



# Full wwPDB X-ray Structure Validation Report ⓘ

May 16, 2018 – 01:38 PM EDT

PDB ID : 6F8R  
Title : Crystal structure of the PDE4D catalytic domain in complex with GEBR-54  
Authors : Prosdocimi, T.; Donini, S.; Parisini, E.  
Deposited on : 2017-12-13  
Resolution : 1.83 Å(reported)

This is a Full wwPDB X-ray Structure Validation Report for a publicly released PDB entry.

We welcome your comments at [validation@mail.wwpdb.org](mailto:validation@mail.wwpdb.org)

A user guide is available at

<https://www.wwpdb.org/validation/2017/XrayValidationReportHelp>

with specific help available everywhere you see the ⓘ symbol.

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The following versions of software and data (see [references ⓘ](#)) were used in the production of this report:

MolProbity : 4.02b-467  
Mogul : 1.7.3 (157068), CSD as539be (2018)  
Xtriage (Phenix) : 1.13  
EDS : rb-20031021  
Percentile statistics : 20171227.v01 (using entries in the PDB archive December 27th 2017)  
Refmac : 5.8.0158  
CCP4 : 7.0 (Gargrove)  
Ideal geometry (proteins) : Engh & Huber (2001)  
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)  
Validation Pipeline (wwPDB-VP) : rb-20031021

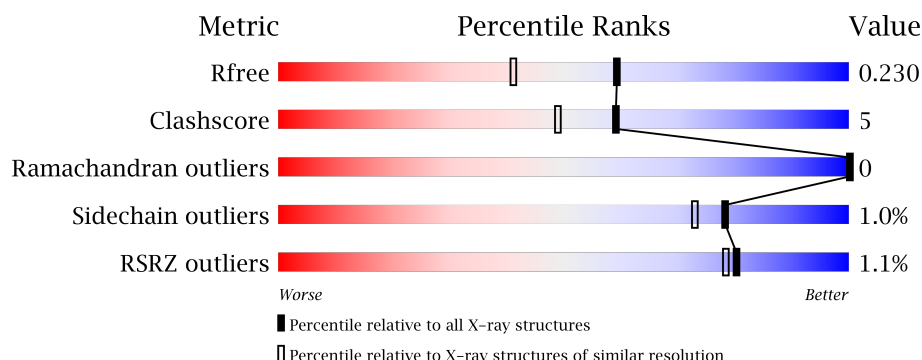
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*

The reported resolution of this entry is 1.83 Å.

Percentile scores (ranging between 0-100) for global validation metrics of the entry are shown in the following graphic. The table shows the number of entries on which the scores are based.



Metric	Whole archive (#Entries)	Similar resolution (#Entries, resolution range(Å))
$R_{free}$	111664	6455 (1.84-1.80)
Clashscore	122126	7349 (1.84-1.80)
Ramachandran outliers	120053	7272 (1.84-1.80)
Sidechain outliers	120020	7272 (1.84-1.80)
RSRZ outliers	108989	6347 (1.84-1.80)

The table below summarises the geometric issues observed across the polymeric chains and their fit to the electron density. The red, orange, yellow and green segments on the lower bar indicate the fraction of residues that contain outliers for  $\geq 3$ , 2, 1 and 0 types of geometric quality criteria. A grey segment represents the fraction of residues that are not modelled. The numeric value for each fraction is indicated below the corresponding segment, with a dot representing fractions  $\leq 5\%$ . The upper red bar (where present) indicates the fraction of residues that have poor fit to the electron density. The numeric value is given above the bar.

Mol	Chain	Length	Quality of chain
1	A	343	 .% 82% 11% 6%
1	B	343	 .% 82% 13% 6%

## 2 Entry composition

There are 6 unique types of molecules in this entry. The entry contains 5983 atoms, of which 68 are hydrogens and 0 are deuteriums.

In the tables below, the ZeroOcc column contains the number of atoms modelled with zero occupancy, the AltConf column contains the number of residues with at least one atom in alternate conformation and the Trace column contains the number of residues modelled with at most 2 atoms.

