Super D2B – A High Resolution 2D Neutron Detector

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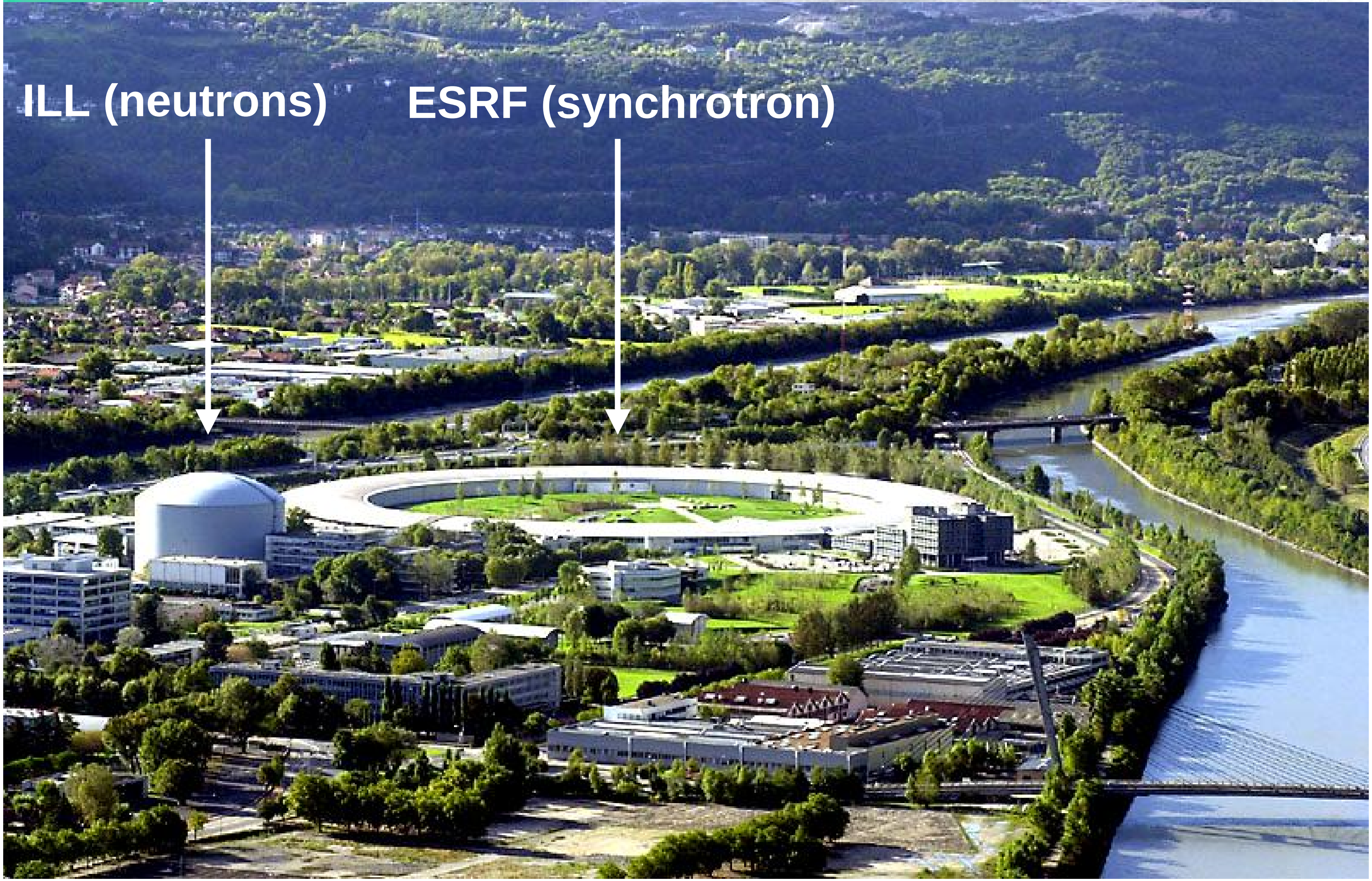
European Neutron & Synchrotron Sources
ILL & ESRF Grenoble

- World's most intense neutron source
- 1280 visiting scientists each year
- 300+ scientific papers each year
- physics, chemistry, biology, materials

- Proposals for experiments welcome
  Next deadline 2 weeks from now
  see: www.ill.fr (visitors club)

ILL member countries are shown in green

European Neutron & Synchrotron Sources
ILL & ESRF Grenoble



The ILL Millennium Programme in Neutron Diffraction

- New 10 year ILL contract with European members
- ILL seeking participation of more European states
- Millennium Programme -> ILL machines by x10 to x20
- New detector and neutron optic technology
- Competition with new US and Japanese neutron sources

Super-D2B proposal to upgrade D2B
A large 2D detector

September 1998: letter to ILL Director A. Leadbetter

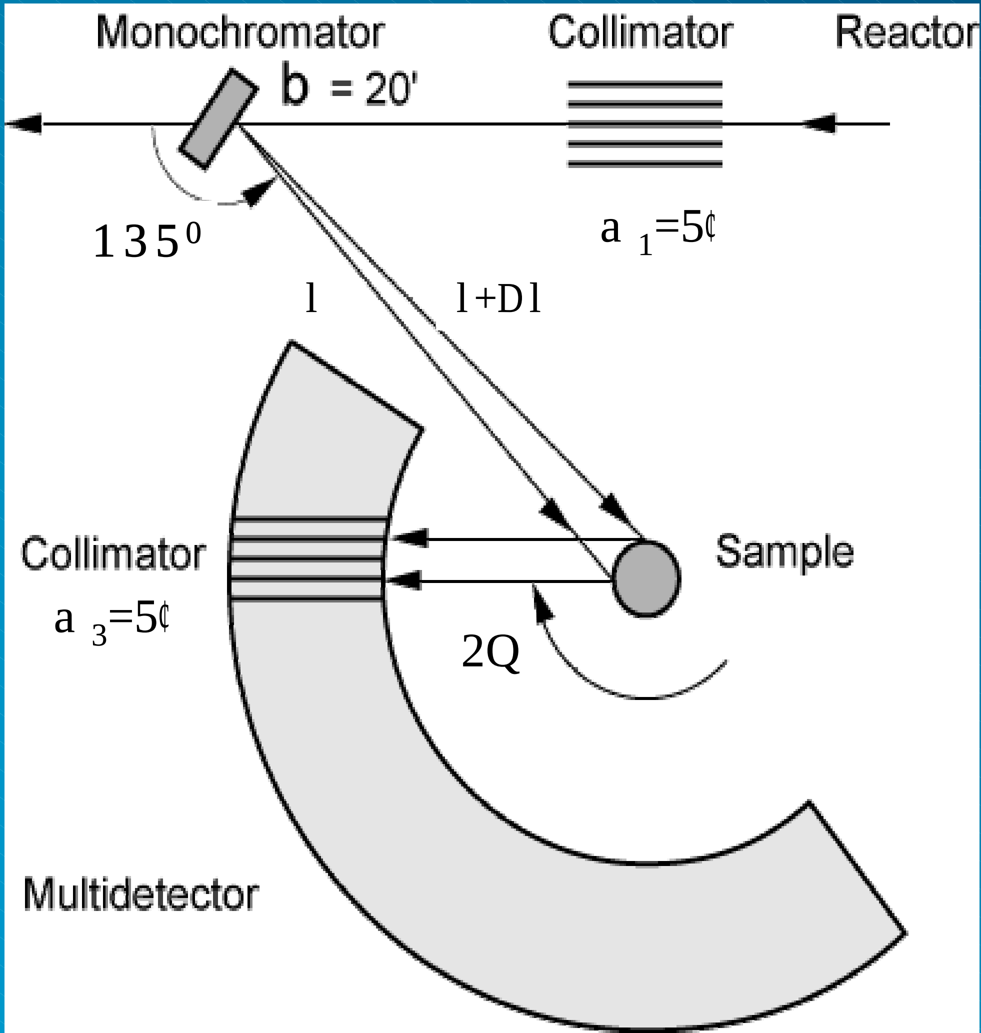
- D2B is 14 years old, and part of the 1984 “2ieme souffle” with D19 and D20
- D2B is one of the most demanded of ILL machines (number of users & papers)
- D2B has produced highly cited papers (zeolites, superconductors, GMR…)
- We propose an order of magnitude gain in intensity, and higher resolution

Why Neutrons ?

- Neutrons electrically neutral & more penetrating than X-rays.
- Neutrons interact with nuclei & locate atoms more precisely.
- Light atoms scatter neutrons as strongly as heavy atoms.
- Neutrons are tiny magnets, & determine magnetic structures.

- But neutron flux is very low compared with that of X-rays

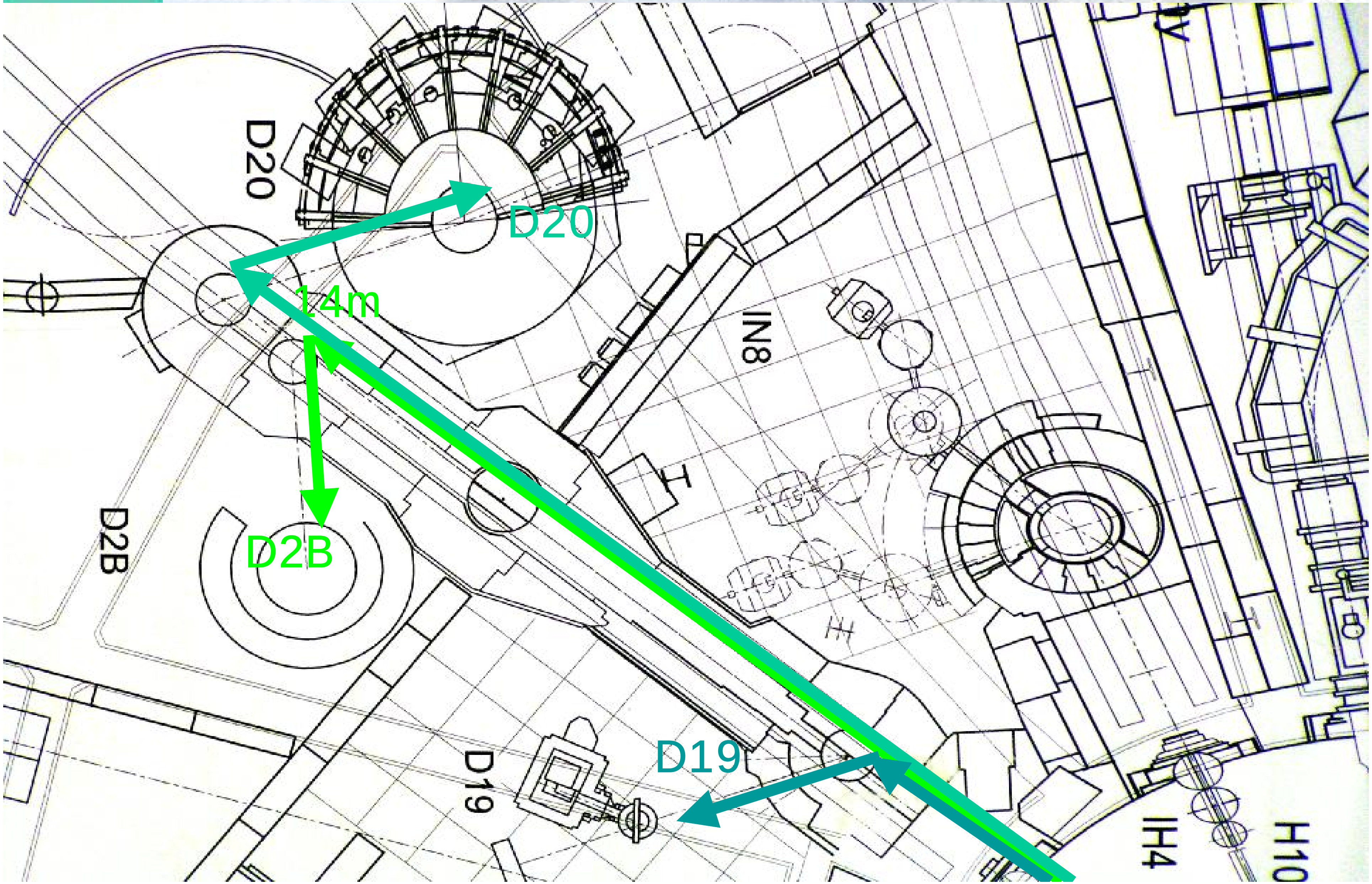
Neutron diffractometers are simple
Use of large multidetectors since 1970



