

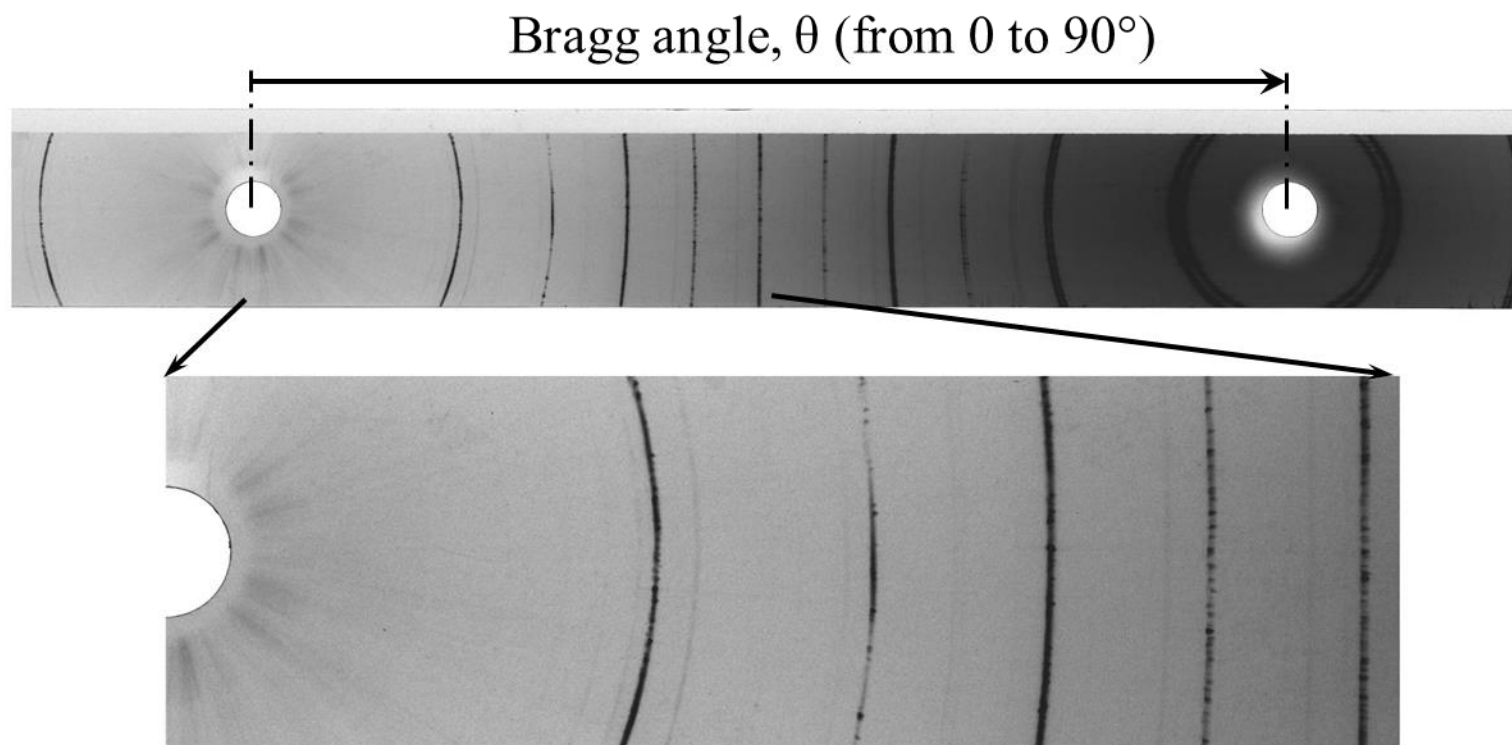
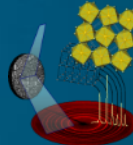


Figure 11.1. Film with the X-ray diffraction pattern of the polycrystalline LuAu, recorded in a Debye-Scherrer camera using Cu $K\alpha$ radiation. Bragg peaks appear as concentric ring segments with varying darkness and curvature. The spottiness of some rings, clearly visible at low Bragg angles in the expanded view, indicates an insufficient number of grains in the irradiated volume of the sample. This result was achieved by annealing the needle at 900°C to promote grain growth.

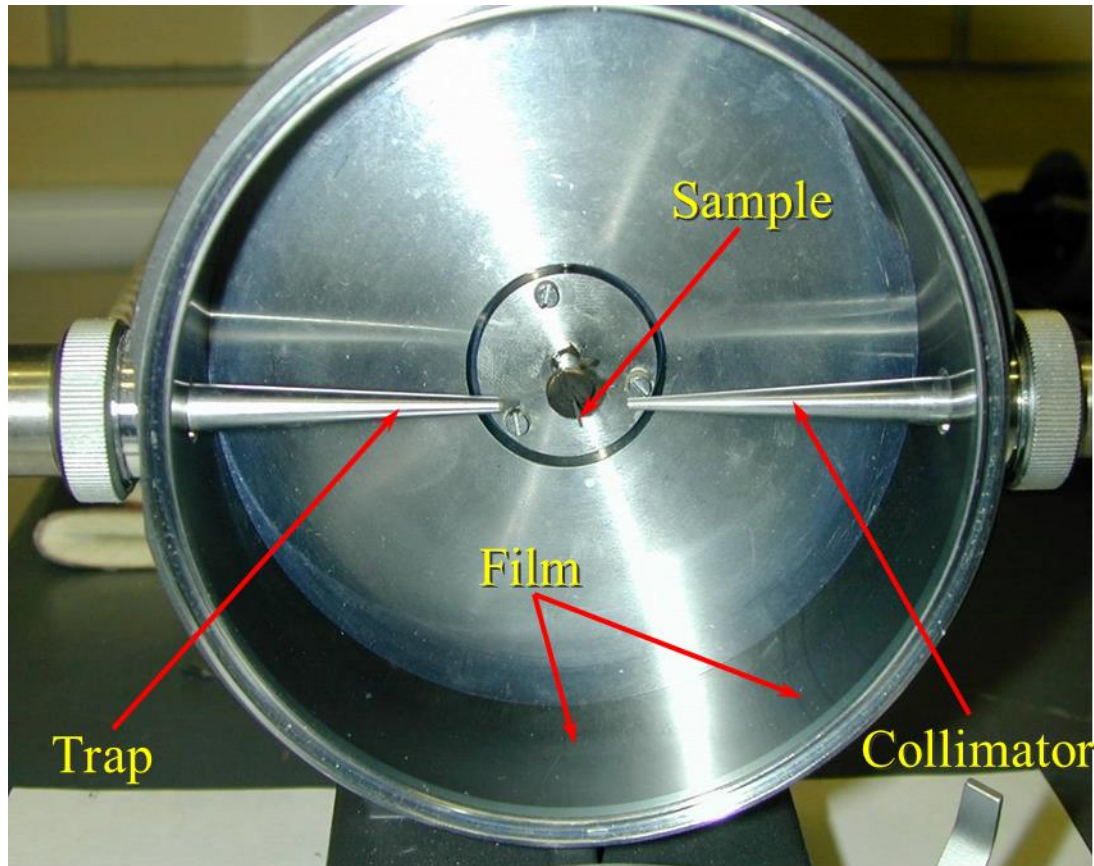
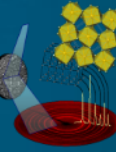


Figure 11.2. Debye–Scherrer camera without a cover, including a cylindrical sample, collimator, incident beam trap, and the location of the X-ray film.

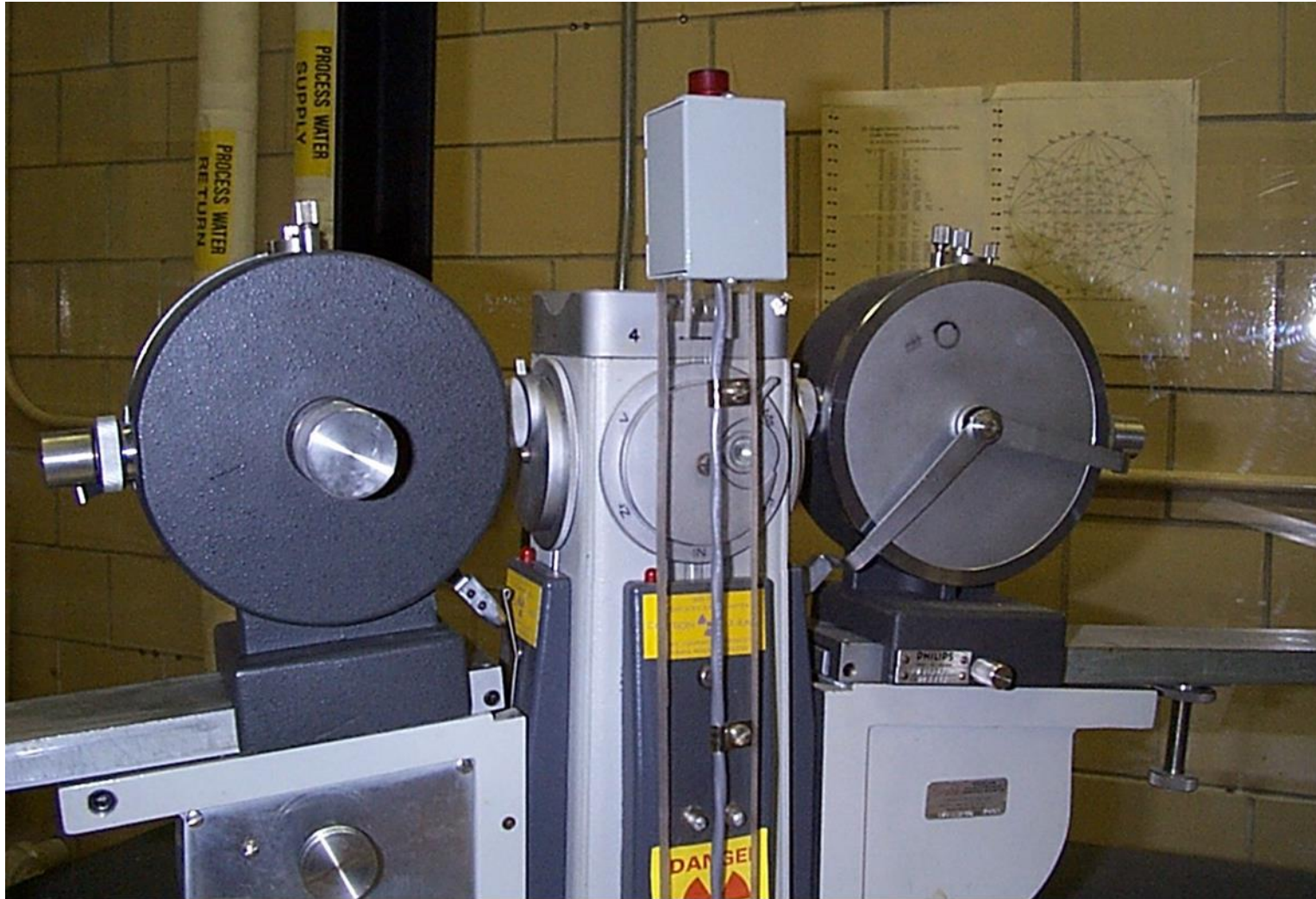
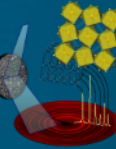


Figure 11.3. Two Debye–Scherrer cameras with covers, which have been loaded with X-ray film and are installed on the X-ray generator, ready for collecting powder diffraction data.

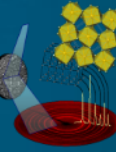


Figure 11.4. The overall view of a powder diffractometer. On the *right*, the radiation enclosure is opened to expose the goniometer, which rests on top of the high voltage power supply. The computer on the *left* is used to manage and control data collection and to carry out preliminary processing of the data, such as conversion from a software-specific binary to ASCII format.

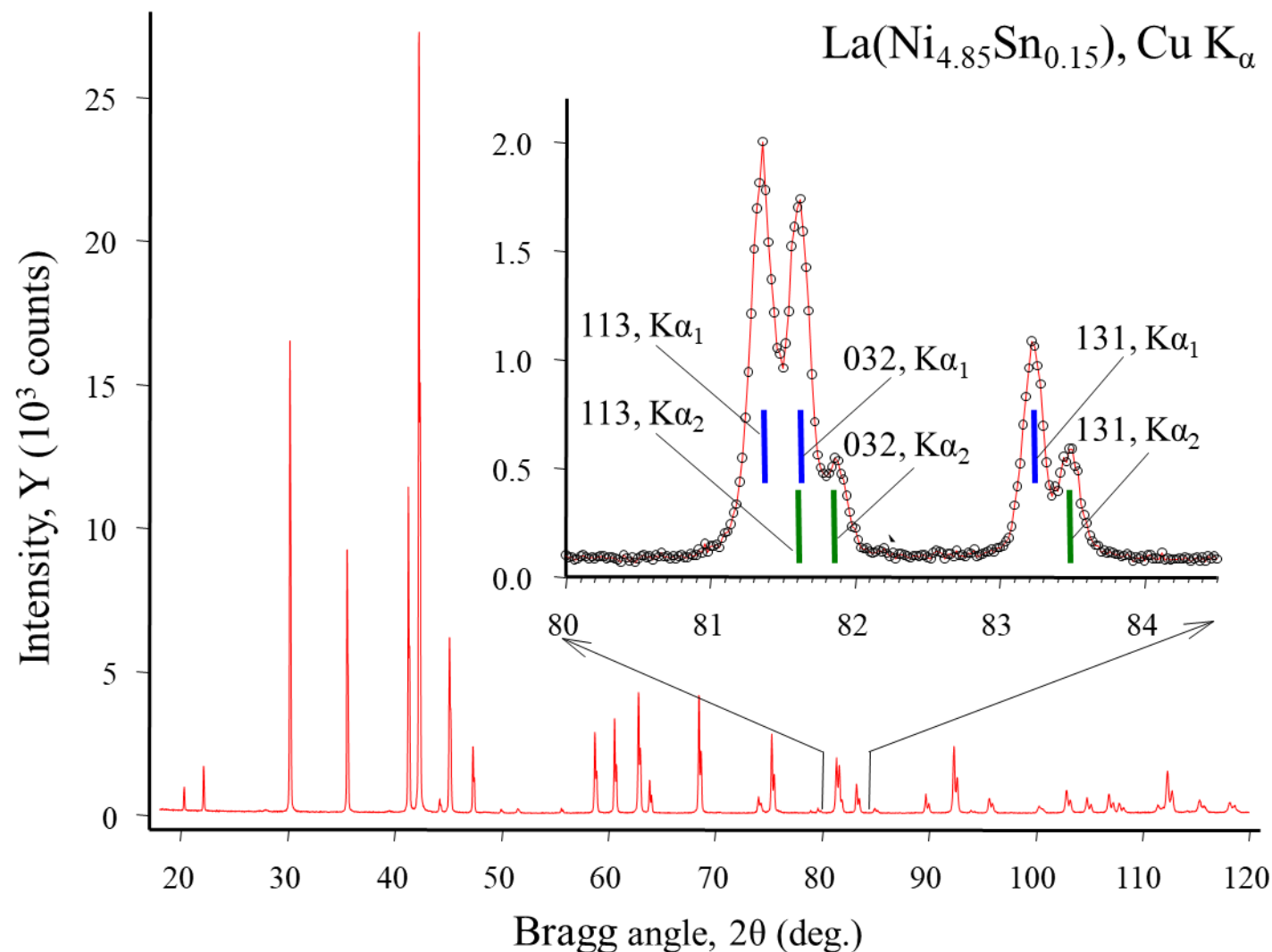
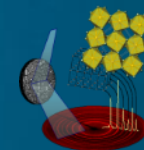


Figure 11.5. The X-ray powder diffraction pattern of polycrystalline $\text{LaNi}_{4.85}\text{Sn}_{0.15}$ recorded on a Rigaku TTRAX rotating anode powder diffractometer using $\text{Cu } K\alpha$ radiation. The expanded view shows three Bragg peaks between 80 and $84.5^\circ 2\theta$. Each of the Bragg peaks consists of nearly resolved $K\alpha_1/K\alpha_2$ doublets. The $K\alpha_2$ component of the strongest Bragg peak (113) at $2\theta \cong 81.6^\circ$ is completely overlapped with the $K\alpha_1$ component of the second Bragg peak (032) at $2\theta \cong 81.6^\circ$. The $K\alpha_1$ component of 113 peak at $2\theta \cong 81.38^\circ$, the sum of the 113 $K\alpha_2$ and 032 $K\alpha_1$ components at $2\theta \cong 81.6^\circ$ and the 032 $K\alpha_2$ component at $2\theta \cong 81.82^\circ$ are well resolved. Every point in the inset represents a single experimental measurement, $Y(2\theta_i)$. The lines connecting the data points are guides for the eye.

