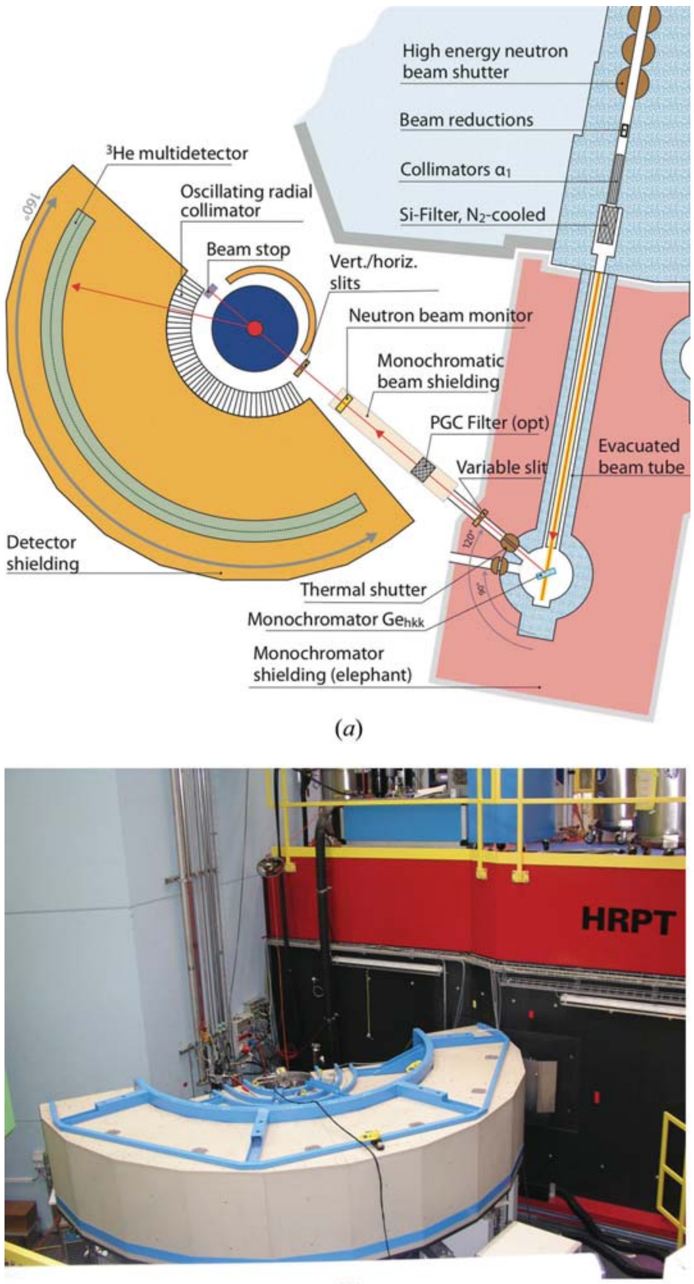
International Tables for Crystallography (2018). Vol. H, Section 2.3.4.1.2, pp. 81–82.



(*a*)

(*b*)

Figure 2.3.15
A constant-wavelength neutron powder diffractometer. This figure shows (*a*) a layout diagram and (*b*) the physical appearance (dominated by the monochromator and detector shieldings) for the HRPT diffractometer installed at the SINQ continuous spallation source. (Figures from https://www.psi.ch/sinq/hrpt/.)

the sample will control the precision with which the scattering angle 2θ can be determined. For a diffractometer detecting neutrons and measuring scattering angles in the horizontal plane (as shown in Fig. 2.3.15) the horizontal divergences are critical, the vertical divergences less so.[11] Indeed, the horizontal divergences are key parameters in the determination of resolution and intensity (Section 2.3.4.1.4); for this reason we denote by α_1, α_2 and α_3 the (half-angle) angular divergences of the primary beam (*i.e.* the beam onto the monochromator), the monochromatic beam (from monochromator to sample) and the diffracted beam (from sample to detector), respectively.

[11] For this reason large vertical divergences are employed to increase intensity; they do however have second-order impacts on the shapes (asymmetry) and positions of diffraction peaks (Howard, 1982; Finger *et al.*, 1994; see also Section 4.2 in Kisi & Howard, 2008).