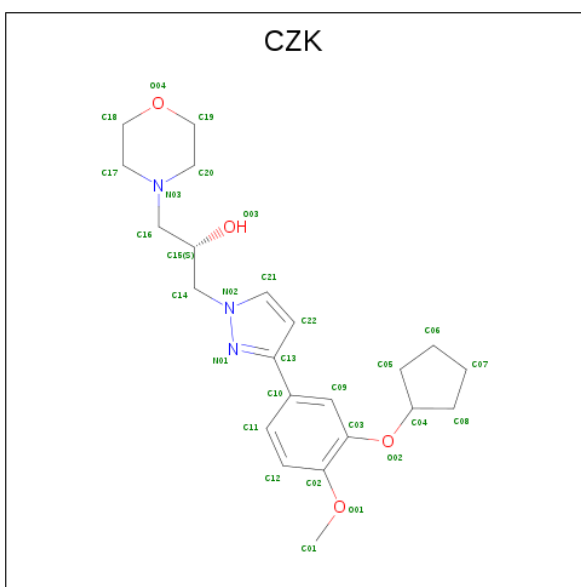
- Molecule 1 is a protein called cAMP-specific 3',5'-cyclic phosphodiesterase 4D.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	322	Total	C	N	O	S	0	5	0
			2626	1665	445	500	16			
1	B	324	Total	C	N	O	S	0	5	0
			2643	1675	449	503	16			

There are 16 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	243	MET	-	initiating methionine	UNP Q08499
A	579	ALA	-	expression tag	UNP Q08499
A	580	HIS	-	expression tag	UNP Q08499
A	581	HIS	-	expression tag	UNP Q08499
A	582	HIS	-	expression tag	UNP Q08499
A	583	HIS	-	expression tag	UNP Q08499
A	584	HIS	-	expression tag	UNP Q08499
A	585	HIS	-	expression tag	UNP Q08499
B	243	MET	-	initiating methionine	UNP Q08499
B	579	ALA	-	expression tag	UNP Q08499
B	580	HIS	-	expression tag	UNP Q08499
B	581	HIS	-	expression tag	UNP Q08499
B	582	HIS	-	expression tag	UNP Q08499
B	583	HIS	-	expression tag	UNP Q08499
B	584	HIS	-	expression tag	UNP Q08499
B	585	HIS	-	expression tag	UNP Q08499

- Molecule 2 is (2 {S})-1-[3-(3-cyclopentyloxy-4-methoxy-phenyl)pyrazol-1-yl]-3-morpholin-4-yl-propan-2-ol (three-letter code: CZK) (formula: C<sub>22</sub>H<sub>31</sub>N<sub>3</sub>O<sub>4</sub>).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
2	A	1	Total	C	H	N	O	0	0
			60	22	31	3	4		
2	B	1	Total	C	H	N	O	0	0
			60	22	31	3	4		

- Molecule 3 is MAGNESIUM ION (three-letter code: MG) (formula: Mg).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
3	B	1	Total	Mg	0	0
			1	1		
3	A	3	Total	Mg	0	0
			3	3		

- Molecule 4 is ZINC ION (three-letter code: ZN) (formula: Zn).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	B	1	Total	Zn	0	0
			1	1		
4	A	1	Total	Zn	0	0
			1	1		

- Molecule 5 is 1,2-ETHANEDIOL (three-letter code: EDO) (formula: C<sub>2</sub>H<sub>6</sub>O<sub>2</sub>).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
5	B	1	Total	C	H	O	0	0
			10	2	6	2		

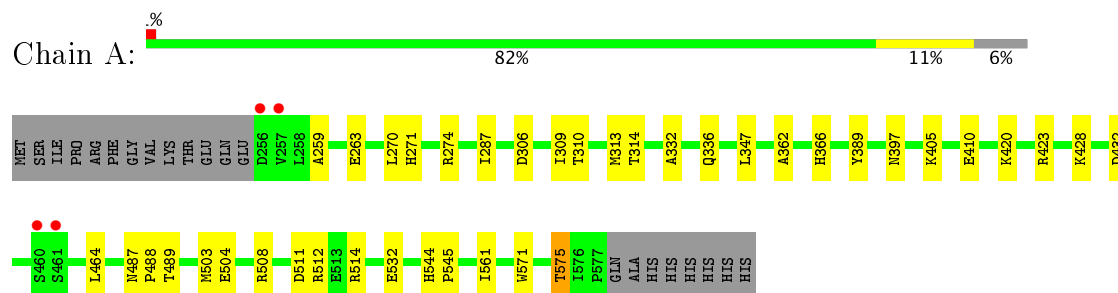
- Molecule 6 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
6	A	306	Total	O	0	0
			306	306		
6	B	272	Total	O	0	0
			272	272		

