Electronic Supplementary Material (ESI) for CrystEngComm.

Supplementary information: Disappearing disorder

Authors

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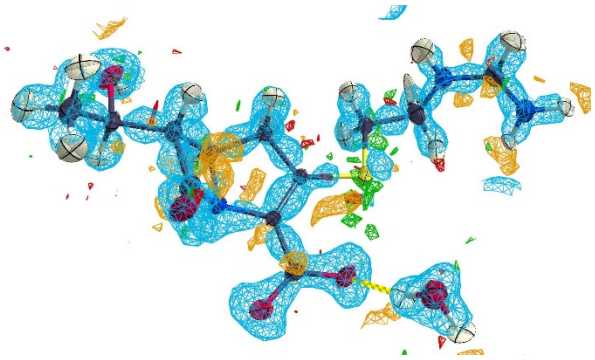
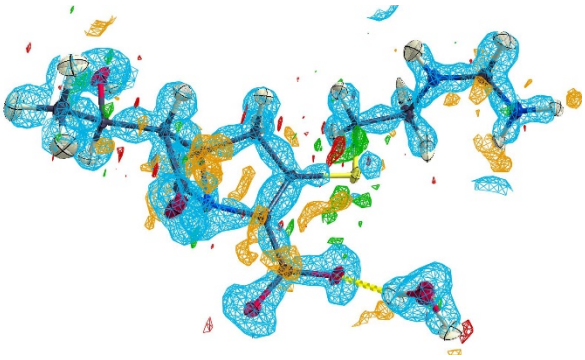
Table 1 Crystal structure determinations on imipenem monohydrate (synchrotron data).

Compound name	Imipenem monohydrate			
Chemical formula	$C_{12}H_{19}N_3O_5S$			
M_r	317.36			
Crystal system, space group	Orthorhombic, $P2_12_12_1$, No. 19			
Temperature (K)	11 K ordered Petra III	11 K disordered Petra III	80 K ordered Petra III	100 K disordered SLS
a, *b*, *c* (Å)	8.174 (4), 11.080 (6), 15.295 (10)	8.184 (9), 11.092 (9), 15.296 (8)	8.19(3), 11.054 (2), 15.31(6)	8.210 (4), 11.1142 (17), 15.372 (4)
α, β, γ (°)	90, 90, 90			
V (Å^3)	1385.2 (13)	1389 (2)	1386 (8)	1402.6 (8)
Z	4			
Radiation type	0.4769 Å	0.4769 Å	0.460 Å	0.6358 Å
μ (mm^{-1})	0.10	0.10	0.10	0.19
Crystal size (mm)	0.075 × 0.012 × 0.001	0.09 × 0.01 × 0.001	0.06 × 0.01 × 0.001	0.09 × 0.02 × 0.0014
Diffractometer	Beam line diffractometer			
Absorption corr.	empirical, SADABS version 2015/1 (G. M. Sheldrick, 2015)			
T_{min}, T_{max}	0.715, 0.862	0.650, 0.744	0.684, 0.744	0.714, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4143, 4143, 3365	4198, 4198, 3668	2378, 2378, 2233	4482, 4482, 4434
R_{int}	0.076	0.070	0.055	0.056
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.076	0.713	0.714	0.749
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.048, 0.084, 1.37	0.039, 0.065, 1.43	0.028, 0.055, 2.35	0.023, 0.056, 2.35
No. of reflections	3365	3668	2234	4434
No. of parameters	190	184	190	184
No. of restraints	—			
H-atom treatment	H Uanis estimated, X—H bond distances from invariom database			
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.56, −0.44	0.50, −0.32	0.39, −0.62	0.51, −0.23
Absolute structure (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).	1223 quotients	1451 quotients	Merged data, no quotients	1913 quotients
Absolute structure parameter	unreliable	unreliable	—	0.040 (19)



