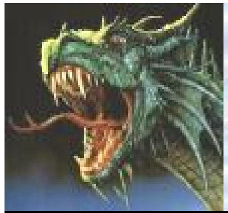
# DRAC or DRACULA ?



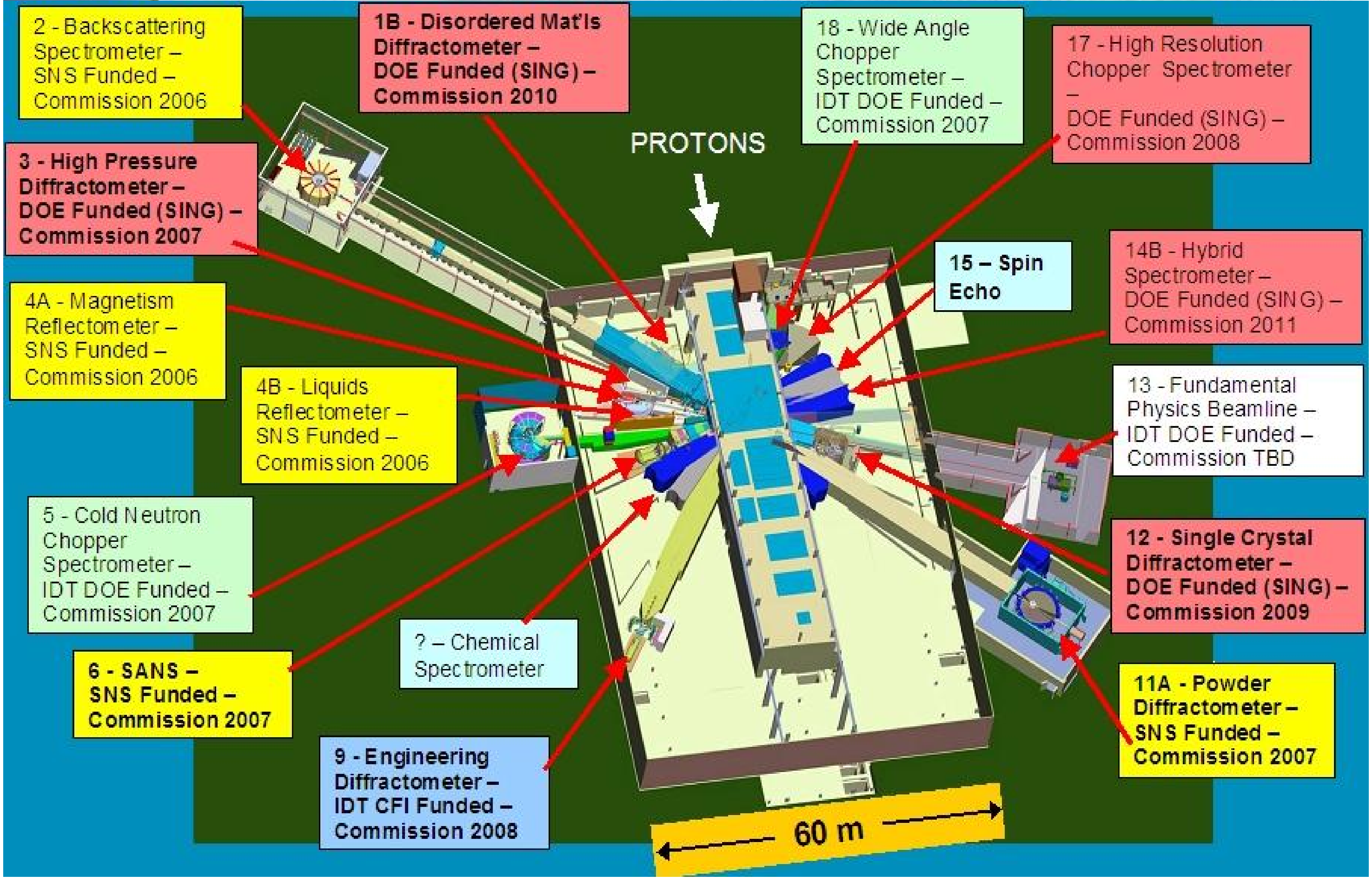
# DRAC or DRACULA ?

**D**iffractometer for
**R**apid
**AC**quisition
**U**ltra
**L**arge
**A**reas

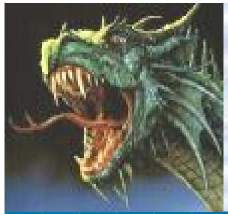
DRAC, first presented at ILL "Instrument Day" 26 Feb 2002
DRAC, highest priority for Instrument Committee 17 Oct 2003

# Hypnotised by the American SNS ?

# Instruments on the American SNS



# POW-GEN3 Powder Diffractometer at SNS





# SNS-EFAC recommendations

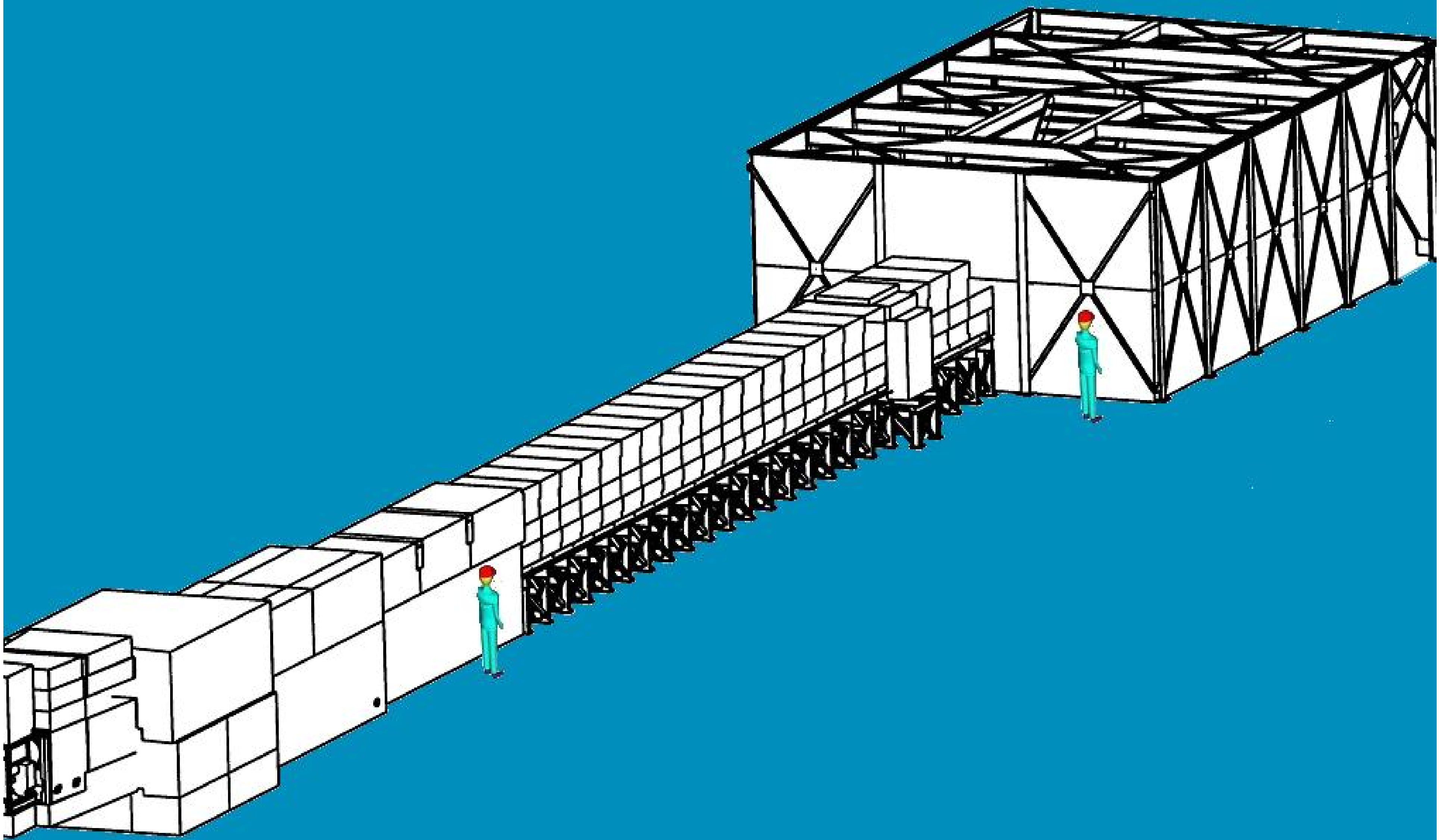
- "…the SNS should immediately begin work on the conceptual design for… a third generation powder diffractometer with a resolution $\Delta d/d$ of ~$1 \times 10^{-3}$ at 90º." Nov 1998.

Note: Moderately high resolution at 90º scattering.

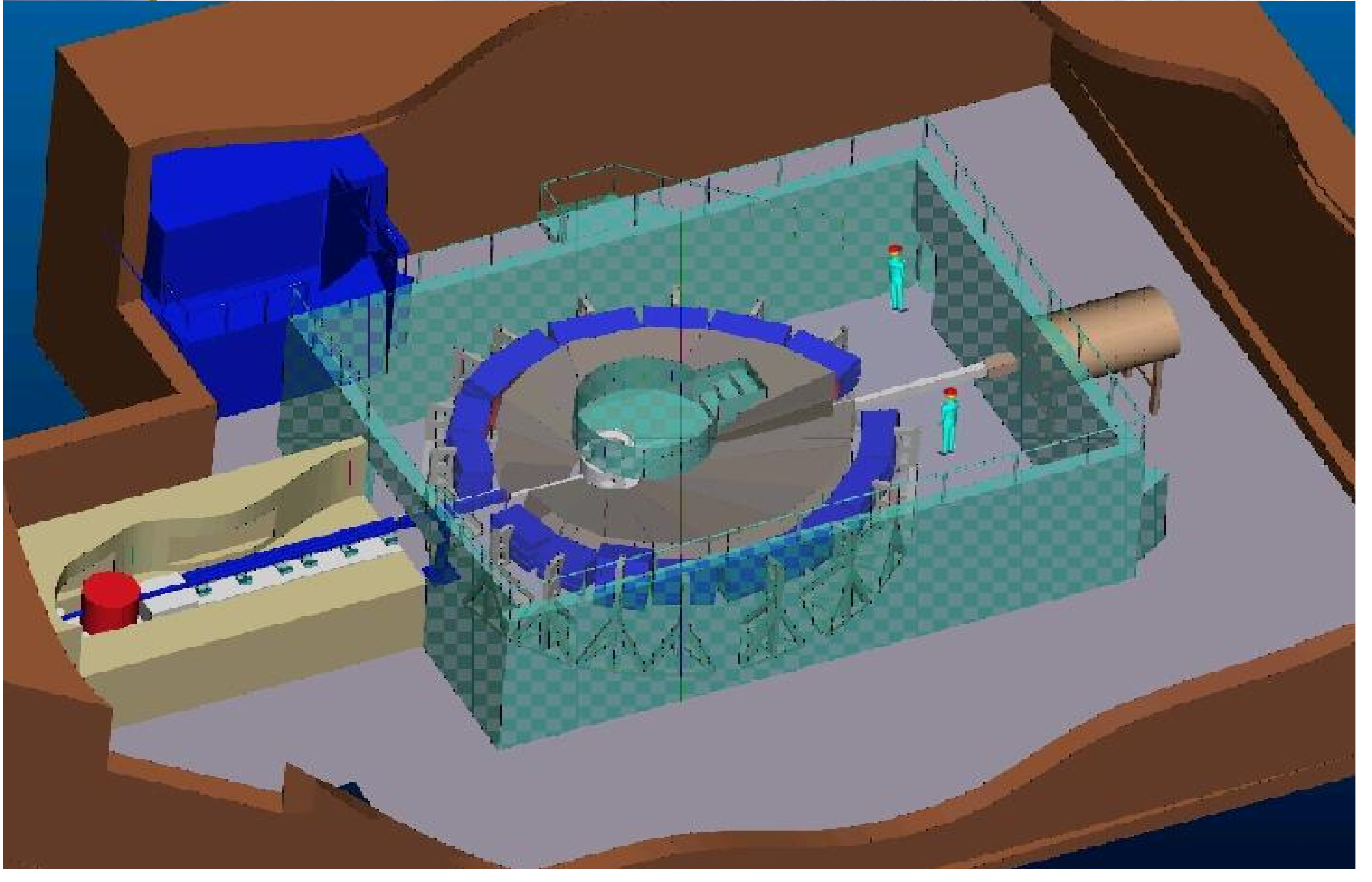
- "SNS without a world-class powder diffractometer on day one is unthinkable." May 2001.

- "1.1 Recommendation: A high level of priority should be assigned to bringing the powder diffractometer (POW-GEN3) into operation as early as possible." Dec 2003.