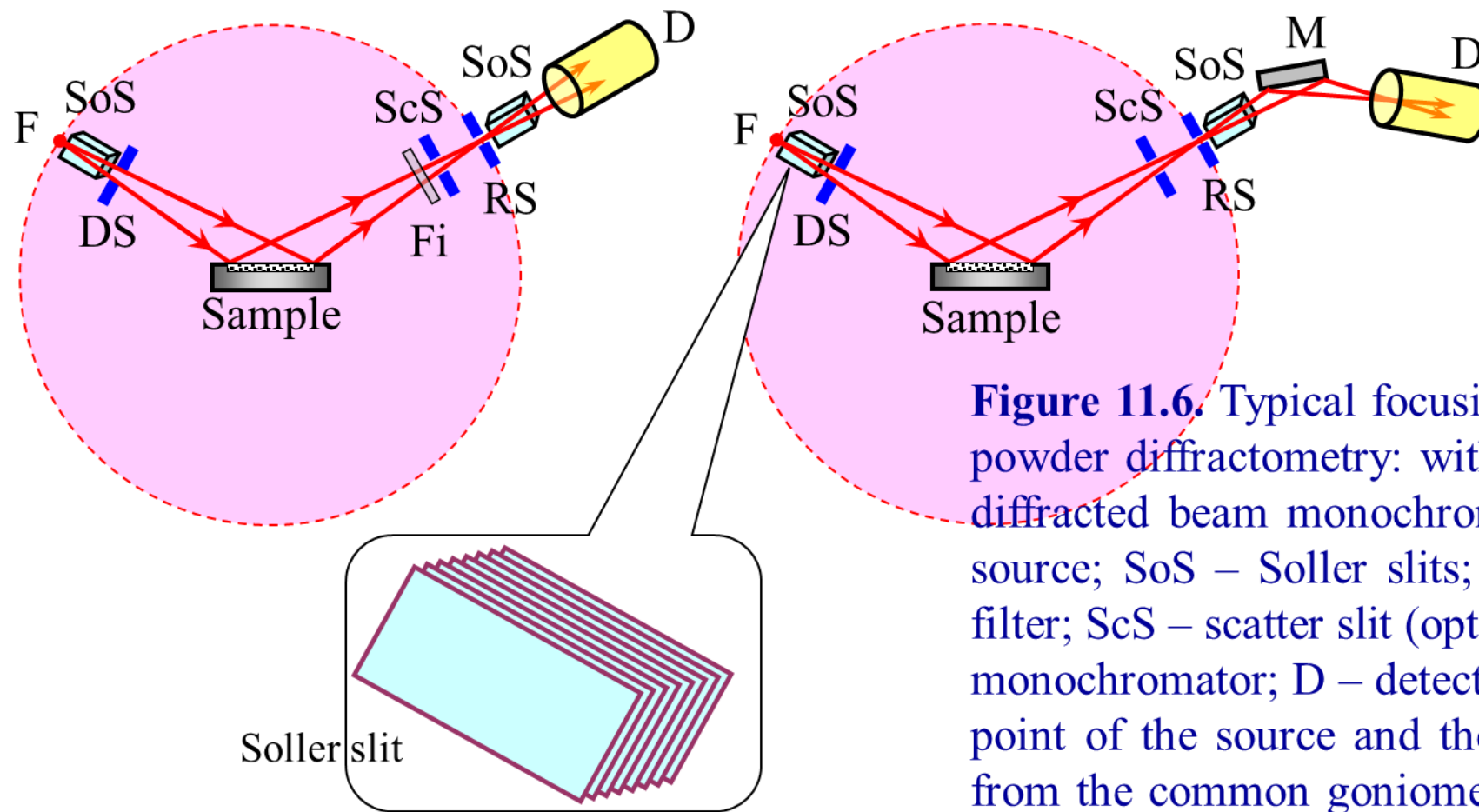
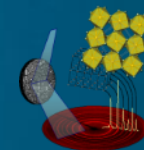


Figure 11.6. Typical focusing optics employed in modern powder diffractometry: without (*left*) and with (*right*) the diffracted beam monochromator. F – focus of the X-ray source; SoS – Soller slits; DS – divergence slit; Fi – β -filter; ScS – scatter slit (optional); RS – receiving slit; M – monochromator; D – detector. In each case, both the focal point of the source and the receiving slit are equidistant from the common goniometer axis, which coincides with the center of the sample. These points are located on the surface of a cylinder known as the goniometer circle, shown using a dashed line.

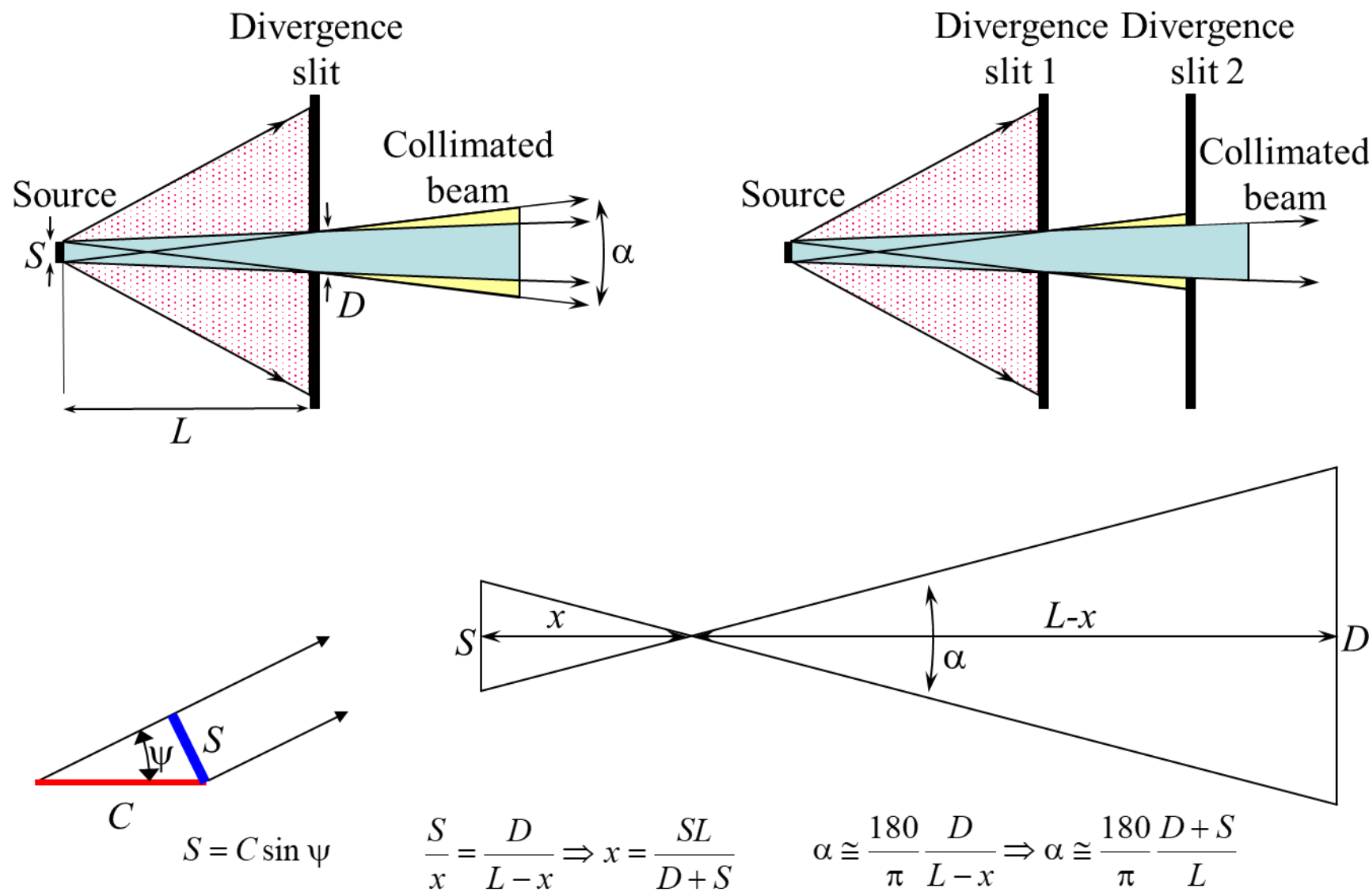
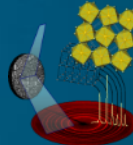


Figure 11.7. The schematic showing collimation of the incident X-ray beam by using a single divergence slit (*top, left*) or coupled divergence slits (*top, right*). The schematic on the bottom left illustrates the size of the source (S) when the projection of the cathode (C) is viewed at a take-off angle, ψ . Equation 11.1 is derived on the *bottom, right*.

$$\alpha(^{\circ}) \cong \frac{180}{\pi} \frac{D + S}{L} \quad \text{Eq. 11.1}$$

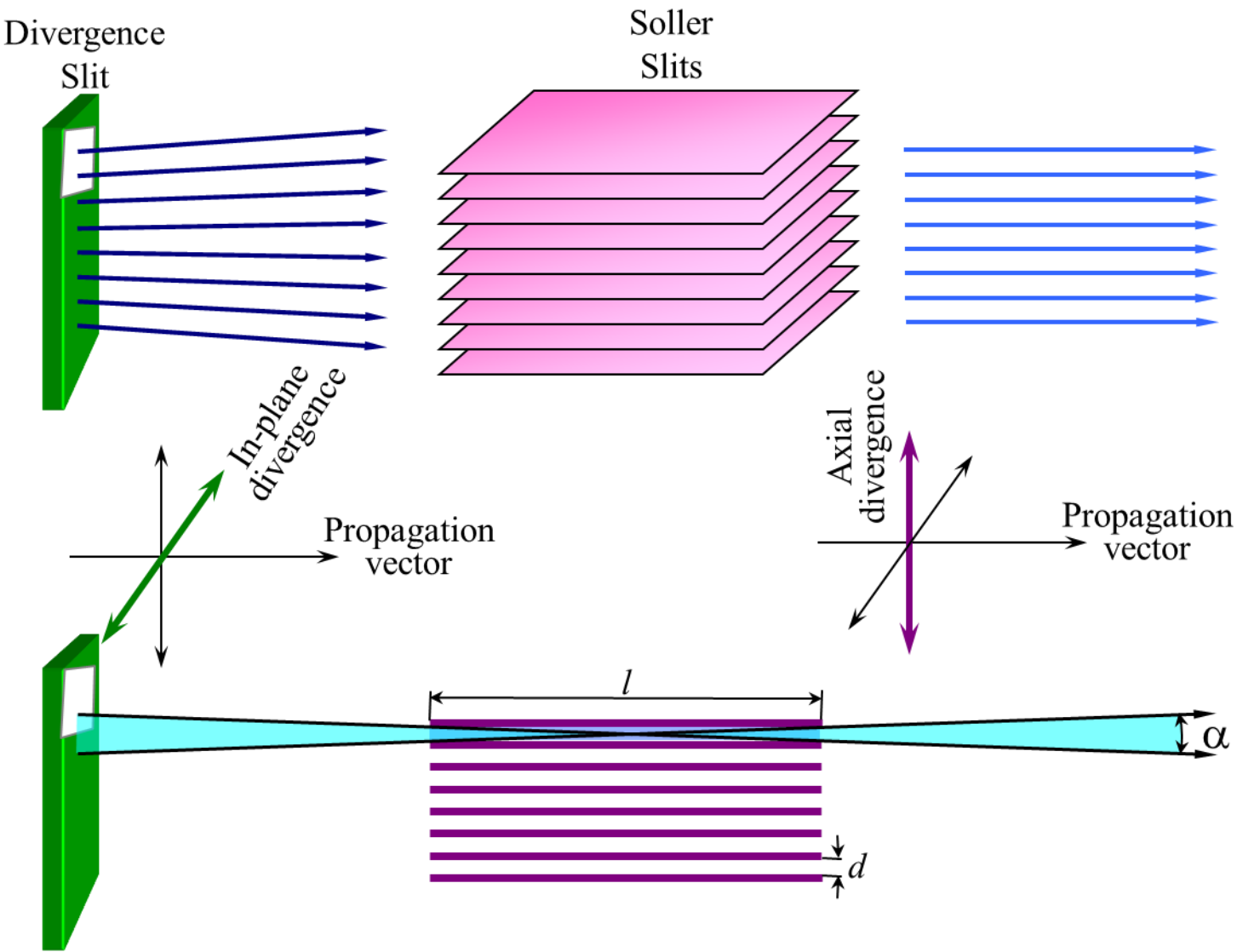
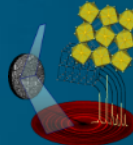


Figure 11.8. The schematic showing how the X-ray beam is collimated by using both the divergence and Soller slits (*top*). The beam, collimated in-plane by the divergence slit, is further collimated axially by the Soller slits. The coordinates in the middle of the drawing indicate the corresponding directions.

