Table 2.10.1
Intensity (counts) and mean deviation in intensity of the main quartz 101 reflection with a stationary sample of -325 mesh quartz powder

Data from Alexander *et al.* (1948) and Klug & Alexander (1954).

	Crystallite size			
Data set	15–50 μm	5–50 μm	5–15 μm	<5 μm
1	7612	8688	10841	11055
2	8373	9040	11336	11040
3	8255	10232	11046	11386
4	9333	9333	11597	11212
5	4823	8530	11541	11460
6	11123	8617	11336	11260
7	11051	11598	11686	11241
8	5773	7818	11288	11428
9	8527	8021	11126	11406
10	10255	10190	10878	11444
Mean % deviation	18.2	10.1	2.1	1.2

Table 2.10.2
Theoretical behaviour of different crystallite sizes of quartz in a volume of 20 mm^3

Data from Smith (2001).

Crystallite diameter (μm)	40	10	1
Crystallites per 20 mm^3	5.97×10^5	3.82×10^7	3.82×10^{10}
No. of diffracting crystallites	12	760	38 000

Debye rings in a capillary (Klug & Alexander, 1954), while the pattern would be granular in reflection. Specimen rotation has also been long employed in reflection geometry (de Wolff, 1958; de Wolff *et al.*, 1959), and a sample spinner is now a standard attachment for commercial diffractometers. When properly applied, the use of a spinner can reduce the standard deviation of the integrated intensity by a factor of approximately 4–5 (~7–8 for peak intensities) (de Wolff *et al.*, 1959), corresponding to a reduction in the effective crystallite size by a factor of 3 (Zevin & Kimmel, 1995). However, depending on the sample, as seen in Table 2.10.2 this can be insufficient on its own as it rotates the specimen only in a single plane. Where a spinner is used in conjunction with a point counter, it is important that the spinner must complete at least one rotation during each step to maximize its effectiveness. In order to further improve the particle statistics it is possible to construct spinners that tilt back and forth along an axis normal to the beam (similar to the capillary concept) in addition to the normal axis of rotation. This is effective in improving particle statistics but adversely affects the para-focusing condition in Bragg–Brentano geometry. It is important to note that specimen rotation improves grain-sampling statistics, but does nothing to alter preferred orientation.

The term 'micronized' is one that is frequently seen in papers on quantitative phase analysis. Potentially any kind of mill could be used to reduce the crystallites down to the desirable μm size range (such as shown in Fig. 2.10.14*a* and *b*). However, most mills use high-energy percussion-like impacts between the grinding media and the sample, which tend to damage the crystal structure in softer materials and induce microstrain into the material. In extreme cases the sample can become completely amorphous. There is also the potential problem of modifying the polymorph with samples susceptible to such changes. The mill produced by McCrone (http://www.mccrone.com) was designed specifically for the preparation of X-ray diffraction and X-ray fluorescence samples, and the shearing milling mechanism minimizes damage *versus* conventional impact milling. It is necessary to use wet milling to produce the best results, so it is up to the analyst to

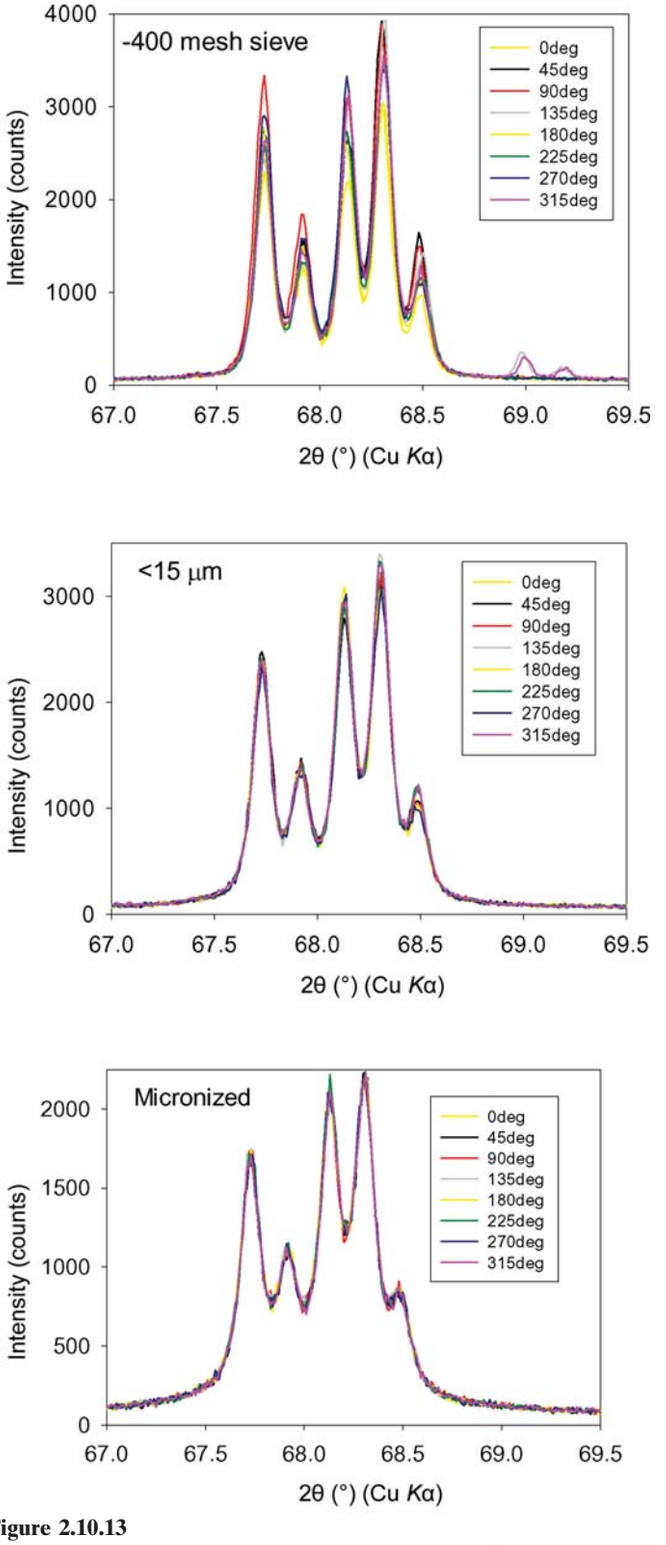

Figure 2.10.13
φ scans of the five fingers of quartz for (*a*) <38 μm, (*b*) <15 μm and (*c*) micronized samples.

choose the best media compatible with both the sample and the polymer micronizing vials. Commonly used are ethanol, isopropyl alcohol, *n*-hexane and water; it is not advisable to use acetone, as this solvent dissolves the polymer jars supplied with this mill. A limitation of most forms of milling is the requirement for a relatively large amount of sample. In the McCrone mill a volume of >1 ml is usually required, although desperate scientists have been known to dilute the specimen with amorphous material, such as silica gel. The analyst should also be aware of the possible contamination of samples by degrading and eroding grinding