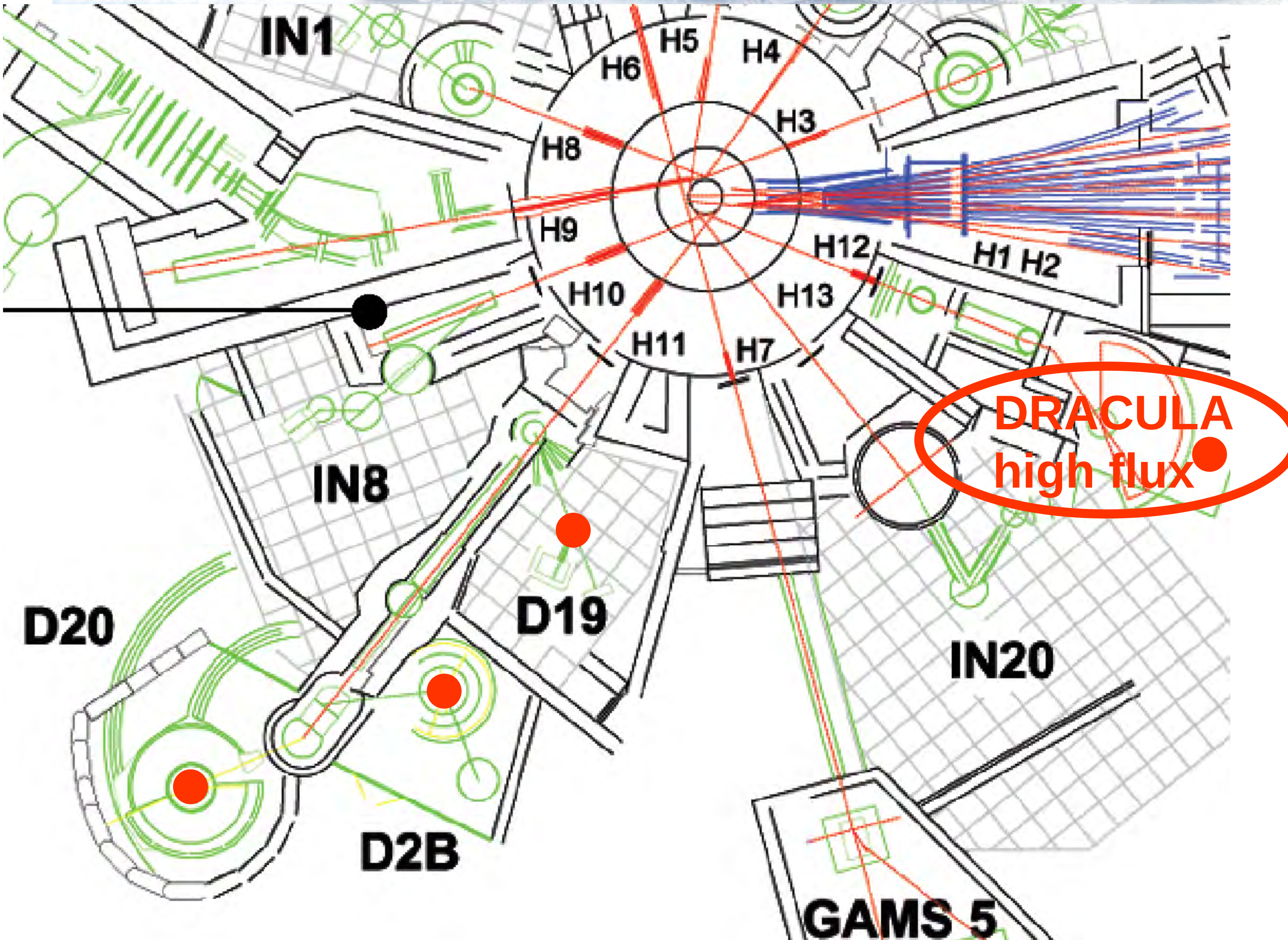


Two rings, similar scattering
Deuterate one ring to label it

Selectively deuterated

Fully hydrogenated

D2B,D19,D20,DRACULA
High Flux-High Resolution Diffractometers

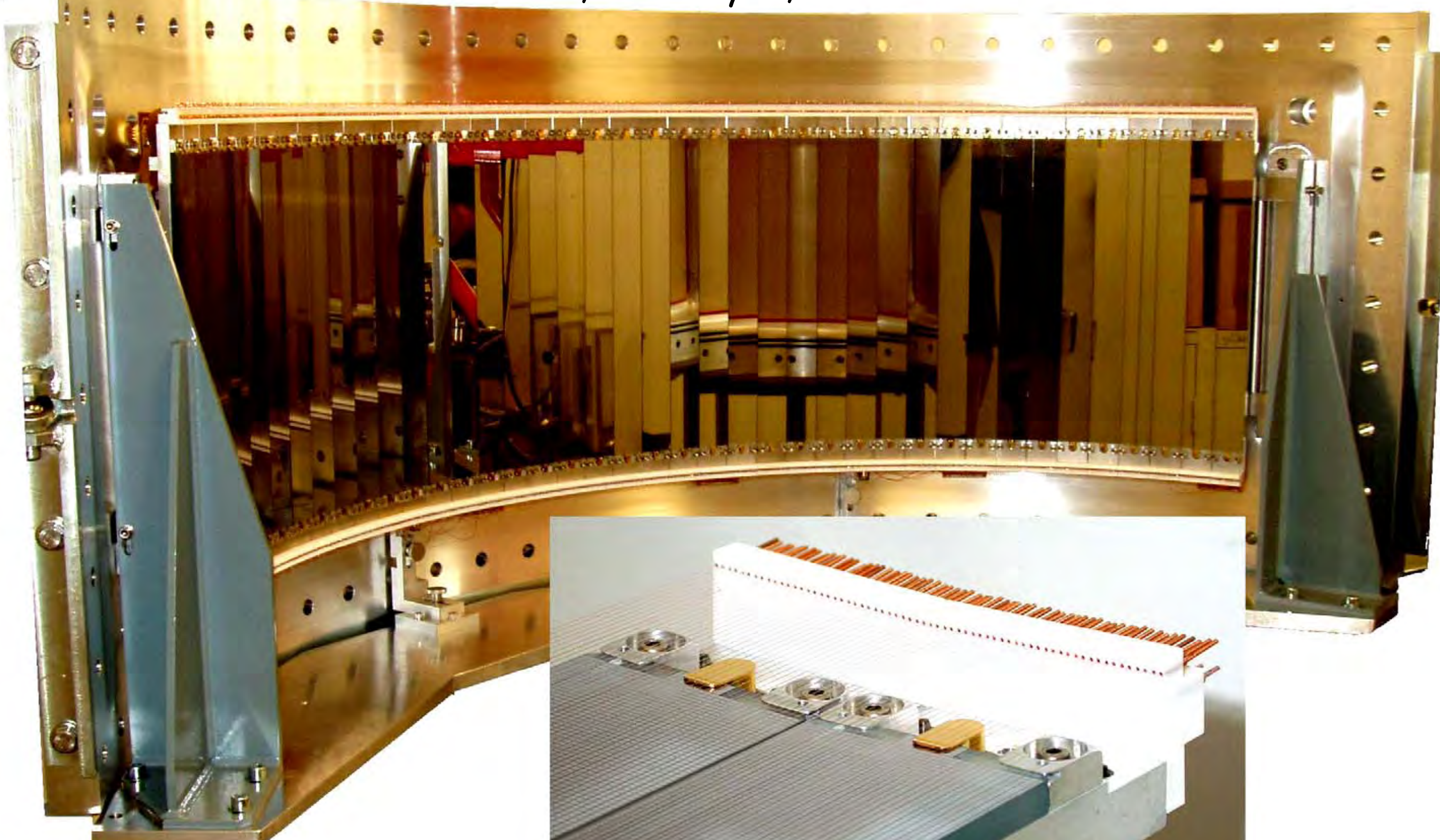


Dracula Ultra Large Area 2D Detector

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

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

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



Comparison of TOF & CW Diffractometers

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

	D20	GEM	DRACULA	SNS
Flux average on sample	5×10^{7}	$\sim 2 \times 10^{6}$	$\sim 10^{8}$	$\sim 2.5 \times 10^{7}$
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 $30^{o} \times 160^{o}$

TOF machines already have big detectors

With big detectors ILL can compete with SNS intensity

New Science on Dracula
Diffractometer for Rapid ACquistion

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

Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focusing)
- 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 (focusing)