$$\alpha(^{\circ}) \cong \frac{180}{\pi} \frac{2d}{l}$$

Eq. 11.2

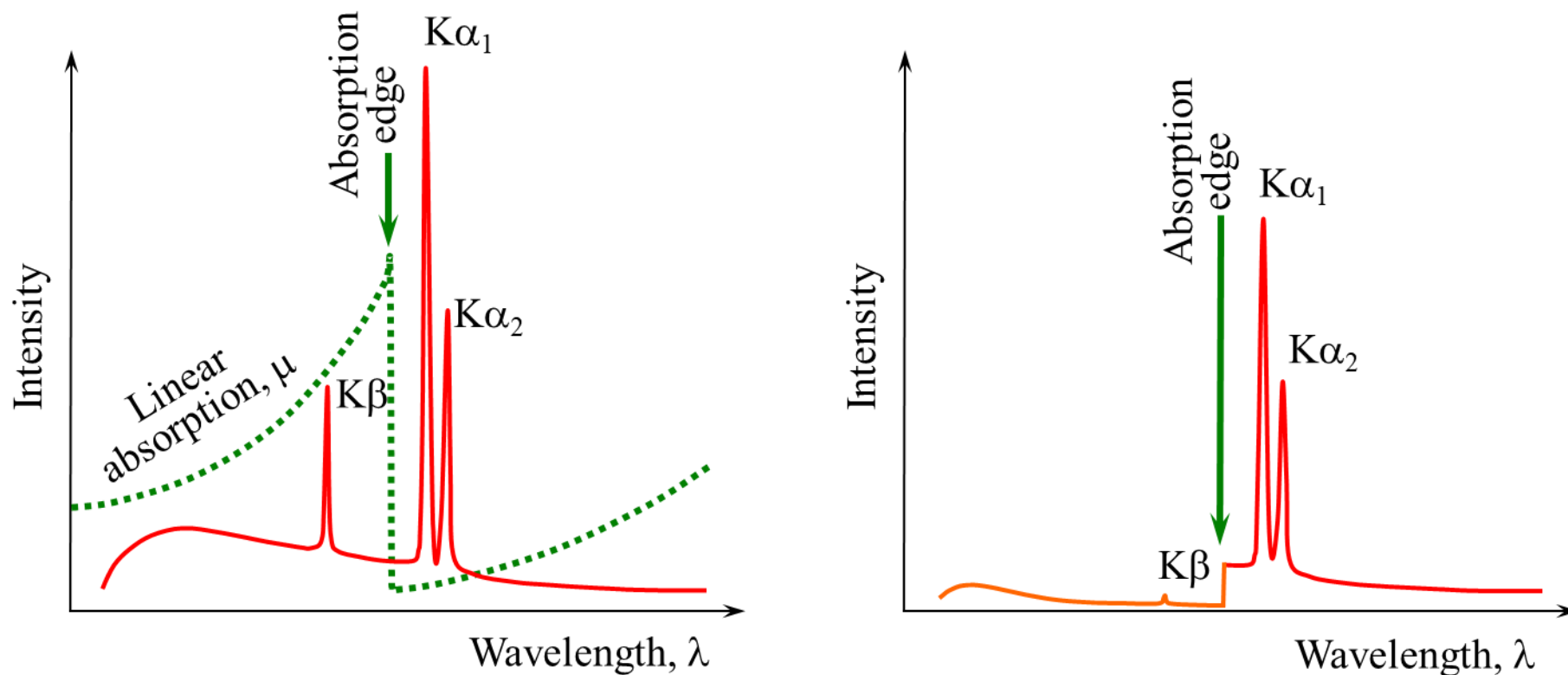
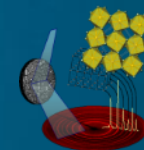


Figure 11.9. *Left* - the schematic of the X-ray emission spectrum shown as the solid line overlapped with the schematic of the $\mu(\lambda)$ function of the properly selected β -filter material (*dotted line*). *Right* – the resultant distribution of intensity after filtering as a function of the wavelength.

$$\frac{I_{K\alpha_1}}{I_{K\beta}} = 5 \frac{\exp(-\mu_{\alpha} t)}{\exp(-\mu_{\beta} t)} \quad \text{Eq. 11.3}$$

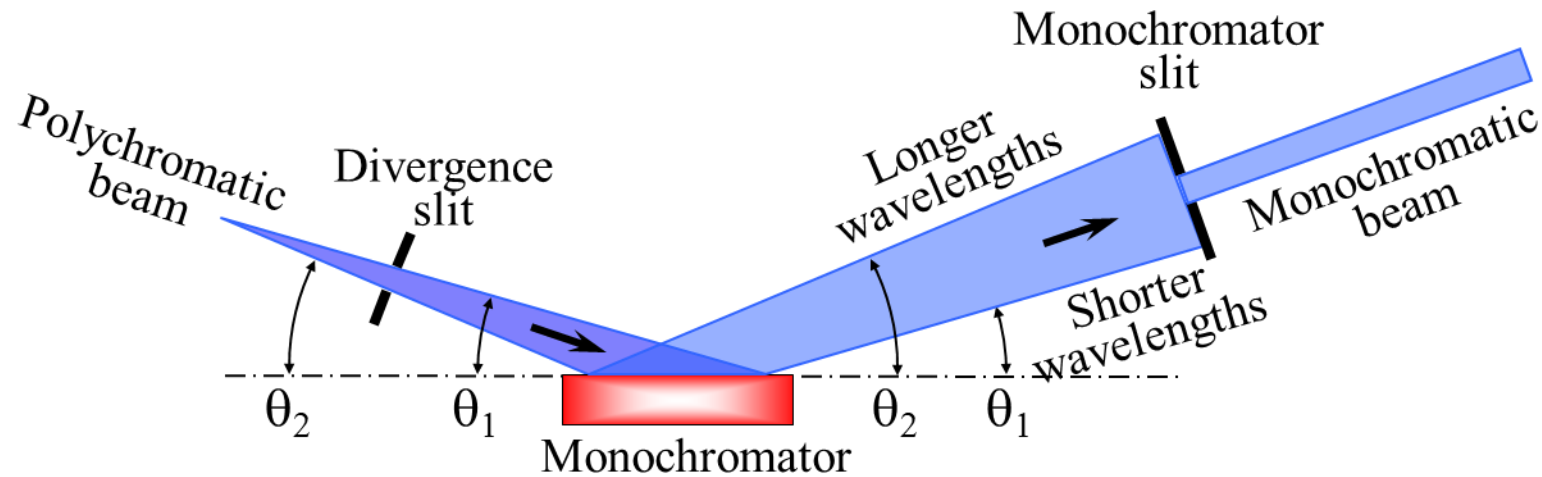
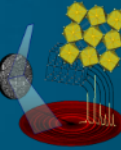


Figure 11.10. The schematic explaining the principle of monochromatization using a single crystal monochromator. Generally, $\theta^M \cong (\theta_2 + \theta_1)/2$. The directions of the propagation vectors are indicated by arrows.

$$2d_{hkl}^M \sin \theta^M = n\lambda_t \quad \text{Eq. 11.4}$$

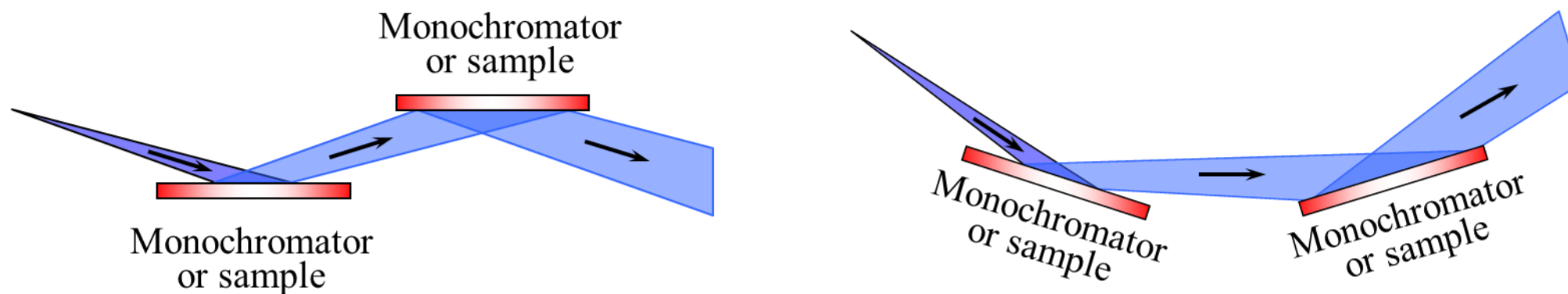
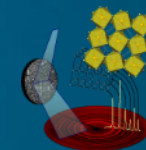


Figure 11.11. Parallel (*left*) and angular (*right*) arrangements of two crystal monochromators or the sample and the crystal monochromator commonly used to improve the monochromatization of the resultant X-ray beam.

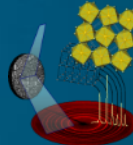


Table 11.1. Comparison of the three common monochromator/sample configurations used in powder diffractometry.

Property	a ^a	b	c
Position in the beam	Diffracted	Diffracted	Primary
Orientation to the sample	Parallel	Angular	Parallel
Shape of the crystal	Flat/curved	Curved	Flat/curved
Intensity loss	Low	Low	Low/high
K α_1 and K α_2 separation	No	No	No/complete
Alignment	Simple	Simple	Simple/complex
Additional torque on the detector arm	High	Low	None
Use with area detectors	Impossible	Impossible	Possible

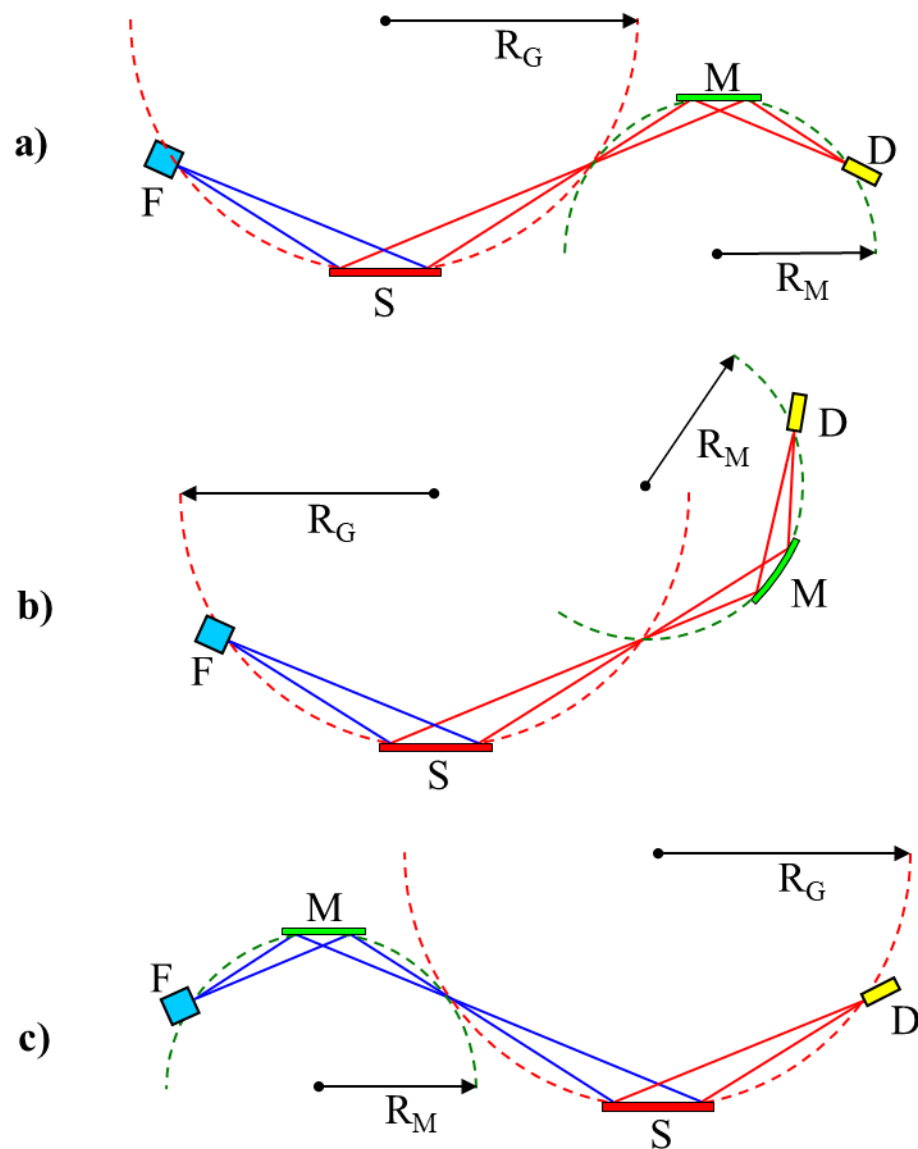
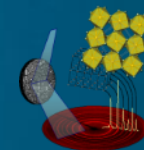


Figure 11.12. The three different monochromator/sample geometries used in powder diffraction: (a) flat *diffracted* beam monochromator, parallel arrangement; (b) curved *diffracted* beam monochromator, angular arrangement, and (c) flat *primary* beam monochromator, parallel arrangement. F – focus of the X-ray source, S – sample, M – crystal monochromator, D – detector, R_M – radius of the monochromator focusing circle, R_G – radius of the goniometer focusing circle.

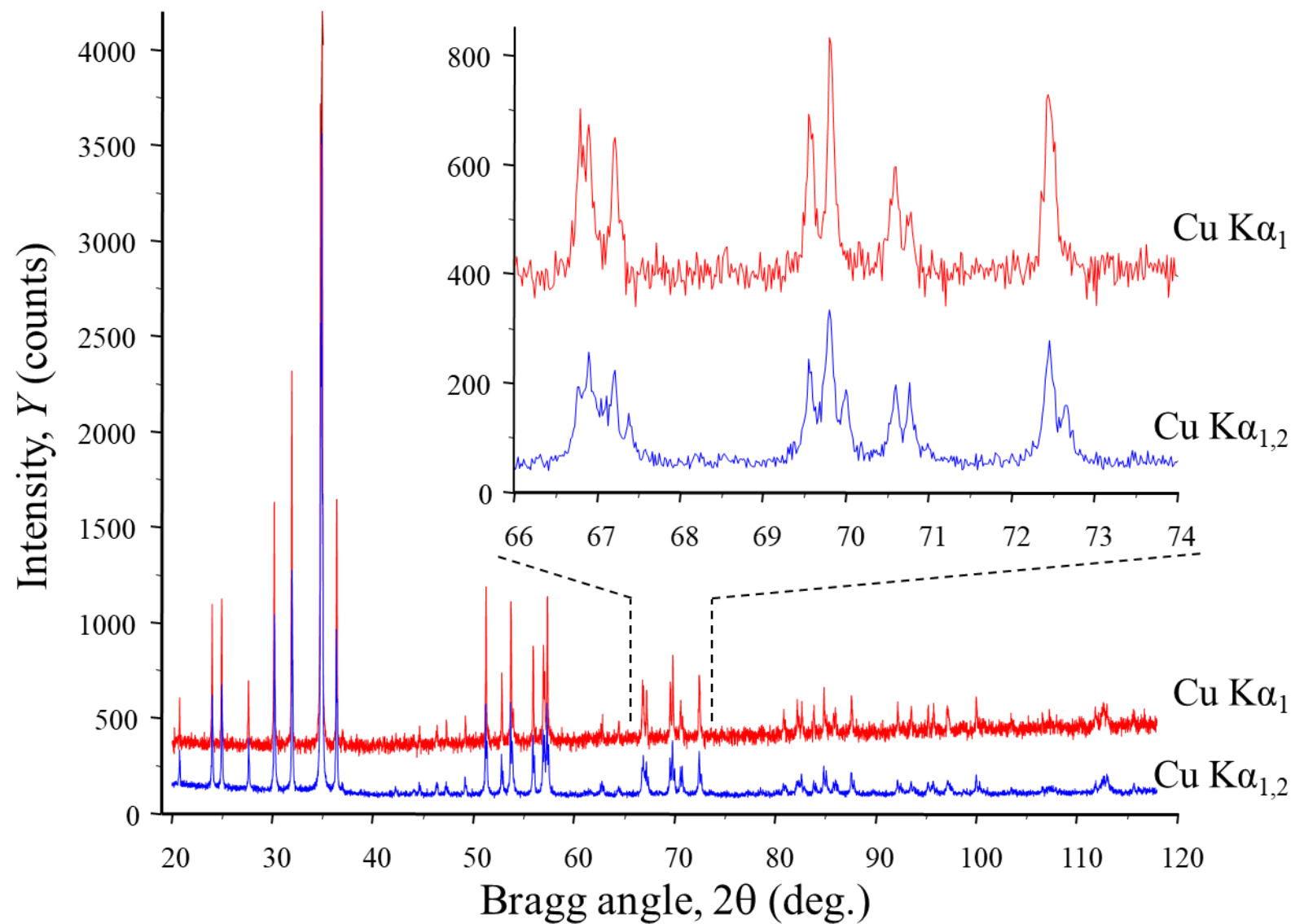
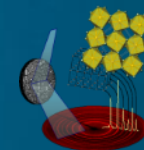


Figure 11.13. Powder diffraction patterns collected from the same $\text{Gd}_5(\text{Si}_{1.5}\text{Ge}_{1.5})$ specimen using $\text{Cu K}\alpha$ radiation. The patterns shown in *blue* were recorded using a strip detector and a diffracted beam monochromator (the $\text{K}\beta$ component of the characteristic spectrum was eliminated but the $\text{K}\alpha_2$ component was not). The patterns in *red* have been recorded using the primary beam Johansson monochromator, which results in purely monochromatic $\text{Cu K}\alpha_1$ radiation. The increased background in the top patterns is due to high fluorescence of Gd.

