

# Full wwPDB X-ray Structure Validation Report (i)

Nov 15, 2021 – 11:04 am GMT

PDB ID : 7AY9

Title: Crystal structure of CK2 bound by compound 7

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Deposited on : 2020-11-11

Resolution : 2.25 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

A user guide is available at

https://www.wwpdb.org/validation/2017/XrayValidationReportHelp with specific help available everywhere you see the (i) symbol.

The following versions of software and data (see references (1)) were used in the production of this report:

MolProbity : 4.02b-467

Mogul : 1.8.4 (270009), CSD as541be (2020)

Xtriage (Phenix) : 1.13

EDS : 2.23.2 buster-report : 1.1.7 (2018)

Percentile statistics : 20191225.v01 (using entries in the PDB archive December 25th 2019)

Refmac : 5.8.0267

CCP4 : 7.1.010 (Gargrove)

Ideal geometry (proteins) : Engh & Huber (2001) Ideal geometry (DNA, RNA) : Parkinson et al. (1996)

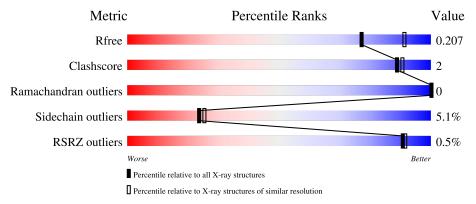
Validation Pipeline (wwPDB-VP) : 2.23.2

# 1 Overall quality at a glance (i)

The following experimental techniques were used to determine the structure: X- $RAY\ DIFFRACTION$ 

The reported resolution of this entry is 2.25 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 $(\# \mathrm{Entries})$	$\begin{array}{c} {\rm Similar\ resolution} \\ (\#{\rm Entries},{\rm resolution\ range}(\mathring{\rm A})) \end{array}$
$R_{free}$	130704	1377 (2.26-2.26)
Clashscore	141614	1487 (2.26-2.26)
Ramachandran outliers	138981	1449 (2.26-2.26)
Sidechain outliers	138945	1450 (2.26-2.26)
RSRZ outliers	127900	1356 (2.26-2.26)

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 of the lower bar indicate the fraction of residues that contain outliers for >=3, 2, 1 and 0 types of geometric quality criteria respectively. 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 <=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	342	90%	6% •
1	В	342	87%	9% • •

The following table lists non-polymeric compounds, carbohydrate monomers and non-standard residues in protein, DNA, RNA chains that are outliers for geometric or electron-density-fit criteria:



Mol	Type	Chain	Res	Chirality	Geometry	Clashes	Electron density
4	EDO	В	416	_	-	-	X



# 2 Entry composition (i)

There are 5 unique types of molecules in this entry. The entry contains 6190 atoms, of which 0 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 Casein kinase II subunit alpha.

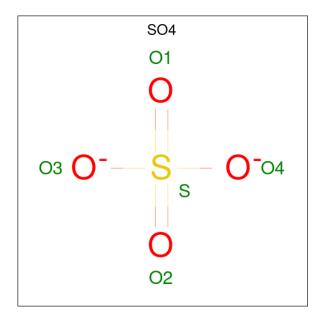
Mol	Chain	Residues	Atoms			ZeroOcc	AltConf	Trace		
1	A	331	Total 2761	C 1771	N 486	O 493	S 11	0	0	0
1	В	331	Total 2748	C 1766	N 480	O 491	S 11	0	0	0

There are 12 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	337	HIS	=	expression tag	UNP P68400
A	338	HIS	ı	expression tag	UNP P68400
A	339	HIS	-	expression tag	UNP P68400
A	340	HIS	ı	expression tag	UNP P68400
A	341	HIS	-	expression tag	UNP P68400
A	342	HIS	-	expression tag	UNP P68400
В	337	HIS	ı	expression tag	UNP P68400
В	338	HIS	-	expression tag	UNP P68400
В	339	HIS	ı	expression tag	UNP P68400
В	340	HIS	-	expression tag	UNP P68400
В	341	HIS	-	expression tag	UNP P68400
В	342	HIS	-	expression tag	UNP P68400

• Molecule 2 is SULFATE ION (three-letter code: SO4) (formula: O<sub>4</sub>S).