Home diffractometer, 300K



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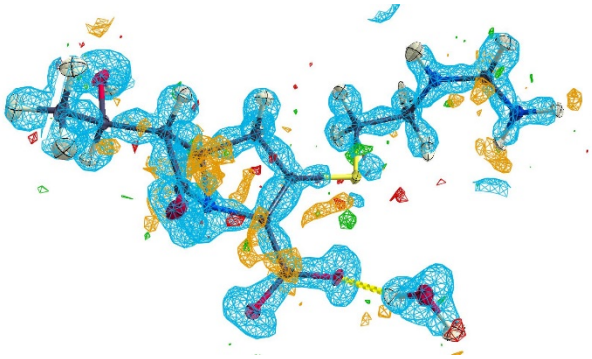

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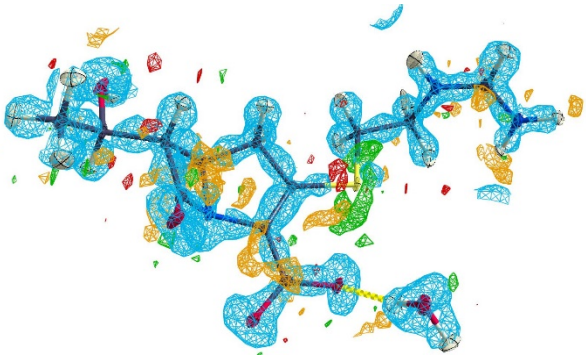

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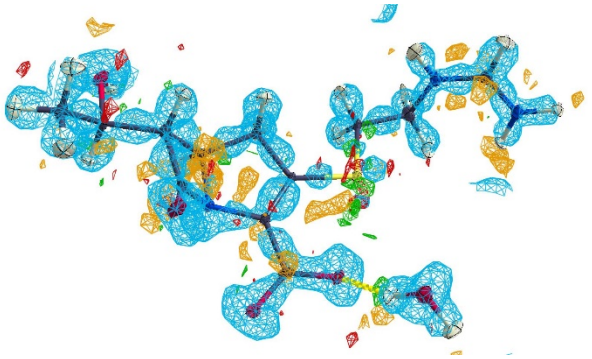
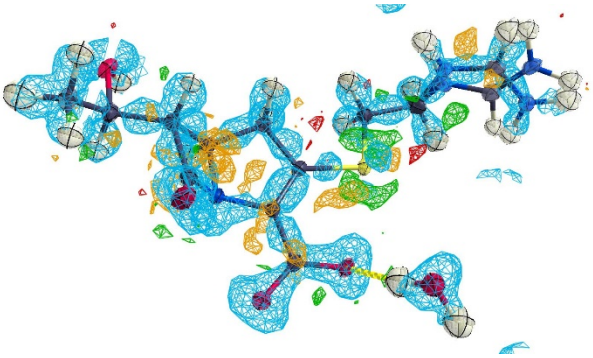
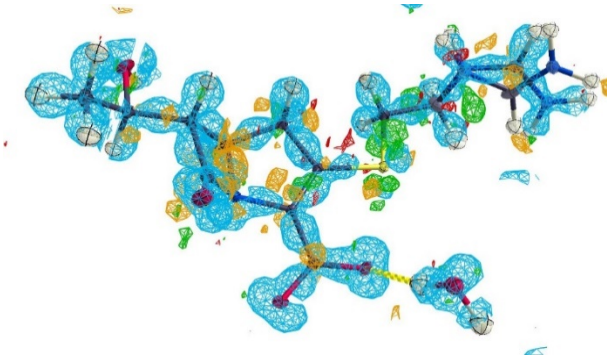


