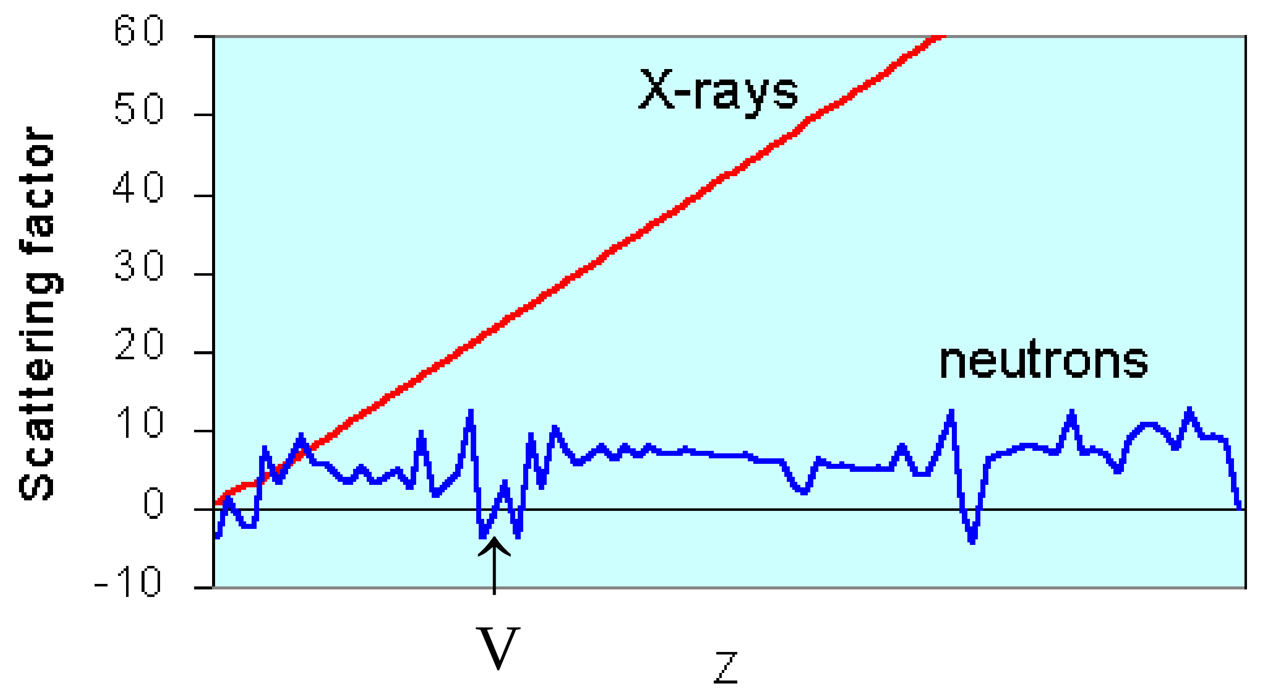
Dyfrakcja neutronów

Neutron Diffraction

X-rays: $f_j \propto Z$ - can be calculated

Neutrons: small dependence of f_j on Z but major part Z independent. f_j must be determined experimentally



Good points/Bad points

- Can detect light atoms
- Can often distinguish between adjacent atoms
- Can distinguish between isotopes
- Can accurately find atoms in presence of very high Z atoms
- Covers a wide range of d-spacings - more hkl **- BUT**
- some atoms/isotopes good neutron absorbers (e.g. Cd, Gd (Gadolinium), ^{6}Li (so use ^{7}Li)
- V has very low, ~0 scattering
- need neutron source
- VERY expensive (~£10,000 per DAY!)