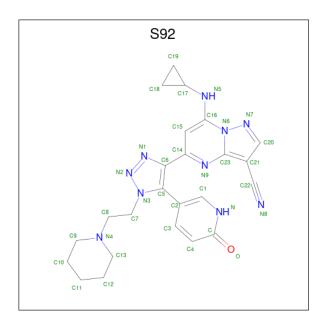




Mol	Chain	Residues	Ato	ms		ZeroOcc	AltConf
2	A	1	Total 5	O 4	S 1	0	0
2	A	1	Total 5	O 4	S 1	0	0
2	A	1	Total 5	O 4	S 1	0	0
2	A	1	Total 5	O 4	S 1	0	0
2	A	1	Total 5	O 4	S 1	0	0
2	A	1	Total 5	O 4	S 1	0	0
2	В	1	Total 5	O 4	S 1	0	0
2	В	1	Total 5	O 4	S 1	0	0
2	В	1	Total 5	O 4	S 1	0	0
2	В	1	Total 5	O 4	S 1	0	0
2	В	1	Total 5	O 4	S 1	0	0

• Molecule 3 is 7-(cyclopropylamino)-5-(5-(6-oxo-1,6-dihydropyridin-3-yl)-1-(2-(piperidin-1-yl)ethyl)-1H-1,2,3-triazol-4-yl)pyrazolo[1,5-a]pyrimidine-3-carbonitrile (three-letter code: S92) (formula:  $C_{24}H_{26}N_{10}O$ ) (labeled as "Ligand of Interest" by depositor).





Mol	Chain	Residues	Atoms				ZeroOcc	AltConf	
9	Λ	1	Total	С	N	О	0	0	
3	А	1	35	24	10	1	0	U	
9	D	1	Total	С	N	О	0	0	
3	Б	1	35	24	10	1	U	0	

 $\bullet$  Molecule 4 is 1,2-ETHANEDIOL (three-letter code: EDO) (formula:  $\mathrm{C_2H_6O_2}).$ 



Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
4	A	1	Total C O 4 2 2	0	0
4	A	1	Total C O 4 2 2	0	0



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Mol	Chain	$oxed{ \mathbf{Residues} }$	Atoms	ZeroOcc	AltConf
			Total C O		
4	A	1	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	0	0
4	A	1	Total C O	0	0
4	A	1	4  2  2	U	U
4	A	1	Total C O	0	0
4	Λ	1	4   2   2	U	
4	A	1	Total C O	0	0
-1	11	1	4 2 2	U	0
4	A	1	Total C O	0	0
	71	1	4 2 2	Ŭ	0
4	A	1	Total C O	0	0
1	11	1	4 2 2	Ŭ	<u> </u>
4	A	1	Total C O	0	0
	11	-	4 2 2	Ů	Ü
4	A	1	Total C O	0	0
	11	1	4 2 2	Ŭ	0
4	A	1	Total C O	0	0
	11	-	4 2 2	Ů	
4	В	1	Total C O	0	0
		_	4 2 2		Ü
4	В	1	Total C O	0	0
_	_	_	4 2 2		
4	В	1	Total C O	0	0
			4 2 2		-
4	В	1	Total C O	0	0
			4 2 2	_	
4	В	1	Total C O	0	0
			4 2 2		
4	В	1	Total C O	0	0
			4 2 2		
4	В	1	Total C O	0	0
			4 2 2		
4	В	1	Total C O	0	0
			4 2 2		
4	В	1	Total C O	0	0
			4 2 2 Total C O		
4	В	1		0	0
4	В	1		0	0
			4 2 2 Total C O		
4	В	1		0	0
			4 2 2	1	



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Mol	Chain	Residues	Ato	ms	ZeroOcc	AltConf
4	В	1	Total 4	C O 2	0	0

#### • Molecule 5 is water.

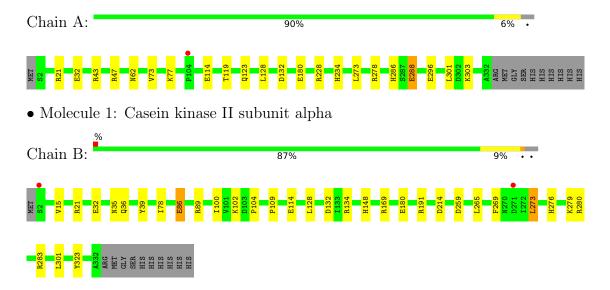
Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
5	A	223	Total O 223 223	0	0
5	В	237	Total O 237 237	0	0



# 3 Residue-property plots (i)

These plots are drawn for all protein, RNA, DNA and oligosaccharide 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: Casein kinase II subunit alpha