The divergences are limited by various forms of collimation. The divergence of the primary beam will be limited in the first instance by the delivery system. For delivery through a simple beam tube of length L, with entrance and exit apertures of dimensions a_1 and a_2, respectively, the angular divergence (half-angle) is given by (as already noted in Section 2.3.3.4)

$$\alpha_1 = \frac{a_1 + a_2}{2L}. \tag{2.3.16}$$

Neutrons emerging from a guide tube would have divergence equal to the critical angle of the guide, $\alpha_1 = \theta_c$. Soller collimators (see below) can be used if there is a need to further reduce the horizontal divergence of the primary beam. The divergence of the monochromatic beam may be limited by slits, or a beam tube. The divergence of the diffracted beam, α_3, is often defined using another Soller collimator. Sometimes this divergence is limited just by the dimensions of the sample and the detecting elements; equation (2.3.16) gives α_3 if it now references the sample and detector element dimensions and the distance between them. Even in this circumstance (as in HRPT), Soller collimators may be used in front of the detector to reduce scattering from ancillary equipment and other background contributions.

Soller collimators (Soller, 1924) are used to transmit beams of large cross section while limiting (for example) horizontal divergence. They are in effect narrow but tall rectangular collimators stacked side by side; in practice they comprise thin neutron-absorbing blades equally spaced in a mounting box. It should be evident from equation (2.3.16) that if the length of the collimator is L and the separation between the blades is a, then the (half-angle) horizontal divergence is a/L. The transmission function for a Soller collimator is ideally triangular. It is technologically challenging to make compact Soller collimators, since, for a given collimation, a shorter collimator needs a smaller blade spacing. One very successful approach, due to Carlile *et al.* (1977), has been to make the neutron-absorbing blades from Mylar, stretched on thin steel or aluminium alloy frames, and subsequently coated with gadolinium oxide paint; these blades are stacked and connected *via* the frames which become the spacers in the final product. The collimators made by Carlile *et al.* were 34 cm long, and the blade spacing was 1 mm, giving a horizontal divergence of 0.17°. Compact Soller collimators of this type (Fig. 2.3.16) are now commercially available, with blade spacings down to 0.5 mm.

Even more compact collimators can be produced by eliminating the gaps in favour of solid layers of neutron-transmitting material; for example, a collimator only 2.75 cm long made by stacking 0.16 mm thick gadolinium-coated silicon wafers gave a divergence of 0.33° (Cussen *et al.*, 2001). Microchannel plates (Wilkins *et al.*, 1989) may offer additional possibilities for collimation and focusing.

2.3.4.1.2. *Monochromators*

The wavelength in a constant-wavelength powder diffractometer is almost invariably selected by a single-crystal monochromator. If the primary beam is incident onto the monochromator in such a way as to make an angle θ_M with a chosen set of planes in the crystal, then the wavelength that will be reflected from these planes is given by Bragg's law,

$$\lambda = 2d\sin(\theta_M),$$

where d is the spacing of the chosen planes. A spread of angles of incidence represented by $\Delta\theta_M$ will result in the selection of a

Figure 2.3.16
Commercially available compact Soller collimators. (Reproduced with permission from Eurocollimators Ltd, UK.)

band of wavelengths $\Delta\lambda$ given by

$$\frac{\Delta\lambda}{\lambda} = \cot\theta_M \Delta\theta_M. \quad (2.3.17)$$

For high-resolution performance we need a rather precisely defined wavelength, so $\Delta\lambda$ should be small; if, on the other hand, intensity is an issue then a wider band of wavelengths needs to be accepted. It should be evident from equation (2.3.17) that a high-resolution diffractometer will operate with a take-off angle from the monochromator, $2\theta_M$, as high (*i.e.* as close to 180°) as practicable, and with tight primary collimation α_1.

It might be noticed that the integer n appearing on the right-hand side of equation (1.1.3) has been omitted from our formulation of Bragg's law. If the Miller indices of the chosen planes are hkl, if the spacing of these planes is d_{hkl}, and if we introduce $d_{nh,nk,nl} = d_{hkl}/n$ [*cf.* equation (1.1.23)], then the factor n is effectively restored. This means that, as well as reflecting the selected wavelength through the hkl reflection, the monochromator has the potential to reflect unwanted harmonics λ/n of the desired wavelength through the nh,nk,nl reflections. This problem can be largely overcome using the hkl planes with h, k, l all odd in crystals with the diamond structure, such as silicon and germanium; for this structure the structure factors [equation (2.3.7)] for the $2h,2k,2l$ reflections are zero so that there is no contamination by $\lambda/2$, and at the shorter wavelengths, $\lambda/3$ and so on, there are very few neutrons in the thermal neutron spectrum (Fig. 2.3.5).