- A "white" beam of neutrons from the reactor is collimated
- A large focussing monochromator selects particular wavelengths
- This small band of wavelengths is scattered by the sample
- A large multi-detector collects the neutrons scattered at all angles

3 Instruments on 1 ILL beam tube

D2B, D19 and D20

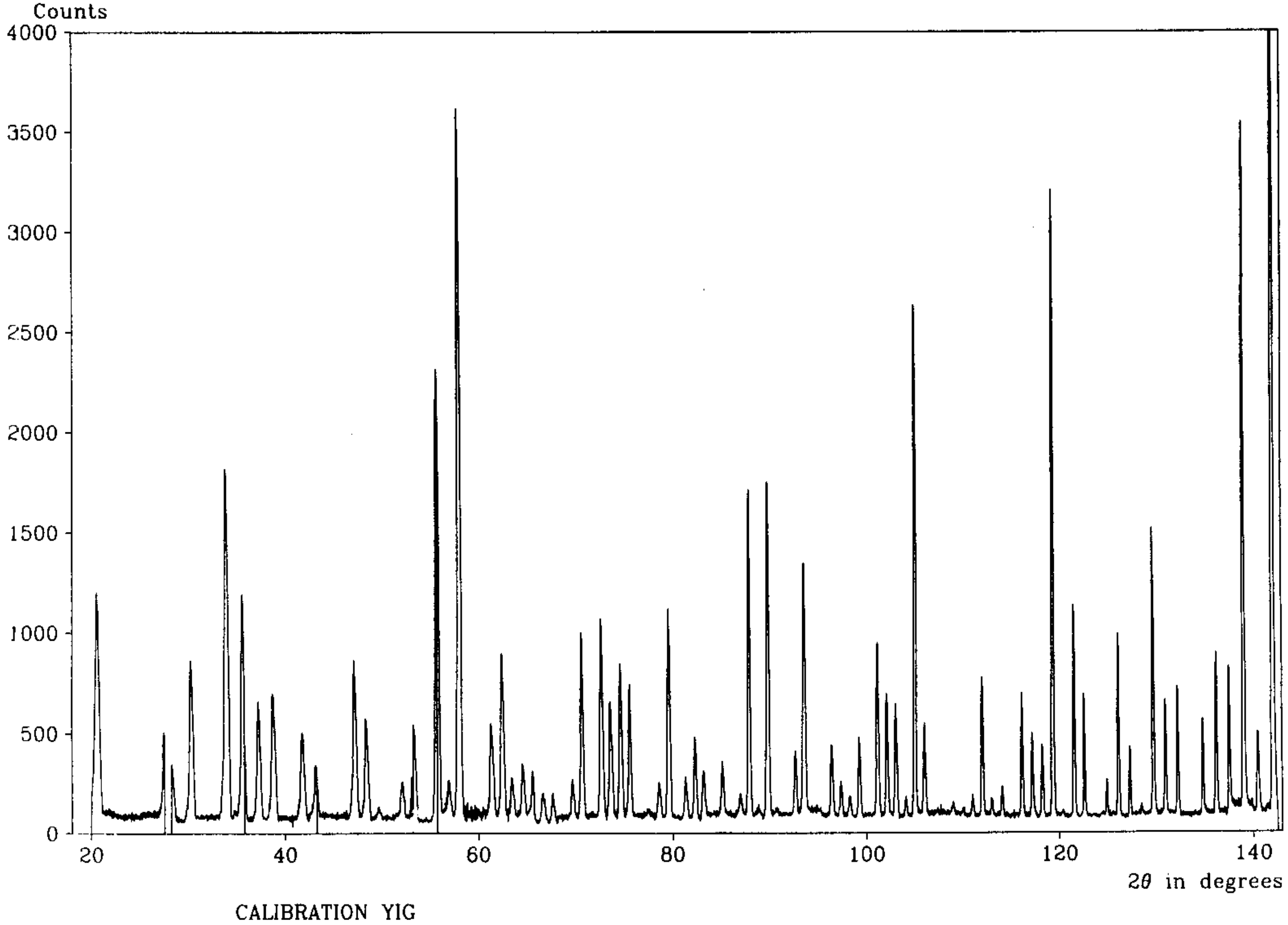


Neutron diffractometers are simple

Use of large multidetectors since 1970

D2B-ILL (1984)

Strong peaks at high angles
High precision structures



D2B-ILL First diffraction pattern (YIG) 1984

Highly cited ILL neutron diffraction papers

http://www.ill.fr/dif/citations/

Large number of citations /www.ill.fr/dif/citations/

- **922** **(D2B) Hwang HY, Cheong SW, Radaelli PG, Marezio M, Batlogg B (1995) Phy.Rev.Lett. 75, 914.**
Lattice effects on the magnetoresistance in doped LaMnO3.

- **856** **(D2B) Cava RJ, Hewat AW, Hewat EA, Batlogg B, Marezio M, Rabe KM, Krajewski JJ, Peck WF, Rupp LW (1990) Physica C. 165, 419.**
Structural anomalies oxygen ordering and superconductivity in oxygen deficient Ba2YCu3Ox.

- **501** **(D1A) Capponi JJ, Tournier R, Chaillout C, Hewat AW, Lejay P, Marezio M, Nguyen N, Raveau B, Soubeyroux JL, Tholence JL (1987) Europhysics Letters. 3, 1301.**
Structure of the 100K superconductor Ba2YCu3O7 between 5-300K by neutron powder diffraction.

- **367** **(D2B) Deteresa JM, Ibarra MR, Algarabel PA, Ritter C, Marquina C, Blasco J, Garcia J, Delmoral A, Arnold Z (1997) Nature 386, 256-259**
Evidence for magnetic polarons in the Magnetoresistive materials

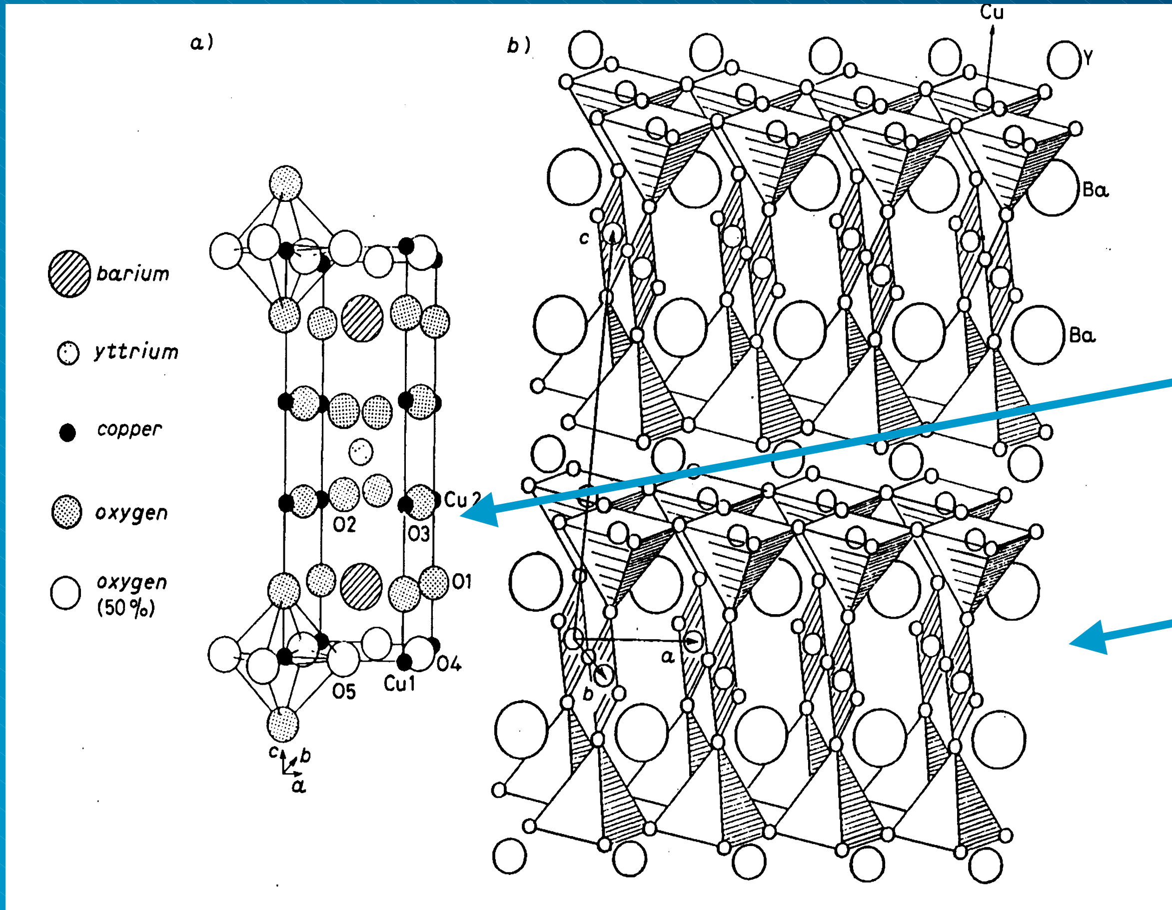
- **337** **(D1A) Fitch AN, Jobic H, Renouprez A (1986) J. Phys. Chem. 90, 1311**
Location of benzene in sodium-Y zeolite by powder neutron diffraction

- **321** **(D2B) Radaelli PG, Cox DE, Marezio M, Cheong SW, Schiffer PE, Ramirez AP (1995) Phys.Rev.Lett. 75, 4488**
Simultaneous structural, magnetic, and electronic-transitions in La(1-x)Ca(x)MnO3 with x=0.25 and 0,5

- **319** **(D2B) Radaelli PG, Cox DE, Marezio M, Cheong, SW (1997) Phys.Rev. B55, 3015**
Charge, orbital, and magnetic ordering in La(0.5)Ca(0.5)MnO3

- **218** **(D2B) Kaldis E, Fischer P, Hewat AW, Hewat EA, Karpinski J, Rusiecki S (1989) Physica C. 159, 668.**
Low temperature anomalies and pressure effects on the structure and Tc of the superconductor YBa2Cu4O8 (Tc=80 K).

Neutrons scatter strongly from light atoms
Oxygen order in High-Tc superconductors

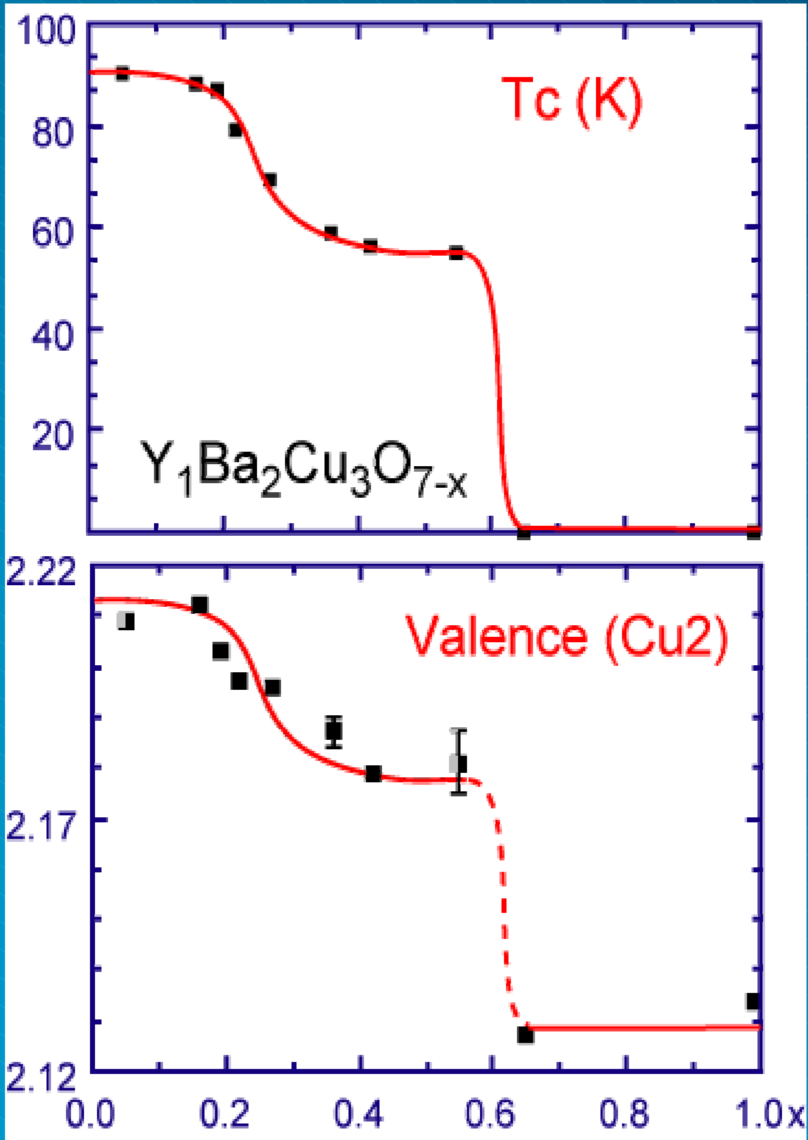


The 90K high Tc Superconductor $Y_1Ba_2Cu_3O_7$

- Left -by X-rays (Bell labs. & others)
- Right -by Neutrons (ILL & others)

Neutrons gave new insight, important in searching for similar materials.