# 4 Data and refinement statistics (i)

Property	Value	Source
Space group	P 43 21 2	Depositor
Cell constants	127.45Å 127.45Å 124.84Å	Domositon
a, b, c, $\alpha$ , $\beta$ , $\gamma$	$90.00^{\circ}$ $90.00^{\circ}$ $90.00^{\circ}$	Depositor
Resolution (Å)	18.18 - 2.25	Depositor
Resolution (A)	90.12  -  2.25	EDS
% Data completeness	99.8 (18.18-2.25)	Depositor
(in resolution range)	99.8 (90.12-2.25)	EDS
$R_{merge}$	0.10	Depositor
$R_{sym}$	(Not available)	Depositor
$< I/\sigma(I) > 1$	4.04 (at 2.25Å)	Xtriage
Refinement program	BUSTER 2.11.7	Depositor
D D	0.166 , 0.207	Depositor
$R, R_{free}$	0.168 , $0.207$	DCC
$R_{free}$ test set	2387 reflections (4.88%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	30.6	Xtriage
Anisotropy	0.284	Xtriage
Bulk solvent $k_{sol}(e/Å^3)$ , $B_{sol}(Å^2)$	(Not available), (Not available)	EDS
L-test for twinning <sup>2</sup>	$< L > = 0.48, < L^2> = 0.32$	Xtriage
Estimated twinning fraction	0.016 for -h,-l,-k	Xtriage
Estimated twinning fraction	0.005 for l,-k,h	Atriage
$F_o, F_c$ correlation	0.95	EDS
Total number of atoms	6190	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	35.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The analyses of the Patterson function reveals a significant off-origin peak that is 32.82 % of the origin peak, indicating pseudo-translational symmetry. The chance of finding a peak of this or larger height randomly in a structure without pseudo-translational symmetry is equal to 8.8397e-04. The detected translational NCS is most likely also responsible for the elevated intensity ratio.

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



<sup>&</sup>lt;sup>1</sup>Intensities estimated from amplitudes.

# 5 Model quality (i)

### 5.1 Standard geometry (i)

Bond lengths and bond angles in the following residue types are not validated in this section: S92, SO4, 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		
MIOI	Chain	RMSZ	# Z  > 5	RMSZ	# Z  > 5	
1	A	0.52	0/2836	0.64	0/3840	
1	В	0.52	0/2823	0.64	0/3825	
All	All	0.52	0/5659	0.64	0/7665	

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 (i)

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	2761	0	2684	5	0
1	В	2748	0	2667	15	0
2	A	30	0	0	0	0
2	В	25	0	0	0	0
3	A	35	0	0	0	0
3	В	35	0	0	0	0
4	A	44	0	66	3	0
4	В	52	0	78	4	0
5	A	223	0	0	1	0
5	В	237	0	0	1	0
All	All	6190	0	5495	21	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 2.

All (21) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Interatomic	Clash
Atom-1	Atom-2	${ m distance}({ m \AA})$	overlap (Å)
1:B:86:GLU:HG3	1:B:89:ARG:HH12	1.48	0.79
1:B:86:GLU:HG3	1:B:89:ARG:NH1	2.19	0.57
1:A:296:GLU:HG2	5:A:622:HOH:O	2.04	0.56
1:A:286:HIS:CE1	1:A:288:GLU:HG2	2.44	0.52
1:B:148:HIS:HE1	1:B:214:ASP:OD2	1.93	0.51
1:B:279:LYS:HB3	1:B:283:ARG:HD3	1.92	0.51
1:B:21:ARG:HB3	5:B:714:HOH:O	2.10	0.50
1:B:39:TYR:HB2	4:B:412:EDO:H21	1.94	0.50
1:B:36:GLN:HG3	1:B:104:PRO:HD3	1.93	0.50
1:B:134:ARG:HG2	1:B:323:TYR:CZ	2.52	0.45
1:B:279:LYS:HA	4:B:413:EDO:H21	1.98	0.45
1:B:269:PHE:HB3	1:B:273:LEU:HD22	2.00	0.44
1:A:303:LYS:HE3	4:A:414:EDO:H12	1.99	0.43
1:A:278:ARG:HG3	4:A:408:EDO:H11	2.01	0.43
1:B:280:ARG:HH11	4:B:413:EDO:H22	1.84	0.43
1:B:276:HIS:HD2	4:B:415:EDO:C2	2.32	0.42
1:A:128:LEU:HD22	1:A:132:ASP:HB3	2.03	0.41
1:B:78:ILE:HD13	1:B:109:PRO:HG2	2.03	0.40
1:B:128:LEU:HD22	1:B:132:ASP:HB3	2.02	0.40
1:B:273:LEU:HD12	1:B:273:LEU:HA	1.97	0.40
4:A:413:EDO:H11	4:A:417:EDO:H22	2.04	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	Favoured Allowed Out		Perce	ntiles
1	A	329/342 (96%)	317 (96%)	12 (4%)	0	100	100