Focusing neutrons to study small samples

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

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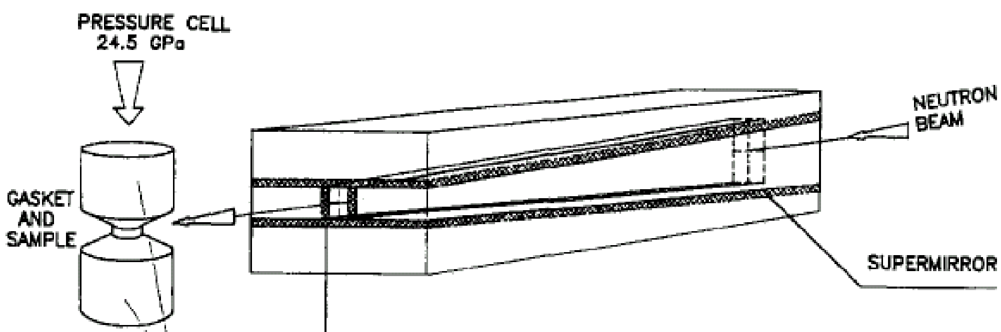
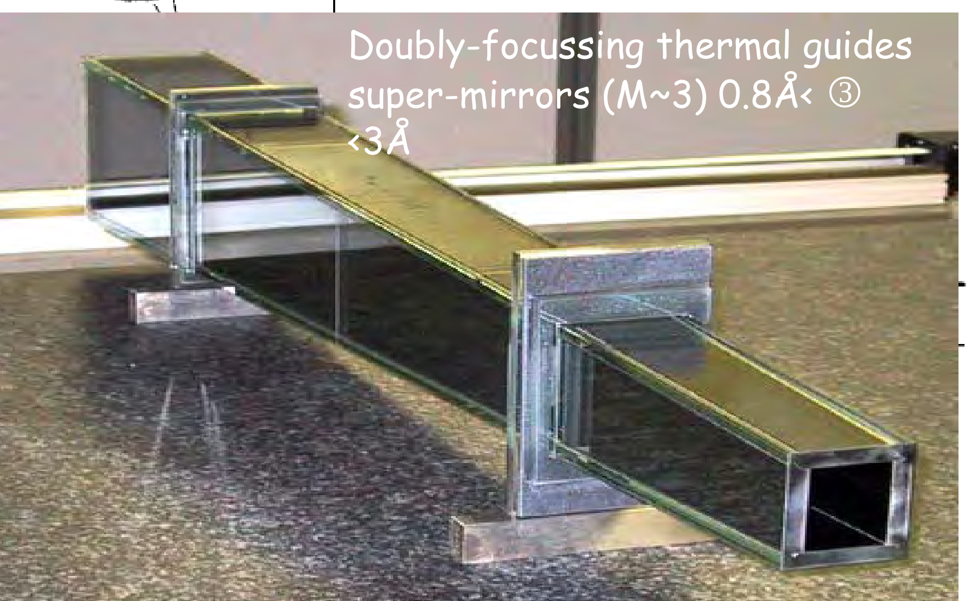
[b]*Russian Research Center Kurchatov Institute, 123182 Moscow, Russian Federation*
[c]*Ecole Centrale de Nantes, 1 rue de la Noé, 44000 Nantes, France*
[d]*Laboratory for Neutron Scattering ETH&PSI, CH-5232 Villigen PSI, Switzerland*





Kirkpatrick–Baez microfocusing optics for thermal neutrons

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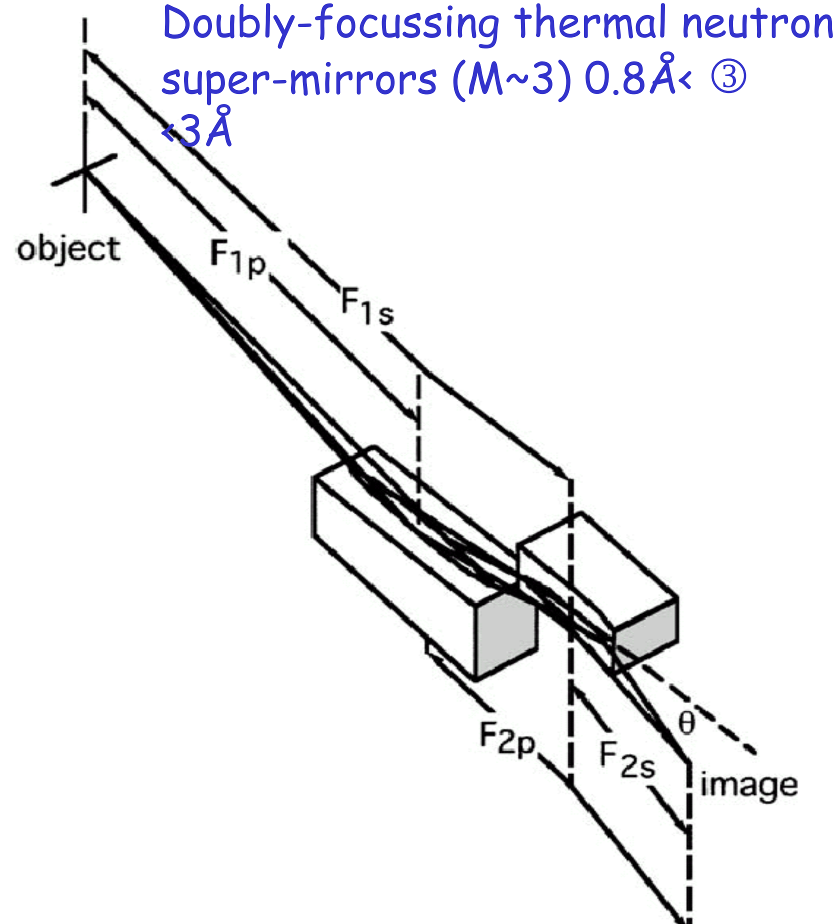
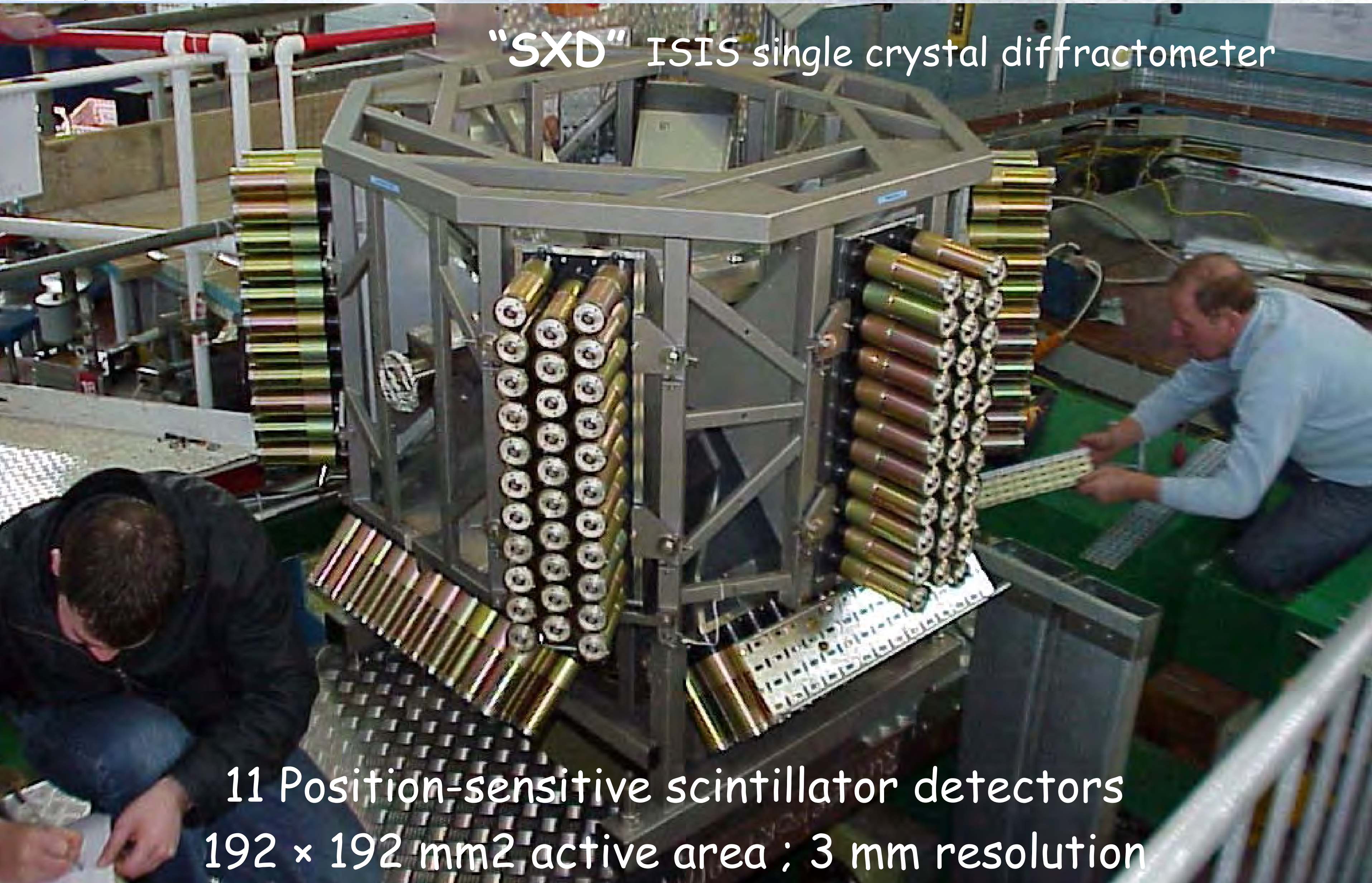
Doubly-focussing thermal neutron super-mirrors (M~3) 0.8Å‹ ③ ‹3Å