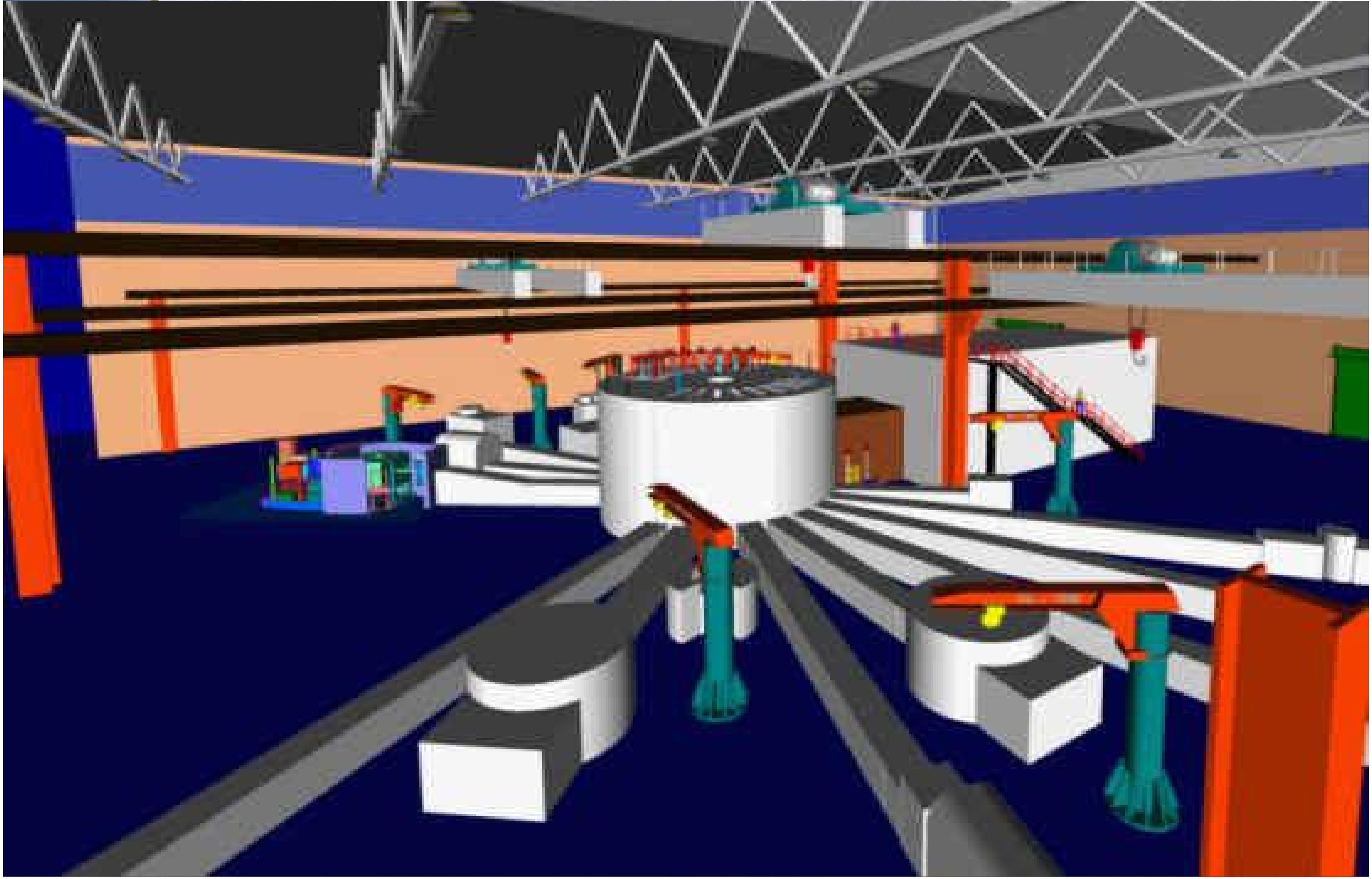
# POW-GEN3 at SNS – a BIG detector



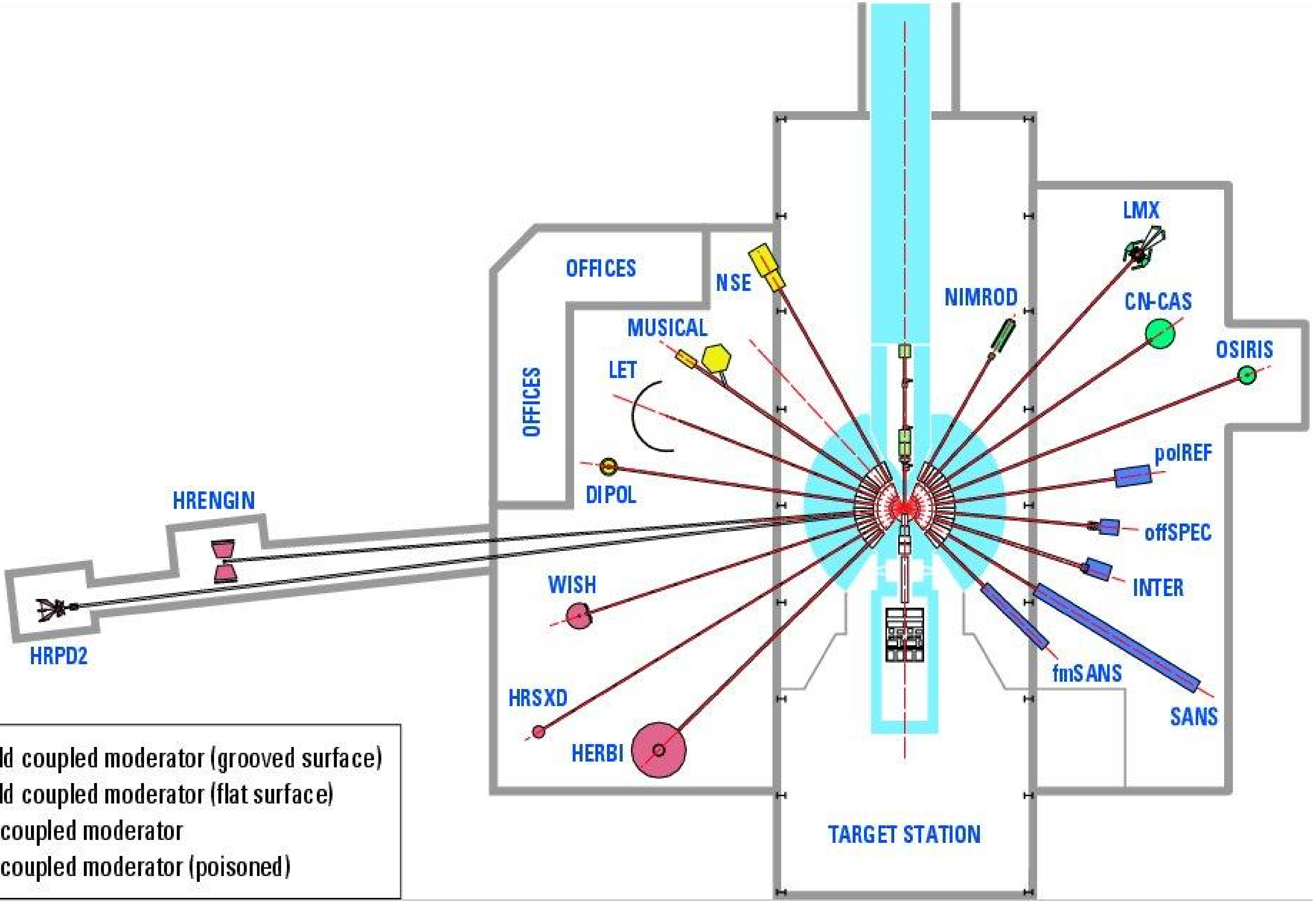
# POW-GEN3 at SNS – a BIG detector



# Target Station-2 at ISIS

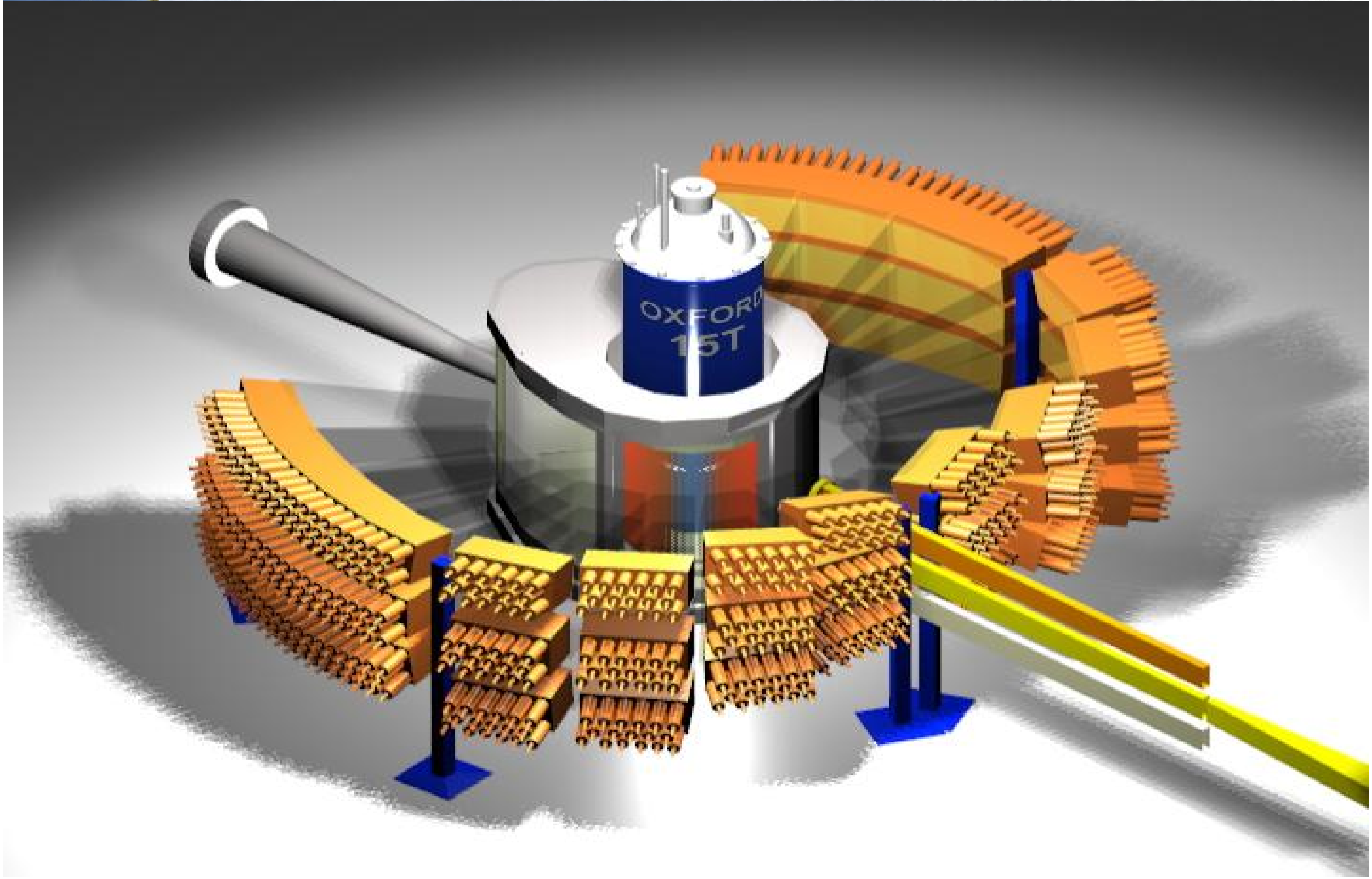


# Target Station-2 at ISIS




Cold coupled moderator (grooved surface)
Cold coupled moderator (flat surface)
Decoupled moderator
Decoupled moderator (poisoned)

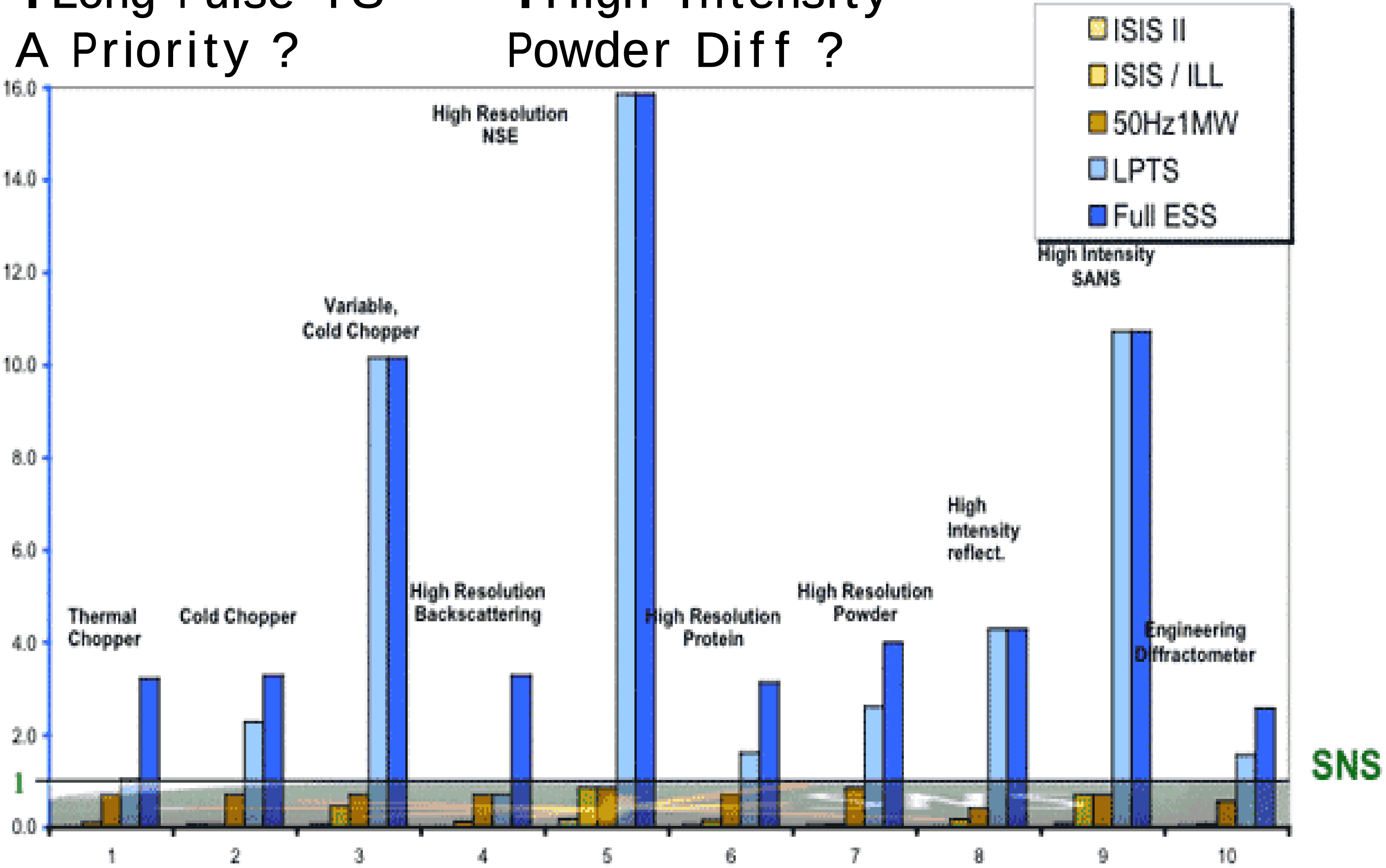
# WISH Powder Diffractometer, TS2-ISIS

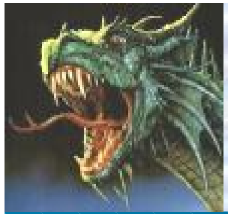


# While we are waiting for the ESS dream...

# While we are waiting for the ESS dream…

- Long Pulse TS A Priority ?
- High Intensity Powder Diff ?