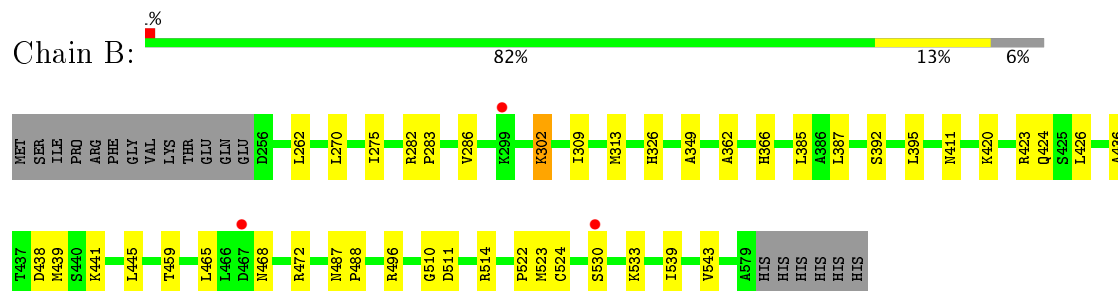
### 3 Residue-property plots [i](#)

These plots are drawn for all protein, RNA and DNA chains in the entry. The first graphic for a chain summarises the proportions of the various outlier classes displayed in the second graphic. The second graphic shows the sequence view annotated by issues in geometry and electron density. Residues are color-coded according to the number of geometric quality criteria for which they contain at least one outlier: green = 0, yellow = 1, orange = 2 and red = 3 or more. A red dot above a residue indicates a poor fit to the electron density ( $RSRZ > 2$ ). Stretches of 2 or more consecutive residues without any outlier are shown as a green connector. Residues present in the sample, but not in the model, are shown in grey.

- Molecule 1: cAMP-specific 3',5'-cyclic phosphodiesterase 4D



- Molecule 1: cAMP-specific 3',5'-cyclic phosphodiesterase 4D



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	64.67Å 98.30Å 120.09Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	49.27 – 1.83 49.27 – 1.83	Depositor EDS
% Data completeness (in resolution range)	98.7 (49.27-1.83) 98.7 (49.27-1.83)	Depositor EDS
$R_{merge}$	0.10	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	1.60 (at 1.83Å)	Xtriage
Refinement program	PHENIX 1.9_1692	Depositor
R, $R_{free}$	0.186 , 0.230 0.186 , 0.230	Depositor DCC
$R_{free}$ test set	3396 reflections (5.01%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	21.1	Xtriage
Anisotropy	0.496	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.35 , 49.0	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.48$ , $\langle L^2 \rangle = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.95	EDS
Total number of atoms	5983	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	23.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 4.05% of the height of the origin peak. No significant pseudotranslation is detected.*

<sup>1</sup>Intensities estimated from amplitudes.

<sup>2</sup>Theoretical values of  $\langle |L| \rangle$ ,  $\langle L^2 \rangle$  for acentric reflections are 0.5, 0.333 respectively for untwinned datasets, and 0.375, 0.2 for perfectly twinned datasets.

## 5 Model quality

### 5.1 Standard geometry

Bond lengths and bond angles in the following residue types are not validated in this section: CZK, ZN, MG, EDO

The Z score for a bond length (or angle) is the number of standard deviations the observed value is removed from the expected value. A bond length (or angle) with  $|Z| > 5$  is considered an outlier worth inspection. RMSZ is the root-mean-square of all Z scores of the bond lengths (or angles).

Mol	Chain	Bond lengths		Bond angles	
		RMSZ	$\# Z  > 5$	RMSZ	$\# Z  > 5$
1	A	0.36	0/2695	0.49	0/3661
1	B	0.34	0/2712	0.48	0/3683
All	All	0.35	0/5407	0.49	0/7344

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

There are no planarity outliers.

### 5.2 Too-close contacts

In the following table, the Non-H and H(model) columns list the number of non-hydrogen atoms and hydrogen atoms in the chain respectively. The H(added) column lists the number of hydrogen atoms added and optimized by MolProbity. The Clashes column lists the number of clashes within the asymmetric unit, whereas Symm-Clashes lists symmetry related clashes.

Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	A	2626	0	2598	27	0
1	B	2643	0	2614	28	0
2	A	29	31	0	0	0
2	B	29	31	0	1	0
3	A	3	0	0	0	0
3	B	1	0	0	0	0
4	A	1	0	0	0	0
4	B	1	0	0	0	0
5	B	4	6	6	0	0
6	A	306	0	0	6	0
6	B	272	0	0	9	0
All	All	5915	68	5218	54	0



The all-atom clashscore is defined as the number of clashes found per 1000 atoms (including hydrogen atoms). The all-atom clashscore for this structure is 5.