Neutrons - precise metal-oxygen distances
Valence Charge and "Charge Transfer"



High-Tc superconductors

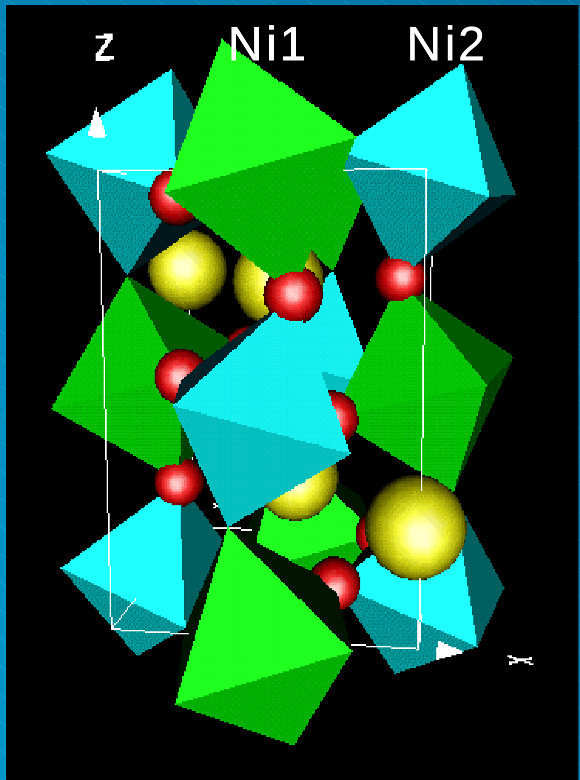
- Charge reservoir concept
- Tc depends on oxidation
- Imagine new charge reservoirs
- Discovery of new materials

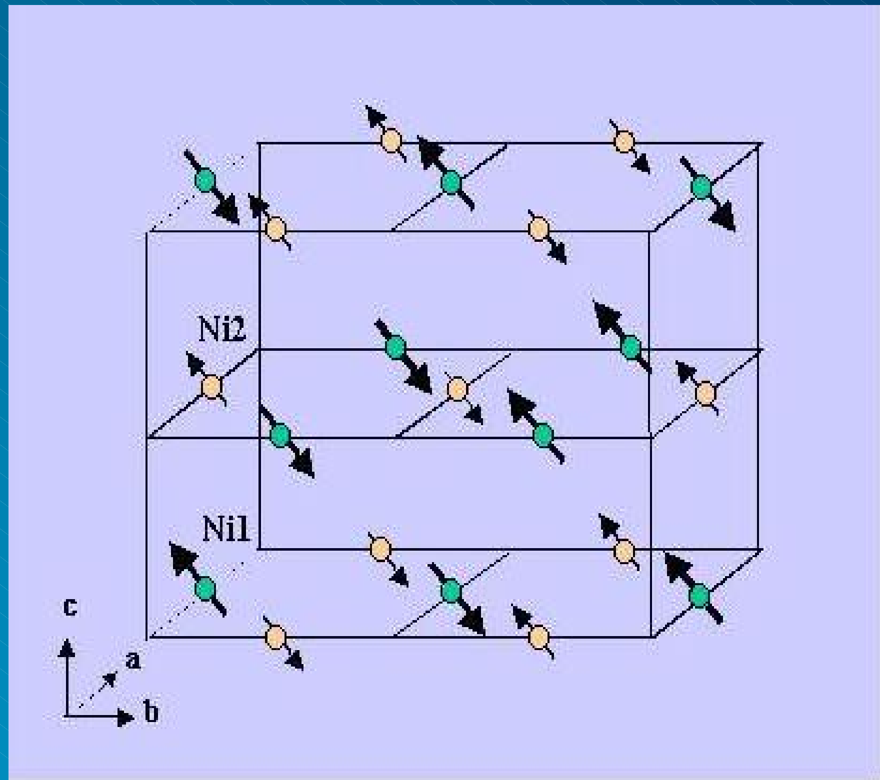
ILL and Bell labs. (1990)

Neutrons – Magnetic Structures
Charge Transfer in YNiO3

Combined ESRF, D1B and D2B data - Alonso J.A. et al (1999) PRL 82, 3873

Metallic Ortho. YNiO3 -> Insulating Mono. YNiO3 T < 582K   Ni valence 3-d, 3+ d

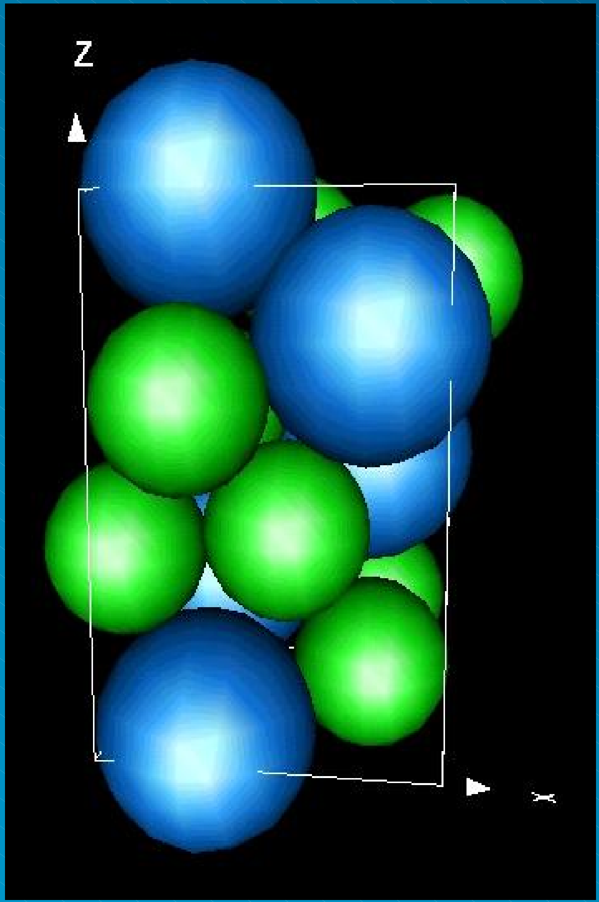




V(Ni1) = 2.62   V(Ni2) = 3.17

M(Ni1) = -1.4 m_B M(Ni2) = 0.7 m_B

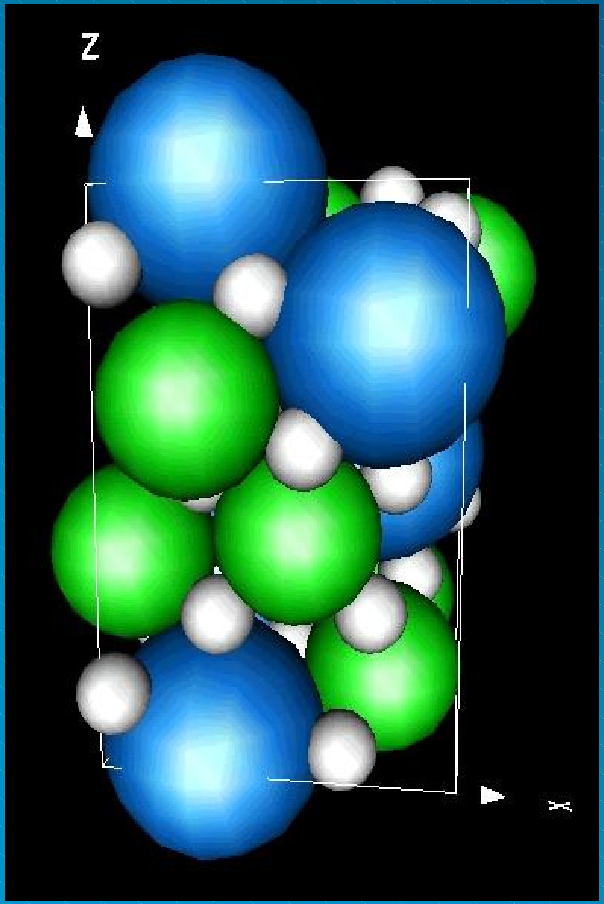
Real Materials, not crystals
Hydrogen in Metals



- Hydrogen storage in metals
  - Location of H among heavy atoms
  - No single crystals
- Laves phases eg $LnMg_2H_7$ (La,Ce)
- Can even find H in Eu on D20 !

Gingl, Yvon et al. (1997) J. Alloys Compounds 253, 313.
Kohlmann, Gingl, Hansen, Yvon (1999) Angew. Chemie 38, 2029. etc..

Real Materials, not crystals
Hydrogen in Metals



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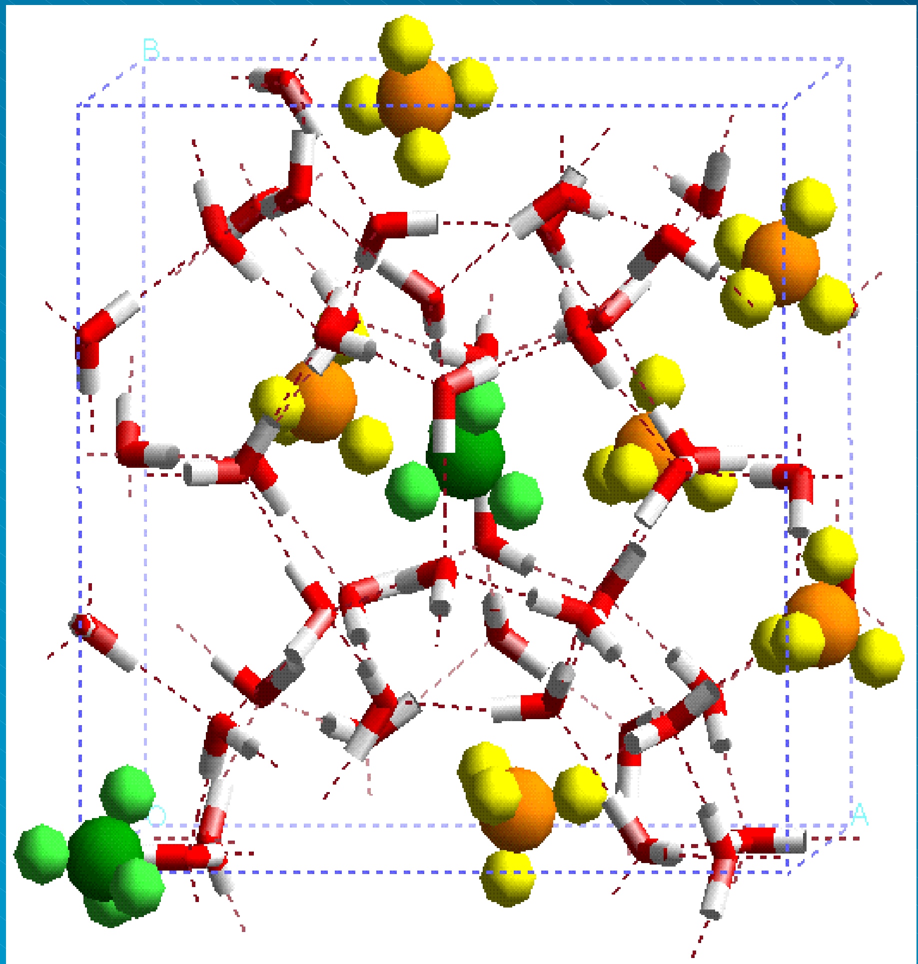
Clathrates - hydrate fuel from the ocean