## Proposed ESS Powder Diffractometers

- **ST05 High-Q Powder Diffractometer** — **HQP**
- **ST06 Liquids & Amorphous Diffractometer** — **LAD**
- **SM10 Single Pulse Diffractometer** — **SPD**
- **SD17 Magnetic Powder Diffractometer** — **MagP**
- **SD18 High Resolution Powder Diffractometer** — **HRPD**
- **LM05 Ultra-high Resolution Powder Diffractometer** — **URPD**
- **LM06 High Pressure Powder Diffractometer** — **HiPD**

# ESS Instrumentation Group Reports, May 2001

## Powder Diffraction Instruments

P.G. Radaelli, S. Hull , H.J. Bleif & A. M. Balagurov

- Given the choice, the 50Hz target is always better than the 10Hz one.
- We dearly miss a truly sharp cold moderator, especially for crystallography requiring high and low Q at the same time.
- The 50Hz target would be the first choice for all powder (and probably single-crystal) diffractometers.

# Powder Diffractometer Efficiency

## ESS Instrumentation Group Reports, May 2001

Powder Diffraction Instruments

P.G. Radaelli, S. Hull , H.J. Bleif & A. M. Balagurov

The average count rate is the product of:

- the flux at the sample
- the detector solid angle and efficiency
- the sample volume

## ESS Instrumentation Group Reports, May 2001

Powder Diffraction Instruments

P.G. Radaelli, S. Hull , H.J. Bleif & A. M. Balagurov

- Reactor-based instruments tend to maximise the source solid angle, by exploiting focusing monochromators (flux)
- Diffractometers at pulsed sources tend to have much larger detector solid angles.

# ESS Instrumentation Group Reports, May 2001

Powder Diffraction Instruments

P.G. Radaelli, S. Hull , H.J. Bleif & A. M. Balagurov

Conclusions:

- TOF machines need a higher flux source (SNS,ESS)
- CW machines need a larger detector
  (which is somewhat less expensive).

# Comparison of TOF & CW Diffractometers

Jorgensen, J.D., Cox, D.E., Hewat, A.W., Yelon, W.B (1984)

"Scientific opportunities with advanced facilities for neutron scattering"

Shelter Island Workshop, 1984

Nuclear Instruments and Methods in Physics Research B12 (1985) 525-561

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

The time-averaged Flux*Detector criterium

D20 has high flux, GEM has a big detector

| | D20 | GEM |
|---|---|---|
| time averaged sample flux | $5x10^7$ | $\sim 2x10^6$ |
| detector solid angle | 0.27 sr | 4.0 sr |
| efficiency | 1.7 | 1 |

# Comparison of TOF & CW Diffractometers

The time-averaged Flux*Detector criterium

So, let's use a big detector too !

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

* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires $30^o x 160^o$

The time-averaged Flux*Detector criterium

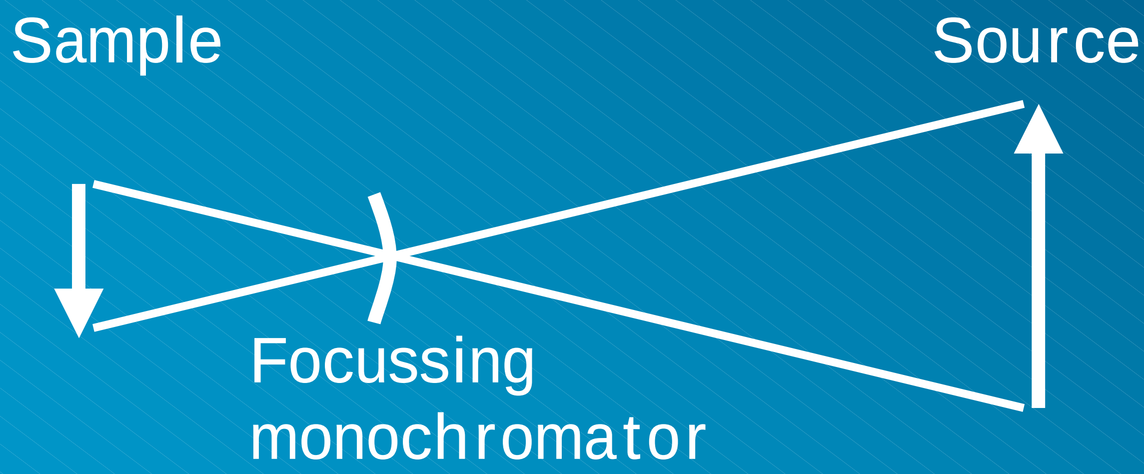
Only then can we compete with the SNS.

| | 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 $30^o x 160^o$

A: Large vertically focusing monochromators ?

No ! Focusing in real space only gives a factor of x2 or x3

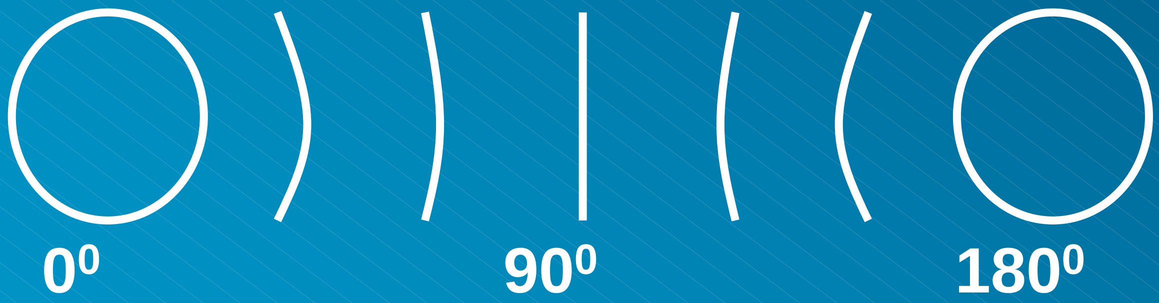


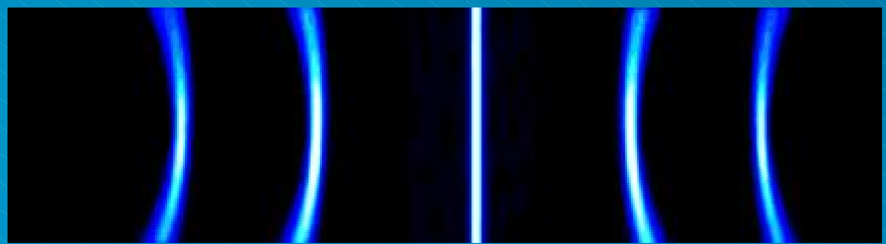

cf use of convergent guide with TOF

# Why is sample flux so high from a reactor?

## A: Large vertically focusing monochromators ?

Slight smearing of diffraction cones due to monochromator vertical divergence (esp. at very low and very high angles).



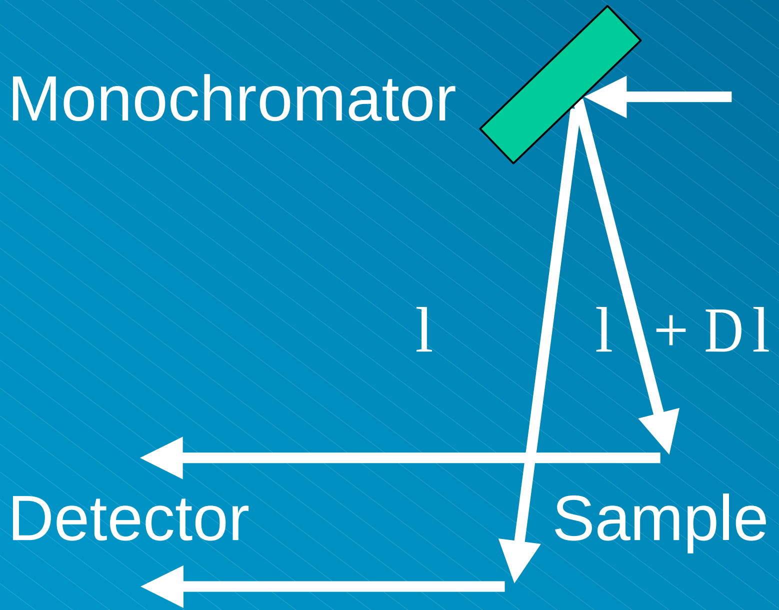



Not a big problem for medium resolution

# 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

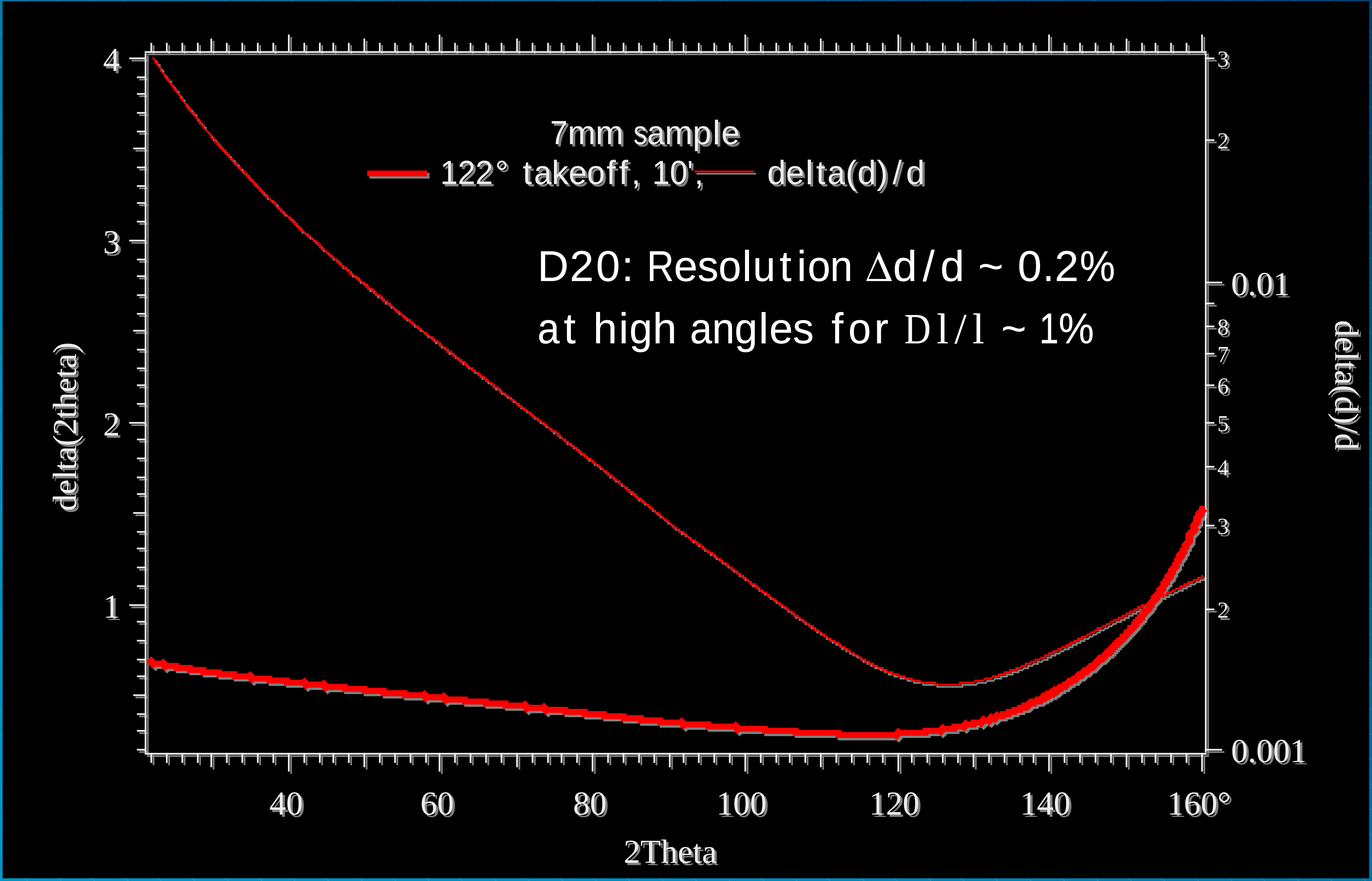



$\Delta d/d$ ~0.1% for $\Delta\lambda/\lambda$ ~1%

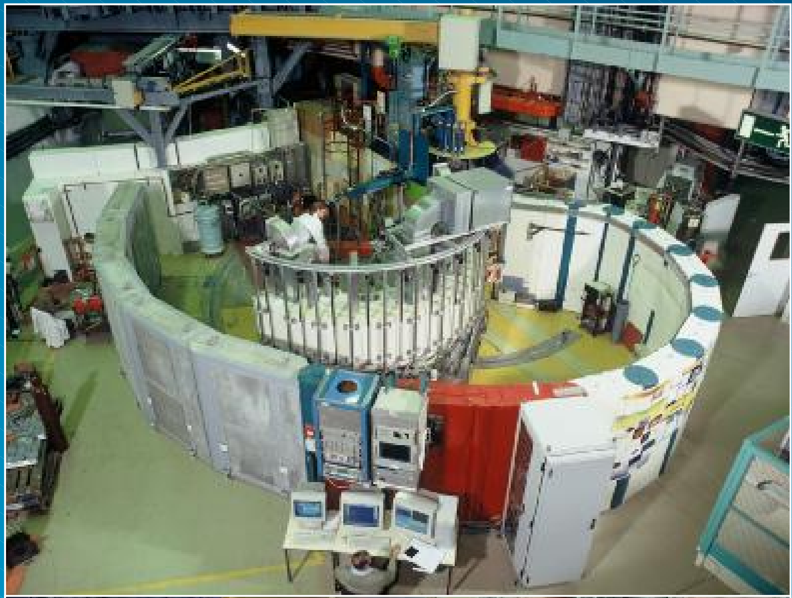
cf TOF $\Delta d/d \sim \Delta\lambda/\lambda$