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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Perce	entiles
1	В	329/342 (96%)	321 (98%)	8 (2%)	0	100	100
All	All	658/684 (96%)	638 (97%)	20 (3%)	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	Rotameric Outliers	
1	A	$296/314 \ (94\%)$	280 (95%)	16 (5%)	22 22
1	В	294/314 (94%)	280 (95%)	14 (5%)	25 28
All	All	590/628 (94%)	560 (95%)	30 (5%)	24 25

All (30) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	21	ARG
1	A	32	GLU
1	A	43	ARG
1	A	47	ARG
1	A	62	ASN
1	A	73	VAL
1	A	77	LYS
1	A	114	GLU
1	A	119	THR
1	A	123	GLN
1	A	180	GLU
1	A	228	ARG
1	A	234	HIS
1	A	273	LEU
1	A	288	GLU
1	A	301	LEU
1	В	15	VAL
1	В	32	GLU



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Mol	Chain	Res	Type
1	В	35	ASN
1	В	86	GLU
1	В	100	ILE
1	В	102	LYS
1	В	114	GLU
1	В	169	ARG
1	В	180	GLU
1	В	191	ARG
1	В	259	ASP
1	В	265	LEU
1	В	273	LEU
1	В	301	LEU

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (12) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	16	ASN
1	A	35	ASN
1	A	126	GLN
1	A	234	HIS
1	A	309	HIS
1	В	16	ASN
1	В	35	ASN
1	В	40	GLN
1	В	126	GLN
1	В	148	HIS
1	В	186	GLN
1	В	276	HIS

#### 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 monosaccharides in this entry.



### 5.6 Ligand geometry (i)

37 ligands are modelled in this entry.

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

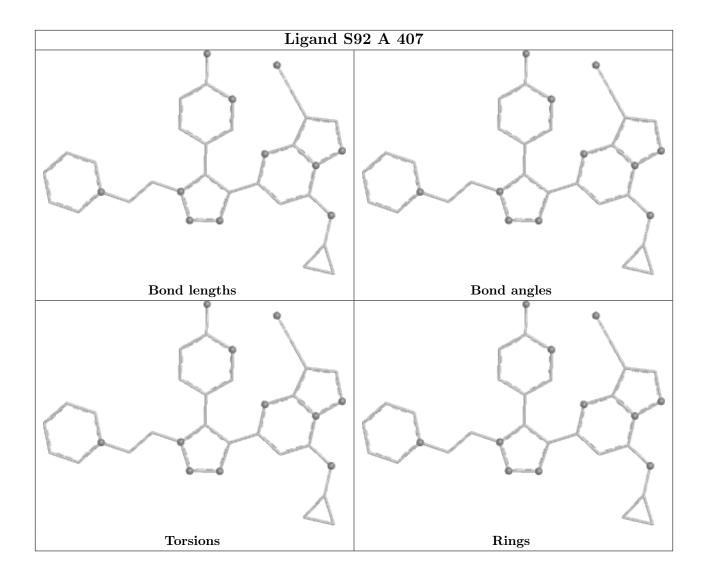
There are no torsion outliers.

There are no ring outliers.