Since 'perfect' crystals (of silicon and germanium, for example) have low reflectivity, for monochromator applications imperfect or 'mosaic' crystals are usually preferred. A mosaic crystal can be pictured as comprising small blocks of crystal with slightly differing orientations, the distribution in angle of these blocks being characterized by a full-width at half-maximum angle, β, known as the 'mosaic spread'. In addition to improving the intensity markedly,[12] this 'mosaic spread' will also increase the range of wavelengths obtained. Crystals intended for use as monochromators are very often deliberately deformed to achieve the desired mosaic structure. Further gains in intensity are sought by using vertically focusing monochromators, since the vertical divergence can be increased without serious detriment to the diffraction patterns. Vertically focusing monochromators usually comprise a number of separate monochromator crystals either individually adjustable (Fig. 2.3.17) or in fixed mountings on a bendable plate.

It is not common to find polarized neutrons being used in neutron powder diffractometers. Nevertheless, we think it appropriate to mention here that one means to obtain a polarized neutron beam is to use an appropriate polarizing crystal monochromator.[13] The 111 reflection from the ferromagnetic Heusler alloy Cu_2MnAl is commonly used for this purpose; the nuclear and magnetic structure factors [equations (2.3.7) and (2.3.8)] are of similar magnitude and they add or subtract depending on whether the neutron spin is antiparallel or parallel to the magnetization of the alloy. The beam reflected from such a monochromator can be polarized to better than 99%.

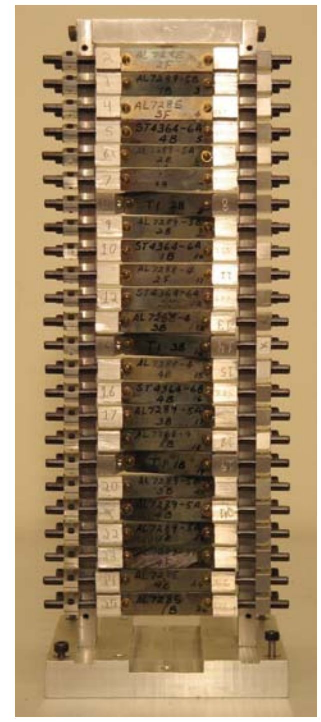


Figure 2.3.17
The vertically focusing monochromator constructed at the Brookhaven National Laboratory (Vogt *et al.*, 1994) and now used by the high-resolution powder diffractometer ECHIDNA at OPAL. The 24 monochromating elements are individually adjustable, and each of these is a 30-high stack of 0.3 mm thick Ge wafers, deformed to yield a suitable mosaic structure and then brazed together. (Reproduced with permission from ANSTO.)

The reader is referred to Section 4.4.2 of Volume C (Anderson & Schärpf, 2006) and to Kisi & Howard (2008) Sections 3.2.1 and 12.3 for further details.

2.3.4.1.3. *Neutron detectors*

Neutrons, being electrically neutral, do not themselves cause ionization and so cannot be detected directly; their detection and counting therefore depend on their capture by specific nuclei and the production of readily detectable ionizing radiation in the ensuing nuclear reaction. Only a limited number of neutron-capture reactions are useful for neutron detection [see Chapter 7.3 of Volume C (Convert & Chieux, 2006)]; they include

$$\begin{aligned}
{}^1_0\mathrm{n} + {}^3_2\mathrm{He} &\rightarrow {}^1_1\mathrm{H} + {}^3_1\mathrm{H} + 0.76\ \mathrm{MeV}\\
{}^1_0\mathrm{n} + {}^6_3\mathrm{Li} &\rightarrow {}^3_1\mathrm{H} + {}^4_2\mathrm{He} + 4.79\ \mathrm{MeV}\\
{}^1_0\mathrm{n} + {}^{10}_5\mathrm{B} &\rightarrow {}^7_3\mathrm{Li} + {}^4_2\mathrm{He} + 2.8\ \mathrm{MeV}\\
{}^1_0\mathrm{n} + {}^{157}_{64}\mathrm{Gd} &\rightarrow {}^{158}_{64}\mathrm{Gd} + \gamma\text{-rays} + \text{conversion electrons}\\
&\qquad + \leq 0.18\ \mathrm{MeV}\\
{}^1_0\mathrm{n} + {}^{235}_{92}\mathrm{U} &\rightarrow \text{fission fragments} + \sim 200\ \mathrm{MeV}
\end{aligned}$$

(*cf.* Section 2.3.3.2).

[12] Most of the improvement is due to a change from a 'dynamical' to a 'kinematic' scattering regime.

[13] Polarized beams can also be produced using suitable mirrors or filters [see Section 4.4.2 of Volume C by Anderson & Schärpf (2006)].