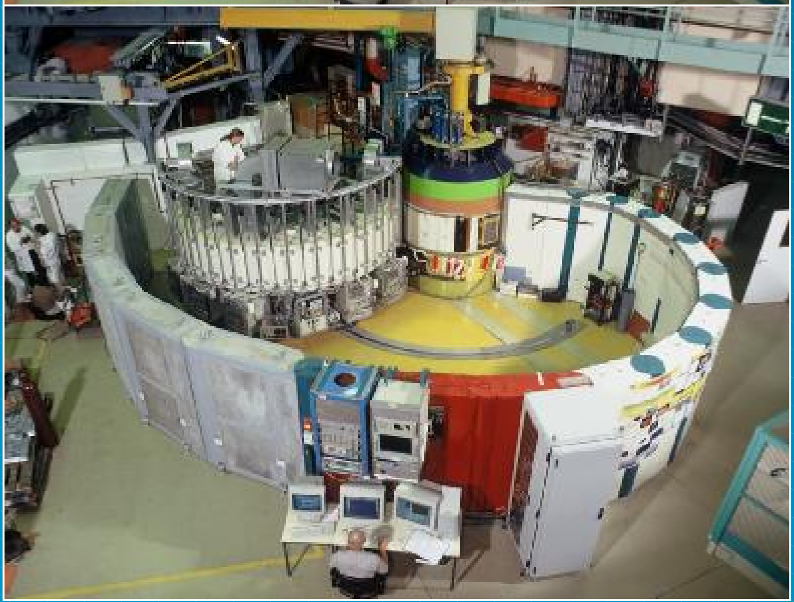
# Does large Dl/l mean low resolution ?

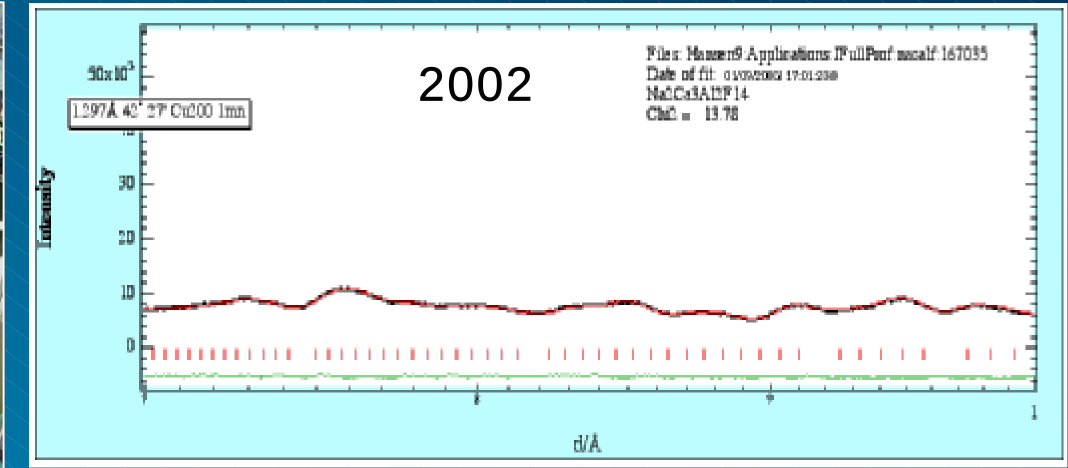
A: No. Resolution is INDEPENDENT of $\Delta\lambda/\lambda$ at focusing angle.



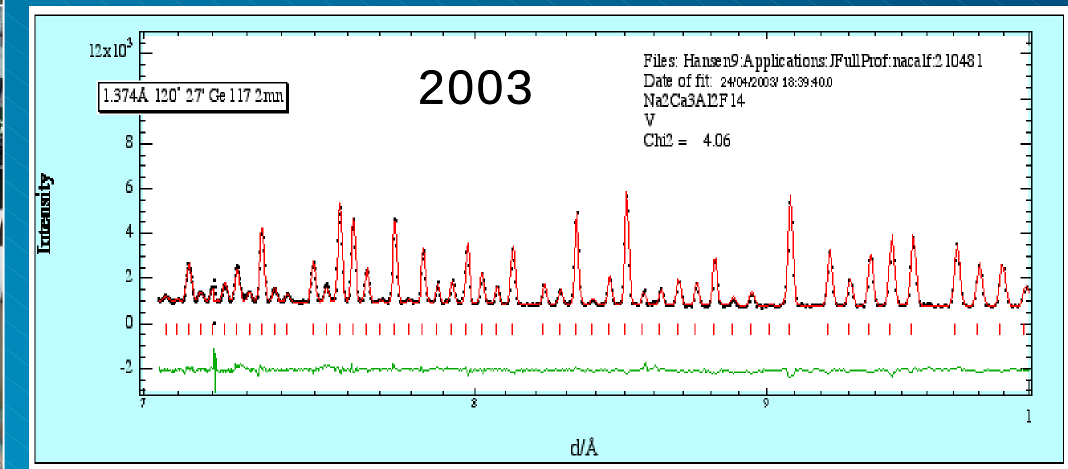
# D20 – Good Resolution but still very fast








Before and After (data in 2 min.)

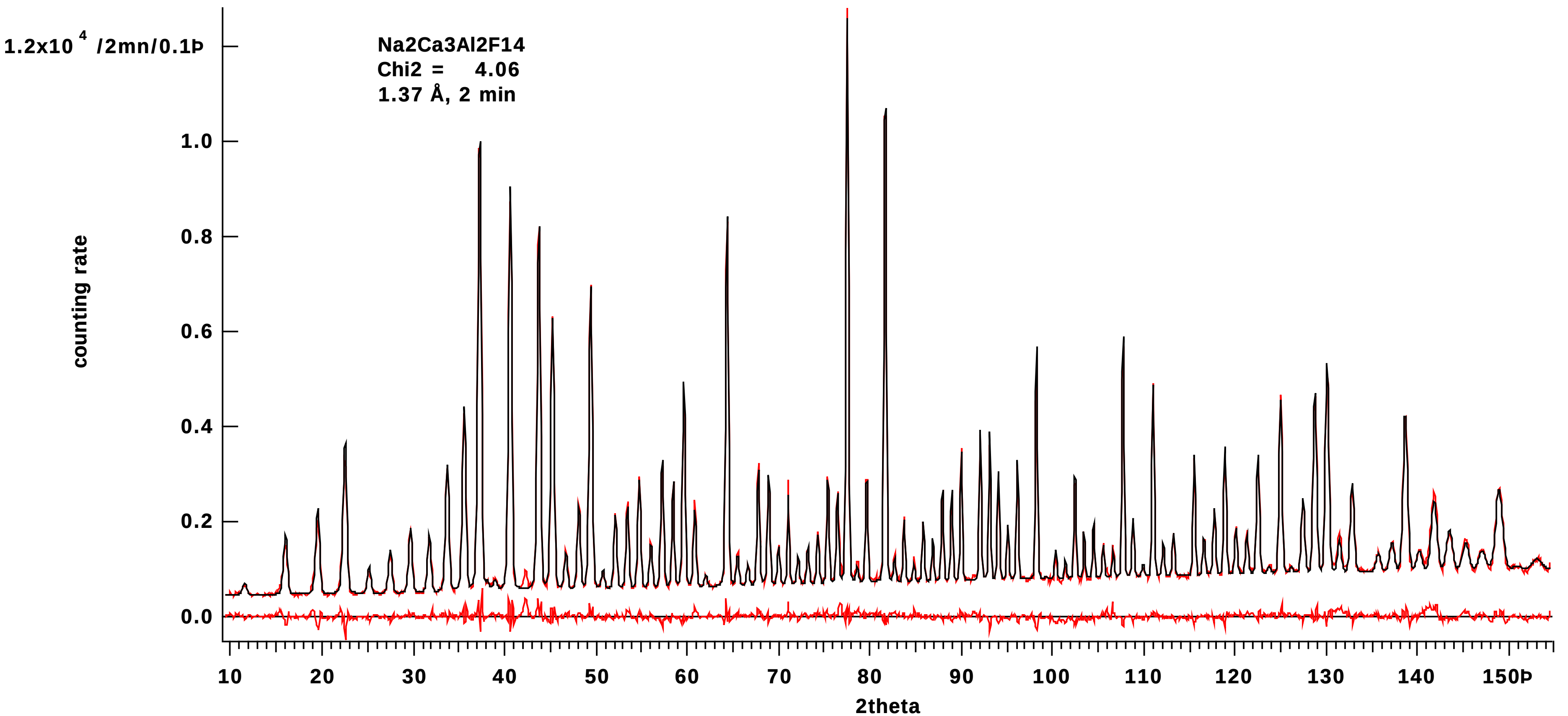



Higher D20 resolution since 2003

# D20 – Good Resolution but still very fast

Thomas Hansen (2003) ILL News, June 2003

2 minute D20 data for a ~700 $mm^3$ sample of $Na_2Ca_3Al_2F_{14}$



# GEM at ISIS, comparable to D20

Radaelli, Hammon & Chapon (2003) Neutroni e Luce di Sincrotrone

~700 minute GEM data for a 2mm$^3$ sample of $Y_3Al_5O_{12}$

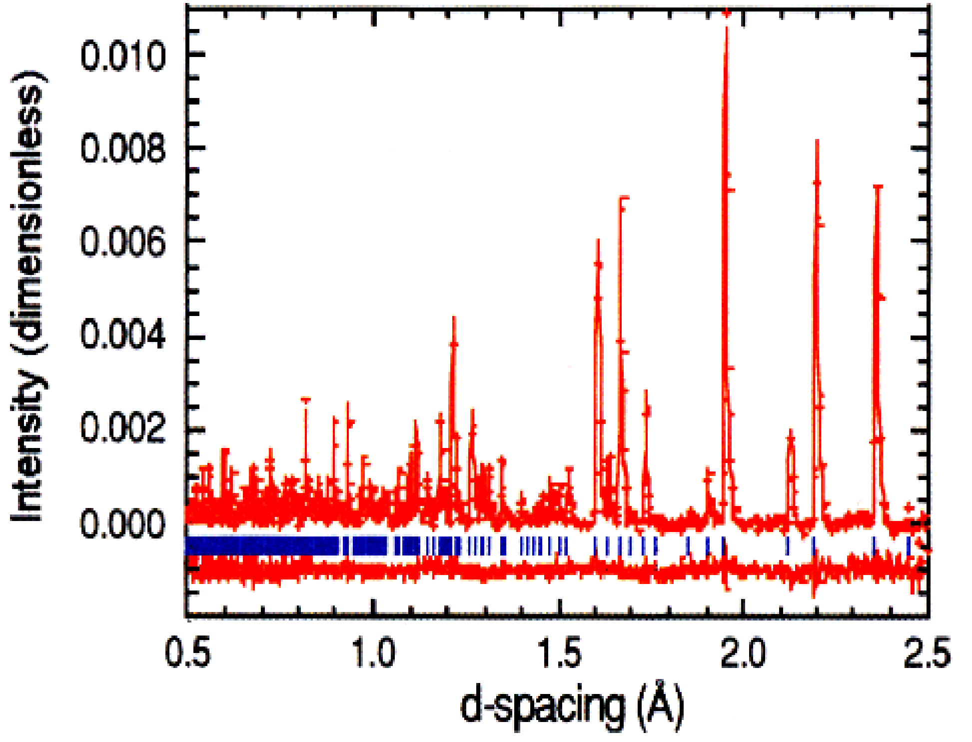
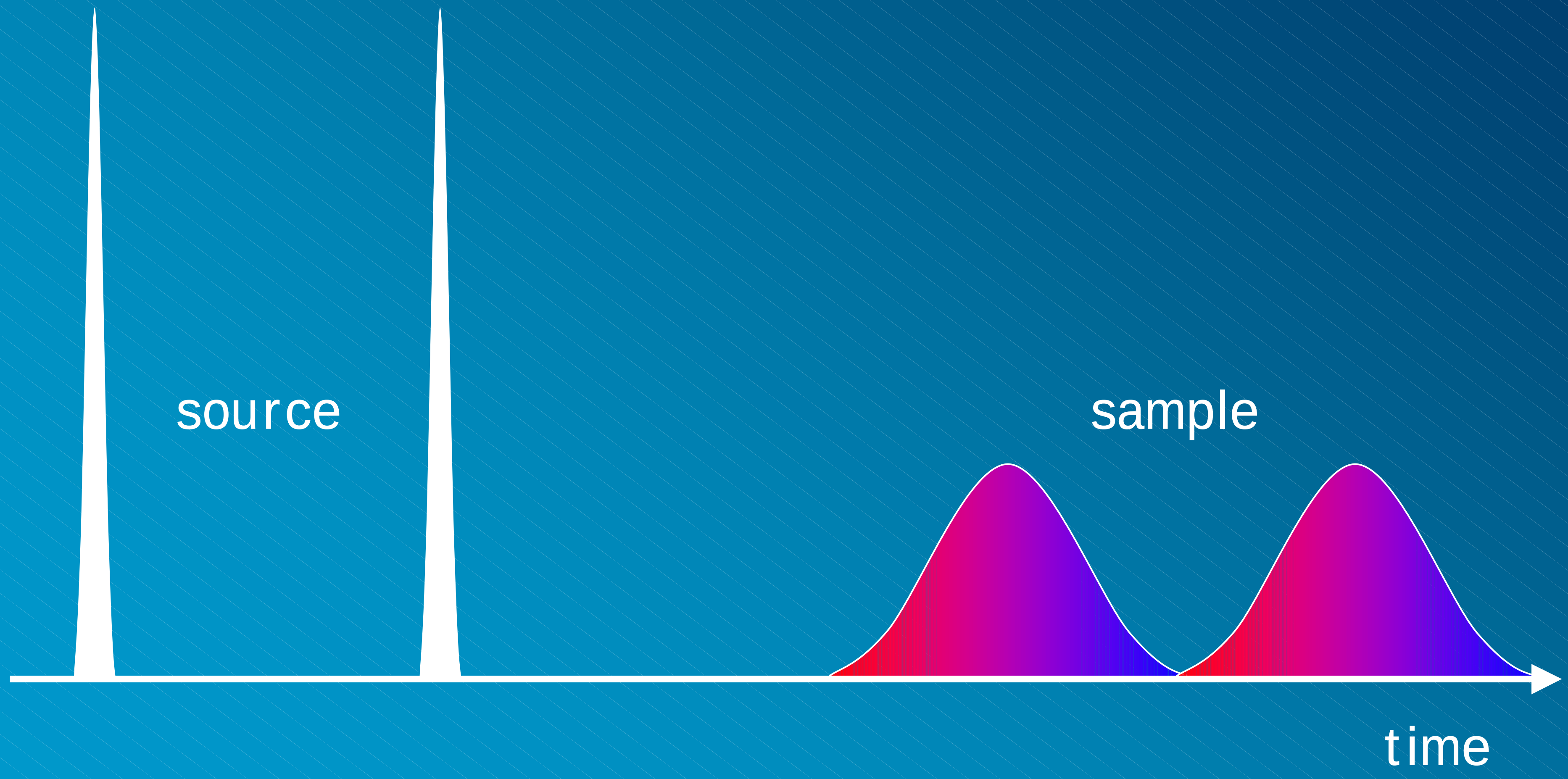


