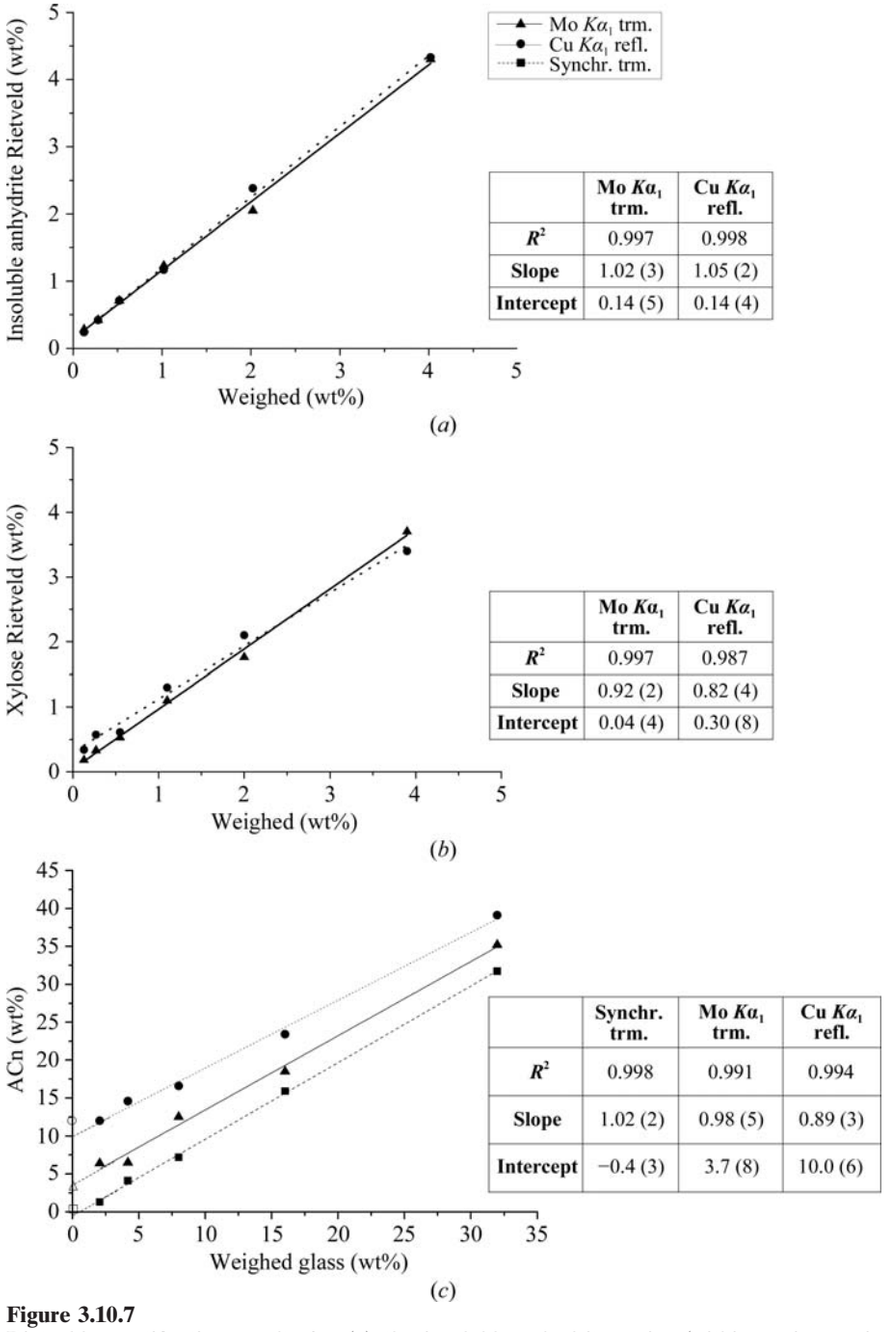
has to be exercised when filling capillaries in order to minimize this problem.