Excellent complementary technique to XRD

More on neutrons

Neutron can be scattered by atoms by:

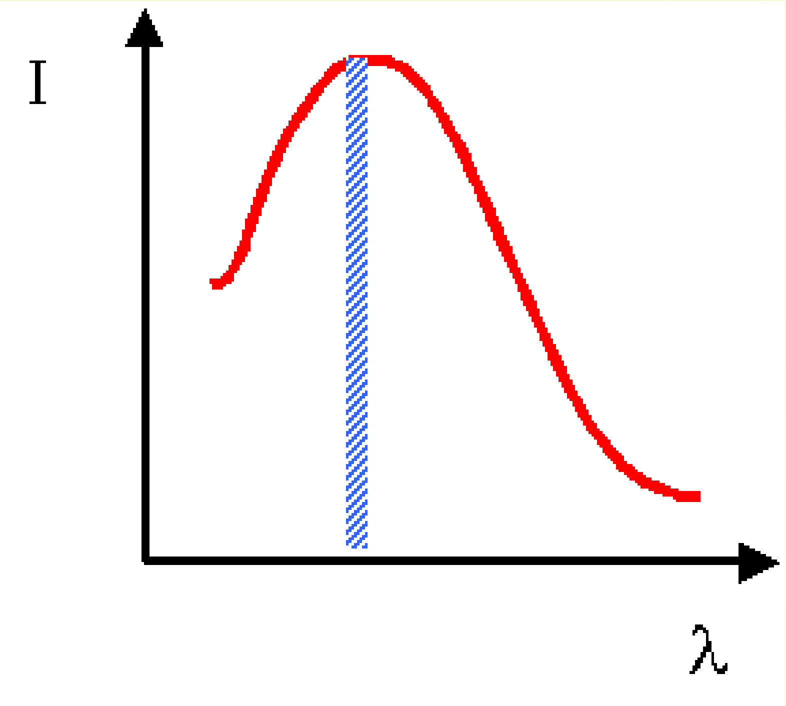
- interaction with nucleus
- interaction with spin of unpaired electrons - magnetic interaction, magnetic scattering. This happens because the neutron has a magnetic moment.

Also the interaction can be:

- elastic (diffractometer) structural studies
- inelastic (spectrometer) loss of energy on scattering gives information on phonon dispersion (effect of vibrations in lattice) and stretching of bonds.

The experiment

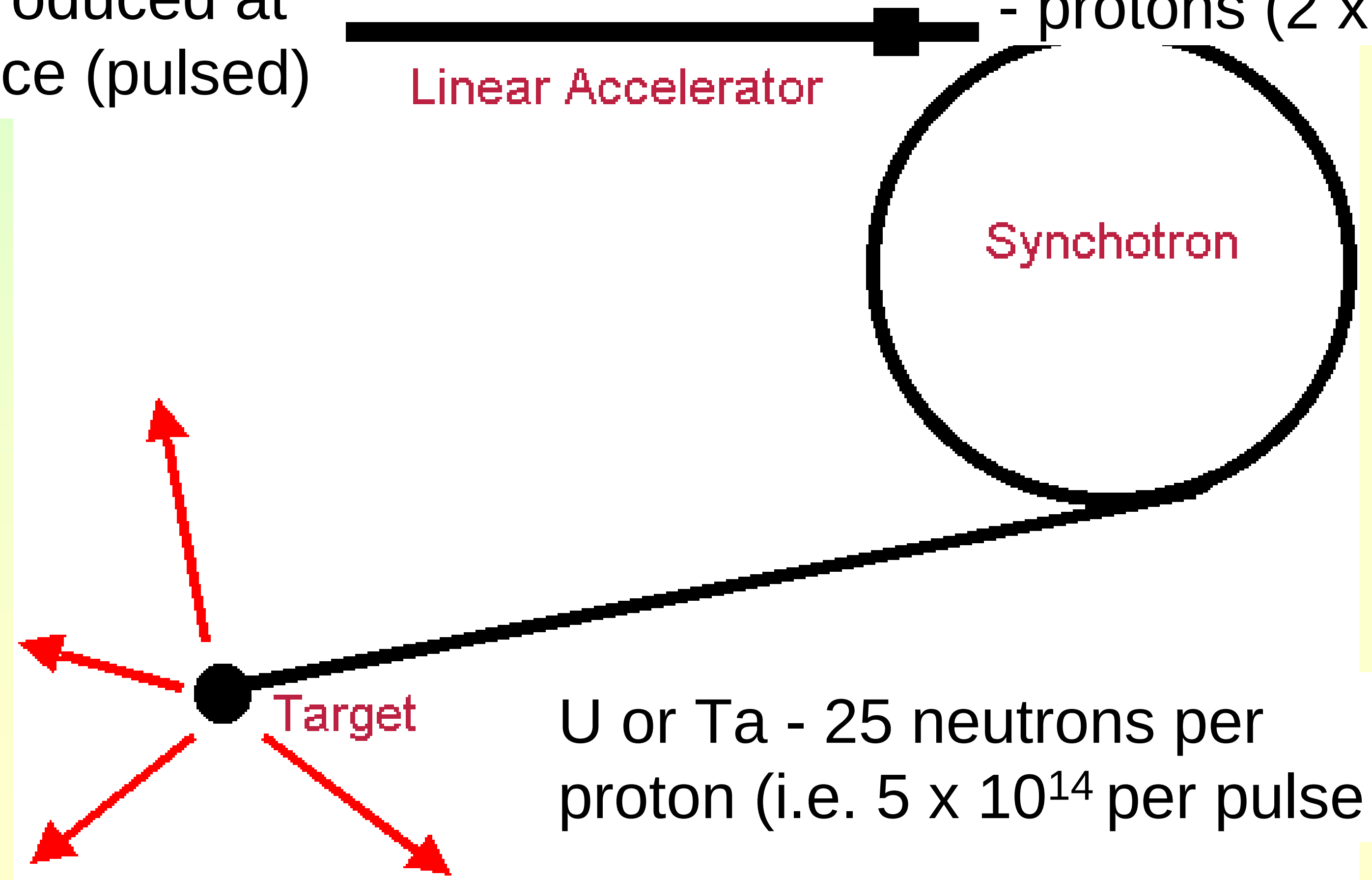
At many sources (e.g. ILL at Grenoble) neutrons are produced by fission in a nuclear reactor and then selected by wavelength - but with neutrons there are no “characteristic” wavelengths:



..so by selecting a wavelength we lose neutrons and lose intensity

ISIS

UK neutron source at Rutherford Appleton Laboratory uses “time of flight” neutron diffraction

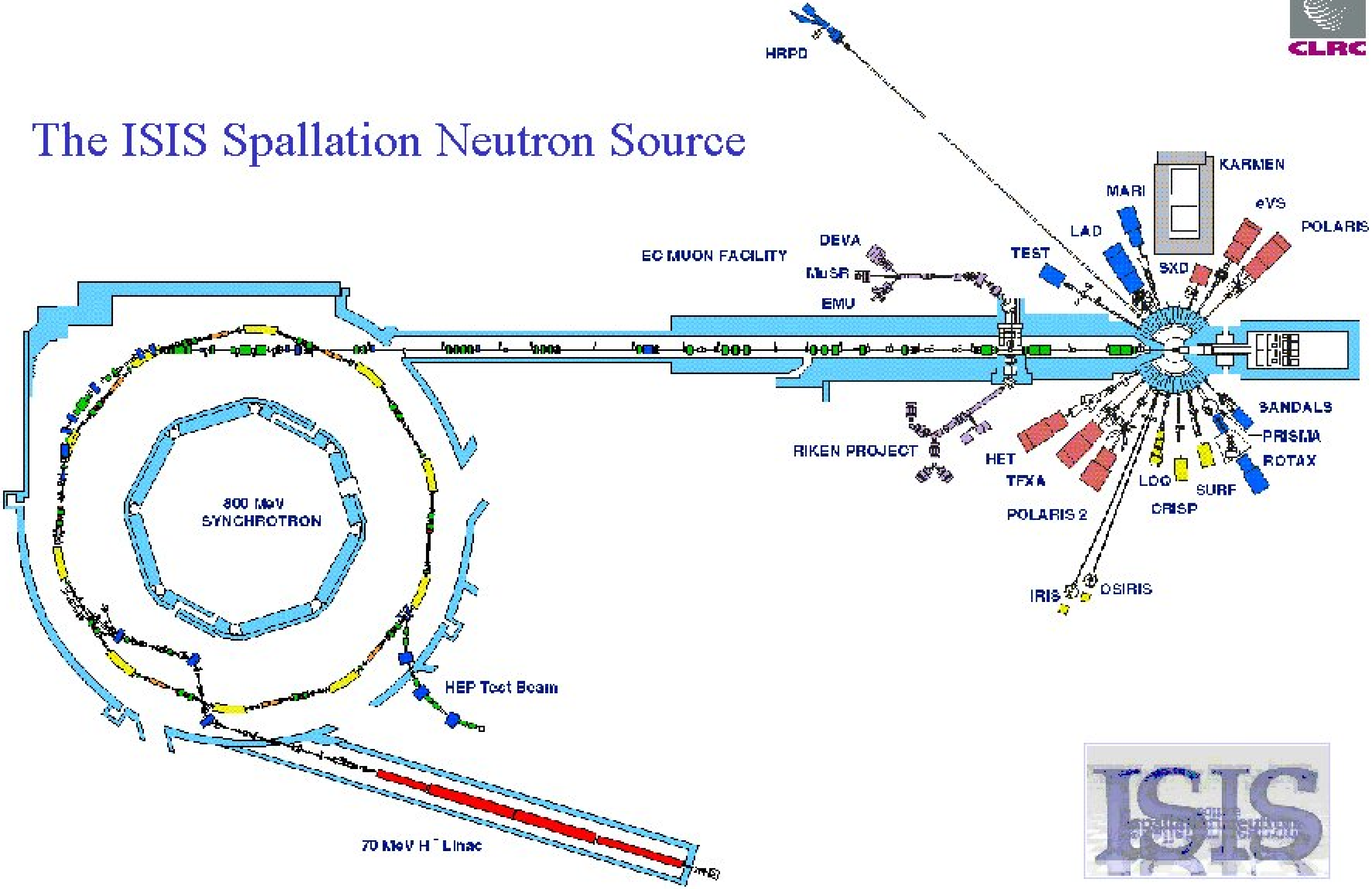


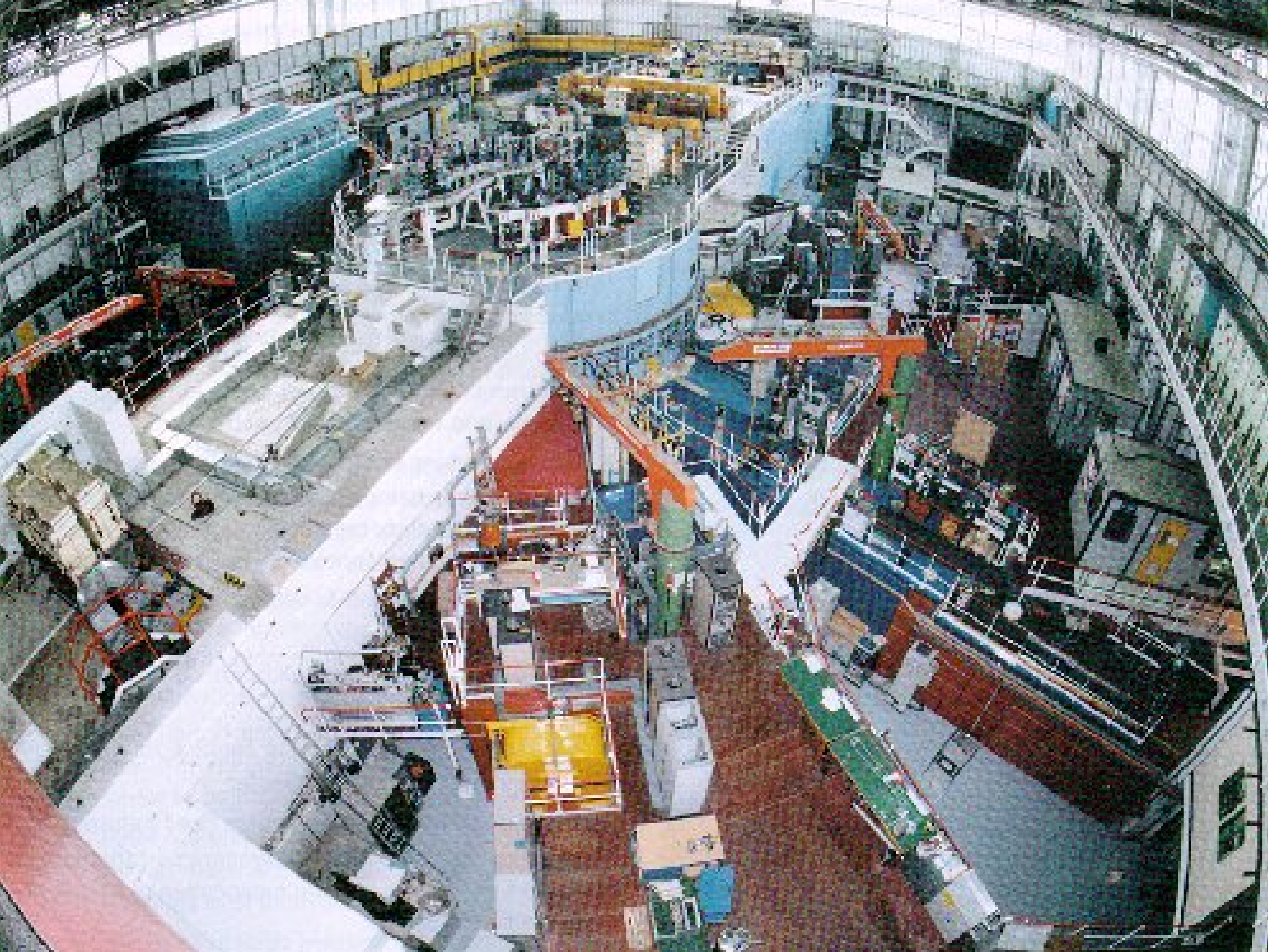
H^- produced at source (pulsed)