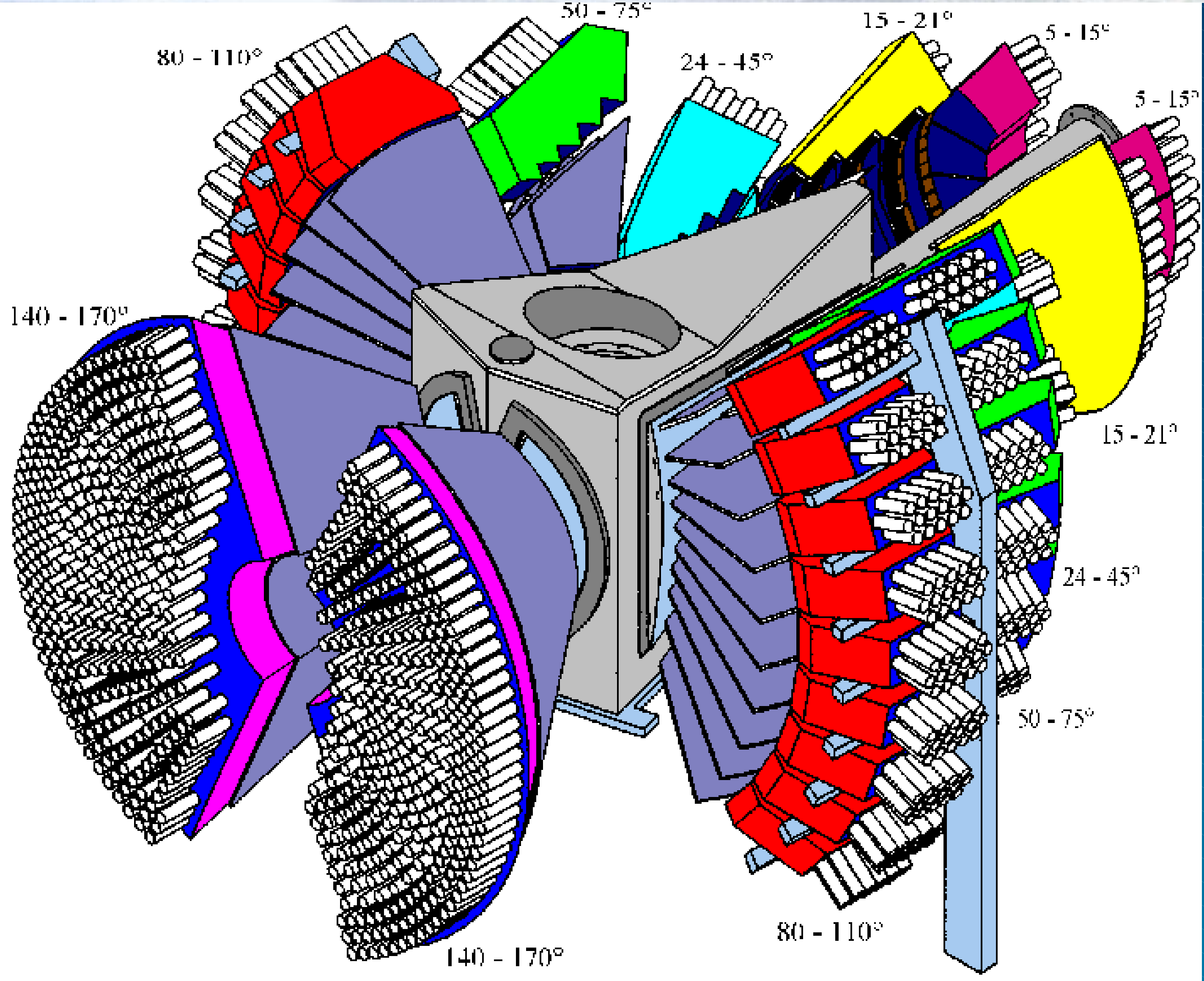

Fig. 7. Rietveld Refinement plot for a 2 mm$^2$ sample of Yttrium Iron Garnet (YAG), after an overnight data collection.

# Why is sample flux low on a pulsed source ?

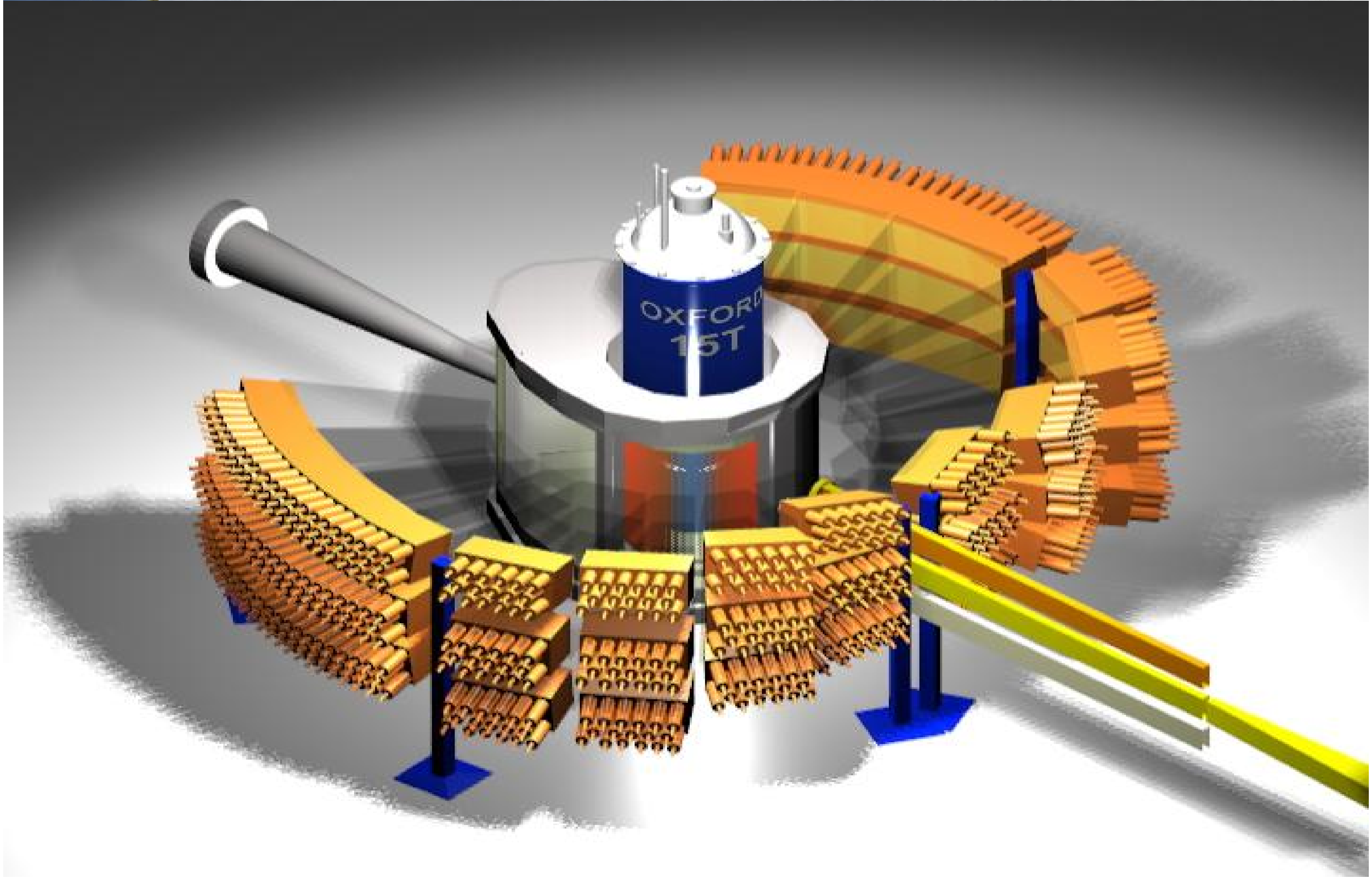
A: The sharp white peaks spread out in wavelength with time
TOF machines have rather constant (low) flux at sample



