Acta Crystallographica Section B
Structural Science, Crystal Engineering and Materials

ISSN 2052-5206

Electronic Supplementary Information (ESI) - Habermehl *et al.*: Structure determination from powder data without prior indexing using a similarity measure based on cross-correlation functions

S1. Details of FIDEL fits and Rietveld refinements

Table S1 lists the major settings of the FIDEL fits. Background corrections were performed using the programs WinX^{POW} 2.15 (STOE & Cie, 2006) or DASH 3.1 (David *et al.*, 2006), or by FIDEL using the Bayesian background correction as implemented in ObjCryst++ (Favre-Nicolin & Černý, 2002).

Loperamide hydrochloride: The position of the chloride anion was included in the FIDEL fits. The relatively low similarity values of the FIDEL fitting are due to the asymmetry of the reflections at low angles. During Rietveld refinement the displacement factor of the chloride ion was fitted separately from the rest of the molecule. The final crystal structure is available as CIF file in the ESI with experimental details listed in Table S3.

Pigment Red 170 α-phase: The start structure was derived from the published structure of the methylated derivative through substitution of the methyl group by hydrogen. Three intramolecular degrees of freedom were included in the fitting with FIDEL: the rotations of both O-C bonds of the ethoxy side group and the rotation of the N-C bond connecting the diazo unit to the benzamide fragment.

Clarithromycin: FIDEL fitting included the variation of five torsion angles for the side groups bound to the macrolide ring. The comparison of simulated powder patterns and the experimental pattern is shown for the starting and resulting structures of the FIDEL fit (Fig. S1) and for the result of the Rietveld refinement (Fig. S2). The final crystal structure is available as CIF file in the ESI with experimental details listed in Table S3.

ETBE α-phase and **P.Y.14**: The molecular geometry resulting from the crystal structure prediction was not changed during the FIDEL fits.

S2. Details of the FIDEL fit example

Table S2 refers to the fitting of a start structure with a strongly deviating simulated powder diagram to an experimental powder pattern of P.Y.14. The results of the fitting are shown in Fig. 2 of the main article. The procedure starts with a raw FIDEL fit with a comparably high value of the neighbourhood half width l=1.0. In the second step a fine fit using a narrow neighbouring range for the pattern comparison is performed with FIDEL.

References

Coelho, A. A. (2007). *TOPAS-Academic V4.1*. Coelho Software, Brisbane, Australia.

David, W. I. F., Shankland, K., van de Streek, J., Pidcock, E., Motherwell, W. D. S. & Cole, J. C. (2006). *J. Appl. Cryst.* **39**, 910–915.

Favre-Nicolin, V. & Černý, R. (2002). *J. Appl. Cryst.* **35**, 734–743.

STOE & Cie (2006). *WinXPOW Version 2.15*. STOE & Cie GmbH, Darmstadt, Germany.

Table S1
Details of FIDEL fits to experimental powder patterns. Two values indicate different settings for the raw and the fine fitting step.

	Background correction	Profile function	*fwhm* / °	Comparison range 2θ / °	l / °	Degrees of freedom
Loperamide hydrochloride	WinX^{POW}	Gauss	0.13	4. - 50.	0.5 / 0.1	3
α-P.R.170	WinX^{POW}	Gauss	0.80	4. - 33.	0.5 / 0.1	3
Clarithromycin	WinX^{POW}	Gauss	0.18	4. - 50.	0.5 / 0.1	5
ETBE (pure α)	FIDEL	Gauss	0.16	5. - 60.	0.5 / 0.1	0
ETBE (mixture)	FIDEL	Gauss	0.30	5. - 60.	0.48 / 0.12	0
P.Y.14	DASH	Lorentz / Pseudo-Voigt	0.32 / ~0.35	5. - 30.	0.5 / 0.1	0

Table S2
Crystal structures of P.Y.14 example in space group $P1$: Random start structure, raw fit with FIDEL, fine fit with FIDEL, and reference structure in $P\bar{1}$ (Z=1) (CSD ref. code BARPAM01) for comparison

	l	$S_{12}(l)$	S^0_{12}	a / Å	b / Å	c / Å	α / °	β / °	γ / °	V / Å^3
Start structure	0.5	0.5688	0.8572	8.184	9.443	11.343	115.67	98.41	103.64	736.3
FIDEL fitted (raw)	1.0	0.9955	0.9940	8.234	9.383	11.863	113.17	98.41	104.14	786.3
FIDEL fitted (fine)	0.1	0.9926	0.9972	8.216	9.383	11.868	113.17	98.31	104.23	784.8
Reference structure				8.214	9.330	11.785	112.68	98.16	105.43	772.3