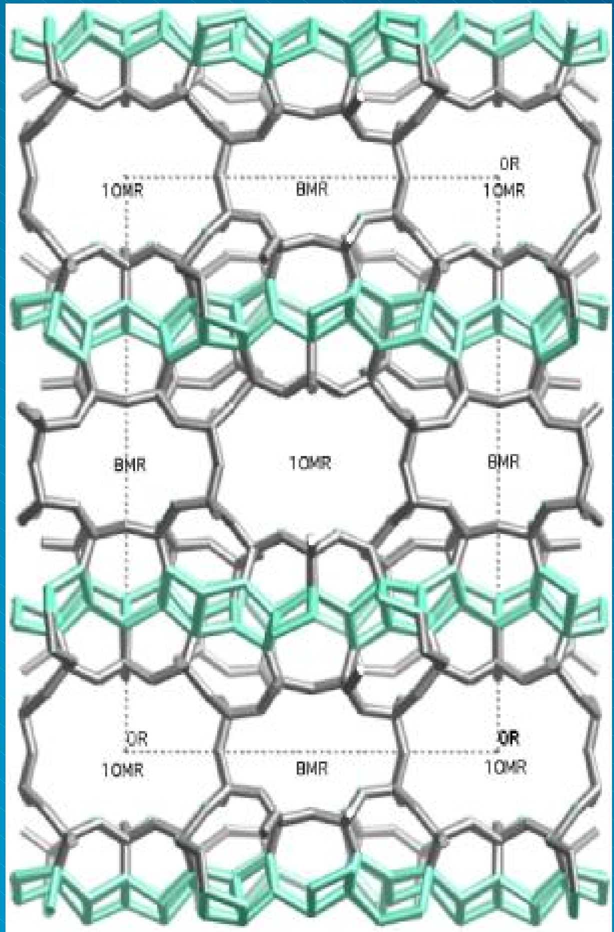


- Clathrates consist of molecular cages that can trap methane (spheres)
- Neutrons are important – they scatter strongly from the light methane atoms
- High pressure compressibility was studied, to help with seismic searches for clathrates

B.Chazallon, A.Klaproth, D.Staykova, W.Kuhs (Göttingen)

EPSRC

Molecular sieves and ion exchangers



- Ion exchangers can remove toxic metals from the environment
- New types of zeolite ion-exchangers are needed to trap specific elements
- Neutron and synchrotron radiation are used to understand ion exchange
- RUB29, a new lithium zeolite for cleaning up radioactive caesium

J.B.Parise, S-H.Park, A.Tripathi,
T.Nenoff, M.Nymann (SUNY & SANDIA)

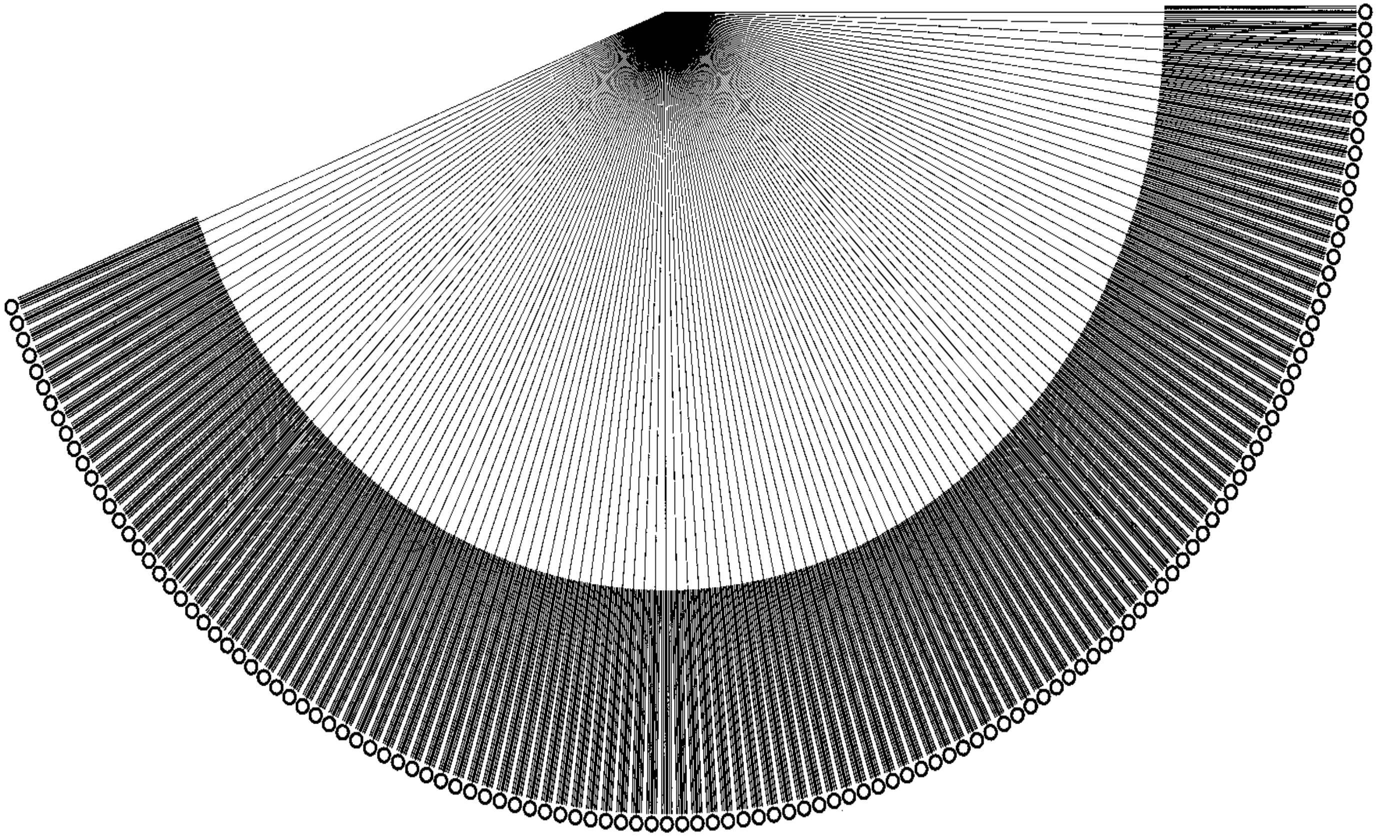
What did we propose ?

- x2 number of detectors 64 -> 128 detectors
- x3 height of each detector/collimator -> 300 mm
- 50% increase in flux in the sample -> horizontally+vertically focussing
- 2D detector to allow correction for curvature of diffraction cones
- New faster electronics and computers (PSD electronics + Linux)
- New cryo-refrigerator (no cryogenics) & 7T superconducting magnet
- Application to UK-EPSRC and French ministry to pay for all this !

Why did it take so long ?

- Most of the time & effort was getting the money (~ 1 million €) !

High resolution 2D linear wire detector
Super-D2B

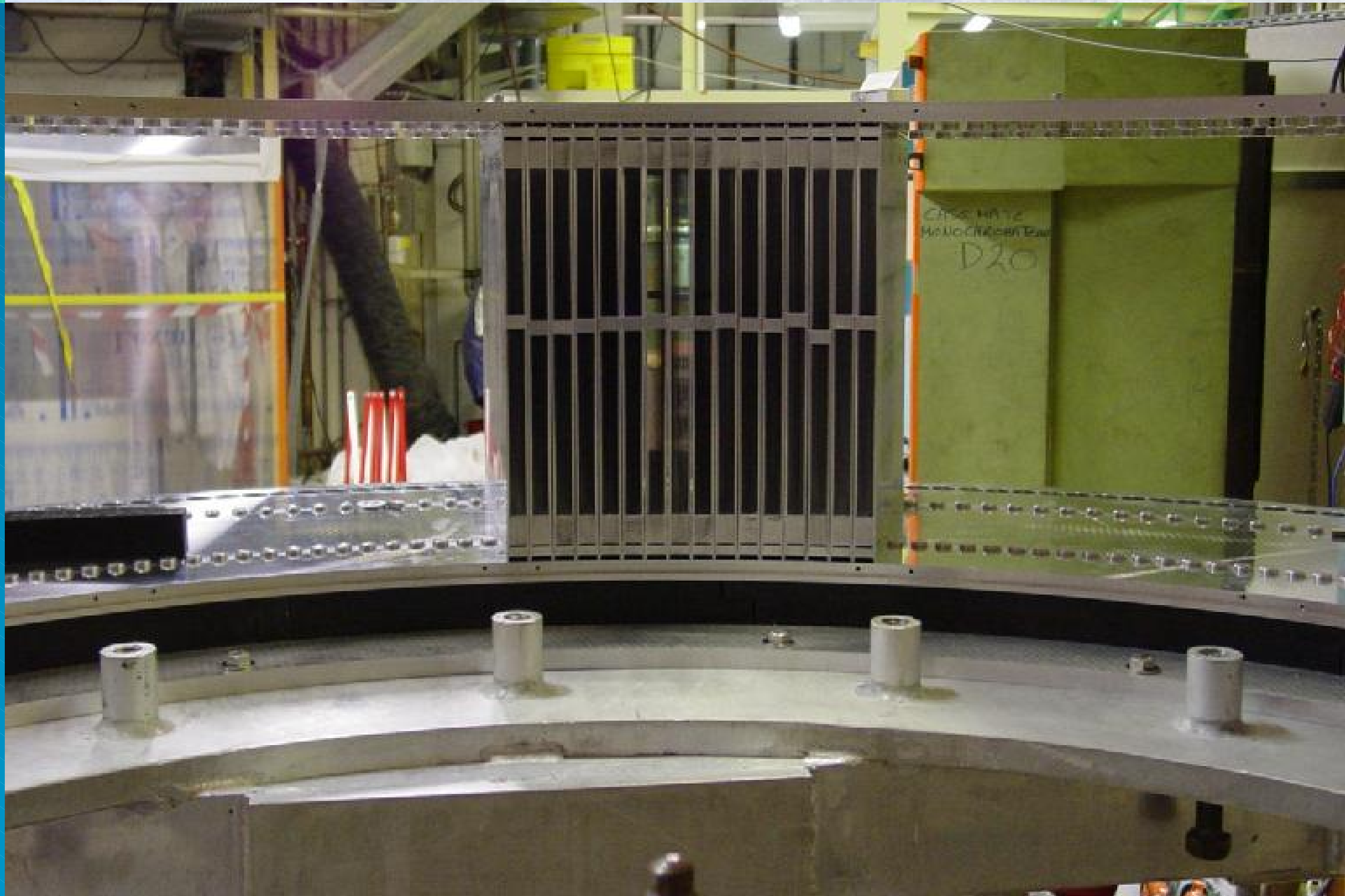


128 x 300 mm high resistive wire detectors, high resolution collimators

High resolution 2D linear wire detector
2003 Super-D2B

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

High resolution 2D linear wire detector
2003 Super-D2B



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

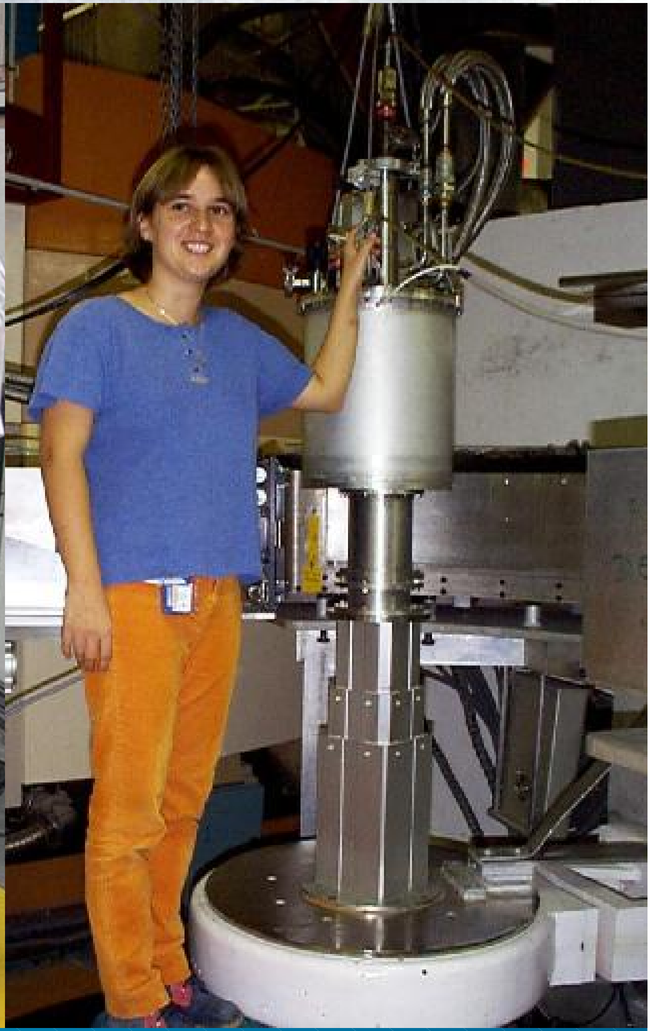
High resolution 2D linear wire detector
2003 Super-D2B