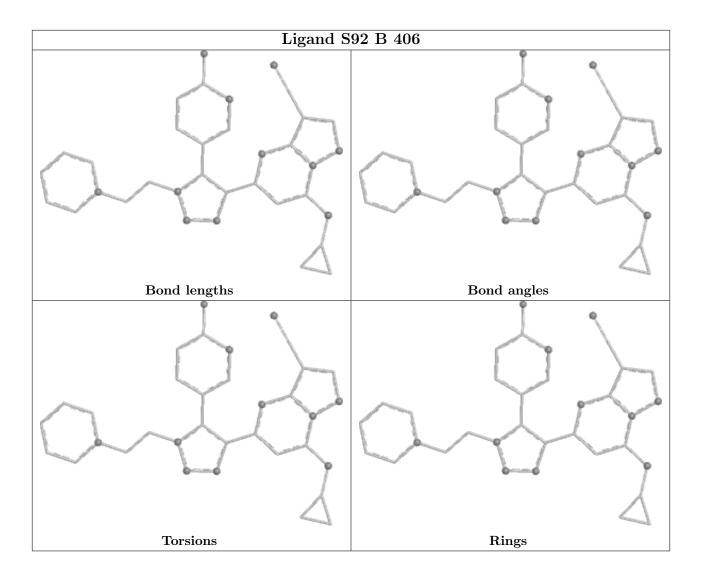
No monomer is involved in short contacts.

The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the validation Tables will also be included. For torsion angles, if less then 5% of the Mogul distribution of torsion angles is within 10 degrees of the torsion angle in question, then that torsion angle is considered an outlier. Any bond that is central to one or more torsion angles identified as an outlier by Mogul will be highlighted in the graph. For rings, the root-mean-square deviation (RMSD) between the ring in question and similar rings identified by Mogul is calculated over all ring torsion angles. If the average RMSD is greater than 60 degrees and the minimal RMSD between the ring in question and any Mogul-identified rings is also greater than 60 degrees, then that ring is considered an outlier. The outliers are highlighted in purple. The color gray indicates Mogul did not find sufficient equivalents in the CSD to analyse the geometry.









# 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,  $95^{th}$  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		$OWAB(A^2)$	Q<0.9	
1	A	331/342 (96%)	-0.15	1 (0%)	94 94	Ŀ	19, 31, 59, 78	1 (0%)
1	В	331/342 (96%)	-0.17	2 (0%)	89 89	)	18, 31, 54, 86	1 (0%)
All	All	662/684 (96%)	-0.16	3 (0%)	91 91		18, 31, 58, 86	2 (0%)

All (3) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	104	PRO	3.1
1	В	2	SER	2.8
1	В	271	ASP	2.4

### 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 monosaccharides 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,  $95^{th}$  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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q<0.9
4	EDO	A	418	4/4	0.57	0.27	62,64,68,69	0
4	EDO	В	419	4/4	0.64	0.31	55,57,60,61	0