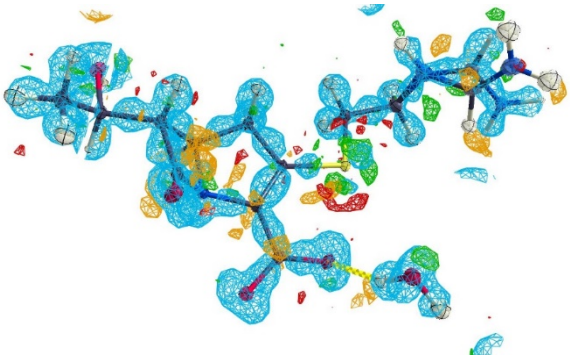
Home diffractometer, 100K



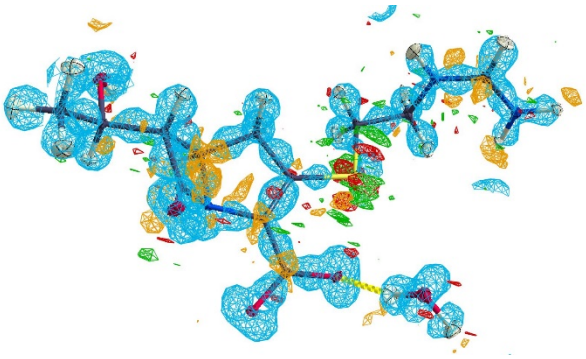
CuKα, 100K, disordered 13.9% occupancy



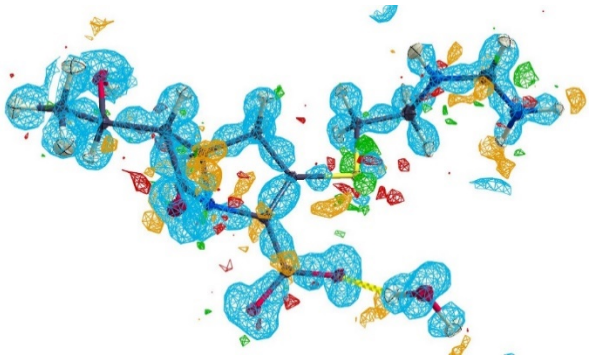
SLS, 100K, disordered, 11.2 % occupancy



Petra III, 11K, disordered, 6.7 % occupancy



PetraIII, 80K ordered,



PetraIII, 11K, ordered

Figures 1 – 10: ORTEP representations generated with MolecoolQt (Hübschle and Dittrich 2011) after Invariom refinement (Dittrich, Hübschle et al. 2013) with XDLSM (Volkov, Macchi et al. 2006) version 2006. These figures also show the deformation and the residual electron density together with the atomic displacements. Measurement temperatures and disorder status are given below the individual figures.

Table 2 Further crystal structure determination and refinement results for oxaceprol.

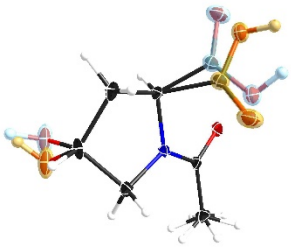
Compound name	Oxaceprol					
Chemical formula	$C_7H_{11}NO_4$					
M_r	173.17					
Crystal system, space group	Orthorhombic, $P2_12_12_1$, No. 19					
Temperature (K)	110 K	135 K	140 K	150 K	160 K	185 K
a, *b*, *c* (Å)	7.317(1), 10.562(8), 10.593(4)	7.316(1), 10.568(8), 10.599(4)	7.315(2), 10.580(4), 10.607(2)	7.3148(14), 10.583(4), 10.6112(17)	7.3148(16), 10.587(7), 10.615(4)	7.3115 (10), 10.603 (12), 10.619 (6)
α, β, γ (°)	90.0, 90.0, 90.0					
V (Å^3)	818.6(7)	819.5(7)	820.9(4)	821.4(4)	822.0(7)	823.2 (10)
Z	4					
Radiation type	Synchrotron (0.6199 Å)					
μ (mm^{-1})	0.09	0.09	0.09	0.09	0.09	0.09
Crystal size (mm)	0.65 × 0.55 × 0.50					
Diffractometer	One-axis diffractometer at beamline PX11					
Absorption corr.	Empirical, SADABS version 2015/1 (G. M. Sheldrick, 2015)					
T_{min}, T_{max}	0.692, 0.746	0.697, 0.746	0.682, 0.746	0.695, 0.746	0.655, 0.746	0.675, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] refl.	5235, 2687, 2686	5227, 2679, 2668	5265, 2681, 2674	5214, 2675, 2675	5243, 2688, 2661	5117, 2651, 2613
R_{int}	0.047	0.048	0.034	0.038	0.067	0.037
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.771	0.771	0.771	0.771	0.771	0.771
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.088, 1.10	0.032, 0.087, 1.10	0.029, 0.082, 1.09	0.030, 0.086, 1.08	0.048, 0.119, 1.10	0.032, 0.092, 1.07
No. of reflections	2687	2679	2681	2675	2688	2651
No. of parameters	140					
No. of restraints	21					
H-atom treatment	Riding hydrogens using AFIX constraints					
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.18	0.22, −0.20	0.19, -0.16	0.17, −0.18	0.34, −0.26	0.18, −0.18
Absolute structure (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).	1039 quotients	1017 quotients	1029 quotients	1026 quotients	1016 quotients	980 quotients
Absolute structure parameter	Unreliable					

Table 3 Further crystal structure determination and refinement results for oxaceprol.