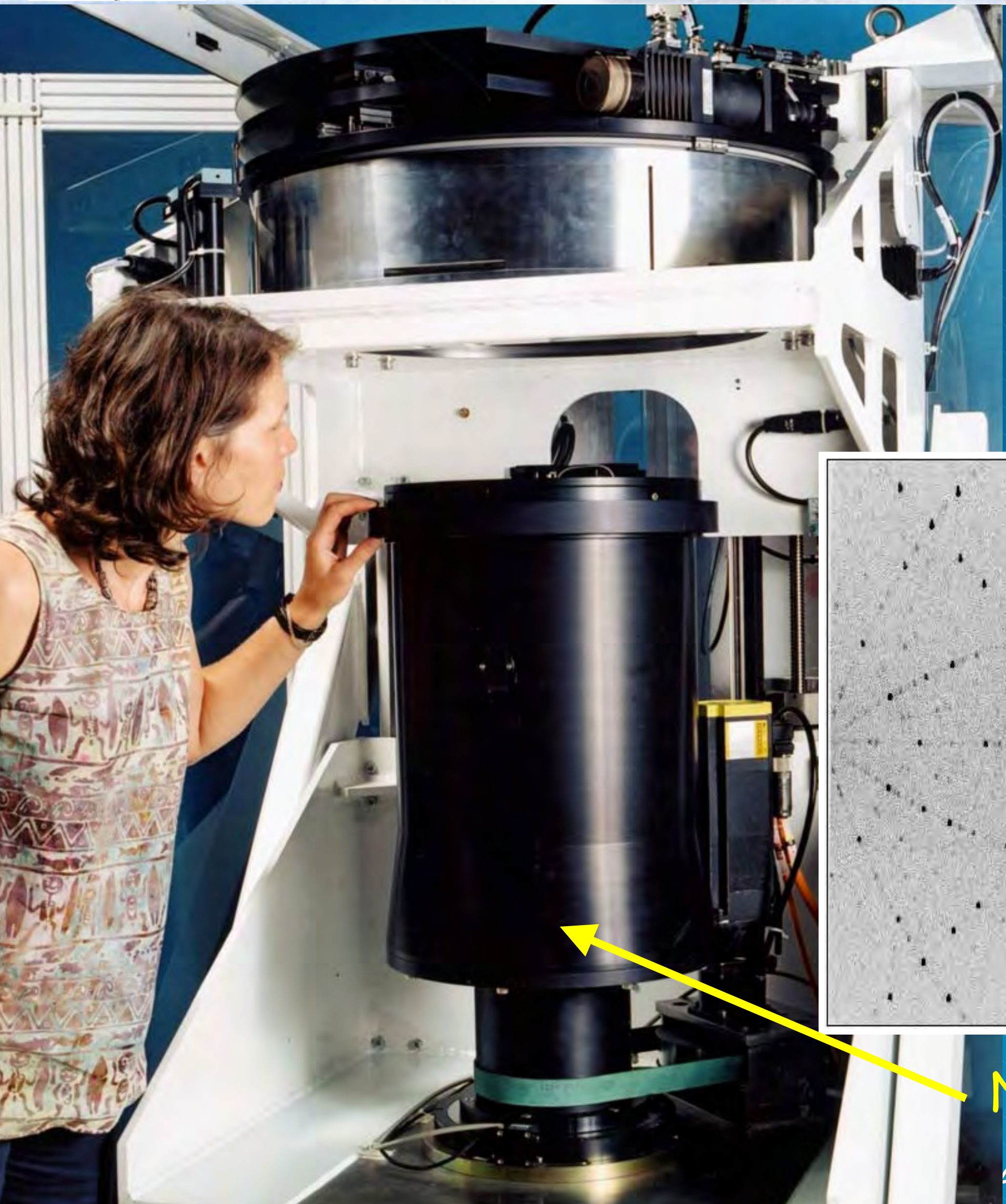
Next Generation Neutron Diffractometers

A very large area detector



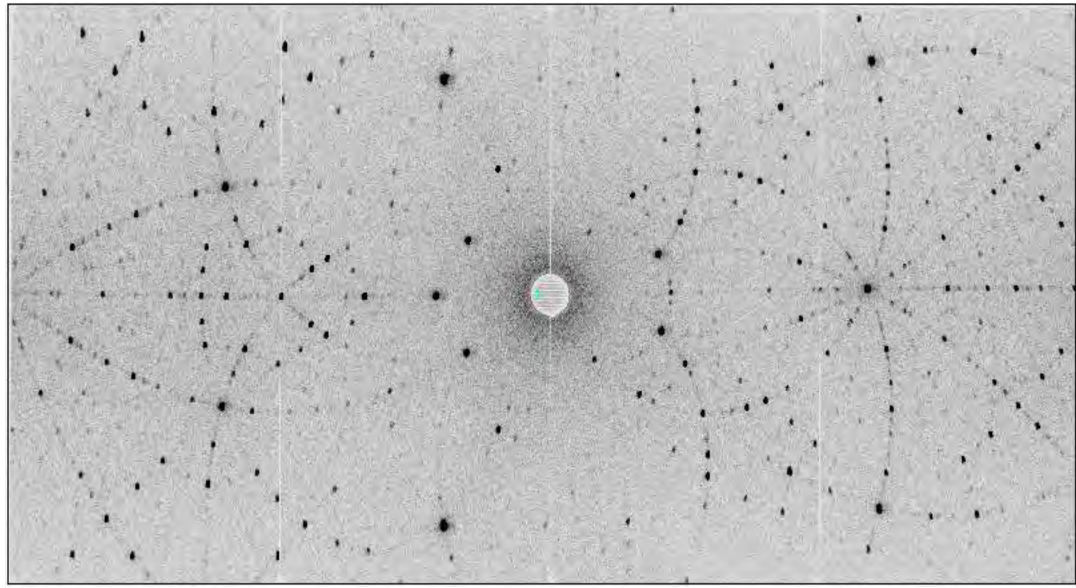
White Beam Neutron Diffraction-Vivaldi

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

SXD (pulsed source) & VIVALDI (continuous source)

- SXD uses energy discrimination (lower background, better resolution)
- VIVALDI uses continuous white beam (flux 2 to 3 orders of magnitude)
- SXD uses PM tubes & co-incidence logic (~3mm detector resolution)
- VIVALDI uses Neutron Image plate (~150 micron resolution)