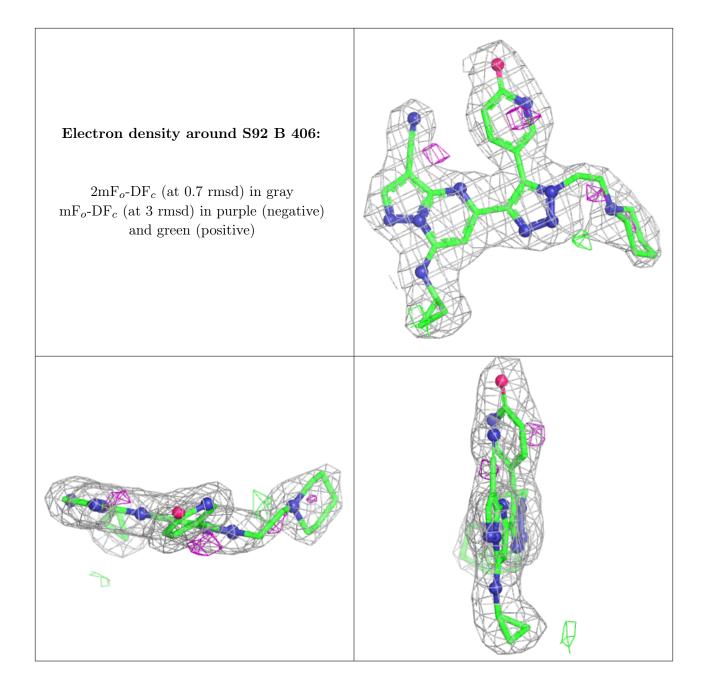


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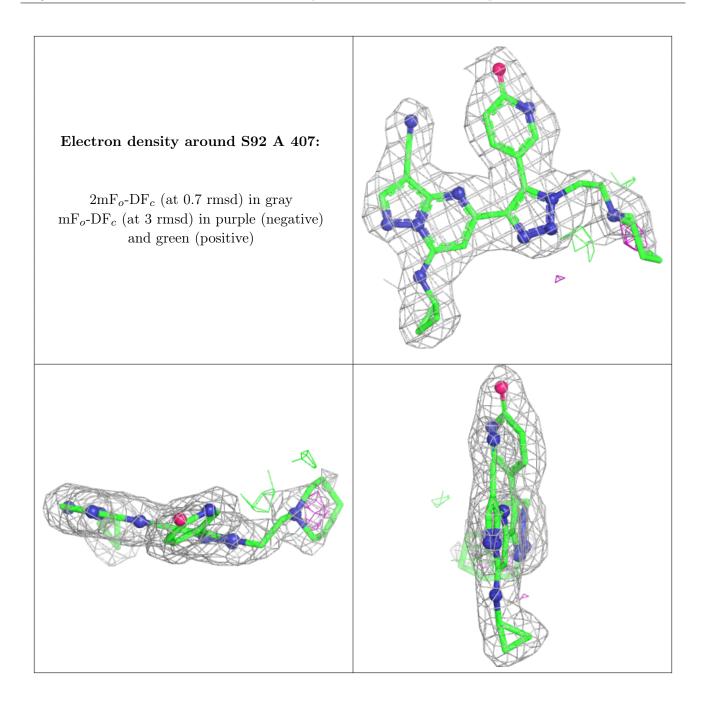
Mol	Type	Chain	Res	Atoms	RSCC	RSR	$\operatorname{B-factors}(\mathring{\mathbf{A}}^2)$	Q<0.9
4	EDO	В	416	4/4	0.66	0.56	60,61,61,61	0
4	EDO	В	413	4/4	0.67	0.25	55,57,59,60	0
4	EDO	В	418	4/4	0.70	0.29	59,60,61,61	0
4	EDO	A	411	4/4	0.70	0.38	62,63,64,66	0
4	EDO	A	410	4/4	0.72	0.36	61,61,62,63	0
4	EDO	A	416	4/4	0.73	0.24	60,61,61,63	0
4	EDO	В	411	4/4	0.74	0.18	65,68,72,74	0
4	EDO	В	414	4/4	0.78	0.16	65,66,69,71	0
4	EDO	В	409	4/4	0.78	0.28	47,47,48,50	0
4	EDO	A	413	4/4	0.82	0.46	46,51,54,56	0
4	EDO	A	417	4/4	0.83	0.30	57,60,63,65	0
4	EDO	A	408	4/4	0.83	0.39	60,60,61,61	0
4	EDO	В	415	4/4	0.83	0.17	50,52,55,55	0
2	SO4	В	405	5/5	0.85	0.52	106,108,109,109	0
4	EDO	В	417	4/4	0.85	0.20	$52,\!55,\!56,\!57$	0
4	EDO	A	414	4/4	0.87	0.23	55,57,59,61	0
4	EDO	A	412	4/4	0.88	0.15	57,57,58,58	0
4	EDO	В	412	4/4	0.89	0.22	47,50,52,54	0
4	EDO	В	408	4/4	0.89	0.16	49,50,50,53	0
4	EDO	A	415	4/4	0.89	0.19	53,53,55,56	0
2	SO4	В	404	5/5	0.89	0.28	84,84,85,86	0
4	EDO	A	409	4/4	0.92	0.25	35,38,44,51	0
2	SO4	В	402	5/5	0.92	0.27	113,113,114,114	0
4	EDO	В	410	4/4	0.92	0.13	41,42,47,48	0
2	SO4	В	403	5/5	0.95	0.12	106,106,107,107	0
2	SO4	A	406	5/5	0.95	0.21	76,78,80,80	0
4	EDO	В	407	4/4	0.95	0.16	35,38,41,47	0
2	SO4	A	404	5/5	0.96	0.18	51,57,59,62	0
2	SO4	В	401	5/5	0.96	0.15	58,58,60,61	0
2	SO4	A	403	5/5	0.97	0.14	59,60,60,62	0
2	SO4	A	402	5/5	0.97	0.13	56,58,61,62	0
3	S92	В	406	35/35	0.97	0.15	13,24,58,59	0
3	S92	A	407	35/35	0.98	0.14	18,26,65,66	0
2	SO4	A	405	5/5	0.98	0.12	70,72,73,74	0
2	SO4	A	401	5/5	0.99	0.16	48,49,51,54	0

The following is a graphical depiction of the model fit to experimental electron density of all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the geometry validation Tables will also be included. Each fit is shown from different orientation to approximate a three-dimensional view.









# 6.5 Other polymers (i)

There are no such residues in this entry.