Compound name	Oxaceprol					
Chemical formula	$C_7H_{11}NO_4$					
M_r	173.17					
Crystal system, space group	Orthorhombic, $P2_12_12_1$, No. 19					
Temperature (K)	200 K	215 K	230 K	250 K	260 K	270 K
a, *b*, *c* (Å)	7.316(3), 10.611(6), 10.6409(18)	7.316(3), 10.616(5), 10.650(3)	7.3155(15), 10.622(7), 10.658(5)	7.3159(17), 10.641(7), 10.698(6)	7.3159(19), 10.646(7), 10.712(6)	7.3156(18), 10.651(7), 10.725(6)
α, β, γ (°)	90.0, 90.0, 90.0					
V (Å^3)	826.1(6)	827.2(6)	828.2(7)	832.8(7)	834.3 (8)	835.7(7)
Z	4					
Radiation type	Synchrotron (0.6199 Å)					
μ (mm^{-1})	0.09	0.09	0.08	0.08	0.08	0.08
Crystal size (mm)	0.65 × 0.55 × 0.50					
Diffractometer	One-axis diffractometer at beamline PX11					
Absorption corr.	Empirical, SADABS version 2015/1 (G. M. Sheldrick, 2015)					
T_{min}, T_{max}	0.578, 0.746	0.636, 0.746	0.695, 0.746	0.693, 0.746	0.695, 0.746	0.682, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] refl.	5281, 2699, 2665	5312, 2702, 2662	5336, 2717, 2707	5309, 2706, 2694	5301, 2697, 2637	5276, 2697, 2666
R_{int}	0.101	0.090	0.052	0.056	0.052	0.054
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.771	0.771	0.771	0.771	0.771	0.771
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.066, 0.161, 1.06	0.058, 0.135, 1.08	0.037, 0.103, 1.10	0.037, 0.100, 1.09	0.038, 0.105, 1.02	0.038, 0.107, 1.07
No. of reflections	2699	2702	2717	2706	2697	2697
No. of parameters	140			152		
No. of restraints	21			—		
H-atom treatment	Riding hydrogens using AFIX constraints					
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.34	0.40, −0.27	0.26, −0.26	0.20, −0.21	0.16, −0.16	0.19, −0.16
Absolute structure (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).	1002 quotients	979 quotients	1032 quotients	1021 quotients	977 quotients	1001 quotients
Absolute structure parameter	Unreliable					

Table 4 Further crystal structure determination and refinement results for oxaceprol.

Compound name	Oxaceprol				
Chemical formula	$C_7H_{11}NO_4$				
M_r	173.17				
Crystal system, space group	Orthorhombic, $P2_12_12_1$, No. 19				
Temperature (K)	280 K	290 K	310 K	320 K	330 K
a, *b*, *c* (Å)	7.3140(15), 10.660(8), 10.738(4)	7.3140(18), 10.665(9), 10.755(4)	7.3119(18), 10.676(10), 10.792(5)	7.3111(16), 10.680(7), 10.813(5)	7.3104(17), 10.684(7), 10.828(2)
α, β, γ (°)	90.0, 90.0, 90.0				
V (Å^3)	837.2(7)	838.9(8)	842.4(9)	844.3(7)	845.7 (6)
Z	4				
Radiation type	Synchrotron (0.6199 Å)				
μ (mm^{-1})	0.08	0.08	0.08	0.08	0.08
Crystal size (mm)	0.65 × 0.55 × 0.50				
Diffractometer	One-axis diffractometer at beamline PX11				
Absorption corr.	Empirical, SADABS version 2015/1 (G. M. Sheldrick, 2015)				
T_{min}, T_{max}	0.638, 0.746	0.595, 0.746	0.588, 0.746	0.610, 0.746	0.609, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] refl.	5285, 2717, 2681	5316, 2723, 2635	5256, 2712, 2642	5281, 2717, 2664	5370, 2750, 2640
R_{int}	0.070	0.065	0.065	0.071	0.076
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.771	0.771	0.771	0.771	0.771
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.055, 0.149, 1.08	0.057, 0.153, 1.05	0.058, 0.156, 1.03	0.060, 0.158, 1.07	0.064, 0.161, 1.06
No. of reflections	2717	2723	2712	2717	2750
No. of parameters	152				
No. of restraints	—				
H-atom treatment	Riding hydrogens using AFIX constraints				
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.30	0.35, −0.26	0.33, −0.30	0.30, −0.26	0.30, −0.23
Absolute structure (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).	982 quotients	966 quotients	970 quotients	985 quotients	945 quotients
Absolute structure parameter	Unreliable				