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

Array of linear wire tubes
2004 Super-D2B

Completed 300 mm high 2D-detector, with 6 Tesla cryomagnet

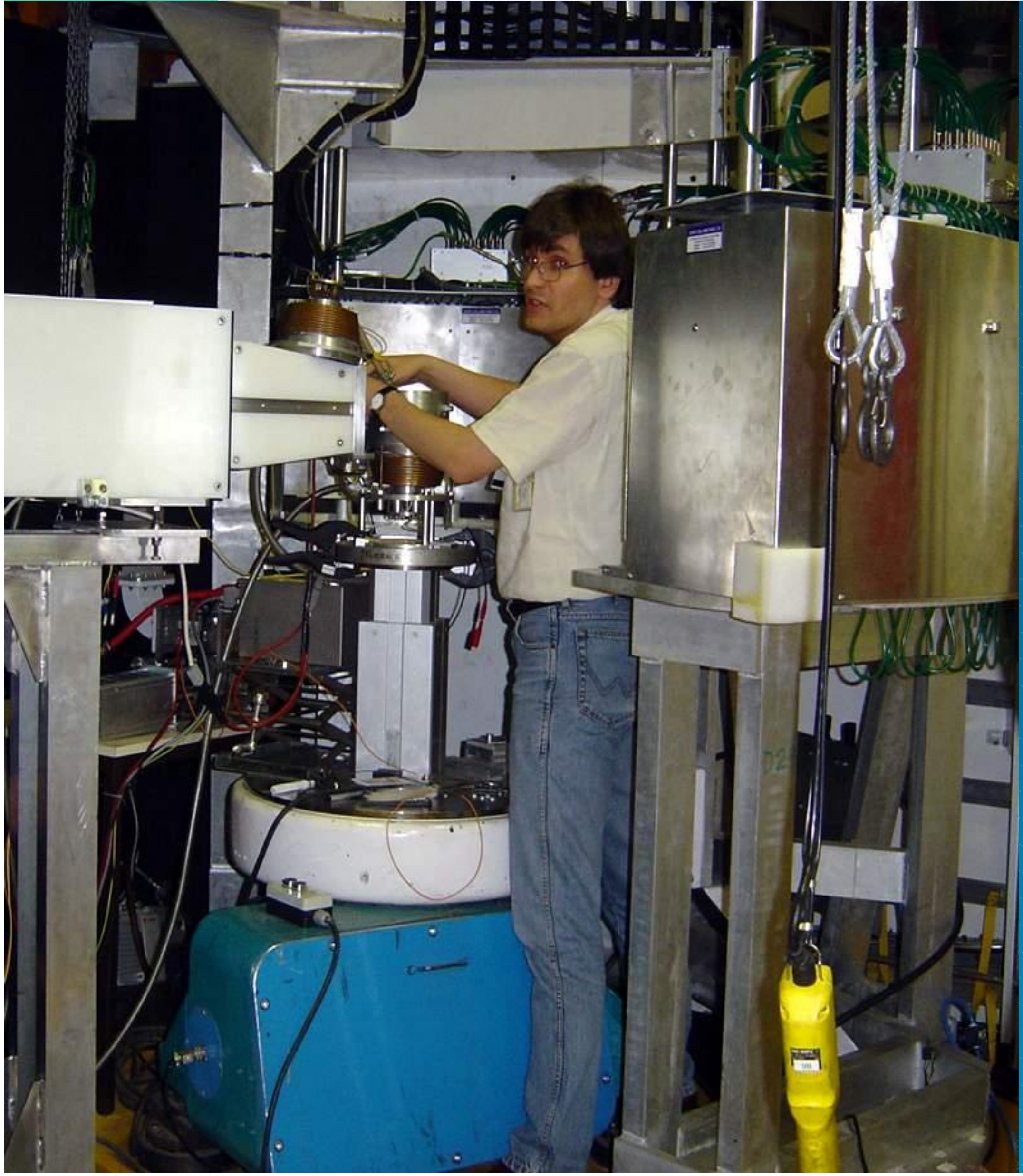
Array of linear wire tubes
2004 Super-D2B



Emma Suard with the ILL-Eurocollimator Team

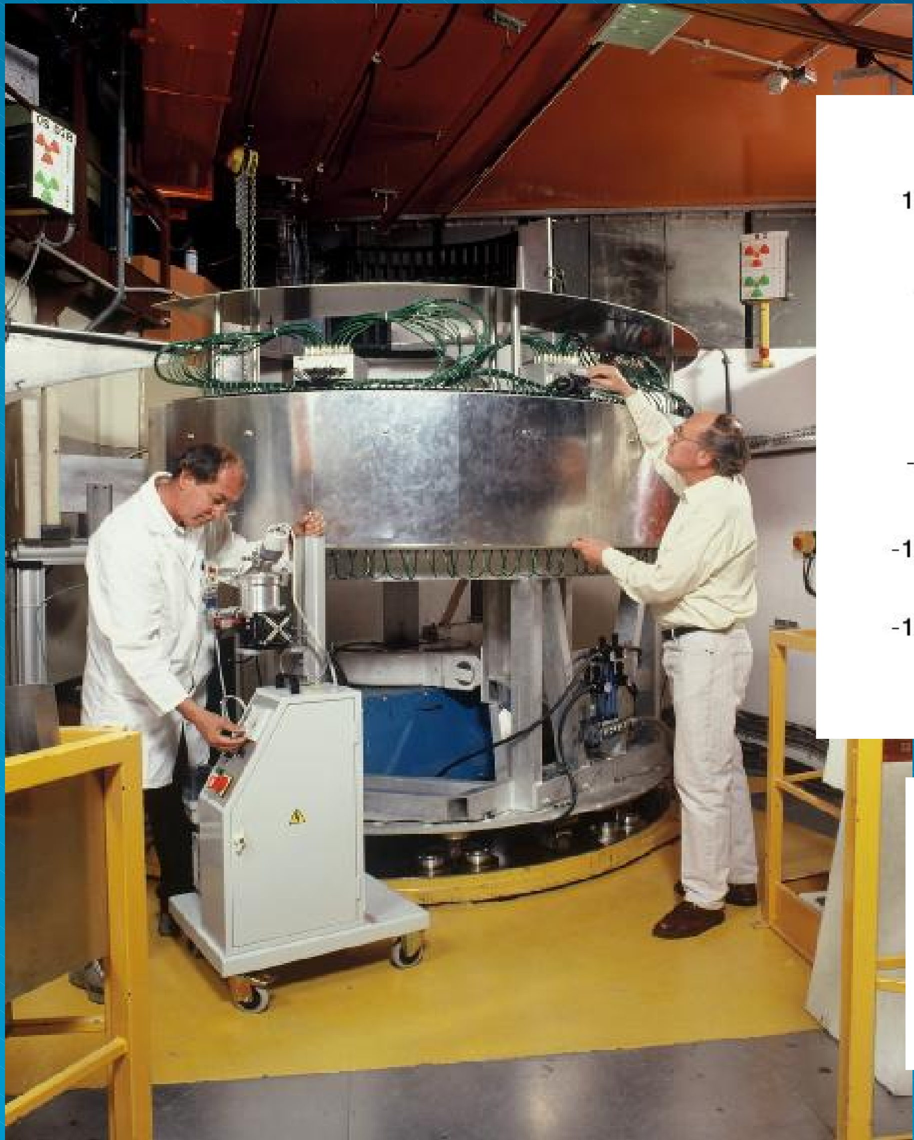
Gwen Rousse & 4K Refrig.

Applications - fast detectors, small samples

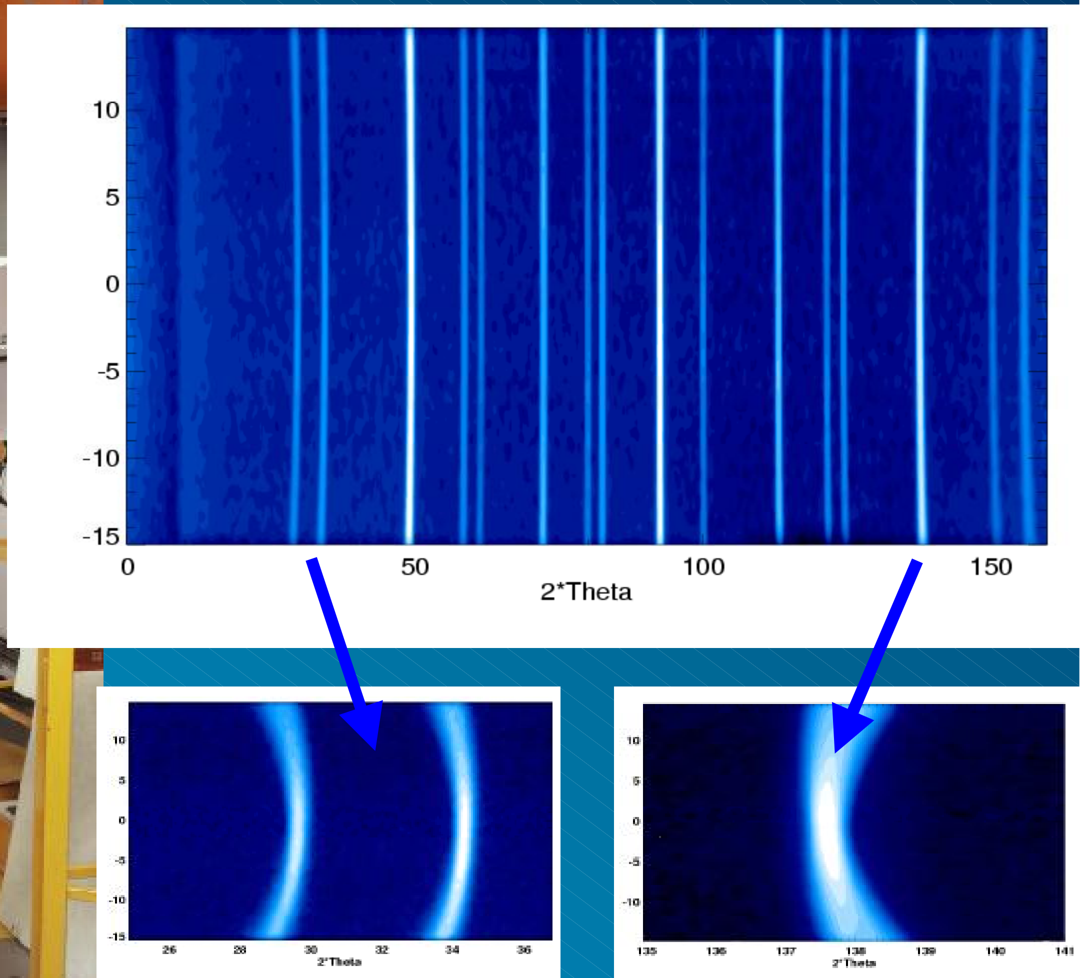


High-T Microwave Furnace
Super-D2B (Boysen et al.)
...with Carsten Korte from Giessen (2004)