White Beam Neutron Diffraction-Vivaldi

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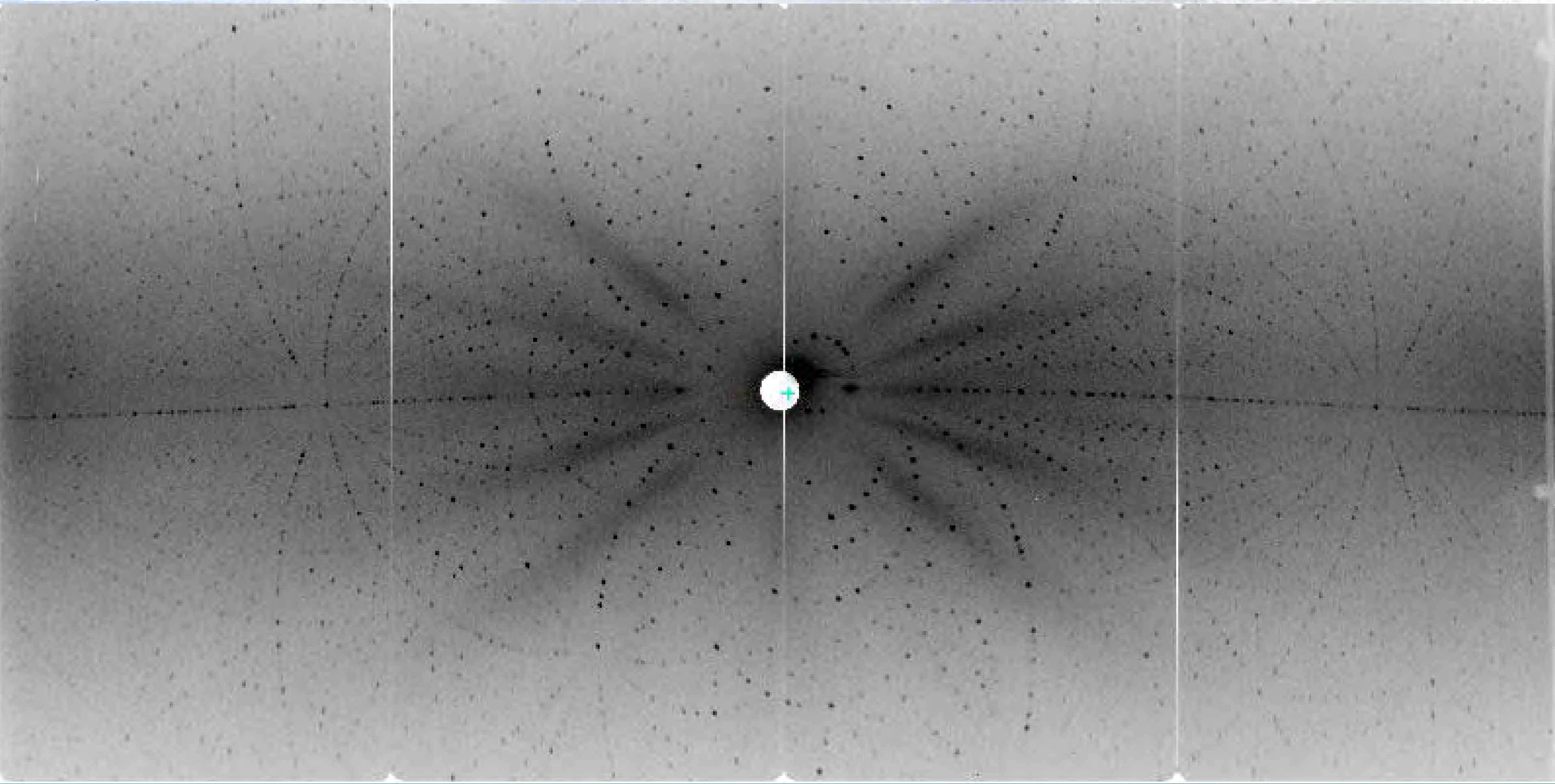
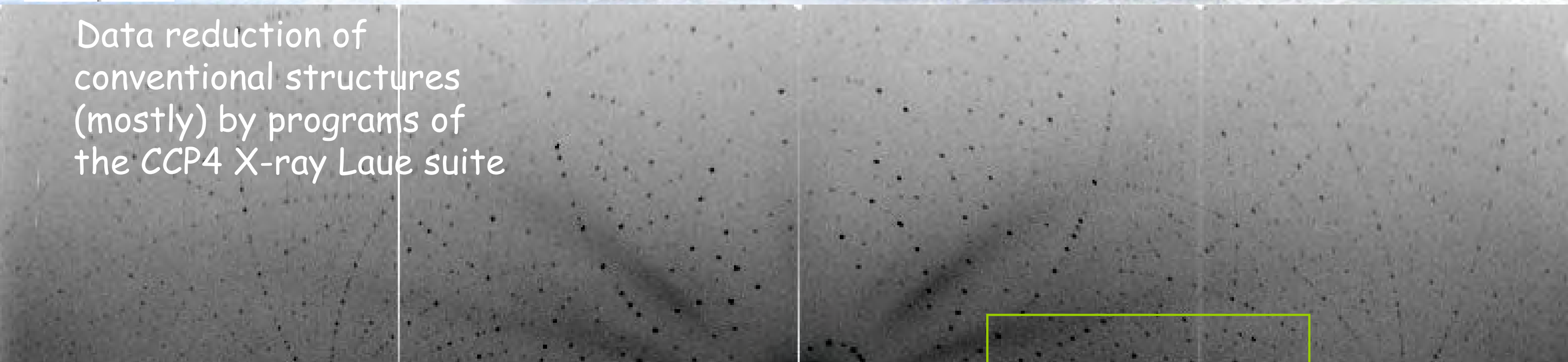


Vitamin B12. Nearly 10,000 measurable reflections
8-hour exposure, $10mm^3$ crystal.

White Beam Neutron Diffraction-Vivaldi

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

Data reduction of conventional structures (mostly) by programs of the CCP4 X-ray Laue suite



Quick inspection of integration ellipsoid boundaries

Improvements needed for spatial overlap

Vitamin B12. Nearly 10,000 measurable reflections

8-hour exposure, $10mm^3$ crystal.

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

Examples of experiments on VIVALDI

Compound	Volume (mm^3)	Cell ($Å^3$)	Exposure (hr)	Peaks seperated	$\langle I/\sigma(I)\rangle$	Time total (hr)*
Vitamin B12	6	8853	2	7596	10.1	60
$Cs_3VCl_6.4H_2O$	2	446	0.7	647	6.6	3
$Na_2Pb(OH)_2$	2	147	0.8	263	7.9	5
$LiAlSi_2O_6$	1.4	389	0.33	442	11.9	2
$Co(NH_3)_6.CuCl_5$	1	2691	0.7	1865	6.0	3
$Co_4C_{22}H_{36}$	0.6	8254	2.5	4340	4.8	22
dabco HBF_4	0.21	473	1	384	5.0	10
$Cd_2Nb_2O_7$	0.07	279	2	204	3.1	*
$C_4H_4N_2O_2$	0.00075	451	4	272	1.1	48
In a TiZr pressure cell at 10 kbar :						
$(NH_4)_2Cr(SO_4)_2.6H_2O$	3.5	726	1	557	5.6	4