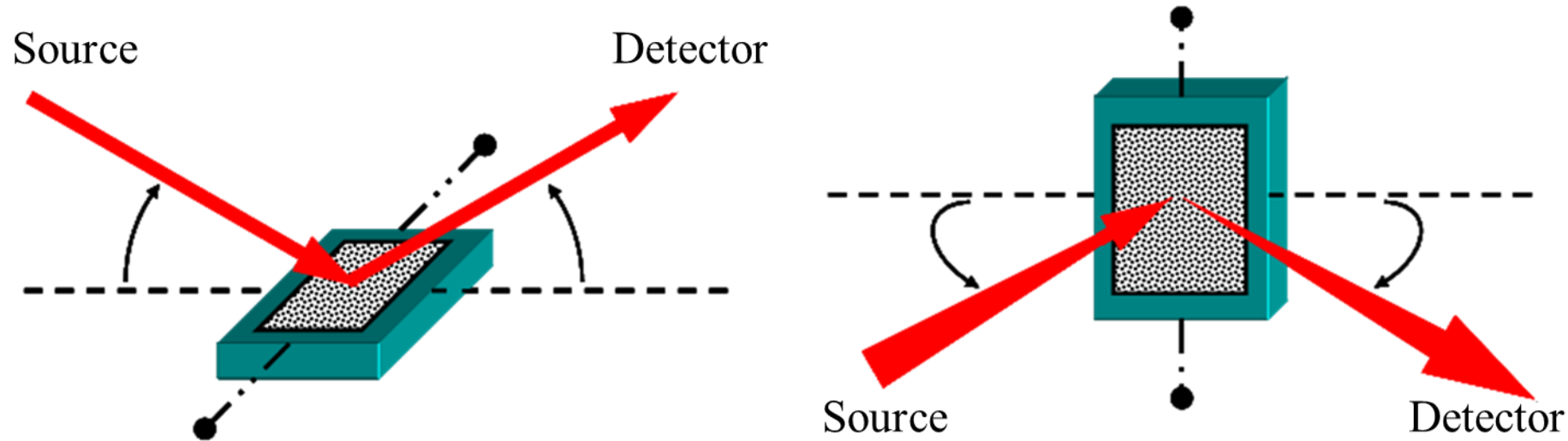
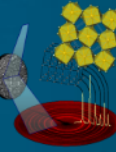
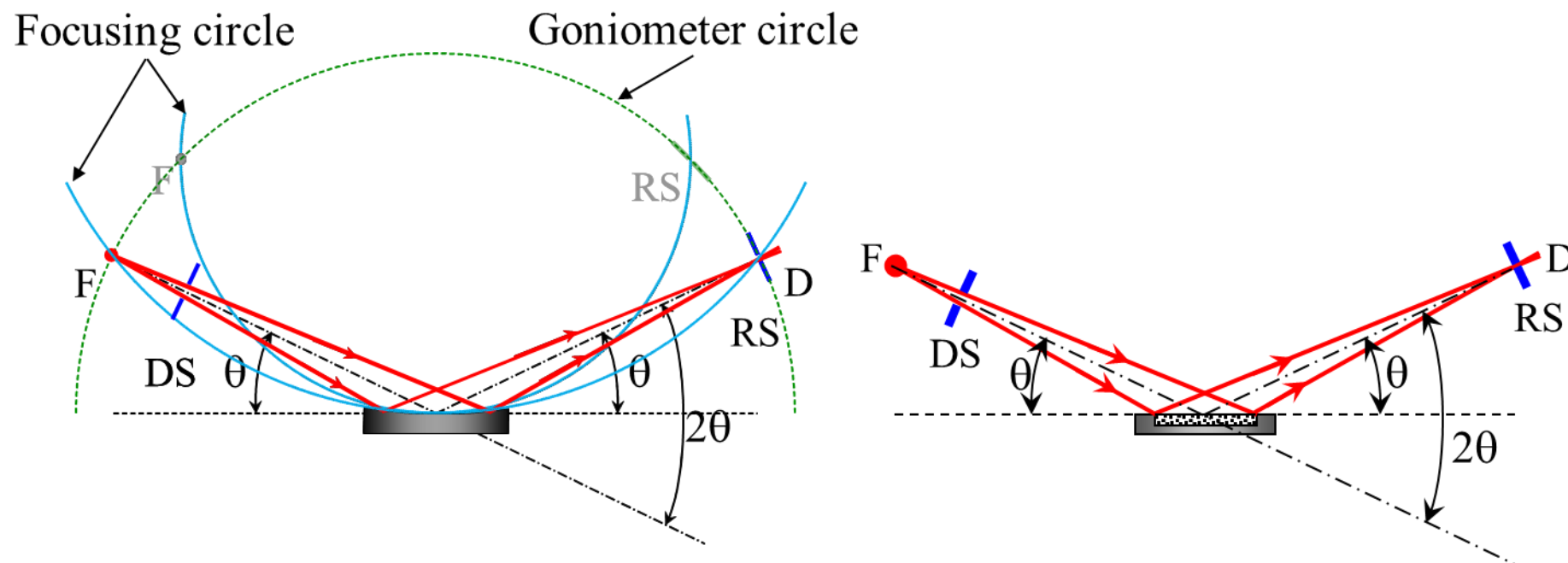
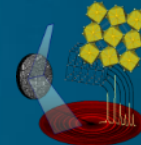
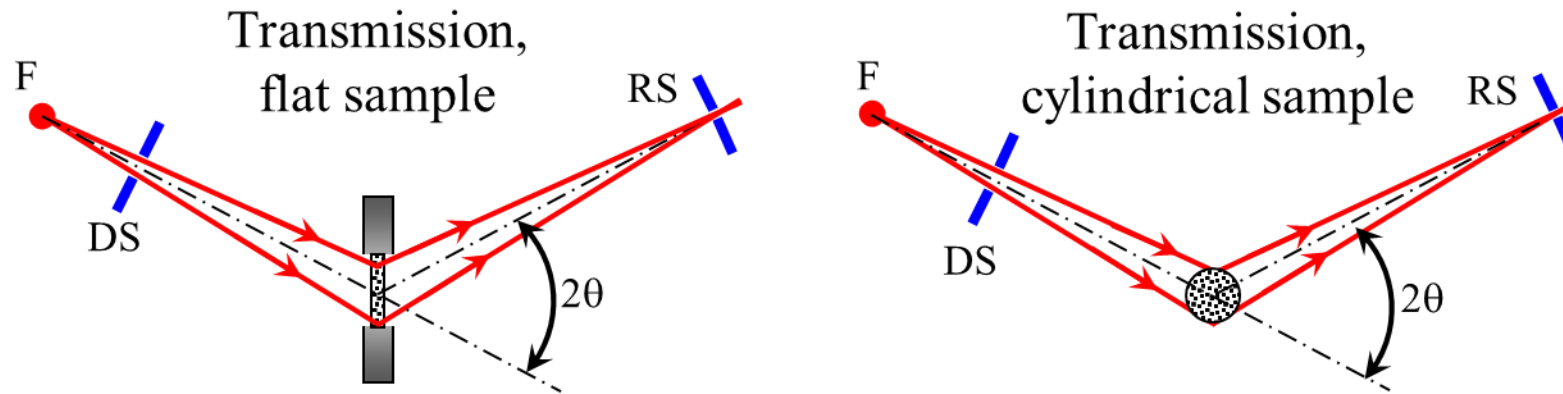
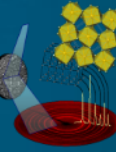


Figure 11.14. Horizontal (*left*) and vertical (*right*) orientations of a flat sample, along with the goniometer axis, which is depicted using a *dash-double-dotted line* with *small circles* at the ends. The *dashed line* indicates the location of the optical axis, which connects the focus of the X-ray tube, the receiving slit, and the sample center.



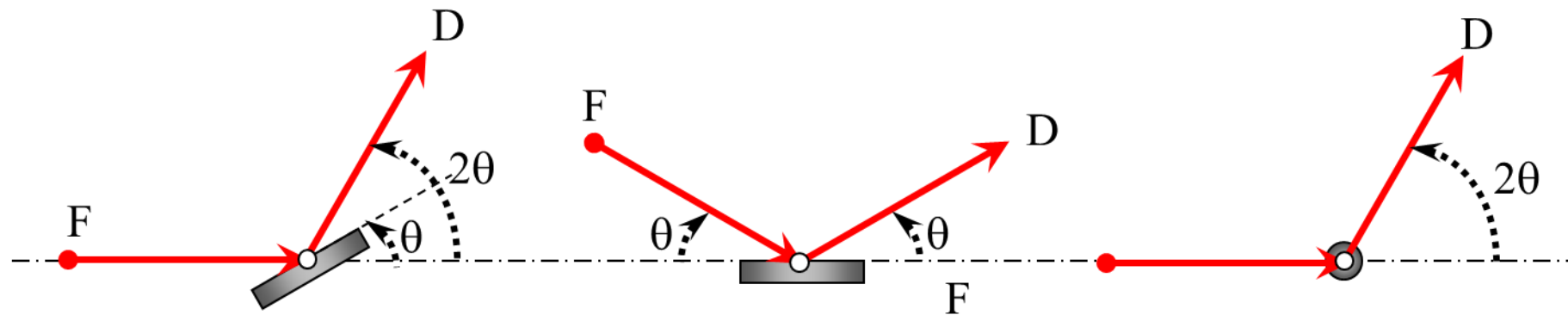
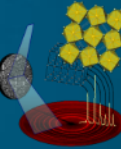
F – focus of the X-ray source, D – detector, θ – Bragg angle, DS – divergence slit, RS – receiving slit.

Figure 11.15. The schematic of the ideal focusing geometry (*left*) and its common modification known as the Bragg–Brentano geometry using a flat sample (*right*) when the self-focused diffracted beam is registered by the detector after reflection from the sample. To achieve ideal focusing of the reflected divergent beam, the curvature of the sample must coincide with the circumference of the focusing circle as indicated by two *circles* of different radius shown in *blue* on the *left*. This is impractical because the curvature of the specimen becomes a function of the Bragg angle, and therefore, flat specimens are employed instead.



F – focus of the X-ray source, θ – Bragg angle, DS – divergence slit, RS – receiving slit.

Figure 11.16. Transmission geometry in the case of flat (*left*) and cylindrical (*right*) samples.



F – focus of the X-ray tube indicating the position of the X-ray source arm, D – detector arm, θ – Bragg angle.

Figure 11.17. Synchronization of the goniometer arms: the X-ray source is stationary while the sample and the detector rotations are synchronized to fulfill the θ – 2θ requirement (*left*); the sample is stationary while the source and the detector arms are synchronized to realize the θ – θ condition (*middle*) – this geometry is in common use at present; only the detector arm revolves around the goniometer axis in the case of a cylindrical sample (*right*). The common goniometer axis (which is perpendicular to the plane of the projection) around which the rotations are synchronized is shown as the open circle in each of the three drawings. The location of the optical axis is shown as the dash-dotted line

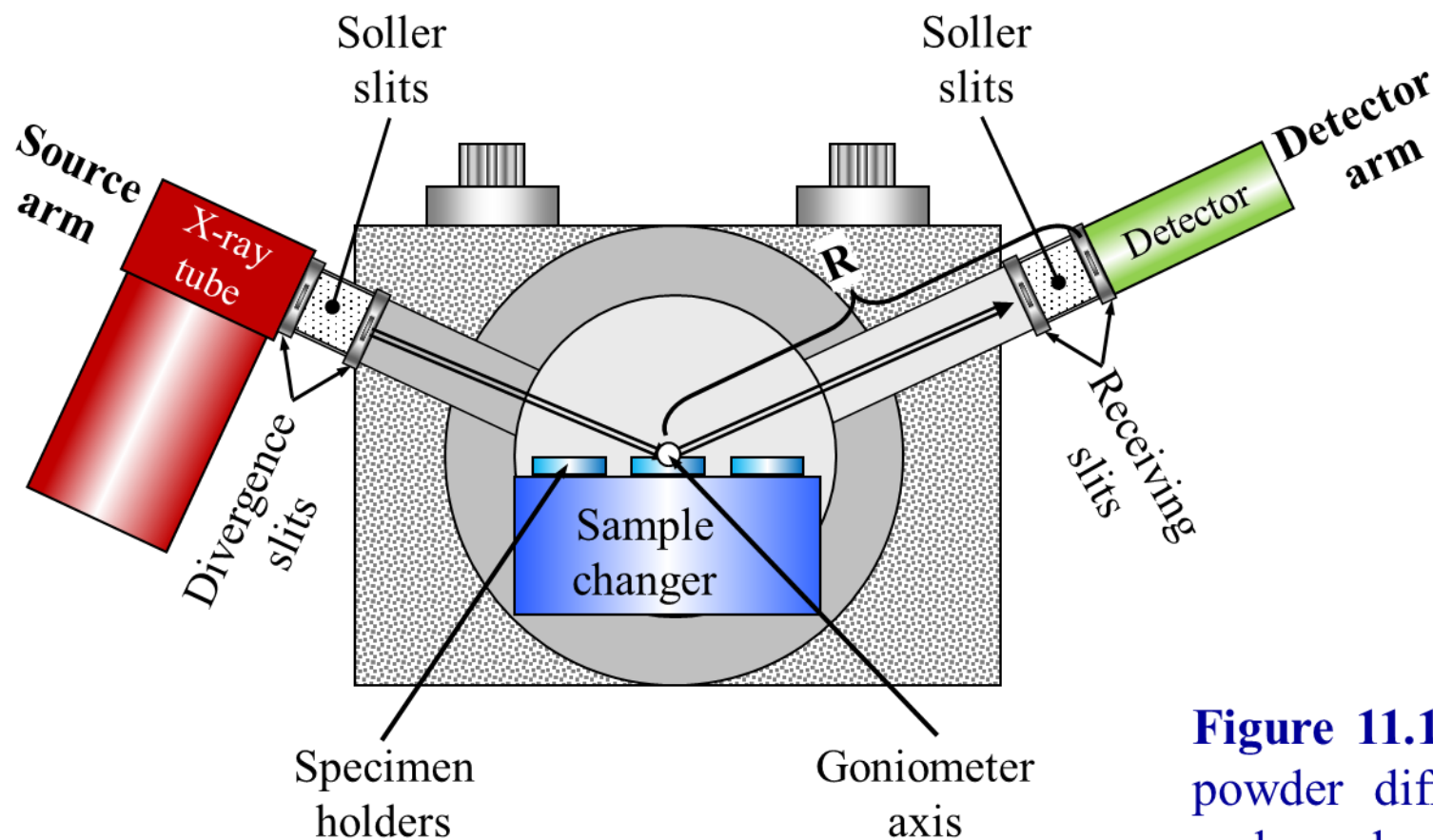
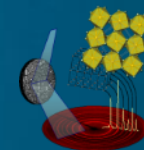


Figure 11.18. The schematic of a goniostat of a powder diffractometer with the horizontal axis and synchronized rotations of both the source and detector arms. R – is the radius of the goniometer.

