International Tables for Crystallography (2018). Vol. H, Figure 3.1.15, p. 231.

its proper mounting and function. The number of defects, loose bolts *etc.*, that can be found this way, even with quite familiar equipment, can be surprising.

Let us briefly review the development of diffraction equipment and the subsequent impact on alignment procedures. The goniometer assemblies used for powder diffractometers utilize a worm/ring gear to achieve rotation of the θ and 2θ axes while allowing for the ~0.002° resolution with the use of a stepper or servo motor actuating the worm gear. 'Home' switches, with a coarse one on the ring gear and a fine one on the worm shaft, allow the software to locate the reference angle(s) of the goniometer assembly to a repeatability of the stepper motor resolution. With the first generation of these automated goniometers, the zero angles were fixed relative to the home positions. With such a design the invariant reference was the receiving slit, and the operator adjusted the height of the tube shield and the angle of the θ stage to realize alignment condition (4). Second-generation machines offered the ability to set the zero angles relative to the home positions (or those of optical encoders) *via* software, in which case the exact angular position of either the X-ray tube focal line or of the receiving slit in θ–2θ space is arbitrary. The operator simply determines the positions where the θ and 2θ angles are zero, and then sets them there. There is no technical reason why the older designs cannot be aligned to the accuracy of newer ones. In practice, however, with older equipment the patience of the operator tends to become exhausted, and a less accurate alignment is accepted. An important consideration in evaluating modern equipment is that it is often the incident optic, not the X-ray source (focal line), that is used as the reference. Which situation is the case can be readily discerned with an inspection of the hardware: if the incident optic is anchored to the instrument chassis, then it is the reference. If it is attached to the tube shield, however, then the source establishes the reference. The NIST equipment has the latter design.

Condition (1) is that the goniometer radius, defined by the source-to-rotation-axis distance, R_1, equals that defined by the rotation-axis-to-receiving-slit distance, R_2. This condition is required for proper focusing and is generally realized with the use of rulers to achieve a maximum permissible error of $R \pm$ 0.25 mm for a nominal $R = 200$ mm diffractometer. Condition (2) concerns the centring of the components in the plane of diffraction or equatorial plane. This condition is assured with the use of straightedges and rulers and, again for a line focus with an 8 to 12 mm source length, the maximum permissible error for deviations along the equatorial plane is ±0.25 mm. One can also consider the takeoff angle at this time; this is the angle between the surface of the X-ray tube anode and the equatorial centre line of the diffractometer incident-beam path. As this angle decreases the resolution is improved at the expense of signal intensity, and *vice versa*, as a consequence of the variation in the size of the source that the specimen 'sees'. However, with modern fine-focus tubes, this is not a major effect. Qualitative experiments at NIST indicate that the exact angle is not critical; a 6° takeoff angle is reasonable.

The third issue concerns the concentricity of the θ and 2θ rotation axes of the goniometer assembly; this is a matter of underappreciated concern. It is not, however, one over which the end user has a great deal of control. Measurement of axes centricity requires the construction of some fairly complex and stiff structures capable of measuring displacements of the order of 1 to 2 µm and rotations of seconds of arc. The objective is to measure both the offset between the two axes and the angle between them. Concentricity errors affect XRPD data in a manner analogous to that of sample displacement; hence a 5 µm concentricity error is of concern. Worse yet is the possibility that some degree of precession occurs between the two axes with the operation of the goniometer. In this case, the performance of the machine will challenge description using established models.

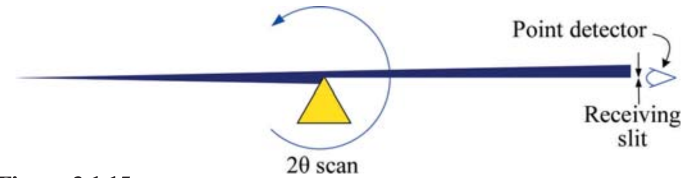

Figure 3.1.15
Diagrammatic view illustrating the use of a knife edge to determine the 2θ zero angle.

Subsequent experiments are performed with the X-rays present in order to achieve conditions (4) and (5). The criteria for proper alignment are universal, but there is a range of experimental approaches by which they can be realized. The specific approach may well be based on the age and make of the equipment as well as the inclinations of the operator. The essence of the experimental design remains constant, however: the operator uses optics mounted in the sample position that will either pass or block the X-ray beam in such a way as to tell the operator if and when the desired alignment condition has been realized. One approach is to use a knife edge mounted as shown in Fig. 3.1.15; a 2θ scan is performed using a point detector with a narrow receiving slit. When the intensity reaches 50% of the maximum, the X-ray source (focal line), the rotation axes of the goniometer and the 2θ (zero) angle are coplanar. However, the problematic presumption here is that the sample stage is aligned so exactly that the rotation axes of the goniometer assembly bisect the specimen surface, and therefore the knife edge, to within a few micrometres. This is equivalent to the z height being zero. The verification of this level of accuracy in stage alignment would be exceedingly difficult *via* direct measurements on the sample stage itself. While many would be inclined to trust the instrument manufacturer to have correctly aligned the stage, at NIST we use an alternative approach.

A straightforward means of addressing this problem is to use a stage that can be inverted, and perform the 2θ zero angle experiment in both orientations. 2θ scans of a knife edge in the normal and inverted positions can be compared to determine the true 2θ zero angle, independent of any z-height issue associated with the stage. It is often useful to draw a diagram of the results in order to avoid confusion; half the difference between the two measured zero angles yields the true one. With this information, the final alignment involves adjusting the specimen z height in the desired stage, which need not be invertible, until what is known to be the true 2θ zero angle is realized. The knife edge can also be used to centre the beam on the rotation axes, as per condition (5). Determination of the θ stage zero angle can be performed using a precision ground flat. An alternative optic to the knife edge is a rectangular 'tunnel' through which the X-ray beam passes. The entrance window of said tunnel may measure 20 to 40 µm in height and 10 mm in width, while the tunnel itself is 5 cm long. It is mounted in the beam path as illustrated in Fig. 3.1.16, with the 20 to 40 µm dimension defining the width of the beam and the 10 mm dimension describing the beam's length. Optics like this can be made of metal but are often made of glass. This optic will pass an X-ray beam only if it is parallel to the direction of the tunnel and can be used to determine both θ and 2θ zero angles. These are the optics used at NIST, *via* an experimental approach that will be discussed below.

  references