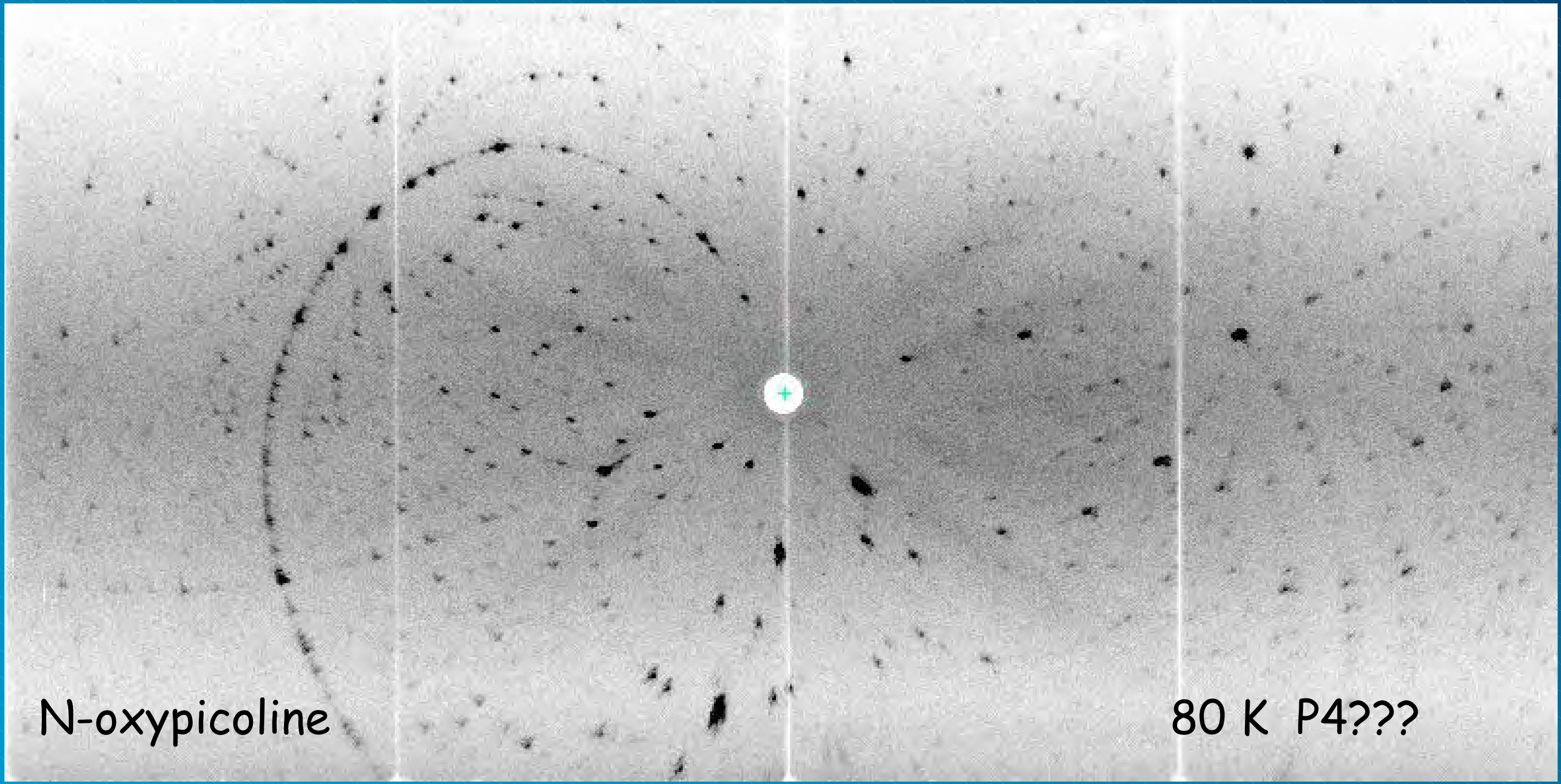
White Beam Neutron Diffraction-Vivaldi

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

Rapid data collection and a global view of reciprocal space

Symmetry and structural parameters as a function of e.g. temperature

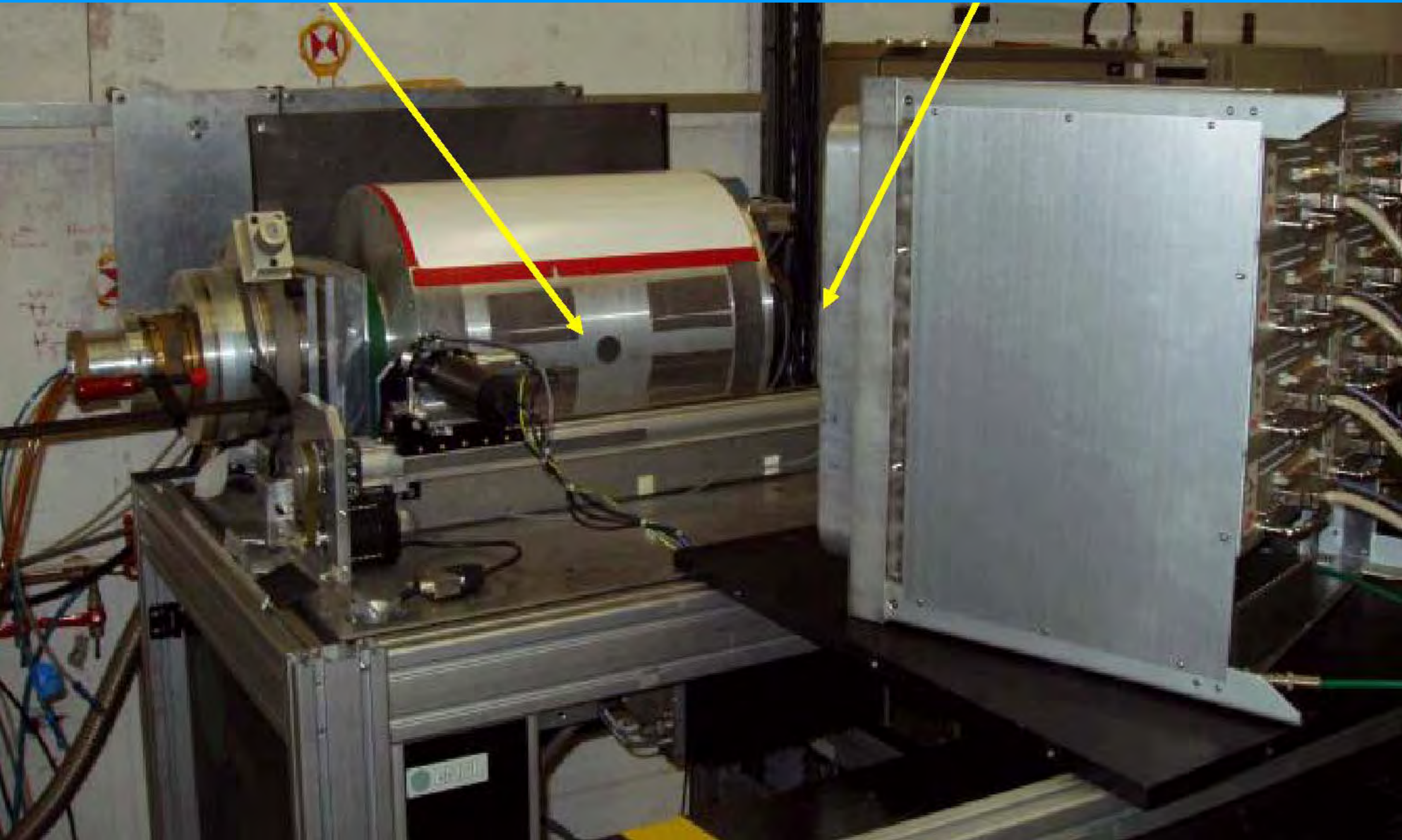
N-oxypicoline (Fillaux, Cousson, Nicolaii, Le Toquin, Paulus, Carlile & McIntyre, 2004)



2D Electronic Detectors & Image Plates

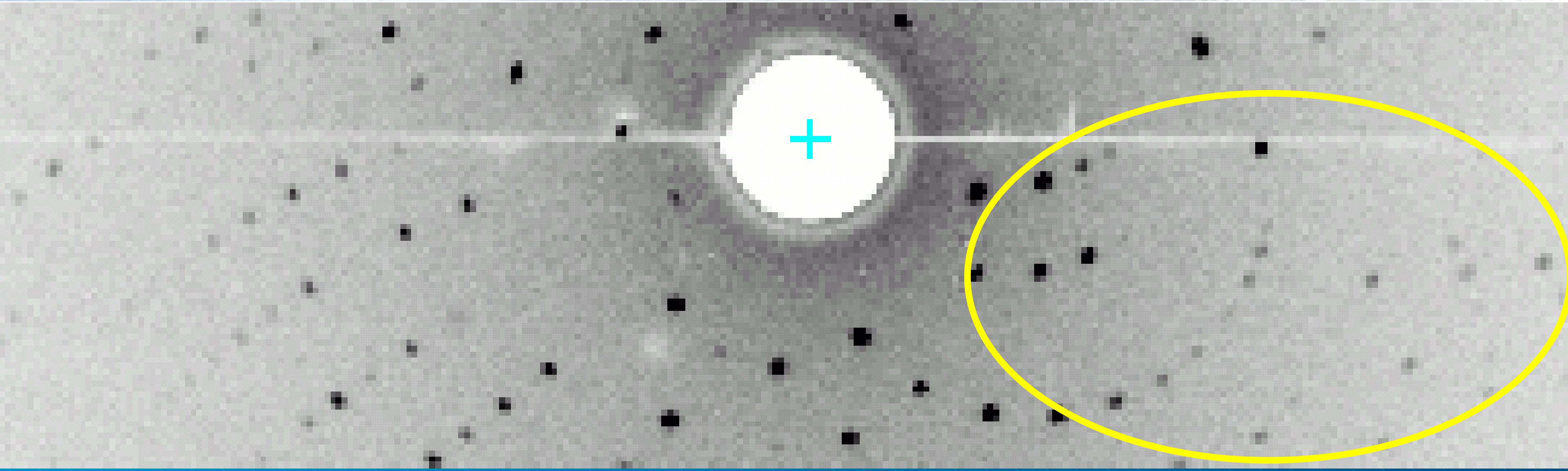
8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