3.10.7. Increasing inorganic crystalline phase content series

Table 3.10.2 reports the RQPA results for six inorganic mixtures with increasing amounts of i-A measured with Mo $K\alpha_1$ (transmission) and Cu $K\alpha_1$ (reflection). The Rietveld plots of the mixture with 4 wt% i-A are shown in Fig. 3.10.6. For most of the samples, the AKLD values (see Table 3.10.2) for Mo $K\alpha_1$ radiation are slightly smaller than the corresponding values obtained for Cu $K\alpha_1$ radiation. For this reason, we can conclude that the Mo $K\alpha_1$ analyses are slightly better than those derived using Cu $K\alpha_1$ radiation.

On the other hand, calcite and gypsum presented preferred orientations, with the axes being [104] and [010], respectively. This effect was modelled using the March–Dollase algorithm. Preferred orientation makes the $0l0$ reflections for gypsum have higher intensities in the Cu $K\alpha_1$ patterns, and smaller intensities in the Mo $K\alpha_1$ patterns, than those calculated from the crystal structure (see insets in Fig. 3.10.6). As a consequence, the refined values for flat samples in reflection and transmission geometries were smaller and larger than 1.0, respectively (Cuesta *et al.*, 2015). Although preferred orientation is present in all patterns, the Cu $K\alpha_1$ patterns were recorded in reflection geometry (flat samples), while the Mo $K\alpha_1$ measurements were collected in transmission (also flat samples). This results in opposite diffraction intensity changes and points towards another (possible) fruitful use: joint refinement of these two types of patterns to counterbalance the effects of preferred orientation in RQPA.



	Mo $K\alpha_1$ trm.	Cu $K\alpha_1$ refl.
R^2	0.997	0.998
Slope	1.02 (3)	1.05 (2)
Intercept	0.14 (5)	0.14 (4)

	Mo $K\alpha_1$ trm.	Cu $K\alpha_1$ refl.
R^2	0.997	0.987
Slope	0.92 (2)	0.82 (4)
Intercept	0.04 (4)	0.30 (8)

	Synchr. trm.	Mo $K\alpha_1$ trm.	Cu $K\alpha_1$ refl.
R^2	0.998	0.991	0.994
Slope	1.02 (2)	0.98 (5)	0.89 (3)
Intercept	−0.4 (3)	3.7 (8)	10.0 (6)

Figure 3.10.7
Rietveld quantification results for (*a*) the insoluble anhydrite series (within an inorganic crystalline matrix), (*b*) the xylose series (within an organic crystalline matrix) and (*c*) the ground-glass series (within an inorganic crystalline matrix) as a function of the weighed amount of each phase. Open symbols represent the derived amorphous contents in the mixtures without any added glass. The results of the least-squares fits are also shown.

Fig. 3.10.7(*a*) shows the quantified i-A contents (wt%), as determined by the Rietveld methodology, as a function of the weighed i-A amount. The two R^2 values for the fits are very close to 1.00, and the intercept values are very close to zero, showing the appropriateness of the Rietveld methodology for quantifying crystalline materials. Furthermore, the slopes of the calibration curves are also 1.00 in both cases. Consequently, this study allows it to be concluded that RQPA for crystalline inorganic phases using powder-diffraction patterns collected using Mo $K\alpha_1$ radiation yields results that are as accurate as those obtained from the well established method using Cu $K\alpha_1$.

3.10.8. Increasing crystalline organic phase content series

Table 3.10.3 shows RQPA results for six mixtures prepared with G, F, L and an increasing amount of X measured with Mo $K\alpha_1$ (transmission) and Cu $K\alpha_1$ (reflection). In general, the values obtained using both radiations are quite similar to the weighed values. The AKLD values and the KLD values for the xylose phase are also reported in Table 3.10.3. The AKLD values from Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations are relatively similar. The main problem for RQPA of organic mixtures measured in reflection geometry is related to the low X-ray absorption of the samples and the transparency effects that lead to poor peak shapes and even some split peaks in the powder patterns, as discussed previously (León-Reina *et al.*, 2016).

Fig. 3.10.7(*b*) shows the quantified xylose contents (wt%) as determined by the Rietveld methodology as a function of the weighed amount of xylose added to the mixtures. The results were plotted to obtain the calibration lines with increasing content of the analyte. Both plots gave R^2 values close to 1.0. However, the slope values were 0.92 and 0.82 for Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations, respec-