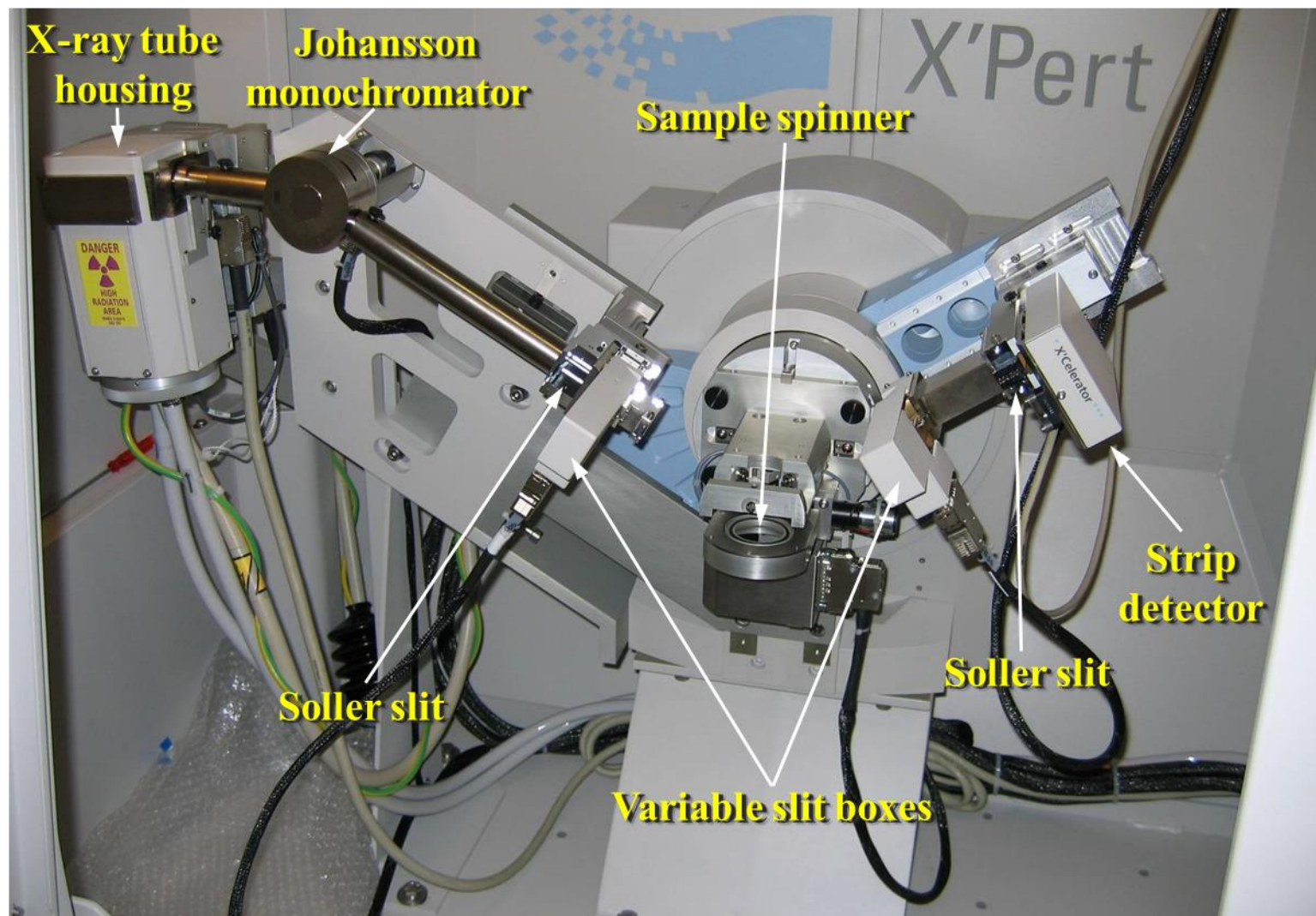
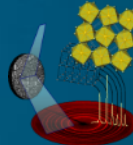


Figure 11.19. The overall view of the goniostat of the Malvern Panalytical X'Pert powder diffractometer. This diffractometer has the horizontal goniometer axis, stationary X-ray source and synchronized rotations of both the detector arm and sample holder. The goniometer is equipped with a Johansson-type primary beam monochromator, which eliminates $\text{Cu K}\alpha_2$ or $\text{Co K}\alpha_2$ wavelengths in addition to $\text{K}\beta$ components of the characteristic spectrum, and a solid-state real-time multiple strip detector, which accelerates data collection.

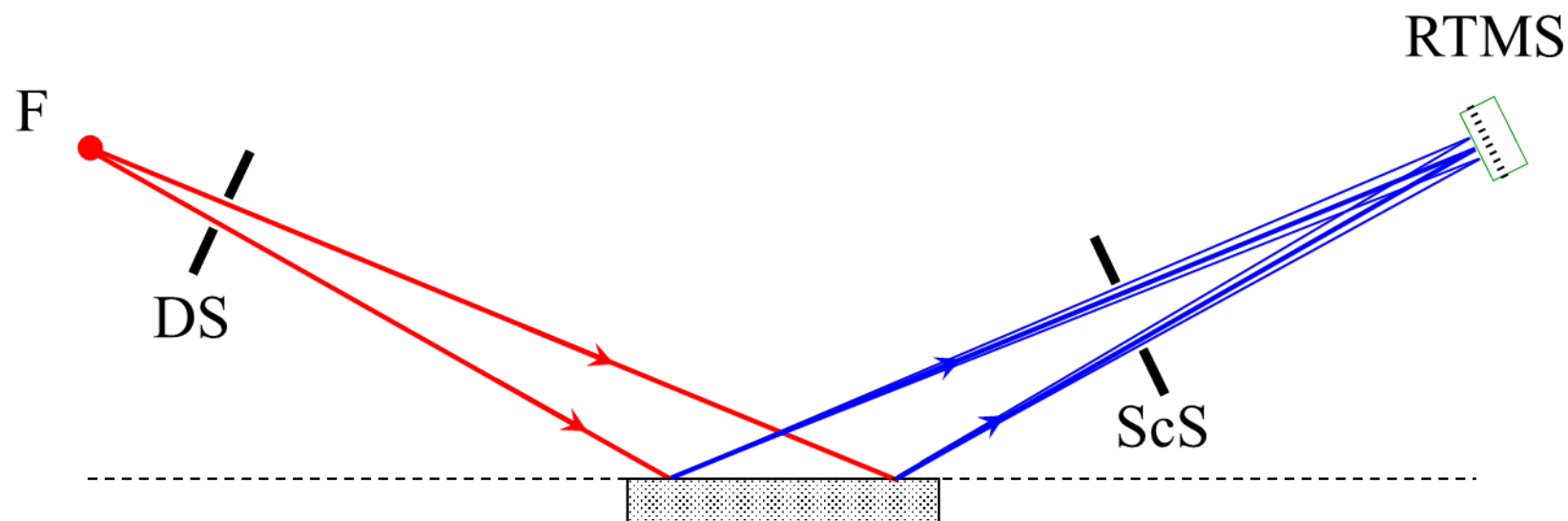
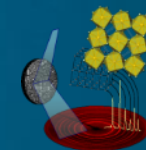


Figure 11.20. The schematic of focusing in the Bragg–Brentano geometry when the real-time multiple strip detector is utilized. F – focus of the X-ray source, DS – divergence slit, ScS – scatter slit, RTMS – strip detector.

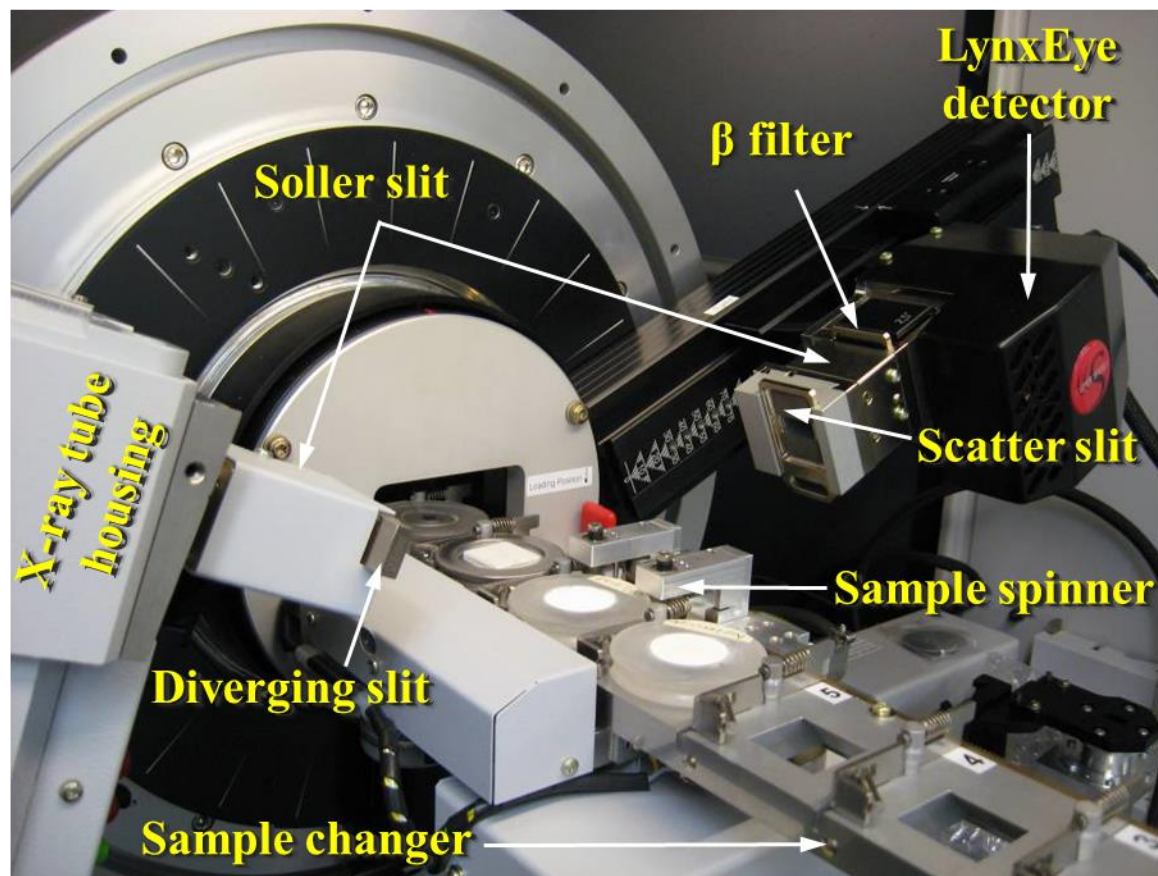
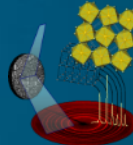


Figure 11.21. The overall view of the Bruker D8 Advance powder diffractometer. It has a horizontal goniometer axis and so-called θ - θ geometry – a synchronized rotation of both the X-ray source and the detector arms. This diffractometer is equipped with Cu K α radiation from sealed tube, 2.5° Soller slits on both incident and diffracted beams, a Ni β -filter, a 9-samples changer with spinner, and a LynxEye multiple strip detector for fast data collection, which measures 3.6° 2θ (usually 180 data points) simultaneously while providing high resolution data.