Table S3
Experimental details of the crystal structure determinations of loperamide hydrochloride and clarithromycin (CIF files in the ESI)

	Loperamide hydrochloride	Clarithromycin
Crystal data		
Chemical formula	$C_{29}H_{34}ClN_2O_2^+ \cdot Cl^-$	$C_{38}H_{69}NO_{13}$
M_r	513.48	747.94
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, $P2_12_12_1$
Temperature (K)	298	298
a, b, c (Å)	16.7095 (8), 12.3773 (5), 13.2135 (5)	20.1492 (4), 23.9545 (5), 8.8534 (2)
β (°)	101.642 (3)	
V (Å^3)	2676.59 (19)	4273.25 (16)
Z	4	4
Radiation type	Cu $K\alpha_1$, λ = 1.54056 Å	Cu $K\alpha_1$, λ = 1.54056 Å
μ (mm^{-1})	2.40	0.71
Specimen shape, size (mm)	Cylinder, 10 × 1	Cylinder, 10 × 0.7
Data collection		
Diffractometer	STOE Stadi-P diffractometer	STOE Stadi-P diffractometer
Specimen mounting	Glass capillary	Glass capillary
Data collection mode	Transmission	Transmission
Scan method	Step	Step
2θ values (°)	$2\theta_{min}$ = 2.000 $2\theta_{max}$ = 100.190 $2\theta_{step}$ = 0.01	$2\theta_{min}$ = 2.000 $2\theta_{max}$ = 69.000 $2\theta_{step}$ = 0.01
Refinement		
R factors and goodness of fit	R_p = 0.043, R_{wp} = 0.060, R_{exp} = 0.027, R_{Bragg} = 0.030, χ^2 = 5.081	R_p = 0.021, R_{wp} = 0.028, R_{exp} = 0.018, R_{Bragg} = 0.008, χ^2 = 2.356
No. of data points	9820	6800
No. of parameters	259	406
No. of restraints	201	359
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined

Computer programs: *WinX^{POW} 2.15* (STOE & Cie, 2006), *TOPAS Academic 4.1* (Coelho, 2007).