All (54) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:A:532:GLU:HG2	1:A:575:THR:HG23	1.56	0.88
1:A:532:GLU:HG2	1:A:575:THR:CG2	2.03	0.87
1:A:504:GLU:OE2	1:A:508:ARG:NH2	2.08	0.85
1:A:511:ASP:OD1	1:A:514:ARG:NH2	2.18	0.75
1:B:438:ASP:HB3	1:B:441:LYS:HD2	1.68	0.73
1:B:487:ASN:HB2	1:B:488:PRO:HD3	1.74	0.70
1:A:310:THR:O	1:A:314[B]:THR:HG23	1.94	0.68
1:B:326:HIS:NE2	6:B:701:HOH:O	2.27	0.68
1:A:508:ARG:NH1	6:A:705:HOH:O	2.29	0.65
1:A:423:ARG:NH1	6:A:706:HOH:O	2.32	0.62
1:A:309:ILE:O	1:A:313[B]:MET:HG3	2.01	0.61
1:B:511:ASP:OD1	1:B:514:ARG:NH2	2.32	0.61
1:B:423:ARG:HD3	6:B:816:HOH:O	2.02	0.59
1:A:487:ASN:HB2	1:A:488:PRO:HD3	1.86	0.58
1:B:302:LYS:NZ	6:B:710:HOH:O	2.38	0.56
1:A:420:LYS:HA	1:A:423:ARG:NH1	2.22	0.55
1:A:405:LYS:HD3	1:B:387:LEU:HD21	1.88	0.54
1:A:270:LEU:HD22	1:A:336:GLN:HG3	1.88	0.54
1:A:571:TRP:O	1:A:575:THR:HB	2.08	0.54
1:B:439:MET:HG2	6:B:935:HOH:O	2.09	0.52
1:A:347:LEU:HD21	1:A:464:LEU:HD12	1.90	0.52
1:A:428:LYS:HE3	1:A:432:ASP:OD2	2.11	0.50
1:B:349:ALA:HA	6:B:830:HOH:O	2.12	0.49
1:B:459:THR:HG22	1:B:465:LEU:HD21	1.94	0.49
1:A:544:HIS:HB3	1:A:545:PRO:HD3	1.95	0.48
1:B:309:ILE:O	1:B:313[A]:MET:HG3	2.13	0.48
1:B:510:GLY:HA3	1:B:524:CYS:O	2.13	0.48
1:A:347:LEU:CD2	1:A:464:LEU:HD12	2.44	0.47
1:B:530:SER:HA	6:B:709:HOH:O	2.13	0.47
1:B:522:PRO:O	1:B:523:MET:HB2	2.14	0.47
1:B:523:MET:SD	2:B:601:CZK:C07	3.02	0.47
1:B:262:LEU:HD23	1:B:275:ILE:HD13	1.97	0.46
1:B:530:SER:OG	1:B:533:LYS:HG3	2.15	0.46
1:A:489:THR:HB	1:A:561:ILE:HG23	1.97	0.46
1:A:389:TYR:CE1	1:A:397:ASN:HB3	2.52	0.45
1:B:496:ARG:HG2	6:B:708:HOH:O	2.16	0.45
1:A:423:ARG:HD3	6:A:856:HOH:O	2.15	0.45

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:A:271:HIS:CE1	1:A:274:ARG:HH21	2.35	0.45
1:A:362:ALA:O	1:A:366:HIS:HB3	2.17	0.44
1:B:411:ASN:ND2	6:B:703:HOH:O	2.28	0.44
1:B:420:LYS:O	1:B:424:GLN:HG3	2.18	0.44
1:B:362:ALA:O	1:B:366:HIS:HB3	2.18	0.43
1:B:436:ALA:HB1	1:B:445:LEU:HD11	1.99	0.43
1:A:287:ILE:HD12	1:A:332:ALA:HB1	2.01	0.43
1:B:283:PRO:HA	1:B:286:VAL:HG22	2.00	0.43
1:A:259:ALA:O	1:A:263:GLU:HG3	2.18	0.43
1:B:395:LEU:HD12	6:B:738:HOH:O	2.19	0.43
1:B:282:ARG:N	1:B:283:PRO:CD	2.82	0.42
1:B:270:LEU:HD21	1:B:275:ILE:HD12	2.01	0.42
1:B:539:ILE:HA	1:B:543:VAL:HB	2.01	0.42
1:A:512:ARG:HD2	6:A:952:HOH:O	2.19	0.41
1:A:306[A]:ASP:OD1	6:A:702:HOH:O	2.22	0.41
1:B:468:ASN:O	1:B:472:ARG:HG3	2.20	0.41
1:A:503:MET:HE1	6:A:808:HOH:O	2.21	0.40

There are no symmetry-related clashes.