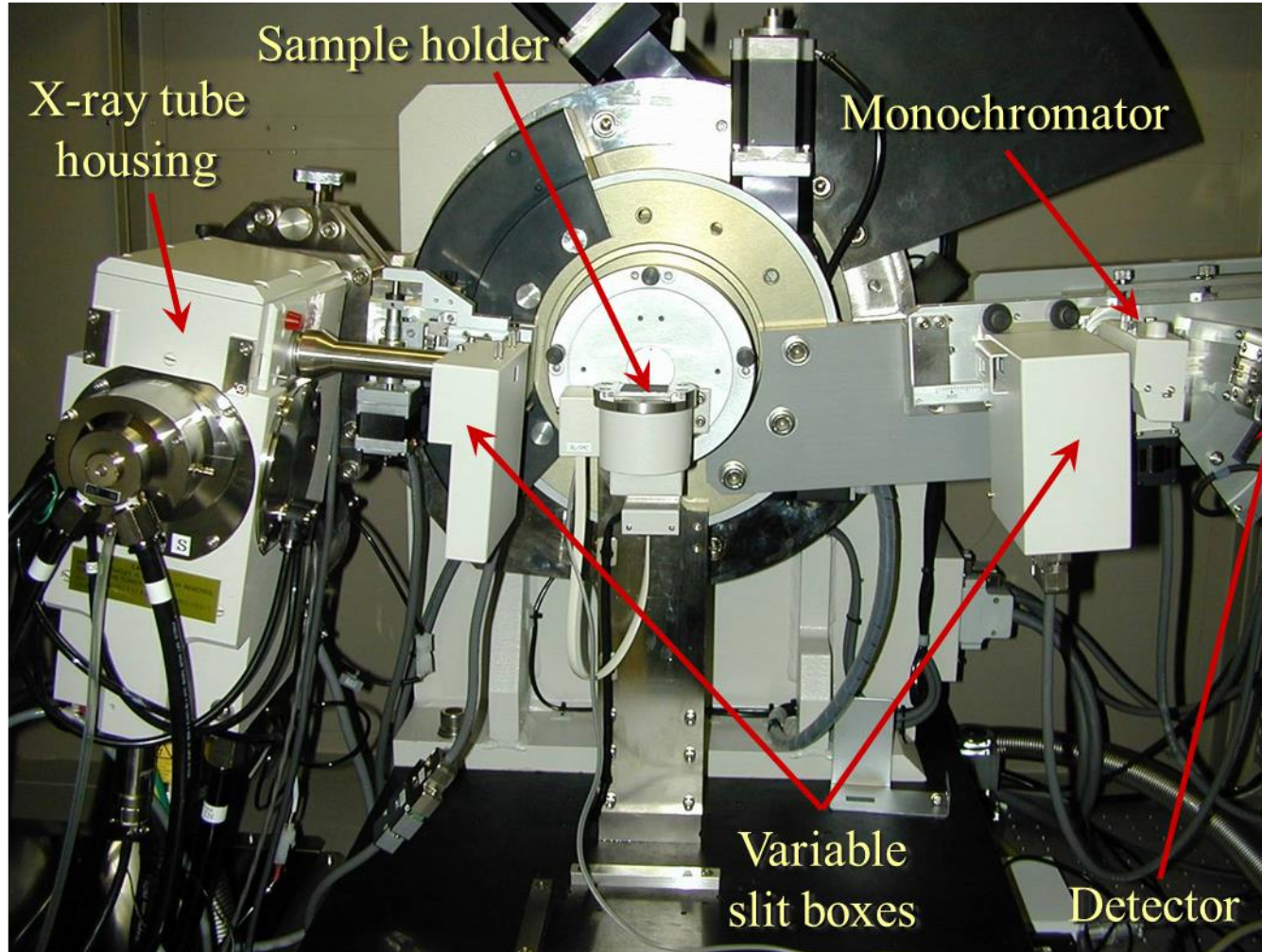
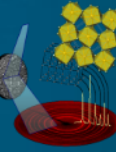
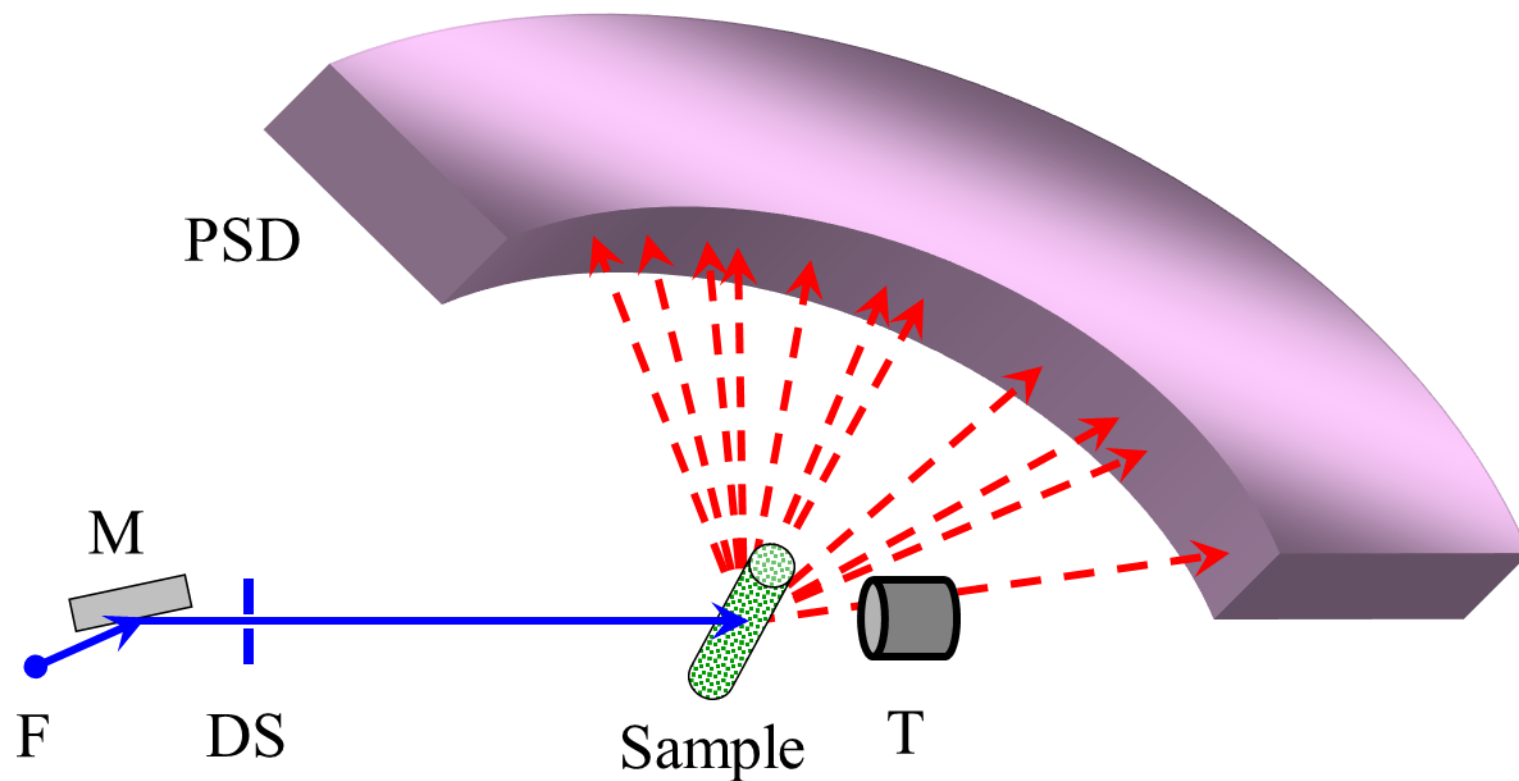
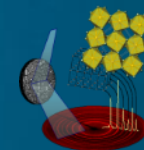


Figure 11.22. The overall view of the goniostat of the Rigaku TTRAX rotating anode powder diffractometer with the horizontal goniometer axis, and synchronized rotations of both the X-ray source and detector arms. This θ - θ goniometer is equipped with variable divergence, scatter and receiving slits, curved crystal monochromator, and scintillation detector.



F – focal point of the X-ray source,
M – monochromator,
DS – divergence slit,
T – incident beam trap.

Figure 11.23. The schematic of a powder diffractometer cylindrical sample in the transmission mode and a curved position sensitive detector (PSD). *Solid arrows* indicate the incident beam and *broken arrows* indicate the diffracted beams pathways.

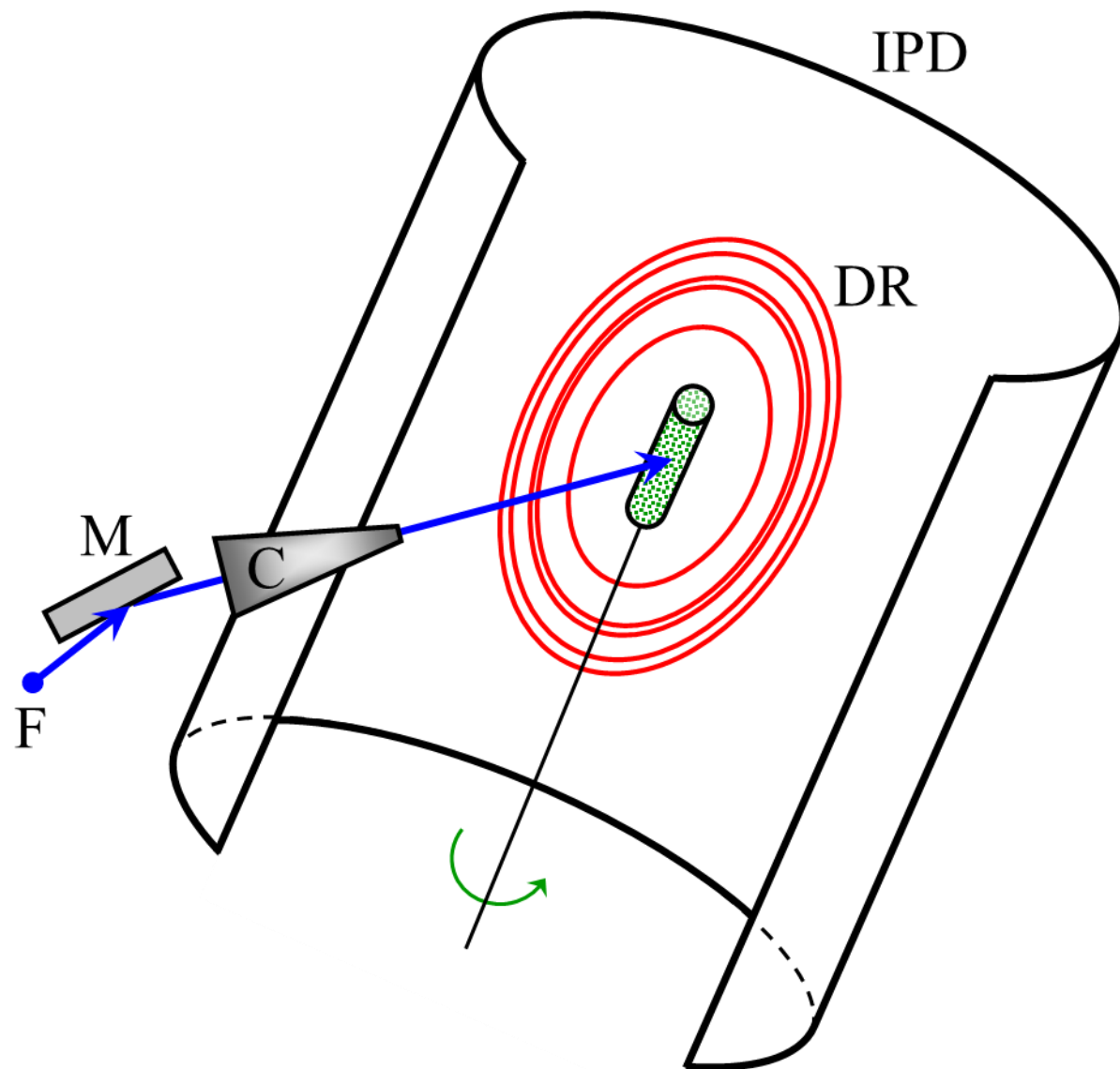
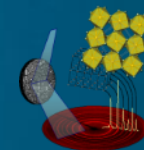


Figure 11.24. The schematic of a powder diffractometer with the vertical goniometer axis, cylindrical sample in the transmission mode, and image plate detector (IPD). Blue arrows show the incident beam path. *Red rings* indicate intercepts of Debye cones with the IPD.

F – focal point of the X-ray source,
M – monochromator,
C – collimator,
DR – Debye rings.

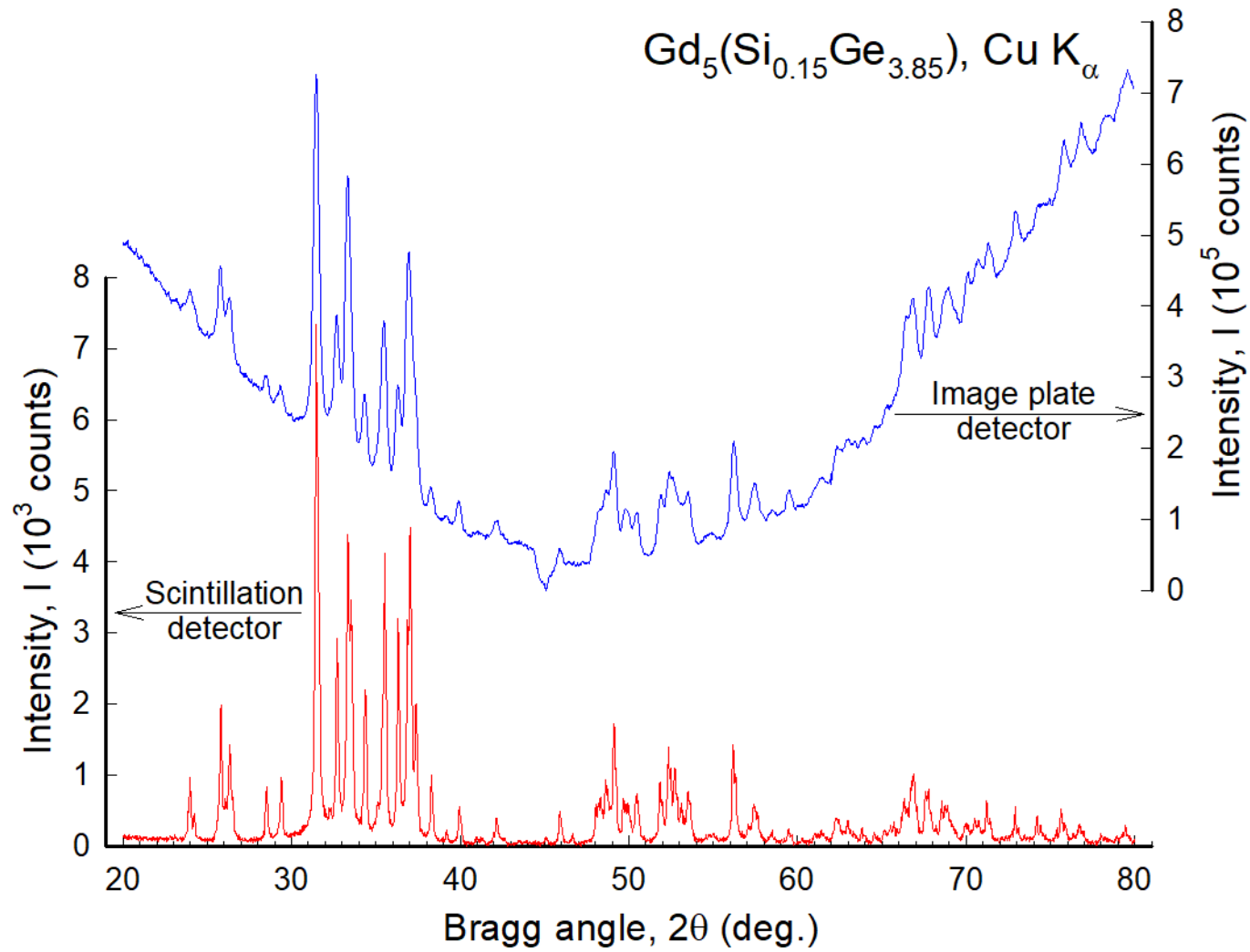
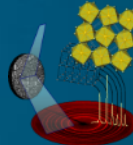


Figure 11.25. Comparison of the X-ray diffraction data collected from the same powder using a point detector with a diffracted beam monochromator in Bragg–Brentano geometry (shown in *red*, left-hand intensity scale) versus an image plate detector with an incident beam monochromator (shown in *blue*, right-hand intensity scale) in the transmission geometry from a cylindrical sample. The extremely high and nonlinear background observed with the image plate detector is due to the strong X-ray fluorescence of Gd atoms interacting with Cu $\text{K}\alpha$ radiation.