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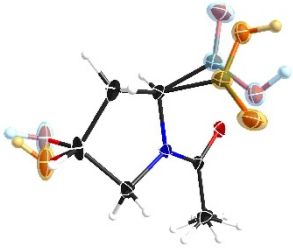

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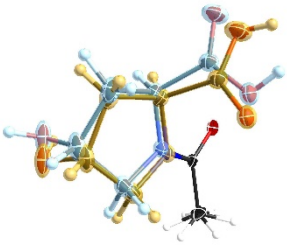

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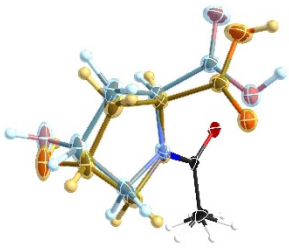

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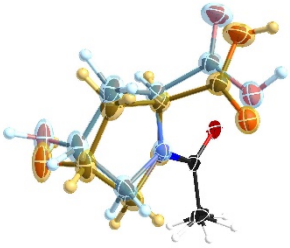

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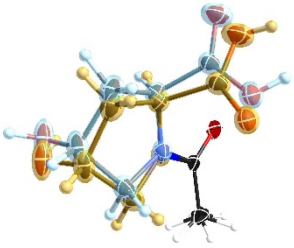

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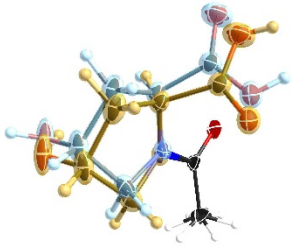

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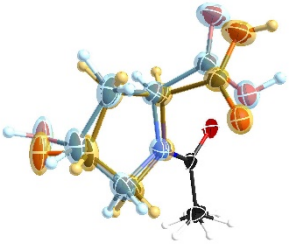

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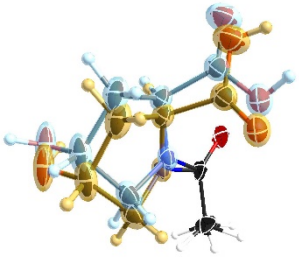

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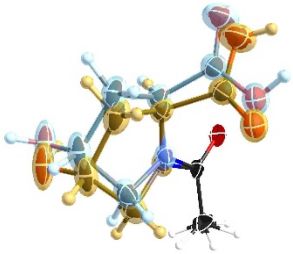

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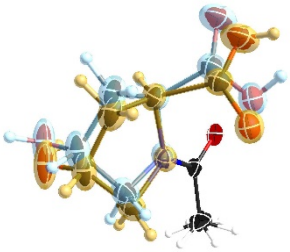

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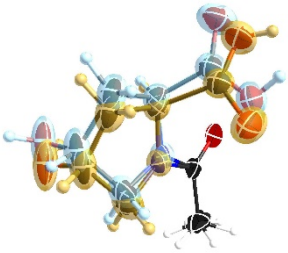

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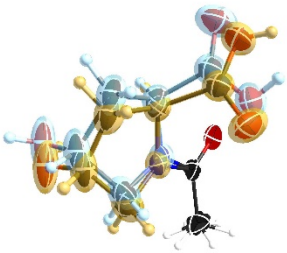

