International Tables for Crystallography (2006). Vol. C, Figure 2.3.2.4, p. 56.

the Bragg equation (Hart, 1981). The accuracy is then limited by the angular accuracy of the diffractometer and the orientation setting.

It is necessary to monitor the monochromatic beam intensity I_0, which changes during the recording due to decreasing storage-ring current, orbital shifts or other factors. This can be done by inserting a low-absorbing ionization chamber in the beam or by using a scintillation counter to measure scattering from an inclined thin beryllium foil, kapton or other low-absorbing material. The data are recorded and used to correct the observed intensities. The monitored counts can also be used as a timer for step scanning if a sufficient number are recorded for good counting statistics.

The entrance slit ES determines the irradiated specimen length, which is equal to ES/sin θ_s. Vertical parallel slits VPS with $\delta \simeq 2^\circ$ are used to limit the axial divergence. The longer the distance between the specimen and detector, the smaller the asymmetry, and a vacuum path should be used to avoid air-absorption losses. The specimen may be used in either reflection or transmission simply by rotating it 90° around the diffractometer axis from its previous position.

The diffracted beam can be defined by a receiving slit (Parrish, Hart & Huang, 1986), horizontal parallel slits HPS [Fig. 2.3.2.4(*a*)] (Parrish & Hart, 1985) or a high-quality single-crystal plate which acts as a very narrow receiving slit [Fig. 2.3.2.4(*b*)] (Cox, Hastings, Thomlinson & Prewitt, 1983; Hastings *et al.*, 1984). If a receiving slit is used, the intensity, profile width and shape are determined by the widths of both ES and RS. If either one is much wider than the other, the profile has a flat top. Increasing the RS width and keeping ES constant causes symmetrical profile broadening and increases the intensity as in conventional focusing diffractometry. There are disadvantages in using a receiving slit because the intensities are low and

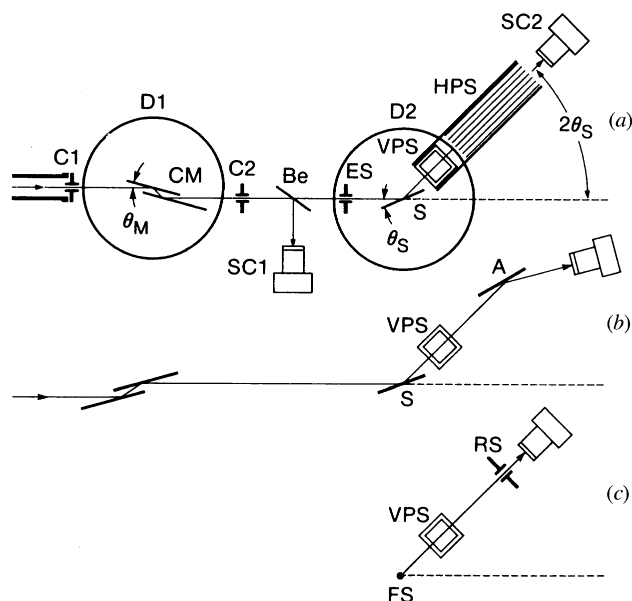

Fig. 2.3.2.4. (*a*) Optics of dispersive parallel-beam method for synchrotron X-rays. *C*1 primary-beam collimator, *D*1 diffractometer for channel monochromator CM, *C*2 antiscatter shield, Be beryllium foil for monitor, SC1 and SC2 scintillation counters, ES entrance slit on powder diffractometer *D*2, VPS vertical parallel slits to limit axial divergence, HPS horizontal parallel slits, which determine the resolution. (*b*) CM in nondispersive setting and crystal analyser *A* used as a narrow receiving slit. (*c*) Fibre specimen FS with receiving slit RS or with position-sensitive detector (not shown) with RS removed.

it causes the same specimen-surface-displacement and transparency errors as the focusing geometries.

A set of horizontal parallel (Soller) slits is advantageous because of the much higher intensity and it eliminates the displacement errors. The profiles of specimens without broadening effects have the same FWHM as the aperture of the slits, equation (2.3.1.7). The FWHM increases as $\tan\theta$ due to wavelength dispersion. By increasing the length of the foils and keeping the same spacing, the aperture can be reduced to increase the resolution without large loss of intensity. A set of 365 mm long slits with 0.05° aperture has been used and even smaller apertures are feasible. Longer slits decrease the fluorescence intensity (if any) reaching the detector. They must be carefully made and aligned to avoid loss of intensity and should be evacuated or filled with He to avoid air-absorption losses.

The use of a crystal analyser eliminates fluorescence and gives the highest resolution powder profiles with FWHM $= 0.02$ to $0.05^\circ 2\theta$, depending on the quality of the crystal (Hastings *et al.*, 1984). The alignment of the crystal is critical and must be done with remote automated control every time the wavelength is changed. Displacement aberrations are eliminated but the intensity is much lower than the HPS because of the small rocking angle and low integrated reflectivity of the crystal.

The correct orientation of crystalline powder particles for reflection is far more restrictive for the parallel beam than the X-ray tube divergent beam. A much smaller number of particles will have the exact orientation for reflection, and thus the recorded intensity will be lower and relative intensities less accurate. If the specimen is stationary, the standard deviations of the intensities due to particle size are six to nine times higher than in focusing methods (Parrish, Hart & Huang, 1986). It also becomes more difficult to achieve the completely randomly oriented specimens required for structure determination and quantitative analysis and, as in X-ray tube data, a preferred-orientation term is included in the structure refinement. It is, therefore, essential to use small particles $< 10\,\mu$m and to rotate the specimen. Some investigators prefer to oscillate the specimen over a small angle but this is not as effective as rotation.

The profiles are virtually symmetrical except at small angles where axial divergence causes asymmetry. The profiles in Fig. 2.3.2.5 show the differences in the shape and resolution obtained with conventional focusing (*a*) and parallel-beam synchrotron methods (*b*). The effect of the higher resolution on a mixture of nearly equal volumes of quartz, orthoclase, and feldspar recorded with X-ray tube focusing methods is shown in Fig. 2.3.2.5(*c*) and with synchrotron radiation in Fig. 2.3.2.5(*d*). The symmetry and nearly constant simple instrument function make it easier to separate overlapping reflections and simplify the profile-fitting procedures and the interpretation of specimen-broadening effects.

The early crystal-structure studies using Rietveld refinement were not as successful with X-ray tube focusing methods as they were with neutron diffraction because the complicated instrument function made profile fitting difficult and inaccurate. The development of synchrotron powder methods with simple symmetrical instrument function, high resolution, and the use of longer wavelengths to increase the dispersion have made structural studies as successful as with neutrons, and have the advantage of orders-of-magnitude higher intensity. Some examples are described by Attfield, Cheetham, Cox & Sleight (1988), Lehmann, Christensen, Fjellvåg, Feidenhans'l & Nielsen (1987), and *ab initio* structure determinations by

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