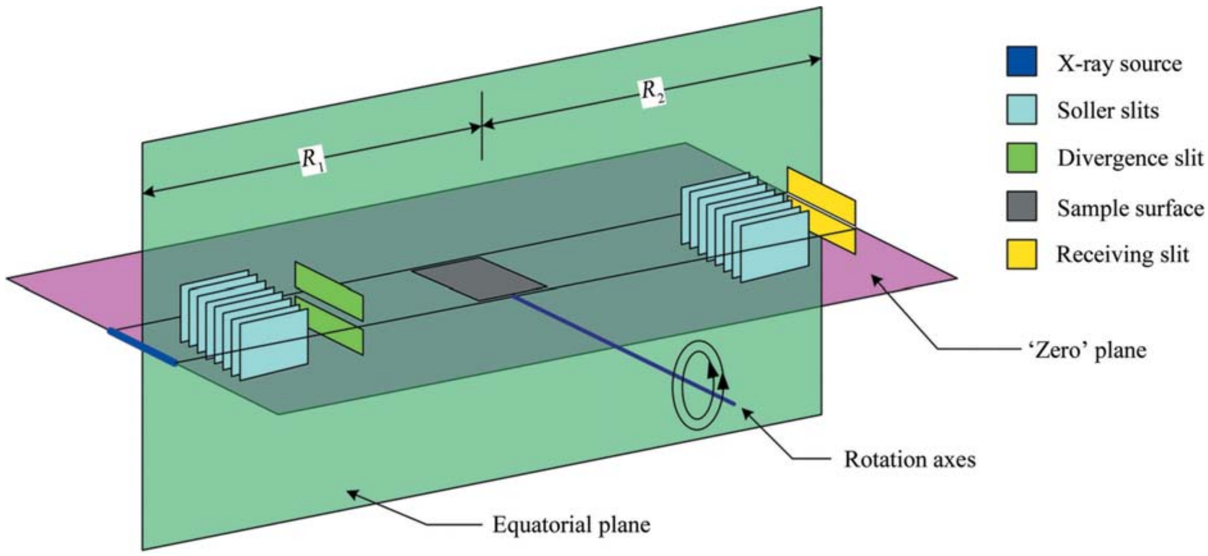

Figure 3.1.14
Diagrammatic explanation of the conditions necessary to realize a properly aligned X-ray powder diffractometer.

mathematical description of an incident spectrum. At best, a 'perfect' focusing crystal will impose an uncharacterized, though somewhat Gaussian, energy filter on the beam it diffracts. However, in certain optics the required bend radius of Johansson geometry is realized by clamping the crystal onto a curved form. The clamping restraint exists only at the edges of the optic, not in the central, active area where it is illuminated by the X-ray beam. The crystal itself however, can minimize internal stress by remaining flat; in this case an anticlastic curvature of the optic results. A 'saddle' distortion across the surface of the diffracting region of the crystal results in a complex asymmetric $K\alpha_1$ spectrum that defies accurate mathematical description. Johansson optics, however, can be bent by cementing the crystals into a preform, yielding an optic of superior perfection in curvature. Fig. 3.1.13 shows data collected from such an optic using an Si single crystal, 333 reflection, as an analyser. Parallel-beam conditions were approximated in this experiment with the use of very fine 0.05 mm incident and receiving slits. The observed symmetric emission profile of Fig. 3.1.13(*a*) can be modelled with a combination of several Gaussians. However, a Johansson optic will scatter 1–2% of high-energy radiation to a higher 2θ angle than the $K\alpha_1$ focal line of the optic. This unwanted scatter is dominated by, but not exclusive to, the $K\alpha_2$ spectrum. Louër (1992) indicated that it can be largely blocked with a knife edge aligned to just 'contact' the high-angle side of the optic's focal line. Alternatively, the NIST method is to use a slit aligned to straddle the focal line. Proper alignment of this anti-scatter slit is critical to achieving a good level of performance with the absence of '$K\alpha_2$' scatter, as illustrated in Fig. 3.1.13(*b*). As will be demonstrated, with use of any Johansson optic the elimination of the $K\alpha_2$ line is of substantial benefit in fitting the observed peaks with analytical profile-shape functions.

3.1.3. Instrument alignment

Modern instruments embody the drive towards interchangeable pre-aligned or self-aligning optics, which, in turn, has led to several approaches to obtaining proper alignment with minimum effort on the part of the user. We will not review these approaches, but instead we decribe here the methods used at NIST, which could be used to check the alignment of newer equipment. With the use of calibration methods that simply characterize the performance (which includes the errors) of the machine in an empirical manner and apply corrections, the quality of the instrument alignment may be surprisingly uncritical for a number of basic applications such as lattice-parameter refinement. However, with the use of the more advanced methods for characterization of the IPF that are based on the use of model functions, the proper alignment of the machine is critical. The models invariably include refineable parameter(s) that characterize the extent to which the given aberration affects the data; the correction is applied, and the results are therefore 'correct'. However, if the instrument is not aligned properly, the analysis attempts to model the errors due to misalignment as if they were an expected aberration. The corrections applied are therefore incorrect in degree and nature and an erroneous result is obtained.

The conditions for proper alignment of a Bragg–Brentano diffractometer (see Fig. 3.1.14) are:

(1) the goniometer radius, defined by the source-to-rotation-axes distance, R_1, equals that defined by the rotation-axes-to-receiving-slit distance, R_2 (to ± 0.25 mm);
(2) the X-ray line source, sample and receiving slit are centred in the equatorial plane of diffraction (to ± 0.25 mm);
(3) the goniometer rotation axes are co-axial and parallel (to ± 5 µm and <10 arc seconds);
(4) the X-ray line source, specimen surface, detector slit and goniometer rotation axes are co-planar, in the 'zero' plane, at the zero angle of θ and 2θ (to ± 5 µm and $\pm 0.001^\circ$); and
(5) the incident beam is centred on both the equatorial and 'zero' planes (to $\pm 0.05^\circ$).

The first three conditions are established with the X-rays off, while conditions (4) and (5) are achieved with the beam present, as it is actively used in the alignment procedure. Neither incident- nor diffracted-beam monochromators are considered; they are simply added on to the Bragg–Brentano arrangement and have no effect on the issues outlined here. Also, in order to execute this procedure, a sample stage that can be rotated by 180° in θ is required. However, this does not need to be the sample stage used for data collection. Before any concerted effort to achieve proper alignment, it is advisable to check the mechanical integrity of the equipment. Firmly but gently grasp a given component of the diffractometer, such as the tube shield, receiving-slit assembly or sample stage, and try to move it in a manner inconsistent with