# TOF machines need big detectors
# GEM Powder Diffractometer, TS1-ISIS

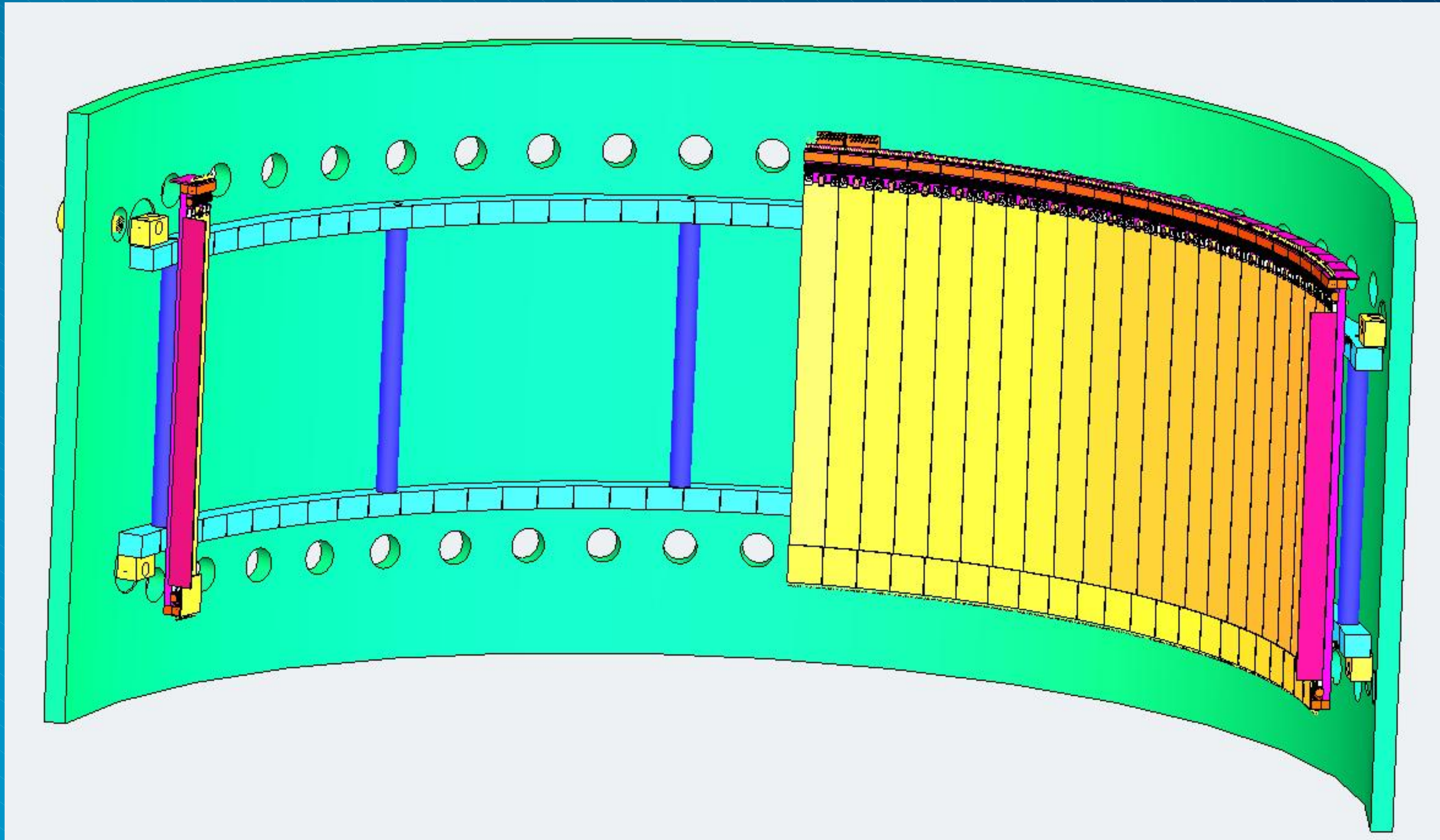


# TOF machines need big detectors
# GEM Powder Diffractometer, TS1-ISIS

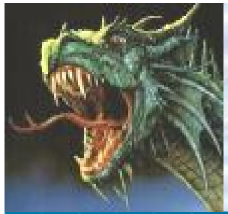


# Can we have a big detector too please?

## D19 Millennium - A Revolution in large 2D Gas Detectors



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



# Comparison of TOF & CW Diffractometers

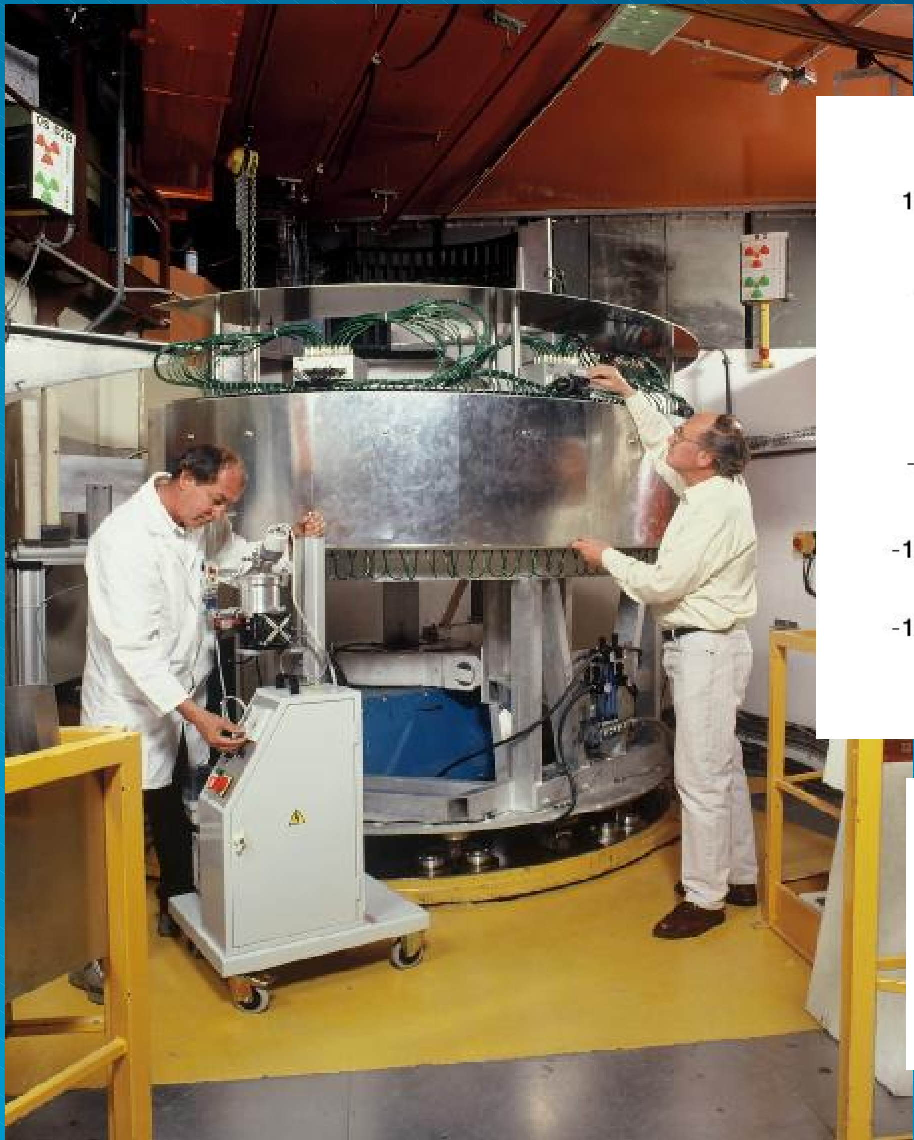
## The time-averaged Flux*Detector criterium

| | 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 |

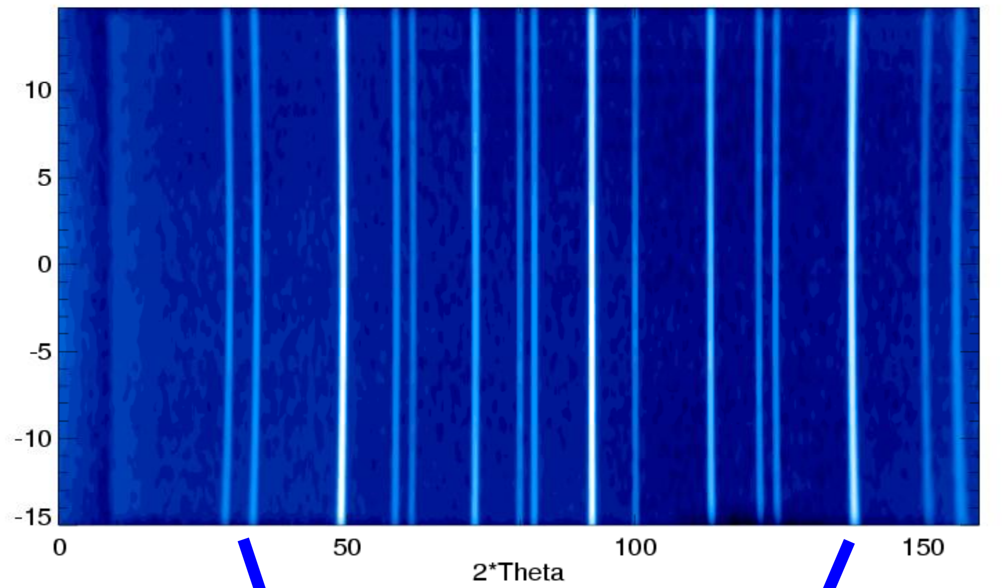
* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires $30^o x 160^o$

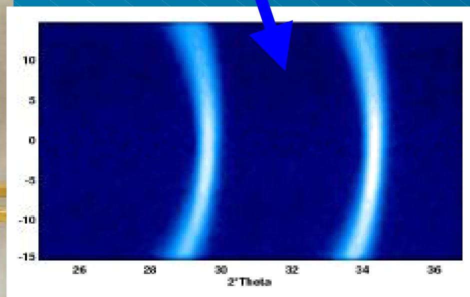
# Array of linear wire PSD-tubes on Super-D2B at ILL

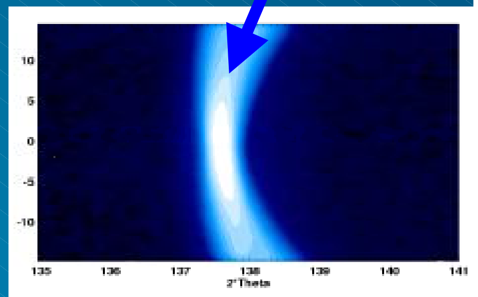
# 2D detectors for CW Powder Diffraction



## UK-EPSRC Super-D2B project at ILL

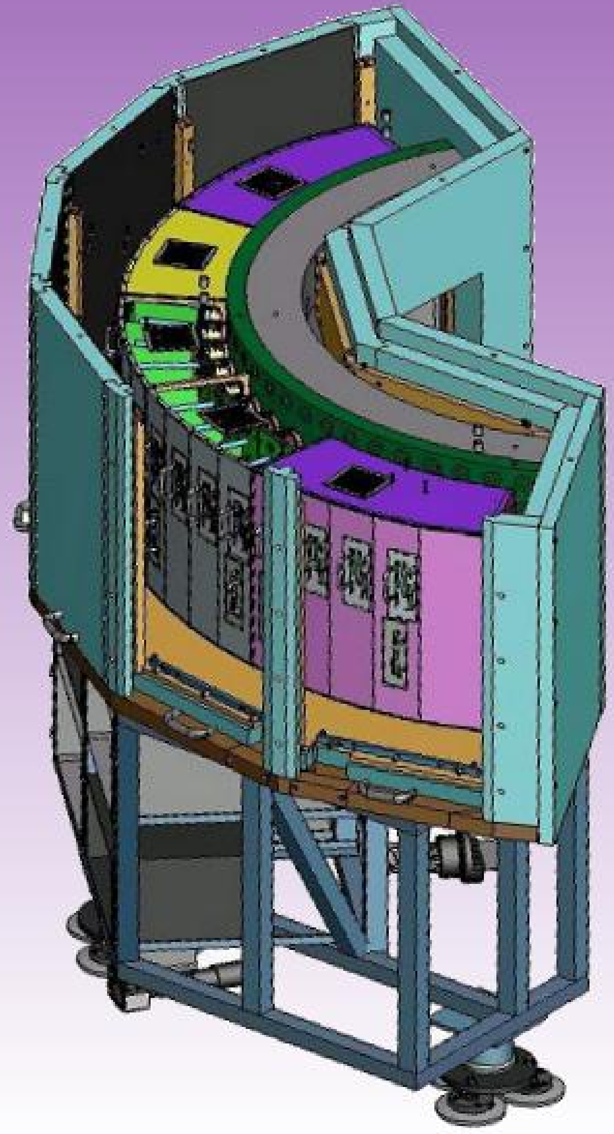




E.Suard, C.Ritter, A.Hewat, P.Attfield... (Edinburgh)

# DRACULA - What do we want to do ?

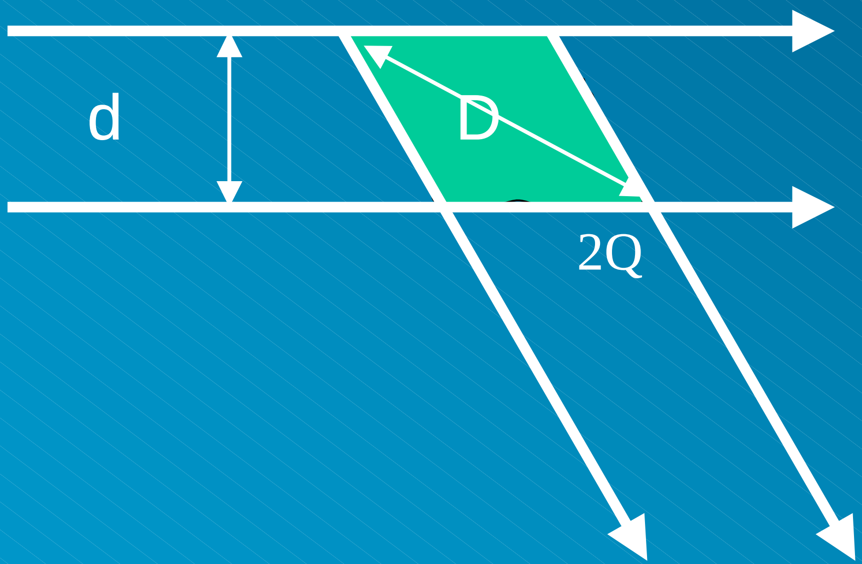


- Order of magnitude smaller samples than D20
- Low background (pressure cell)
- Large, compact 2D area detector (cf D19)
- Radial collimator

# DRACULA – Small Samples, Low Background

## Large detector & radial collimator near 90º scattering

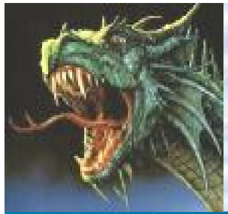
± 15º vertical as for the new D19 detector cf ±7º for new Paris-Edinburgh cell
± 30º horizontal ie 2Q = 60º - 120º (range of scattering angles for pressure cell)




d = diameter of the incident beam
D = diameter of scattering volume
= d/sinQ
= dÖ2 (minimum at 2Q = 90º)
= 2d (maximum at 2Q = 60º & 120º)

