International Tables for Crystallography (2018). Vol. H, Figure 3.1.17, p. 232.

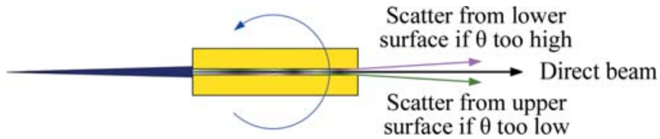

Figure 3.1.16
Diagrammatic view of the glass tunnel for determination of θ and 2θ zero angles.

If a diffractometer is being commissioned for the first time, or if major components have been replaced, it is appropriate to use fluorescent screens to achieve a rough alignment and to ensure that the incident beam does indeed cross the goniometer rotation axes and enter the detector; otherwise one may waste time looking for the beam. It is critical that these experiments are performed with the tube at operating power and that the equipment is at thermal equilibrium. Thermal effects will cause the anode to expand and contract, which will typically cause the position of the source to change. This is particularly critical when using optics to prepare the incident beam, as the performance of the optics can change markedly with movement of the source.

The objective of the first experiment using X-rays is to achieve parallelism between the line source of the tube anode, or focal line of the Johansson optic, and the receiving slit. A 5 µm platinum pinhole, which was originally manufactured as an aperture for transmission electron microscopy, is mounted in the sample position and used to image the focal line of the source onto the receiving slit (Fig. 3.1.17). This experiment is the one exception to the operating-power rule, as otherwise *Bremsstrahlung* will penetrate the platinum foil of the pinhole and produce confounding results. Success can be realized with settings of 20 kV and 10 mA; these reduced power settings are not thought to affect the angle between the tube anode and receiving slit (which is the issue addressed in this experiment). The incident slit is opened to the point at which the line source itself is imaged, not the incident slit. The Soller slits, and the post-monochromator if there is one, must also be removed to allow for the axial divergence that is needed for the success of this experiment. The pinhole images the line source onto the receiving slit; as the angle between the two decreases, progressively larger lengths of the receiving slit are illuminated during a 2θ scan. The tilt of the X-ray tube shield is varied and sequential 2θ scans are collected. As parallelism is approached, the profiles will exhibit a progressive increase in the maximum intensity value, with corresponding decreases in breadth. Conclusive results are shown in Fig. 3.1.18. It should be noted that this is a very difficult experiment to perform because the beam is essentially open and scatter is abundant. Shielding must be installed such that the detector can see only the signal that passes through the pinhole. The pinhole itself should also be shielded to minimize the area of (relatively transparent) platinum exposed to the direct beam.

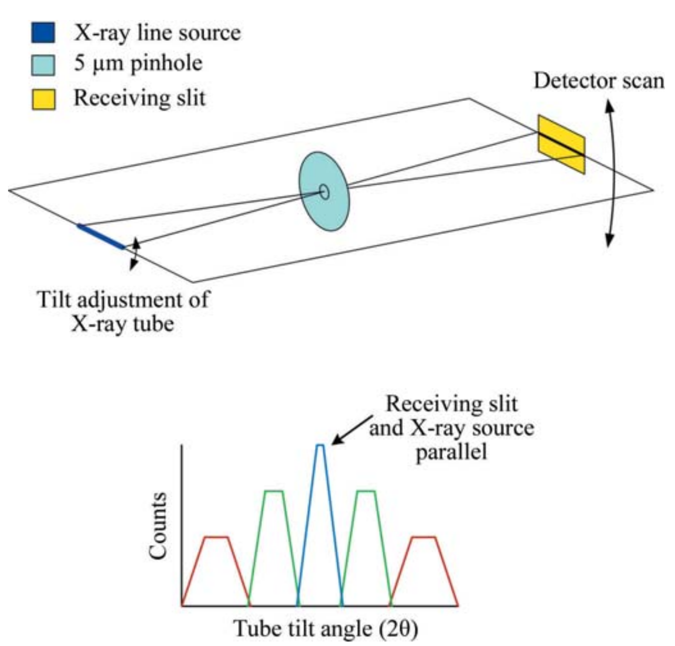

Figure 3.1.17
Design of experiments using a pinhole optic to align the X-ray source with the receiving slit.

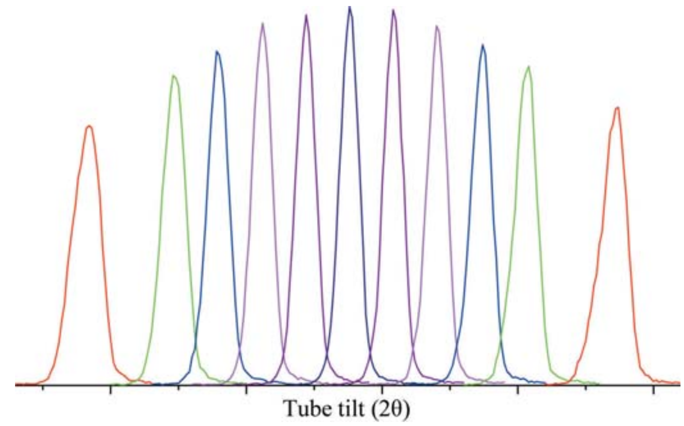

Figure 3.1.18
Successful results from the pinhole experiment showing variation in profile shape with successive adjustment of tube tilt; the central peak of highest intensity indicates the state of parallelism between the source and the receiving slit.

We now proceed to determine the θ and 2θ zero angles using the glass-tunnel optic. Initial experiments should be performed without a post-monochromator, as its presence tends to complicate finding the beam. However, it should be installed as experiments progress, as it will lead to an increase in resolution; it may alter the wavelength distribution slightly and its mass will change the torque moment on the 2θ axis. The latter two factors may alter the apparent 2θ zero by several hundredths of a degree. It is best to use a minimum slit size for the incident beam that will fully illuminate the entrance to the tunnel optic to avoid undue levels of scatter. The receiving slit should be the smallest size available, 0.05 mm in our case. The first experiment will determine a first approximation of the zero angle of θ. The tunnel optic is used, with a θ scan being performed with an open detector. Once an approximate zero angle of θ is determined, the receiving slit is installed and a 2θ scan is performed with θ at its zero point. Thus, we now have a qualitative idea of both zero angles. Then an experiment is performed as shown in Fig. 3.1.19; sequential 2θ scans are performed as θ is stepped through its zero point by very small steps (0.004° in the case of our experiment). The tunnel scatters radiation from its upper and lower surfaces when it is not parallel to the central portion of the beam, resulting in a lobe on each side of the direct beam in Fig. 3.1.19. When θ is at the desired zero angle, the direct beam is transmitted with minimum intensity in the lobes.

Once the zero positions of the θ and 2θ angles are determined, the stage is inverted and this set of experiments is repeated. It is desirable to drive the stage by 180°; however, remounting the stage in an inverted position is acceptable if the mounting structure centres the stage to within a few micrometres. Again, it is often useful to draw a diagram of the results from these two zero-angle determinations to ensure that the data are interpreted

  references