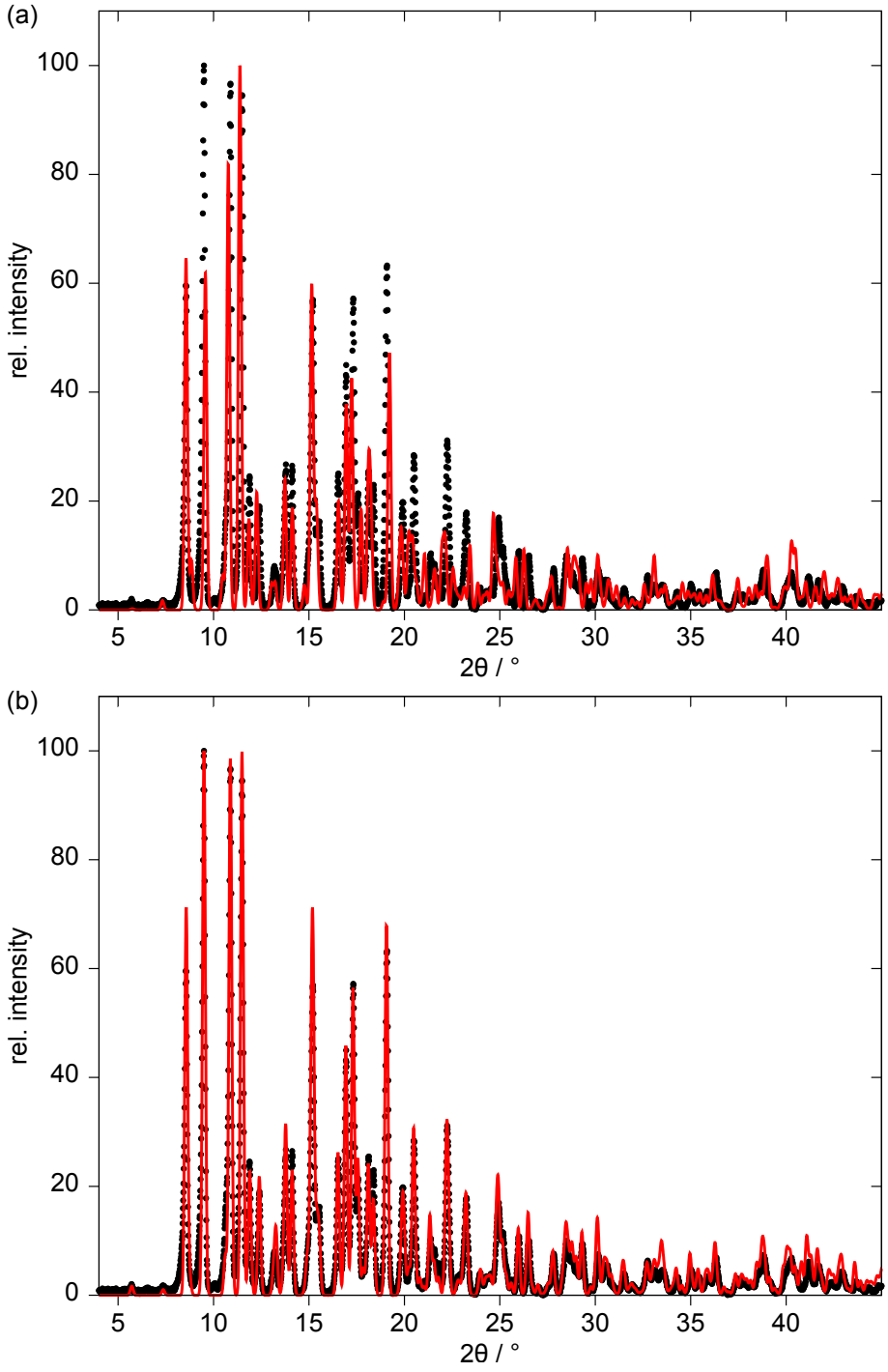

Figure S1
Experimental X-ray powder diagram of clarithromycin (dots) and simulated diagrams (red lines) (a) before optimisation with FIDEL, (b) after optimisation with FIDEL. Experimental data are background corrected.

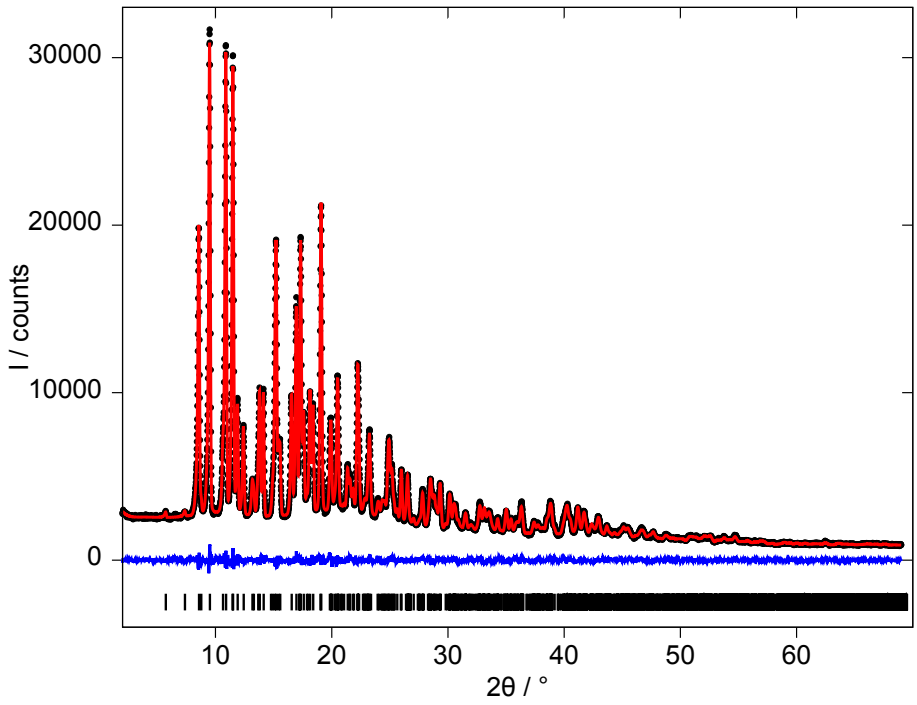

Figure S2
Rietveld refinement of clarithromycin: Experimental X-ray powder diagram (dots), simulated diagram of refined structure (red line), difference plot (blue line) and reflection positions (black)