D = 5mm – 7mm for 2Q = 60º - 120º

**Scattering limited to a very small sample volume**



# DRACULA – Small Samples, Low Background

## Can we obtain all d-spacings with a $2\Theta$ range of 60º-120º ? (i.e. with a very small scattering volume)

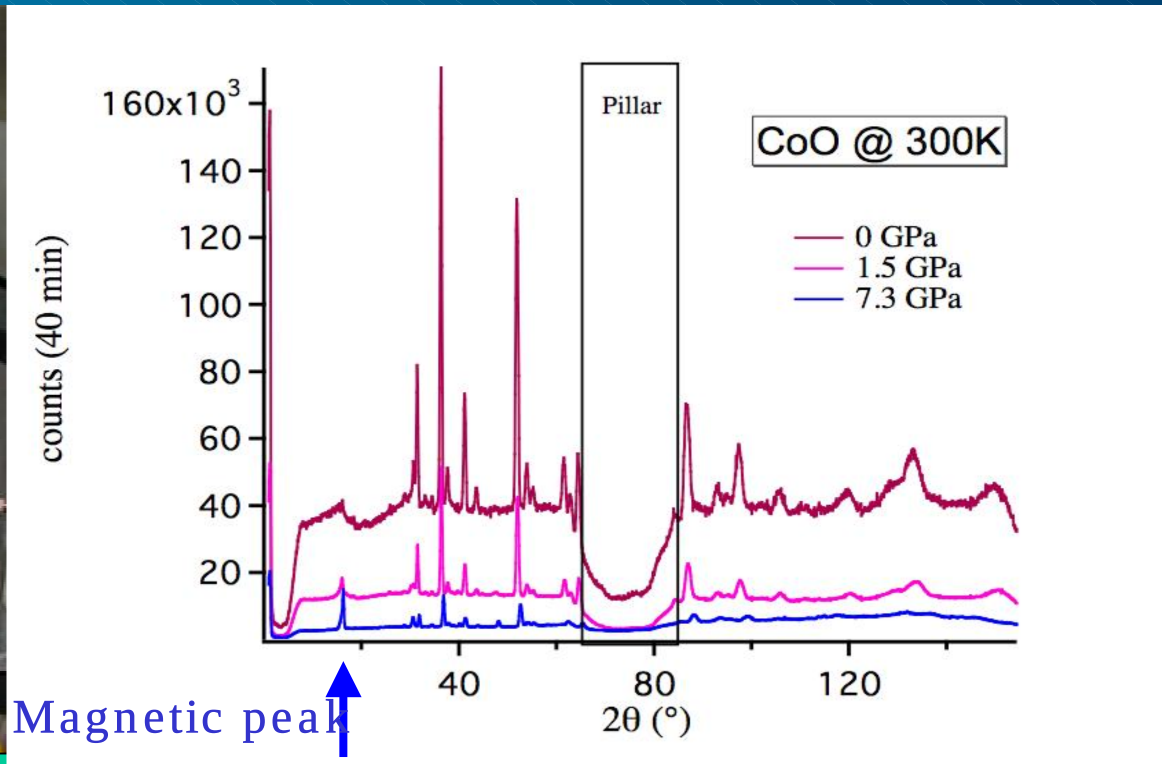
Use a large focusing Ge monochromator near 90º to obtain several $\lambda$

| | | |
|---|---|---|
| [115] -> 1.54Å; | | d= 0.889Å - 1.54Å |
| [113] -> 2.44Å; | (graphite filter) | d= 1.39Å - 2.44Å |
| [111] -> 4.61Å; | (beryllium filter) | d= 2.66Å - 4.61Å |

# D20 with “large” Paris-Edinburgh Pressure Cell (50 Kg)

Kernavanois et al. (2003) Advanced Millennium Pressure Project

40 minute D20 data for a 100 mm³ sample of CO at 7.3 GPa




BUT low temperatures –> smaller cells -> 1-10 mm³ samples

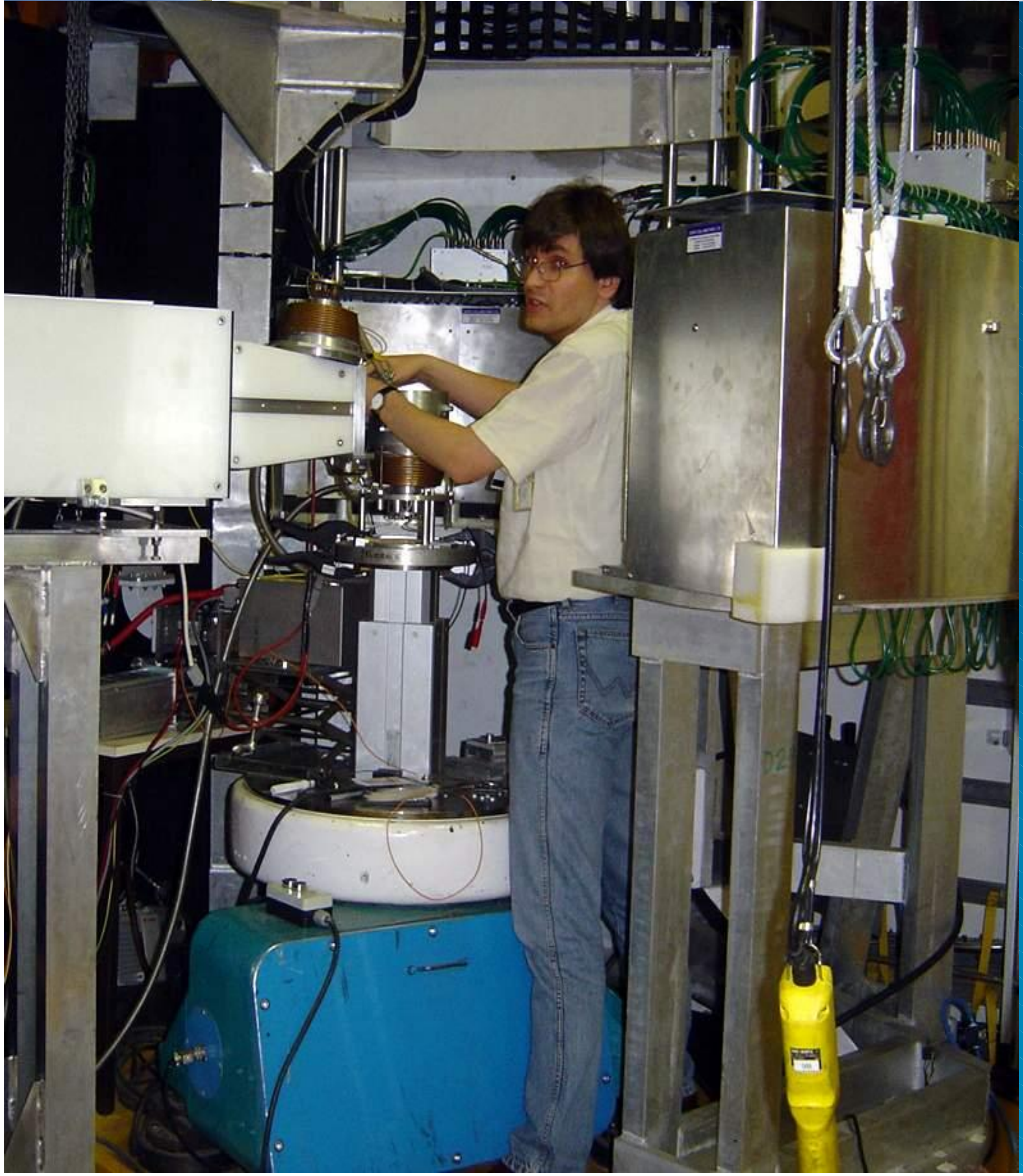
# Applications - fast detectors, small samples

## Very fast chemical and electrochemical kinetics


- The explosive SHS reaction was studied in real time with neutrons
- The reaction is exothermic, & heats the sample to 2200ºC 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)

# Applications - fast detectors, small samples



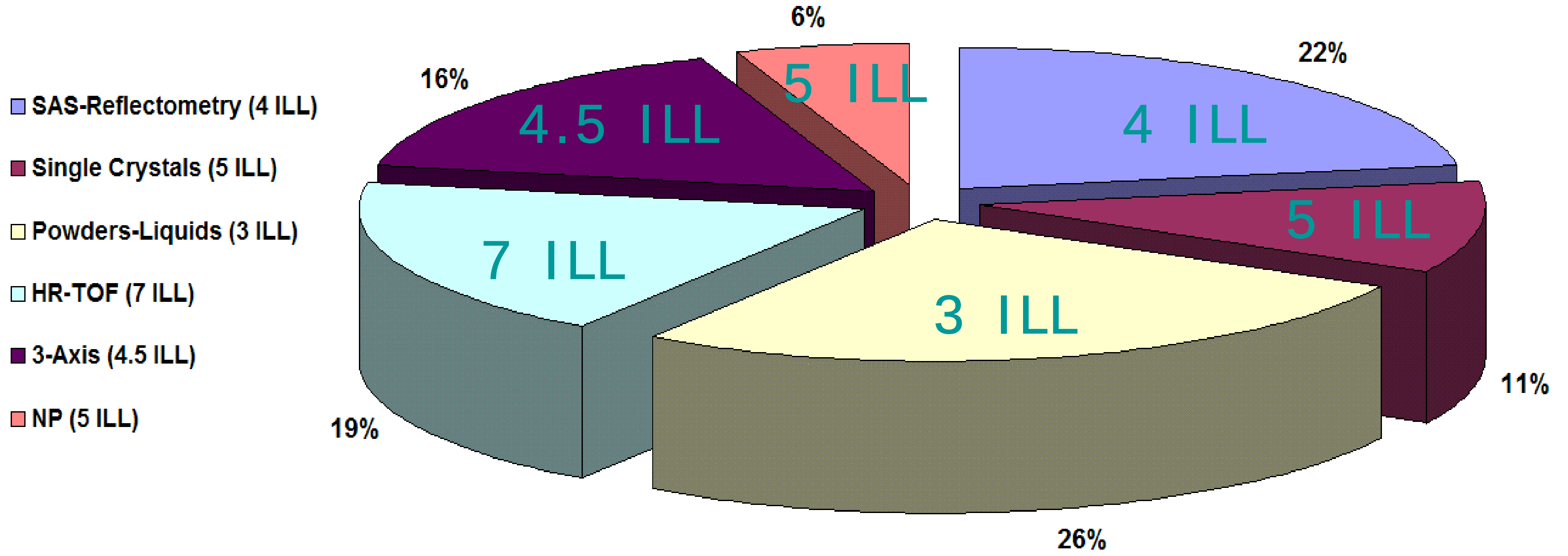
High-T Microwave Furnace
Super-D2B (Boysen et al.)
...with Carsten Korte from Giessen (2004)

## Convert D20 to DRACULA ?

- D20 has only recently been finished & is now working well
- D20 is the ILL's most requested machine (57 proposals)
- Only 2 modern powder machines for 22% of all proposals

# DRACULA–Why do we need a new machine ?

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




D2B, D4, D20

## Proposed ESS Powder Diffractometers

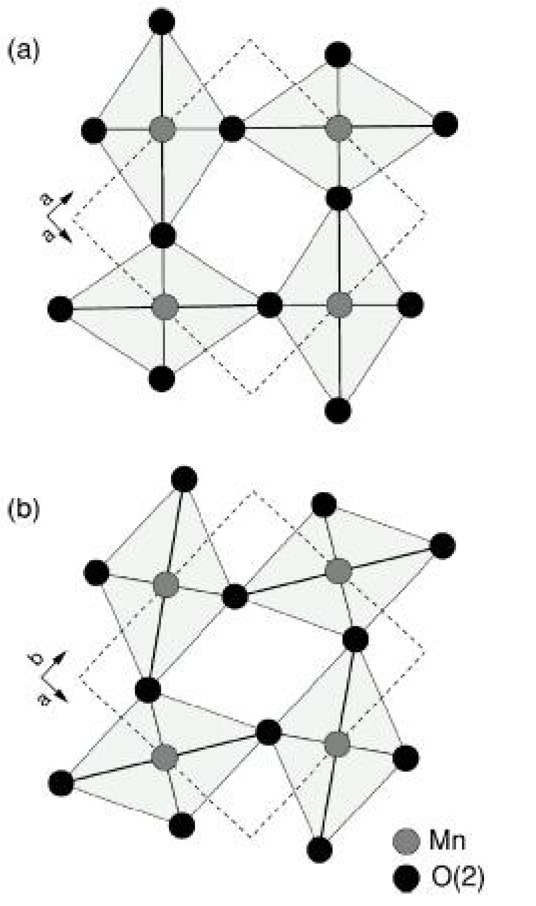
| | | |
|---|---|---|
| ST05 | High-Q Powder Diffractometer | HQP |
| ST06 | Liquids & Amorphous Diffractometer | LAD |
| SM10 | Single Pulse Diffractometer | SPD |
| SD17 | Magnetic Powder Diffractometer | MagP |
| SD18 | High Resolution Powder Diffractometer | HRPD |
| LM05 | Ultra-high Resolution Powder Diffractometer | URPD |
| LM06 | High Pressure Powder Diffractometer | HiPD |

## ILL 2-axis diffractometers that might use large 2D detectors

- **High intensity, small sample powder diffractometer** — **DRACULA**
- **Liquids & Amorphous Materials Diffractometer** — **super-D4**
- **Long Wavelength Magnetism/Biology Diffractometer** — **super-D16**

# Measurement of the local Jahn-Teller distortion in $LaMnO_{3.006}$

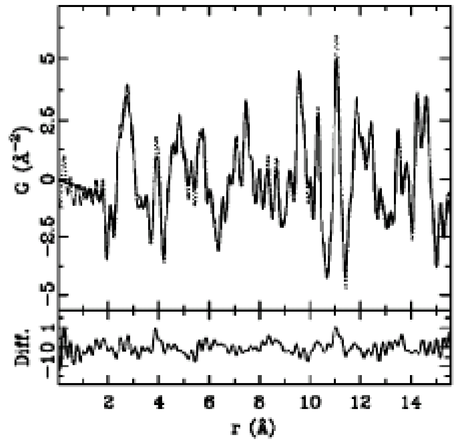
Th. Proffen, R. G. DiFrancesco, and S. J. L. Billinge (1999) **60**, 9973