Electrons stripped - protons (2 x 10^{13})

U or Ta - 25 neutrons per proton (i.e. 5 x 10^{14} per pulse

The ISIS Spallation Neutron Source





Time-of-flight

Bragg equation - $2d_{hkl}\sin\theta = \lambda$

We are measuring d, so two variables, θ and λ

In X-ray powder diffraction, λ is constant, θ variable

In time-of-flight (t.o.f), θ is constant, λ variable

This takes advantage of the full “white” spectrum

Two basic equations:

$$\lambda = \frac{h}{mv} \qquad\qquad v = \frac{L}{t}$$

where m,v = mass, velocity of neutron

L = length of flight path t = time of flight of neutron

Time-of-flight equation

Combine:

$$\lambda = \frac{ht}{mL} = 2d\sin\theta$$

$$\boxed{t = \frac{2mL}{h} d\sin\theta}$$

L is a constant for the detector, h, m are constants so:

$$\mathbf{t \propto d}$$

d-spacings are discriminated by the time of arrival of the neutrons at the detector

The biggest error in the experiment is **where** the neutrons originate

This gives an error in the flight path, L

typical value ~5cm

$$\frac{\Delta L}{L} = \frac{\Delta t}{t} = \frac{\Delta d}{d}$$

Hence as L increases, error in d is reduced - <u>resolution</u> of the instrument is improved

e.g. instrument at 10m compared to instrument at 100m

100m = HRPD, currently highest resolution in the world

Example 1

Calculate the velocity of a neutron that would have the same wavelength as CuKα radiation (λ=1.54Å); mass of neutron = 1.675 x 10^{-27}kg; h=6.626 x 10^{-34} Js

$$\lambda = \frac{h}{mv} \Rightarrow v = \frac{h}{m\lambda}$$

$$v = \frac{6.626 \times 10^{-34}}{1.675 \times 10^{-27} \times 1.54 \times 10^{-10}}$$

$$\mathbf{v = 2.57 \times 10^{3}\ ms^{-1}}$$

Example 2

Silicon has a cubic unit cell. A neutron diffraction experiment using a detector at 10m and θ=45° reveals that the (111) reflection of silicon has a time of flight of 11200 microseconds. What is the unit cell of silicon?

(h=6.626 x 10^{-34} Js; mass of neutron = 1.675 x 10^{-27}kg)

$$\boxed{t = \frac{2mL}{h} d \sin\theta}$$

$$d = \frac{ht}{2mL\sin\theta}$$

$$d = 3.13Å$$

d-spacing eqn: $$\frac{1}{3.13^2} = \frac{3}{a^2}$$

$\Rightarrow$ **a**=5.42Å