2D detectors for CW Powder Diffraction



UK-EPSRC Super-D2B project at ILL

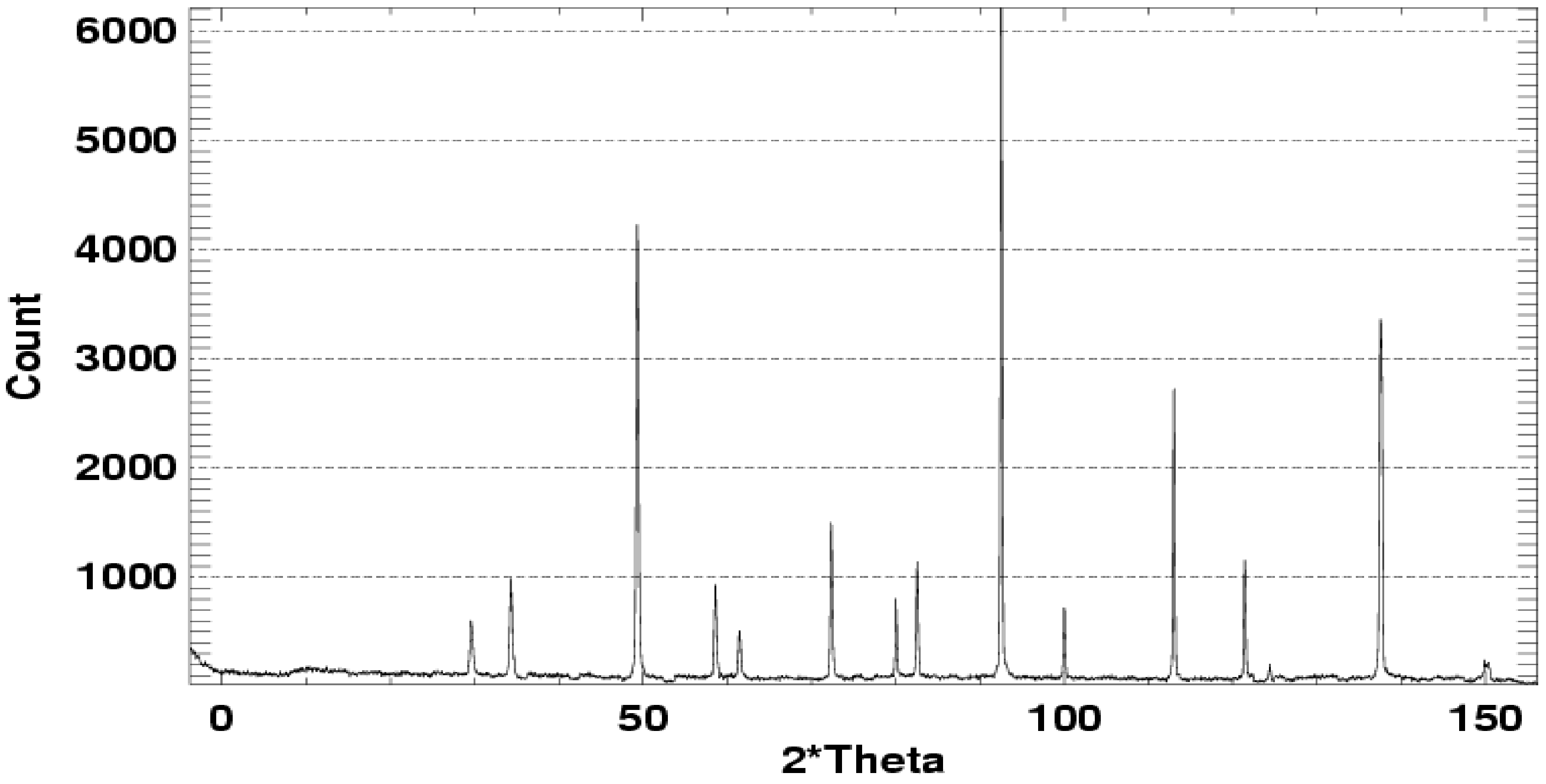


E.Suard, C.Ritter, A.Hewat, P.Attfield… (Edinburgh)

CeO2 NIST standard

2-theta scan in 10 min, no corrections

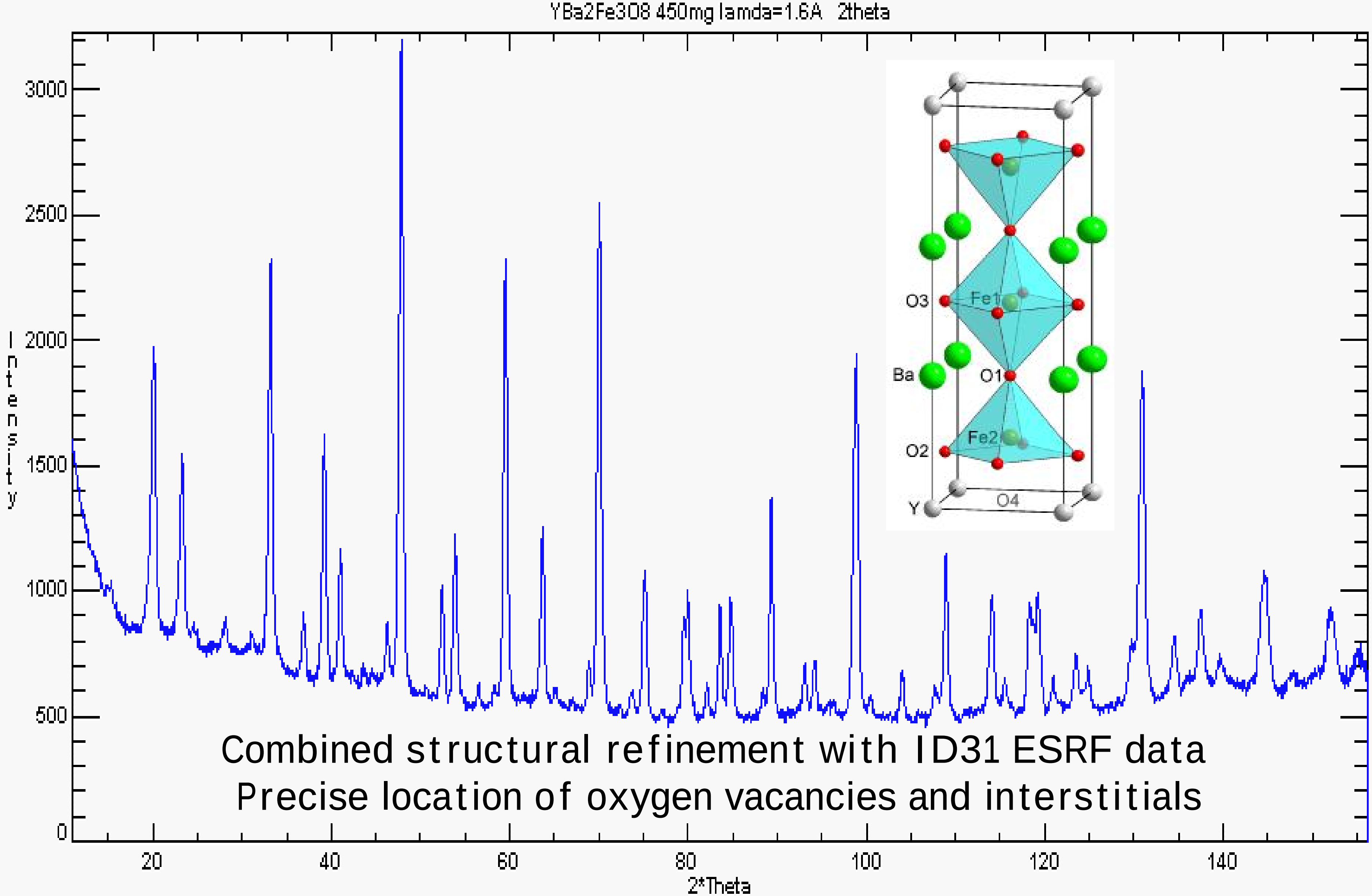
suard suar 80 80 135 135 slits open D2B CeO2 HI



d2b Date 16-Apr-03 Time 14:36:10 Run 056620 /d2bcal.zone

$YBa_2Fe_3O_{8+w}$ 450 mg measured in 5 hours

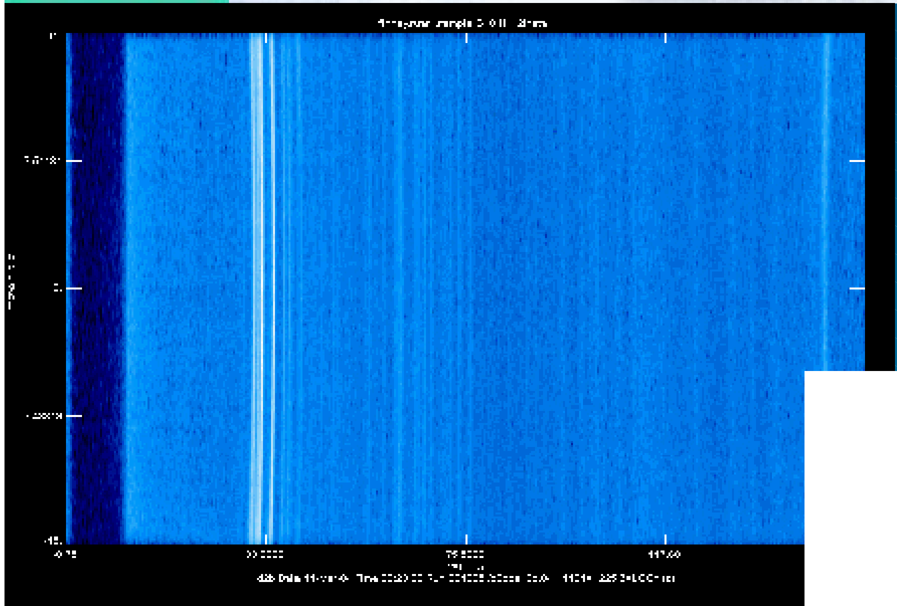
P. Karen, E. Suard, F. Fauth. (2004) Inorg.Chem. (submitted)



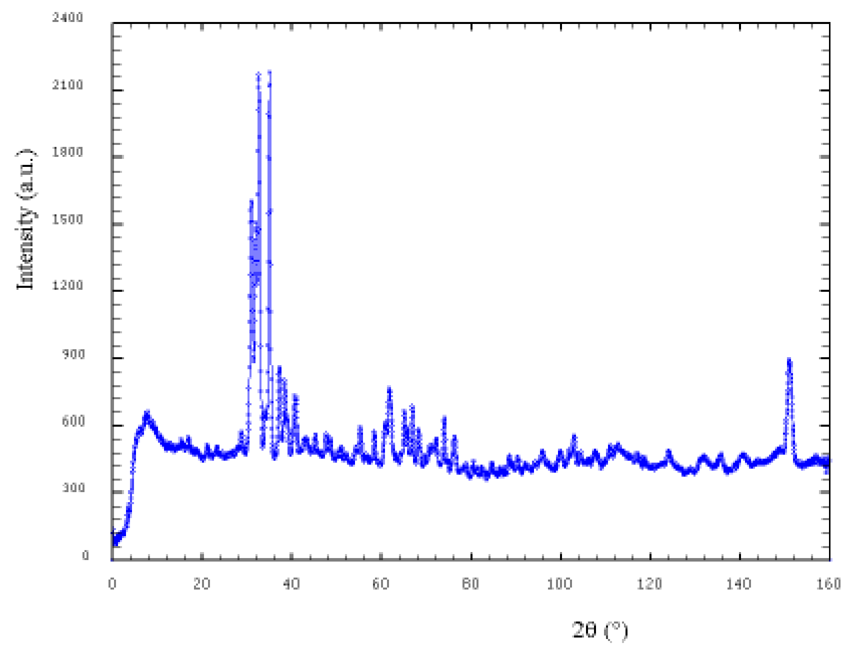
Combined structural refinement with ID31 ESRF data
Precise location of oxygen vacancies and interstitials