LADI image plate vs D19 proto detector

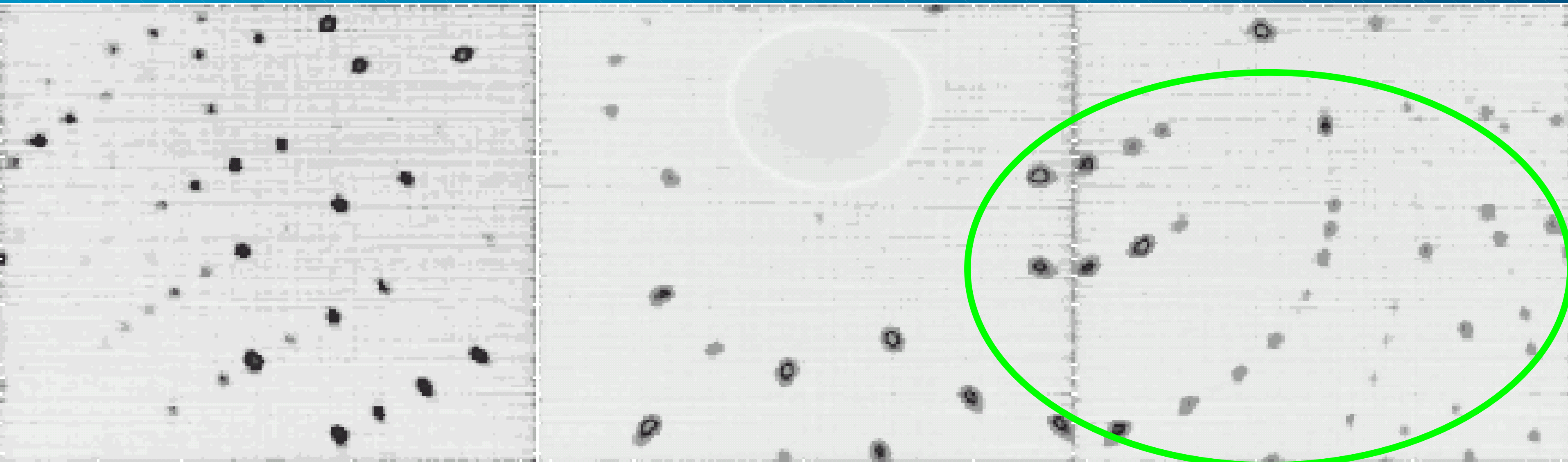


2D Electronic Detectors & Image Plates

8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

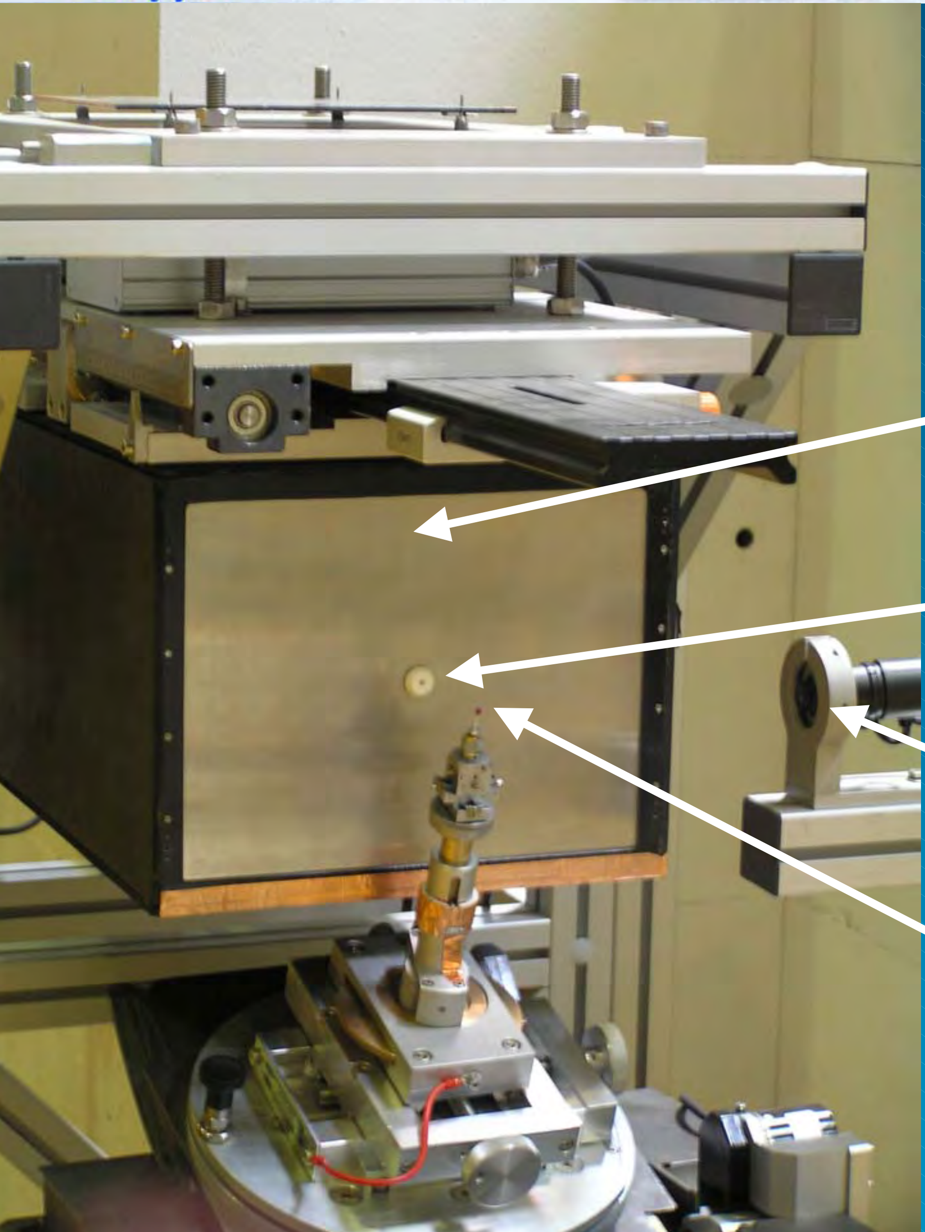


LADI image plate (12 hours above) vs D19 proto detector (1 hour below)



ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



Orient Express

On H24 thermal neutron guide

Dual lens-coupled chilled CCD's

CCD detectors in backscattering

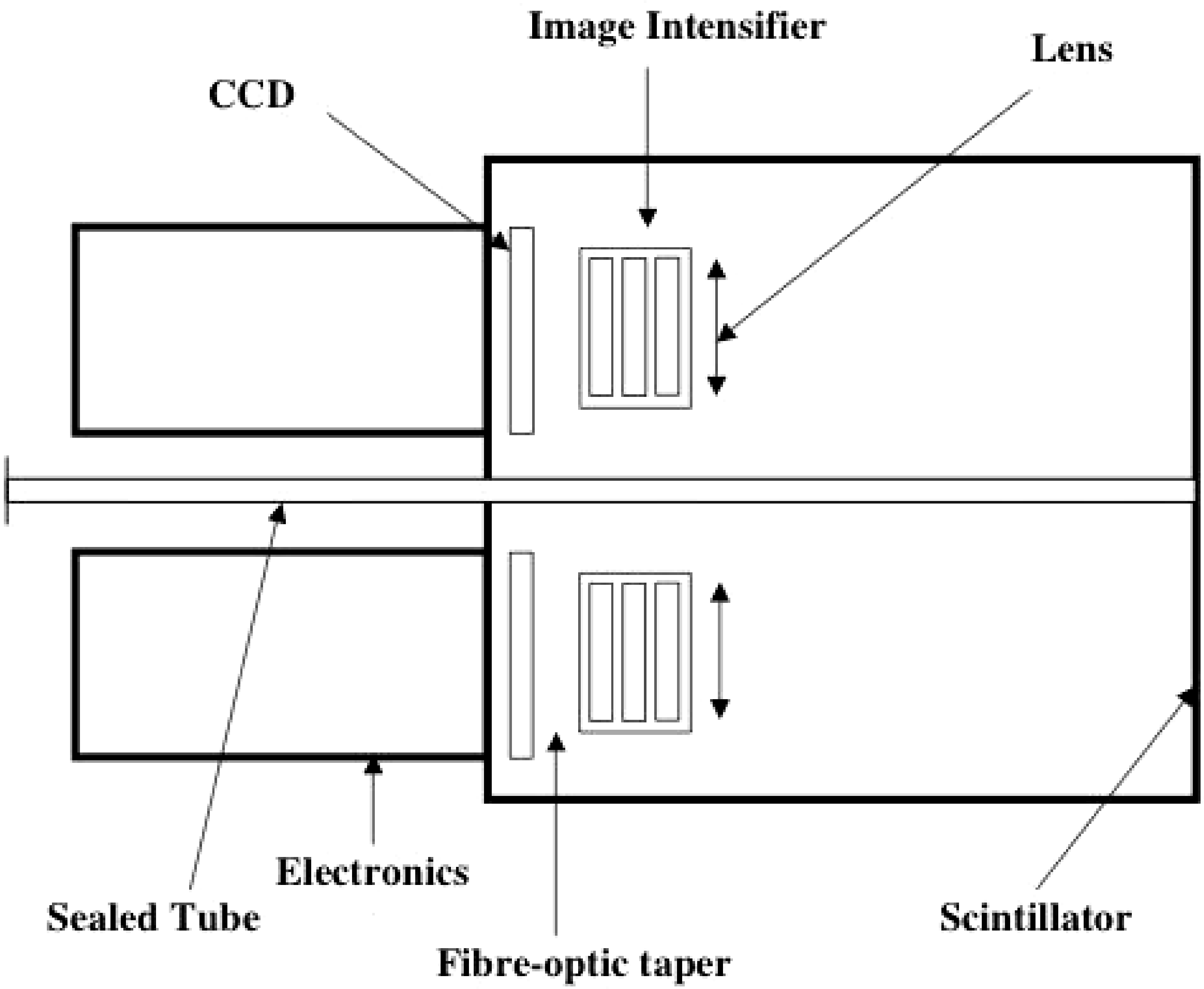
Neutron beam

TV camera for crystal alignment

Crystal on goniometer

ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



ORIENT EXPRESS, a new CCD detector

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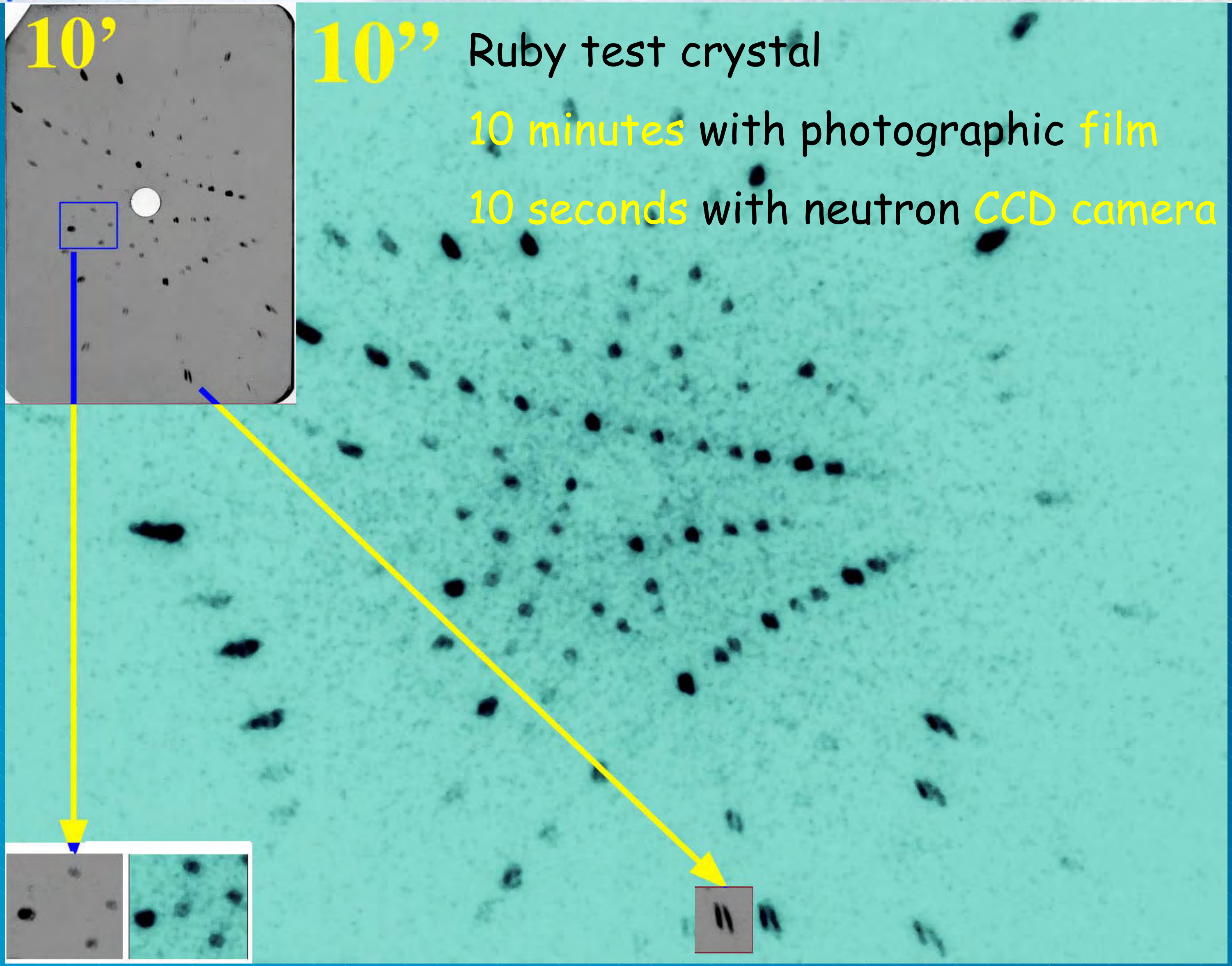


