Table 3.10.3
RQPA for the crystalline organic mixtures measured with Cu $K\alpha_1$ and Mo $K\alpha_1$ radiations

Weighed amounts (wt%) are also shown for the sake of comparison. Absolute values of the Kullback–Liebler distance (AKLD) for each mixture and the KLD value for xylose are also included. Trm, transmission; rfl, reflection.

	GFL_0.0X			GFL_0.25X			GFL_0.50X		
Phases	wt%	Mo trm	Cu rfl	wt%	Mo trm	Cu rfl	wt%	Mo trm	Cu rfl
G	33.4	33.8 (1)	33.5 (3)	33.3	33.6 (1)	33.1 (2)	33.2	32.3 (2)	33.5 (2)
F	33.5	31.7 (1)	32.7 (3)	33.4	32.3 (1)	34.3 (2)	33.3	32.1 (2)	33.4 (2)
L	33.1	34.5 (1)	33.7 (3)	33.0	33.7 (1)	32.0 (2)	33.0	35.0 (3)	32.5 (2)
X	—	—		0.27	0.33 (4)	0.57 (9)	0.55	0.53 (8)	0.61 (9)
AKLD sum		0.0362	0.0150		0.0216	0.0231		0.0410	0.0096
(X) KLD		—	—		−0.001	−0.002		0.000	−0.001

	GFL_1.0X			GFL_2.0X			GFL_4.0X		
Phases	wt%	Mo trm	Cu rfl	wt%	Mo trm	Cu rfl	wt%	Mo trm	Cu rfl
G	33.0	34.7 (1)	33.6 (2)	32.7	32.2 (1)	31.5 (2)	32.0	32.8 (1)	33.6 (2)
F	33.1	32.6 (1)	33.7 (2)	32.8	31.7 (1)	34.4 (2)	32.2	30.7 (1)	32.5 (2)
L	32.8	31.6 (2)	31.4 (2)	32.5	34.3 (1)	32.0 (2)	31.8	32.9 (1)	30.5 (2)
X	1.1	1.10 (5)	1.3 (1)	2.0	1.76 (5)	2.1 (1)	3.9	3.70 (5)	3.4 (2)
AKLD sum		0.0338	0.0280		0.0363	0.0339		0.0361	0.0372
(X) KLD		0.000	−0.002		0.003	−0.001		0.002	0.005

Table 3.10.4
Rietveld quantitative phase analyses of the CQZ_*x*Gl mixture, where quartz (Q) is the internal standard, to derive amorphous content (am), obtained from SXRPD, Mo $K\alpha_1$ and Cu $K\alpha_1$ patterns

Absolute values of the Kullback–Liebler distance (AKLD) for each mixture and the KLD value for the amorphous content are also included. Trm, transmission; rfl, reflection.

	Weighed			Synchrotron trm				
Mixture	C wt%	Z wt%	Gl wt%	C wt%	Z wt%	Am wt%	AKLD sum	Am KLD
CZQ_0Gl	50.01	49.99	0.00	49.9 (1)	49.6 (1)	0.4 (1)	0.0050	—
CZQ_2Gl	48.98	48.96	2.05	49.7 (1)	49.0 (1)	1.3 (1)	0.0169	0.009
CZQ_4Gl	47.93	47.91	4.17	47.9 (1)	47.6 (1)	4.5 (1)	0.0066	−0.003
CZQ_8Gl	46.00	46.00	7.99	46.6 (1)	45.9 (1)	7.5 (1)	0.0120	0.005
CZQ_16Gl	41.99	41.99	16.01	42.0 (1)	41.6 (1)	16.4 (1)	0.0079	−0.004
CZQ_32Gl	34.00	34.00	31.99	34.0(1)	33.7 (1)	32.3 (1)	0.0061	−0.003

	Mo $K\alpha_1$ trm					Cu $K\alpha_1$ rfl				
Mixture	C wt%	Z wt%	Am wt%	AKLD sum	Am KLD	C wt%	Z wt%	Am wt%	AKLD sum	Am KLD
CZQ_0Gl	47.5 (1)	49.0 (1)	3.5 (1)	0.0358	—	47.2 (1)	40.8 (1)	12.0 (1)	0.1305	—
CZQ_2Gl	45.9 (1)	47.7 (1)	6.4 (1)	0.0679	−0.023	47.4 (1)	40.6 (1)	12.0 (1)	0.1440	−0.036
CZQ_4Gl	46.5 (1)	47.0 (1)	6.5 (1)	0.0422	−0.019	45.8 (1)	39.7 (1)	14.6 (1)	0.1641	−0.052
CZQ_8Gl	42.6 (1)	44.8 (1)	12.5 (1)	0.0832	−0.036	45.3 (1)	38.1 (1)	16.6 (1)	0.1522	−0.058
CZQ_16Gl	39.9 (1)	41.7 (1)	18.5 (1)	0.0475	−0.023	40.9 (1)	35.8 (1)	23.4 (1)	0.1388	−0.061
CZQ_32Gl	31.7 (1)	33.1 (1)	35.2 (1)	0.0635	−0.031	32.2 (1)	28.7 (1)	39.1 (1)	0.1403	−0.064

tively. Slope values close to 1.0 mirror accurate analyses. Furthermore, the *y*-intercept values were 0.04 and 0.30 for Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations, respectively. A *y*-intercept value close to 0.0 mirrors accurate analyses. Hence, it can be concluded that slightly more accurate analyses are obtained for Mo $K\alpha_1$ powder diffraction in transmission when compared with Cu $K\alpha_1$ powder diffraction in reflection for organic crystalline samples.

3.10.9. Increasing amorphous content series within an inorganic crystalline phase matrix

Fig. 3.10.8 shows Mo $K\alpha_1$ (transmission), Cu $K\alpha_1$ (reflection) and SXRPD (transmission) raw patterns for the mixtures with increasing amounts of glass. It is important to highlight that the increase in the background due to the glass is very modest even for ∼32 wt% of glass. Table 3.10.4 shows the RQPA of these mixtures, prepared with C, Z and an increasing amount of Gl, for the three radiations. The glass-free sample may contain amorphous material from the employed phases. Hence, we used the SXRPD data to calculate a correction factor for quartz to yield zero amorphous content for the glass-free sample (León-Reina *et al.*, 2016).

The linear fit to the amorphous content values obtained using SXRPD was very good, R^2 = 0.998, with the slope being 1.00 within the errors (see Fig. 3.10.7*c*). This plot also shows the quantified amorphous contents, in weight percentage, as a function of the amount of added ground glass, measured with Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations. Open symbols indicate the derived amorphous contents obtained with the internal-standard method in the mixture without any added glass, CZQ_0Gl. Both R^2 values are quite close to 1.00, showing the consistency of the internal-standard methodology. However, the slope values were 0.98 and 0.89 for Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations, respectively. Furthermore, the *y*-intercept values were 3.7 and 10.0 for Mo $K\alpha_1$ and Cu $K\alpha_1$ radiations, respectively. Again, slope values close to 1.0 and *y* intercepts close to 0.0 mirror accurate analyses. It must also