180 mg Ice, high pressure at 80K

J. Finney, E.Suard (2004)

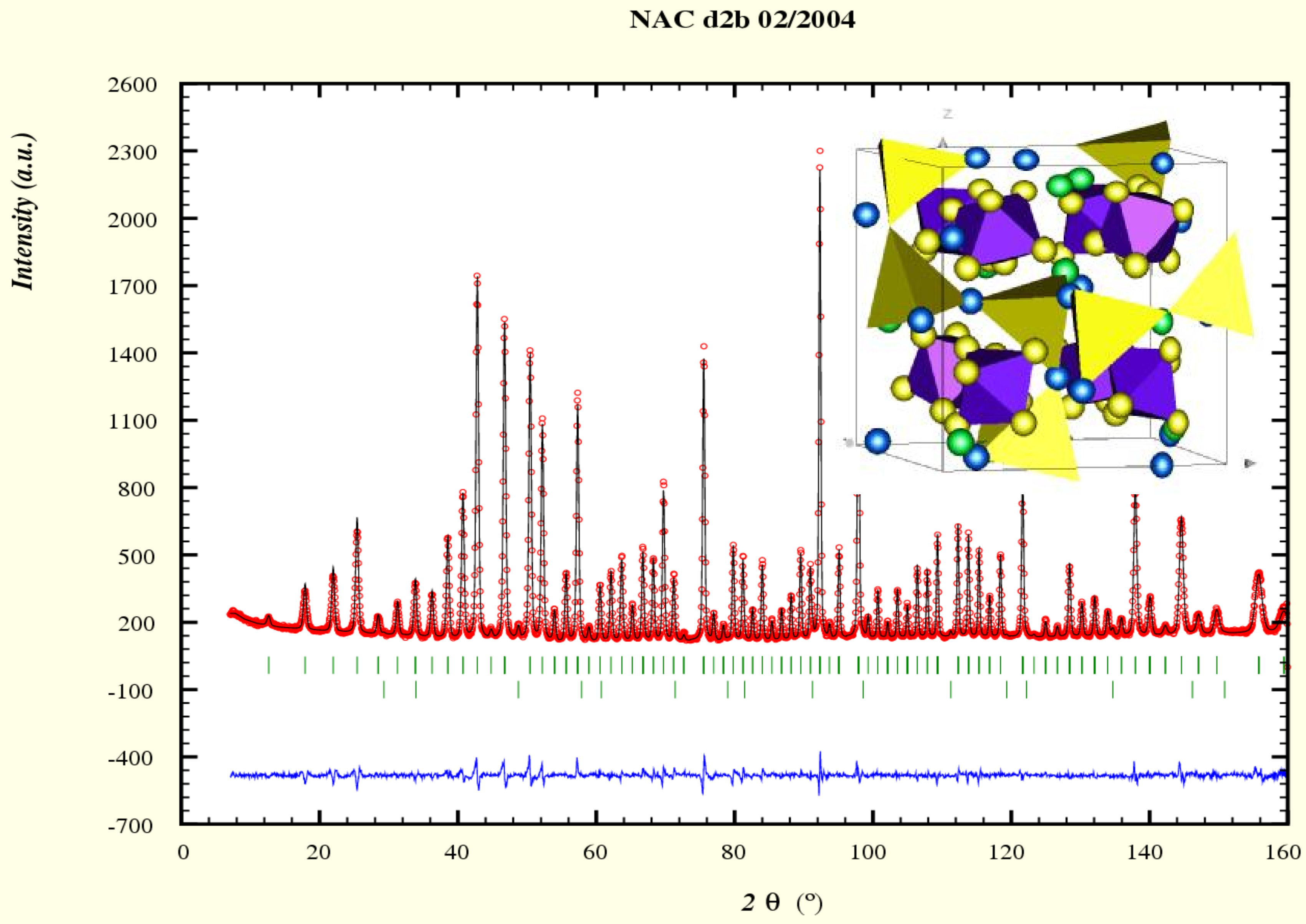


180 mg Ice sample measured at high pressure and 80K in the D2B displex (J. Finney, E.Suard)



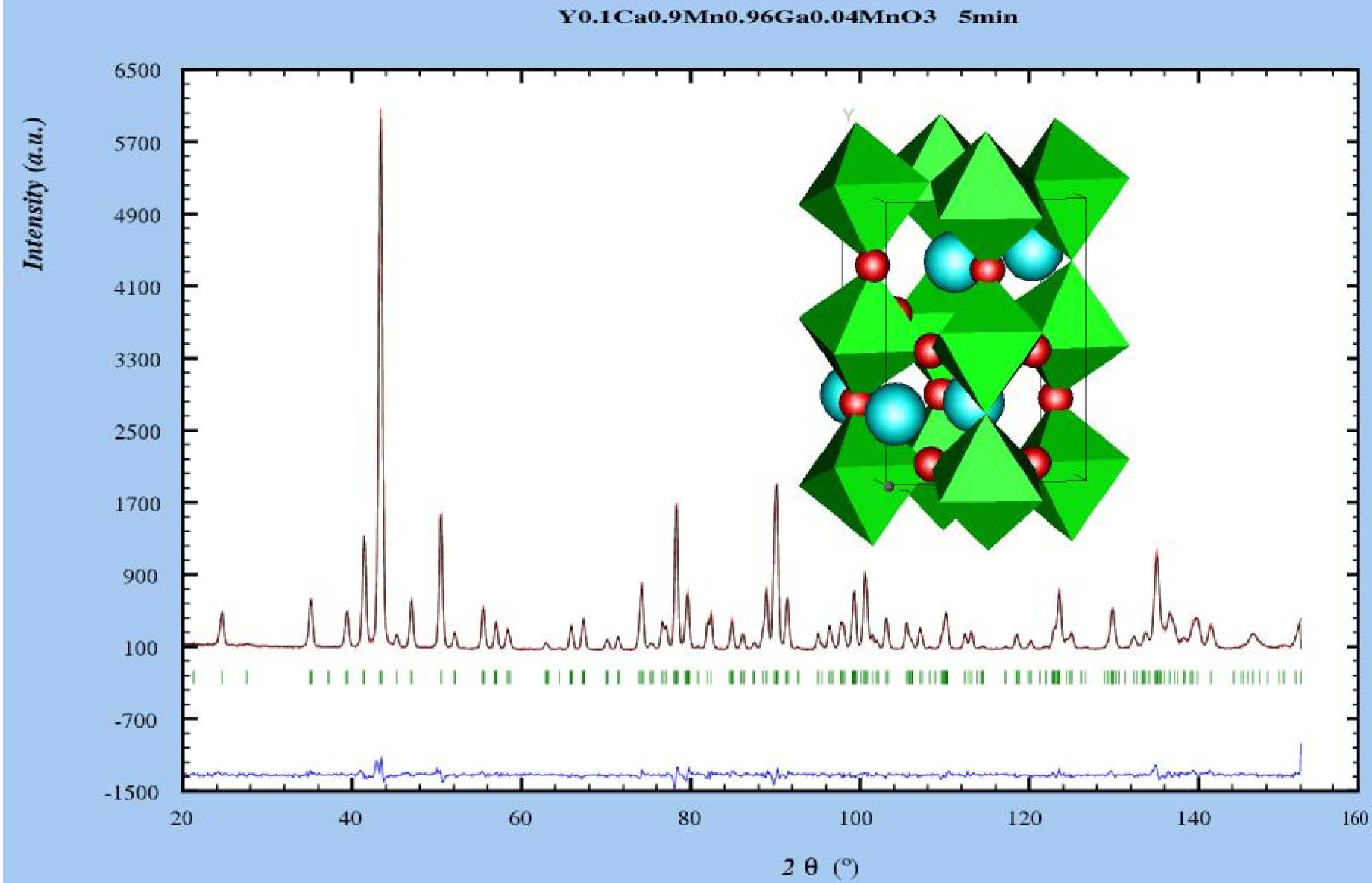
$Na_2Ca_3Al_2F_{14}$ standard on super-D2B

E. Suard, C. Ritter, ILL

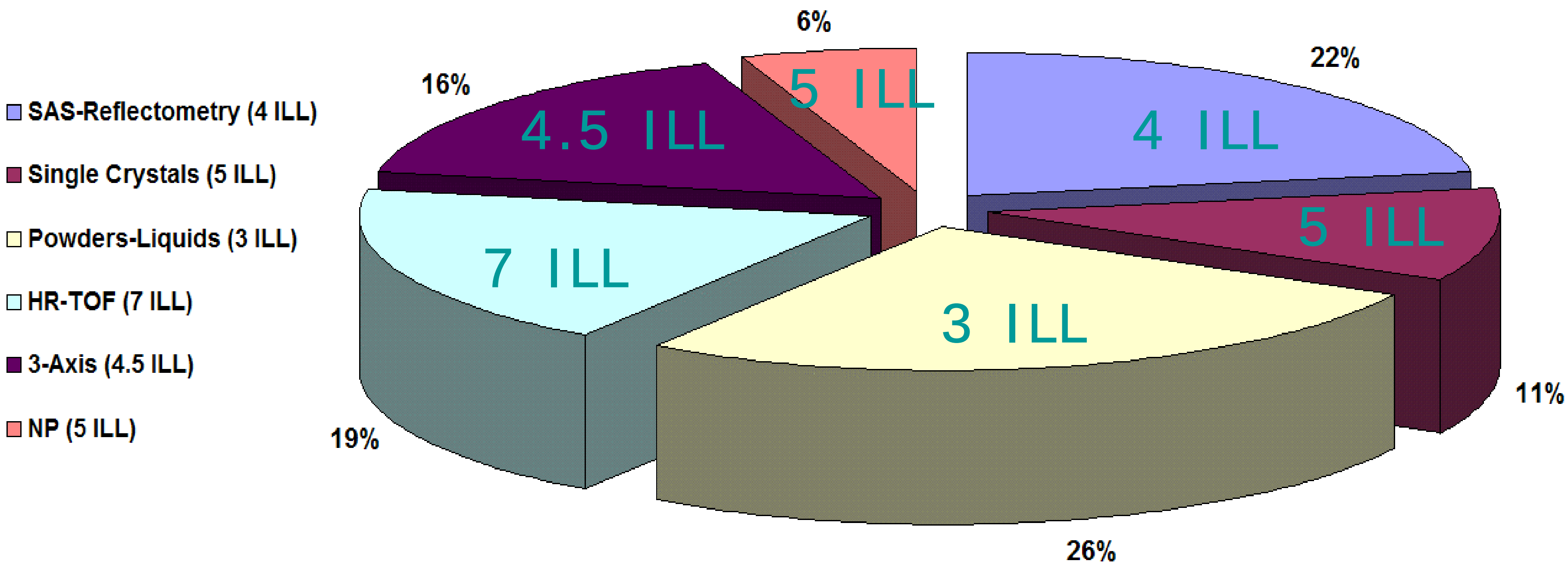


$Y_{0.1}Ca_{0.9}Mn_{0.96}Ga_{0.04}O_3$ measured in 5 min.