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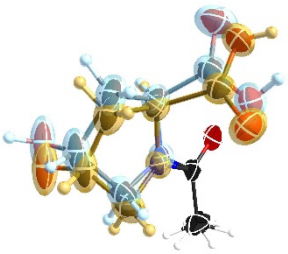

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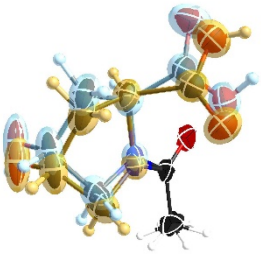

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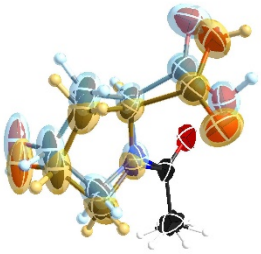

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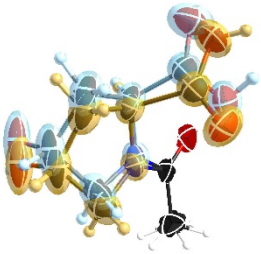

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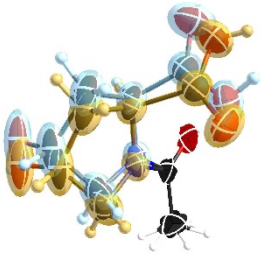

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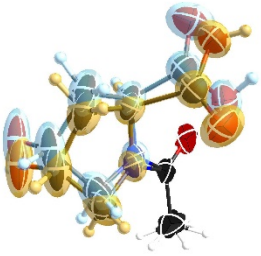

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Figures 11 – 29: ORTEP representations generated with SHELXLE (Hübschle, Sheldrick et al. 2011) after BODD (Lübben, Wandtke et al. 2019) refinement with SHELXL (Sheldrick 2015) version 2018/3. Measurement temperatures are 10, 23, 100, 110, 135, 150, 160, 185, 200, 215, 230, 250, 260, 270, 280, 290, 310, 320, 330 K.

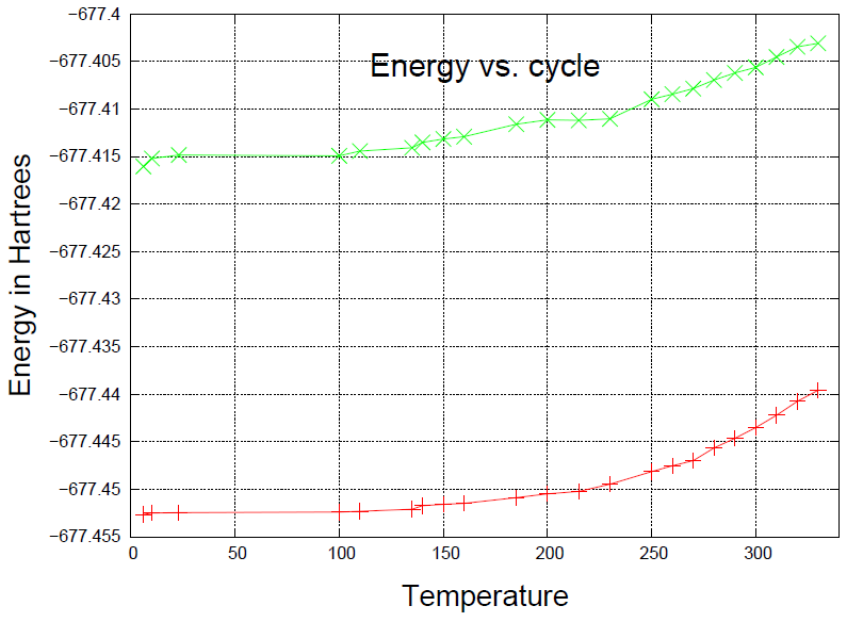

Figure 30: Comparison of the GFN2-xTB energy (Bannwarth, Ehlert et al. 2019) of the 15-molecule cluster for the archetype a (lower values) and b (higher values). Measurement temperatures of 10, 23, 100, 110, 135, 150, 160, 185, 200, 215, 230, 250, 260, 270, 280, 290, 310, 320, 330 K provided the respective unit-cell constants.