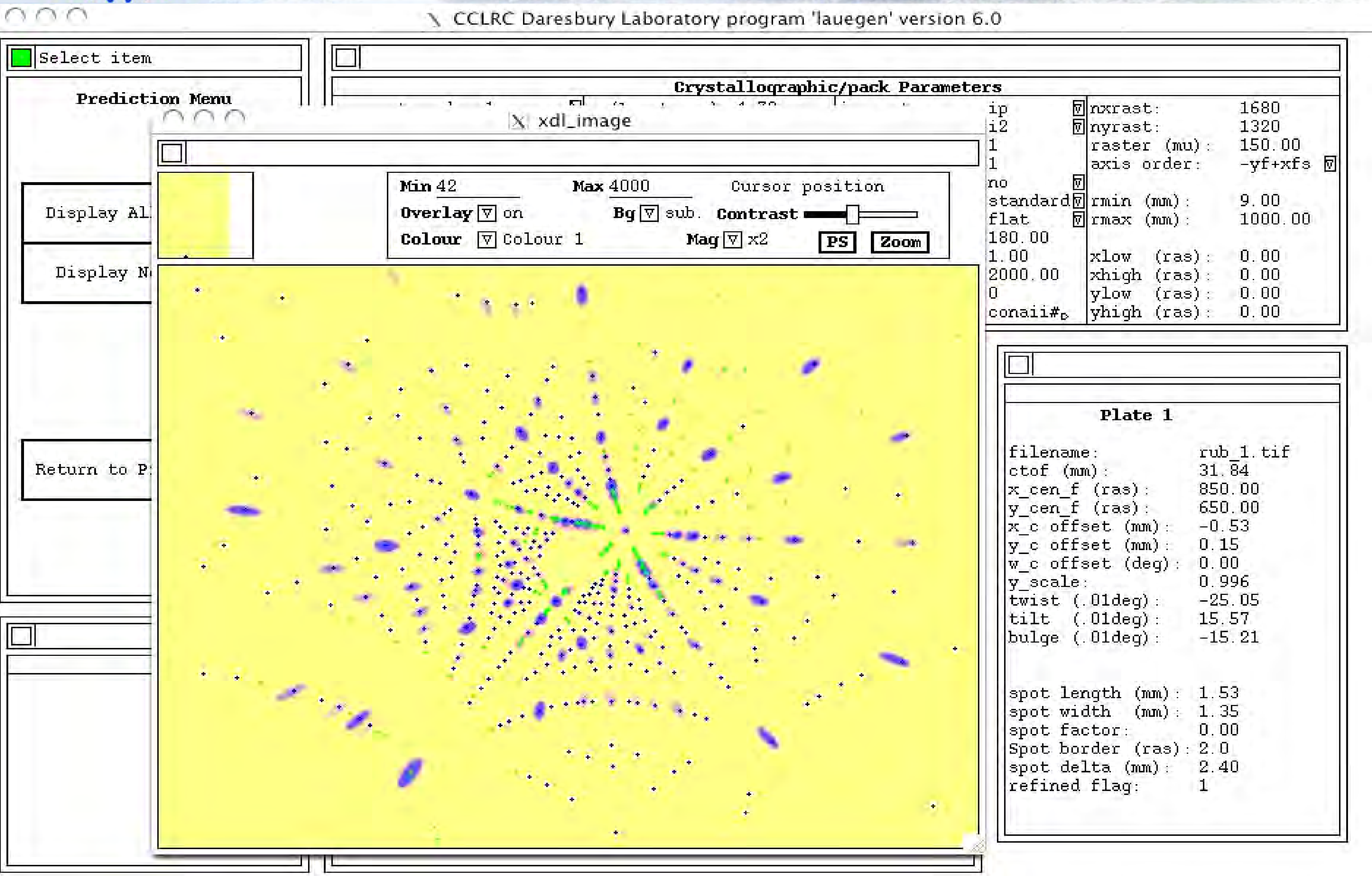
Ruby test crystal

10 minutes with photographic film

10 seconds with neutron CCD camera

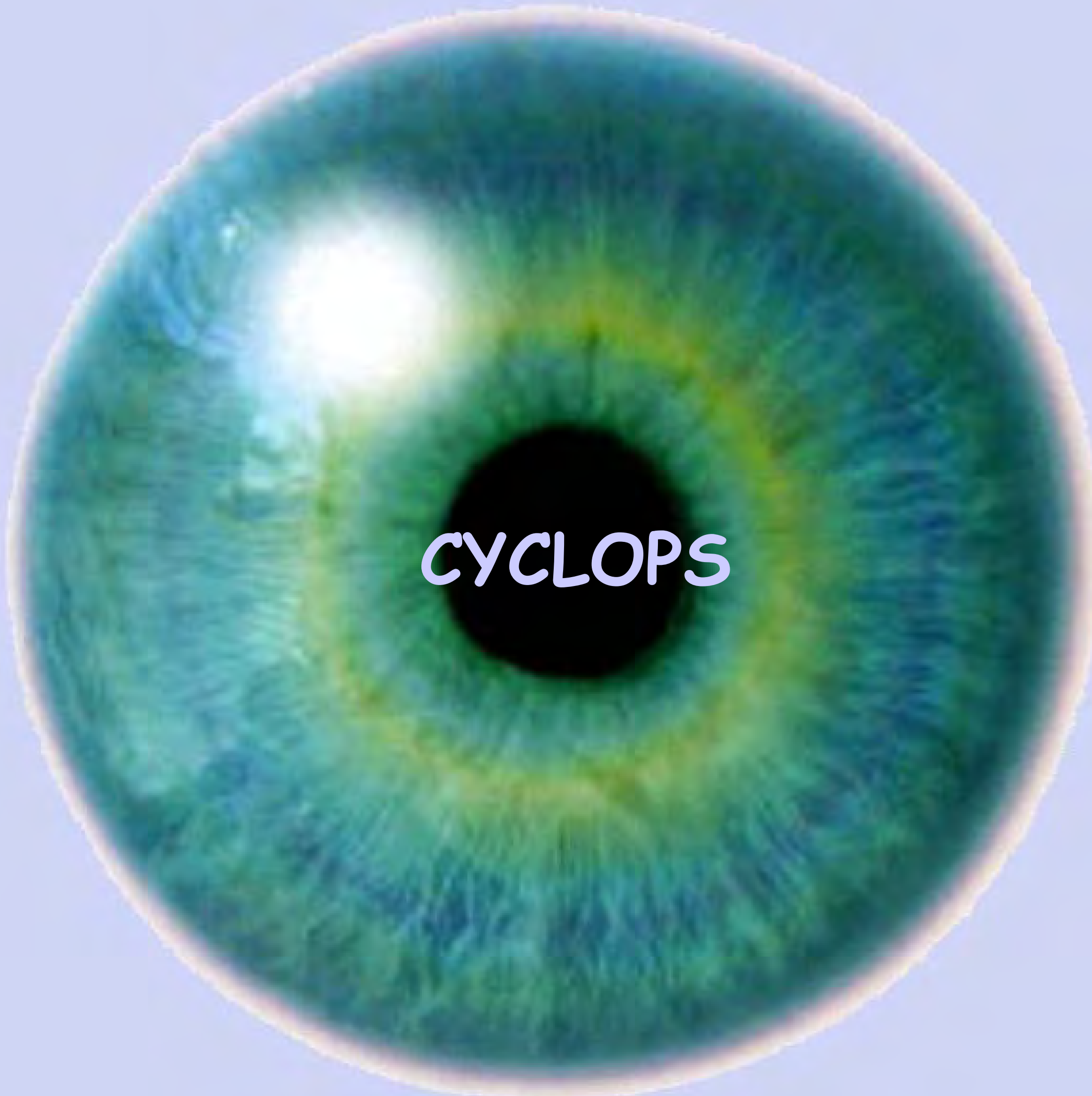
ORIENT EXPRESS, a new CCD detector

Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble



Next Generation Neutron Diffractometers

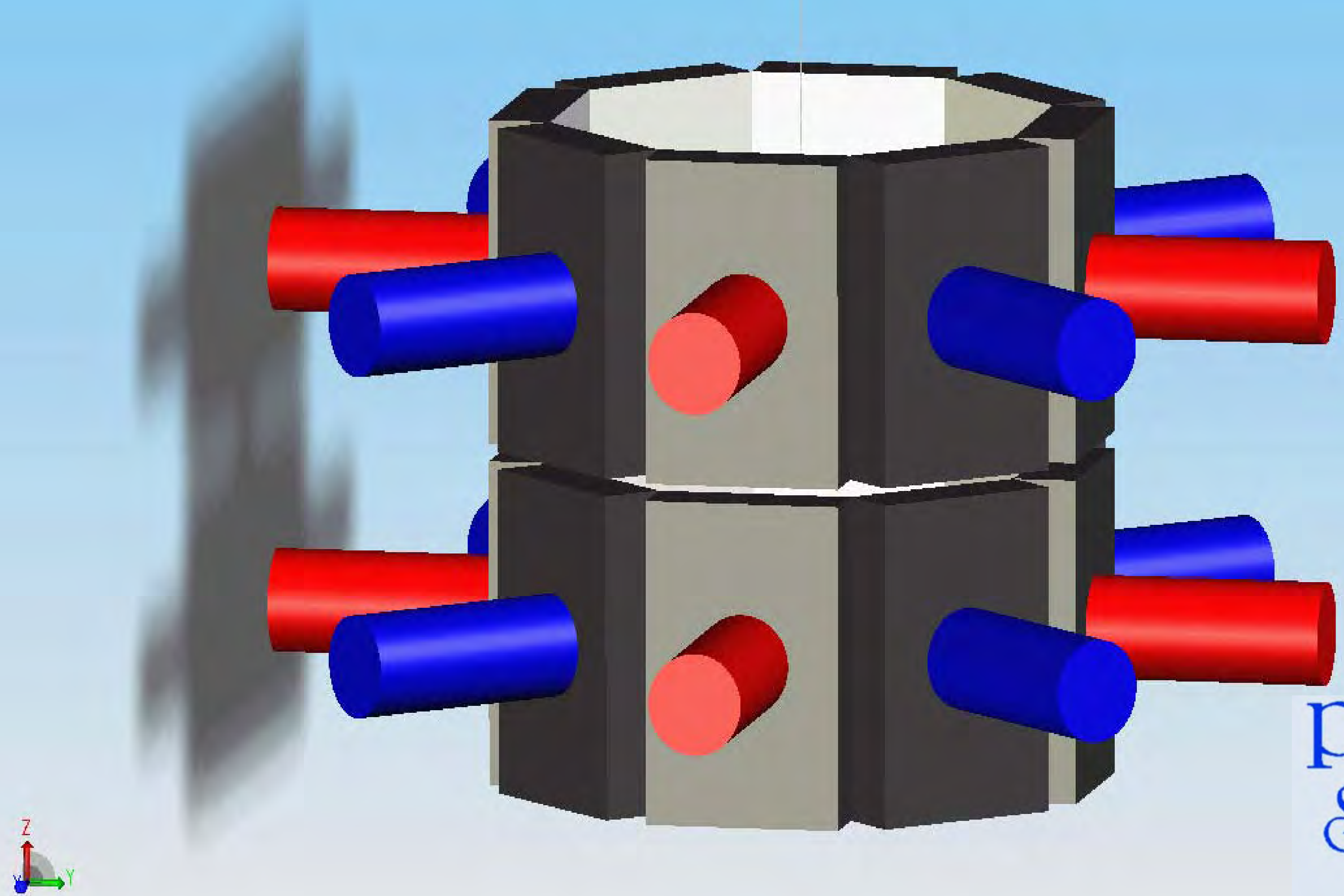
CYlindrical CCD Laue Optics Photo Scintillator



Next Generation Neutron Diffractometers

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

Double octagonal array of CCD neutron cameras



Photonic Science

CYCLOPS, a new type of neutron CCD detector

- Double octagonal array of CCD neutron cameras
- 70% of 4π
- 25% neutron efficiency (best image plates ~20%)
- 170x170 micron resolution
- 20 million 16-bit pixels
- Read out time of <1 second