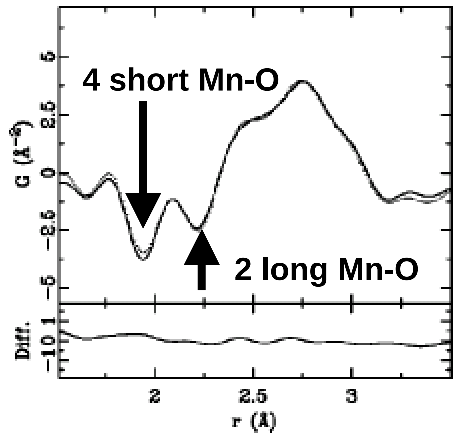



- Rietveld -> Average structure
- PDF -> Local details

- Rietveld NPD -> CMR distortion
- PDF -> Confirms JT distortion

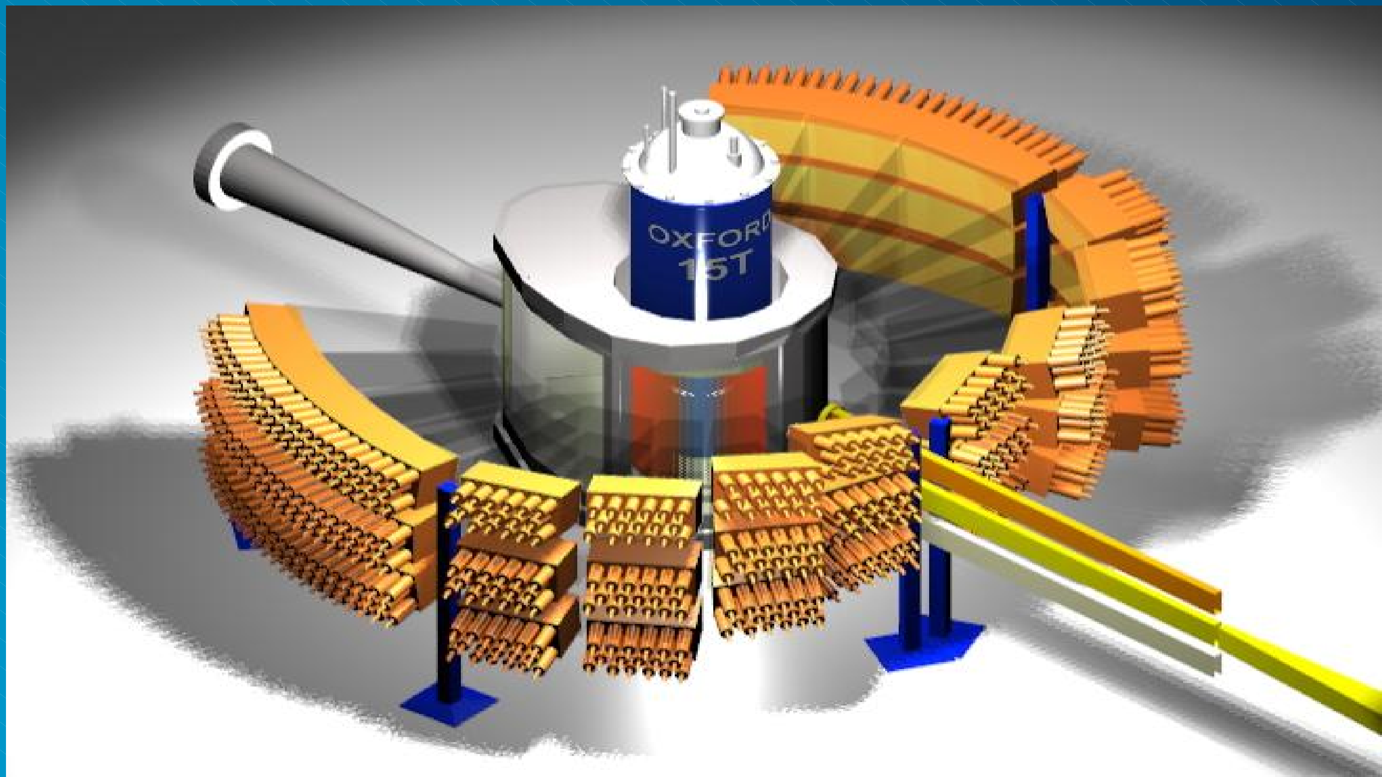
- PDF is increasingly important
- Super-D4/D9 diffractometer



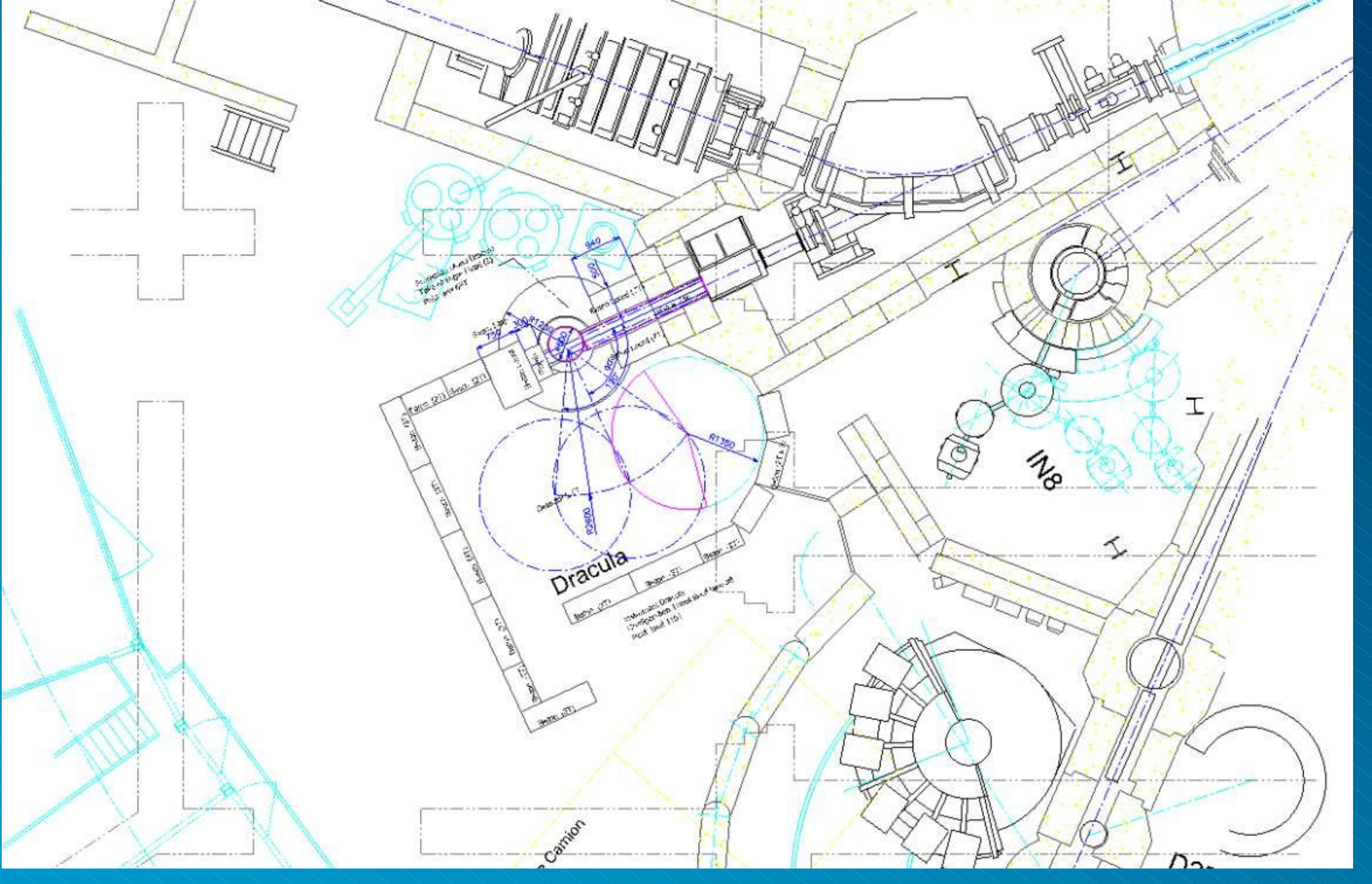



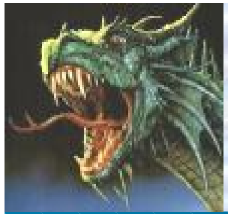
# Magnetism and Biology Dracula (super-D16)
# High resolution with long wavelengths

- ILL lacks a wide-angle, cold neutron machine like ISIS-WISH
- WISH is designed for long-period structures – magnetism & biology
- D16 is a good candidate – exists already, 90° take-off, long λ.
- Replace current small “bidim” on D16 by large D19-type banana ?



# DRACULA on H9 (replacing the Tomography station)



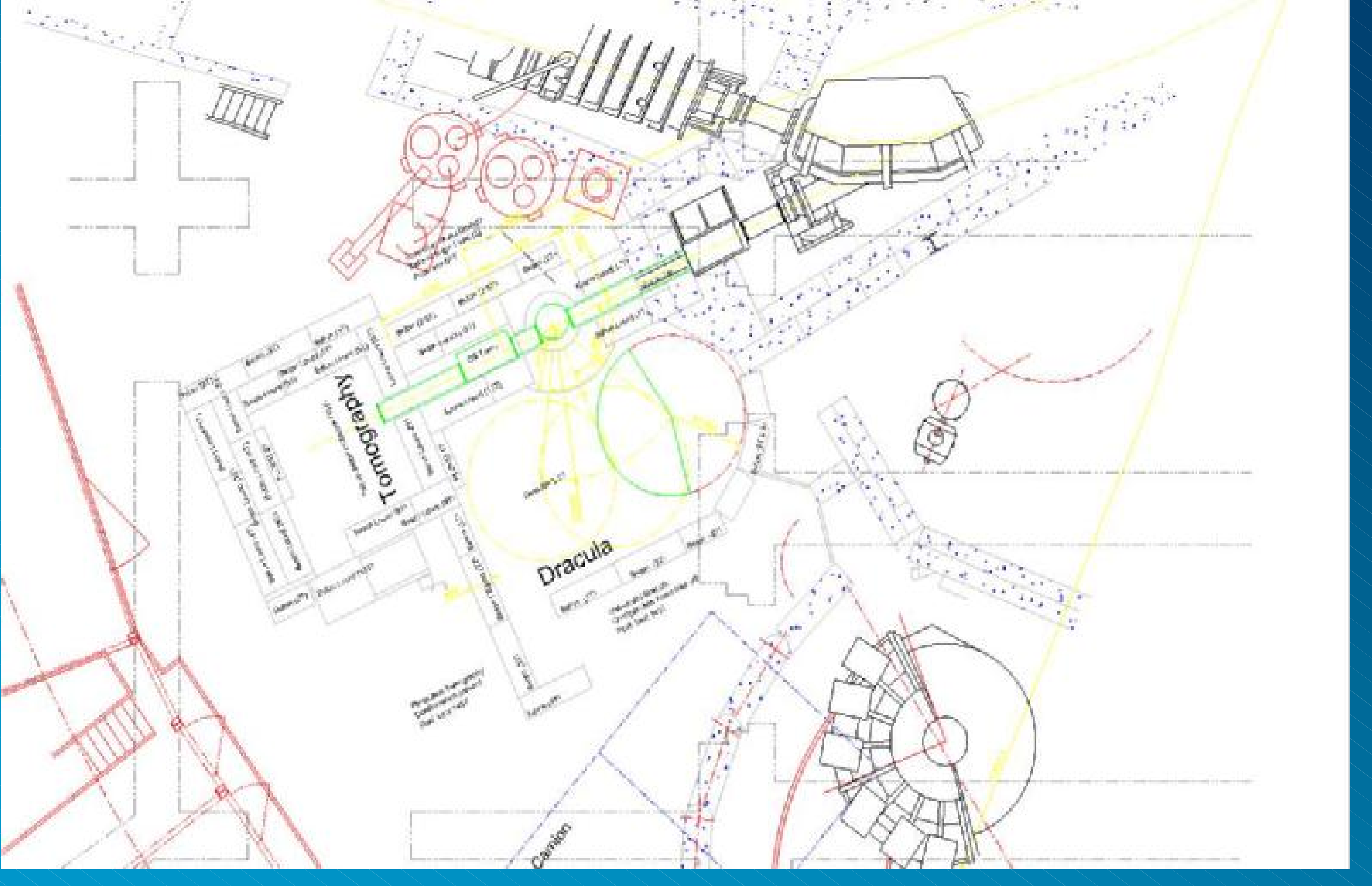




## DRACULA on H9 (replacing the Tomography station)

- Dracula would weigh about the same as Tomography
- Dracula slightly restricted by Lohengrin chariot/magnet
- But the big intensity gain comes from wavelength focussing
- For that, we need a wide monochromator, not so tall
- But it would be nice to find a place for tomography...

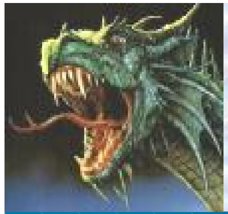
# DRACULA on H9 (co-existing with Tomography station?)



# Thermal DRACULA on a High Flux Beam

## DRACULA on H9 (co-existing with Tomography station?)

- Tomography would be moved back ~4m
- Tomography could be supported using a pillar in level-B
- A detailed floor load calculation has been commissioned
- Tomography would benefit by having better resolution
- Tomography would benefit from a better, larger casemate
- Dracula monochromator would absorb ~15% of white beam





## DRACULA cost and feasibility

- Dracula would use tested D19-type 2D-detector
- Dracula would also be useful for non-powder diffraction
- Dracula casemate optics would be similar to that of D20
- Dracula mono. would be less high, but horizontally focussing
- Cost and time-scale for Dracula can be easily calculated
- Dracula is a “no-risk” project that will give ILL a big lead

# DRACULA – Strategy or Submission ?

# DRACULA – Strategy or Submission ?

Can we compete with the Americans while waiting for ESS?

(Free Advice to ILL Directors)

- Use our natural advantage – time average flux on sample
- Use big detectors, as on pulsed neutron sources
- Do not assume that the SNS will be a long time coming
- Do not wait until the SNS is operational before reacting