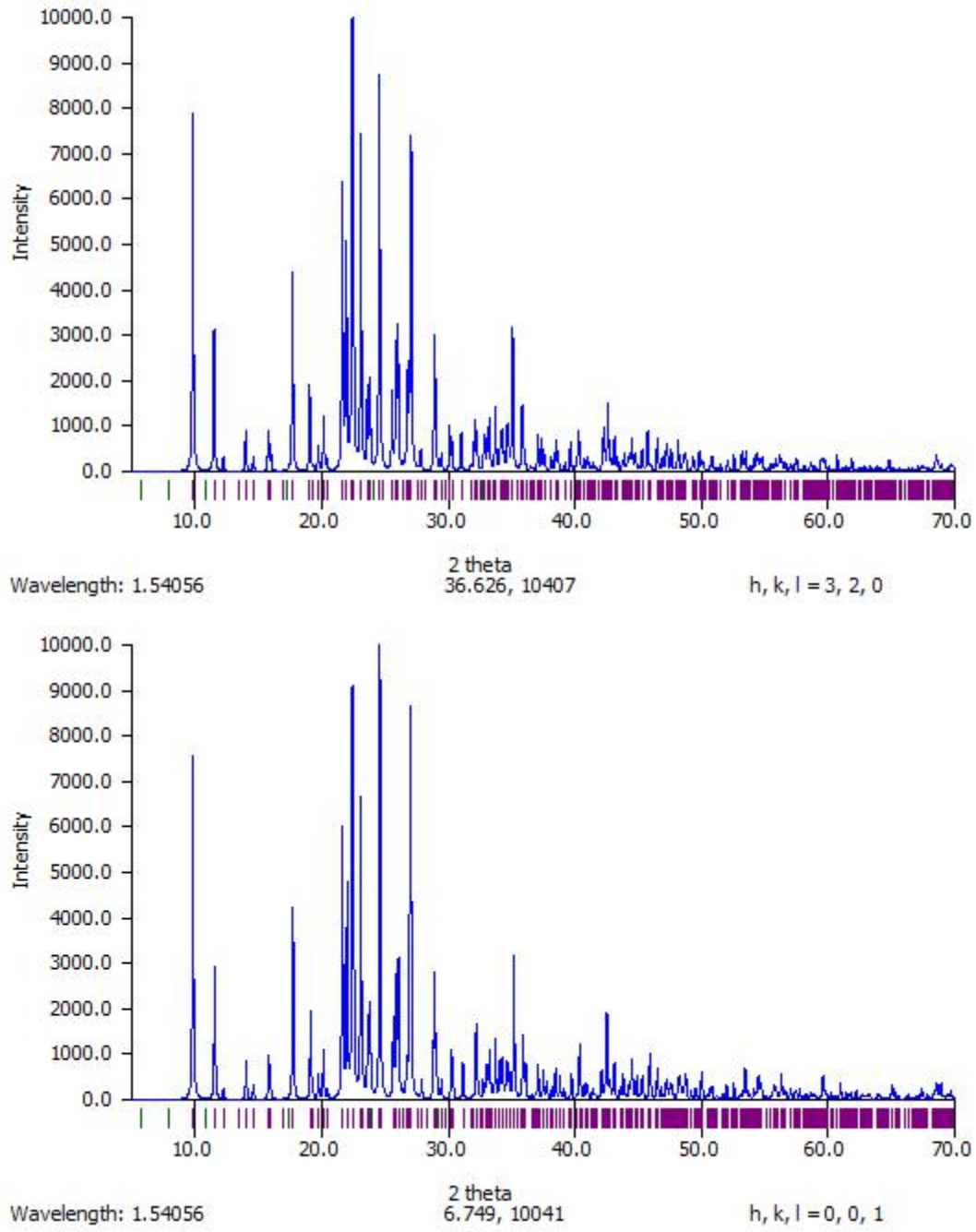

Figure 31: Comparison of simulated powder X-ray diffractograms of imipenem monohydrate at 100K from the CuKα measurement with 13.9 % disorder (top) and the 100K measurement with MoKα without disorder (bottom). Figures generated with Mercury from the single-crystal data assuming Cu Kα radiation (Macrae, Bruno et al. 2008).