## 5.3 Torsion angles [i](#)

### 5.3.1 Protein backbone [i](#)

In the following table, the Percentiles column shows the percent Ramachandran outliers of the chain as a percentile score with respect to all X-ray entries followed by that with respect to entries of similar resolution.

The Analysed column shows the number of residues for which the backbone conformation was analysed, and the total number of residues.

Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles	
1	A	325/343 (95%)	320 (98%)	5 (2%)	0	100	100
1	B	327/343 (95%)	323 (99%)	4 (1%)	0	100	100
All	All	652/686 (95%)	643 (99%)	9 (1%)	0	100	100

There are no Ramachandran outliers to report.

### 5.3.2 Protein sidechains [i](#)

In the following table, the Percentiles column shows the percent sidechain outliers of the chain as a percentile score with respect to all X-ray entries followed by that with respect to entries of similar resolution.

The Analysed column shows the number of residues for which the sidechain conformation was analysed, and the total number of residues.

Mol	Chain	Analysed	Rotameric	Outliers	Percentiles	
1	A	299/313 (96%)	297 (99%)	2 (1%)	85	82
1	B	300/313 (96%)	295 (98%)	5 (2%)	63	53
All	All	599/626 (96%)	592 (99%)	7 (1%)	78	67

All (7) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	410	GLU
1	A	575	THR
1	B	302	LYS
1	B	385	LEU
1	B	392[A]	SER
1	B	392[B]	SER
1	B	426	LEU

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. There are no such sidechains identified.

### 5.3.3 RNA [i](#)

There are no RNA molecules in this entry.

## 5.4 Non-standard residues in protein, DNA, RNA chains [i](#)

There are no non-standard protein/DNA/RNA residues in this entry.

## 5.5 Carbohydrates [i](#)

There are no carbohydrates in this entry.

## 5.6 Ligand geometry

Of 9 ligands modelled in this entry, 6 are monoatomic - leaving 3 for Mogul analysis.

In the following table, the Counts columns list the number of bonds (or angles) for which Mogul statistics could be retrieved, the number of bonds (or angles) that are observed in the model and the number of bonds (or angles) that are defined in the Chemical Component Dictionary. The Link column lists molecule types, if any, to which the group is linked. The Z score for a bond length (or angle) is the number of standard deviations the observed value is removed from the expected value. A bond length (or angle) with  $|Z| > 2$  is considered an outlier worth inspection. RMSZ is the root-mean-square of all Z scores of the bond lengths (or angles).

Mol	Type	Chain	Res	Link	Bond lengths			Bond angles		
					Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2
2	CZK	A	601	-	31,32,32	1.15	2 (6%)	40,43,43	1.86	7 (17%)
2	CZK	B	601	-	31,32,32	1.07	0	40,43,43	3.05	17 (42%)
5	EDO	B	604	-	3,3,3	0.41	0	2,2,2	0.57	0

In the following table, the Chirals column lists the number of chiral outliers, the number of chiral centers analysed, the number of these observed in the model and the number defined in the Chemical Component Dictionary. Similar counts are reported in the Torsion and Rings columns. '-' means no outliers of that kind were identified.

Mol	Type	Chain	Res	Link	Chirals	Torsions	Rings
2	CZK	A	601	-	-	0/16/33/33	0/4/4/4
2	CZK	B	601	-	-	0/16/33/33	0/4/4/4
5	EDO	B	604	-	-	0/1/1/1	0/0/0/0

All (2) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	A	601	CZK	C10-C13	-2.68	1.44	1.48
2	A	601	CZK	C09-C03	2.29	1.42	1.38

All (24) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	B	601	CZK	O02-C03-C09	-7.56	106.05	123.68
2	A	601	CZK	O02-C03-C09	-6.33	108.92	123.68
2	B	601	CZK	C09-C10-C13	-4.58	114.32	120.59
2	B	601	CZK	C22-C13-N01	-2.98	106.68	110.30
2	B	601	CZK	C10-C13-N01	-2.59	116.97	120.98
2	B	601	CZK	O01-C02-C03	-2.19	112.39	115.41
2	B	601	CZK	C21-C22-C13	2.02	106.31	105.21