C. Ritter, ILL

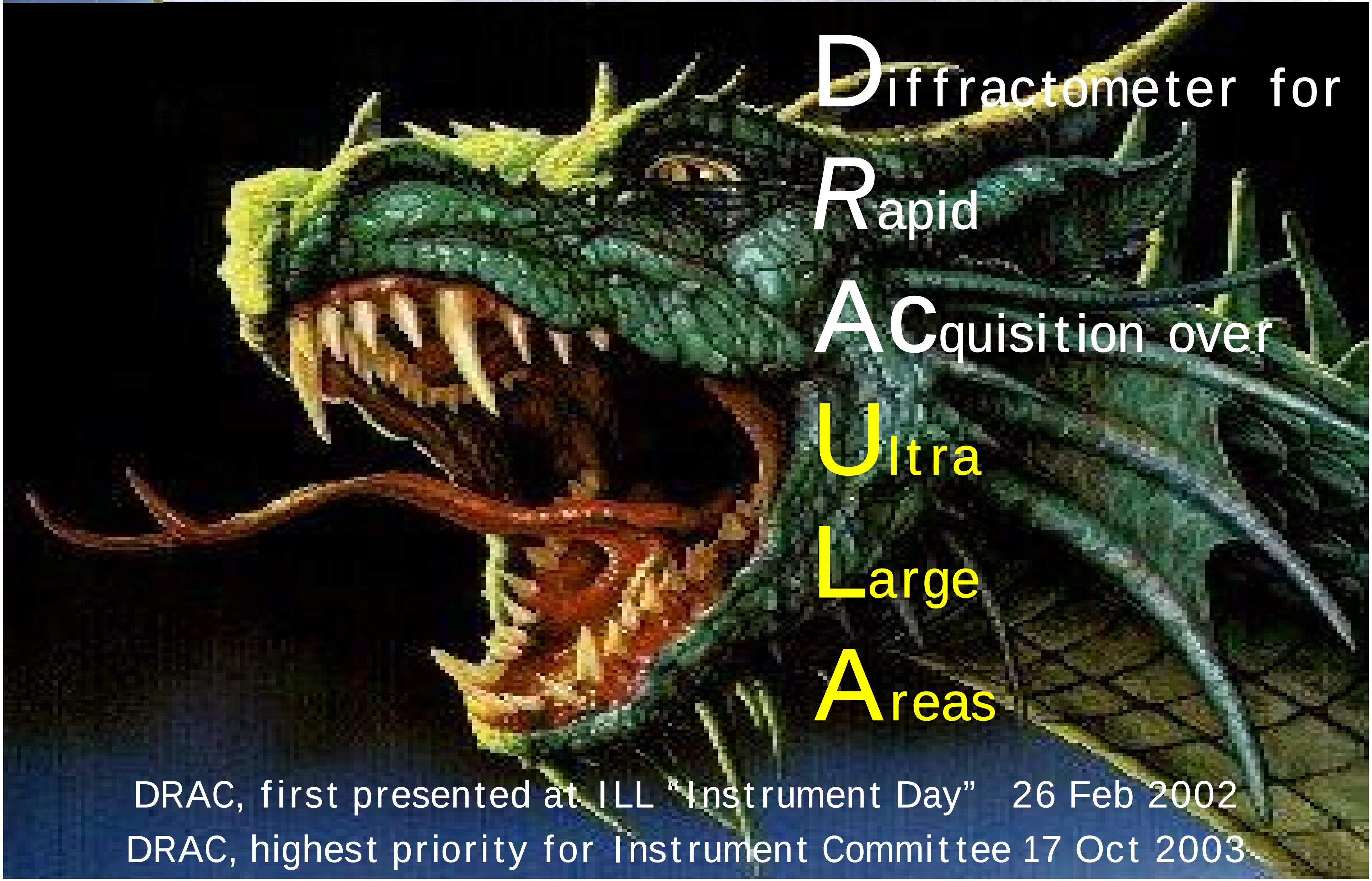


Only 3 ILL machines for more than ¼ of all ILL proposals



D2B, D4, D20

DRACULA – Ultra fast, very small samples
But only medium resolution



Diffractometer for
Rapid
ACquisition over
Ultra
Large
Areas

DRAC, first presented at ILL "Instrument Day" 26 Feb 2002
DRAC, highest priority for Instrument Committee 17 Oct 2003

Comparison of TOF & CW Diffractometers

The time-averaged Flux*Detector criterium

Then can we compete with the new US-SNS

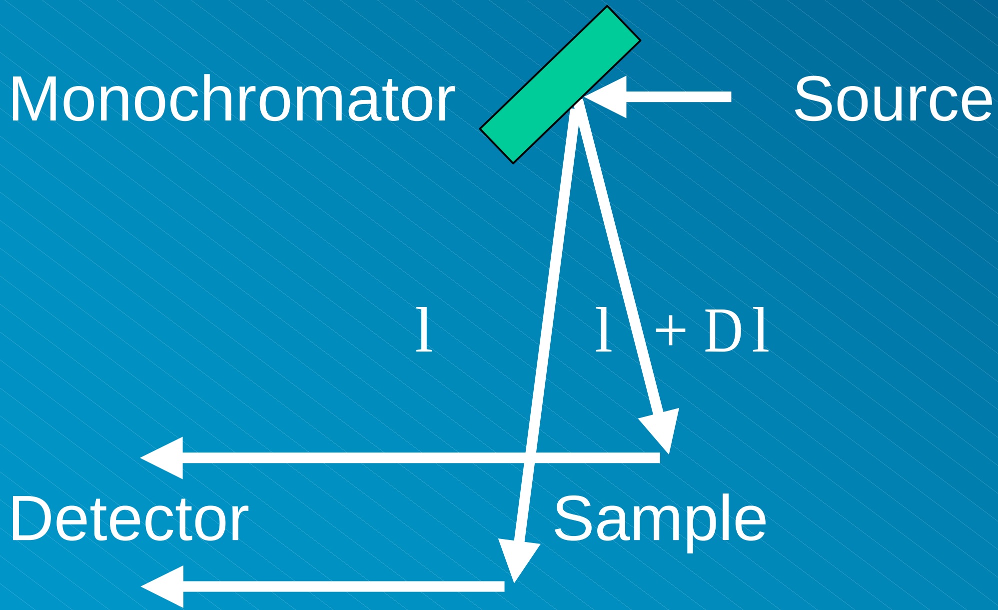
	D20	GEM	DRACULA	SNS
time averaged sample flux	$5x10^7$	$\sim 2x10^6$	$\sim 10^8$	$\sim 2.5x10^7$
detector solid angle	0.27 sr	4.0 sr	1.5 sr*	3.0 sr
efficiency	1.7	1	18	9

* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires 30^ox160^o

Why is sample flux so high from a reactor?

A: Large wavelength-band focusing monochromators ?

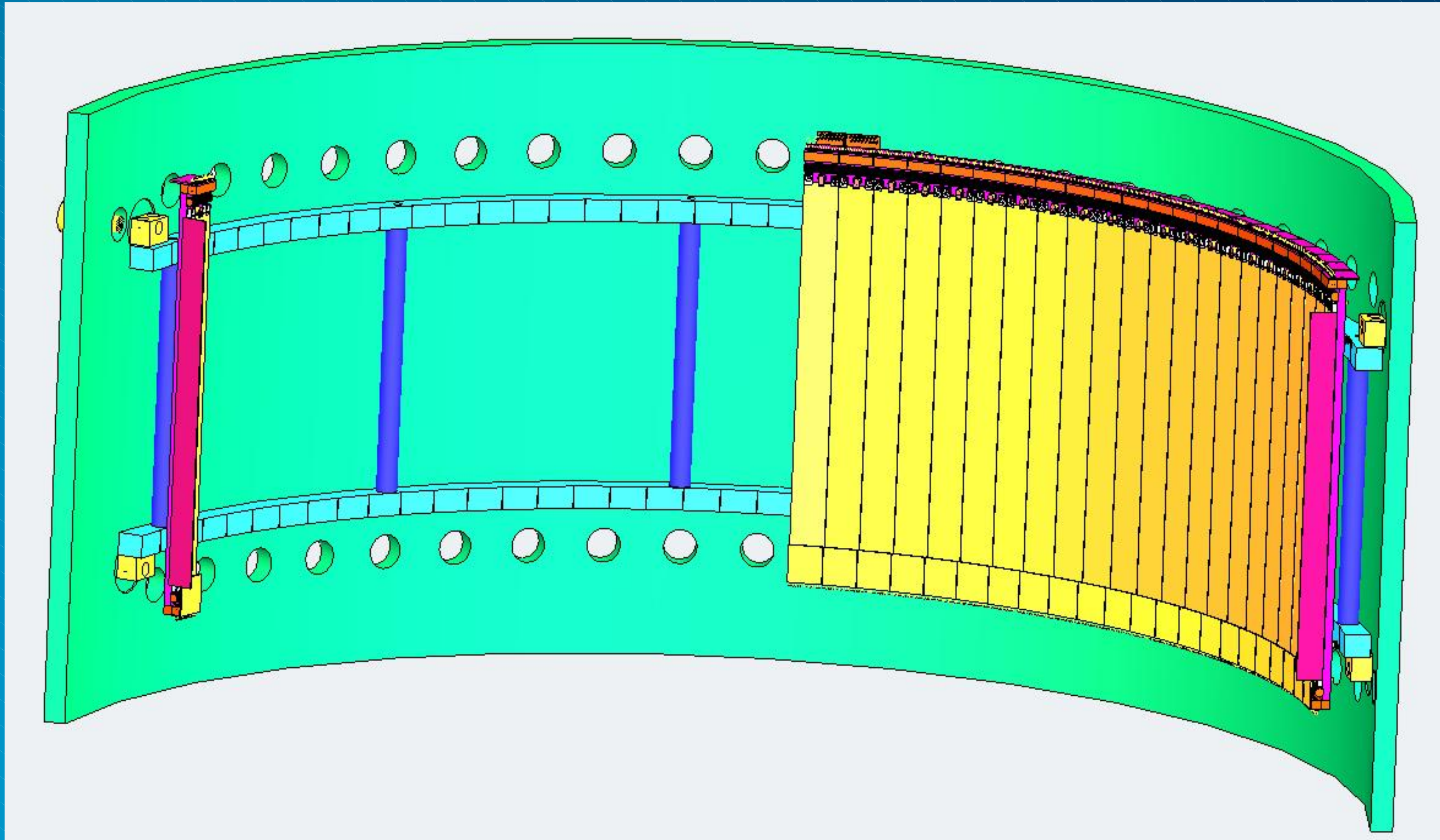
Yes ! Focusing in reciprocal space can give a factor of x10



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

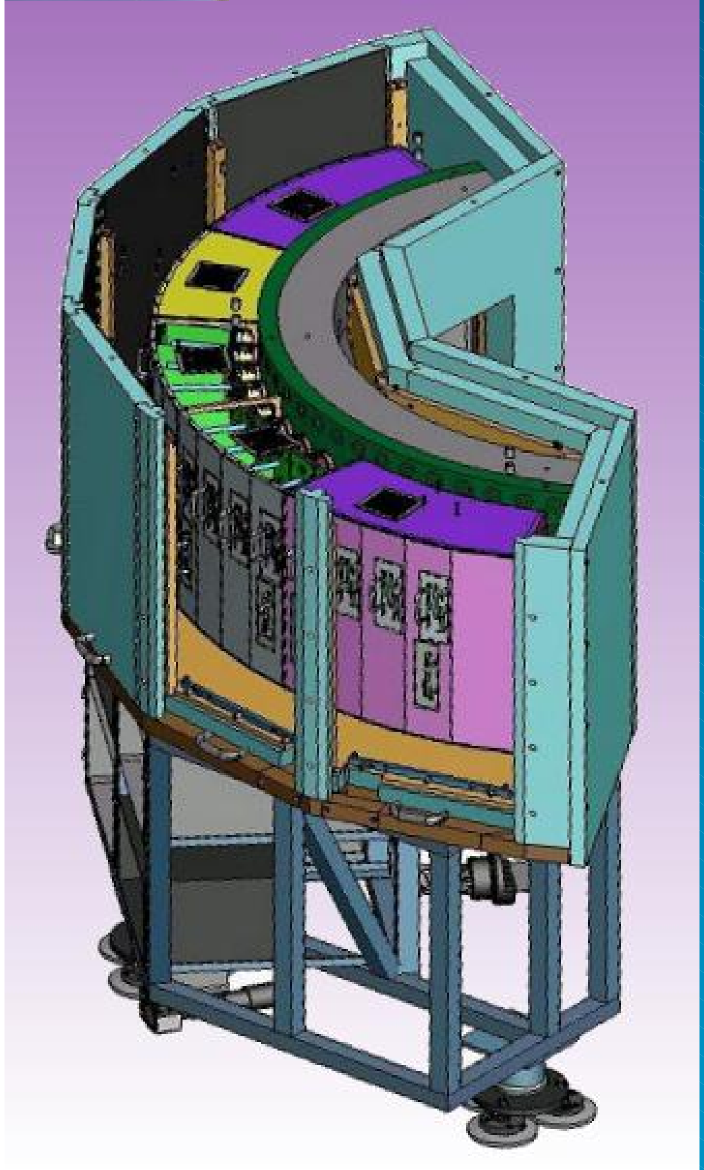
cf TOF $\Delta d/d \sim \Delta\lambda/\lambda$

D19 Millennium - A new type of large 2D multiwire detector



2D with Solid Angle > 1 steradian c.f. 0.27 on D20

DRACULA - What do we want to do ?



- Order of magnitude smaller samples than D20
- Low background (pressure cell)
- Large, compact 2D area detector (cf D19)
- Radial collimator

Large 2D area detectors for neutrons
x10 increased efficiency, existing sources

Super-D2B, new high resolution powder diffractometer
DRACULA, new high flux powder diffractometer
SPODI (Munich FRM-II) Ralph Gilles et al.