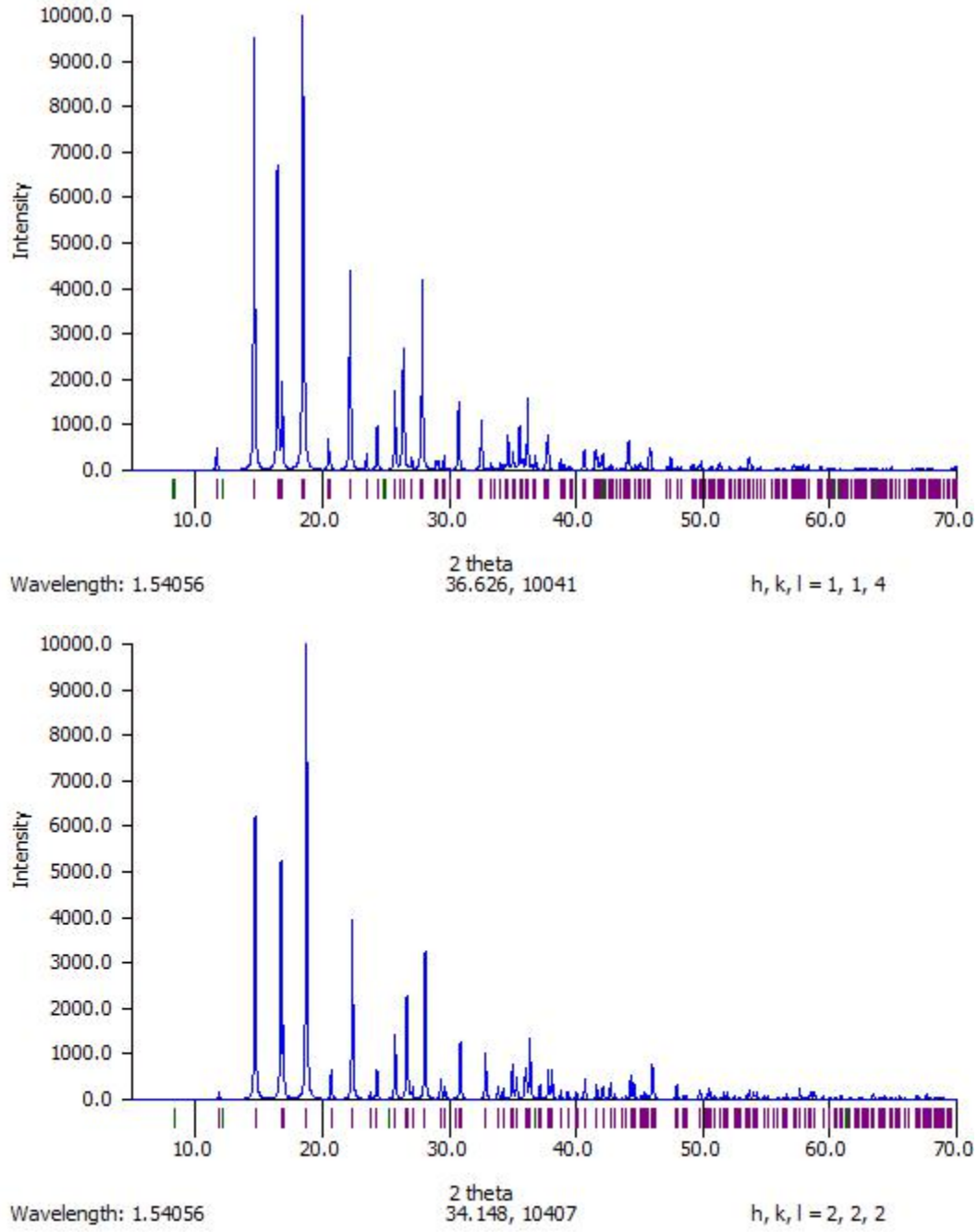

Figure 32: Comparison of the simulated powder X-ray diffractograms of oxaceprol at 290 K from the Synchrotron measurement with 12.3 % disorder (top) and the 294 K measurement with MoKα without disorder (bottom). Figures generated with Mercury from the single-crystal data assuming Cu Kα radiation (Macrae, Bruno et al. 2008).

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