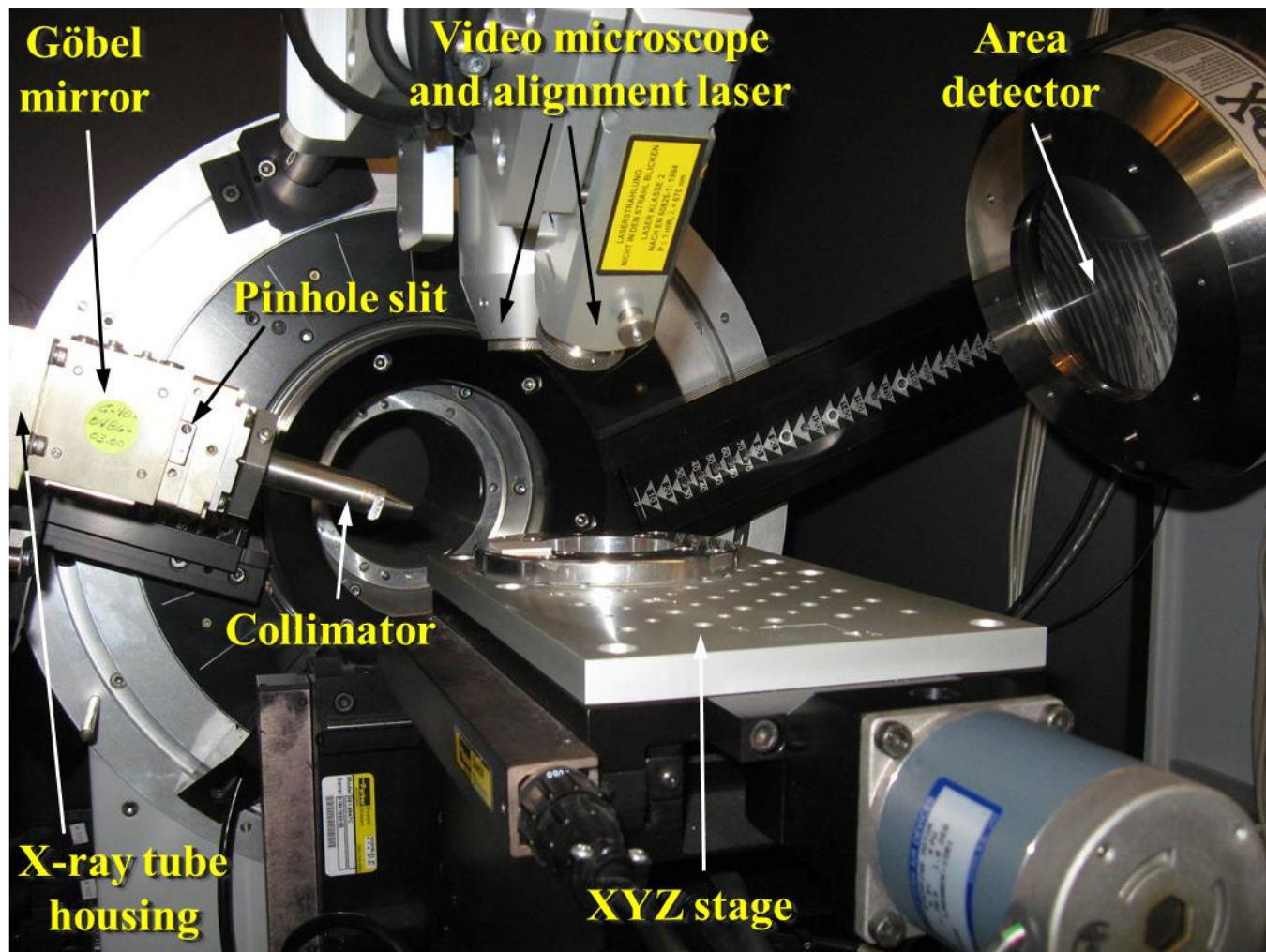
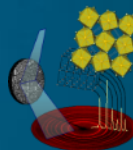


Figure 11.26. Overall view of the Bruker D8/C2 Discover goniometer configured for powder diffraction with GADDS (General Area Detector Diffraction System). The goniometer axis is horizontal, with independent rotations of both the X-ray source and detector arms. This goniometer is equipped with a Göbel mirror that produces a parallel beam, an XYZ sample stage, a video camera with a laser alignment system, and an area detector, specifically, the HiStar multi-wire gas proportional detector.

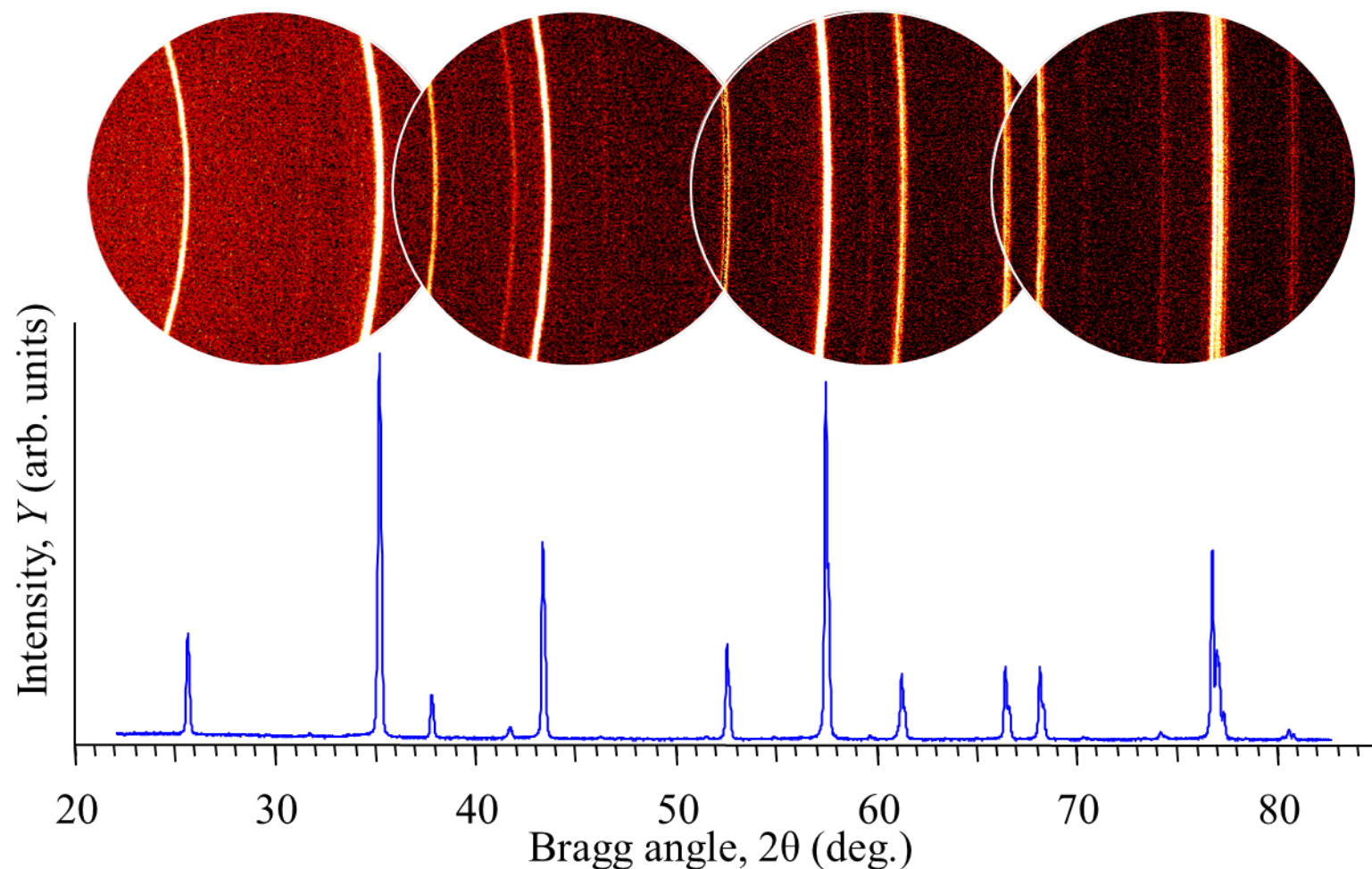
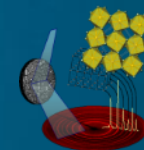


Figure 11.27. Examples of two-dimensional diffraction images (*top*) from a corundum plate (SRM 1976) collected on D8/C2 Discover-GADDS powder diffractometer using Cu K α radiation, Göbel mirror, HiStar detector at 30 cm with the resolution of 1024 $\times$ 1024 pixels. The plot at the *bottom* shows the corresponding one-dimensional pattern after the integration of the diffracted intensities along the Debye rings.

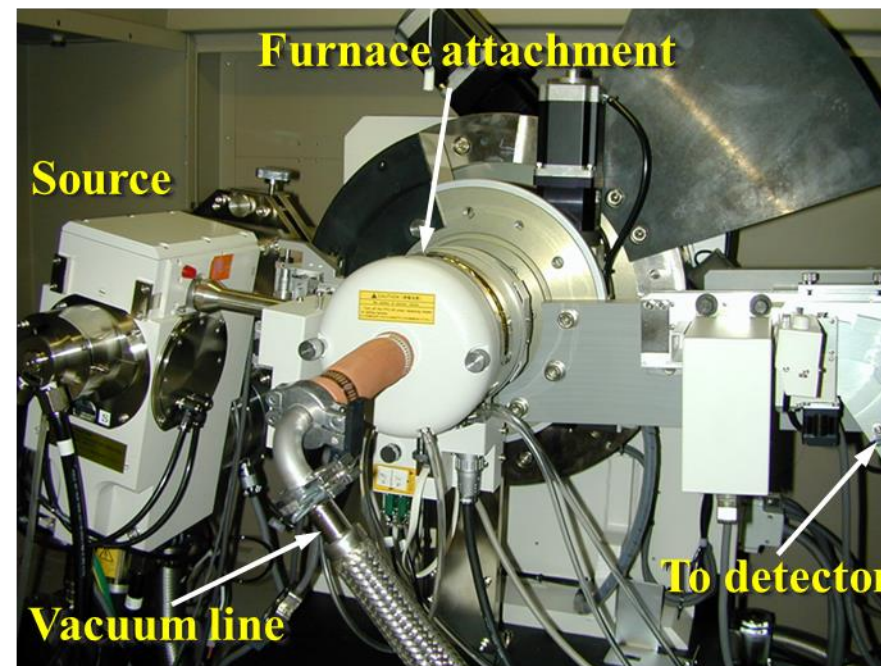
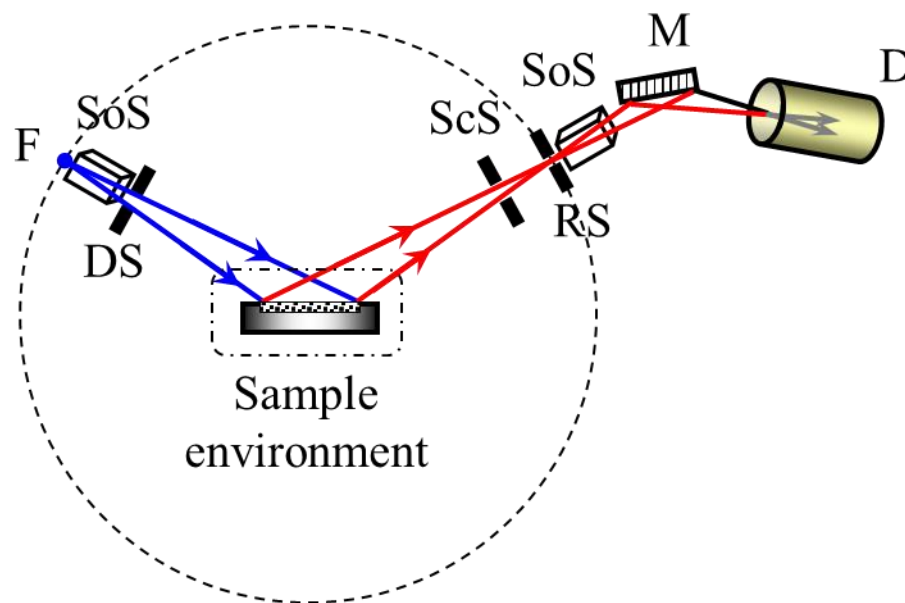
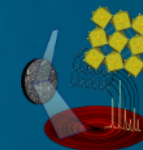


Figure 11.28. The schematic of the optics of a powder diffractometer with diffracted beam monochromator and a sample in a controlled environment (*left*), and a photograph of the Rigaku TTRAX goniometer with a furnace attachment (*right*).

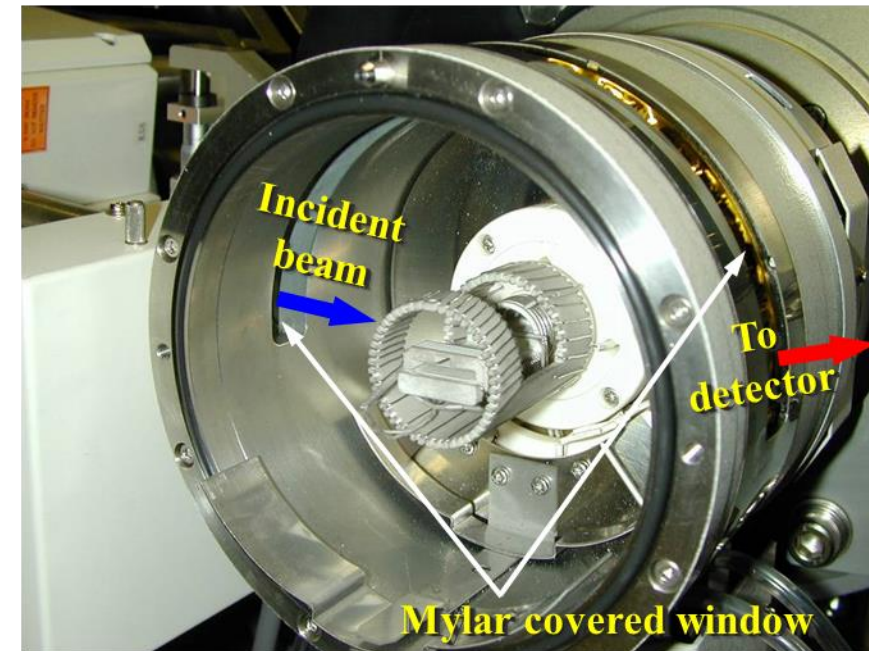
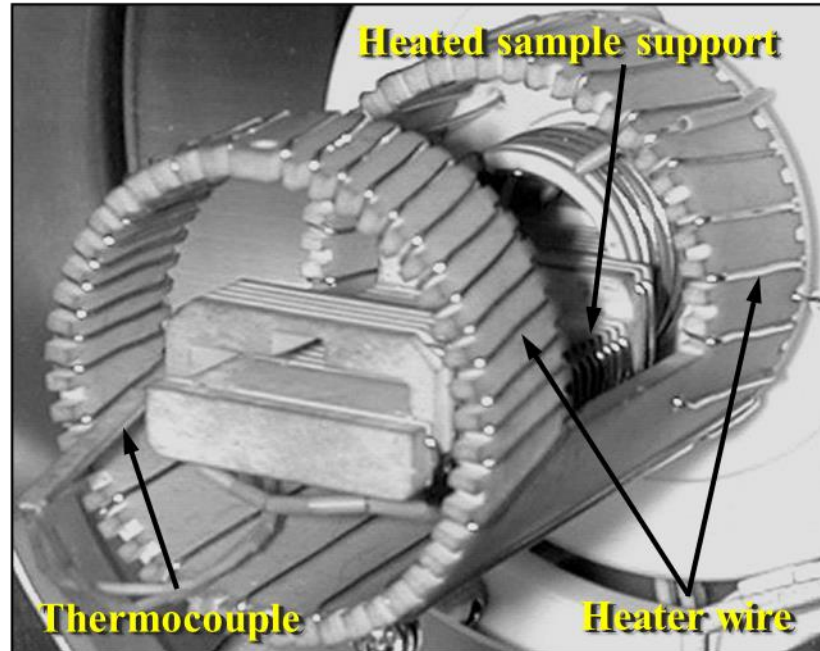
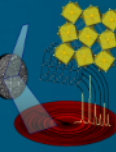


Figure 11.29. Sample heater (*left*) and the overall view of the high temperature furnace attachment with the cover removed to expose the inside (*right*).

