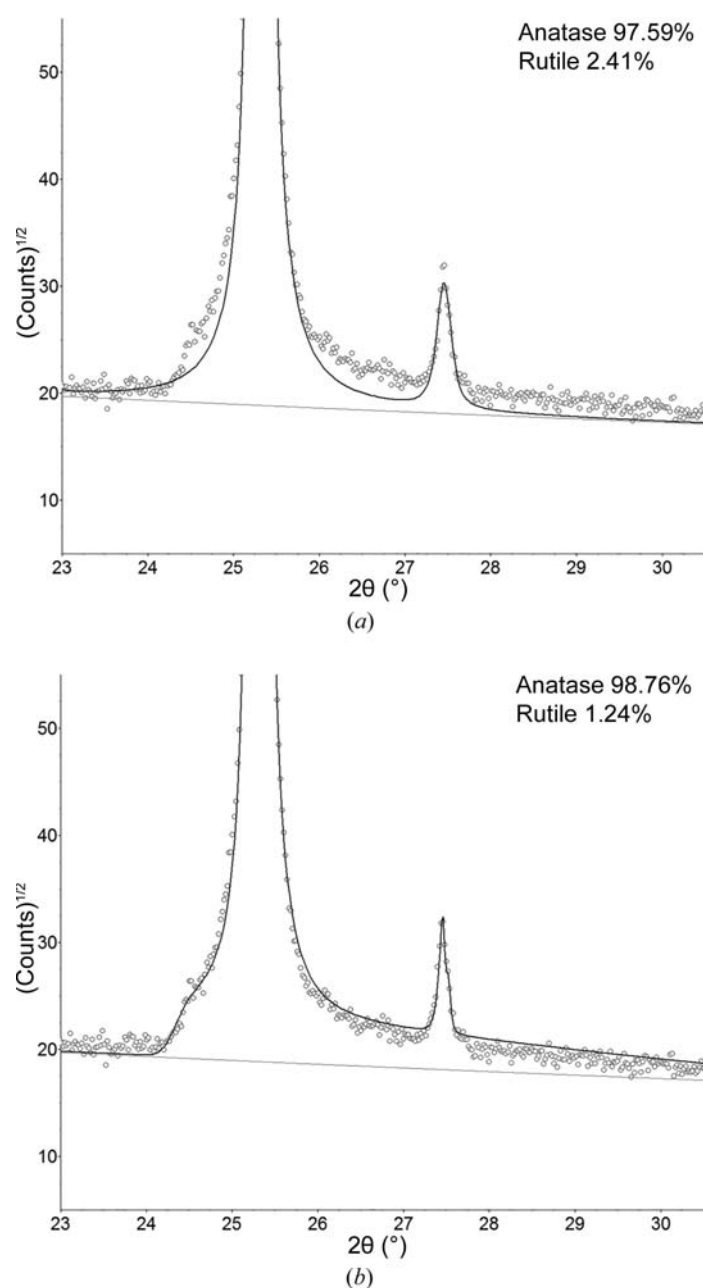


3.9. QUANTITATIVE PHASE ANALYSIS

Table 3.9.6

Compositional analysis of the Dillinger Hütte iron-ore certified reference material SX 11-14, (i) derived from QPA results, taking into account the nominal stoichiometry of the phases (XRD) and (ii) the certified analyses (Cert) (Knorr & Bornefeld, 2013)

Phase	wt%		Fe	FeO	SiO ₂	Al ₂ O ₃	MgO	CaO	K ₂ O	Na ₂ O	C
Haematite	0.37		0.26	—	—	—	—	—	—	—	—
Goethite	3.86		2.43	—	—	—	—	—	—	—	—
Magnetite	85.97		62.21	26.68	—	—	—	—	—	—	—
Quartz	5.73		—	—	5.73	—	—	—	—	—	—
Gibbsite	0.71		—	—	—	0.46	—	—	—	—	—
Talc	1.79		—	—	1.13	—	0.57	—	—	—	—
Orthoclase	0.30		—	—	0.19	0.05	—	—	0.05	—	—
Albite	0.89		—	—	0.60	0.18	—	—	—	0.10	—
Calcite	0.40		—	—	—	—	—	0.22	—	—	0.19
			Fe	FeO	SiO ₂	Al ₂ O ₃	MgO	CaO	K ₂ O	Na ₂ O	C
		XRD	64.89	26.68	7.66	0.70	0.57	0.22	0.05	0.10	0.19
		Cert	65.55	27.20	7.47	0.27	0.56	0.42	0.06	0.08	0.12
		Bias	-0.66	-0.52	0.19	0.43	0.01	-0.20	-0.01	0.02	0.07

**Figure 3.9.21**

Profile fit of anatase and rutile (a) without and (b) with a $K\beta$ filter absorption-edge correction.

result may then be evaluated by comparing the calculated elemental abundances with those determined by traditional chemical-analysis techniques. However, for the best level of agreement, this method requires that the composition of the crystalline phases be well defined. A complication, in particular for minerals, is that idealized compositions may be reported but do not necessarily match the actual composition of the species present in the sample. Where possible, detailed phase analysis using microbeam techniques should be undertaken to establish the true composition for each phase. A complication that serves to decrease the agreement is that chemically based compositional analysis does not distinguish between crystalline and amorphous phase content, while the diffraction-based QPA usually measures only the crystalline phases. Generally, the composition of amorphous phases may not be known accurately and even highly crystalline material can contain amorphous components because of non-diffracting surface layers of the grains (Cline *et al.*, 2011).

An example demonstrating the level of agreement that can be achieved is that of the iron-ore certified reference material SX 11-14 from Dillinger Hütte (Fig. 3.9.22). The material is moderately complex and consists of nine distinct mineral species. The data were measured with Co $K\alpha$ radiation and analysed using Rietveld-based QPA in *TOPAS* (Bruker AXS, 2013). The phase abundances are converted to elemental and oxide compositions for comparison with the certified elemental analyses (Table 3.9.6). There is excellent agreement between the XRD results and the chemical analysis with bias values better than ± 1 wt%.

3.9.10.3.6. Phase-specific methods: diffraction SRMs, round-robin samples and synthetic mixtures

In contrast to elemental compositional analysis, where standard reference materials (SRMs) are widespread, there are only a very limited number of SRMs available for diffraction-based QPA. Prominent examples are SRMs for the cement industry [NIST reference material clinker 8486 (Stutzman & Leigh, 2000) and ordinary Portland cement NIST SRM 2686] or ceramics materials (silicon nitride CRM BAM-S001) (Peplinski *et al.*, 2004). Similar to elemental standards, the certified values do not necessarily represent the true composition. Rather, they are published values that are typically averaged over the results from different independent methods, instruments and laboratories. Therefore, confidence limits of concentrations are provided that may be much larger than estimated standard deviations of concentrations within a single laboratory.