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	A	601	CZK	C10-C13-N01	2.03	124.13	120.98
2	B	601	CZK	C10-C09-C03	2.19	123.04	120.08
2	A	601	CZK	C18-C17-N03	2.22	113.17	110.11
2	A	601	CZK	C03-O02-C04	2.42	128.74	120.36
2	B	601	CZK	O04-C18-C17	2.63	117.58	111.81
2	A	601	CZK	C21-C22-C13	2.76	106.71	105.21
2	B	601	CZK	O01-C02-C12	3.11	129.61	124.36
2	B	601	CZK	C13-N01-N02	3.36	110.29	105.42
2	B	601	CZK	C14-C15-C16	4.19	120.55	110.79
2	B	601	CZK	C01-O01-C02	4.40	124.06	117.53
2	A	601	CZK	C19-C20-N03	4.51	116.32	110.11
2	A	601	CZK	O02-C03-C02	4.57	131.14	117.01
2	B	601	CZK	C18-C17-N03	4.67	116.54	110.11
2	B	601	CZK	C11-C10-C13	4.71	128.81	121.30
2	B	601	CZK	O02-C03-C02	5.56	134.21	117.01
2	B	601	CZK	C19-C20-N03	6.11	118.52	110.11
2	B	601	CZK	C22-C13-C10	8.10	136.35	128.77

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

1 monomer is involved in 1 short contact:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
2	B	601	CZK	1	0

## 5.7 Other polymers [i](#)

There are no such residues in this entry.

## 5.8 Polymer linkage issues [i](#)

There are no chain breaks in this entry.

## 6 Fit of model and data [i](#)

### 6.1 Protein, DNA and RNA chains [i](#)

In the following table, the column labelled ‘#RSRZ> 2’ contains the number (and percentage) of RSRZ outliers, followed by percent RSRZ outliers for the chain as percentile scores relative to all X-ray entries and entries of similar resolution. The OWAB column contains the minimum, median, 95<sup>th</sup> percentile and maximum values of the occupancy-weighted average B-factor per residue. The column labelled ‘Q< 0.9’ lists the number of (and percentage) of residues with an average occupancy less than 0.9.

Mol	Chain	Analysed	<RSRZ>	#RSRZ>2	OWAB(Å <sup>2</sup> )	Q<0.9
1	A	322/343 (93%)	-0.18	4 (1%) 79 77	10, 19, 35, 55	0
1	B	324/343 (94%)	0.05	3 (0%) 84 82	12, 22, 41, 52	0
All	All	646/686 (94%)	-0.07	7 (1%) 80 78	10, 21, 39, 55	0

All (7) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	460	SER	3.7
1	A	257	VAL	3.2
1	A	461	SER	3.1
1	B	467	ASP	2.5
1	B	530	SER	2.4
1	B	299	LYS	2.2
1	A	256	ASP	2.2

### 6.2 Non-standard residues in protein, DNA, RNA chains [i](#)

There are no non-standard protein/DNA/RNA residues in this entry.

### 6.3 Carbohydrates [i](#)

There are no carbohydrates in this entry.

### 6.4 Ligands [i](#)

In the following table, the Atoms column lists the number of modelled atoms in the group and the number defined in the chemical component dictionary. The B-factors column lists the minimum, median, 95<sup>th</sup> percentile and maximum values of B factors of atoms in the group. The column labelled ‘Q< 0.9’ lists the number of atoms with occupancy less than 0.9.

Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors( $\text{\AA}^2$ )	Q<0.9
2	CZK	B	601	29/29	0.70	0.29	22,38,50,54	60
2	CZK	A	601	29/29	0.84	0.17	20,33,51,59	0
3	MG	A	604	1/1	0.86	0.07	33,33,33,33	0
5	EDO	B	604	4/4	0.94	0.19	22,26,29,30	0
3	MG	A	603	1/1	0.98	0.10	22,22,22,22	0
3	MG	B	602	1/1	0.99	0.12	10,10,10,10	0
3	MG	A	602	1/1	0.99	0.10	9,9,9,9	0
4	ZN	A	605	1/1	1.00	0.06	15,15,15,15	0
4	ZN	B	603	1/1	1.00	0.07	17,17,17,17	0

## 6.5 Other polymers [i](#)

There are no such residues in this entry.