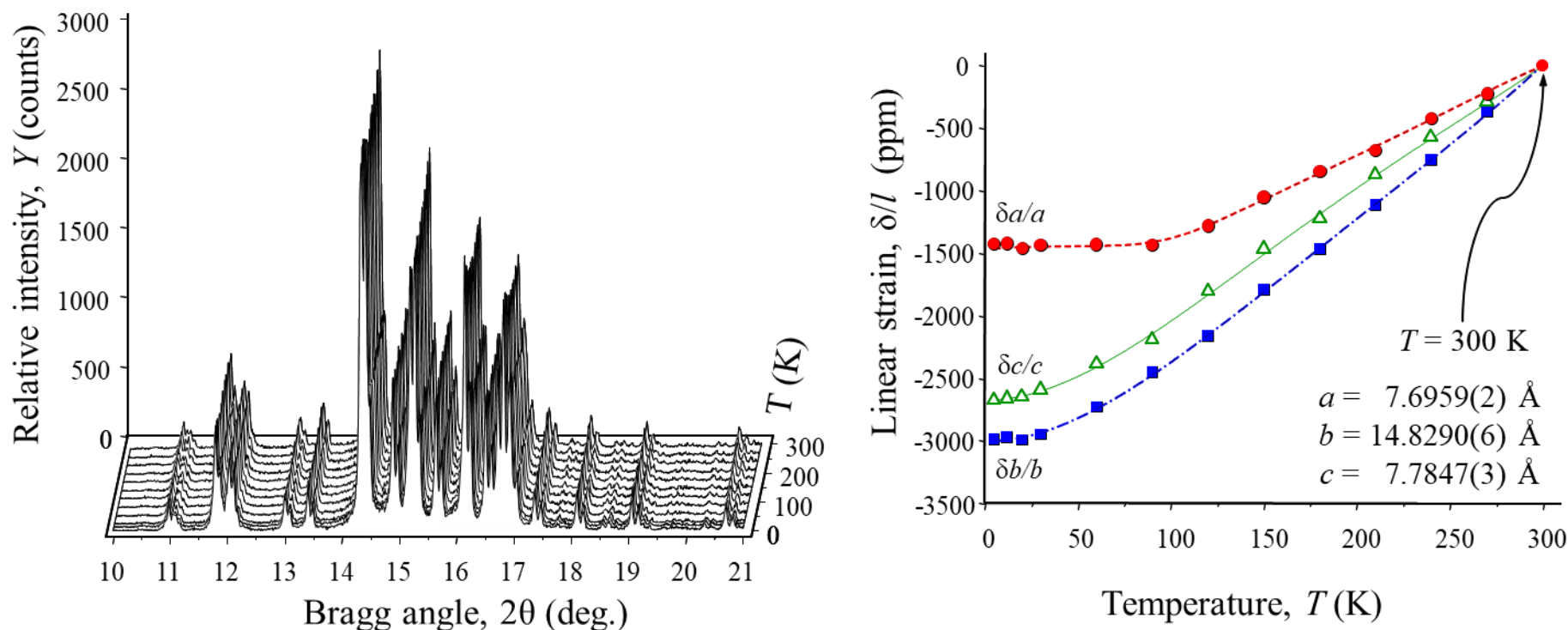
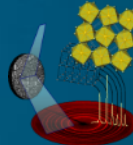


Figure 11.30. A series of powder diffraction patterns of Gd_5Ge_4 collected between 5 and 300 K (*left*) and anisotropic linear thermal expansion along the three independent crystallographic direction of the orthorhombic lattice of the compound computed from the powder diffraction data (*right*). The data were collected between 7° and 42° 2θ using equipment seen in Figure 11.32, but for clarity only small fragments are shown on the *left*. Error bars on the right are smaller than the size of the data points.

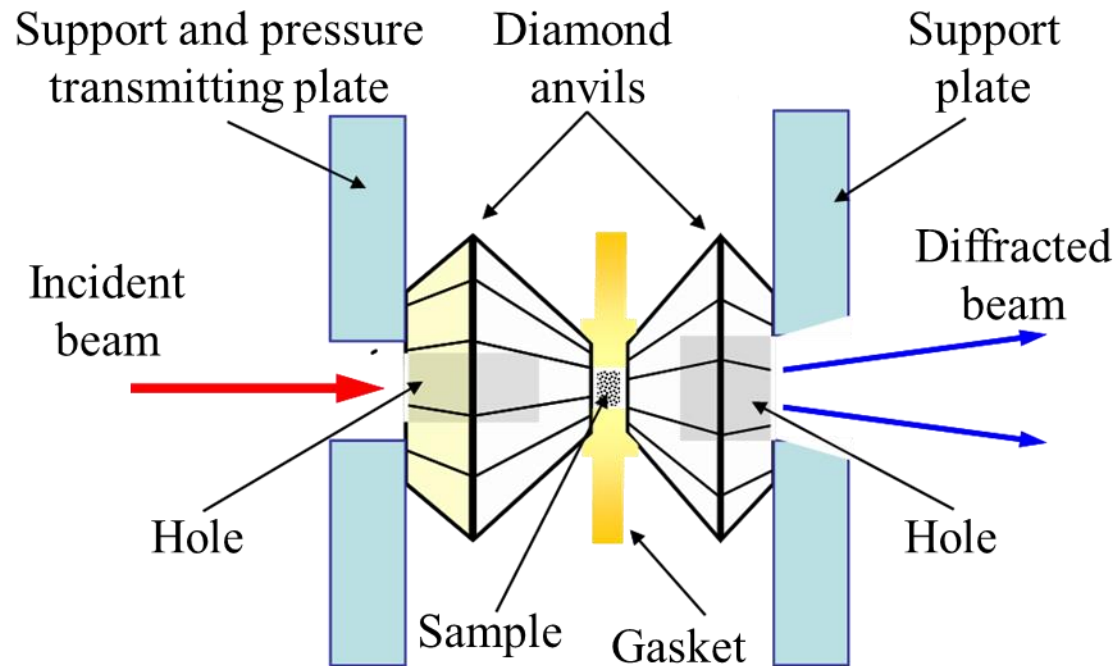
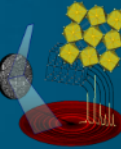


Figure 11.31. The schematic of a diamond anvil cell (*left*), commonly known as Bridgman anvils, and an actual Diacell Bragg-(S) Plus (*right*), which uses conical Boehler-Almax anvils for high pressure X-ray research. The sample is placed inside an opening in a metal gasket, typically 250 μm in diameter. The diamonds have hollow centers to reduce X-ray absorption. The anvils are pressurized either by a screw or a hydraulic press. The pressure-transmitting medium is usually alcohol, such as methanol, or fluorinert.

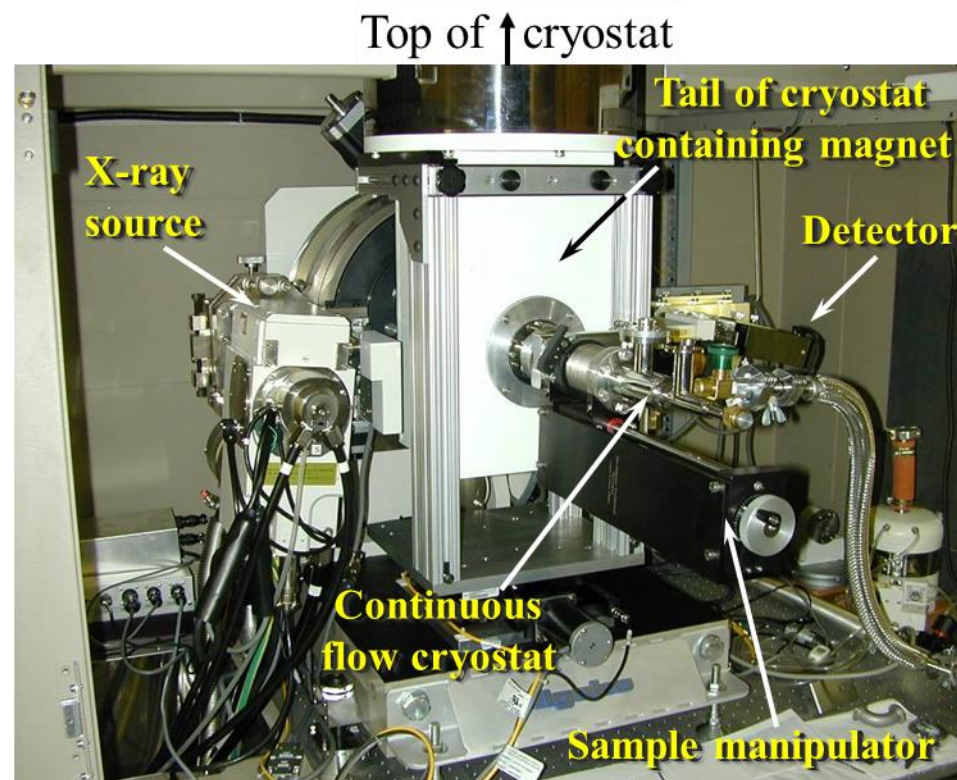
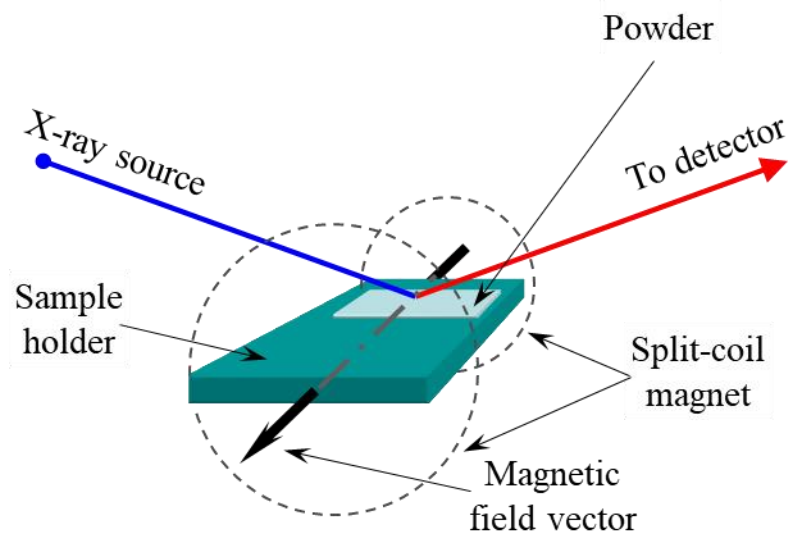
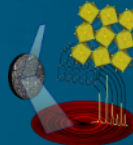


Figure 11.32. A simplified schematic of the sample holder surrounded by a split coil magnet and the X-ray beam path from the source through the split coil to the detector (*left*), and the view of the bottom part of a cryostat containing a split coil superconducting magnet mounted on a Rigaku TTRAX powder diffractometer (*right*).

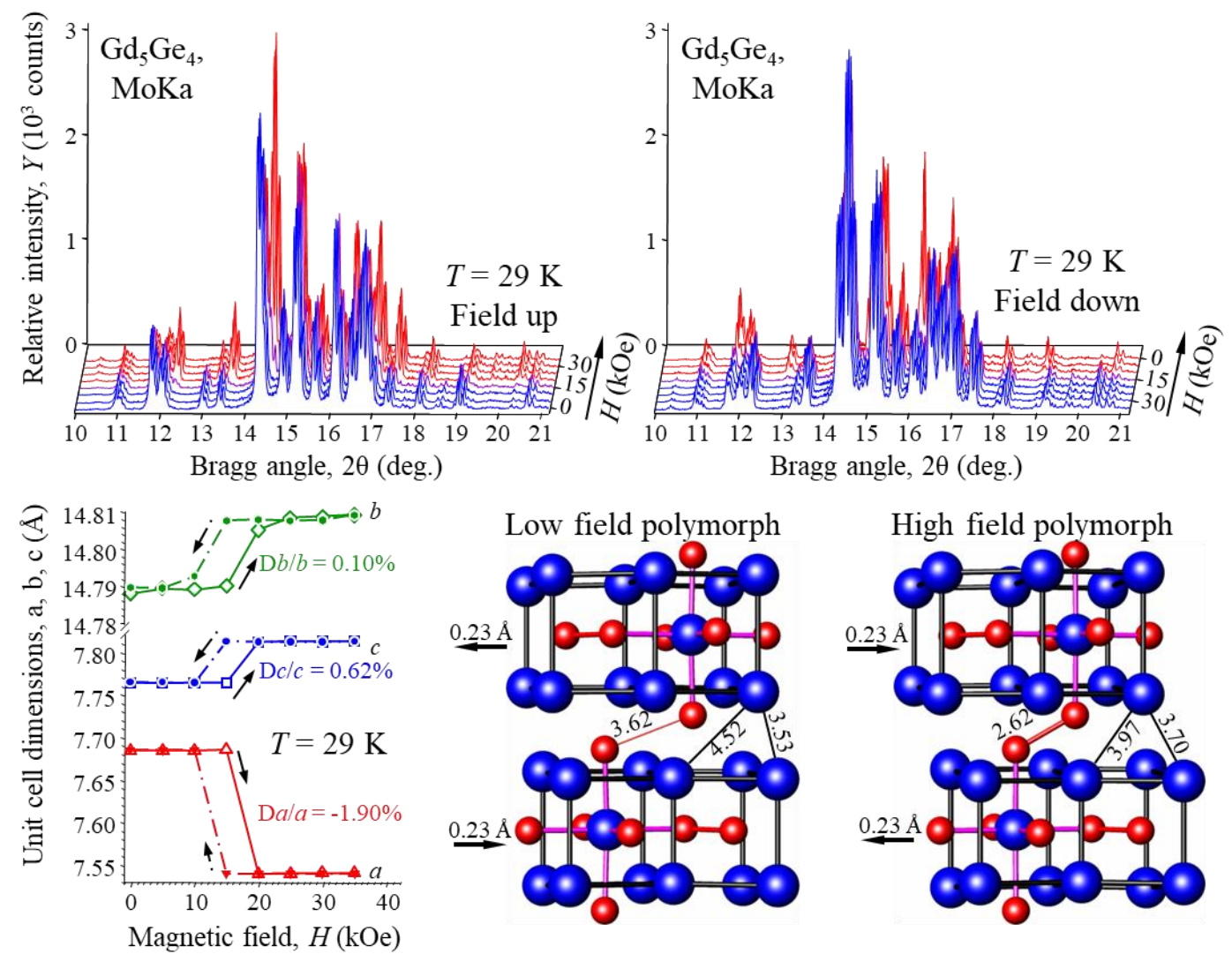
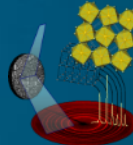


Figure 11.33. (*Top*) Two series of powder diffraction patterns of Gd_5Ge_4 collected isothermally at $T = 29$ K with the magnetic field varying between 0 and 35 kOe. (*Bottom*) Unit cell dimensions as functions of magnetic field and details of the field-induced crystallographic phase transformation in the compound.