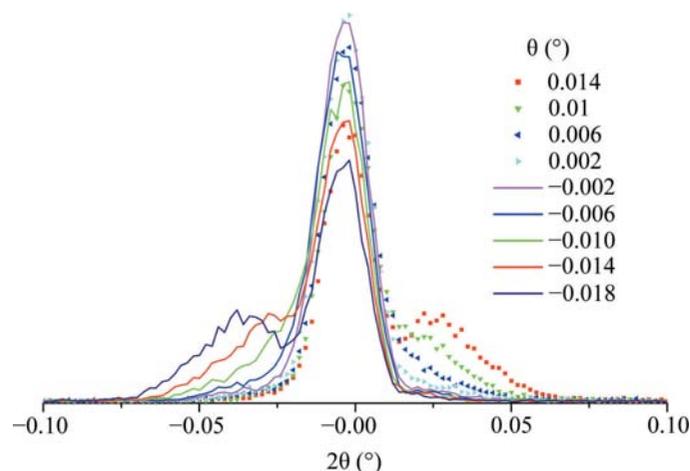
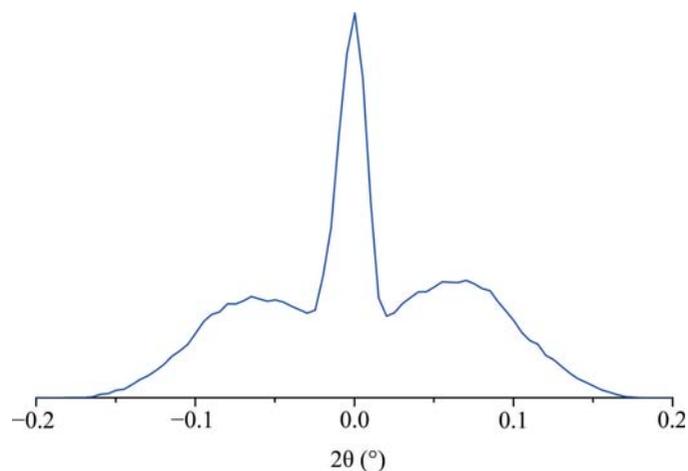


## 3.1. OPTICS AND ALIGNMENT OF THE LABORATORY DIFFRACTOMETER



**Figure 3.1.19**  
Results from  $2\theta$  scans at successive  $\theta$  angles using the glass tunnel to determine the  $\theta$  and  $2\theta$  zero angles.



**Figure 3.1.21**  
Final results from a  $\theta$ - $2\theta$  scan using the glass tunnel, indicating the correct determination of  $\theta$  and  $2\theta$  zero angles.

correctly, as shown in Fig. 3.1.20. In this example, the sample height is displaced in the positive  $z$  direction, otherwise the positions of orientation 1 and  $180^\circ$  from orientation 1 would be reversed. The operator should verify that fully self-consistent results are obtained with respect to the four zero angles shown in Fig. 3.1.20. Because the beam is divergent, the difference between the two  $\theta$  zero angles will not be precisely  $180^\circ$ , as shown in Fig. 3.1.20. Again, half the difference between the two measured  $2\theta$  zero angles yields the true one, with respect only to the locations of the X-ray source and the goniometer rotation axes. Using the data of Fig. 3.1.20 and the goniometer radius, the  $z$ -height error on the stage in question could be computed and an adjustment made; this should be followed by repeating the two zero-angle measurements and checking for self-consistency to provide additional confidence in the alignment.

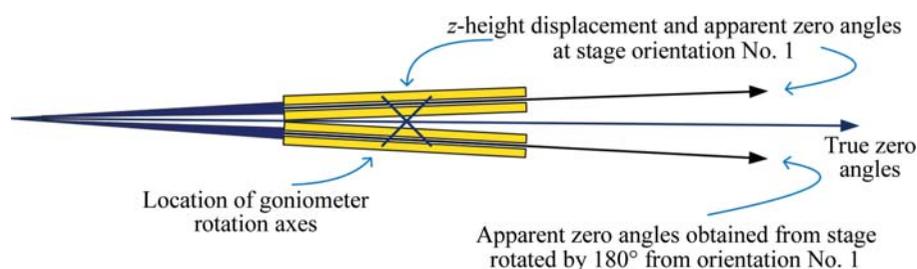
The final task is to mount the stage to be used in subsequent data collection and adjust its sample height until the known true  $2\theta$  zero angle is obtained. The final experiment is a  $\theta$ - $2\theta$  scan of the tunnel optic to yield data of the kind shown in Fig. 3.1.21. The symmetry of the lobes on each side of the peak from the direct beam is indicative of the correct  $\theta$  zero angle setting. This final high-resolution experiment is an excellent indicator of the state of the alignment of the instrument. These experiments, when used in conjunction with profile fitting, can yield measurements of the zero angles with an uncertainty for  $\theta$  and  $2\theta$  of  $\pm 0.001^\circ$ . Given the high certainty with which the zero angles are determined, they would then not be refined in subsequent data analyses. The alignment of the incident-beam slit, issue (5), is accomplished with a scan of the direct beam. If the machine is

equipped with a variable-divergence incident-beam slit, it is important to evaluate it at several settings because changes in the centre line of the beam may occur as the divergence angle is altered. Use of an excessively narrow receiving slit should be avoided for scans of the direct beam, since the thickness of the metal blades used for the slit itself may be larger than the width of the slit, leading to a directional selectivity as the scan is performed.

The alignment presented here was carried out using a scintillation detector; however, much of it could be performed using a PSD in 'picture-taking' mode. In any case, the count rates have to be monitored to ensure that they are within the linear range of the detector (5000 to 10 000 counts per second), otherwise anomalous results are obtained. Attenuating foils that are flat and in good condition can be used to reduce the intensity. It should also be stressed that if the observations made during the experiments do not meet expectations, something is wrong and the desired outcome, *i.e.* the correct alignment, will not be realized. Drawing a diagram of the X-ray beam path can be very useful for discovering the cause of apparently unexplainable observations. Also, throughout these experiments it is appropriate for the operator to try various additional settings to ensure that the machine is operating as expected. Anomalous observations can almost always be explained in a quantitative manner with appropriate investigation. Patience is required.

In the past, achieving acceptable performance with a Johansson optic was considered so problematic that they were under-used, despite the improvements in the data quality they provided. Modern instrumentation can provide their advantages

with dramatically reduced effort. The NIST Johansson IBM, however, was derived from an older design that was originally supplied with a Siemens D500, circa 1987. It uses a Huber 611 monochromator housing that provides 5 degrees of freedom in the positioning of the optic: the  $a$  distance, the takeoff angle, crystal  $2\theta$ , tilt and azimuth. For aforementioned reasons, we installed a modern Johansson optic manufactured by Crismatec (now part of Saint Gobain). There are two stages to the procedure for aligning the machine equipped with the IBM: first, the crystal optic itself is aligned



**Figure 3.1.20**  
Diagram of hypothetical results from two zero-angle measurements (Fig. 3.1.19) with the sample stage in the normal and inverted positions to determine the true  $2\theta$  zero angle of the goniometer assembly in the absence of a  $z$ -height error from sample-stage misalignment.